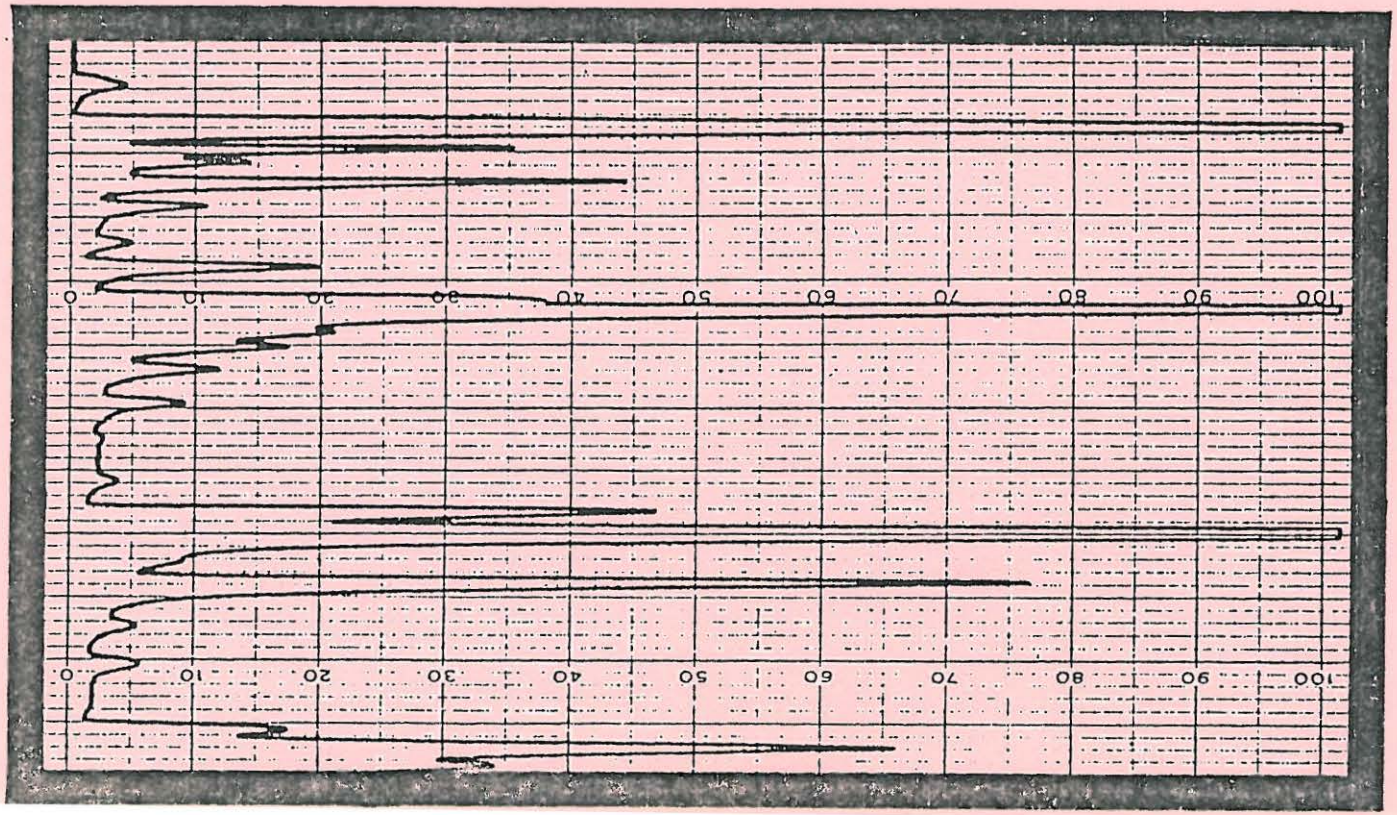
Arson Analysis Newsletter



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The AAN solicits contributions from forensic scientists, arson investigators, and interested parties which have some unique or routine analysis which helps in the identification of flammable liquid or explosive residues.

December 1979

ARSON ANALYSIS NEWSLETTER

VOLUME 3, NUMBER 5

COLLECTION AND DETERMINATION OF ACCELERANT VAPORS FROM ARSON DEBRIS

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THE COLLECTION AND DETERMINATION OF ACCELERANT VAPORS FROM ARSON DEBRIS

INTRODUCTION

The process of obtaining accelerant from arson debris is a vital step in the analysis; therefore, the technique is of much interest. Various methods have been proposed, such as heated head space (1), steam and vacuum distillation (2), and solvent extraction (3). Each technique has advantages and disadvantages (4,5). Consequently, a need for a simple and efficient method still exists.

The most efficient technique appears to be collection and concentration of vapors, but in some cases concentration of head space vapors is unsatisfactory. Then some alternative method is necessary for collection and concentration of sufficient samples for analysis. In these alternative techniques, the separation of the accelerant from the carrier is a relative cumbersome process. The literature indicates that concentration techniques such as vapor trapping on adsorbent materials such as activated charcoal or gas chromatographic column packing materials (6) are preferred over the use of dry ice-acetone baths and other cryogenic methods.

For some time, this laboratory has been using a gas purging adsorption elution technique that appears to have some advantages over present techniques. It evolved from a method that was developed by this laboratory for the collection and determination of ethylene glycol dinitrate, nitroglycerine and trinitrotoluene explosive vapors (7).

The debris is purged with heated nitrogen which is then drawn into an adsorption tube, where the accelerant is retained and concentrated. The adsorption tube is extracted with carbon disulfied (CS_2) and the extract is analyzed by gas chromatography (GC) and infrared spectroscopy (IR).

Other examples of adsorption elution techniques for concentration of hydrocarbon vapors also appear in the literature (8,9).

APPARATUS

Adsorbent and Collection Column Preparation.

The column for the collection of accelerant vapors consisted of a 5 3/4" long Kimble disposable pipet containing approximately 3" of 50-200 mesh activated coconut charcoal (Fisher Scientific Company) between glass wool plugs (Fig. 1). The charcoal was heated to 400°C for four hours, cooled in a desiccator, placed in an airtight container until used. Each new batch of activated charcoal was tested for residual hydrocarbons by analyzing the

CS_2 extracts of a blank column. The carbon disulfide used in the analysis was also analyzed for trace impurities by gas chromatography.

ACCELERANT VAPOR COLLECTION SYSTEM

The system used to collect and concentrate the accelerant vapors is shown schematically in Figures 1 and 2, and pictorially in Figures 3, 4 and 5. The system consists of 3 paint cans, tubing, a collection column, a tank of water-pumped nitrogen gas, two thermostatically controlled hot plates, and a vacuum pump capable of drawing 2 to 3 liters of nitrogen gas/minute through a filled collection tube.

EXPERIMENTAL

For experimental purposes, gasoline and fuel oil #2 were used because they cover the range of hydrocarbons found in most accelerants. Samples of 1-5 µl accelerant were added to cotton and placed in a sealed paint can. Holes the size of an eight-penny nail were made in the bottom of the can to allow nitrogen to flow through the sample. Two holes were made in the top of the lid, with a drill or tapered punch, that would accomodate a piece silicone rubber tubing into which were placed a collection column and a thermometer. Since silicone rubber tubing withstands heat and does not contaminate the sample, it was used throughout the system. The tapered end of the collection column was inserted

into the rubber tubing leading to the vacuum pump. This technique prevents the adsorbent from being pulled through the tube.

The system works as follows: Can A (Fig. 2) is used to preheat the nitrogen coming from the tank. It contains 6 lbs. of glass marbles which act as a heat exchanger. Gas enters can A through a ¼" diameter tubing and exits through 1/8" diameter tubing; this technique restricts the gas flow and increases the retention time of the gas in can A. The gas then enters can C through a ¼" diameter tubing. Can C contains aluminum foil that acts as a heat exchanger to provide a quantity of nitrogen that is further heated and available for purging the sample in can B. Should can C become contaminated from falling sample, the aluminum foil can be replaced and the can heated to expel any residual hydrocarbons. With extensive contamination, the can and foil should be replaced. The head of nitrogen at the interface of cans B and C should be sufficient to supply a quantity of nitrogen to fill can B and slightly exceed the flow created by the vacuum pump pulling nitrogen through the collection tube. Can B and C are fitted loosely to allow the slight excess of nitrogen to escape. Can C, which is open at the top, is joined to can B with several thicknesses of aluminum foil overlaid with masking tape, (Fig. 4). Three inch fiberglass insulation wrapped in foil is placed around the cans to prevent heat escape.

The hot plate under can A is maintained at 110°C, and the temperature of the hot plate under can C is varied to attain the desired temperature in can B. Nitrogen from the gas cylinder is applied to the system at a flow rate slightly greater than 3 liters/minute. With the pump on, heated nitrogen is pulled through the system, and accelerant vapors are adsorbed in the collection tube. Collection time can vary from 30 to 60 minutes depending on the type and quantity of debris. The collection tube is removed from the system and 2-3 ml CS_2 is added to the tube. Approximately 0.5 ml eluate is collected into a small screw cap glass vial. Approximately 5 µl of the carbon disulfide extract is chromatographed on a Perkin-Elmer gas chromatograph Model 900 using the following parameters:

Column length:	Nine feet
Coating:	5% diisodecyl phthalate with 5% Bentone 34.
Support:	Chromosorb W
Mesh:	80-100
Carrier gas:	Helium
Rate:	45 ml/minute
Pressures:	Inlet: 60 psig, Outlet: atmospheric
Temperatures:	Injector: 230°C Column: 50-150°C Detector: 230°C
Temperature Rates:	4°/minute
Initial hold:	6 minutes

The remainder of the eluate was evaporated on a salt plate and analyzed by IR spectroscopy.

RESULTS AND DISCUSSION

Since most accelerants encountered in arson cases have vapor pressures within the range of gasoline on the light hydrocarbon end and fuel oil #2 on the heavy hydrocarbon end, it was decided to use these fuels to demonstrate the feasibility of the method.

At ambient temperature (about 25°C) and a nitrogen flow of approximately 3 liters/minute through the system, sufficient quantities of gasoline vapor were collected on the adsorption column in fifteen minutes to obtain a chromatogram which could be identified.

After 45 minutes at the above conditions, the fuel oil #2 vapors collected gave a chromatogram that followed the pattern of fuel oil #2 up to about C_{12} hydrocarbons. A peak also appeared at about C_{15}. A procedure that was finally adopted was to start the vapor collection with the preheated nitrogen flowing through the system until the temperature in the sample can B, is in the range of 50 to 75°C. The collection continues for an additional fifteen minutes. In the temperature range of 50 to 75°C, sufficient quantities of accelerant were collected to obtain a chromatogram using a 1 µl sample of gasoline and 5 µl of fuel oil #2 that did not differ significantly from the original liquid. Figures 6 and 7 are examples of chromatograms that were obtained in this work.

Fractionation of a hydrocarbon mixture may occur if the temperature in the sample can is too low to volatilize the higher boiling components. It is most likely to occur when the debris contains an accelerant such as a fuel oil. The resulting chromatogram would then indicate a light hydrocarbon fraction.

Experiments conducted with water saturated gasoline samples showed that water vapor did not interfere with the collection of gasoline vapor.

Several advantages are apparent in this type of system. The material does not have to be transferred to another container. A physical separation from the carrier is unnecessary because nitrogen as the carrier separates automatically. Large amounts of solvents are also unnecessary. Approximately 2 to 3 ml CS_2 is all that is needed to elute the adsorbed accelerant from subsequent analysis. Consequently, repeated injections can be made into the gas chromatograph to achieve optimum attentuation. If an arson case consists of many items (cans), the vapors can be collected on the adsorption columns one day and analyzed at a convenient time. Once the vapors are adsorbed or collected, they are not depleted from the adsorbent until they are eluted. Originally, the can containing the debris was placed directly on a hot plate and room air was drawn through the debris. However, it was decided to use nitrogen gas to prevent oxidation of the debris and to assure that contamination vapors from the atmosphere did not enter the collection tube.

The sensitivity of the method requires that the debris that is sent to the laboratory for arson analysis be examined before analysis, because materials such as burned polyurethane foam, burned pine wood, and other types of burned organic products generate peaks which can be easily mistaken for accelerant peaks. A control sample that is known not to contain accelerant should be requested. The control sample is partially burned, placed in a sealed can, and analyzed for comparison purposes. If a control sample is not available, a sample of the debris is thoroughly heated to expel residual vapors and then treated as a control to determine if peaks are generated from the material itself.

CONCLUSION

Designs other than the one presented can be devised to obtain similar results. However, the principle of using gas as a purging medium along with adsorption and elution as a method of collecting vapors has many advantages over existing methods.

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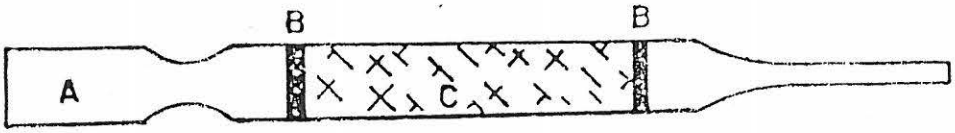

FIG. 1 - Vapor collection column: A-disposable glass pipet; B-glass wool; and C-activated charcoal, 50-200 mesh.

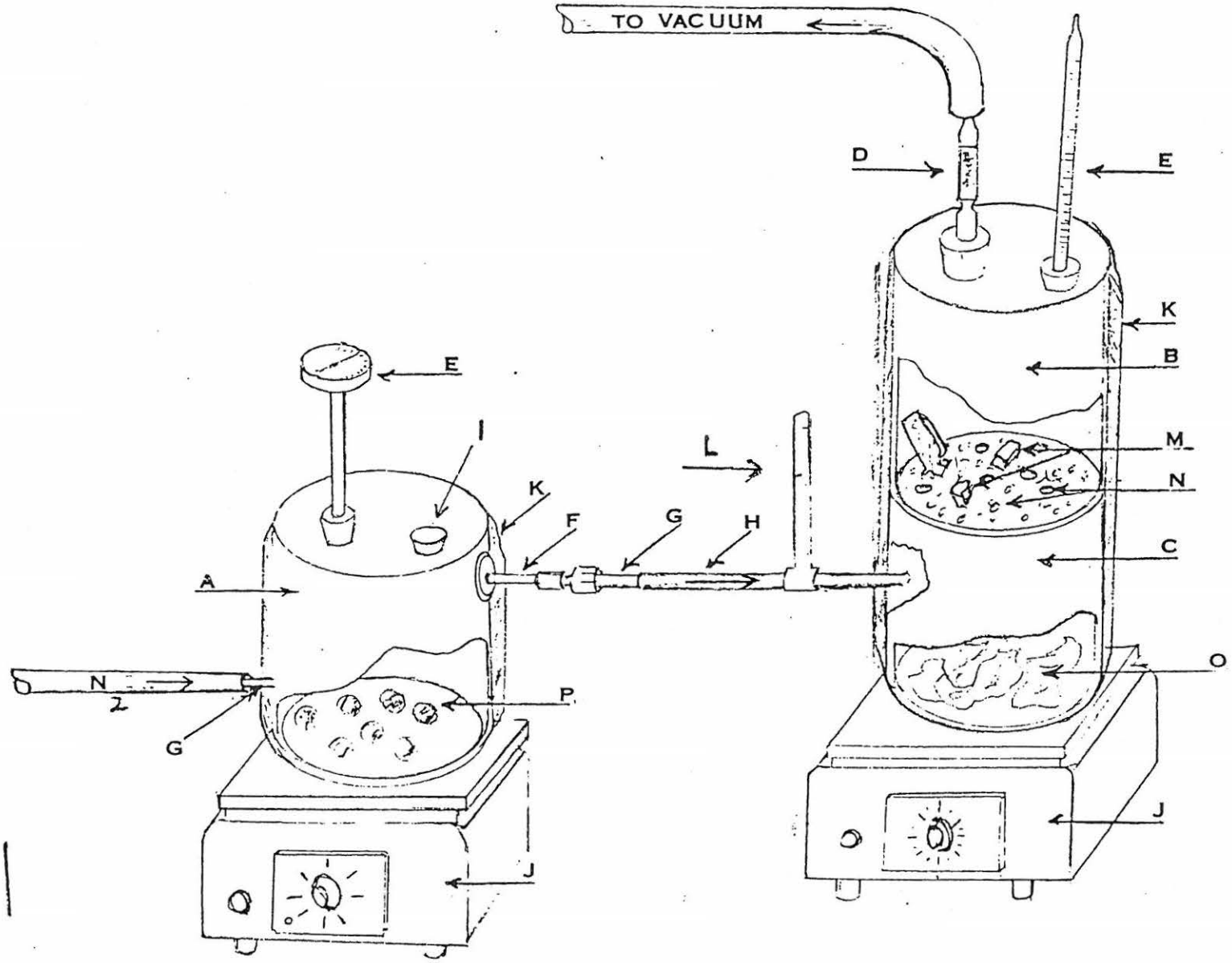

FIG. 2 - Accelerant vapor collection system: A- gallon paint can; B- unused metal container (paint can in pint, quart, or gallon size); C- unused metal container (paint can in pint, quart, or gallon size); D- collection column; E- thermometer; F- 1/8" copper tubing; G- ¼" copper tubing; H- silicone rubber tubing; I- rubber stopper; J- hot plate; K- insulation; L- flowmeter; M- debris; N- 8 penny nail holes; O- aluminum foil; P-glass marbles.

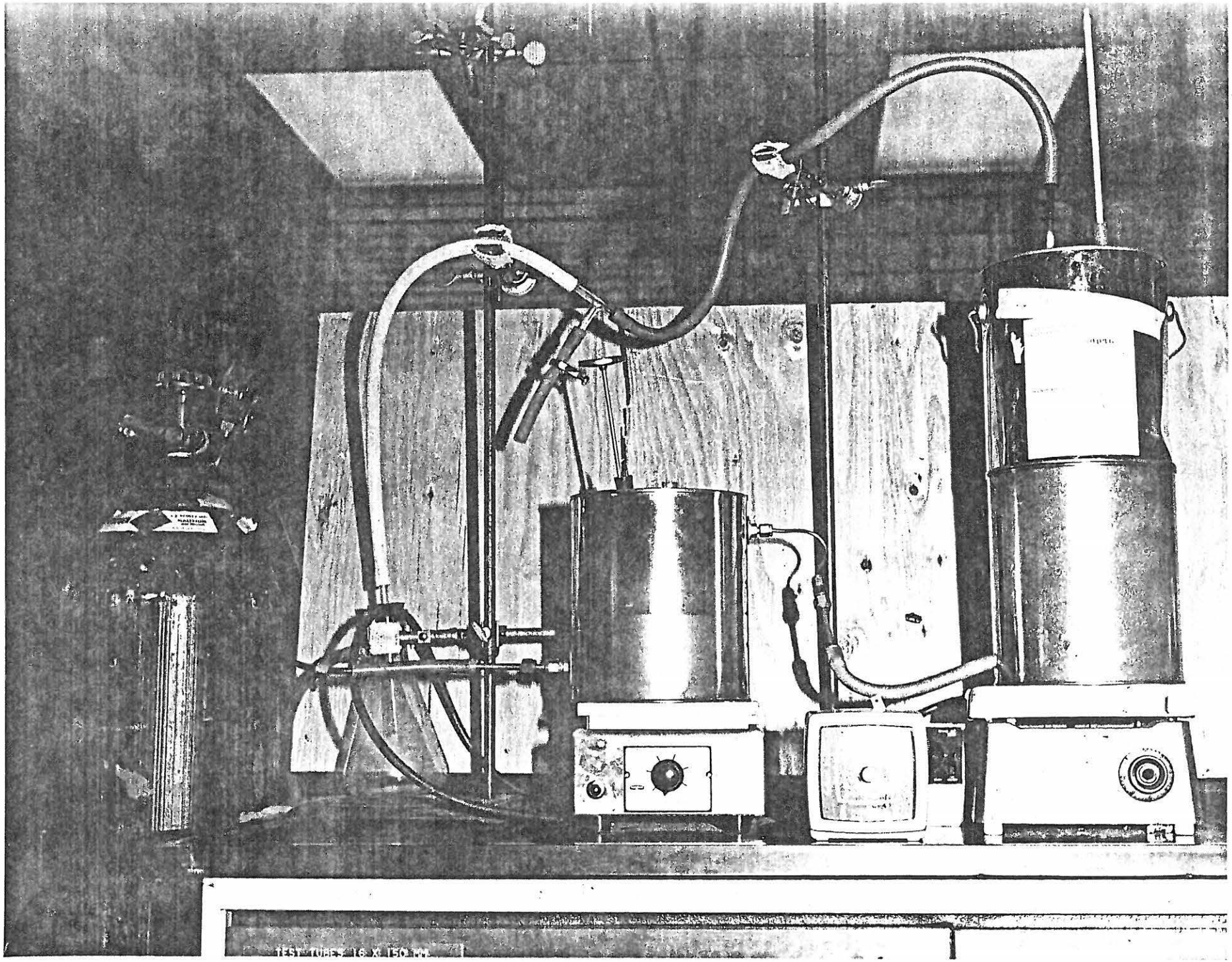

FIG. 3 - A photograph of the apparatus for the collection of accelerant vapors, showing the initial arrangement of the cans.

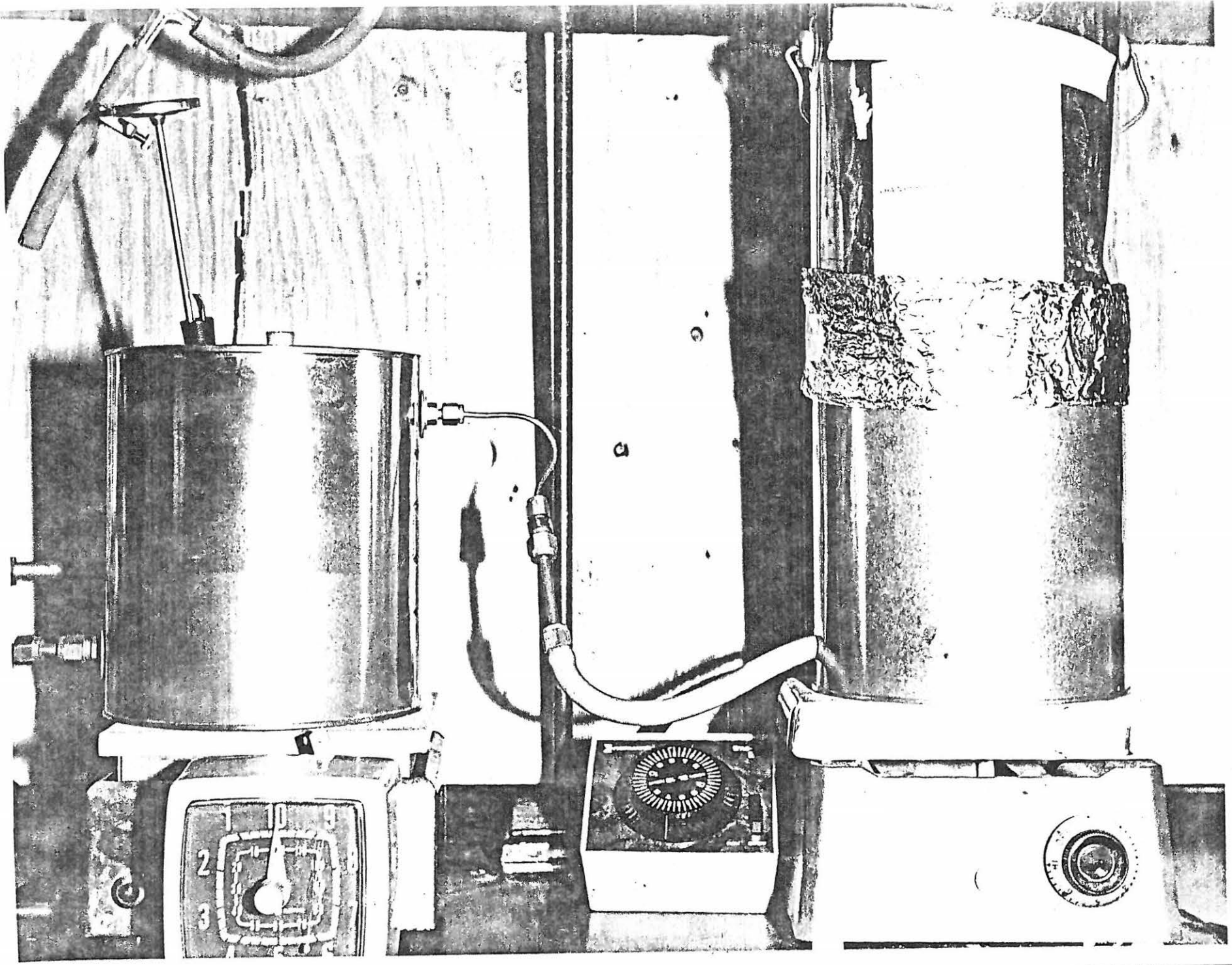

FIG. 4 - A photograph showing the joining of cans C and B with aluminum foil.

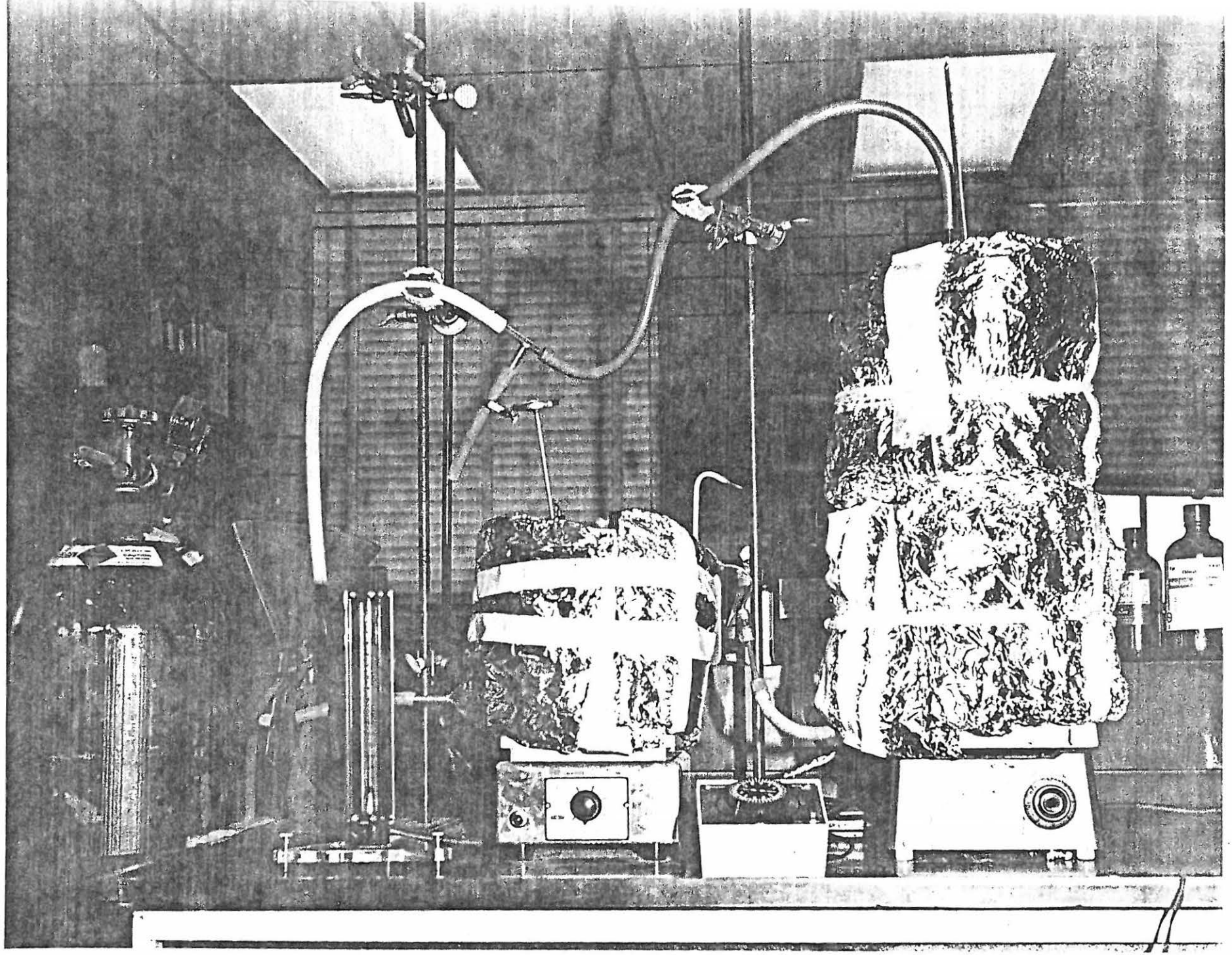

FIG. 5 - A photograph showing the final arrangement of the apparatus for the collection of accelerant vapors.

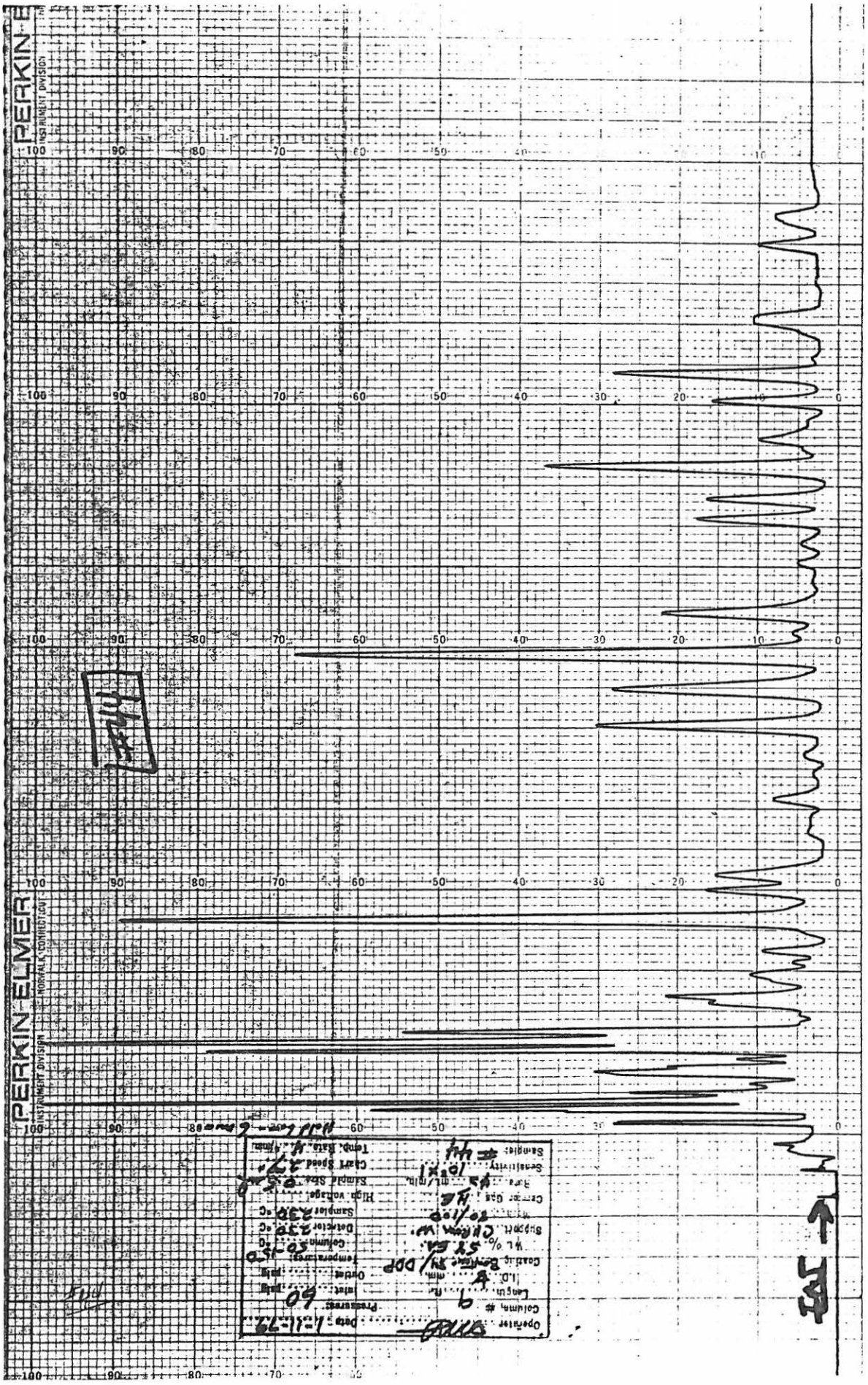

FIG. 6 - A gas chromatogram of gasoline vapors.

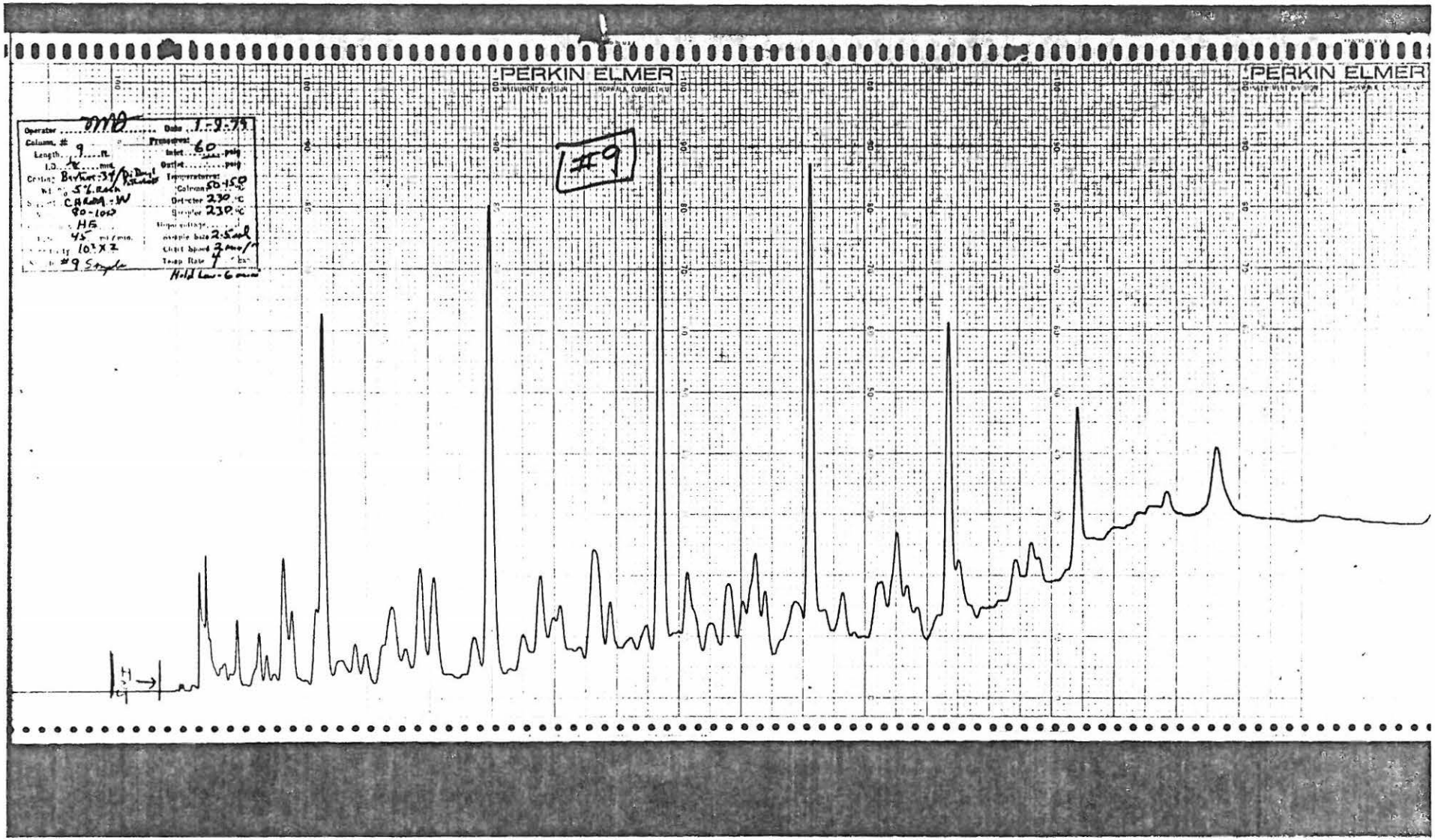

FIG. 7 - A gas chromatogram of fuel oil #2 vapors.

Evaluation of a Portable Gas Chromatograph for Arson Analysis: Column Selection

by
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Abstract:

A series of nine column packings were tested using a GOW-MAC "portable" gas chromatograph equipped with thermal conductivity detector. Gasoline, gum spirits of turpentine, and fuel oil, flammable liquids commonly found in arson debris, were chromatographed. It appears from the results that the SP2100/1.75% Bentone 34 column packing yielded the more favorable chromatograms for interpretation using (5) five microliter injections.

Introduction

The use of gas chromatography as a technique for identification of flammable liquids has been reported by several authors.[1-6] The Aerospace Corporation report of 1976 prepared for LEAA discusses the use of gas chromatography for detection of arson accelerants.[7] Portable field gas chromatographs are being used in Ohio and West Virginia in arson investigation.[8] The Aerospace report suggests several criteria for a suitable gas chromatographic detector in arson investigations, namely: sensitivity, portability, moderate cost, and simplicity.[9]

The main consideration of this report is the sensitivity (response to flammable liquids) of a portable gas chromatograph with the emphasis on column packing selection. The gas chromatograph used in this report was equipped with a thermal conductivity detector, which is generally less sensitive than a flame ionization detector; however, the thermal conductivity detector is generally

less expensive and requires only one compressed gas, the carrier gas, for operation. Due to the use of a thermal conductivity detector, it was necessary to inject liquid samples for favorable chromatograms: injection of vapor samples yielded, in nearly all cases, unfavorable chromatograms for interpretation.

Instrumentation

GOW-MAC series 150 Thermal Conductivity detector gas chromatograph equipped with Linear recorder and helium (.995%) carrier gas with Hydro-Purge on line filter.

Operating Parameters

The thermal conductivity current was set at 200 DC milliamperes. The flow rate of the carrier gas was set at 30 milliliters per minute. Column oven temperatures used were 100 and 150^{o}C isothermal. Chart recorder speeds were .5 and 1.0 centimeters per minute.

Columns Selected

The nine column packings were selected mainly on the basis of gas chromatographic literature on columns recommended for hydrocarbons, and to a lesser extent on cost; several of the packings selected are stock items and generally less expensive.

The nine column packings selected were:

1. 5% SP-2100 plus 1.75% Bentone 34 on 100/120 mesh Supelcoport.

2. 7% THEED on 100/120 mesh Chromosorb P.

3. 5% Carbowax 20M on 60/80 mesh Chromosorb WAW.

4. 5% Dexsil on 80/100 mesh Chromosorb WAW.

5. Porapak QS, 80/100 mesh.

6. Chromosorb 102, 80/100 mesh.

7. Tenax GC, 60/80 mesh.

8. 5% SE 30 on 100/120 mesh Chromosorb WAW, DMCS.

9. Durapak, n-octane on 120/150 mesh Poracil C.

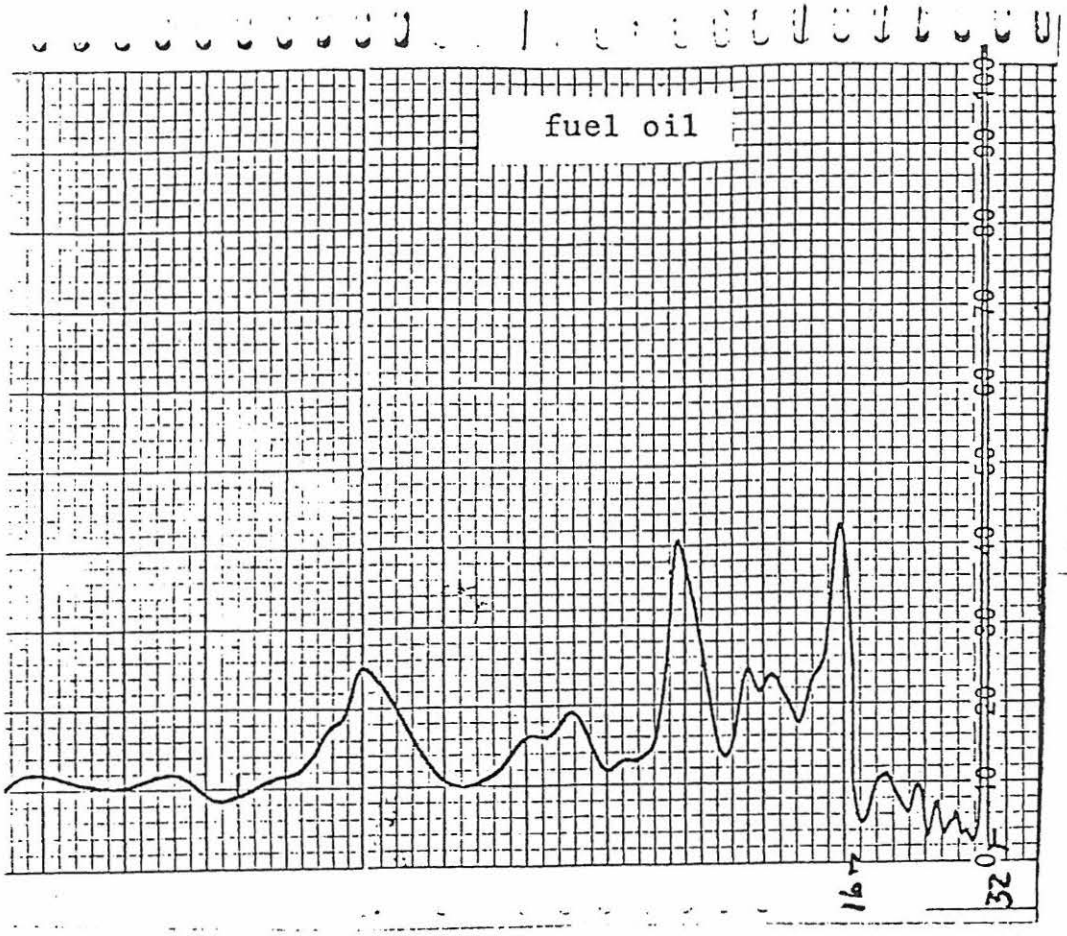
All of the columns were 6 foot by 1/8 inch diameter stainless steel. These columns are generally stock columns. All columns were conditioned for approximately 2 hours.

Samples

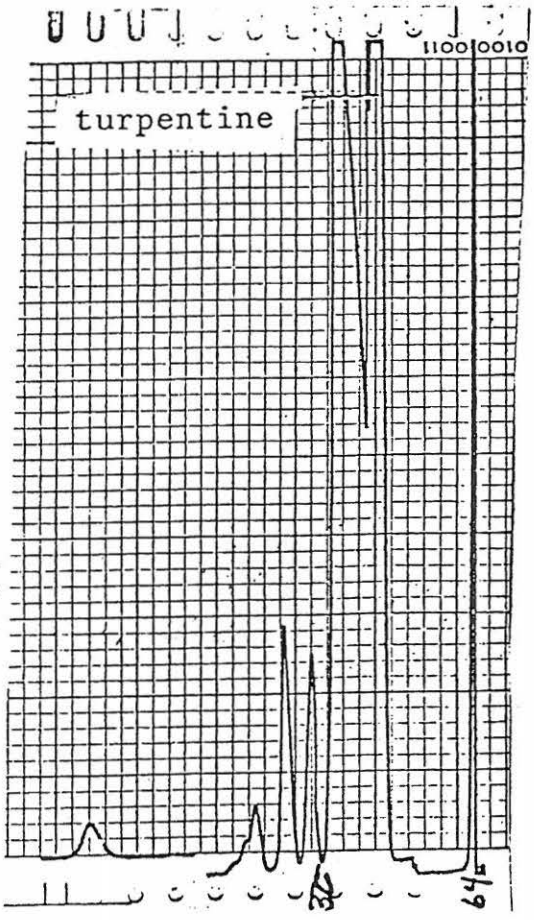
The samples tested were gasoline, gum spirits of turpentine, and fuel oil, since these flammable liquids exhibit a wide range of hydrocarbon components and boiling temperatures.

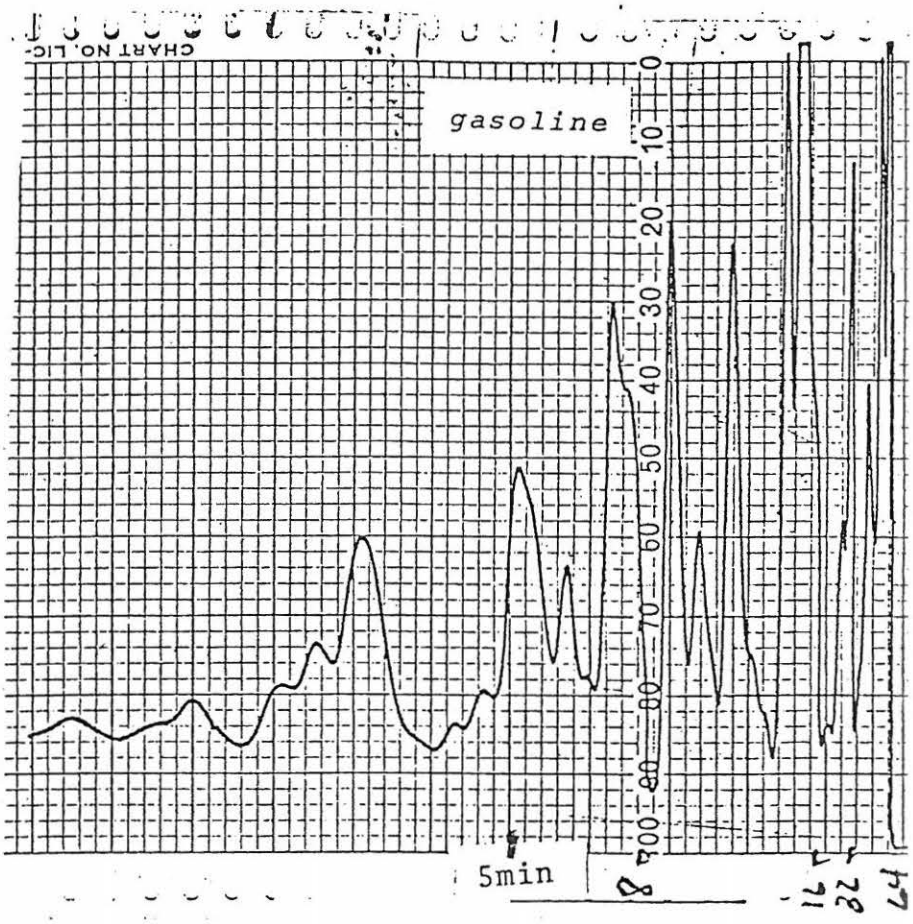
Results

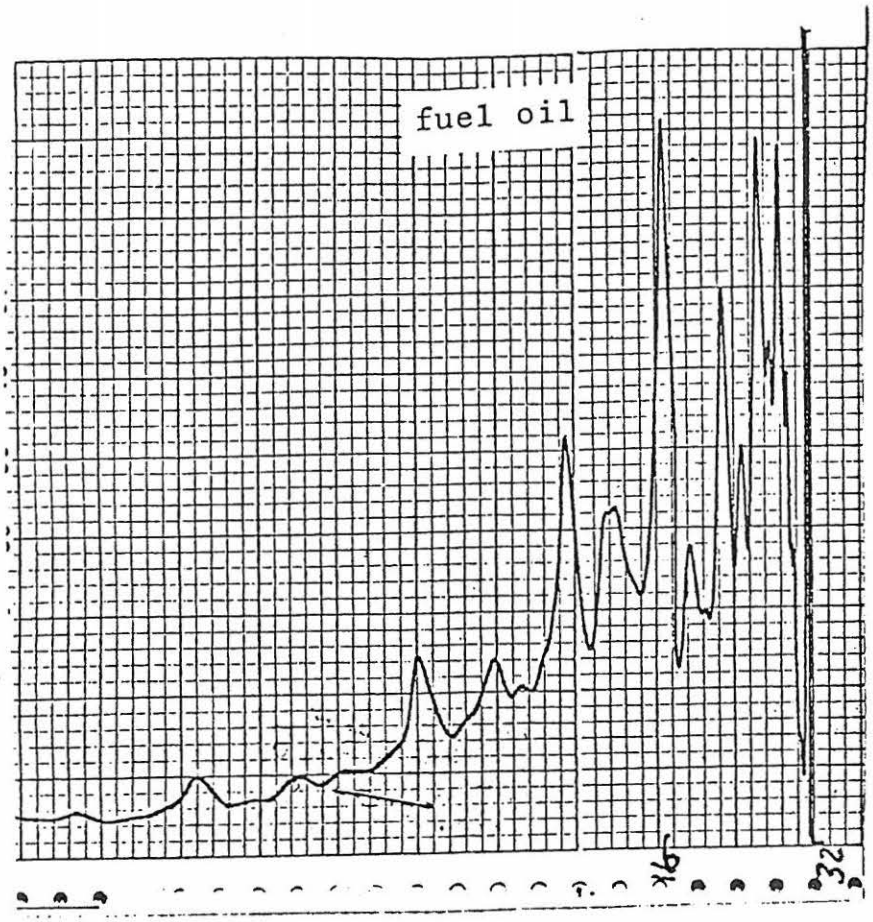
The following chromatograms reflect the column packings, oven temperature (isothermal), quantity injected, chart speed and results for the flammable liquids tested. For virtually all samples, attenuation (sensitivity) changes were necessary for suitable chromatograms for interpretation.



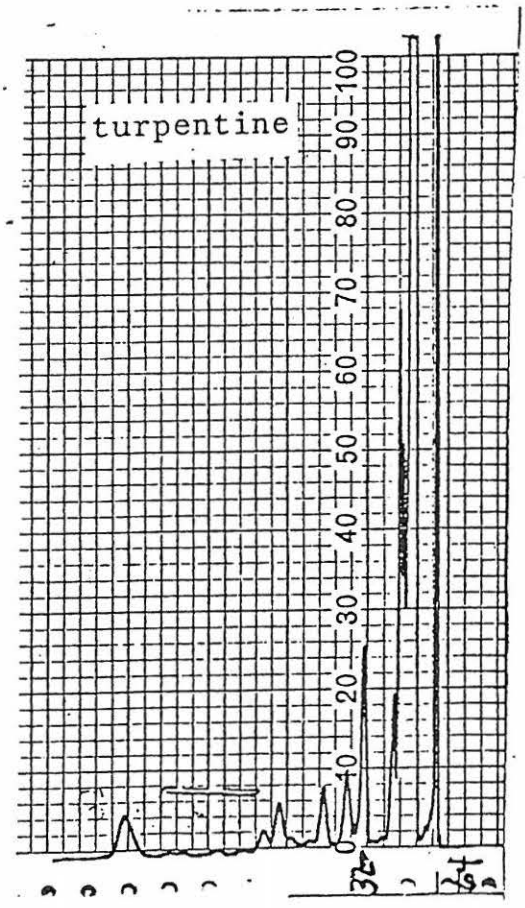
5% SP 2100/1.75% Bentone 34
100°C Chart 1 cm/min
5 microliter

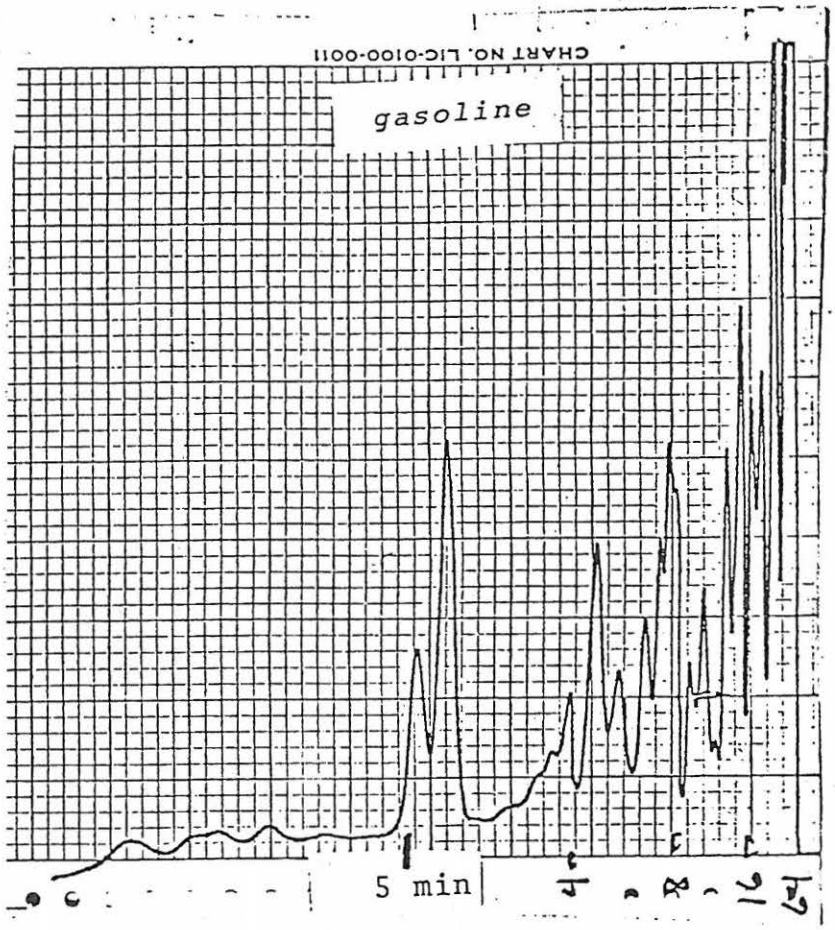


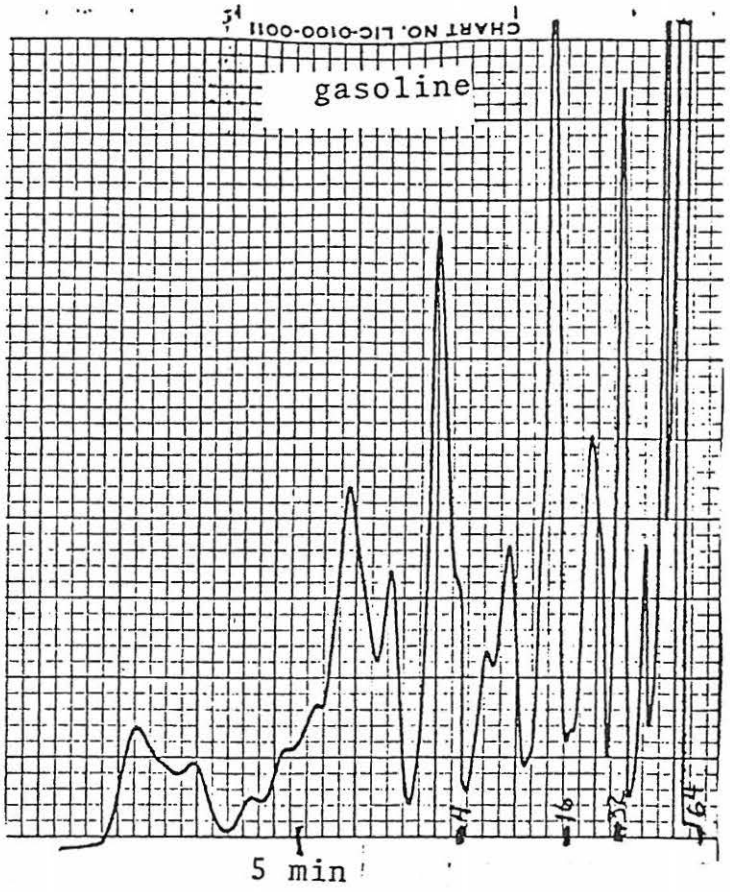




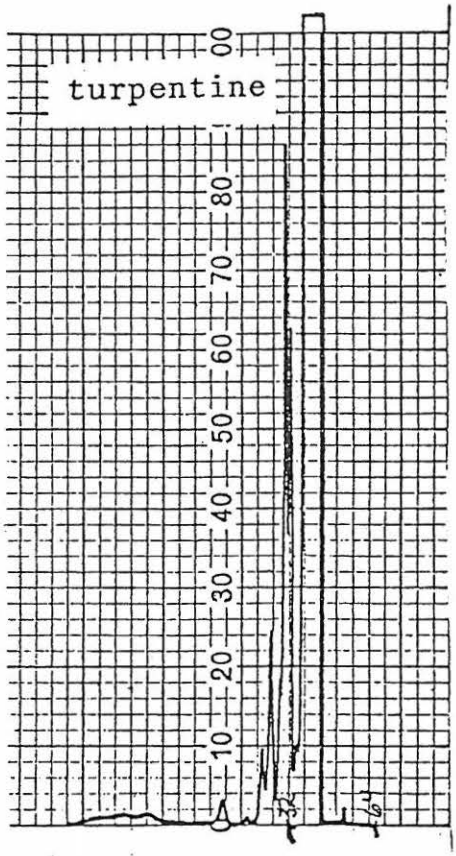
5% SP 2100/ 1.75% Bentone 34
150°C Chart 1cm/min
5 microliter

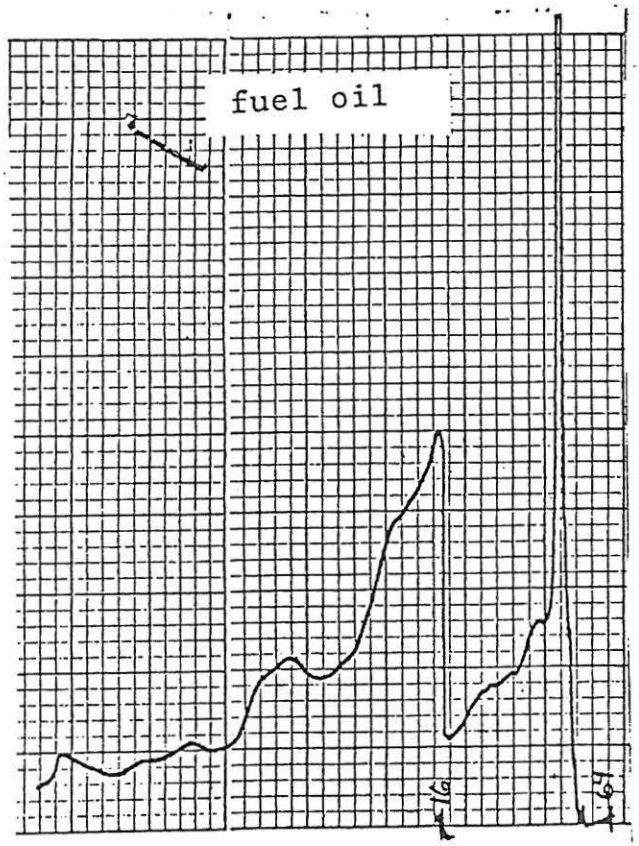


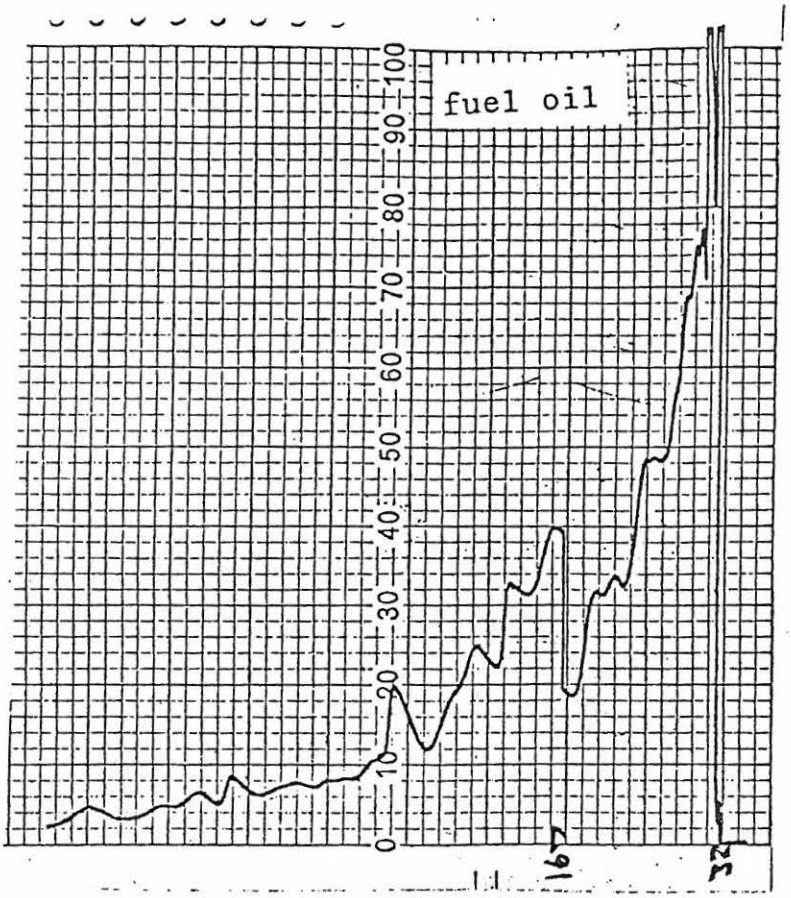




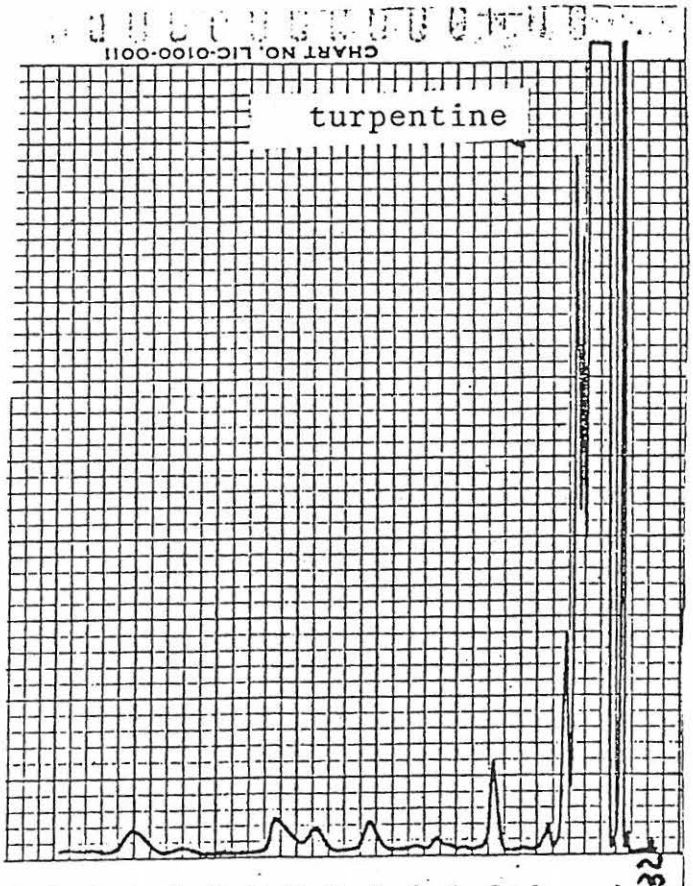
7% THEED
100°C Chart 1cm/min
5 microliter

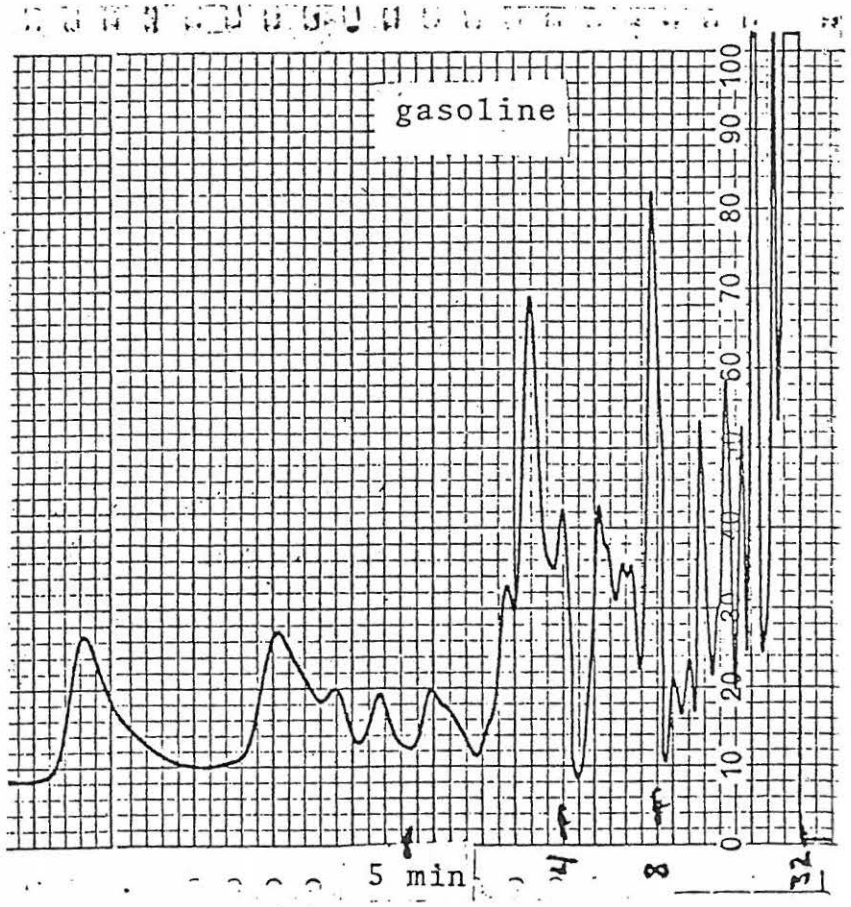


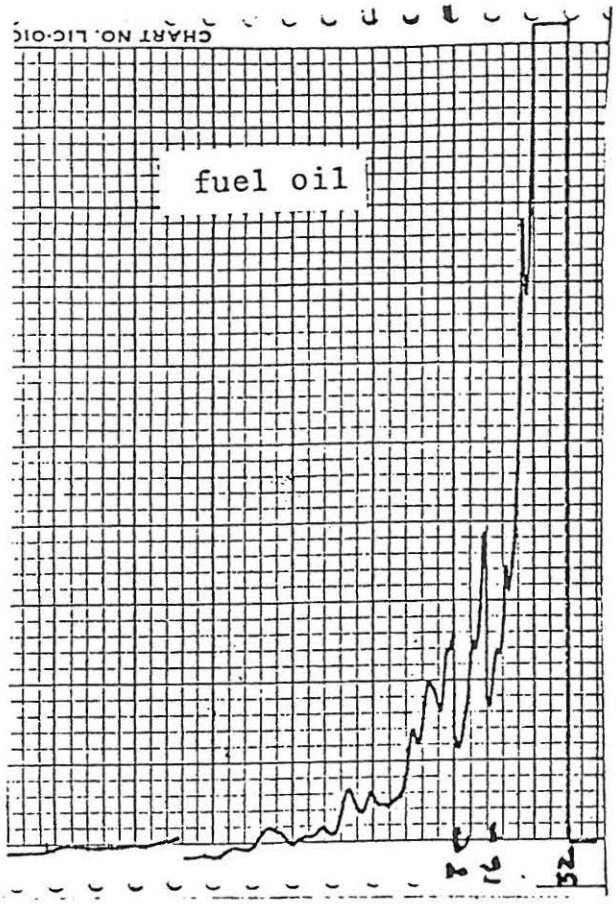




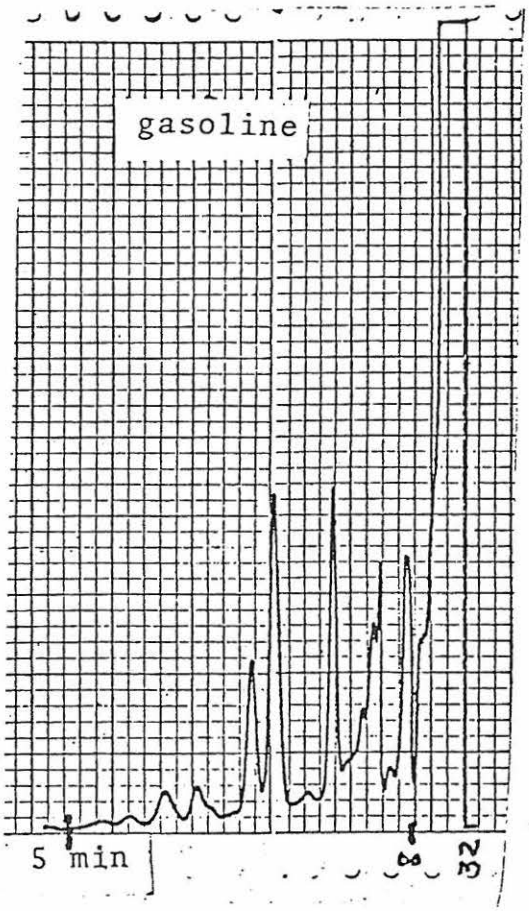
5% Carbowax 20M
100°C Chart 1cm/min
5 microliter

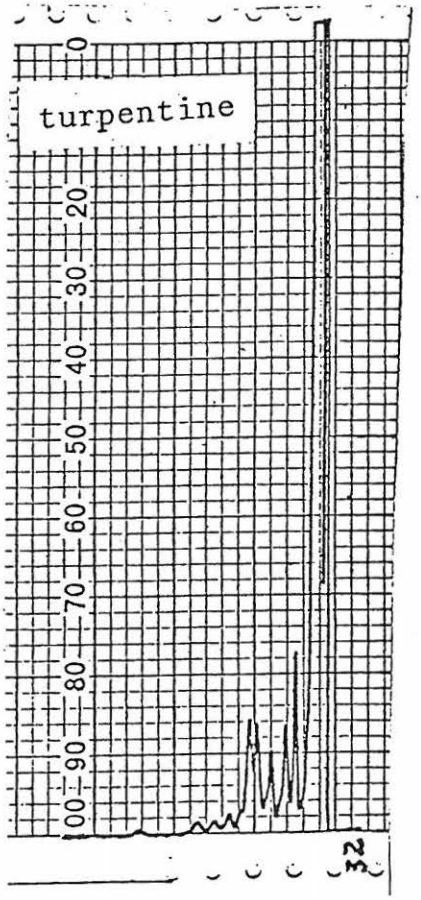


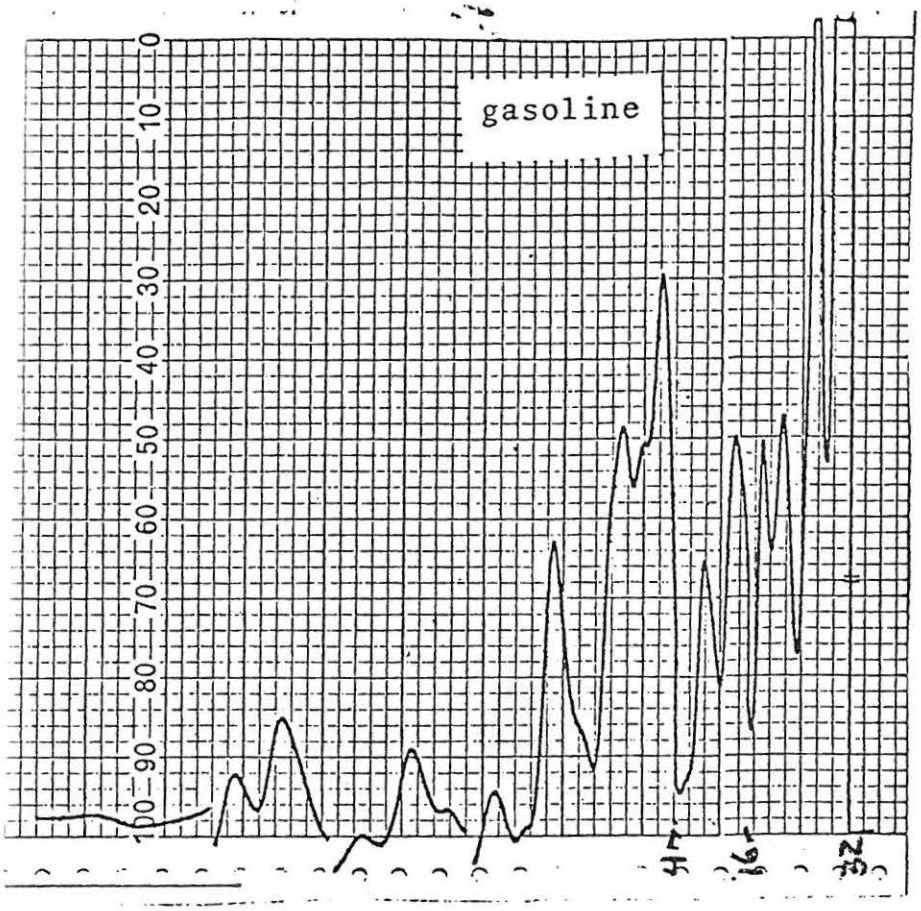


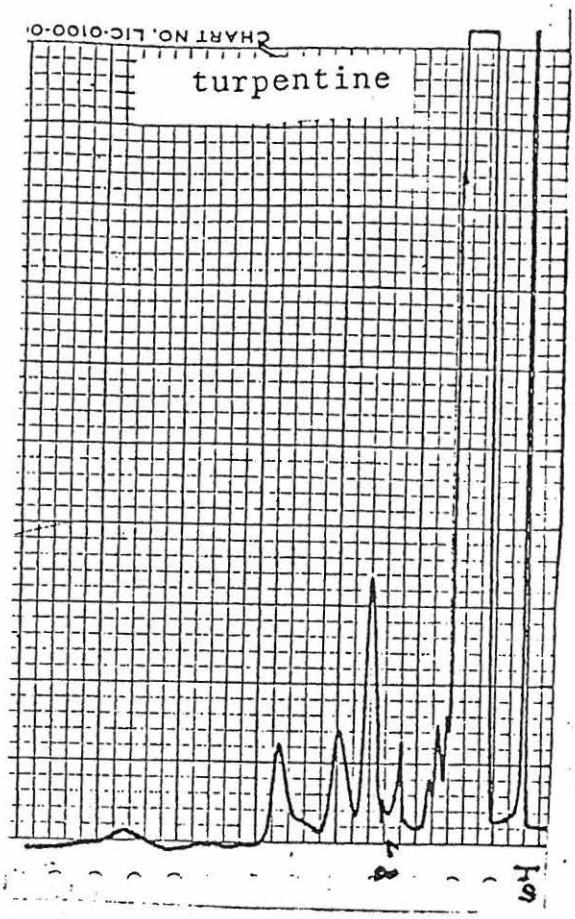


5% Carbowax 20M
150°C Chart 1cm/min
5 microliter

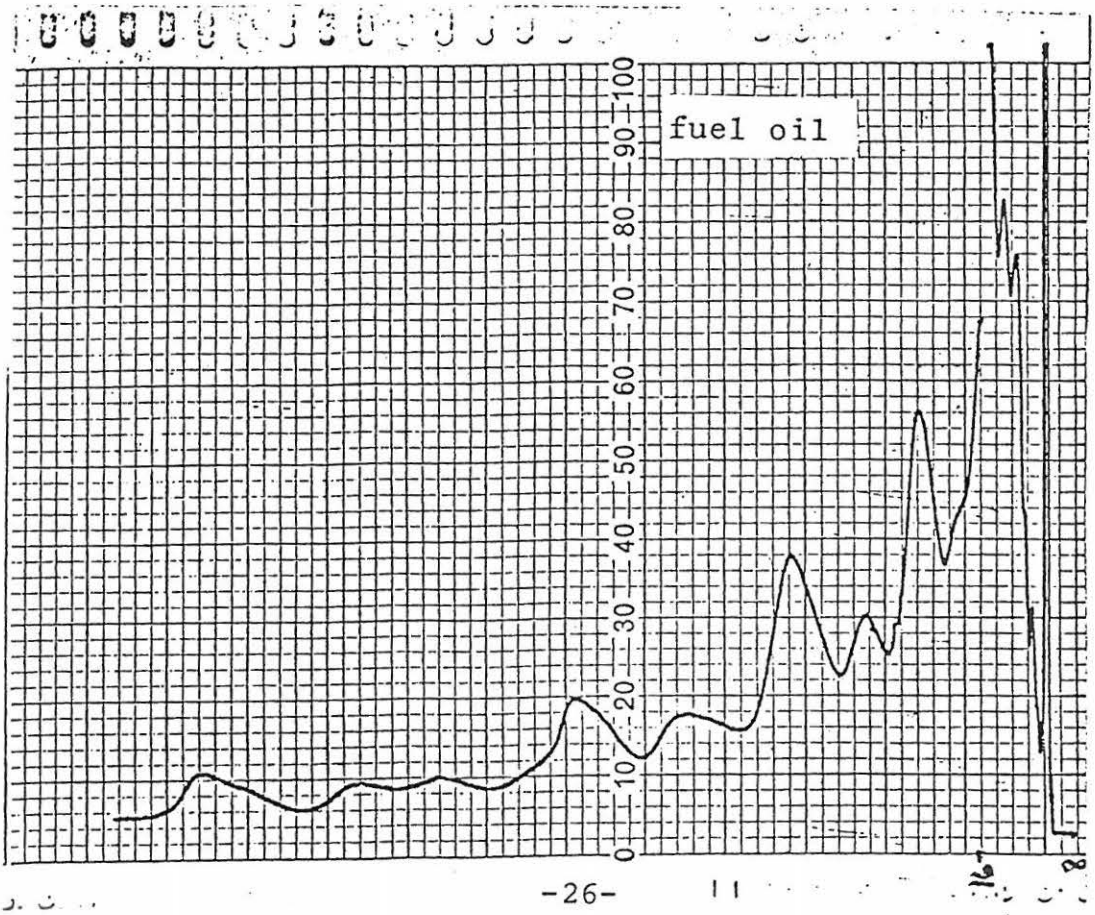


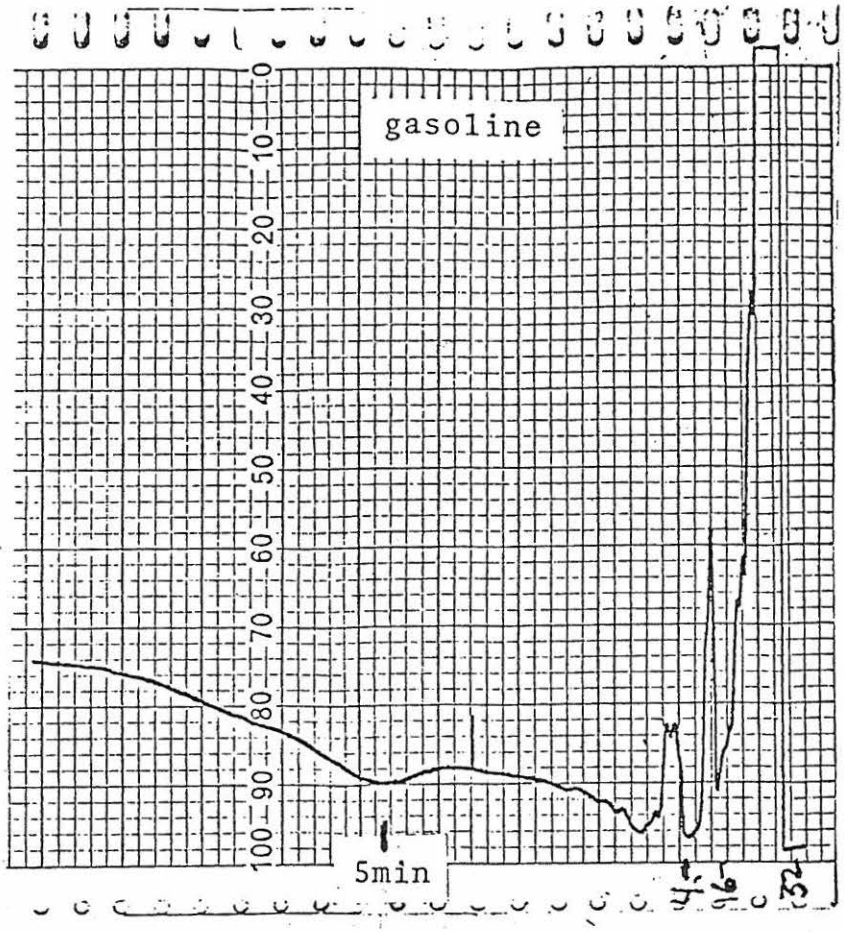




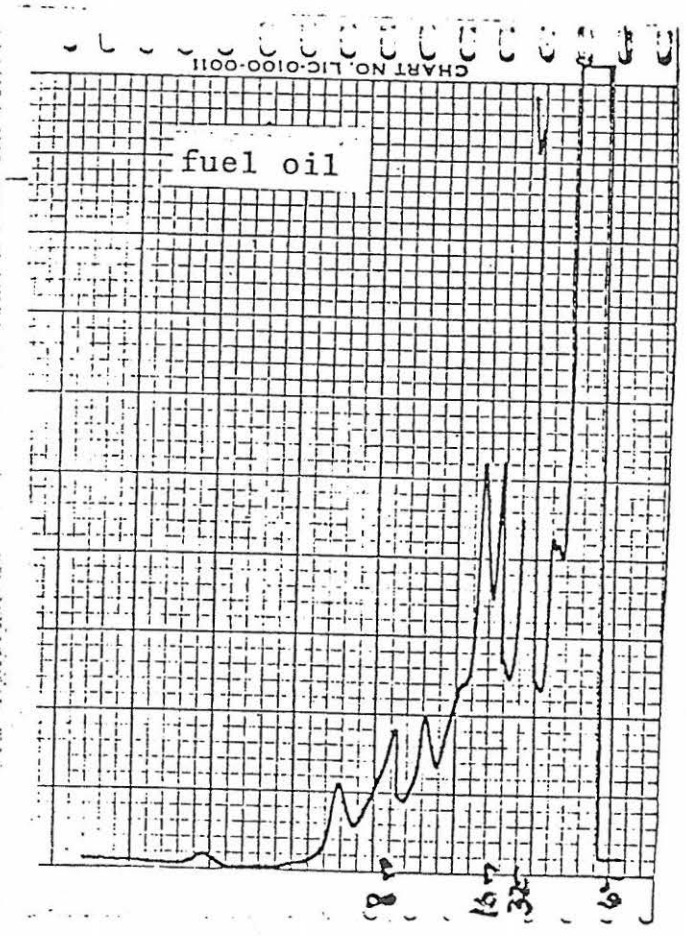


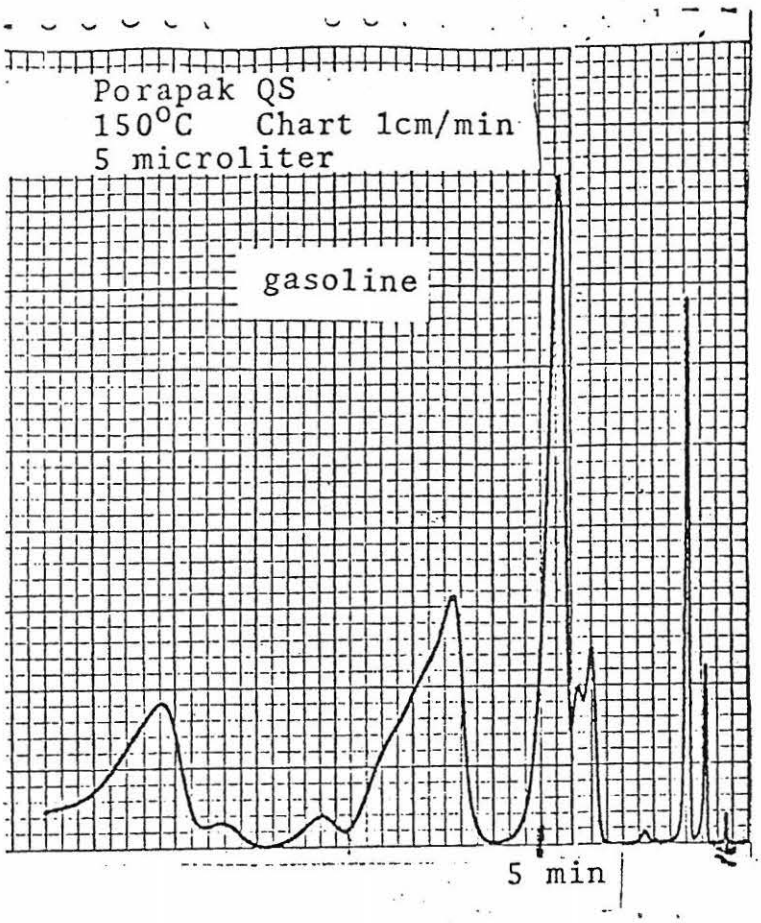
5% Dexsil
100°C Chart 1cm/min
5 microliter

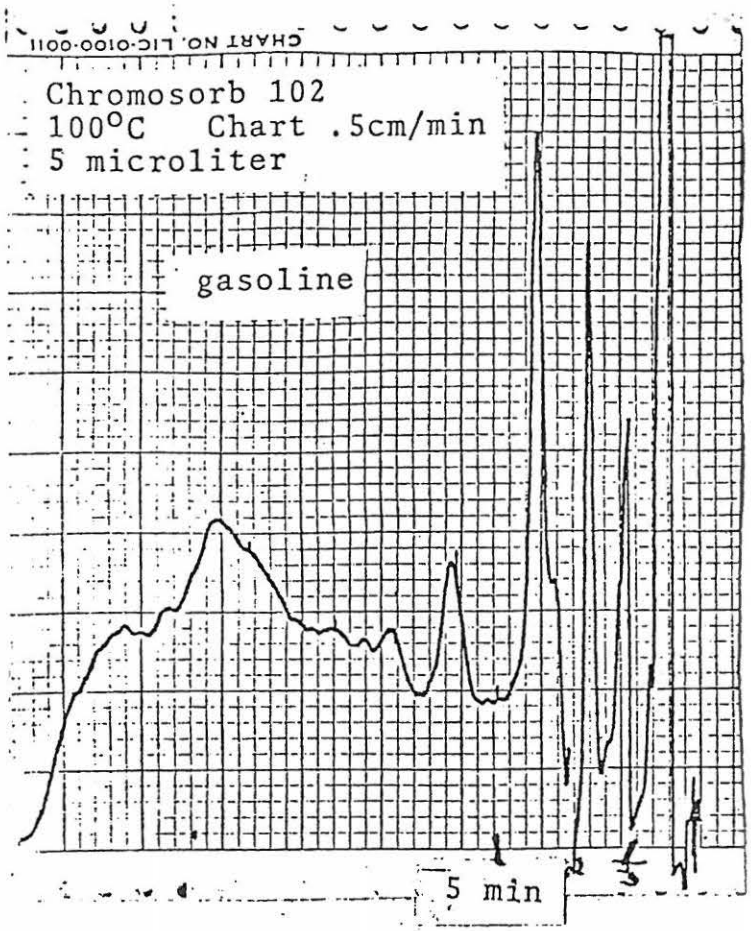


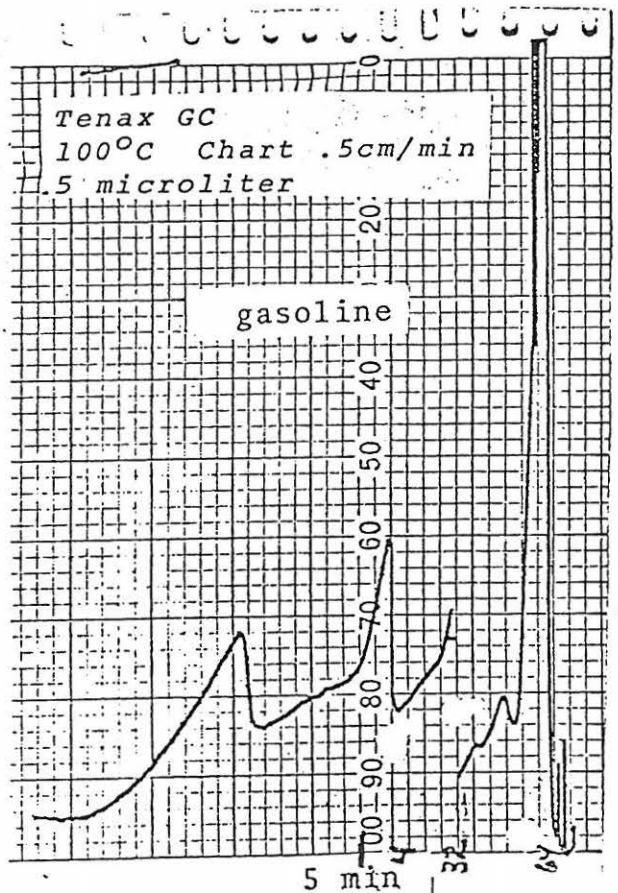


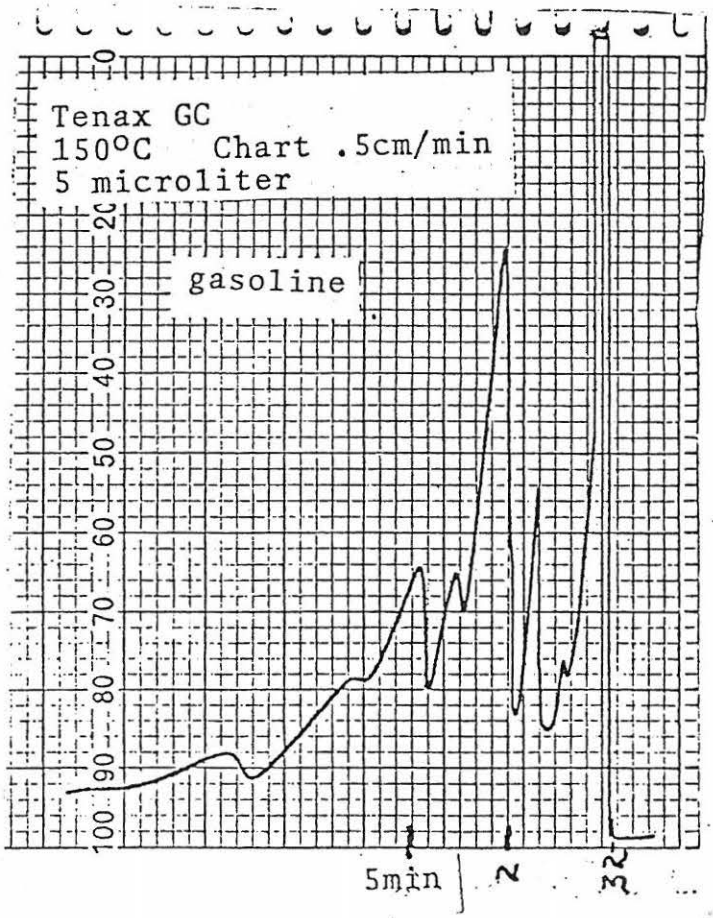
5% Dexsil
150°C Chart 1cm/min
5 microliter

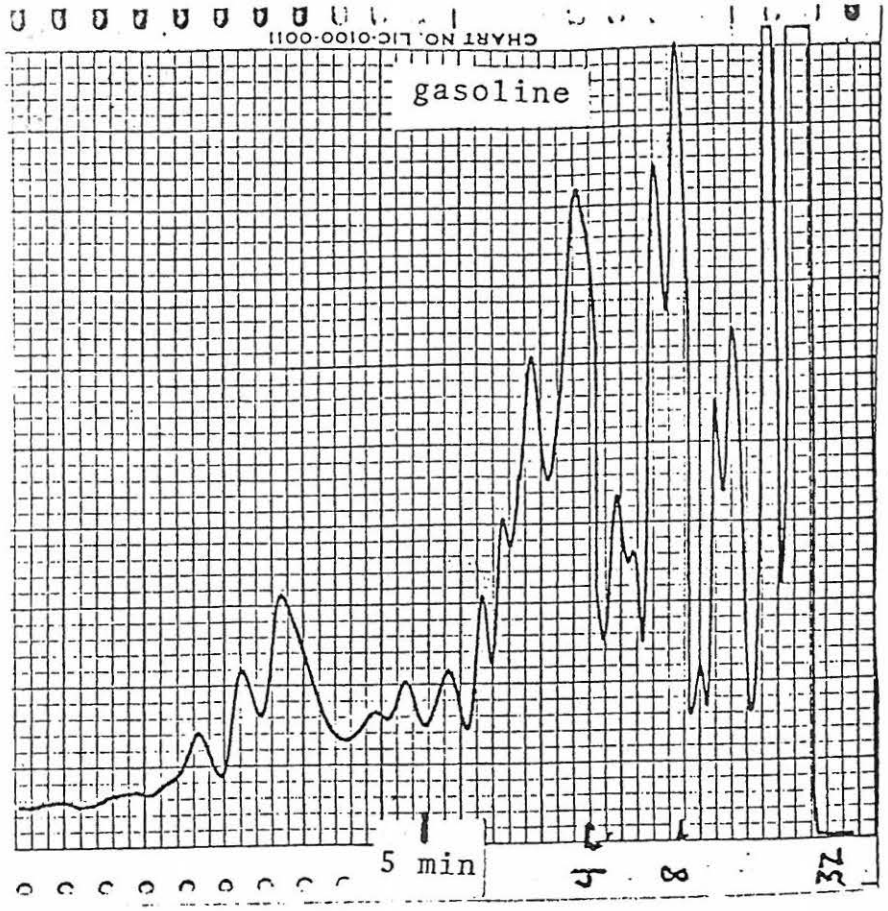



Porapak QS
150°C Chart 1cm/min
5 microliter
gasoline
5 min

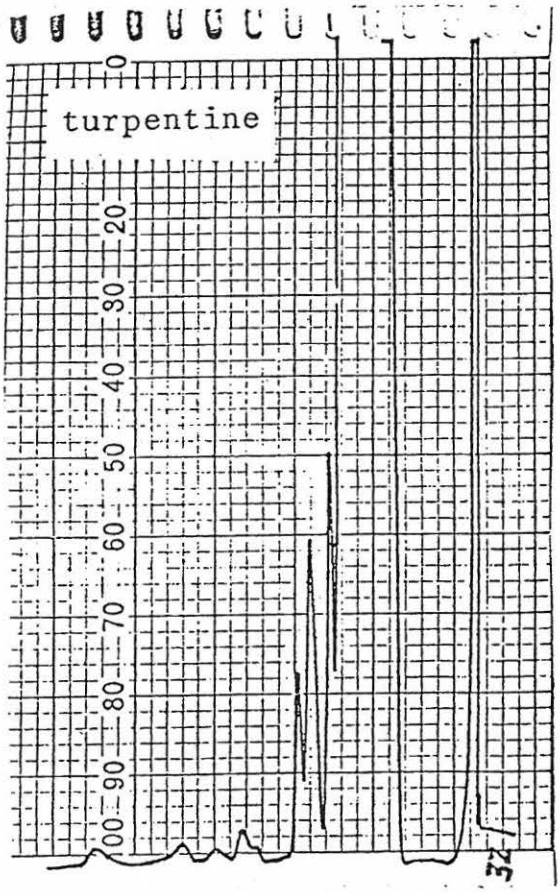

Chromosorb 102
100°C Chart .5cm/min
5 microliter
gasoline
5 min

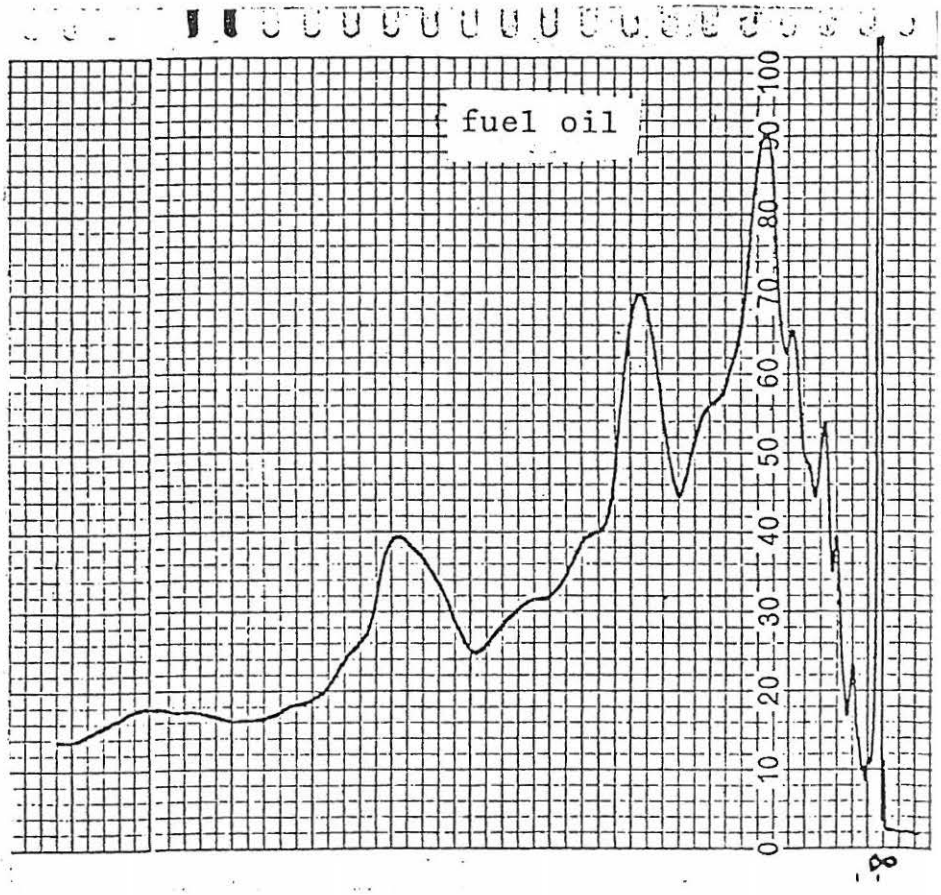

Tenax GC
100°C Chart .5cm/min
.5 microliter
gasoline
5 min

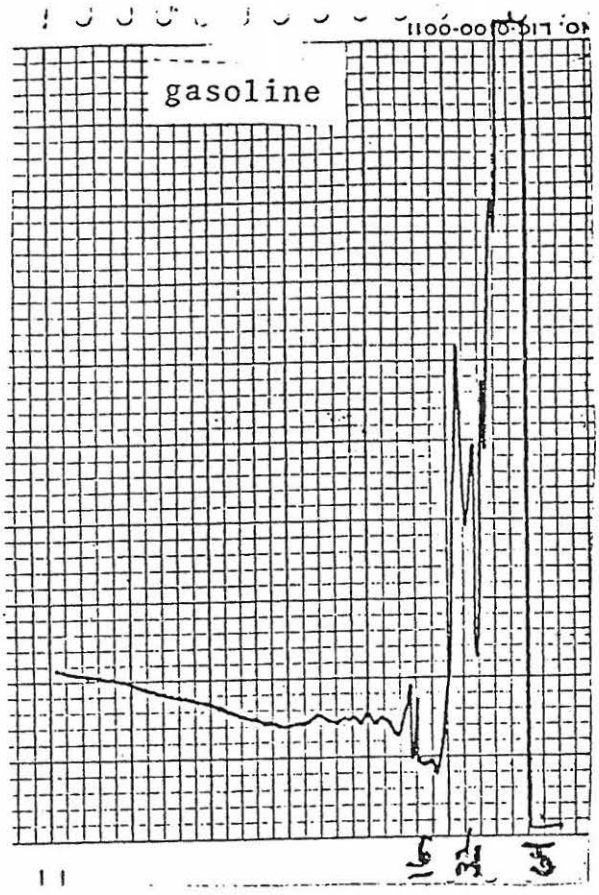

Tenax GC
150°C Chart .5cm/min
5 microliter
gasoline
5min



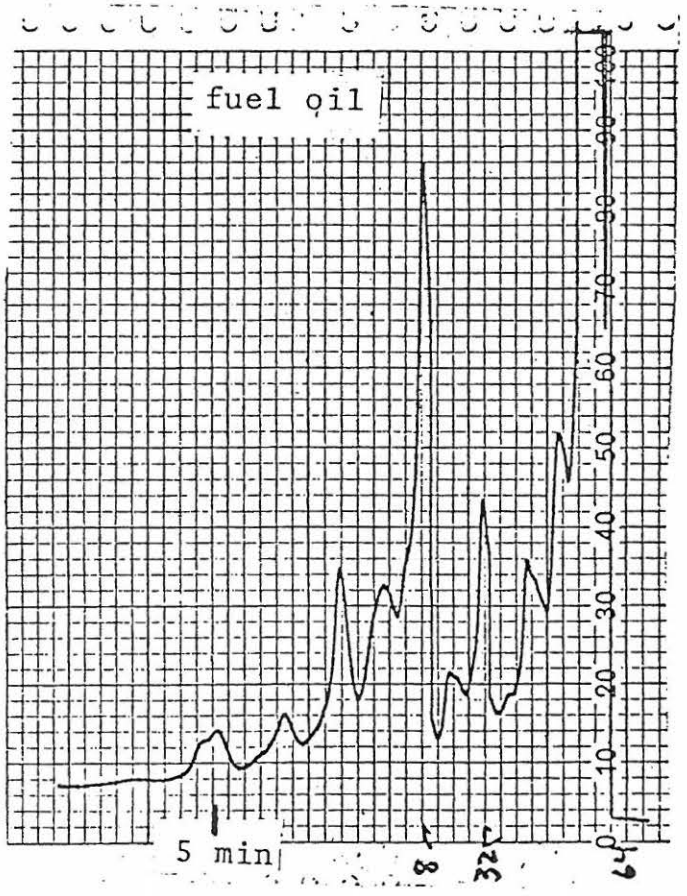
SE-30
100°C Chart 1cm/min
5 microliter

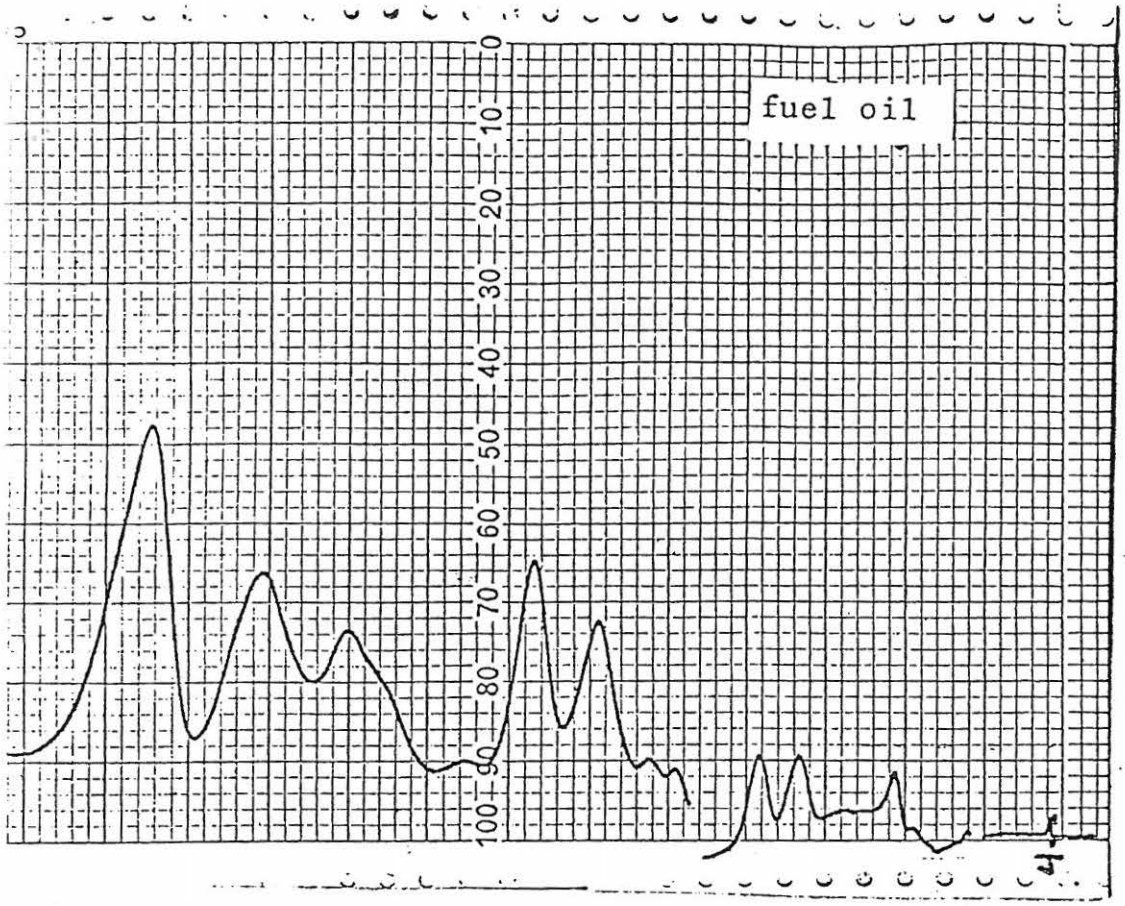




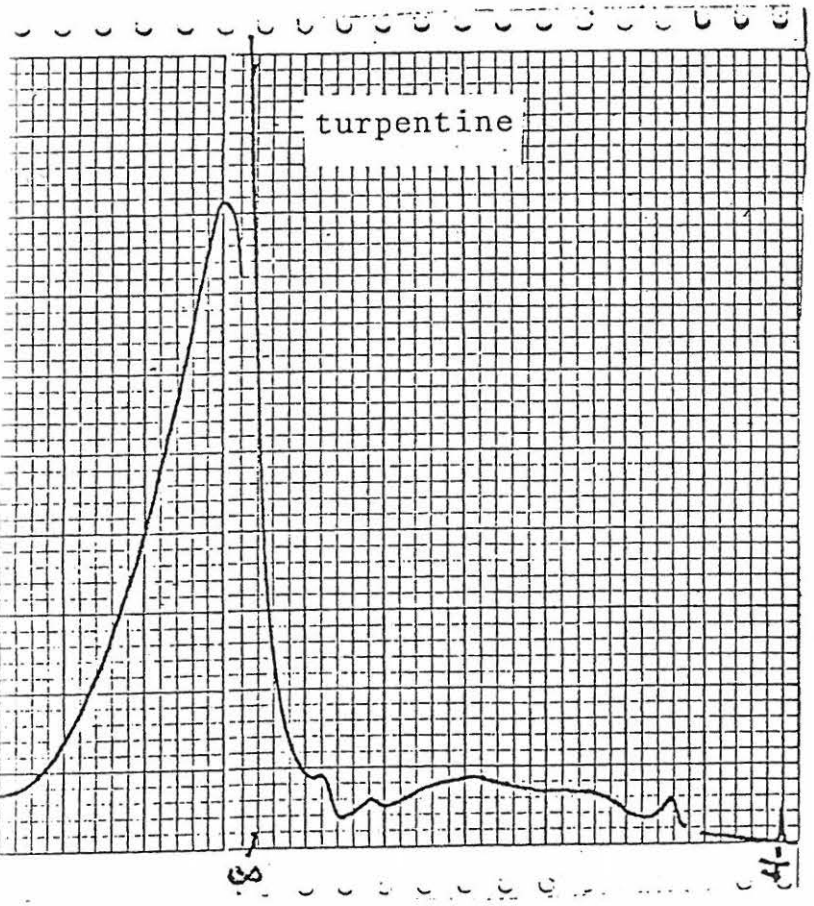


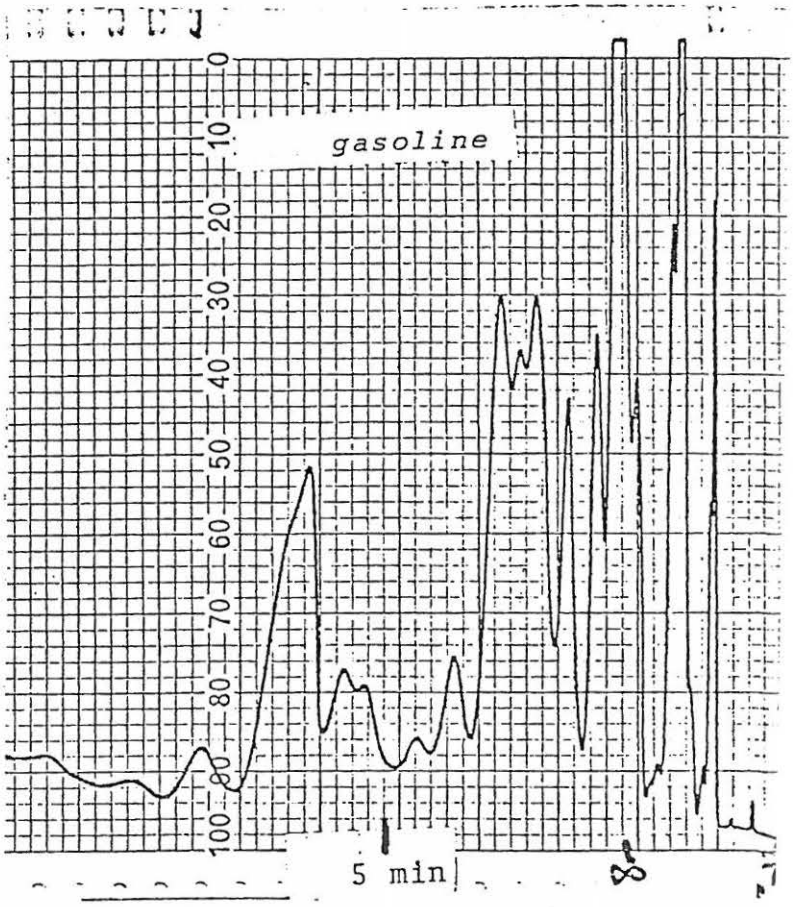
SE-30
150°C Chart 1cm/min
5 microliter





Durapak
100°C Chart 1cm/min
5 microliter





Discussion

Chromatograpms that appeared highly unfavorable for interpretation at 100°C or 150°C are not included in this report. Also, vapor (head space) injections proved to be generally unfavorable and are not included.

At 100°C isothermal, gasoline and turpentine for the majority of column packings yielded adequate results, and at 150°C fuel oil yielded adequate results. Fuel oil, which is less volatile than gasoline or turpentine, proved to be a decisive flammable liquid in determining operating oven temperature. It appears from the column packings tested, that an oven temperature of 150°C isothermal is required for favorable and easily recognizable chromatograms for fuel oil.

The reported chromatograms represent an evident variety of results, and the SP2100 plus 1.75% Bentone 34 column packing appears to yield the most favorable chromatograms at 150°C for the flammable liquids and column packings tested.

Sample Collection

Since the gas chromatograph tested was equipped with a thermal conductivity detector, it required liquid injections for favorable chromatograms.

The following proposed field technique was used successfully under laboratory condition:

1. Clean cotton swabs were saturated with 3 to 5 drops of hexane and the area containing a flammable liquid was swabbed.

2. 3 to 5 drops of hexane were again added to the cotton swab, and 10 microliters of solution was withdrawn from the swab into a 10 microliter syringe.
3. The 10 microliter sample was injected into the gas chromatograph.

The hexane did not significantly interfere with the chromatogram; however, the chromatogram did require attenuation changes. It should be noted that this sample collection technique was tested only under laboratory conditions, and it is not known at this time how effective the technique may prove to be in the field.

Conclusion

On the basis of the nine column packings tested under the operating conditions used in this evaluation, it appears that the 6 foot by 1/8 inch stainless steel column packed with SP2100/1.75% Bentone 34 yielded the most favorable chromatograms at 150°C isothermal for interpretation using liquid injections.

Acknowledgments

The author would like to thank Dr. James Howenstine, Director of Forensic Sciences Division, Michigan State Police, Lt. Donald Collins, Laboratory Commander Michigan State Police Scientific Laboratory, the members of the Michigan State Police Forensic Science Laboratory in Madison Heights, Michigan, and Mr. Jeff Lawson of GOW MAC, for their comments and cooperation.

Footnotes

1. D. M. Lucas, "The Identification of Petroleum Products by Gas Chromatography", Jour. of Forensic Science, Vol. 5, No. 2, 1960, pp. 236-243.

2. W. J. Chisum and T. R. Elzerman, "Identification of Arson Accelerants by Gas Chromatographic Patterns Produced by a Digital Log Electrometer", Jour. of Forensic Science, Vol. 16, 1971, pp. 280-291.

3. C. R. Midkiff and W. D.Washington, "Gas Chromatographic Determination of Traces of Accelerants in Physical Evidence", Jour. of Assoc. of Official Analytical Chemists, Vol. 55, No. 4, July, 1972, pp. 84-845.

4. L. Benjamin, "Qualitative Analyes of Lacquer and Similar Solvent Mixtures by Gas Chromatography", Analytical Chemistry, Vol. 47, No. 11, Sept. 1975, pp. 1851-1853.

5. C. E. Yates, "Recovery and Identification of Residues of Flammable Liquids from Suspected Arson Debris", American Chemical Society Symposium Series 13, Forensic Science, G. Davis (editor), 1975.

6. I. Yip and E. Clair, "Rapid Analyses of Accelerants in Fire Debris", Jour. of Canadian Society of Forensic Science, Vol. 9, No. 2, June, 1976, pp. 75-80.

7. U. S. Department of Justice, Law Enforcement Assistance Administration, Survey and Assessment of Arson and Arson Investigation , by the Aerospace Corporation, Law Enforcement Development Group, Pubn. No.ATR-76 (7918-05)-2, (October, 1976), pp. 79-87.

8. Ibid., p. 83.

9. Ibid., p. 85.

EXPLOSIVE DEVICE

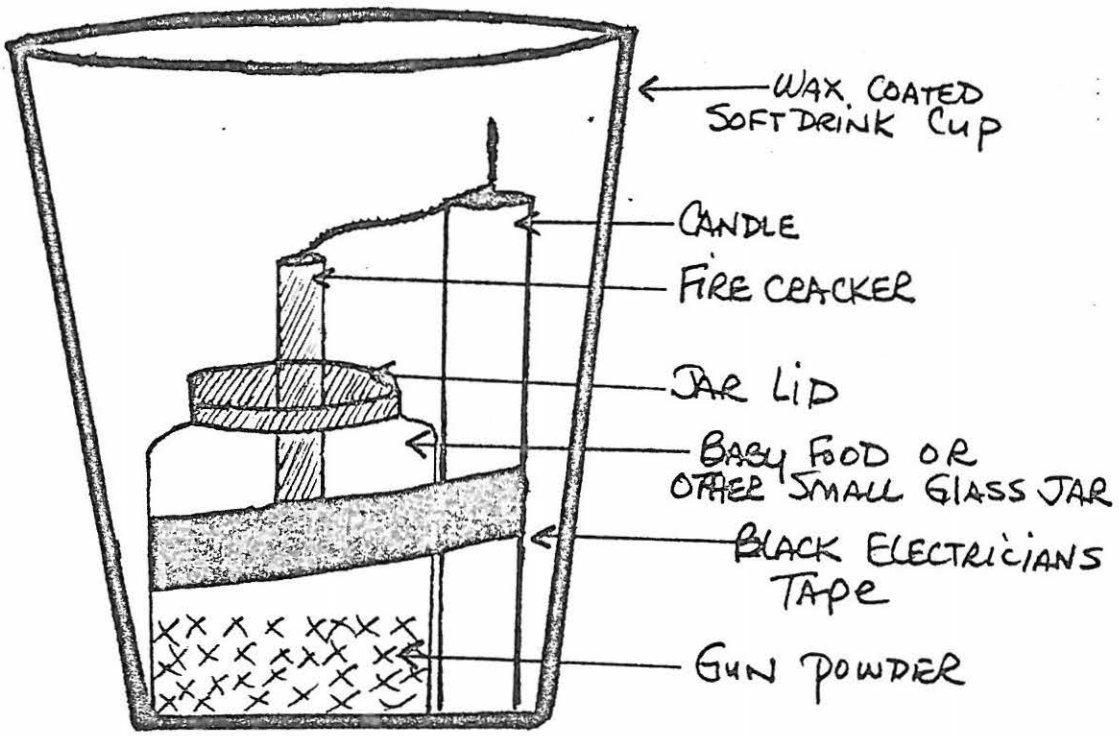
At the bottom of this page is a drawing of an explosive device that has been used at least twice in suspected labor union incidents in the Fresno area.

The device is floated in the diesel tank of large trucks. Since a small time delay allows the suspect to get away from the scene before the explosion takes place, it is difficult to get a suspect.

The main tip off that this type of device has been used is the broken jar found in the bottom of the diesel tank. A usable latent print has been lifted from one of the two incidents in Fresno.

If any such fires have been occurring in your area, please contact the Fresno Regional Laboratory.

Gary V. Cortner
Fresno Regional Laboratory



**Submitted by: Department of Justice
Division of Law Enforcement
Investigative Services Branch
Fresno Regional Criminalistics Laboratory

AA Notes

DECEMBER 1979

Mr. William Dean
3159 Eden Avenue
Cincinnati, OH 45219

During the recent Midwestern Association of Forensic Scientists (MAFS) meeting in Springfield, Illinois, several items of interest to arson analysts were discussed. The meeting included a one day workshop on GC-MS with emphasis on capillary column GC. The workshop was conducted by a representative from the Dupont Corporation at the Illinois State Forensic Lab in Springfield. The workshop consisted primarily of a survey of equipment and techniques including the jet separator, ion sources, mass analysers, detectors, WCOT and SCOT columns, sample size and loading, injection techniques, and mass spectra of arson materials. The following are a few of the hints which may be of interest to other analysts.

The instructor recommended avoiding nickel capillary columns but thought the fused silica (flexible) type were worth looking into He also recommended using a cigarette lighter to straighten out the capillary column ends and stressed the need to ensure the column ends were cut perfectly square. A ragged column end could result in troublesome dead volume.

To avoid contamination don't use a soap bubble meter to measure flow rate; instead use a 5µl syringe to inject a sample of butane from a "Bic" lighter, and monitor mass 43 or 58 on the mass spec. The carrier gas linear velocity equals the length of the column divided by the retention time of the butane peak:

$$\text{Vel} = \frac{L(\text{cm})}{R_t(\text{sec.})}$$

A good target range is 15-30 cm/sec. In general it is better to use a head pressure gauge rather than a rotameter to monitor flow through capillary columns. For a 0.25mm I.D. WCOT column the following pressures were recommended as a starting point (with the injection split open).

	HEAD PRESSURE	COLUMN LENGTH
WCOT	12-15 LB	20 meters
	30	40
	60	50+
SCOT	10	40

Analysts should avoid using soap solution to test for leaks. It is better to cool down the system and test the connections with methanol or methylene chloride.

For arson samples it was suggested that the can of debris be heated to 100°C in an oven for an hour or so and that a 100µl to 2ml gas tight syringe be used for injection. The best syringes are those that have a valve at the base of the needle. The sample volume can then be compressed before injection.

Two useful references on interpreting mass spectra are: Mass Spectral Correllations, an ACS publication by McLafferty and W.A. Benjamin Co. publication Interpretation of Mass Spectra also by McLafferty.

Later in the meeting D. Michael Kurz, Illinois State University presented an introduction to the chemistry of fire and Mr. Arthur Towner, Chief Electrical Inspector for Grand Rapids Michigan discussed cases involving the use/missuse of fuses, circuit breakers etc. He also provided copies of the "Electrical Protection Handbook" published by Bussman which is an excellent technical reference. You may be able to get a copy by writing the company. Ask for Bullentin SPD-78. The address is:

Bussman Manufacturing Division
McGraw - Edison Company
502 Earth City Plaza
P.O. Box 14460
St. Louis, MO 63178

There is a strong possibility of an Arson Roundtable discussion at the next MAFS meeting around May 8, 1980. This will be a combined meeting in Louisville, Ky. with the Southern, Mid-Atlantic, and Northeastern regional societies. The last combined meeting with SAFS was outstanding, this next one should be even better.

William L. Dean
Hamilton County Cornorer's Lab
Cincinnati, OH

AAN Notes

DECEMBER 1979

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