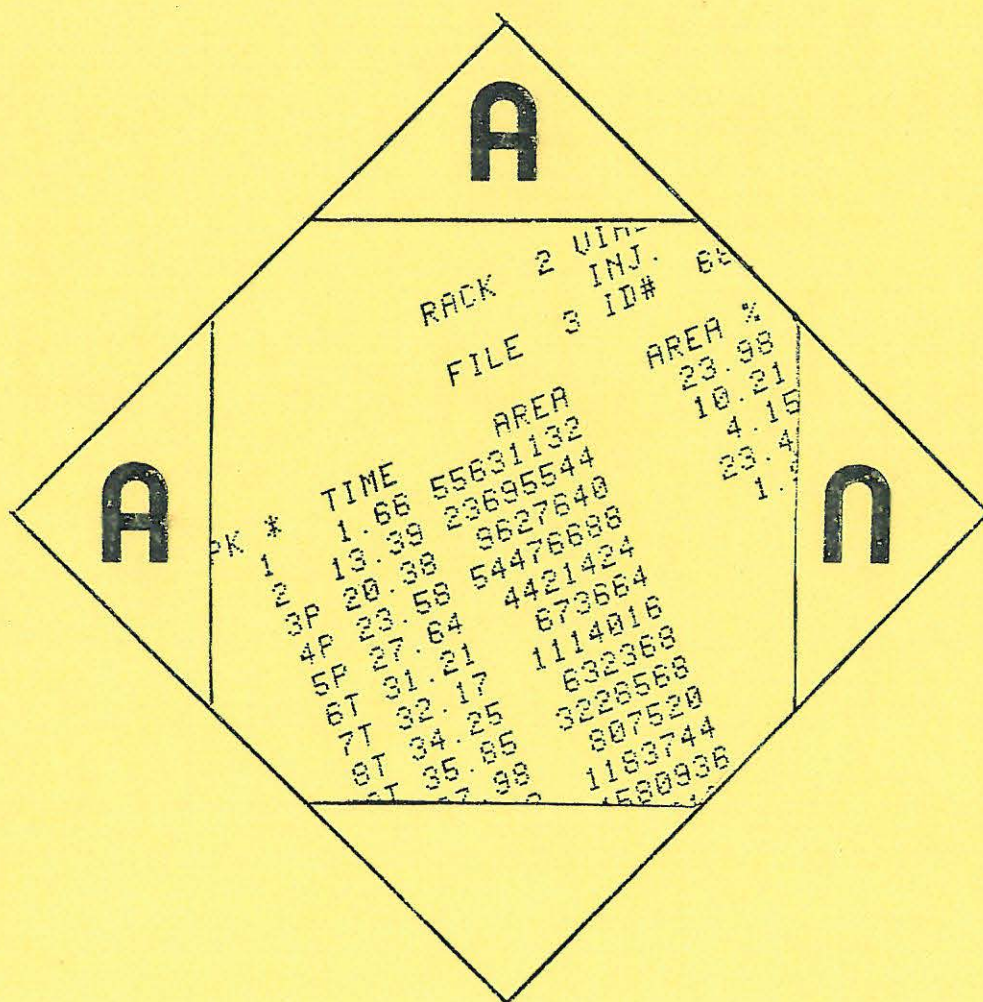


ARSON ANALYSIS NEWSLETTER

VOLUME 1 NO.6

9/77



ARSON ANALYSIS NEWSLETTER (AAN)

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Systems Engineering Associates
7349 Worthington-Galena Road
Columbus, Ohio 43085
Attention: R. N. THAMAN

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.



State of New Jersey

DEPARTMENT OF LAW AND PUBLIC SAFETY DIVISION OF STATE POLICE

RECORDS AND IDENTIFICATION SECTION
POST OFFICE BOX 7068
WEST TRENTON, NEW JERSEY 08625
(609) 882-2000

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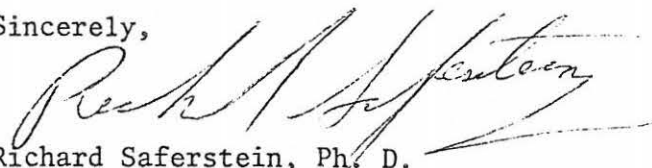
Hats off to Arson Analysis Newsletter for opening the lines of communication for those of us concerned with the detection of accelerants in arson investigations. After reading the articles and letters appearing in Newsletter it is quite apparent to me that your publication can serve as an effective medium for exchanging information and data on this very vital subject. However, the contents of your past issues does reveal one distressing observation, that is, the wide variation in techniques and methods presently being used by crime laboratories to examine debris suspected of containing some type of accelerant. This observation is further fortified by the recently published results of a survey relating to accelerant detection methodology (1). While most of us seem to be using gas chromatography little agreement exists concerning column packings and related gc conditions. Likewise, a significant number of laboratories have incorporated IR into their analytical procedures. Furthermore, methods of sample recovery vary between crime laboratories, these include headspace, steam distillation, solvent extraction and vacuum distillation.

If continuing progress is to be made in accelerant analysis, and if the exchange of analytical data is to be facilitated, then the need for some degree of standardization becomes apparent. For example, articles in Newsletter have cited the problems of distinguishing pyrolysis products from

those of added flammable liquids (2,3). In the future some of your contributors may want to publish chromatograms and/or IR spectra of the degradation products they have encountered in case work. However, while this type of information will surely begin to fill a very troublesome void in our knowledge, the data may prove meaningless to those readers not employing comparable techniques and procedures. In a field where publications are sparse, the likelihood of such occurrences are disconcerting.

It seems to me that no valid reason exists to justify the wide diversity of techniques presently used by crime laboratories for accelerant analysis. Undoubtedly, this situation arose out of our lack of communication in past years. This is especially true of gas chromatographic procedures. Why can't the forensic community agree to standardize on one or two liquid phases suitable for hydrocarbon analysis? The time is ripe for change and Newsletter can be the catalyst to effect standardization. I don't think it would be out of order to suggest that a committee representing a cross-section of Newsletter subscribers be organized to study and recommend solutions to this problem. In any case, our failure to recognize and correct this situation will hamper our ability to exchange meaningful analytical data in future issues of Newsletter.

Sincerely,

A handwritten signature in dark ink, appearing to read 'Richard Saferstein', with a long horizontal flourish extending to the right.

Richard Saferstein, Ph. D.
Chief Forensic Chemist

RS:kw

P.S. At present our New Jersey State Police laboratories are routinely using headspace for analyzing suspected accelerants. We're using a 8' X 1/8" stainless steel column packed with 7% Bentone 34 and 10% didecylphthalate. Oven temperature is isothermal at 100°C.

- (1) Loscalzo, P. J., "A Study to Determine the Limit of Detectibility of Gasoline Vapor From Different Surfaces After Various Periods of Combustion and Collection Delay", Masters Thesis, John Jay College, New York City, 1977.
- (2) Thaman, R. N., Arson Analysis Newsletter, Vol. I. No. 1 (1976) pp. 9 - 19.
- (3) Graves, R. L., Hunter, D., and Stewart, L. E., Arson Analysis Newsletter, Vol. I., No. 5 (1977) pp. 5 - 12.

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PHONE: 609-267-8282



August 18, 1977

Systems Engineering Associates
7349 Worthington-Galena Road
Columbus, Ohio 43085

Attention: R. N. Thaman

Dear Dr. Thaman:

In response to our recent telephone conversation, enclosed please find a copy of survey and results conducted by Mr. Peter Loscalzo, under the guidance of Dr. Peter DeForest and myself. From the results, it seems that there exists very little standardized guidelines or reference materials for arson investigation. Therefore, the investigator has a scant amount of established criteria with which to compare his own results. The first step to solve this problem would be for you to request data concerning arson from each investigative laboratory with which you deal. Included in this data would be a copy of gas chromatogram marked with known peak of regular gasoline and list the column used and all operation parameter. Your compilation of this material would enable all arson investigators like myself to have some type of reference source.

Furthermore, the investigators would be afforded the opportunity to consider other methods of analysis and perhaps some nationwide standard for arson investigation would ensue. If you prefer for me to take over this work I will be glad to do so.

Sincerely,

Jew-ming Chao, Ph.D.
Laboratory Director

Enclosure
JC:dlj

ARSON QUESTIONNAIRE

1. Does your laboratory examine evidence from suspected arson cases
for the presence of flammable hydrocarbons? yes no
 2. Usual method of analysis:
Gas Liquid Chromatography Mass Spectrometry Other
If "other" please specify: _____
 3. Usual method of sample recovery for analysis:
heated headspace vapors steam distillation
Other:(please specify) _____
 4. If you use GLC, please furnish the information below:
Liquid phase _____ Solid Support _____
Column Type: glass stainless steel capillary
Column Length: _____
 5. If you use GLC, do you employ any of the following techniques in your
sample analysis:
temperature programming attenuation programming
both simultaneously
 6. From your experience, which of the following would you consider a
major problem in the detection if a flammable hydrocarbon from
suspected arson residues" (Please rate each item consecutively from
1 to 4 where 1= most major problem and 4= least major problem)

Evidence not collected in sufficient time after extinguishment
Fire not extinguished in sufficient time to obtain accelerant
detection
Evidence improperly collected and/or preserved before submission
to laboratory
Other:(specify) _____
 7. Which accelerant has your laboratory most often encountered from
arson evidence? _____
 8. Do you wish the results of this survey? _____
- NAME: _____
TITLE: _____
DATE: _____

RESULTS OF ARSON DETECTION SURVEY

Total questionnaires mailed.....	181
Less no answers to Jan. 1, 1977.....	62
Total questionnaires returned.....	119
Less returns without bona fide laboratories & mailing errors.....	6
Less FBI (refused to supply the requested information).....	1
Total bona fide criminalistic labs that answered the survey.....	112
Less laboratories that did not do arson examinations.....	16
Total bona fide laboratories surveyed that are performing arson examinations.....	96

Methods of Analysis

GLC	93
IR	18
GLC - MS	7
Sp Gr - RI	3
NMR	2
UV	1
GC	1
Flash Point	1
AA	1

Methods of Sample Recovery

Heated Headspace Vapor	71
Steam Distillation	42
Solvent Extraction	18
Non-Heated Headspace Vapor	5
Vacuum Distillation	3
Glycol Distillation	1

Column Construction

Stainless Steel	56
Glass	32
Capillary	9
Aluminum	1

COLUMN PACKINGS

Liquid Phases

SE - 30	28
OV - 101	12
Apieson-L	12
OV-1	8
OV-17	6
Bentone-34/DIDP	3
SR-2100	3
SE-30/OC-550	2
SE-52	2
DC-200	2
Carbowax	2
Various Others	10

Solid Supports

Chromosorb W-HP 80/100	27
Chromosorb Q 80/100	12
Chromosorb W 60/80	7
Chromosorb W-AW	4
Firebrick C-22	4
Supelcoport	4
Silnox 101	2
Various Others	5

Arson Survey Results Continued

GLC Operational Techniques

Temperature Programming	71
Attenuation Programming	4
Both Simultaneously	15
Digital Log Recorder	3

Responses to Problems in Arson Analysis

Statement 1 : Evidence not collected in sufficient time after extinguishment.

Statement 2 : Fire not extinguished in sufficient time to obtain accelerant for detection.

Statement 3 : Evidence improperly collected and/or preserved before submission to laboratory.

Statement 4 : Problem other than the above.

Statement Number	Rated First	Rated Second	Rated Third	Rated Fourth
1	13	23	31	4
2	21	19	40	12
3	51	18	15	6
4	6	11	5	69

A considerable number of responses offered comments about some probable sources of problems in arson analysis. These comments are listed below as they appeared in the individual questionnaires:

"Time lag between evidence collection and analysis."

"Evidence not collected from proper area."

"Too much unimportant and meaningless debris collected, everyone involved is wasting time."

"Lack of even common sense in fire scene examination to determine areas of potential residues."

"Failure to recognize suitable evidence."

"Elimination or reference samples not submitted."

"Laboratory proximity to fire site."

"Insufficient sample collected."

Arson Survey Results Continued

"Submission of control samples. Ex: unburnt carpet or floor tile. Headspace vapors often contain combustion products of substrate."

"Sample preservation techniques."

"Case backlog---lengthy time before analysis---loss of accelerants."

"Sample from improper locations."

"The problem is getting good evidence to analyze. The analysis is straightforward and easily interpreted."

"Reference and 'known' materials rarely obtained or located."

"Insufficient residue in sample."

"Insufficient sample or wrong materials selected."

"Evidence taken from wrong areas."

"Alteration of the accelerant by the fire and contamination from hydrocarbons other than accelerant."

"Wrong debris collected."

"Evidence submitted to laboratory after long period of time."

"Masking of accelerant due to severe burning of or nature of substrate."

"Improper sampling."

"Not finding the origin, therefore improper evidence is collected."

"Controls and standards found at scene not collected."

"Evidence not collected at proper site, i.e. improper point of origin determination by investigator."

"Being able to financially afford better instrumentation for more up-to-date and proficient analysis."

Accelerant Frequency

Gasoline	86
Kerosene	4
Diesel Fuel	1

ADSORPTION-ELUTION TECHNIQUE FOR CONCENTRATION
OF HYDROCARBON VAPORS

Ronald E. Baldwin

South Carolina Law Enforcement Division
P. O. Box 21398
Columbia, S. C. 29221

In analysis of some samples submitted to our laboratory for identification of accelerant residues, it has been desirable to concentrate accelerant vapors in the headspace to a liquid for analysis by gas chromatography or by gas chromatography-mass spectrometry. Cases where this was necessary were usually those which have been subjected to extensive evaporation before the evidence was collected, or submitted in containers having large headspace volume. Opening these containers for conventional techniques such as steam distillation or solvent extraction would result in loss of accelerant residues, while a direct injection of headspace air would result in insufficient sample being injected. A liquid sample has been found to be preferable to a large volume of headspace air for gas chromatography-mass spectrometry and for capillary column gas chromatography.

Hydrocarbons in the headspace are adsorbed by drawing air in the original evidence container through a disposable pipette filled with Florisil (a product of the Floridin Company) between cotton plugs. The large end of the pipette was attached to a vacuum, while the small end was inserted into a small hole in the sample container. Flow rate was controlled by a needle valve and was about 50 ml per minute. These

hydrocarbons are then removed by washing the column with 5 ml of carbon disulfide. The carbon disulfide was then evaporated at room temperature by application of a vacuum.

The Florisil used here was first extracted with methanol for 5 hours in a Soxhlet extraction apparatus, dried, and then activated at 300 degrees for one hour.

Figures 1 and 2 are gas chromatograms of kerosine and gasoline samples run on a 10% OV-101 on Chromosorb P, 100/120 mesh, 1/8 inch by 10 feet column. The column temperature was programmed from ambient to 225 degrees at 16 degrees per minute. Figure 2 was obtained by drawing the headspace air containing vapor from 5 microliters of kerosine in one liter of air through the column. The total volume of air drawn through the vented container was two liters. Figure 3 was obtained under the same conditions using 5 microliters of gasoline.

There was little change in the chromatograms of kerosine, while the lower-boiling components of gasoline failed to be adsorbed and were lost. This loss of lower-boiling components becomes appreciable for components which boil below 125 degrees. However, many samples which have been severely evaporated have only the heaviest components remaining, and identification of the accelerant must of necessity be made from these.

In our opinion, concentration of hydrocarbons in headspace samples by adsorption and elution offers advantages over steam distillation or solvent extraction for samples containing only trace amounts of accelerant residue, or for samples from which a large fraction of the accelerant has evaporated in regard to time required for the operation and efficiency of sample recovery.

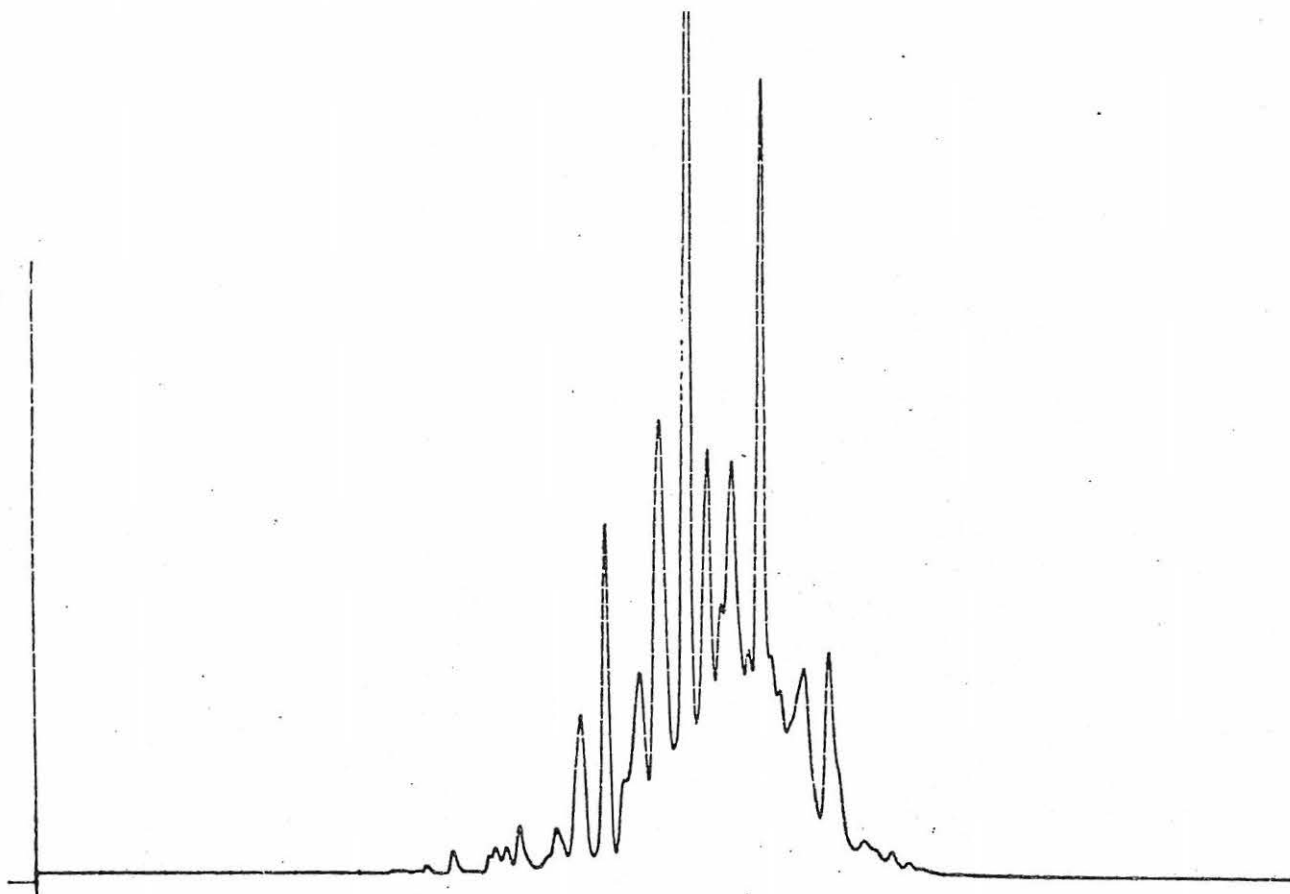


FIGURE 1: Gas Chromatogram of a Kerosine Sample

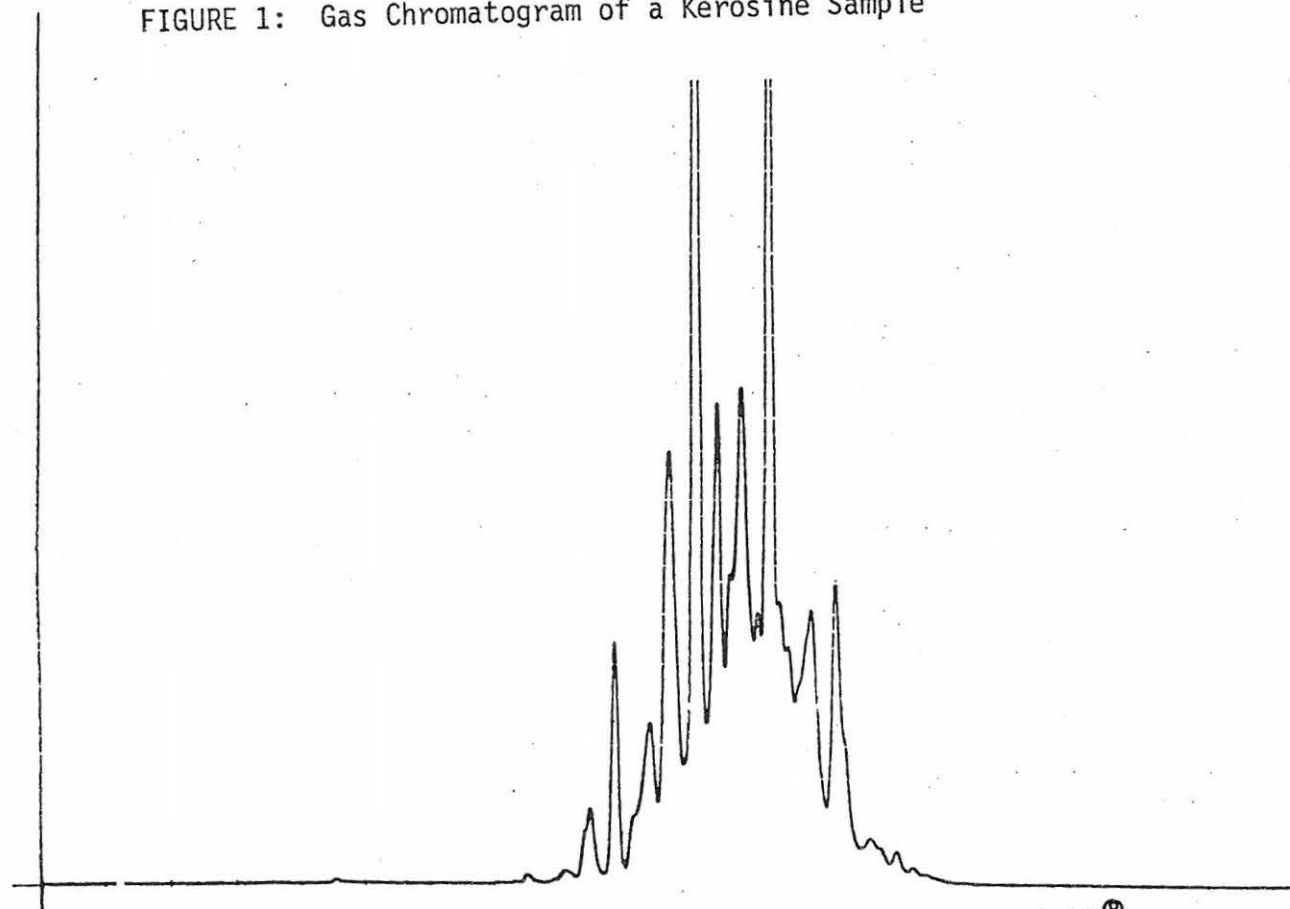


FIGURE 2: Gas Chromatogram of Sample Trapped on Florisil[®]

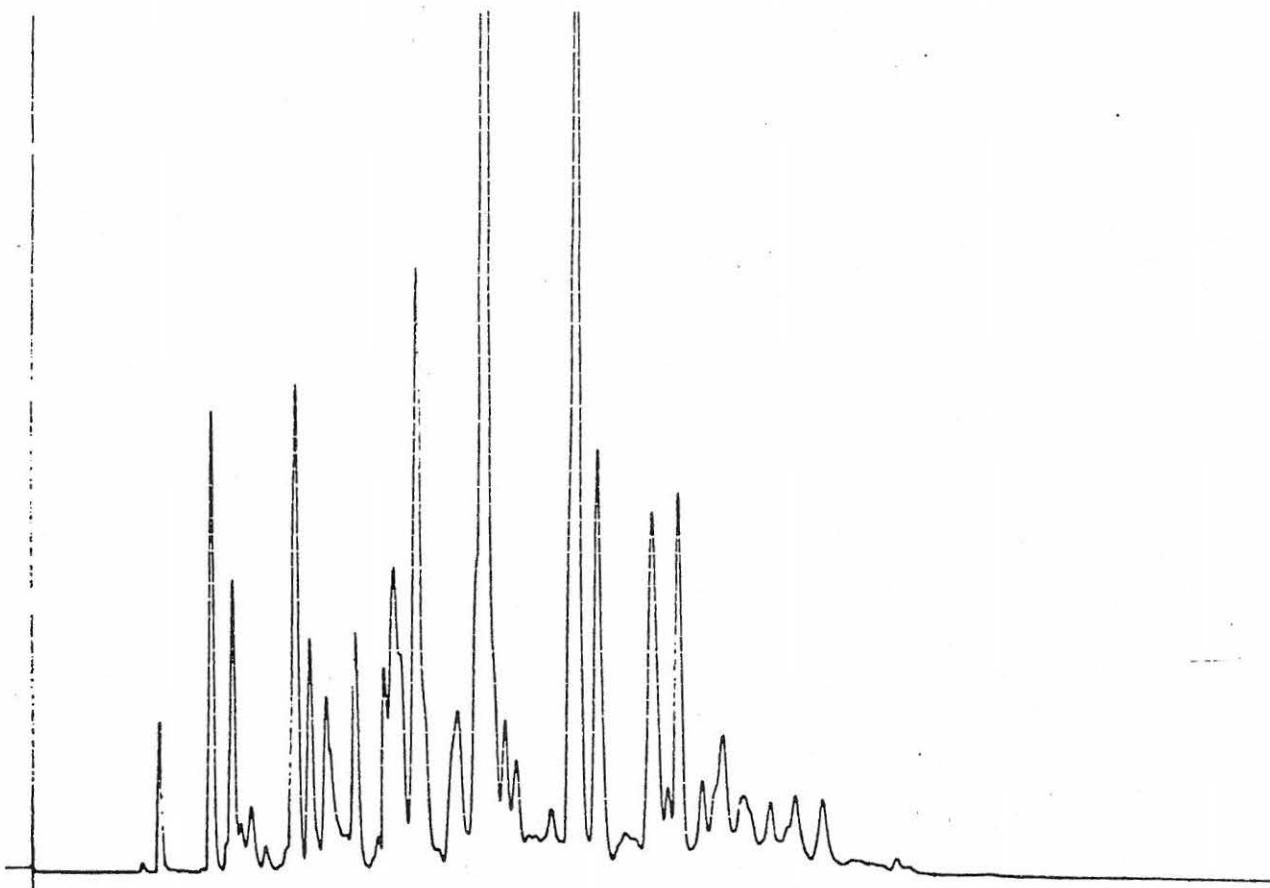


FIGURE 3: Gas Chromatogram of Gasoline Sample

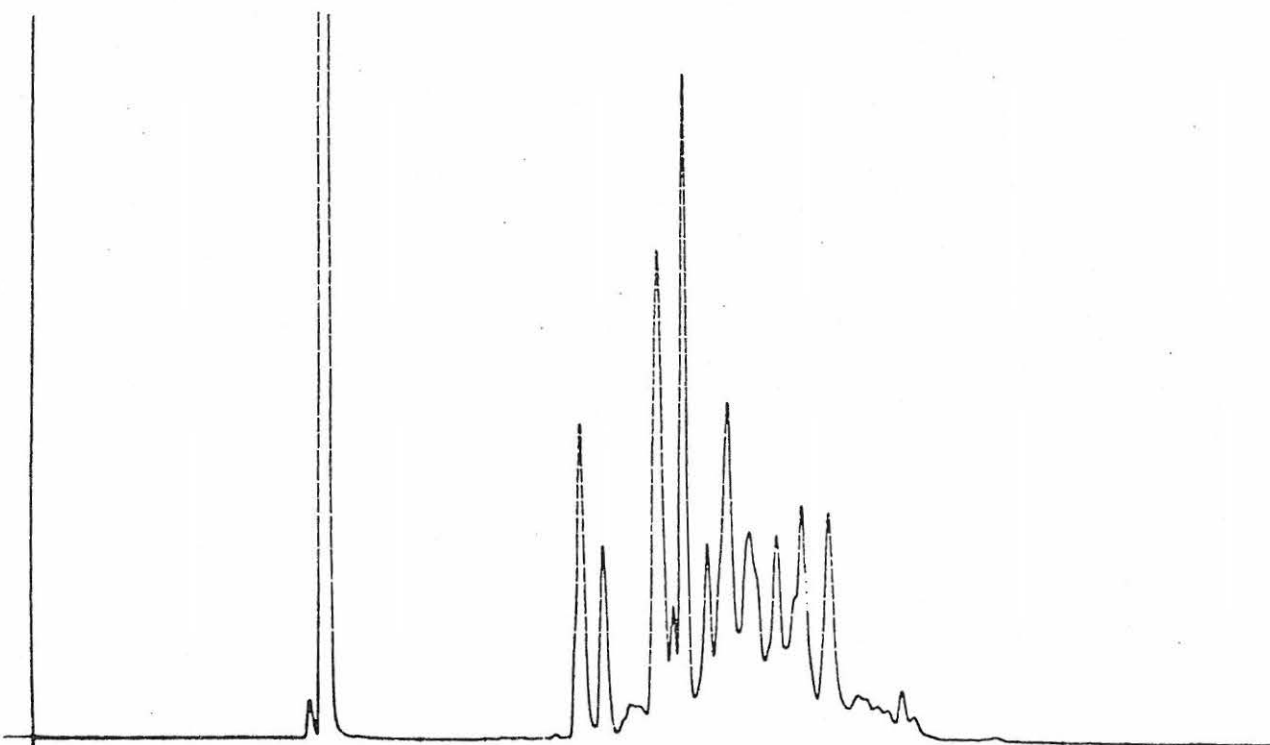


FIGURE 4: Gas Chromatogram of Sample Trapped on Florisil[®]

- * Three recent articles of interest to forensic scientists analyzing fire debris have come to the attention of the AAN:

Rapid Identification of Light Petroleum Products
By Gas Chromatography, K. Leung, H.L. Yip, Can. Soc.
Forens. Sci. J. Vol 3, No. 2 (1970) pp. 42-51.

A Rapid Analysis of Accelerants In Fire Debris
Ivan H. Yip & E. G. Clair, Can. Soc. Forens. Sci.
J. Vol 9, No. 2 (1976) pp. 75-80.

Vacuum Distillation For The Recovery Of Fire Accelerants
From Charred Debris, R. Hrynchuk, R. Cameron, P.G. Rodgers
Can Soc. Forens. Sci. J. Vol 10 No. 2 (1977) pp. 41-50.

- * A "spot reagent" called RHODOKRIT H-630 has come to the attention of the AAN. The reagent reportedly when applied to fire debris will cause a red coloration to develop where a petroleum distillate was present. The reagent was first mentioned in the Fire and Arson Investigator in the October-December 1953 issue. If this rings a bell with anyone please drop the AAN a line describing its bad/good points. Any information received will be published in the next AAN.
- * A good article appeared in Applied Spectroscopy Vol. 31, No. 4 July/August 1977, p. 298-307 describing the analysis of trapped atmospheric contaminants desorbed from charcoal tubes. The method is easily adopted to the analysis of fire debris.
- * A new journal called FIRE RESEARCH is being published by Elsevier Sequoia S.A. P.O. Box 851, 1001 Lausanne 1, Switzerland. The first issue had some very interesting articles concerning flammability decomposition product prediction and analysis of polymeric materials involved in a fire.
- * The enclosed Differential Infrared Spectrum has occurred repeatedly in CCl₄ extracts of samples, mainly plastic, at SEA. Until recently the origin of the chemical was assumed to be a plasticizer or monomer from a polymer. The material has now been placed in the group of polyvinyl

ethers. Infrared spectra in Hummel & Scholl's Infrared Analysis of Polymers, Resins & Additives An Atlas has provided the answer. The book is published by Wiley-Interscience. The most characteristic band 9.0μ is due to asymmetrical C-O-C stretching.

DIFFERENTIAL INFRARED SPECTRUM, CCl_4 , 0.1 cm SPACER (PLASTIC SAMPLE)

WAVELENGTH IN MICRONS

