

BILL DEAN

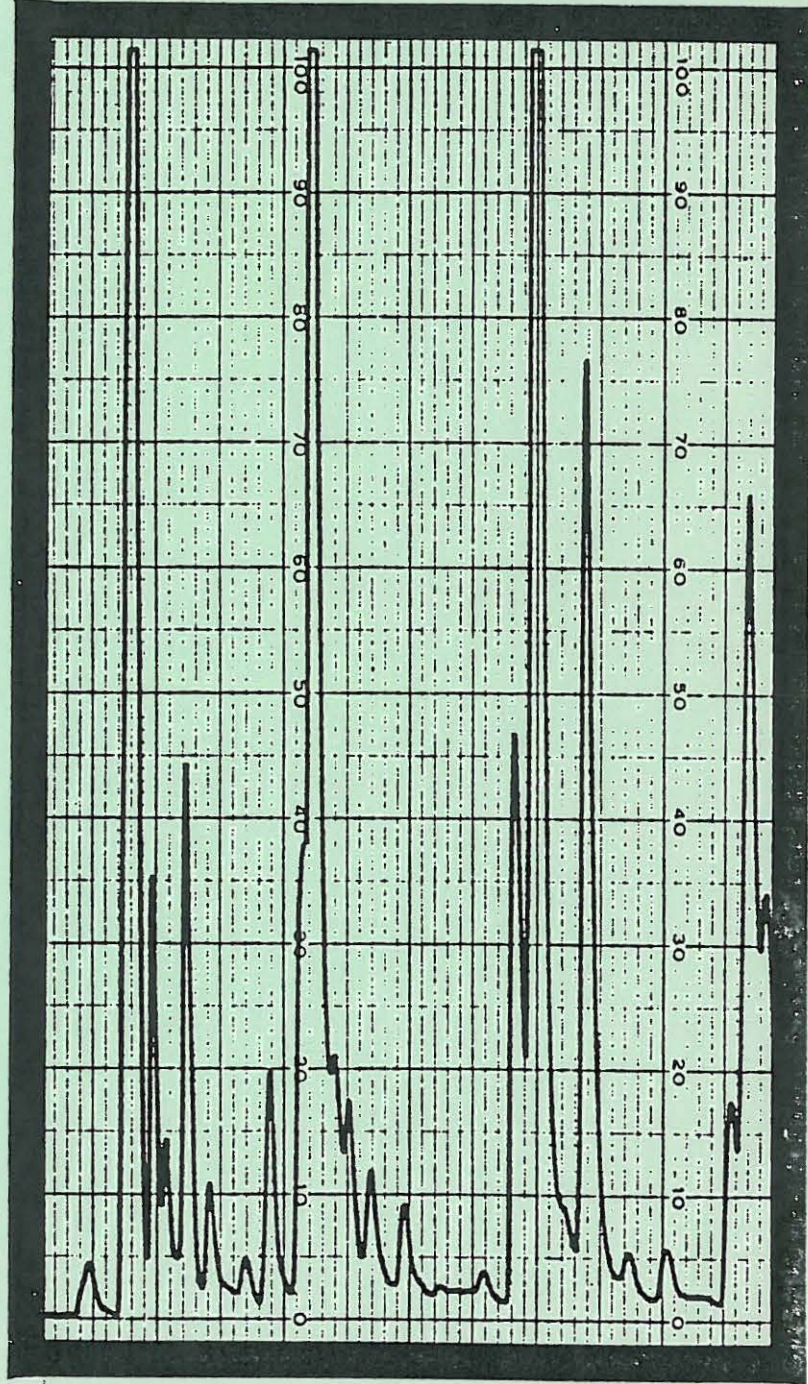
POLYSTYRENE

JOHN

JUHALA

Arsen Analysis Newsletter

OCTOBER 1979
VOLUME 3
NUMBER 4



ARSON ANALYSIS NEWSLETTER (AAN)

Published bimonthly by Systems Engineering Associates.
The AAN is available for a subscription price of \$6.00
per year for subscribers within the United States and
\$20.00 per year for subscribers outside of the United
States.

Send all subscriptions and manuscripts to :

Systems Engineering Associates
7349 Worthington-Galena Road
Columbus, OH 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.

EDITOR: Ronald N. Thaman, Manager-Analytical Chemistry,
Systems Engineering Associates, Columbus, OH.

ASSISTANT EDITORS: Jew-ming Chao, Ph.D., Laboratory Director
Burlington County Public Safety, Woodlane
Road, Mt. Holly, NJ.

John D. DeHaan, Dept. of Justice, Division
of Law Enforcement, Investigative Services
Branch, P.O. Box 13337, Sacramento, CA.

Blair W. Schultz, Illinois Bureau of Scientific
Services, 515 East Woodruff Road, Joliet, IL.

Robert Kopec, Sanford Regional Crime
Laboratory, P.O. Drawer 1737, Sanford, FL.

ANY REPUBLICATION OF THE ARSON ANALYSIS NEWSLETTER (AAN)
WITHOUT THE EXPRESS WRITTEN PERMISSION OF THE EDITOR OF
THE AAN, IS STRICTLY PROHIBITED.

ARSON ANALYSIS NEWSLETTER

VOLUME 3, NUMBER 4

	<u>Page</u>
DETERMINATION OF FIRE DEBRIS VAPORS USING AN ACID STRIPPING PROCEDURE WITH SUBSEQUENT GAS CHROMATOGRAPHIC AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS. By John A. Juhaala.	1
REPORT ON CALIFORNIA CONFERENCE OF ARSON INVESTIGATORS, 1979 ANNUAL SEMINAR. By John D. De Haan.	20
NOTES: C.A.C. TOUR OF UNION OIL RESEARCH CENTER (3-2-78). By Duane Mauzey.	25
<u>AAN</u> Notes	28

DETERMINATION OF FIRE DEBRIS VAPORS USING AN ACID STRIPPING
PROCEDURE WITH SUBSEQUENT GAS CHROMATOGRAPHIC AND
GAS CHROMATOGRAPHY/MASS SPECTROMETRY ANALYSIS

John A. Juhala, Ph.D.
Michigan State Police
Bridgeport Crime Laboratory
P.O. Box H, 6296 Dixie Highway
Bridgeport, Michigan 48722

In many crime laboratories it is common practice to screen fire debris samples, submitted in connection with an arson investigation, using headspace vapor samples for gas chromatographic analysis. The resulting chromatograms are then compared with the chromatograms obtained from flammable liquid standards and the unknown vapors are identified when a reliable match is obtained. For the experienced examiner, the majority of chromatograms encountered can be interpreted without recourse to the reference collection and can either be identified as a given flammable liquid or rejected as products of combustion of various building materials without specific components of the chromatogram being identified.

However, with the increasing use of various types of polymers in the building and automotive industries, the products of combustion can often yield a chromatogram which bears a striking resemblance to some of the less complex common flammable liquids. Many lacquer thinners and enamel reducers contain only five or six components, and the chances of obtaining a debris sample which has one or more peaks with retention times close to those obtained for components of these flammable materials are fairly good. In these instances

the discrimination between a chromatogram which represents a flammable liquid and one which does not is more difficult. An additional problem arises because of the fact that different brands of the same type of product containing flammable liquids vary in formulation and it is difficult for the laboratory to keep on hand all possible brands of a particular product. If there is any doubt in the analyst's mind as to whether or not the chromatogram represents a flammable liquid, he will report it negative. One would like to determine with the greatest scientific certainty whether or not the chromatogram represents a flammable liquid, possibly one with which the analyst is not familiar, or is merely the result of the combustion of various polymers.

In order to make a better decision as to the significance of a particular chromatogram in those cases which are not straight forward, it is necessary to identify some of the peaks in the chromatogram; preferably by some technique which is not solely dependent upon G.C. retention times. Since the concentrations of residual vapors found in debris samples are often very low, the application of infrared and ultraviolet spectroscopy is generally difficult since it usually requires liquid extractions and subsequent concentration steps which may lead to the loss of volatile components and coextraction of artifacts. Gas chromatography and mass spectrometry both have the requisite sensitivity to detect flammable liquids at the concentrations encountered

in fire debris samples. We present here the analysis of a fire debris sample using a series of chemical reactions in conjunction with gas chromatography which will render the G.C. results more specific, and the application of combined GC/MS techniques as a further aid in characterizing the sample.

EXPERIMENTAL

Questioned Sample

The fire debris sample was one of several received at the laboratory in connection with the suspected arson of a restaurant.

Gas Chromatography

The gas chromatographic analyses were carried out using a Varian 2700 gas chromatograph equipped with FID and a dual pen recorder. The three columns used were as follows: 8' x 1/8" stainless steel, packed with 15% Apiezon L on 45/60 mesh Gas Chrom RA, operated isothermally at 160°C with a nitrogen carrier of 20 cc/min; 10' x 1/8" stainless steel, packed with 15% FFAP on 80/100 mesh Gas Chrom Q, operated isothermally at 100°C and a nitrogen carrier of 20 cc/min; and a 6' x 1/8" stainless steel column, packed with 5% Di-n-decyl phthalate and 5% Bentone 34 on 100/120 mesh Gas Chrom Q operated isothermally at 105°C and a nitrogen carrier of 20 cc/min. One ml samples of headspace vapor were used for screening the debris on the three columns.

Acid Stripping Procedure

The stripping procedures used are classical analytical techniques. The oxygenated compounds are removed by taking advantage of their solubility in mineral acids and the aromatics which remain in the mineral acid insoluble fraction are nitrated and subsequently removed with dimethyl sulfate.

Comercially prepared dual layer charcoal adsorption tubes were used to trap the organic vapors for the acid stripping procedures. The procedures used are ones familiar to the petroleum industry and have been adapted for use with vapor samples by White, etc. al. (1) and Levadie and MacAskill (2, 3).

A fire debris sample suitable for stripping was obtained as follows: The ends of a flame sealed charcoal tube were broken off and the tube was attached to the suction end of a standard atomizer bulb with the opposite end of the adsorption tube inserted into a hole punctured in the lid of the sample container. Approximately 500 ml of vapor were withdrawn from the sample container through the charcoal tube which traps the organic constituents. A known vapor sample of a mixture of ethyl acetate, ethanol, benzene, xylenes, and isoamylalcohol was obtained in similar fashion using a second charcoal trap. This mixture contains many of the common components of quality lacquer thinners. The charcoal tubes were then cut open and the charcoal from each

tube placed into separate 5 ml serum vials. In a fume hood 1 ml of CS₂ was added to each vial which was then capped with a serum vial septum and the vials rotated for thirty minutes on a serological rotator to elute the adsorbed vapors. This procedure yields CS₂ solutions having approximately the same composition as the original headspace vapors.

After the adsorbed vapors were eluted from the charcoal into the CS₂ 2 ul of each of the resulting solutions was chromatographed using the FFAP column and the conditions described previously. This provides the reference chromatograms for comparison with those obtained from the stripped samples.

As much as possible of the CS₂ solutions were then transferred from the serum vials, using a hypodermic syringe and needle, to a set of 5 ml screw-capped septum vials which contained 0.5 ml of a mixture of sulfuric and phosphoric acids (1 ml conc. H₂SO₄; 1.7 ml (85%) phosphoric acid). The vials were capped, shaken for one minute, and the layers allowed to separate. This treatment will remove all of the compounds containing oxygen. The carbon disulfide (upper) layers were then chromatographed using 1 ul samples of the liquid.

As much as possible of the CS₂ layer was transferred to a second set of 5 ml screw-capped vials, each containing 2 ml dimethyl sulfate, 2 ml of sulfuric acid, and 1 drop of concentrated nitric acid. The mixtures were shaken for

one minute and allowed to separate into layers. Two- μ l portions of each of the carbon disulfide layers (upper) were chromatographed. The aromatic components have been stripped from this fraction and the remaining peaks in the chromatograms are the alkyl portion.

GC/MS

The gas chromatograph/mass spectrometer used was a Finnigan Model 3100F equipped with a data system. The CI mode was used with isobutane as a carrier and reagent gas at a source temperature and pressure of 130°C and 500 μ respectively. The FFAP column of the gas chromatograph described above was operated isothermally at 115°C. Data on standards of benzene, toluene, ethylbenzene and styrene were obtained under these conditions as well as on a 2 ml headspace sample from the fire debris sample.

RESULTS AND DISCUSSION

The chromatograms obtained on the three columns from the headspace vapors of the fire debris sample are shown in Figs. 1, 2 and 3. Apiezon L is our usual screening column, and the pattern observed is relatively simple and resembles that obtained from lacquer thinners and also some polymers. Examination of the retention times obtained on the three columns showed peaks corresponding to benzene, toluene, and ethylbenzene. The largest peak in the chromatogram still remained unidentified as none of the available standards had retention times which matched this peak. The large increase in retention times for this peak on the FFAP and Bentone columns

as compared to the Apiezon L column is typical of an aromatic compound. The peak corresponding to ethylbenzene was disturbing because of the apparent lack of any xylenes which are normally present in much large quantities than ethylbenzene in mixed aromatic solvents.

Since a GC/MS is not available in our laboratory the acid stripping techniques described above were used to gain additional information about the compounds identified from retention data. It is first necessary to extract the components of interest from the fire debris matrix. Aspirating the headspace through a charcoal trap provides a convenient method of accomplishing this. This same procedure provides a rapid and effective method for collecting vapor samples from the scene of a fire or underground leak and could conceivably eliminate the need for collecting debris samples in many instances.

The chromatograms of the carbon disulfide eluted vapors from the fire debris sample and the standard mixture are shown in Figs. 4 and 5. These provide reference chromatograms to be compared with the chromatograms of the stripped samples. With the exception of an early peak due to the carbon disulfide the chromatograms are unchanged by the adsorption-elution process.

The first stripping step with a sulfuric/phosphoric acid mixture removed any oxygen containing compounds present. The resulting chromatograms for the fire debris and standard

samples are shown in Figs. 6 and 7. The standard sample which originally contained ethyl acetate, ethanol and methyl isobutyl ketone is now missing these oxygen bearing compounds. No change has occurred in the chromatogram of the fire debris sample and therefore no oxygen compounds were present in the sample.

The second step of the stripping procedure removed any aromatic compounds and the resulting chromatograms are shown in Figs. 8 and 9. The aromatic compounds remaining in the known standard after the preceding step have now been removed by this step. All the peaks from the debris chromatogram have also been removed which is in agreement with their tentative identification as benzene, toluene and ethylbenzene. The tentative identification of the largest peak as an aromatic compound is also confirmed by its absence from the stripped sample.

With a view towards identifying the compound producing the most intense peak in the chromatogram and the possibility that this would make it possible to explain the presence of benzene, toluene and ethylbenzene, the sample was taken to another laboratory where G.C./M.S. capabilities were available.

Using GC/MS the presence of benzene, toluene and ethylbenzene were confirmed and a molecular weight of 104 was obtained as being the large unidentified peak. This corresponds with the molecular weight of styrene. An examination of the structure of polystyrene (Fig. 10) reveals that its thermal degradation

could readily yield styrene, ethylbenzene, toluene and benzene.

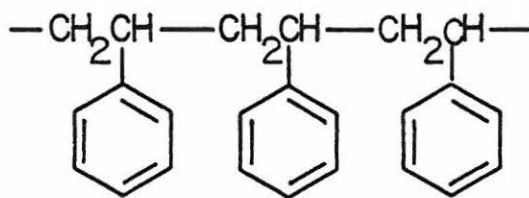


Fig. 10

This hypothesis was tested by burning polystyrene in air and subjecting the vapors to GC/MS analysis. The products observed were styrene, ethylbenzene, toluene and benzene with the relative concentrations the same as those observed for the fire debris sample. Thus the aromatics found in the debris sample are probably the result of some polystyrene plastic item consumed in the fire, and not the residue from an arsonist's flammable liquid.

CONCLUSIONS:

The identification by gas chromatography of benzene, toluene, and ethylbenzene in a debris sample could easily tempt an analyst to report the presence of a flammable liquid (or vapors) which would be true in the strictest sense. However, such a report could in a situation such as that described above, lead the investigator to treat the case as an arson and possibly make a false arrest when only a child's plastic toy may have been left on the floor. The widespread use of polymers coupled with the extreme sensitivity of the gas chromatograph should serve to make the analyst cautious

when an unfamiliar pattern with a few peaks having retention times similar to known flammables is obtained from a debris sample. Further work may be necessary to justify the conclusion "flammable liquid."

The charcoal absorption-acid stripping technique described is very useful for working with vapor samples and provides information about the chemical composition of the compounds present. The GC/MS with its sensitivity and specificity is ideally suited to further analyze suspected arson samples when the gas chromatograph does not provide a definite answer.

ACKNOWLEDGEMENTS

I would like to thank John Snyder of our East Lansing Laboratory for the GC/MS work.

REFERENCES

1. White, L.D., Taylor, D.G., Maurer, P.A., and Kuperl, R.E., Am. Ind. Hyg. Assoc. J., 31, 225-232, (1970)
2. Levadle, B. and MacAskill, S., Anal. Chem., 47, 1851-1853 (1975)
3. Levadle, B. and MacAskill, S., Anal. Chem., 48, 76-78 (1976)

FIGURE 1

1 cc HEADSPACE
Sample 6
Apiezon L

Tentative Identification
of Peaks.
1. Benzene
2. Toluene
3. Ethylbenzene
4. Unknown

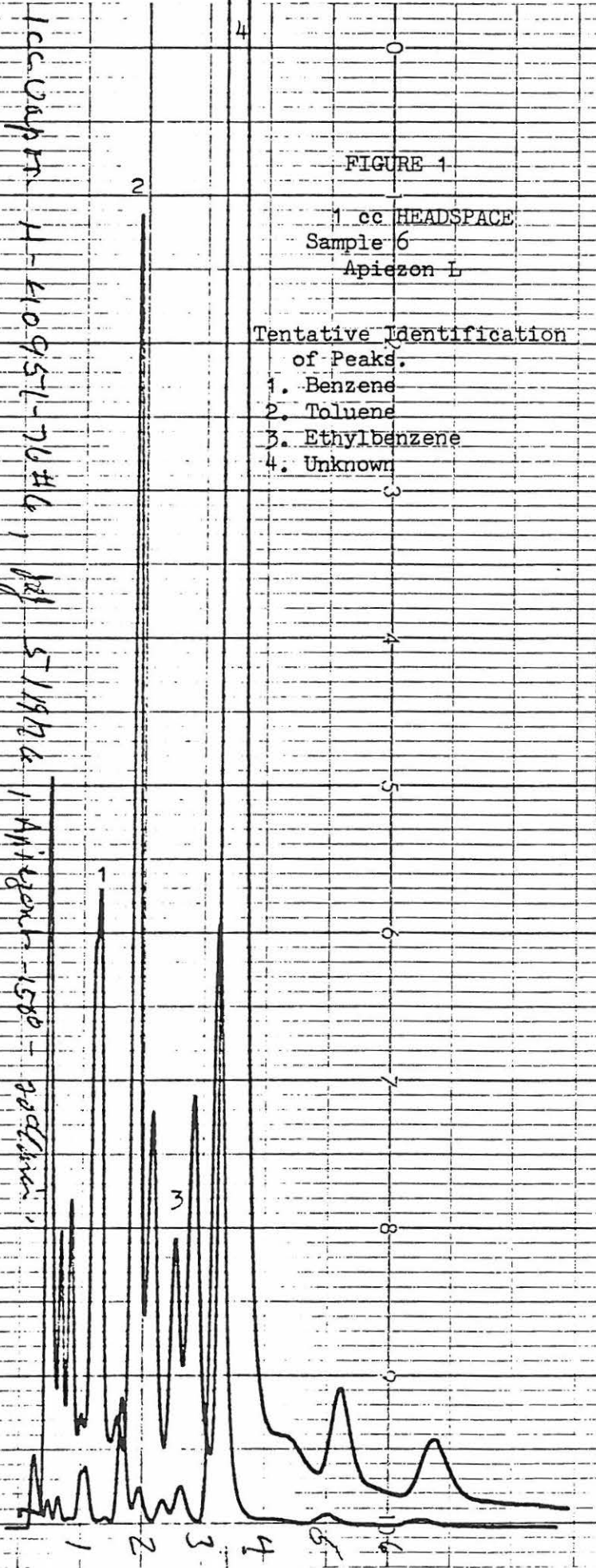


FIGURE 2

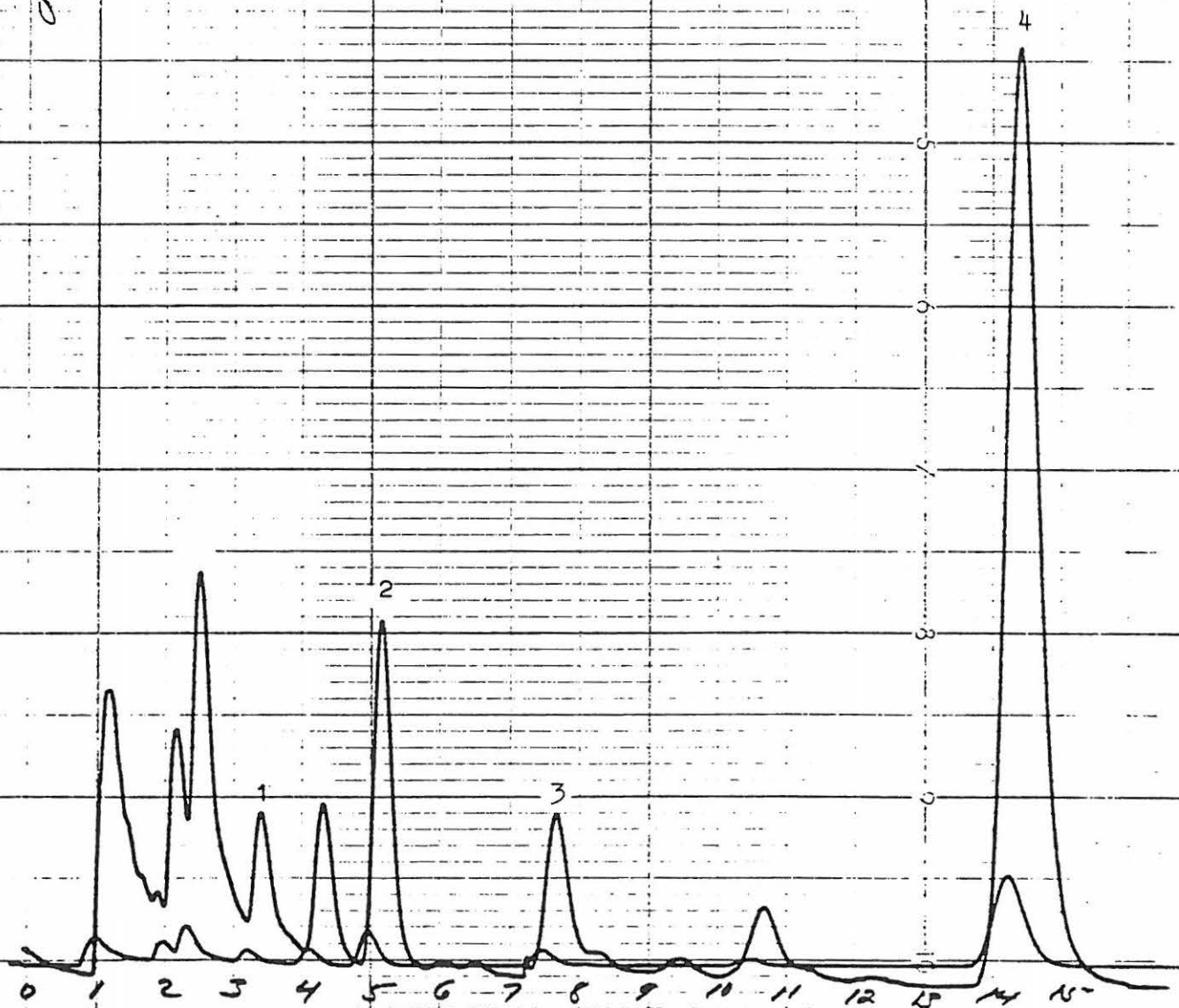
1 cc HEADSPACE

SAMPLE #6

FFAP

Tentative Identification of Peaks,

1. Benzene
2. Toluene
3. Ethylbenzene
4. Unknown



100, 1 carbon

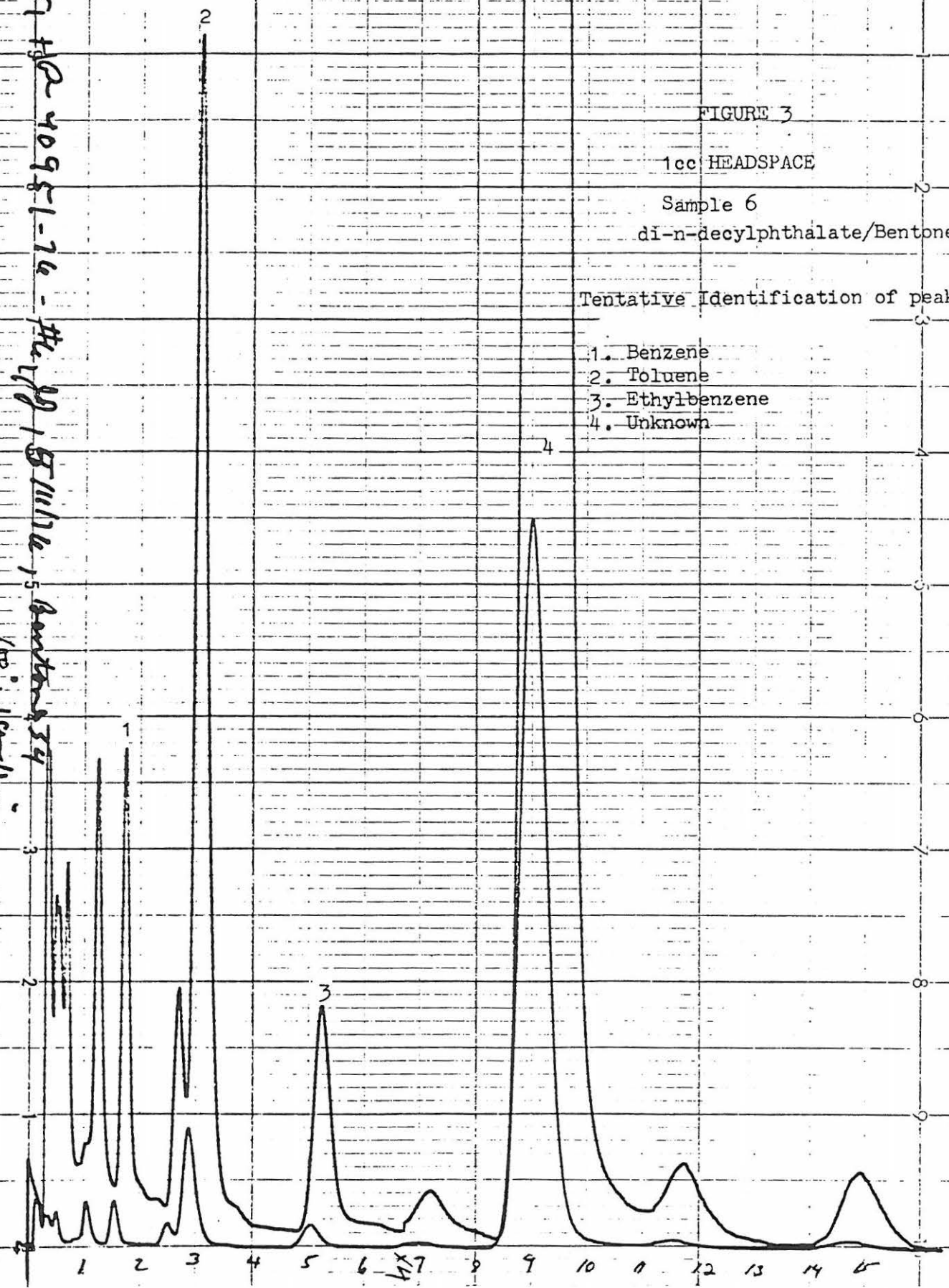


FIGURE 3

1cc HEADSPACE

Sample 6

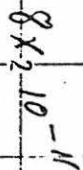
di-n-decylphthalate/Bentone 34

Tentative Identification of peaks

1. Benzene
2. Toluene
3. Ethylbenzene
4. Unknown

1 |ul on FFAP

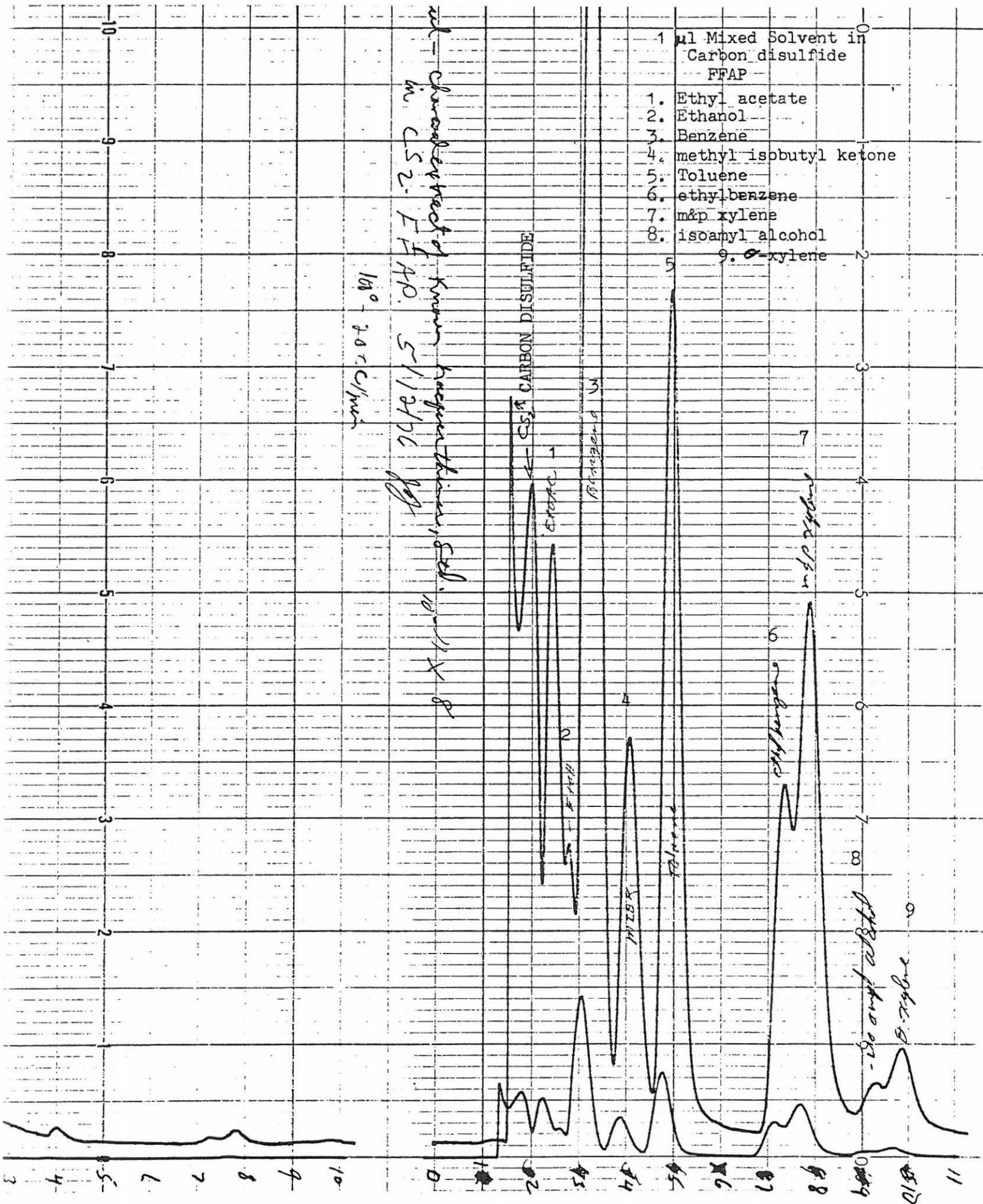
1.	Benzene	
2.	Toluene	
3.	Ethylbenzene	
4.	Unknown	



1.	Ethyl acetate	
2.	Ethanol	
3.	Benzene	
4.	methyl isobutyl ketone	
5.	Toluene	
6.	ethyl benzene	
7.	m&p xylene	
8.	isoamyl alcohol	
9.	o -xylene	N

 $10^9 - 20 \text{ } ^\circ\text{C}/\text{min}$

ul - cleared track of known fugitives, Ed. 10-11 X 8
in CS-2. FH AP. 5/1/27/76 Jof



1 ml Extracted acid stripped Acrylonitrile Std

FIGURE 6

1 μ l mixed solvent standard
after removal of oxygen
compounds by acid stripping.

- 1. Benzene
- 2. Toluene
- 3. Ethylbenzene
- 4. m & p-Xylene
- 5. o-Xylene

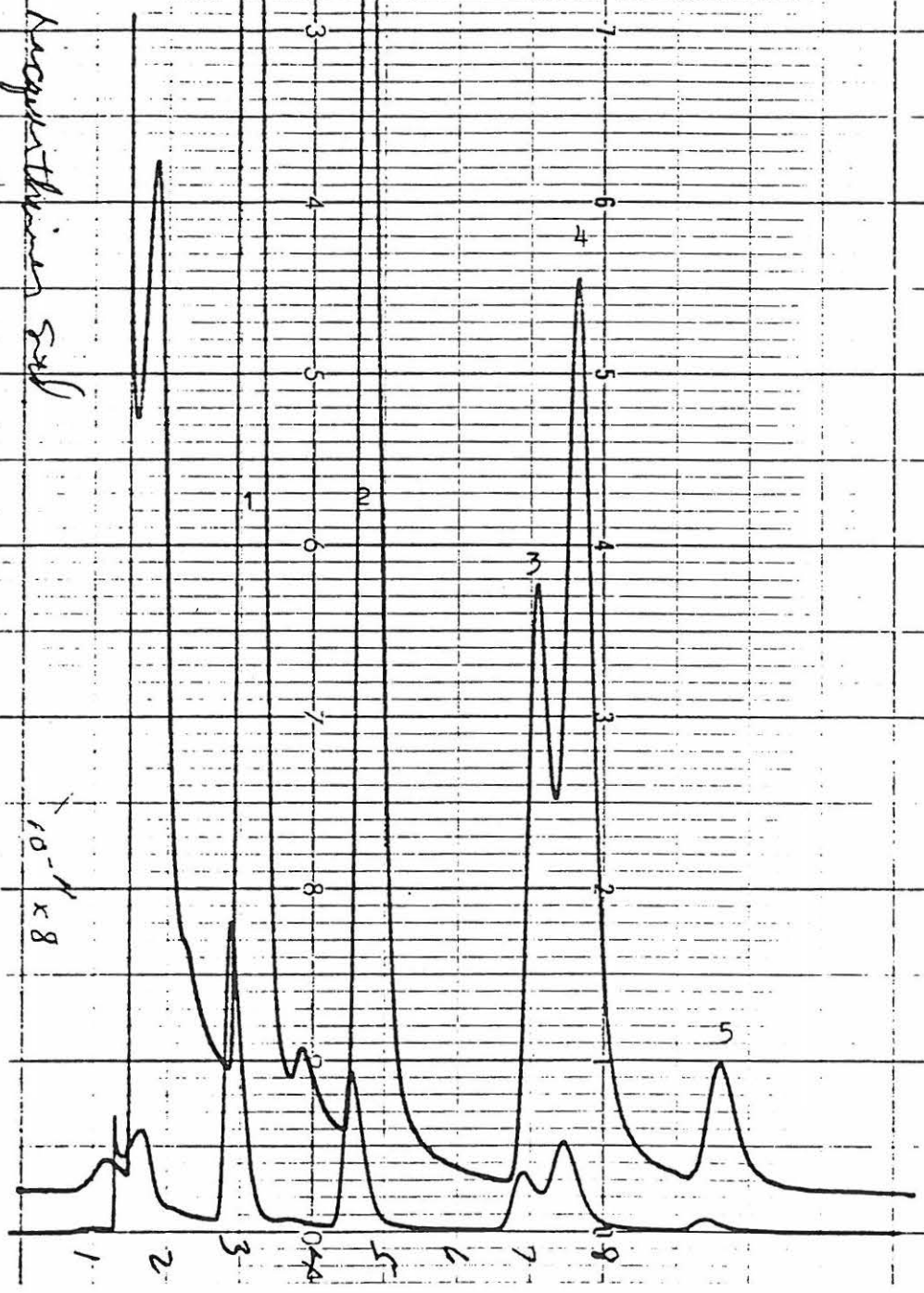
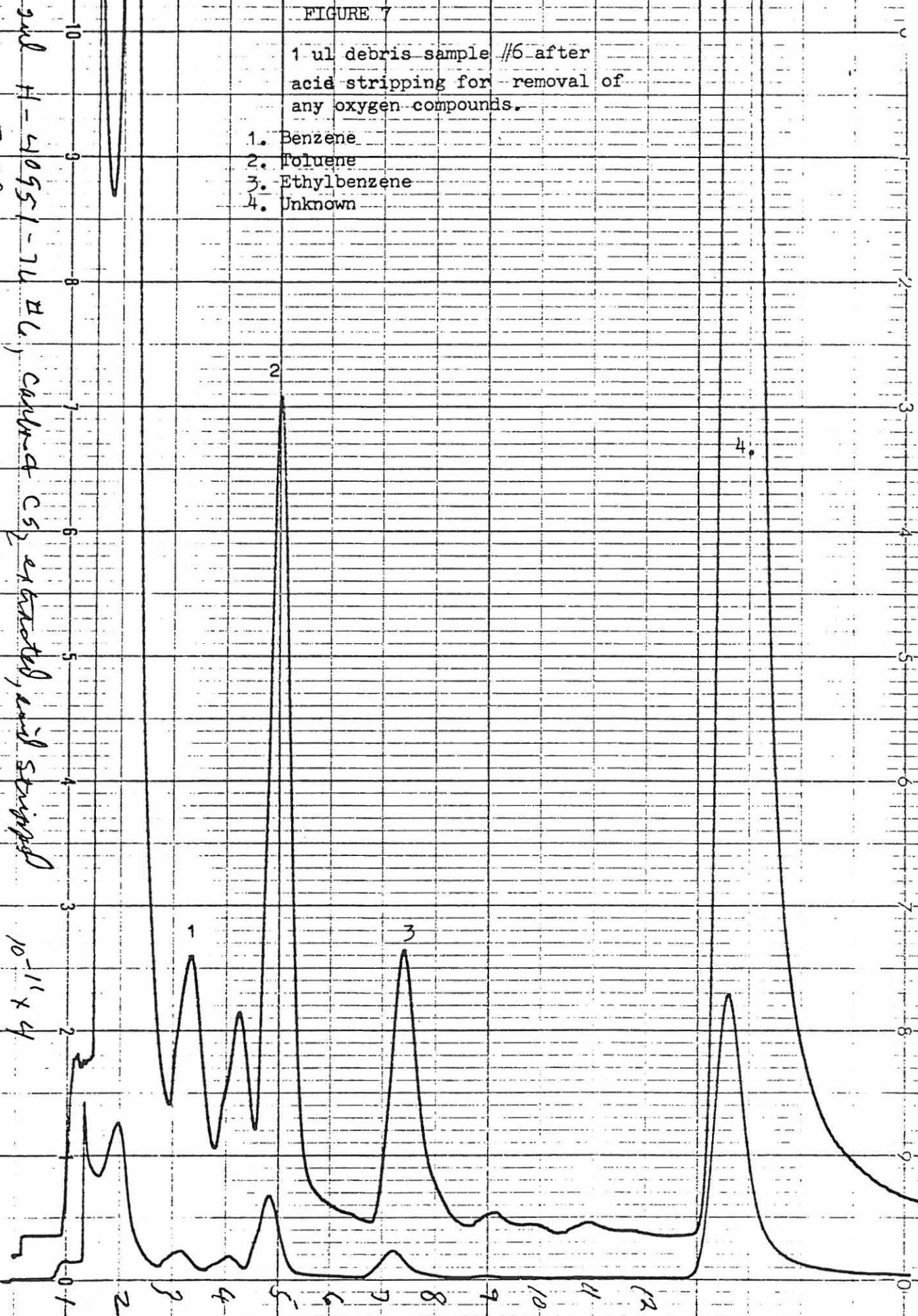


FIGURE 7

1 ul debris sample #6 after
acid stripping for removal of
any oxygen compounds.

1. Benzene
2. Toluene
3. Ethylbenzene
4. Unknown



Jul 4-40951-76 HL, carbon C52 extracted, and stripped

FAAB-1000-100 5/12/76

$10^{-11} \times 4$

FIGURE 8

Mixed Solvent Standard after acid stripping
for removal of aromatic compounds.

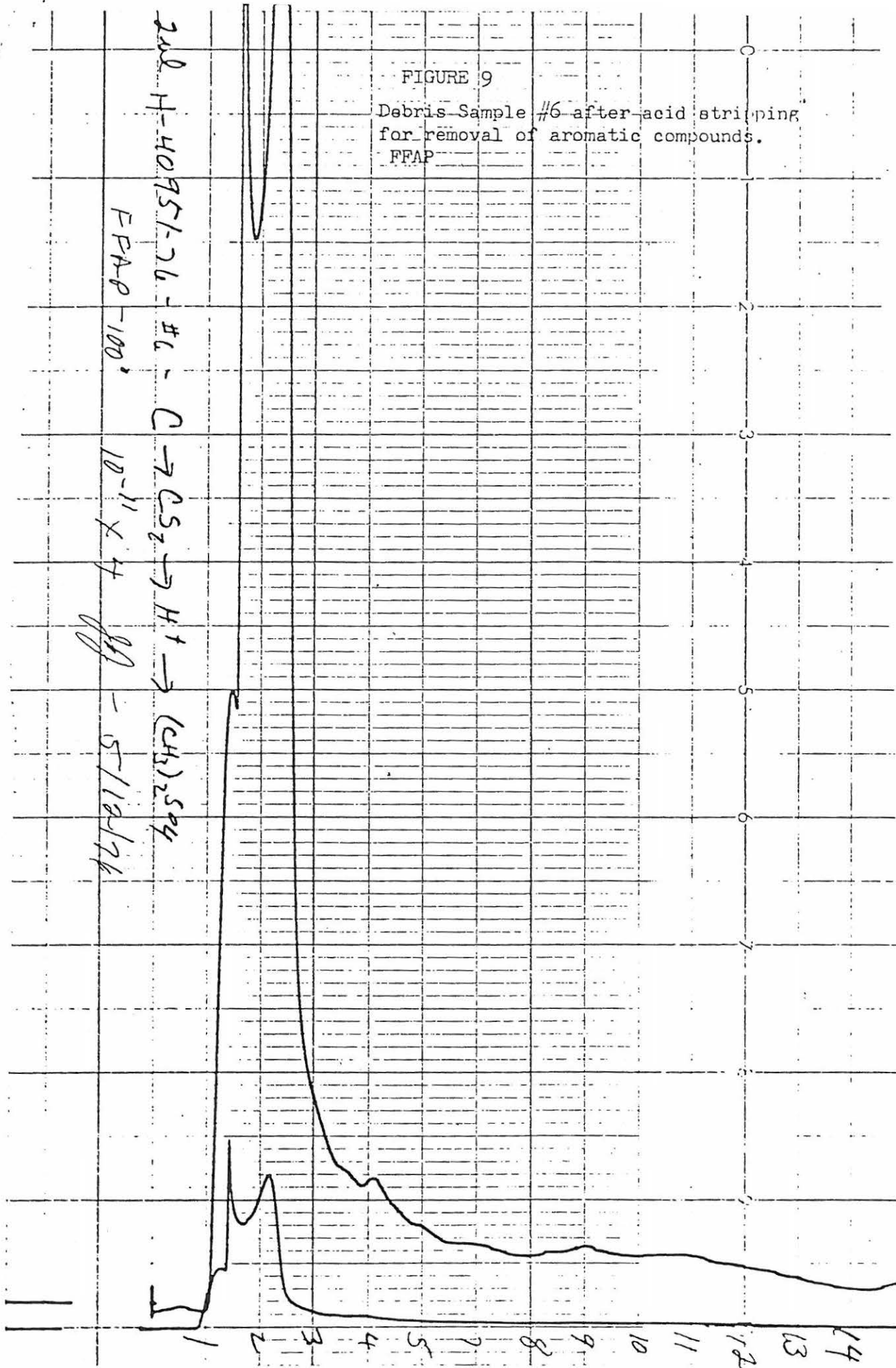
FFAP



oil - heptane 100% acid stripped, Dime Sol standard, 10-11 x 8
FFAP - 100%

FIGURE 9

Debris Sample #6 after acid stripping
for removal of aromatic compounds.
FFAP



Report - California Conference of Arson Investigators - 1979 Annual Seminar.

Submitted by - John D. DeHaan, Information Officer, Calif. Dept. of Justice.

The Information Officer had the opportunity to attend the CCAI meeting held in Sacramento on June 11th and 12th. The following report is a condensation of the presentations of the speakers. Appended to this report is a copy of the program and various handouts from the meeting.

Michael McCrystle, Special Agent with the FBI, provided an outline of the development of case law involving arson evidence. He touched upon a series of landmark cases in the period 1967 to 1978 which bear upon the admissibility of evidence recovered at fire scenes under a variety of circumstances. The first case cited is Camara vs. Municipal Court of San Francisco, 1967. In this case, a housing authority inspector, while conducting an annual safety inspection of a living area, was refused entry by the tenant. The California Supreme Court held that a warrant was required for entry no matter what statutory authority was present. An 'inspection warrant' which functions as an administrative search warrant, can be issued under such circumstances. The probable cause for the issuance of such a warrant can include: elapsed time since last inspection, a preset schedule of such inspections or conditions visible from the exterior dwelling.

See vs. Seattle. A fire chief, while doing a routine fire inspection, demanded entry to a commercial warehouse. He was denied entry by the owner who claimed this was an invasion of his expected rights of privacy. The Supreme Court held that due to considerations of public safety, different standards were expected of commercial areas and that inspections prior to or commensurate with licensing or granting a permit were authorized.

Another important case was U.S. vs. Biswell in 1972, where a sporting goods/firearms dealer gave consent for a search which ultimately revealed the presence of sawed-off illegal weapons. He claimed that the consent search was not legitimate; the U.S. Supreme Court held that the firearms business is closely regulated by the Government for the purposes of public safety, and businesses conducting firearms trade must expect such inspections, and that consent in this case was submitting to a lawful authority.

A similar case was Marshall vs. Barlow where an OSHA inspector doing an inspection by statutory authority in Pocatello, Idaho was denied entry by a plant manager. The manager subsequently obtained an injunction prohibiting re-entry of the OSHA inspector on the grounds that he had certain expected standards of privacy. The Supreme Court held that a plumbing and electrical business such as his was not closely regulated by the Government and that his expected standards of privacy were higher than those for the firearms or liquor trades.

The landmark decision that was expected to have the greatest impact on arson cases is Michigan vs. Tyler, 1978. The order of events was summarized as: 2400 hours - fire in progress, 0200 hours - fire chief arrives and is given containers of suspected flammables recovered from the interior of the dwelling by the fire suppression crews, 0400 - the fire was extinguished. An attempt was made at 0400 to enter the building; however, due to heat, smoke and darkness, no suitable search could be carried out. At that time, all fire and police personnel were secured from the scene. At 0800 hours the fire chief returned with a fire inspector who examined the building with no results. At 0900 an assistant chief and a police investigative team re-examined the building and found evidence of an arson trailer. At that

time, the Michigan State Police Arson Investigation team was requested. That team arrived later than day for examination, photography and collection of evidence at the scene during several subsequent searches. The U.S. Supreme Court held that the returns to the crime scene at 0800 and 0900 were permissible since they were essentially a continuation of the examination from 0400 (which had to be discontinued due to poor conditions). The evidence gathered by the Michigan State Police Arson Team, however, was suppressed. The court held that the prosecution's contention that all rights of privacy by the owner were given up by committing a crime; i.e., the destruction of that property, was not substantiable. On the other hand, an investigation begun and continued for a reasonable amount of time for the benefit of public safety at the scene was permissible. Prior to an extended examination or repeated examinations over a long period of time, a warrant was required to notify the owner of the intended search. The definition of "reasonable period of time" is entirely flexible, depending on the type of building involved, the time and nature of suppression efforts, the complexity of the scene and the extent of emergency situations. The court reinforced the statement that it is the duty of fire officials to determine the cause of fires. The circumstances defining "a reasonable length of time" for an investigator to conduct a warrantless search must be considered by the investigator at the time of the incident. If any doubt, obtain a warrant. Agent McCrystle recommended the November - December 1978 issue of the FBI Law Enforcement Bulletin with its article on crime scene searches for further information.

Another landmark case involving reasonable period of searches was also decided in 1978 with Mincie vs. Arizona. A drug arrest/raid ended in a shootout with an agent, Mincie, girlfriend, and an associate shot. The scene was searched at that time for additional confederates. A shooting team was called in to search the crime scene since an agent and several suspects had been shot. The subsequent crime scene search lasted four days with considerable destruction of the house and its contents. The court held that evidence in plain view discovered during a search for other confederates or in similar emergency situations is admissible. Once the emergency situation ends, however, the requirement for a warrant prior to further searching resumes. Once again, the determination of "reasonable time" and "end of emergency" rests with the investigative officer and his superiors, but are open to interpretation by the courts.

Two additional points were raised in the matter of crime scene searches. If a fire official has obtained an administrative inspection warrant and is conducting a search pursuant to that warrant, criminal evidence in plain view from where the investigator has a legitimate right to be; i.e., where he is allowed to go under the protection of that inspection warrant, is admissible in criminal proceedings. It is interesting to note that an insurance investigator is considered a private citizen in the eyes of the law while working on his own, and not subject to the same Fourth Amendment proscriptions. If he is engaged by a public fire or police agent to work as an associate in an investigation, he becomes legally a deputy agent and is subject to the same laws as police and other public officials.

Other significant cases included: Romero vs. Superior Court (1968) In this case, the search of a burning building for a victim revealed weapons and dynamite. A locker suspected of containing a large quantity of ammunition was opened by the fire department. In emergency situations, Section 13875 of the California Health and Safety Code grants peace officer duties on firemen at the scene. Thereby, this search which was conducted for safety reasons was legitimate. .

In 1970, Swann vs. Superior Court helped define "reasonable rights" of expected privacy. After an April 4th fire, a search conducted on April 24th of the boarded-up, burned premises was held to be illegal and evidence gained therein was suppressed because the boarding up indicated an expectation of privacy. In 1973, U.S. vs. Green allowed the submission of 29 counterfeiting plates which were discovered at the point of origin of a fire by an investigating fire marshal. The fire marshal was acting in line with his legislative authority in conducting a search for the seat of the fire which happened to be coincidental with the plates. In 1974, U.S. vs. Delgado held that a search of a desk drawer was O.K. if the evidence present might be susceptible to fire and water damage.

The next speaker was Roger Venturi of the Attorney General's Office who spoke on the matter of eavesdropping. The key points raised were that visual and aural enhancement of sights or sounds were admissible as long as the activities in question were visible to the unaided senses. The key word here being "unaided". If one uses a high powered telescope to observe activities which would not be visible to the unaided eye, then evidence revealed by that observation would not be permissible. Society has dependence on a reasonable right of privacy; i.e., members of that society can go about their business without fear of being under continual remote observation. Night vision devices such as infrared or starlight scopes are excluded unless there is public advertising of the fact that in a particular area there is an "absence of privacy" as a result of such remote monitoring. Mr. Venturi proceeded to give a series of case-work examples of monitoring situations with the legal interpretation of each situation.

Pete Cleaveland of KGO-TV in San Francisco discussed relations with the public media such as television and newspapers. He revealed that members of the recognized press have a legitimate right to entry to restricted areas such as the police boundaries around, say, a fire scene. However, they do not have the right to violate a recognized crime scene. Members of the press can be ejected from the immediate vicinity of the crime scene for the sake of preservation of evidence. Once members of the press are inside a safety perimeter, their safety is a shared responsibility, This presumably meaning they have a right to some protection by police and fire officials; however, they cannot deliberately take undue risks and expect protection from those hazardous conditions. Mr. Cleaveland warned of the hazards of dealing with the "live" mini-cam. He stressed the hazards of offering off-the-cuff descriptions of a fire scene or suspects which might later prejudice a case. He stressed that the public media could be used in a variety of ways to assist the investigative process. This assistance could take the form of circulation of composite drawings or mug shots of suspects along with requests for information from the general public. The media can also support the educational process of public safety agencies such as bring the magnitude of the arson problem to the attention of the public. Finally, the media can sometimes be of assistance in providing videotape records of crime scenes for later investigative review. He stressed that access to videotape records, films, and scripts was available only by subpoena duces tecum which specifically named the information desired from those files. Access of police agencies to press materials is limited only to material which appeared on the air (including scripts). This does not include records of the reporter such as his notebook or other private materials. If videotapes or films are to be requested, the subpoenaing agency must pay to have it duplicated. The costs of such duplication are not the responsibility of the station.

Frank Wandell, Special Agent, ATF, gave a brief description of the ATF's involvement in arson cases. He stressed that the ATF is concentrating on commercial victims whose property damage exceeds \$100,000 and whose fires indicate the use of an incendiary device. He related that the ATF is not investigating arsons per se;

they are investigating fire cases which use incendiary or explosive devices. He cited the support of the ATF laboratories and their various abilities which closely parallel the abilities of state and county laboratories, with additional expertise on explosive device identification and characterization of flammable vapor explosions. He revealed that in the first six months of 1979 the ATF laboratory at Treasure Island had conducted examination of approximately 62 arson cases. Turn-around time for most cases was around one week, and all submitted cases are finished within two to three weeks from the time of their submission.

Probably the most interesting presentation of the conference was presented by Gordon DaMant of the Department of Consumer Affairs Laboratory in Sacramento. This laboratory is charged with the analysis and characterization of consumer products and their physical and chemical properties. The laboratory does not deal with building structural components. He revealed that of the 10,000 fatal fires per year in the U.S., approximately 75% of all fatalities occur in residences. An estimated 56% of all fatal fires are related to smoking materials, 13.8% related to heaters, 7.5% are related to electrical failures, 7% related to cooking operations or utensils, and an estimated 4.2% related to arson. Due to the magnitude of the involvement of smoking materials in home furnishings in such a large number of fatal fires every year in the U.S., it was decided that studies of cigarette ignition properties of various home furnishings would be of considerable value. DaMant has been responsible for an extensive survey of cigarette products which recorded the time of burning (including the maximum, minimum and overall time of burning for a variety of atmospheric conditions), the extinguishment percentage and the temperatures produced by burning cigarettes. It was revealed that modern production cigarettes contain a complex chemistry of burning and moisture controls in the tobacco and the paper. In addition to such chemical controls, there are variations in the variety and cut of tobacco used. While measuring the maximum temperature produced by a cigarette at various times during its burning, it was discovered that the temperatures reached, as the tobacco burned to the butt end of the cigarette, could be almost 200° higher than those produced elsewhere in the cigarette. In addition to maximum temperature, the maximum heat flux peaks at the end of the cigarette burn as well. This characteristic revealed that temperatures and energies at the end of the burn are critical to the evaluation of fabric-flammability hazard. Some interesting points of reference from the accumulated data included: highest temperature produced: 1440° F (Raleigh); minimum temperature: 760° F, maximum average temperature was 1149° F (Raleigh-King) which was also the fastest burning cigarette. Of the Chesterfield-King cigarettes ignited, 60% went out when left unattended. This can be compared to Camels in which zero percent extinguished themselves when not smoked.

The typical mattress fire is a smoldering fire, started by a cigarette, which very rarely goes to flaming combustion. This can be considered an extreme household hazard since 80% of fire-related deaths in households are due to smoke inhalation. In 1974, the U.S. established flammability standards for mattresses sold in this country. This flammability standard, however, applies to cigarette resistant mattresses. Cigarette-resistant mattresses are not necessarily flame-resistant or even retardant since polyurethane foam mattresses are very resistant to cigarette smoking but are very susceptible, when treated, to flaming combustion. The coverings on upholstered furniture can provide some interesting variations on fire hazards. Some dyes used in printed fabrics can create localized hazards by rendering the fabric much more susceptible to ignition than otherwise would be the case. It should be noted that the heavier the upholstery, the higher the smoking fire risk. Many tests done by Department of Consumer Affairs involve covering a

cigarette with a single layer of paper or cloth. Even this light covering contains sufficient heat to produce burning temperatures on the order of 100° C higher than the same cigarette under the same conditions but left uncovered. According to DaMant, polyurethane is used in 95% of all furniture made in the United States. Unfortunately, there is no way to predict the time frame of smoldering versus flaming combustion in modern upholstered furniture. This time can be anywhere from one hour to as many as nine or more hours. For characterizing upholstery materials, two categories are used. The first category, cellulosic, includes cotton, rayon and linen. This class of fabric is susceptible to ready smoldering but is more resistant to flaming combustion. The second category, thermoplastic, includes nylon, polyesters, acrylics, and wools. As a general case, thermoplastics are more resistant to smoldering but tend to be more susceptible to flaming combustion. A natural fiber, wool, tends to offer the best protection against both classes of combustion.

Since 1976, all polyurethane foam used in furniture sold in California (except decorative pillows and outside furniture) is required to be fire-retardant. Several manufacturers have developed fire-retardant foams, however, they have met resistance in marketing because of their higher cost. Consumers, when offered the choice between cost and fire hazard, often trade off to save themselves that extra little bit, and manufacturers, to be competitive, use the cheaper, more hazardous, material. The fire retardants used in polyurethane may be neutral towards the smoldering properties of the material; however, some may actually make the foam more susceptible to smoldering than would otherwise be the case. Boric acid is used as a most common flame retardant in cotton batting for upholstery and cellulose insulation.

DaMant has also been involved in an extensive program for evaluating fire hazards of jail furnishing with an eye towards improving the safety and security of correctional facilities. This is a rather challenging process because the furnishing must not only be low cost but provide resistance to both smoldering; i.e., accidental ignition, as well as flaming combustion; i.e., intentional incendiarism. The DCA laboratory offers extensive information resources on the flammability of various home furnishings and would be of great potential value to DOJ criminalists. Gordon DaMant can be reached at 3485 Orange Grove Avenue, North Highlands, California 95660. The phone number there is area code (916) 920-6951.

During a lunch break of the conference, an attempt was made to illustrate the hazards of Cadweld welding material. This copper-based thermite-like material has been used as an incendiary in a series of destructive structure fires in northern California. The demonstration at the conference failed to duplicate the ignitability and destructive potential revealed in earlier laboratory tests of this material. Handouts describing the product and the characteristics of its residues were distributed to all in attendance. Sufficient residue was obtained from a limited ignition to provide some exemplars for visual recognition.

Finally, Dr. Corey Clark, a psychiatrist with the Sacramento Child Guidance Center, offered a discussion of child fire-setter types and the potentials for treatment of various categories of fire-setters. He revealed that problem setters have been found to be as young as five to seven years of age and the truly psychotic or seriously neurotic child fire-setters cannot be rehabilitated with any degree of success. He outlined the seriousness of the problem and the difficulties and costs of maintaining treatment centers for the youthful arsonist.

NOTES: C.A.C. TOUR OF UNION OIL RESEARCH CENTER (3-2-78)

Submitted By: Duane Mauzey
California Department of Justice
Santa Barbara Regional Lab.
820 Francis Botello Rd., Goleta, CA 93017

3952 Valencia, Brea (714) 528-7201
Union Oil Manufactures:
Gasoline - leaded premium & unleaded regular
Polymers - latex and hot metal adhesives
Lubricants - automotive and industrial
Asphalt - road and roofing material.

Gasoline - Bill Mallet, additive chemist

Differences due to:

- | | |
|-------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1. Origin of crude
(viscosity
volatility
parafinicity
non HC, mainly S) | 2. Refining process
cracking - breaking C_{12} + to $C_4 - C_{12}$
reforming - make aromatics
alkylation - change olefins to aromatics
hydrocracking - olefins to alkanes
isomerization - straight chain to branched |
|-------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

Crude while in ground,
may lose volatiles and
increase viscosity and lower
volatility, parafin and
S content varies from
one crude to another

olefins polymerize and form gums, are
removed by reforming, alkylation, and
hydrocracking; cracking increases yield
of barrel of crude; all except cracking
also serve to increase octane rating

Gasoline

Union oil uses a single plate distillation for gasoline (ASTM D76)
and takes a 85°F to 410°F cut for C_4 to C_{12} hydrocarbons

Methods of I.D.:

Physical
Gravity (A.P.I. std.)
ASTM D-86 distillation
Reid vapor pressure
Octane

Chemical
FIA (AOS) - this is a method
involving the fluorescence of aromatics
in gasoline, we can't do it except by HPLC
using a combined UV and RI detector

For forensic purposes,
specific gravity is the
most useful physical
property for telling
gasolines apart

Gums - ASTM D-381
Sulfur - % by EDX
GLC - fingerprint on an
8 ft. $\frac{1}{4}$ " packed column of
silicone oil programmed for one hour
continuous rise from -40°C to 540°C

To identify a hydrocarbon as gasoline, a minimum requirement is a good GC fingerprint from a suitable column and program plus an ir spectrum. In order to compare two gasoline samples, one must do the - 40°C to 540°C programmed run, the attenuation is adjusted so that the largest peak does not go off scale, and it is not changed during the run. Pb, Br, and S are determined by EDX. Trace levels (ppm) of various elements are determined by AA or emission spec. Dyes are not useful for comparison as most oil companies use the same dyes from the same manufacturer. Additives vary considerably among refiners.

Pb - EDX or AA

Pb akyl - GC EC

Mg

Dyes - mainly azo, premium is red, regular is orange, union unleaded regular has no dye; the dye is to differentiate between grades of gasoline, not refiners.

Anti oxidants

Corrosion inhibitors - mainly amines

Anti-icers

freeze point - DMF

surfactants

Detergents and disperants - primary and secondary amines, and succinamides

To look at additives, concentrate gasoline to 0.5% of original volume under N₂ and using vacuum; no heat, or gums will form.

Lubricating oils - Rich Mayer

A large variety of lubricating oils are manufactured. They are formulated for specific uses, and therefore differ in makeup, viscosity, and additives. Not all lubricants are hydrocarbons. Motor vehicle oils are dyed according to intended use. There are three basic hydrocarbon oils; Parafinic, naphthalinic, and aromatic. All HC oils have these components, but differ in which one is the major component. For any one particular type of lubricant, they are all much alike. However, when used, they pick up differences due to additive depletion and trace metal and dirt contamination from whatever the oil is lubricating. Characterization of oil, whether new or used, is by viscosity, GC, ir, HPLC, EDX, and emission spec.

Asphalt - Fred Crow

Tar is from coal oil distillation, pitch is the residue. Properly, asphalt is the residue left from the fractional distillation of crude oil.

Distillate: Gasoline and light solvents
Kerosene and light burner oil
Diesel oil
Lubricating oil

Residue: Asphalt

Liquid asphalt

Grade	SC - 0 to 5	Road oils - lowest viscosity
	MC - 0 to 5	Cutback, or mixed, with kerosene
	RC - 0 to 5	Cutback, or mixed, with gasoline (naptha)

Emulsified asphalt - instead of cutback, mixed with a water/soap solution to keep it liquid at lower temperatures and to reduce pollution when spread for use.

Paving, roofing, and pipe joining asphalt are not cutback. Lowest solvent content of the asphalts, highest viscosity, highest asphaltene content, $(C_{89}H_{104}S_3N_2O_2)_2$ as an example of polynuclear asphaltene. Asphalt contains HC, S² less than 7%, N less than 1%. The HC are saturated, parafinic, olefinic, aromatic, heterocyclic, and polynuclear. M.W. is from 300 to 5000.

Asphalt is characterized by the asphaltenes, resins, and oils in it. Asphalt oxidizes with age, increasing the asphaltene content and lowering the viscosity and solvent content. After 5 to 10 years, roof asphalt and road oils become indistinguishable. However, roof asphalt can be distinguished from non roof grade asphalt and road oils (the Williamson Gang) by several methods. One is by the type and amount of solvent present. Roofing asphalt has low levels of heavy solvents. If gasoline or kerosene are present, the asphalt is not roofing grade. Viscosity is a useful comparative technique. It requires about 50g of material. A glass tube is used, and time to pass two marks on the tube is measured for the material at 140°F with vacuum pulling to assist movement, and again at 275°F free flow with no vacuum. Penetration can be measured with a needle and a 100g weight. There is little change in asphalts over a short period, one or two years. One can separate out the solvent and compare that by GC. Ductility at 77°F can be measured, roofing asphalt is near 0 ductility. Also, roofing asphalt has the highest flashpoint index. See ASTM Methods.

The people named above are chemists specializing in particular areas of petroleum chemistry. They can be contacted for help on solving your analytical problems.

A A Notes

October 1979

*John De Haan of the California Department of Justice, Technical Support Unit is planning a side-by-side comparison of portable hydrocarbon detectors or "sniffers" which are in common use by arson investigators or laboratories. If you have any data from such comparisons conducted by your agency or would like to suggest "sniffers" for inclusion in this test, please contact De Haan at:

California Department of Justice
Bureau of Forensic Services
Technical Support Unit
P.O. Box 13337

(916) 322-3968

Since there will be no funds available for purchase of instruments for this test, please supply the name and address of the manufacturer of each device so that they can be contacted to arrange for "evaluation" loaners. Results of this comparison will be published in AAN.

*Interesting Court Cases submitted by Charles R. Midkiff, Chief, Chemical Branch, Scientific Services Division, Department of Treasury, Bureau of Alcohol, Tobacco and Firearms:

Destruction of Potentially Exculpatory Evidence Brings Dismissal of Arson Indictment

The supreme Court of Arizona recently affirmed the dismissal of an indictment for arson where the state had negligently destroyed evidence collected at the scene of the fire. The destruction of the evidence was discovered when the defendant requested that it be provided for examination by defense experts. The evidence which had been seized included a water heater flex pipe, a connecting pipe and two plastic gas caps. At an evidentiary hearing, a police detective testified that no attempt had been made to process any of the items for latent fingerprints but conceded that latents could have been lifted if they were present. The court held that the defendant had been denied due process by the negligent destruction of the seized evidence and stated that tests were not made which could have been made. The court concluded that it cannot be determined later whether or not exculpatory evidence would have been developed. State v. Hannah 583 P. 2d 888 (Ariz. 1978)

AA Notes

October 1979

Interesting Court Cases submitted by Charles R. Midkiff (Con't.)

Failure of Prosecution to provide Laboratory Report is Basis for Reversal of Arson Conviction

A defendant convicted of arson contended that he was entitled to a new trial because he had been denied access to a potentially exculpatory laboratory report. The conviction had been based in part on testimony at trial by a witness for the prosecution that he had smelled kerosene on the defendant's clothes. At the time of his arrest, defendant's clothes had been seized and sent to the state crime laboratory for examination. The laboratory report indicated that no kerosene or other flammable liquids were detected. Before trial, defendant's attorney had filed a motion for voluntary discovery, with which the prosecution had generally complied. The lab report, however, was not provided to the defense, who was apparently unaware of its existence. The North Carolina Supreme Court held that the conviction must be reversed and remanded for a new trial. The court indicated that the report contained information of great potential value to the defendant, particularly in view of the damaging effect of the testimony presented for the prosecution. The court found that even a good-faith failure to comply with the state's discovery statutes required reversal and a new trial. State v. Jones 248 S.E.2d 858 (N.C. 1978)

A A Notes

October 1979

THE AAN WELCOMES THE FOLLOWING NEW SUBSCRIBERS

Henry N. Schladt
Pikes Peak Community College
5675 South Academy Blvd. - Box 19
Colorado Springs, CO 80906

Ventura County Sheriff's Office
Attn: Business Office
501 Poli Street
Ventura, CA 93009

Jimmy Barnhill
Northwest Louisiana Criminalistics
1115 Brooks Street
Shreveport, Louisiana 71101

Harold G. Evans
Chemist
P.O. Box 425
71 Main Street
Springvale, Maine 04083

Department of Law Enforcement
Bureau of Scientific Services
P.O. Box 437, Highway 51
DeSoto, IL 62924

Thomas J. Hopen
Trace Evidence Coordinator
Alabama Dept. of Forensic Sciences
P.O. Box 2344
Montgomery, AL 36103

Richard W. Henderson, Ph.D.
Francis Marion College
Florence, South Carolina 29501

Raymond J. Davis
Consultant in Forensic Science
4275 Sorrel Way Northeast
Bainbridge Island, WA 98110

Albert J. Graziloli
Pikes Peak Community College
5675 South Academy Blvd. - Box 19
Colorado Springs, CO 80906

Accessions Department
British Library - Lending Division
Boston Spa, Wetherby
Yorkshire LS 23 7BQ, England

David M. Larson
Commanding Officer
Negaunee Forensic Laboratory
180 US 41-E
Negaunee, MI 49866

Allan E. Gilmore
Director of Crime Laboratory
Sacramento County Crime Laboratory
4400 V Street
Sacramento, CA 95817

Division of State Police
North Regional Laboratory
Route 46
Little Falls, NJ 07424

Division of State Police
South Regional Laboratory
Route 30
Hammonton, NJ 08037

J. Wesley Sammis, P.E. C.S.P., President
LoCon, Inc.
P.O. Box 596, Suite C
Haddonfield, NJ 08033

Don Hicks
C.G.S. Dev. Lab
121 South Frazier
Conroe, TX 77303

A A Notes

October 1979

Ladis Barr
C.G.S. Dev. Lab
1212 South Frazier
Conroe, TX 77303

Ms. Lindy Bond
AID Consulting Engineers, Inc.
2828 Merrell Road
Dallas, TX 75229

*The third and last part of the series of articles by Dr. John I. Thornton will appear in the next issue of the AAN.

*Have a happy holiday season from the staff of the AAN.