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ARSON ANALYSIS NEWSLETTER (AAN)

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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.

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"The enclosed report is a summary of the proceedings held in November 1978 for the criminalists in the Department of Justice Laboratory system who are responsible for arson cases... It should be noted that the opinions expressed in this report are those of the participants alone and not of the Department of Justice system."

John D. DeHaan
Department of Justice
Division of Law Enforcement
Investigative Services Branch

REPORT ON CONGRESS OF CRIMINALISTS: ARSON

The third Congress of Criminalists was held on November 21, 1978 at the Advanced Training Center. The topic was Arson. In attendance were the following individuals: Bill Johnston, Redding; Hubert Frank, Salinas; Mike Potts, Santa Barbara; Ray Davis, Santa Rosa; Lansing Lee, San Rafael; Rich Lynd, Modesto; Lou Maucieri, A & T; and John DeHaan, Coordinator. In addition, two members of the Alcohol, Tobacco, and Firearms Laboratory in San Francisco, Elliot Byall and Steve Roberts, were present to provide additional information. Due to prior court commitments, Gary Cortner of Fresno and Bob Baker of Sacramento were unable to attend the morning session but were present in the afternoon. The objective of the meeting was the improvement of the accuracy and effectiveness of arson analysis in DOJ laboratories. Discussions on virtually all phases of arson investigation were lively and well participated in.

It was the consensus of the gathered criminalists that they most frequently encounter arson when it is a part of a cover-up of, or in conjunction with, another major crime, usually murder. These cases therefore represent the standard conditions under which the DOJ criminalists find themselves involved with arson investigation. As participants, it was felt that the crime scene security and responsibility were the most critically underdeveloped areas. The crime of arson classically falls in the gap between fire investigation and police investigation and, as a result, the police and fire authorities themselves are often in disagreement as to who maintains control of a fire scene as it is being processed. It was suggested and agreed upon that any steps crime laboratory personnel can take to establish and maintain a working contact between fire and police personnel in their own jurisdiction would be beneficial.

It was the universal consensus of the participants that, due to the complexity of arson scenes and the specialized knowledge required for their proper interpretation, the general practioner is not qualified to properly conduct an arson scene investigation. The criminalist is serving the best interests of the investigation when he is present to act as a technical advisor on the collection and preservation of the evidence involved. In this role he can be expected to provide valuable assistance to the investigators in the possible interpretation of items of evidence as they are encountered at the scene. Those individuals who have participated in arson scene investigations agreed that proper lighting facilities are virtually non-existent. It should be recommended that the crime scene capabilities of every laboratory include some form of auxiliary lighting beyond battery powered flashlights to aid in the examination of arson-related crime scenes.

The group then turned its attention to the types of materials encountered, with special emphasis on unusual accelerants. Gasoline was the most commonly encountered accelerant, with kerosene and paint thinners generally being the "second choice". School fires have involved the use of dust mop dressing (from the janitor's supplies), rubber cement, and various glues. One lab reported the use of the 60% alcohol solution which is sold as a hospital-strength commercial disinfectant. Amyl nitrate was reportedly used in a printing shop fire; plastic cleaner and dye solvents have been detected in shoe store and shoe repair shop fires. Auto body shop fires involve paint thinners, cleaning solvents and body filler solvents. Office fires have been started from copier toners and alcoholbased mimeograph fluids. The use of rubber innertubes has also been reported. They are readily available and, once ignited, are very difficult to extinguish.

They may be recognized as a charred, crusty mass surrounded by a heavy liquid pyrolyzate around the perimeter. Styrofoam and hydraulic brake fluid have also been involved.

It was commented here that virtually all the arsons we are involved in are started by amateurs using what is at hand. The "set" of the professional arsonist may be so subtle that its remains are not detected. He will often select a means for maximum damage in the minimum amount of time rather than make do with the hap-hazard effects of available accelerants.

The ATF reports a larger incidence of 'Molotov cocktail' fires than the DOJ labs. This is thought to be due to the ATF's involvement with device-oriented fires rather than the general population of arson-suspected fires in the DOJ service area.

Evaluations of various packaging methods and the problems and the advantages of each were discussed. Many labs reported persistent problems with getting submitting agencies to package evidence properly. They report the continued use of the wrong materials including paper and plastic bags. It was determined that metal paint cans remain the most useful container for arson debris. One participant reported the detection of volatile thinner components from the alkyd paint liner in one brand of can, and it was recommended that spot checks be conducted on stocks of these cans maintained by laboratories for this type of case. In some instances, it would be worthwhile to also check the cans used by major submitting agencies in each area. It was pointed out the the can-type packaging container offers the best means for the investigator to secure, seal, and label the exhibits of interest. One can impress investigators with the fact that the sealed, labeled can holds up very well in the judicial process. This is, of course, of great interest to the investigators and can be stressed as a major selling point to get them to use proper containers.

It was the consensus of the group that the crime laboratories' best impact on arson investigation can come at this juncture. It was the recommendation of this group that participation of the laboratories in the preparation of various types of training packages specifically designed for, or aimed at, local law enforcement and fire investigators would be most helpful. Short packages on identification and collection of physical evidence at arson scenes could be very beneficial as long as they show the chain of development of the evidence, including how the analysis is conducted in the laboratory and what the shortcomings are in the various packaging techniques. These training packages might be in the form of training manuals such as that currently being prepared by the California District Attorneys Association, videotaped presentations such as that currently being prepared by the Yolo County Sheriff's Department, or slide tape packages such as those prepared within our own department in the past.

In regard to packaging, labs should continue to evaluate alternative methods. The availability of nylon bags from Soplaril Ltd., in England, was reported some time ago in TIE LINE. The ATF reports that polypropylene bags are usable and are available here in the U.S. The cooking type, "browning bags" available in supermarkets, should be checked for their usefulness as packaging materials.

Since corrosion problems have been reported with long-term storage of wet debris in metal cans, it was suggested that criminalists place the debris in a plastic bag before repackaging it in it's can. The distillate or extracted material can be sealed in an all-glass ampoule, labeled, and packed into the can for ready reference at later proceedings.

All of the individuals who had occasion to use various electronic vapor detectors recorded problems with false positives as well as false negatives. The range of false results and the variations of sensitivity limits between instruments has posed some problems for the investigators in the past. Publication and circulation of evaluations of various types of "sniffers", (whether those evaluations are carried out in-house or by other laboratories), should be considered. would enable all labs to accurately inform investigators as to the pros and cons of the use of various types of detectors. Extremely sensitive detectors such as the Grace Industry's "Electronic nose" have provided users with some serious false-positive problems. The investigator brings in some really "hot" debris, only to be told that no fuels or accelerants were detected, only combustion by-products. Nearly all detectors react to combustible hydrocarbons of all types and the nature of their responses may have to be explained to investigators supported with lab feedback on sample quality and specific results. In some cases, the investigators have to be cautioned to zero their detectors in clean air periodically and to check the debris trap on the JW detector. Such sniffers are available in many of the DOJ labs but they have not been found to be of much use in screening samples of debris prior to further analysis.

Virtually all of the labs present use some form of heated headspace sampling for screening of arson debris specimens. It was agreed that really high temperatures (greater than 100°C) create more problems than they solve (i.e., volatilization of oils or asphalt residues). If a material is not appreciably volatile at 50 to 75°C, it probably won't be of much use as an accelerant. A variety of opinions was offered on the volume to be sampled from a debris can; most labs use 1 to 3 cc to ensure a representatively large sample. One analyst maintains that such large injection volumes disturb the GC column excessively and uses 100 microliters withdrawn from the can with a gas-tight syringe. Several labs use disposable 3 cc plastic syringes which have been prewarmed to preclude condensation upon drawing of the headspace. Others use all-glass syringes (such as Precision Scientific's 5 cc gas sampling syringe, \$35) with storage in a heated oven or above ascarite to insure cleanliness. It was suggested that syringe purity checks of reusable glass syringes or random checks of the disposable plastic syringes be carried out routinely. It was pointed out that if one draws a sample of room air prior to the insertion of the needle into the septum of the debris can, when the plunger is depressed, any small plug of septum rubber in the needle is blown into the can and does not offer blockage to the withdrawal of the headspace.

Certain precautions as to the interpretation of negative results or marginal results were discussed at this time. A number of the participants claimed reliance on heated headspace comparisons of the characterization of certain classes of petroleum distillates; i.e., gasolines and lacquer thinners. This was especially true when the heated headspace revealed the presence of large concentrations of a readily identifiable product such as gasoline. At this point, some participants would compare the headspace results with those of headspaces of various suspected compounds while others would attempt various extraction procedures. Although headspace sampling GC techniques have certain shortcomings. it was the consensus of the group that for the characterization of certain types of volatile accelerants, the technique was adequate for investigative purposes. As a screening test, the headspace technique is extremely valuable because it tells us how much material we might be able to recover from a debris sample and it also tells us if we have a simple one or two component mixture which might be an alcohol- or acetone-containing compound. In these cases, the headspace results can be used to help the investigator decide as to which recovery method is going

to be the most successful for the material in question. If further extraction is pursued, the headspace results are a good back-up in case the extraction, for some reason, fails to recover any volatiles.

There remains considerable reliance on organoleptic characterization of volatile materials. The potential health hazards involved with the inhalation of vapors from certain burned synthetic materials (which are becoming more and more common) may warrant reduction of reliance on this technique.

Various techniques for sample recovery were discussed. Most individuals rely on simple steam or water distillation. The advantages of vacuum distillation for certain types of samples or substrates were discussed. The technique has been found to be of use where large, bulky samples (which were not amenable to containment in a boiling flask) contain relatively small quantities of suspected accelerant. It is also of use where the substrate would not withstand boiling in a water solution. It was recommended that some of the laboratories look into building or acquiring capability for vacuum distillation. With regards to the use of liquid nitrogen for cold traps in vacuum distillation, the ATF laboratory warned about using liquid nitrogen, for without the proper precautions, one can condense liquid oxygen in the cold trap and then suffer the risk of some spectacular accidents when restoring the condensate to room temperatures.

The ATF reported on the use of total vacuum distillation evaluated by their Cinncinnati lab. This procedure removes all volatile materials from a specimen and causes their absorption on cocoanut charcoal. The charcoal is then washed with carbon disulfide and the extract injected on the GC. Several laboratories reported the use of solvent extraction using pentane or hexane, however it has not been found usable in most situations. ATF reportedly has used carbon disulfide which extracts all of the materials of interest. Unfortunately, it also removes the tars and oils which produce severe background contamination in subsequent GC injections. When one has a relatively clean material, such as gravel or glass as a substrate, carbon disulfide extraction is quite usable.

All of the laboratories reported reliance on gas chromatography for the characterization of products by headspace sampling alone or by headspace combined with liquid injections. Only four of the laboratories present reported the routine use of the log electrometer for recording GC data. One laboratory reported difficulty using the log function of the HP integrator with nonreproducible false positives being created. None of the other laboratories reported this problem. The log function eliminated problems of sampling size and allows a high throughput of samples without the necessity for repetitive injections with variations of the attenuator setting.

The group compared their basic instrumentation complement among themselves and discovered that most laboratories use the same general equipment (6-12 ft, 1/8" glass or stainless columns, using SE-30, OV-1, OV-101, or SP 2100). To date, no one has used SCOT columns with any regularity; capillary columns were used in Redding and the evaluation was that the column had a great deal of potential. Fresno has had a capillary column and splitter for some time. They were very pleased with the results it was producing; however, the column is very dated at the moment. The ATF reports using an SP-2100 column with good success; they report a very good resolution and a much longer service life over SP-1200. They also use a $2\frac{1}{2}\%$ Bentone $34 + 2\frac{1}{2}\%$ SP-2100 on Supelcoport. This packing produces excellent separation of the three xylenes and ethyl benzene, which are added as octane boosters for gasoline. Under the conditions used in the ATF lab in San Francisco, those four compounds come out as a discrete "fingerprint" group separate from the alkanes in that region.

Lou Maucieri had been in touch with Steve Allen of the FBI lab; they report using a SCOT column of Dow 550 silicone oil, 100 ft in length. The column temperature is programmed from 60 to 145°C. They report a total of 75 to 125 peaks in automotive gasoline. GC/MS is used on a few cases. The additive packages are used only when comparing two liquid samples and this involves comparison of the lead alkyl packages. Phosphorus compounds are not yet characterized. Head-space sampling is used to detect single component accelerants such as alcohol. If the headspace is negative, then the exhibit goes to Latents. Steam distillation is used for some exhibits. Solvent extraction using nanograde pentane is a routine means of extraction. They also report the use of the 200 ft. Squalene capillary GC column for comparing two liquids. This column is run from 0 to 105° C, yielding about 200 peaks for automotive gasoline.

Due to the favorable results achieved by various research application laboratories with capillary chromatography, it was the consensus of the group that labs in our system should investigate capillary GC capability. It was felt that a splitter in capillary column for a total investment of 500 to 700 dollars would be a worthwhile investment for labs who do a great deal of this type of work. It is unfortunate that the one laboratory that has had this capability now finds themselves unable to replace an aged and probably exhausted GC column.

One laboratory has experimented with subambient programming down to -60° C. It reported that it can be a useful technique; however, a tank of CO₂ lasts a maximum of two days. One has to be certain of getting the special cylinder with the dipping tube or delivery tube down to the bottom of the tank since the delivery system has to pick up the liquid at the bottom of the tank, not the head-space gas in the cylinder.

With regards to other analytical techniques, the infrared analysis of distillate is only used in a few labs. The additional information gained from this analysis is still open to debate; it was the general consensus, however, that this sort of information could be useful in supplementing the analyst's knowledge about a particular sample, particularly in regards to comparisons with suspected source materials. The EDX analysis of residues for lead and bromine from leaded gasoline is not in routine use. Several laboratories use refractive index for characterization of the materials, however, it was pointed out that the refractive index is largely dependent on the volatility of the sample. Automotive gasoline can vary in refractive index from about 1.43 to as much as 1.6, depending on the extent to which it has been allowed to evaporate.

Bill Johnston reported on the work he did with a Varian gas chromatograph which had been fitted with a sulfur flame photometric accessory. He was able to conduct a limited sampling of gasolines and was able to discriminate most of the gasolines present from one another. Very little information is available as to the source of sulfur compounds in gasolines or their variability from various crude oil sources or refineries. It was recommended that this technique be evaluated further for possible application in the comparison of lubricating oils. Until this time, very little analytical capabilities are available for these comparisons. It was pointed out that this same type of detector can be used to detect phosphorus and sulfur compounds simultaneously as well as lead alkyls. This versatility may offer some encouragement for its further forensic evaluation since one detector would be usable for both sulfur and lead content characterization.

Hubert Frank reported on his development of the electron capture detector and its use in characterizing lead alkyl and halogenated scavenger profiles of various pump gasolines. Its instances of casework application showed that it can offer some invaluable information, especially where source-origin comparisons are to be made.

Considerable variations were found in the levels of interpretation of volatile accelerant residues. It was the general consensus that the peculiar properties of gasolines permit their ready characterization by gas chromatographic technique with negligible risk of misidentification as long as the gasoline is not greatly weathered or has combusted to any large extent. The characterization of other petroleum products is open to debate, however. Because none of the other commonly encountered petroleum distillates; e.g., paint thinner, charcoal lighter, petroleum ether, etc., have a precisely formulated pattern of contents, as does gasoline, their specific identification is very limited. One can characterize these products; however, due to the multiplicity and probable overlap of various products with similar properties, any such characterization may not be completely specific without a complete scheme of analysis including infrared, elemental analysis, refractive index, specific flashpoint and other properties. It was felt by several of the participants that the time involved in the isolation and complete characterization of such products is not warranted for most investigative reports. The ATF, for instance, will identify a volatile material as a gasoline on the basis of headspace but will only characterize other materials as petroleum hydrocarbons.

Reservations were expressed regarding the identification of the material, on the basis of its GC properties only, as a "flammable". It was pointed out that a volatile hydrocarbon such as methylsalicylate is non-flammable, and that there are probably other similar materials. This is one of the reasons behind the isolation of a liquid distillate and its identification on the basis of all of its chemical and physical properties. The California Penal Code defines a "flammable material" as that having a flashpoint not over 150°F. As a rule, we cannot give that information because we do not have the equipment to do accurate flashpoint determinations. At the present time there are no public laboratories in northern California who have the capability of doing subambient flashpoint testing. This is another field for further investigation with the suggested acquisition of at least one subambient flashpoint testing device.

It is important to note that a negative headspace reaction means that no volatiles were detected. This does not necessarily exclude the presence of solid residues of chemical incendiaries or non-volatile flammables such as diesel fuels. Each container should be opened and the contents visually examined for the presence of fragments of cloth, wick, or Molotov cocktail, non-volatile oils, or even debris that has been packed in a plastic bag and then subsequently sealed in a metal paint can.

It was felt that gasoline will continue to be the most commonly used volatile accelerant. Increasing environmental pressure, however, will eventually bring about gasolines with no heavy metal additives or halogenated compounds. At that time, the forensic scientist will be forced to rely on more sophisticated techniques for detection of aromatic compounds and the detection of sulfur, phosphorus, and possibly nitrogen-containing compounds as a means of characterizing such fuels. A number of laboratories present reported the characterization and separation of gasoline dyes as a means of comparing gasolines. At one time it was felt that such dyes would be characteristic for a particular manufacturer and particular grade of gasoline. This does not appear to be the case at this time, with many gasolines of different manufacturers using very similar dye packages. The marketing pattern and production pattern of gasolines in the northern California area are such that the dyes cannot be used as reliable indicators of manufacturer origin or time of production. The presence of the dyes themselves has been used to strengthen the identification of the petroleum product as a gasoline because most other petroleum distillates will not contain such dye packages.

Along the lines of instrumentation, one of this group's recommendations is that evaluations of procedures new to the forensic scene such as the nitrogen-phosphorus, ECD, and flame photometric detectors, be carried out and their results be circulated among the interested laboratories. In addition, evaluation of other variations like capillary columns or SCOT columns should be continued. Encouragement should be offered to line personnel for the investigation of alternative procedures for the detection and characterization of arson accelerants. These alternative procedures may allow laboratories to improve their capability in the comparison of suspected common-origin gasolines. This sort of comparison remains a frequently requested service. For those DOJ personnel in regional and satellite labs, a listing of the capabilities of each core laboratory was requested. This listing would include a description of the technical resources on hand including specialized equipment (NP, ECD, GC columns, and the like) as well as literature resources or bibliographies. The development of training resource materials aimed at investigators should be considered a top priority item. These resource materials may include videotape or slide packages which would guide the investigator in improving his use of physical evidence and its interpretation. Contributions of time made in such training efforts should be considered for compensation. Subscription to, and participation in, relevant publications such as the Arson Analysis Newsletter should be encouraged. type of publication offers faster and more complete circulation of relevant arson information than the normal forensic literature sources.

The idea of quality control specimens of various types was well received. The availability of proper QC samples would be extremely helpful to all individuals involved in arson accelerant analysis. It was suggested that "open" studies with subsequent publication of the chromatograms and other analytical data would test the "technique" capabilities of various laboratories, and not just the individuals in particular labs. These "first level" specimens would provide inter-lab comparisons of the instrumental capabilities; i.e., whether Sacramento's Victoreen is giving results equivalent to Fresno's H-P and Redding's Perkin-Elmer. Samples of various types of accelerants, either raw or in various substrates, could then be circulated for further testing. This bi-level interlaboratory review could pave the way for a voluntary standardization of techniques and the general improvement of the quality of information all labs could provide.

JOHN D. DE HAAN Criminalist

GAS CHROMATOGRAPHIC ANALYSIS OF NONVOLATILE ACCELERANTS

By

Ronald N. Thaman

The three most widely used methods for flammable liquid recovery

Systems Engineering Associates

include: 1) headspace analysis 2) steam distillation and 3) solvent extraction. All three methods have no difficulties when the suspected flammable liquid is volatile (i.e. gasoline, paint thinner etc.). The more nonvolatile the flammable liquid, the greater the differences between the three methods of analysis. Headspace analysis (ambient or heated) suffers when flammable liquids such as fuel oil and beyond are suspect. The higher molecular weight components of fuel oil are not volatile enough to become detected by gas chromatography. 2 Steam distillation has certain advantages over the various headspace procedures but still has an upper limit depending on the vapor pressure of the hydrocarbon of interest. "For sucessful steam distillation, the insoluble substance to be distilled should have a vapor pressure of at least 5-10 mm at 100°C. Estimated vapor pressures of normal hydrocarbons are as follows at 100°C: nC_{10} 77mm, nC_{12} 19mm, nC_{14} 4.5mm and nC_{16} < 1mm. Generally steam distillation suffers from recovery when the hydrocarbon range goes beyond C15. Solvent extraction does not suffer from the problem associated with headspace or steam distillation methods for fire debris analysis.

 ${\rm nC}_{30}$ and beyond. Although some contaminants arise from solvent

A solvent such as ethyl ether is soluble with normal hydrocarbons up to

extractions, the use of differential infrared spectroscopy and suitable control samples eliminate any of the problems normally associated with solvent extractions. 5,6

The use of a solvent such as ethyl ether for solvent extractions will leach out of the fire debris hydrocarbons up to carbon lengths of C₃₀ to C₄₀. It is therefore essential to choose a temperature program for the gas chromatograph which allows for the most nonvolatile components of the residue to elute. Our laboratory uses the following temperature program: 80°C hold 10 minutes, 80°C to 325°C at 10°C per minute, hold 325°C for 30 minutes. The long hold time at 325°C allows the column to become "clean" for the next injection.

During the past year our laboratory has noticed an increasingly larger number of nonvolatile residues on fire debris samples.

Many of the residues can be explained as mineral oils which are expected on certain types of fire debris. A brochure on mineral oil 1 lists some of the uses of the product: air filters, baby oils, canning industry, cosmetic creams, food processing, hair preparations, industrial dust control and textile processing oils. It is not uncommon to find mineral oil on fire debris consisting of carpet.

A most interesting nonvolatile hydrocarbon accelerant, paraffin, has been found recently on a variety of samples. Paraffin is a mixture of solid hydrocarbons having the general formula $C_nH_{2n} + 2$ and having a flash point of 390°F (C.C.) and boiling point greater than

700°F. 8,9 The uses of paraffin range from lubricants, candle making, home canning, and cosmetics to fire place logs. Figure 1 shows two chromatograms of paraffin one from Parowax and the other from a Sterno Log. Parowax is used for candle making and home canning; and if heated above its auto-ignition temperature, (473°F) it will self ignite. The other use of parowax which is most interesting in fire debris analysis is tis use in prepared fire-place logs.

Headspace analysis will not discover paraffin or mineral oils that have been used as accelerants. Steam distillation may recover some of the lighter ends of paraffin but would certainly have difficulty in recovering C_{40} hydrocarbons. Further research is continuing in analyzing various fireplace logs to attempt a brand name determination.

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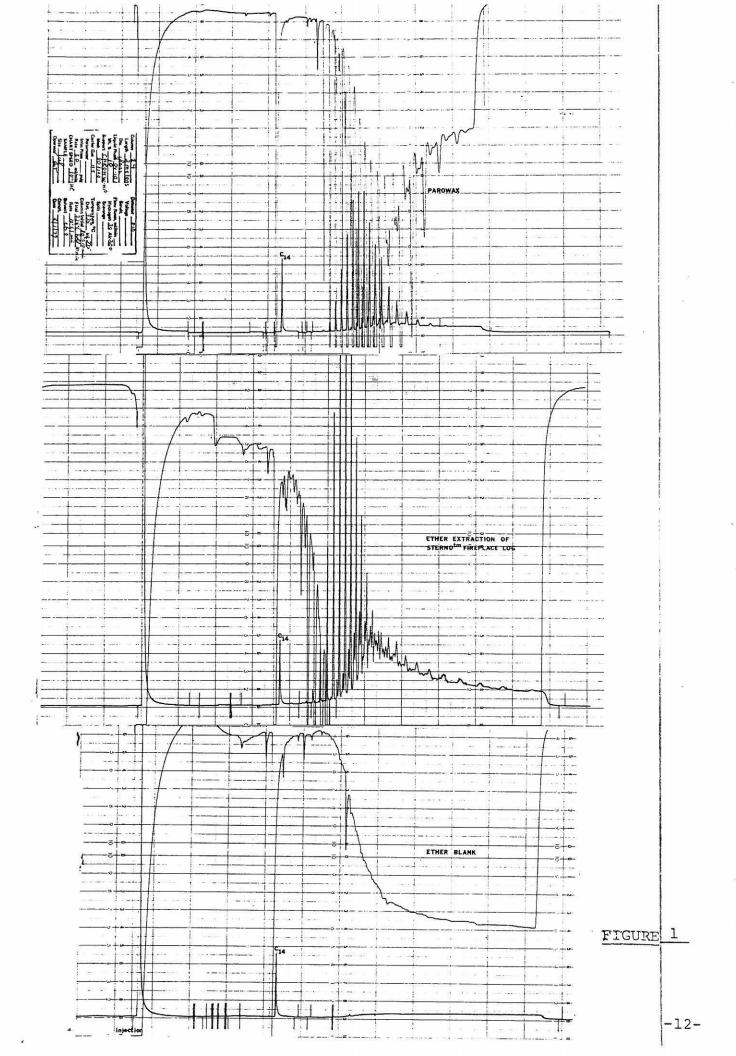
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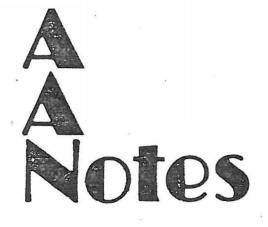
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FEBRUARY 1979

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Investigation of Methods for Detection and Control of Pyrotechnic Dust Fires and Explosions, NESTLE W.R., Report, 1977 ARCSL-CR 77020: order No. AD-B020480 From NTIS.

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