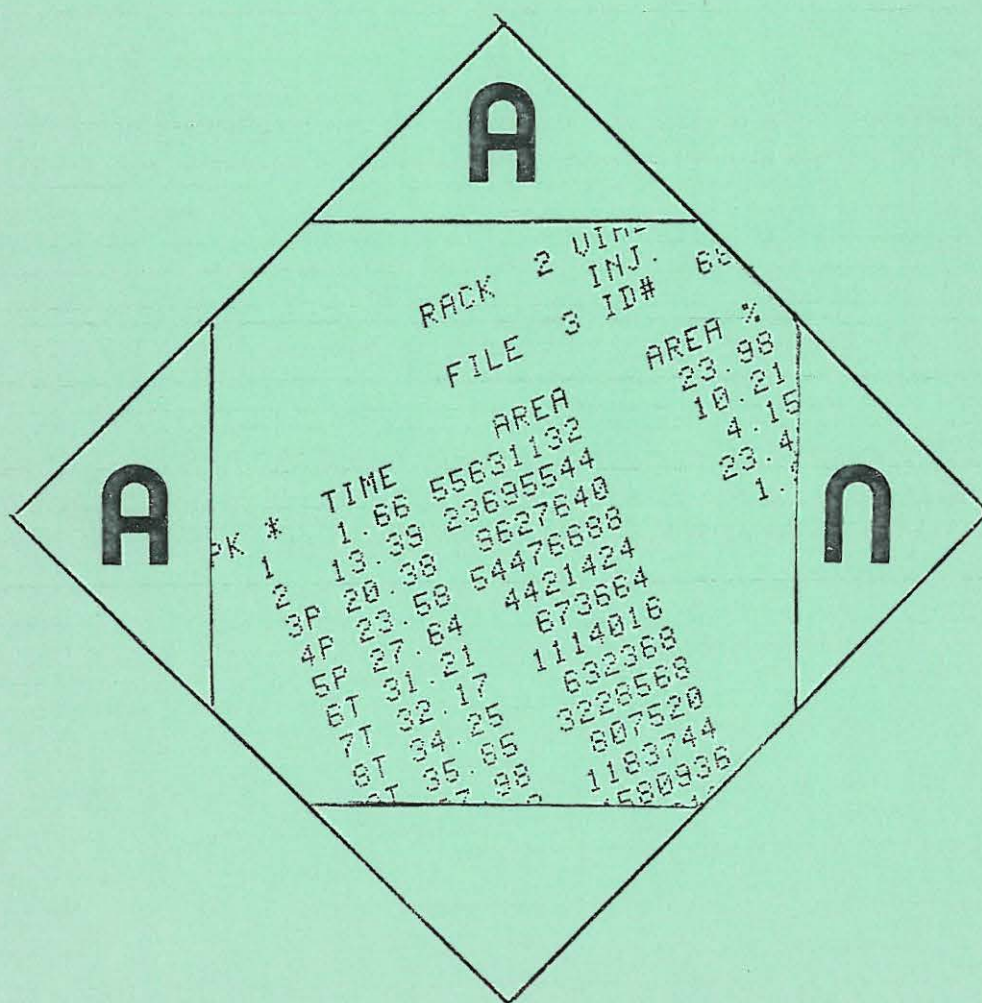


# ARSON ANALYSIS NEWSLETTER

VOLUME 1 No.5



ARSON ANALYSIS NEWSLETTER (AAN)

Published bimonthly by Systems Engineering Associates.  
The AAN is available for a subscription price of \$5.00  
per year.

Send all subscriptions and manuscripts to:

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.

## USE OF DEDICATED MINI-COMPUTERS IN ARSON INVESTIGATION

John N. Lomonte  
Institute of Forensic Sciences  
P.O. Box 35728  
Dallas, Texas 75235

In the analysis of fire residues for accelerants, the use of instrumentation with integral dedicated mini-computers is most valuable. Headspace analysis of cans containing fire residues is done by gas chromatography/mass spectrometry for volatiles identification. Surfaces of building materials are inspected by energy-dispersive X-ray technics to detect metallic additives present in some accelerants. Steam distillates are also analyzed by both methods.

The analysis of the headspace gas of samples taken and preserved at fire scenes is most useful for positive identification of volatile components. The limitations on these identifications are those of mass spectrometry. For example, paraffinic hydrocarbons are difficult to identify by mass spectrometry as their mass spectra consist mostly of mass 43 and 57 peaks. Samples having these peaks but lacking significant higher mass peaks, especially those of even-numbered masses indicative of molecular ions, cannot be identified by mass spectrometry. However, headspace samples which exhibit mass 43 and 57 peaks generally yield large steam distillates so that identification by other methods can be done.

Samples showing large mass 93 peaks indicated the presence of terpenes which are normal wood components, especially in pine wood. GC/MS analysis of the distillate can reveal the presence of diterpenes (mass 136), sesquiterpenes (mass 204) and triterpenes (mass 272) which are

normally present in certain woods. However, if diterpenes occur with practically no sesquiterpene or triterpene, the possibility of the use of turpentine as an accelerant is suspected.

The accelerant most commonly found is gasoline and, in this instance, mass spectral analysis is extremely useful. The components most indicative of gasoline are the aromatic hydrocarbons. Aromatic hydrocarbons give strong molecular ions characteristic of the individual compound. These compounds can be individually identified as there are no interferences with the molecular ions from one aromatic hydrocarbon to another. For example, toluene (MW=92) gives a peak at mass 92. Xylenes, the next higher homolog (MW=106) does not fragment to yield a mass 92 peak, but gives a strong mass 91 instead. Similarly, C3 benzenes (MW=120) do not yield a mass 106 peak which could interfere with xylenes, but do produce mass 105 peaks. Extremely small amounts of these hydrocarbons can be detected by doing limited mass searches for the molecular ions of the hydrocarbons. Once the location of these ions is determined, the mass spectrum of the channel containing a molecular ion can be recalled from the computer memory, background and earlier eluting compounds subtracted, and a positive identification of the component made from the net mass spectrum.

GC/MS analysis is also useful when lacquer thinner and other such mixtures are used as accelerants as the esters and ketones present give readily identifiable mass spectra.

In addition to the aromatic hydrocarbons being an indicator of gasoline-type accelerants, the presence of lead and/or bromine is highly useful. Bromine has been detected in many "unleaded" fuels, as well as lead and bromine in "leaded" gasolines. This is strongly supportive

evidence when GC/MS and IR data indicate a gasoline. Energy-dispersive X-ray is used not only to detect this lead and bromine in liquid samples and in distillates, but is also useful in analysis of residues on building materials and other surfaces. A case history can best illustrate this:

A restaurant was destroyed and arson was suspected.

Samples submitted by the fire investigators failed to reveal conclusive evidence of an accelerant, yet an accelerant was suspected because of burn patterns. The fire scene was revisited fully two weeks after the fire. Suspicious burn patterns were observed in two locations, one under a metal counter and the other at the baseboard floor interface in another room. Analysis of the concrete floor under the counter revealed traces of lead but no distillate was obtained in steam distillation.

Pieces of the stainless steel counter were removed, including a control; EDX showed strong lead and bromine on the underside of the shelf, but not on the top surface. The control contained no evidence of these elements. Even more striking was the analysis of the wood baseboard at the other suspected point of origin. The entire eight feet of baseboard was taken, with one end being the control. EDX revealed strong lead and bromine on the back side at the suspected point of origin, but no lead or bromine was detected in the other end of the same piece of wood.

In our opinion, computer aided analysis of samples from suspected arson cases is such an invaluable aid in positive identification of accelerants that reliance on pattern recognition and matching by non-specific technics is no longer necessary.



Robert L. Graves, Daniel Hunter and LeRoy E. Stewart  
U. S. Treasury Department  
Bureau of Alcohol, Tobacco and Firearms  
Cincinnati Regional Laboratory

ACCELERANT ANALYSIS: GASOLINE

The most common accelerants employed by the arsonist are flammable liquids based on hydrocarbons. We will describe our laboratory's procedure for the detection of one such accelerant, namely gasoline.

Our normal procedure calls for the following steps in the treatment of a sample: (1) extraction and concentration; (2) gas-liquid chromatography (GLC) and (3) comparison to chromatograms of known accelerants. The analysis of the evidence is relatively simple if the accelerant is extracted from the sample matrix, free of extraneous compounds. This unfortunately is not the case very often. When background peaks derived from the sample matrix interfere in the analysis, we resort to ancillary techniques such as re-chromatography after addition of known compounds, lead determination and infrared spectrometry.

Sample extraction and concentration is usually necessary since there will be a small amount of accelerant distributed throughout the debris (charred wood, carpet, burned plastics, etc.). The sample is placed in a four-liter glass reaction vessel to which

is connected a light oil trap and a reflux condenser. The debris is covered with distilled water and refluxed until sufficient sample is collected in the light oil trap for analysis. Short refluxing times are best. As refluxing times are increased more interfering materials will be extracted from the sample matrix.

A 0.2 - 0.3 ul portion of the upper layer collected in the light oil trap is then injected into a gas-liquid chromatograph equipped with temperature programming. Our conditions for GLC follow:

Instrument:	Perkin-Elmer 900 gas chromatograph
Column:	Supelco GP (5% SP-1200/5% Bentone 34 on 100/120 mesh Supelcoport) in a 6' x 1/8" stainless steel column.
Carrier Gas:	Helium at 25 ml/min
Detector:	Flame Ionization at 250° C
Injection port:	225° C
Temperature program:	40° C, 3 min., 12° C/min to 160° C

We have found these conditions to be the best compromise between time of analysis and resolution of peaks.

The chromatogram thus produced is compared to chromatograms of known hydrocarbon mixtures analyzed under identical conditions.



Evaporated gasoline samples mimic burned/weathered gasoline. The chromatograms shown in figure 1 are of gasoline sampled over increasingly longer time periods of evaporation. As the more volatile components dissipate, the less volatile components concentrate as can be seen by comparing the tracings shown in figure 1.

The above procedure may fail to identify the accelerant because of interfering hydrocarbons, which are indigenous to the sample matrix. These hydrocarbons are derived from pyrolysis of wood, breakdown products of plastics, glues, etc. Figure 2 illustrates this point. Shown are chromatograms of steam distillates of some common materials that are often found in arson debris. Comparison of these chromatograms with those in figure 1 show that any recognizable pattern for gasoline may be obliterated. This is especially likely to happen when the gasoline is present in relatively low concentration. When this occurs we must resort to other methods to prove the presence of gasoline.

Confirmation of an uncertain analysis may be made by adding one or more components known to be present in, and preferably unique to, the suspected accelerant and again subjecting the sample to GLC. A relative increase in peak heights of the added components and the absence of new peaks should be the result if the conclusion drawn from the previous test was correct. In figure 3 are shown several components of gasoline that may be used as markers.

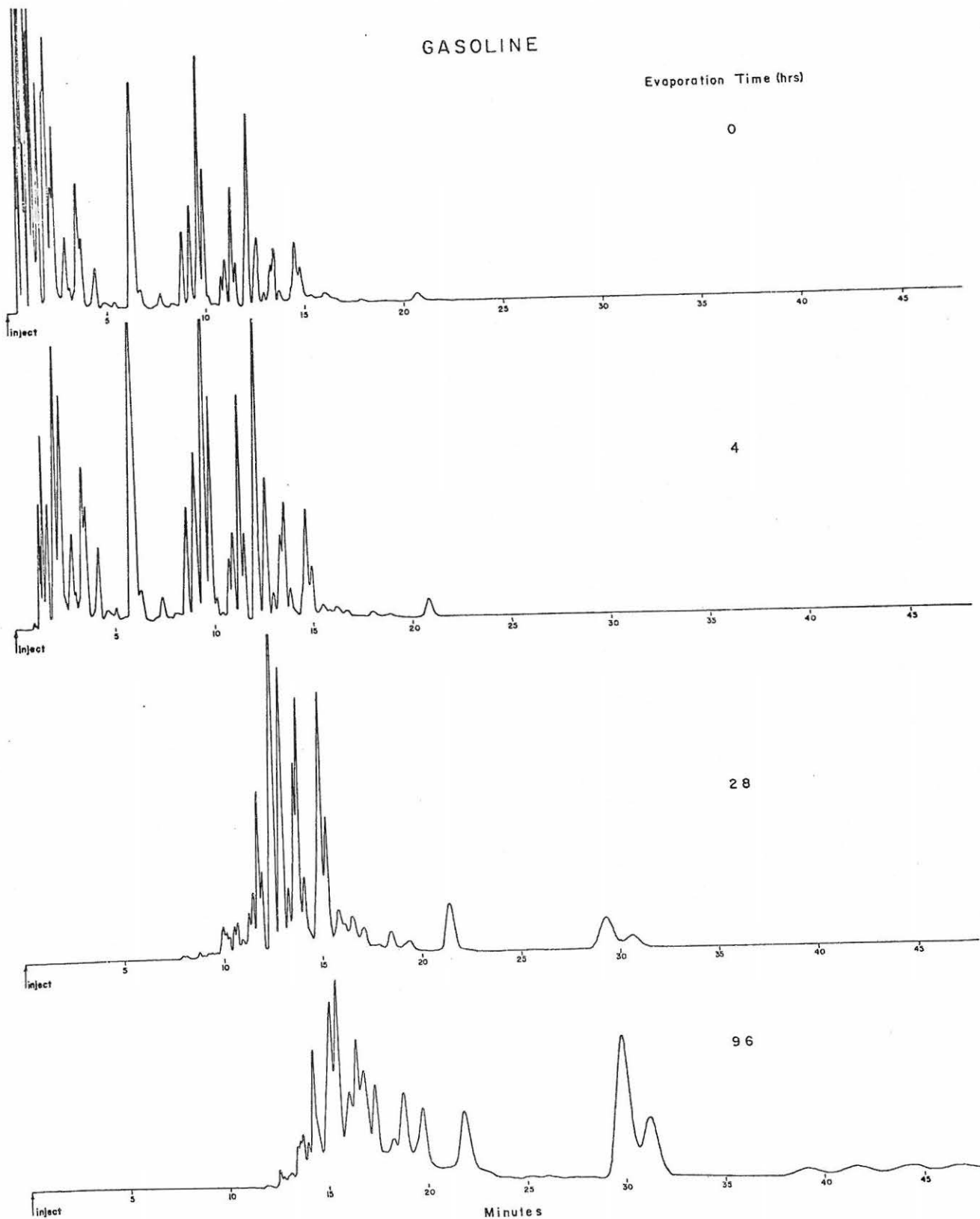


FIGURE 1

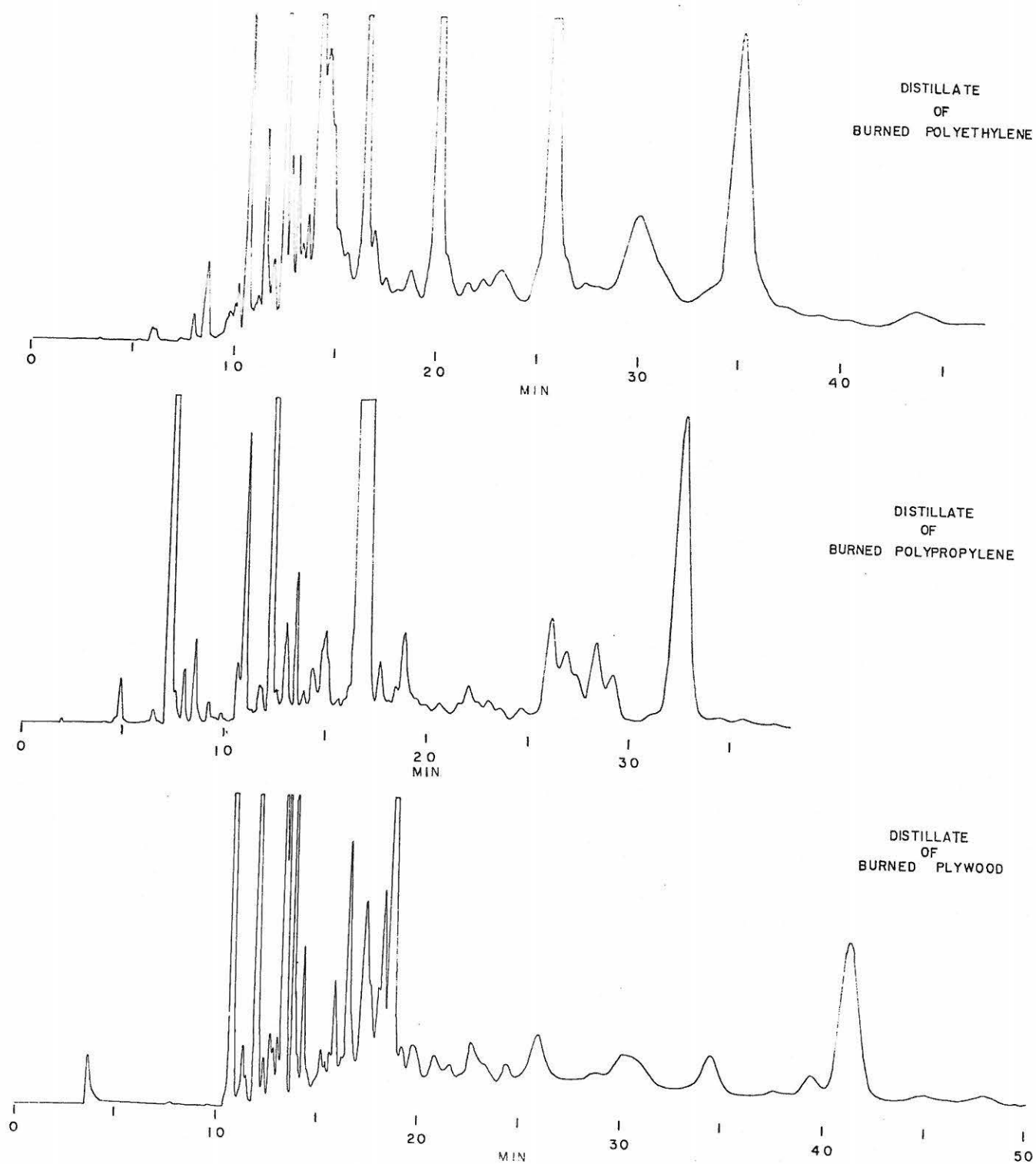


FIGURE 2

- 1 Benzene
- 2 Toluene
- 3 Ethyl Benzene
- 4 p-Xylene
- 5 m-Xylene
- 6 o-Xylene

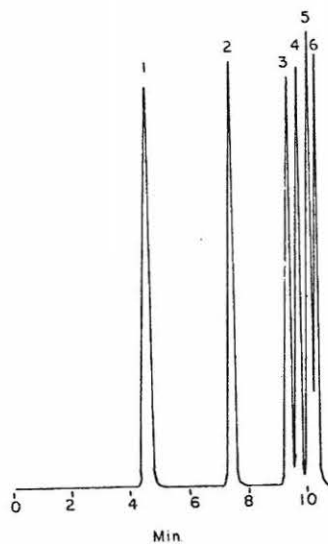


FIGURE 3

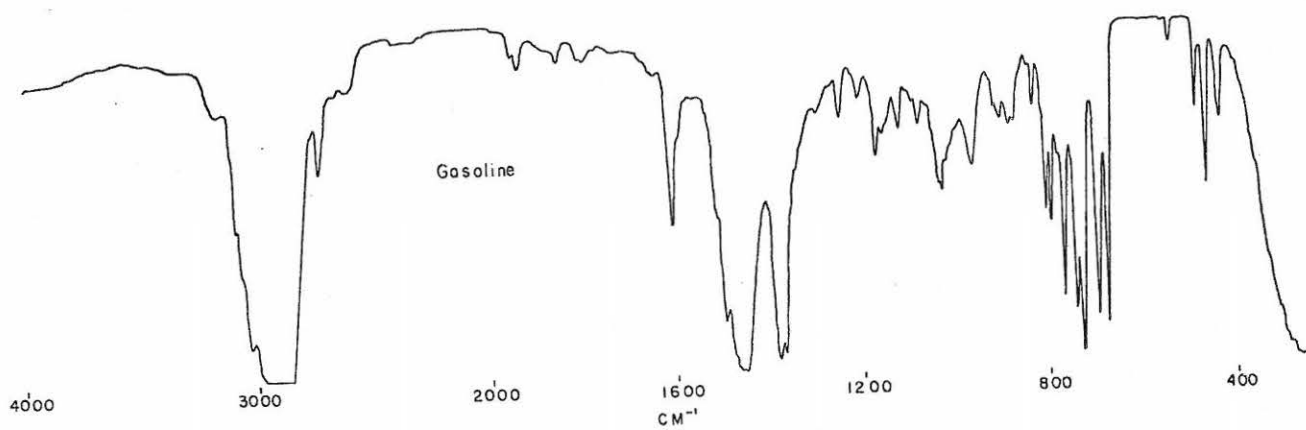


FIGURE 4

The resolution of these compounds on Supelco GP was the major reason for its selection as the GLC stationary phase.

Since Organo-lead compounds are still used to increase the octane rating of many gasolines, an examination for lead is conducted. We make use of an energy dispersive X-ray analyzer (EDAX International, Inc.) because sample preparation is normally quicker and easier than for other methods.

Other procedures for lead analysis, such as atomic absorption spectrometry or colorimetric methods could be used. If possible, the lead content of a control sample from a section of the debris not suspected of containing any accelerant is compared with both the suspect section, and with the distillate of the debris. Presence of lead in the suspect section, and/or distillate, but not in the control, is presumptive evidence of gasoline.

The distillate may then be analyzed by infrared (IR) spectrometry. Figure 4 is an IR spectrum of a typical gasoline. Infrared spectra are used in a similar fashion as were the gas chromatograms. Standard IR spectra are recorded for various materials as was done for GLC standards. In order to interpret the spectra obtained from the sample distillate, comparison is made with spectra for known distillates and evaporated accelerants.

These methods may still be insufficient to unequivocally identify the accelerant. We are currently investigating high pressure liquid chromatography as a possible method for accelerant detection. By making use of ultraviolet (UV) light and refractive index (RI) detectors connected in series, we should be able to generate a profile of just the aromatics (UV detector) as well as a profile of all the constituents present (RI detector). The chromatograms of the accelerants under investigation will, hopefully, deviate enough from chromatograms of debris material to make a positive identification possible.



# AUTOMATED ANALYSIS OF FIRE SAMPLES

BY

RONALD N. THAMAN

SYSTEMS ENGINEERING ASSOCIATES  
7349 Worthington-Galena Road  
Columbus, Ohio 43085

The modern forensic laboratory uses the Gas Chromatograph (GC) as the first and sometimes only tool for the analysis of fire debris. But national trends and statistics show that arson is galloping along at an accelerated pace. How can the forensic laboratory keep up with this pace? Is the answer tying up a scientist playing nursemaid to the gas chromatograph? The purpose of this paper is to show the utility of an automated, computer-controlled gas chromatograph used in fire debris analysis.

Figure one (1) shows the basic components of the system to be described. The gas chromatograph used is a Varian 2800 series coupled with a Varian A-25 dual-pen recorder, Varian autosample and Varian CDS-111 computer.

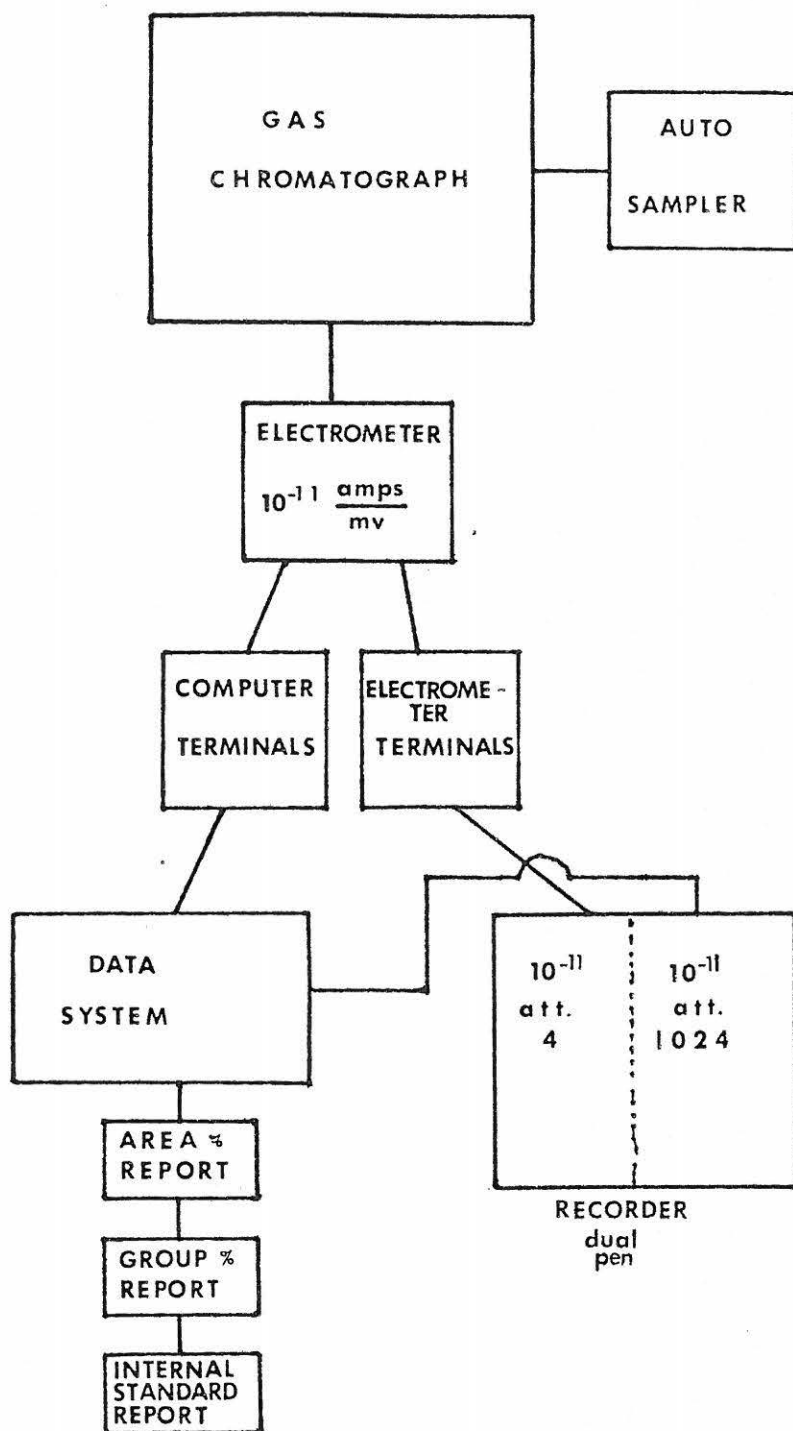


FIGURE 1

## Automated Analysis of Fire Samples

The name of the game in GC analysis of fire debris is speed and accuracy. Whenever an instrument is automated, the scientist must not become a slave to his/her creation but rather create a system which will do the job more efficiently and more reproducibly than he can.

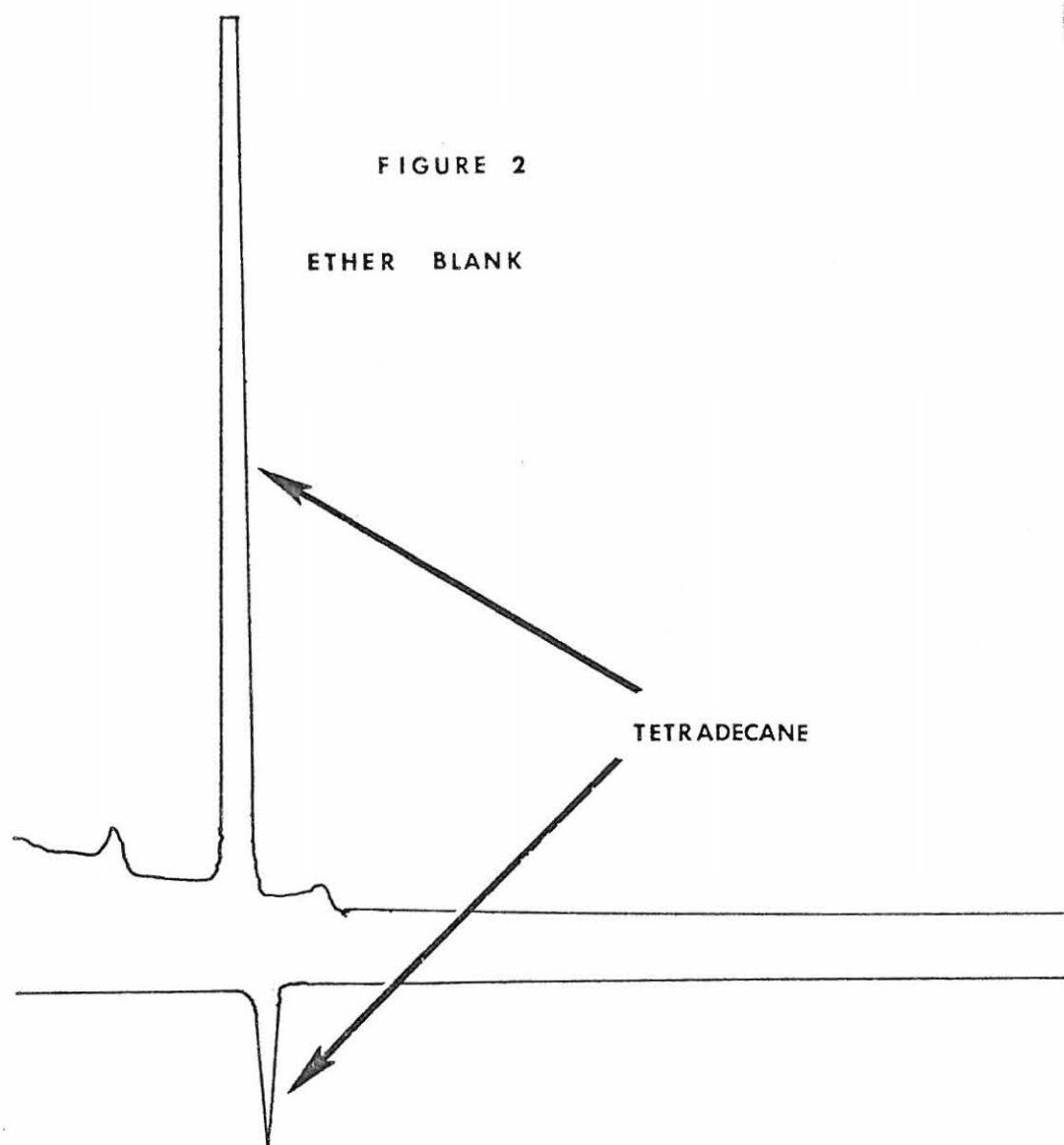
The main features of the system can be summarized as follows:

- \* proof that the syringe is clean before injection of sample
- \* proof that the temperature program has functioned properly each and every run
- \* proof that the correct quantity of sample was injected
- \* quantitation of results via an internal standard

These features can be seen by reviewing the data from a typical "positive" sample run. Figure two (2) shows the GC of the "ether blank" as it will be called. The ether blank proves (for court) that the rinsing cycle of the autosampler has completely cleansed the syringe before injection of the next sample. Any threat of cross contamination between samples must be eliminated. It should be noted that the GC signal is displayed at two attenuation settings. This feature is essential for the analysis of fire debris since the concentration of an accelerant in any group of samples varies considerably.

FIGURE 2

ETHER BLANK



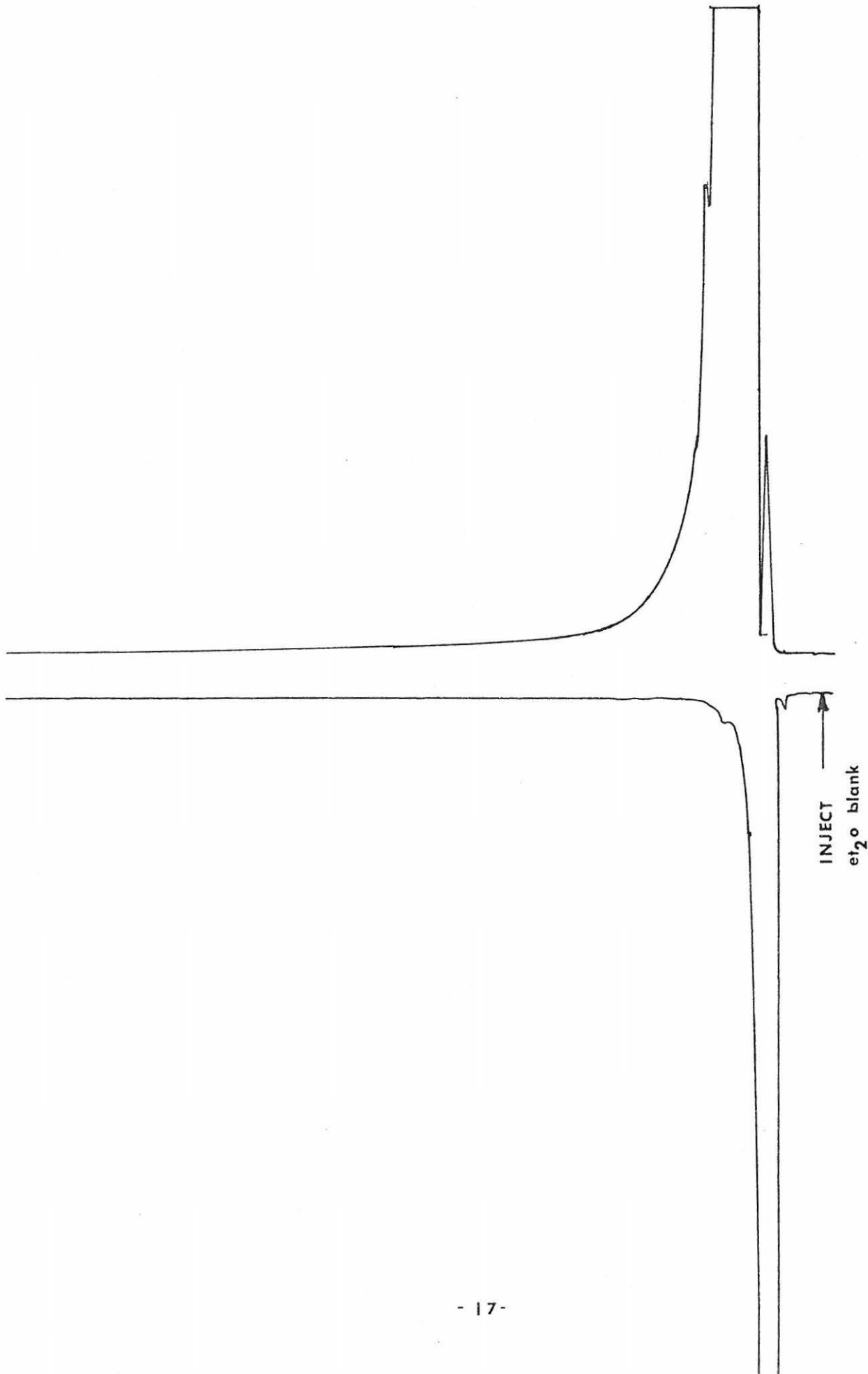
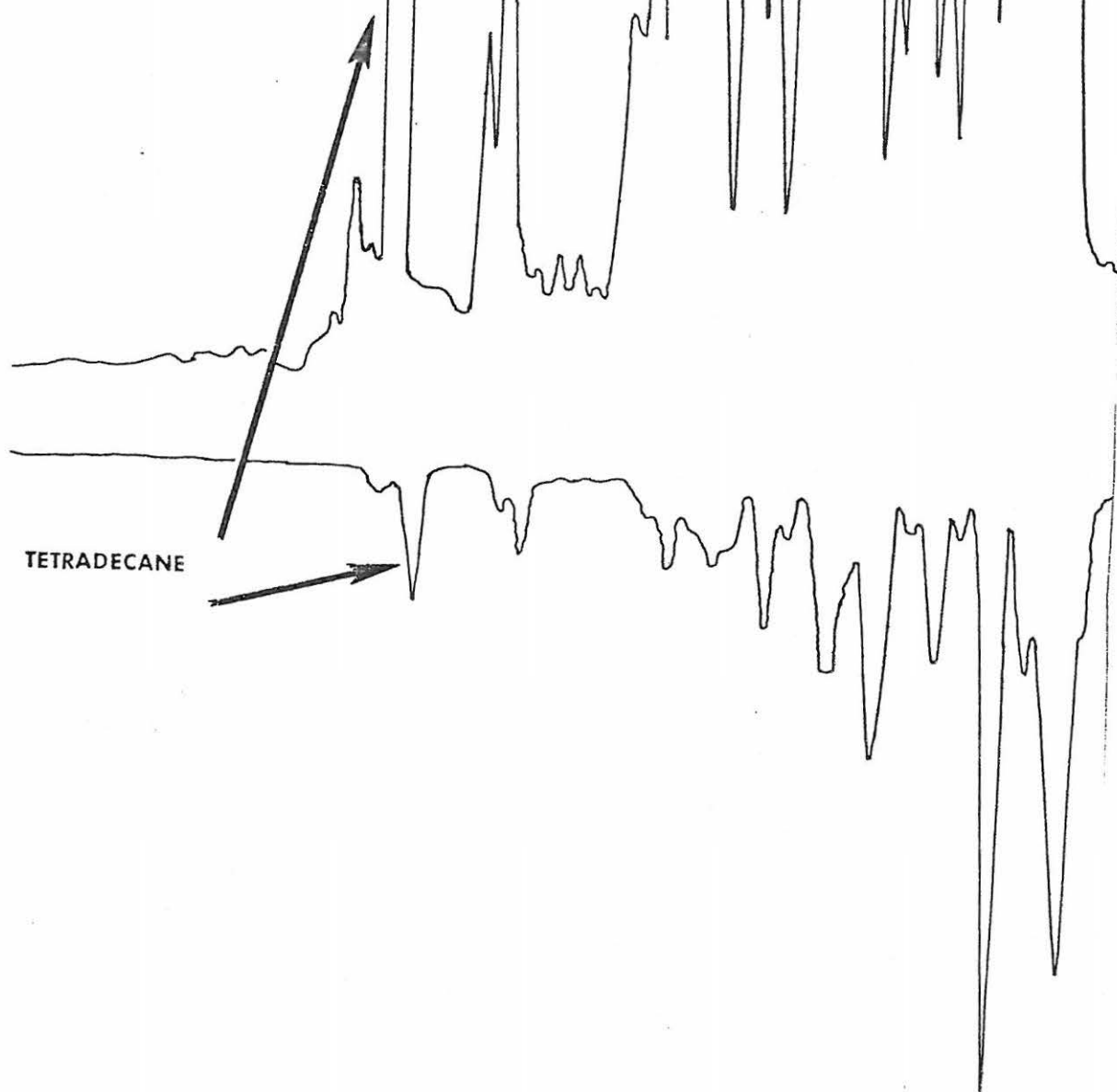
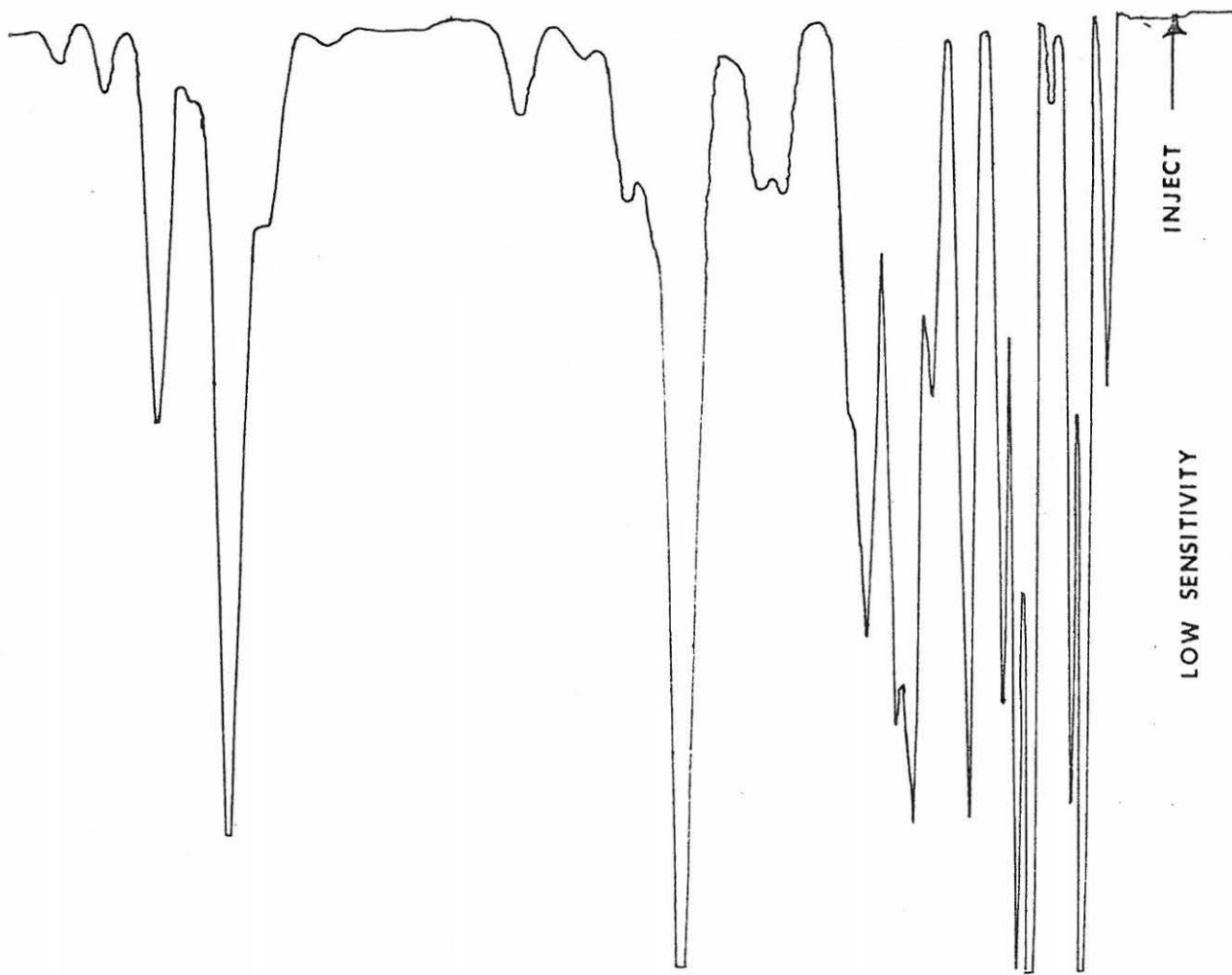
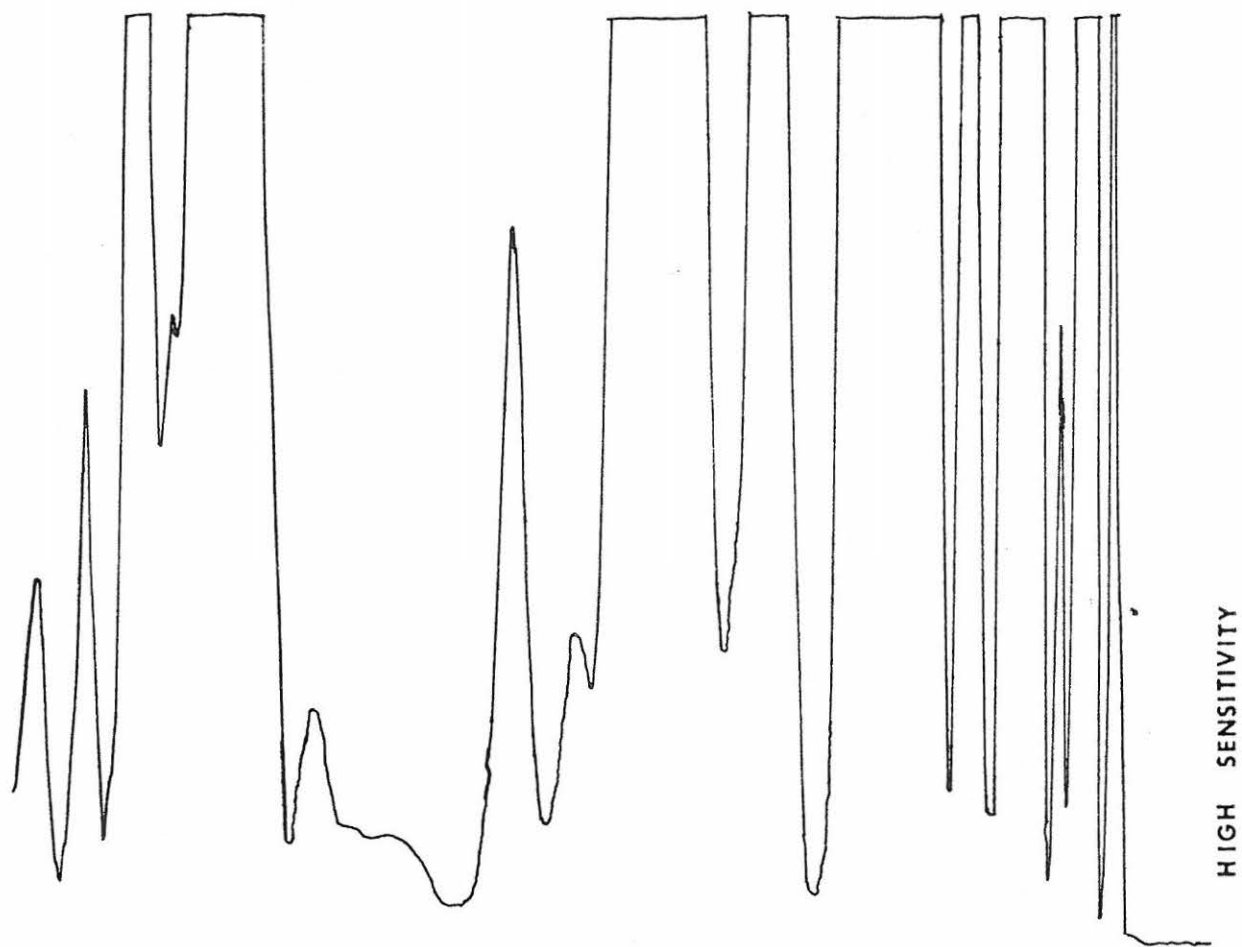


FIGURE 2

GASOLINE







## Automated Analysis of Fire Samples

By displaying the signal at two extremes, the need for additional "runs" is eliminated, speeding up the processing of evidence.

The computer used in this automated GC allows up to nine (9) separate reports to be calculated from one run.

Figure three (3) shows the "ether blank" computer output. File one (1) is a strict area % report. File two (2) groups the data in twenty (20) two-minute intervals from retention times of zero (0) to forty (40) minutes. Such grouping makes comparison more clear and is similar to the "Spec-Finder" system in infrared. File three (3) uses tetradecane as an internal standard (1233.00 ppm) and calculates all peaks in relation to the internal standard.

The system described will analyze up to twenty (20) arson samples which include twenty (20) ether blanks and ether washes. The temperature program, oven cool-down, etc. are all controlled by the computer.

FIGURE 3 ETHER BLANK PRINTOUT

RACK 11 VIAL 3  
INJ. 1  
FILE 1 ID# 49

PK #	TIME	AREA	AREA %
1P	25.75	14806728	93.55
2T	29.64	1021408	6.45
TOTAL		15828136	100.00

ETHER PEAK ELIMINATED USING  
"FORCED BASELINE"

GROUPING CODE RACK 11 VIAL 3  
INJ. 1  
FILE 2 ID# 49

PK #	TIME	AREA	AREA %
1G	25.01	14806728	93.55
2G	29.01	1021408	6.45
TOTAL		15828136	100.00

TETRADECANE RACK 11 VIAL 3  
V# 1 INJ. 1  
FILE 3 ID# 49

PK #	TIME	AREA	INT. STD
1P	25.75	14806728	1233.00
T	29.64	1021408	85.06
TOTAL		15828136	85.06

SAMP 1.000000  
STD/CAL 1233.0000  
SCALAR 1.000000

RACK 11 VIAL 4  
INJ. 1  
FILE 2 ID# 50

PK #	TIME	AREA	AREA %
1	1.66	19400802	3.17
2P	2.28	8875528	1.45
3P	2.86	53526376	8.73
4P	3.34	32099240	5.24
5P	3.90	20561768	3.35
6P	4.76	48198884	7.86
7P	5.40	13907488	2.27
8P	5.74	18687544	3.05
9P	6.73	17974816	2.93
10P	8.46	58165260	9.49
11P	10.14	1923972	.31
12P	10.81	9993692	1.63
13P	12.76	8202340	1.34
14P	13.99	50462768	8.23
15P	14.95	24037936	3.92
16P	16.18	8343688	1.36
17P	17.24	32901336	5.37
18P	18.10	24644240	4.02
19P	18.84	9333600	1.52
20P	19.64	16504336	2.69
21P	20.23	13185792	2.15
22P	21.03	8347848	1.36
23P	21.72	11858896	1.93
24P	22.52	8933952	1.46
25P	23.54	5635448	.92
26P	24.12	1990096	.32
27P	24.71	5168800	.84
28P	25.72	27471584	4.48
29P	26.47	1839952	.30
30P	27.22	1624544	.27
31P	28.23	2891696	.47
32P	29.14	1301024	.21
33P	29.62	3063648	.50
34P	30.52	2444464	.40
35P	31.48	1264784	.21
36P	32.34	4080952	.67
37P	36.07	7739080	1.26
38P	41.30	2807088	.46
39P	44.71	817864	.13
40P	45.83	529912	.09
41P	48.87	1612552	.26
42P	56.23	2066224	.34
43P	59.86	11418384	1.86
44P	65.46	1195632	.20
45P	66.04	1140280	.19
46P	67.00	2693472	.44
47P	67.59	2089192	.34
TOTAL		612959774	99.99

FIGURE 3 GASOLINE PRINTOUT

RACK 11 VIAL 4  
INJ. 1  
FILE 2 ID# 50

PK #	TIME	AREA	AREA %
1G	1.50	19400802	4.05
2G	3.01	47954048	10.02
3G	5.01	13686052	2.86
4G	7.01	17974816	3.75
5G	9.01	58165260	12.15
6G	11.01	11917664	2.49
7G	13.01	58665108	12.25
8G	15.01	24037936	5.02
9G	17.01	41245024	8.62
10G	19.01	50482176	10.54
11G	21.01	33392536	6.98
12G	23.01	14569400	3.04
13G	25.01	34630480	7.23
14G	27.01	3464496	.72
15G	29.01	7256368	1.52
16G	31.01	3709248	.77
17G	33.01	4080952	.85
18G	37.01	7739080	1.62
19P	41.30	2807088	.59
20P	44.71	817864	.17
21P	45.83	529912	.11
22P	48.87	1612552	.34
23P	56.23	2066224	.43
24P	59.86	11418384	2.39
25P	65.46	1195632	.25
26P	66.04	1140280	.24
27P	67.00	2693472	.56
28P	67.59	2089192	.44
TOTAL		478742046	100.00

RACK 11 VIAL 4  
 V# 1 INJ. 1  
FILE 3 ID# 50

PK #	TIME	AREA	INT. STD
	1.66	19400802	870.76
P	2.28	8875528	398.36
P	2.86	53526376	2402.41
P	3.34	32059240	1440.70
P	3.90	20561768	922.87
P	4.76	48199884	2163.34
P	5.40	13907488	624.21
P	5.74	18687544	838.75
P	6.73	17974816	806.76
P	8.46	58165260	2610.62
P	10.14	1923972	86.35
P	10.81	9993692	448.54
P	12.76	8202340	368.14
P	13.99	50462768	2264.91
P	14.95	24037936	1078.89
P	16.18	8343688	374.49
P	17.24	32901336	1476.70
P	18.10	24644240	1106.10
P	18.84	9333600	418.92
P	19.64	16504336	740.76
P	20.23	13185792	591.81
P	21.03	8347848	374.67
P	21.72	11858896	532.26
P	22.52	8933952	400.98
P	23.54	5635448	252.93
P	24.12	1990096	89.32
P	24.71	5168800	231.99
1P	25.72	27471584	1233.00
P	26.47	1839952	82.58
P	27.22	1624544	72.91
P	28.23	2691696	129.79
P	29.14	1301024	58.39
P	29.62	3063648	137.50
P	30.52	2444464	109.71
P	31.48	1264784	56.77
P	32.34	4080952	183.16
P	36.07	7739080	347.35
P	41.30	2807088	125.99
P	44.71	817864	36.71
P	45.83	529912	23.78
P	48.87	1612552	72.38
P	56.23	2066224	92.74
P	59.86	11418384	512.49
P	65.46	1195632	53.66
P	66.04	1140280	51.18
P	67.00	2693472	120.89
P	67.59	2089192	93.77
TOTAL		612859774	26276.30

SAMP 1.000000  
 STD/CAL 1233.0000  
 SCALAR 1.000000

# AAN notes

---

- \* An article describing the effects of various fluorides on the reaction between Potassium Chlorate and Magnesium. Thermogravimetric as well as Differential Thermal Analysis are presented. THERMAL ANALYSIS: INORGANIC MATERIALS & PHYSICAL CHEMISTRY, VOLUME 2 SCHWENKER & GARN, ACADEMIC PRESS, 1969.
- \* HPLC was used to analyze TNT, RDX and HMX. Analytical Chemistry, Volume 49, No. 7, June 1977, p. 1039. Analysis of Explosives by High Performance Required Chromatography and Chemical Ionization Mass Spectrometry.
- \* The AAN is currently being abstracted by Chemical Abstracts Service (CAS). Abstracts of chemical related articles appear in CA Selects Forensic Chemistry, a publication of CAS.