-Arson———Analysis————Newsletter=

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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. Articles herein express the views and opinions of the authors, which are not necessarily those of the AAN or Systems Engineering Associates (SEA).

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"COMPARISON OF FIRE DEBRIS ANALYSIS TECHNIQUES"

Jack Nowicki¹ and Christine Strock²

Fire debris evidence containing the distinct odor of an accelerant can be tested by various techniques. However, if an odor is not noticeable, only the more sensitive detection methods are feasible.

Some of the techniques used in fire debris examination in forensic laboratories to ascertain the presence of accelerants are solvent extraction, heated headspace sampling, steam distillation, charcoal elution under vacuum, sorbent trap concentration followed by thermal desorption, and various forms of charcoal placed within the evidence container followed by elution with an organic solvent or by thermal desorption. All of the techniques yield samples which can be injected into a gas chromatograph for analysis. The resulting chromatograms, when compared to known standards, can determine the presence of an accelerant.

The purpose of this investigation was to determine, using simulated fire debris samples, the efficiency of the above methods. This was accomplished by calculating what percentage of a known amount of accelerant added to the sample was actually extracted and injected into the gas chromatograph.

MATERIALS:

Gasoline was chosen as the accelerant to be tested. For each method, a $1.0~\mu l$ sample (measured with a $10.0~\mu l$ Hamilton syringe) of 80% weathered Shell

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gasoline (regular grade) was applied to a 1.0 cm² sample of polyurethane foam carpet padding prior to the analysis. This method of sample preparation approximated actual fire debris evidence.

The amount of accelerant extracted from the debris by each method was monitored by the area under the peak of a characteristic component of gasoline. The peak for 1,2,4-trimethylbenzene with an approximate retention time of 12 minutes was selected as the component to be monitored. (Figure 1)

GAS CHROMATOGRAPH OPERATING PARAMETERS

Instrument:

Perkin-Elmer Sigma 115

Column:

12' x 1/8" stainless steel; 3% SP-2100

methyl silicon on 100/120 mesh

Supelcoport

Detector:

Flame Ionization Detector (FID)

Injector Temperature:

230°C

Detector Temperature

230°C

Program:

50°C for 4 minutes; to 225°C at 8°C/min;

225°C for 5 minutes

Carrier Gas:

Nitrogen, 20 ml/min

Hydrogen:

30 ml/min

Air:

30 m1/min

After preparing a dilution series of the 80% weathered gasoline standard, a calibration curve relating the area of the 1,2,4-trimethylbenzene peak to the amount of gasoline present was drawn. In this way, for each sample run in this experiment the percentage of gasoline recovered out of the original 1.0 μ l in the sample could be directly extrapolated from the 1,2,4-trimethylbenzene peak area.

RUH

1

GASOLINE

METHODS & RESULTS

HEADSPACE SAMPLING

A sample was placed in a 1.0 quart (0.95 1) friction lid paint can. The can was sealed and a hole punched in the lid. This hole was covered with transparent tape and the can was heated in an oven (Grieve Corp. Model LW200C) set at 80°C ($\pm 10^{\circ}$ C) for 20 minutes. Contents were sampled using a 10.0 μ l disposable syringe. Contents of can were "pumped" with syringe prior to sampling to ensure an even distribution of contents.

Sample size, temperature, and presence of water were the parameters varied to determine the optimum conditions for this method. Water was introduced by including a 1" x 1" Kimwipe dampened with water which was included with the sample. (Table 1)

Table 1 RESULTS OF HEADSPACE SAMPLING TECHNIQUES

I. Sample Size Variations (80°C; 20 min.)

Sample Size	Peak Area	
2.0 ml	0.0237	
5.0 ml	0.0906	
10.0 ml	0.2104	

II. Temperature Variations (5.0 ml samples; 20 min.)

Temperatures	Peak Area	
Room Temperature	0.0004	
80°C	0.0873	
100°C	0.1146	
120°C	0.1489	

III. Effect of Water on Temperature Variations (5.0 ml samples; 20 min.)

Temperature	Peak Area	
80°C	0.0715	
100°C	0.0761	
120°C	0.1149	

Under the conditions specified in this experiment, the maximum values could be obtained by heating the sample to 120°C. Increasing the sample size to 10.0 ml did not decrease the resolution of the characteristic peaks. Inclusion of water with the sample decreased the value of the gasoline detected by approximately 20%.

SOLVENT EXTRACTION

A sample was placed in a 10 ml beaker and covered with 4.0 ml n-pentane (Infrared Spectranalyzed; Fisher Scientific Co.). The solution was covered for five minutes, shaken, and the pentane was poured off and analyzed. Undiluted pentane gave negative results, so the solution was concentrated to 0.60 ml prior to analysis. (Table 2)

Table 2 RESULTS OF SOLVENT EXTRACTION

Average	Area

Unevaporated Solvent

0

Evaporated Solvent

0.0465

STEAM DISTILLATION

A sample was refluxed in a standard steam distillation apparatus for one hour. The sample was removed from the heat and allowed to cool until the condensation stopped. Five drops of n-hexane (UV-Burdick and Jackson Laboratories, Inc.) was added to the distillate and the organic layer removed by pipette. A 1.0 μ l sample was analyzed.

Unevaporated and evaporated (to 0.60 ml) samples were analyzed. Both methods gave negative results.

CHARCOAL ADSORPTION/ELUTION

The sample was enclosed in a 1.0 quart (0.95 1) can and sealed. Two small pieces (1") of black electrical tape were placed near the perimeter of the lid and small holes were punched in the lid through the tape.

The general procedure involved preparing a disposable pasteur-type pipette (5 3/4") packed with a small cotton plug, followed by activated charcoal, 50/200 mesh (Fisher Scientific Co.), topped with another small cotton plug. The tapered end of the pipette was placed in one of the holes in the lid. The other end of the pipette was placed under a vacuum produced by a pump (Gast Mfg. Corp. Model 5KH3 6KN90X) set at 22.5" Hg.

An empty pipette connected to a filter was placed in the other lid hole to provide a source of filtered air for circulation.

After the volatile compounds present were adsorbed on the charcoal, the compounds were eluted by placing the charcoal-filled pipette in a shell vial, tapered end down, and adding solvent to the larger end of the pipette and evacuating the solvent with the aid of a rubber bulb. A 1.0 μ l sample of the eluate was then analyzed. (Figure 2)

Several parameters were varied in the above method in an attempt to determine the most efficient method. (Table 3). First, the amount of charcoal used was varied, holding all other conditions constant. Next, various amounts of eluting solvent were used. Two solvents were examined, pentane and carbon disulfide. Heating the sample in an oven as opposed to a heating mantle was examined. Finally, the effect of concentrating the sample by evaporation using heat and a stream of dry air was determined.

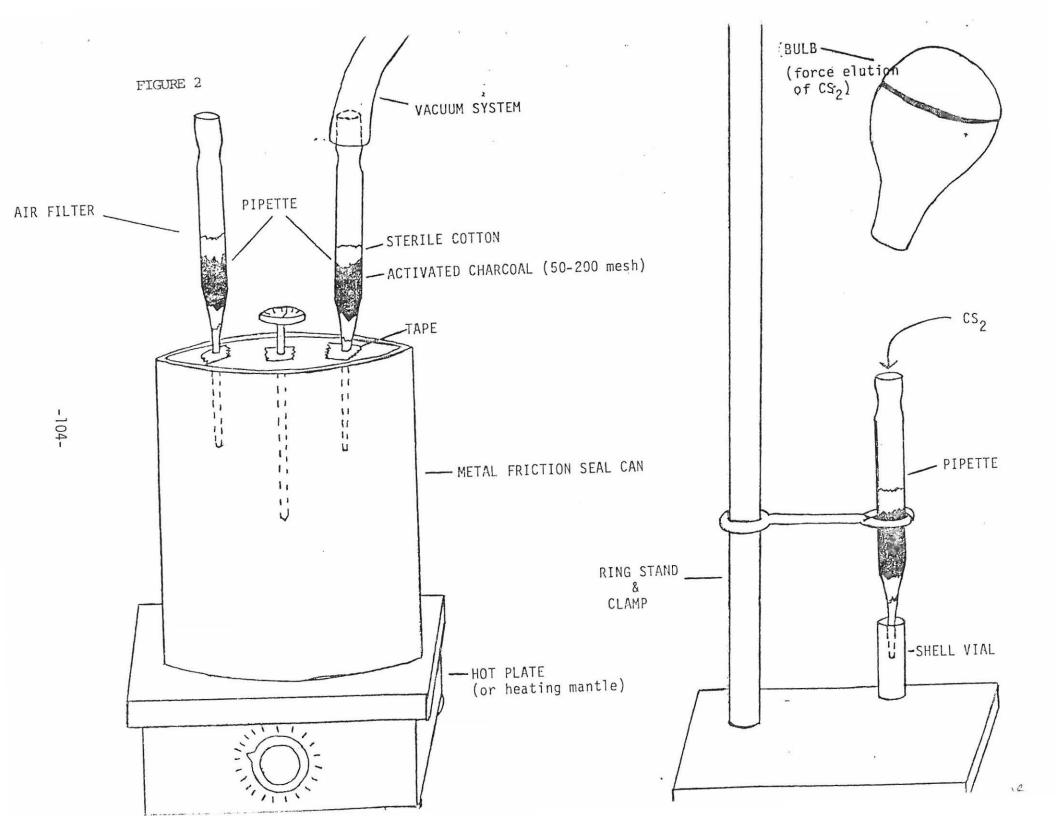


Table 3 RESULTS OF CHARCOAL ADSORPTION/ELUTION METHODS

I. Amount of Charcoal (80°, 15 min., elute with 0.5 ml pentane)

			,	,
			Peak Area	
	1.0 cm		0.0380	
	2.0 cm		0.0427	
	3.0 cm		0.0505	
II. Solv	ent (80°, 15 min	., eluate eva	aporated to 0.1 ml)	
			Peak Area	
1.0 ml	Pentane		0.2102	
	Carbon disulfid	е	1.9846	
2.0 ml	Pentane		0.2708	
	Carbon dilsulfi	de	0.6455	
3.0 ml	Pentane		0.3307	
	Carbon disulfid	е	1.3277	
III. Hea	ating (15 min., 1.	0 ml., CS ₂ e	vaporated to 0.1 ml)	
			Peak Area	
Lab Ove	en	800	1.9846	
		120°	1.2678	
Glas-Co	ol Heating Mantle	e 100°	3.2999	
IV. Eva	poration Time	0.1 ml, eva	80°, 15 min., 1.0 ml CS ₂ aporation time controlled under stream of dry air)	by heating on
			Peak Area	
1	5 minutes		2.0060	

Based on various combinations of operating parameters, the most effective charcoal elution technique was to use approximately 3 cm of charcoal, with the can in a heating mantle set at 100°C. Vacuum should be applied for fifteen minutes and the charcoal eluted with 1.0 ml of CS₂. The solvent should be

2.4881

3.9238

5.2940

30 minutes

40 minutes

4 hours

allowed to soak in the charcoal for at least 5 minutes prior to evacuation. Evaporation should be performed as slowly as possible since the amount of volatile component retained is inversely related to the rate of evaporation.

SORBENT TRAP/THERMAL DESORPTION

This method is an adaption of sorbent trap concentration followed by thermal desorption reported by Sasferstein and Park.¹ Volatile components are trapped in a cartridge filled with an adsorbent material by purging with an inert gas; the sample is then thermally desorbed and deposited directly onto the gas chromatograph column.

The sample was sealed in a can and placed in the Chemical Data Systems' Arson Concentrator. The sampling container has adjustments for temperature and gas flow rate. After heating the can and contents, the heating source was turned off and the volatile components were adsorbed onto a Tenax GC cartridge by flushing the system with dry air.

The cartridge was removed and inserted into the Chemical Data Systems 320 Concentrator which trapped the organic compounds and then evacuated them directly onto the gas chromatograph column for analysis. The trap was $24" \times 1/8"$ OD x .0931" ID stainless steel, Tenax G.C.

The program used required a 10 minute desorption at 250°C, a cool down time of 3 minutes, a drying time of 1 minute followed by a 4 minute transfer to the column at 250°C. The trap was reconditioned by heating at 275°C for 5 minutes prior to analyzing the next sample.

Several problems were encountered with the CDS system. Only one of the three Tenax cartridges provided with the instrument gave any results at all. Also, the results obtained from this cartridge were variable. Over several trials, the peak area varied from a low of 1.0186 to a best run of 9.5850. We were not

able to determine in the time available for this project whether this wide variation in results was inherent to the instrument or if it was due to a problem in our experimental method.

SUMMARY

Table 4 summarizes the peak areas determined for each of the methods described, along with the calculated percent gasoline recovered as extrapolated from the calibration curve. It should be stressed that the quantitative aspects of this experiment are, at best, only "ball-park" figures. The real value of these figures lie in their relative measure of the efficiency of a technique. It can be seen from Table 4 that the optimized charcoal adsorption/elution technique was approximately 25 times more efficient than headspace sampling under these experimental conditions, and over 100 times more efficient than solvent extraction.

Table 4 COMPARISON OF A MICROLITER SAMP			
Method	Average Area	% Gasoline Recovered (Extrapolated from Calibrated Curve)	
Sorbent Trap/Thermal Desorption	9.5850*	11.2500	
Charcoal Elution	5.2940	6.2136	
Headspace Sampling	0.2104	0.2469	
Solvent Extraction	0.0465	0.0546	
Steam Distillation	-		
*va	riable results		

OTHER TECHNIQUES

Due to time limitations on this project, some of the more recently reported techniques were not included in this study. This includes the use of charcoal-coated absorption wires followed by solvent elution² or thermal desorption with a Curie point pyrolyzer.^{3,4} Future work will concentrate on these areas.

ACKNOWLEDGEMENTS

The authors wish to acknowledge financial support during the project from the Dean's Fellowship Program of St. Xavier College. Also, thanks are due to Mary Staniszewski for Figure 2.

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- 1. Saferstein, R. and Park, S. A., "Application of Dynamic Headspace Analysis to Laboratory and Field Arson Investigations," <u>Journal of Forensic Sciences</u>, 27 (3), pp. 484-494, 1982.
- 2. Juhala, J. A., "A Method For Adsorption of Flammable Vapors by Direct Insertion of Activated Charcoal Into the Debris Samples," Arson Analysis Newsletter, 6 (2), pp. 32-36, 1982.
- 3. Twibell, J. D. and Home, J. M., "Novel Method for Direct Analysis of Hydrocarbons in Crime Investigation and Air Pollution Studies," <u>Nature</u>, 268 (31), pp. 711-713, 1977.
- 4. Andrasko, J., "The Collection and Detection of Accelerant Vapors Using Porous Polymers and Curie Point Pyrolysis Wires Coated with Active Carbon," Journal of Forensic Sciences, 28 (2), pp. 330-340, 1983.

Article secured for the AAN by:

Blair W. Schultz Bureau of Scientific Services 515 East Woodruff Road Joliet, Illinois 60432

Case Study

I thought that the following information might be of some interest to the readers of the AAN. A recent case contained some partially melted, plastic-looking material among other charred debris. After concentrating the vapors of the sample with the charcoal trap method, the gas chromatogram revealed a large peak just after benzene in retention time. A mass spectrum of the peak was obtained, but it could not be identified at that point. The infrared spectrum of some of the plastic-looking material matched that of polymethyl methacrylate (plexiglas). The monomer of plexiglas is methyl methacrylate and its structure is:

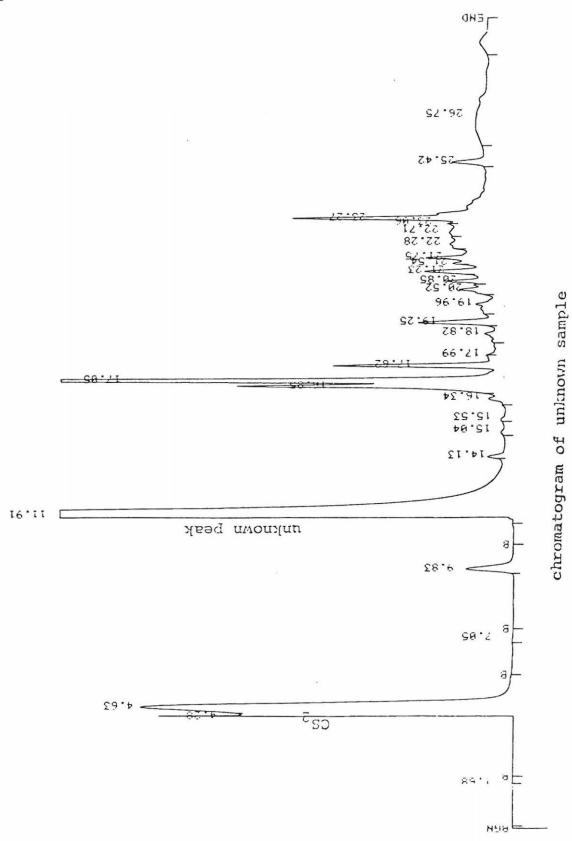
This additional information aided the interpretation of the mass spectrum. The major peaks were identified as follows: the molecular ion was m/e 100; the fragments at m/e 85, m/e 69 and m/e 41 represented the loss of CH $_3$, 0-CH $_3$ and C=0 respectively from the molecular ion.

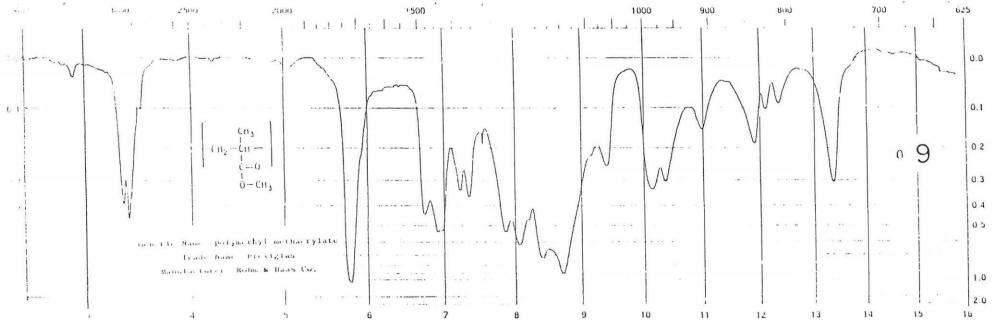
Information from the fire investigator indicated the presence of plexiglas. The sample was taken just outside a door that may have had a plexiglas window.

Finally, a standard of methyl methacrylate was acquired, and its mass spectrum corresponded to that of the unknown peak.

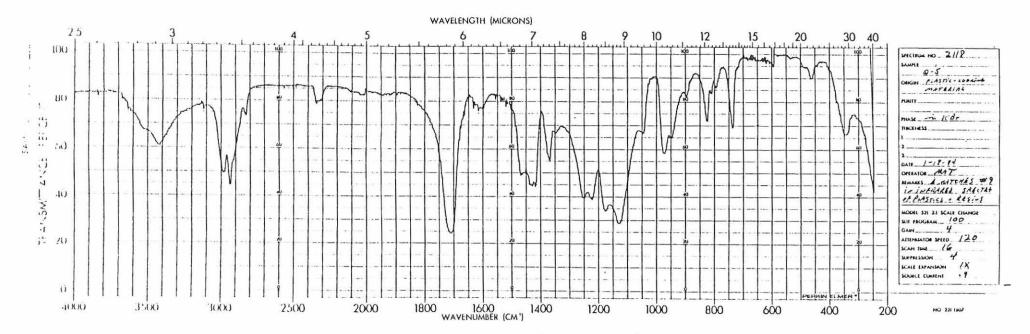
Submitted by:

Michael A. Trimpe Criminalist Institute of Forensic Medicine Toxicology and Criminalistics 3159 Eden Avenue Cincinnati, Ohio 45219





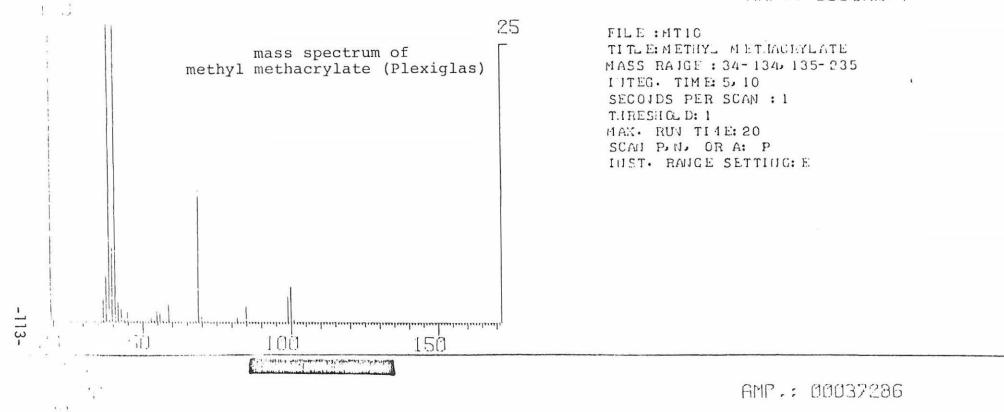
IR spectrum of polymethyl methacrylate (Plexiglas)

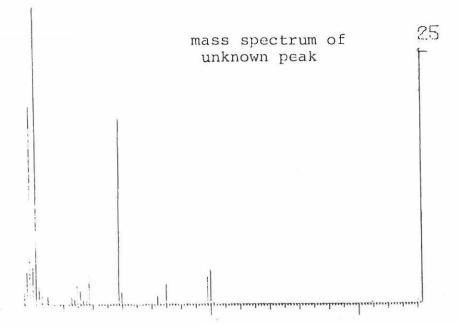


IR spectrum of unknown sample

.

AMP.: 00052274





* * * LETTER TO THE EDITOR * * *

Dear Editor:

Although I have not used or seen RHODOKRIT H-630 which was referred to by Bill Dietz in the May/July, AAN, I did recently run across several references to the use of colorimetric procedures for the detection of hydrocarbons, which may be of interest to the subscribers of AAN. The one from Kirk's book mentions the use of Scharlach R which produces a red lake in the presence of hydrocarbon solvents. Apparently, the reaction depends upon the dye dissolving the liquid hydrocarbon residues and one might presume that residues from polymers would not interfere since they most likely would solidify after the fire debris cooled and would no longer dissolve the Scharlach R. Possibly this is the same dye used in RHODOKRIT H-630? The second reference is, I'm quite sure, the same one you refer to although I believe the publication was called The International Association of Arson Investigators Newsletter back in 1953 when the article first appeared. It appears that the Newsletter was either merged with or replaced by the Fire and Arson Investigator sometime in 1960. The article was reprinted in "Selected Articles for Fire and Arson Investigators", in 1975. I do not have any of these documents, but I'm sure we're talking about the same article. The last one is rather formidable, but if any AAN reader reads Russian I'm sure a copy of the article could be obtained. Possibly an english translation is available from the source of these references.

- 1. P.L. Kirk, "Crime Investigation: Physical Evidence and the Police Laboratory", New York, Interscience Publishers (1966), p. 717.
- H.P. Wonderling, "Arsonists -- Their methods and the evidence", <u>The International Association of Arson Investigators Newsletter</u>, Oct.-Dec. 1953. Reprinted in "Selected Articles for Fire and Arson Investigators", Marlboro, Massachusetts, International Association of Arson Investigators (1975).
- 3. K. Ol'Khovshaya, "Colormetric determination of hydrocarbons, gasoline, kerosene, and white spirit in the air of industrial installations", Gigiena Truda i Professional'nye Zabolevaniya, Vol. 15, No. 11, 1971, pp. 57-58 (Russian).

These references were all found in Arson and Arson Investigation; Survey and Assessment, J.F. Boudreau, Q.Y. Kwan, W.E. Faragher and G.C. Denault, published by the National Institute of Law Enforcement and Criminal Justice in 1977.

Perhaps someone else has more to contribute on the subject.

Sincerely,

John A. Juhala, Ph.D. Chemistry/Biology Group Leader Michigan State Police Forensic Science Division Bridgeport, Michigan 48722



As you can see, this late issue of the AAN is a combined issue. The assistant editors have assured me that future articles will be forthcoming to ensure that the AAN can publish 4 issues in 1984. I sincerely hope that the AAN meets its publication dates in 1984, but feel compelled to state that unless more contributions are received, the sure death of the AAN will result.

Accordingly, the AAN will <u>not</u> accept subscriptions for longer than one year until further notice. Hopefully, this policy will be changed if reader apathy is erased.

Ron Thaman Editor, AAN

* * * FROM THE JOURNALS * *

THE ROLE OF THE ATF FORENSIC CHEMIST ON THE NATIONAL RESPONSE TEAMS. Daniel D. Garner, Ph.D. and Elliott B. Byall, Ph.D., ATF Forensic Science Laboratories, National Laboratory Center, Rockville, MD, and San Francisco Laboratory Center, Treasure Island, CA, respectively.

EXPLOSIVE DEBRIS - PRE-ANALYSIS CLEAN-UP TECHNIQUES USING BONDED PHASE ABSORBENTS. Richard A. Strobel and Richard E. Tontarski, Jr., Chemists, Bureau of Alcohol, Tobacco and Firearms, National Laboratory Center, 1401 Research Blvd., Rockville, MD 20850.

EVALUATION OF CAPILLARY GAS CHROMATOGRAPHY COUPLED WITH A MASS SELECTIVE DETECTOR FOR FORENSIC APPLICATIONS. David T. Stafford, Ph.D. and Amado Maijub, University of Tennessee, 3 N. Dunlap Street, Memphis TN 38163.

A QUANTITATIVE METHOD FOR THE INVESTIGATION OF SELF-HEATING PROCESSES AS FIRE CAUSES. Guenter Hellmiss, Dr. rer.nat., Wolfgang Schwanebeck, Dr. Ing., Bundeskriminalamt, Thaerstr. 11, D-6200, Wiesbaden, W. Germany.

(The above 4 articles were presented at the AAFS meeting in Anaheim, California -- submitted by John DeHaan.)

* * * * *

ANALYSIS OF ARSON ACCELERANTS BY SHORT CAPILLARY COLUMN GAS CHROMATOGRAPHY. Chris W. Beheim, B.S., Alaska State Troopers Crime Laboratory, Anchorage, Alaska.

TRACE EVIDENCE AND THE POLARIZING MICROSCOPE: 1 EXPLOSIVES. Walter C. McCrone, Ph.D., Mcrone Research Institute, Chicago, IL.

BOMBER OR BOMBING VICTIM: CASE STUDY OF A BOMBING USING A CLAYMORE MINE. Richard A. Stroebel, B.S. and Laurence E. Casey, Bureau of Alcohol, Tobacco and Firearms, 1401 Research Blvd., Rockville, MD 20850.

FORENSIC EVIDENCE IN THE LAS VEGAS HILTON FIRE: IMPACT WITHOUT TESTIMONY. Richard E. Tontarski, Jr., Bureau of Alcohol, Tobacco and Firearms, Rockville, MD; Bernard A. Schwartz, U.S. Consumer Product Safety Commission, Gaithersburg, MD; and John F. Rice, Bureau of Alcohol, Tobacco and Firearms, Las Vegas, NV.

RESIDUE MATERIALS FOR LABORATORY TESTING IN AN ARSON INVESTIGATION.

John Reed Davis, B.S.M.E., P.E., Consulting Engineer, Springfield, PA.

(The above 5 articles were presented at the AAFS meeting in Cincinnati, Ohio -- submitted by John DeHaan.)



NATIONAL ORGANIZATION FOR VICTIM ASSISTANCE

1757 Park Road, N.W. Washington, D.C. 20010 (202) 232-8560

PRESS RELEASE

APRIL 2, 1984 -- FOR IMMEDIATE RELEASE CONTACT: Adele D. Terrell -- (202) 232-8560

10th ANNUAL NORTH AMERICAN VICTIM ASSISTANCE CONFERENCE

The National Organization for Victim Assistance is pleased to announce its Tenth Annual Victim Assistance Conference to be held at the Savery Hotel in downtown Des Moines, Iowa, October 7 through 10, 1984. The theme of this year's conference is, "1975-1984: A Decade of Accomplishment".

Workshops at the conference will again reflect the wide range of interests of people active in the victims' movement. One workshop track will cover special victims such as battered spouses, surviving family members of homicide victims, and sexual assault victims. Another track, on victims' movement organizations, will cover self-help therapy groups, special interest coalitions, and victim rights' advocacy groups. Other tracks are on advanced program management, legislation, and victim counseling.

On Sunday, October 7, NOVA will sponsor pre-conference workshops for four allied professional groups: law enforcement, prosecution, the mental health community, and the judiciary.

For further information on the conference, contact:

Conference Coordinator

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Fact Sheet

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NEW RESOURCE WILL HELP CRIME PREVENTION/SECURITY PRACTITIONERS

To help crime prevention/security practitioners find relevant research on crime prevention topics and to discover how other communities address problems of crime and fear of crime, the National Institute of Justice/NCJRS announces a series of bibliographies--topical searches on crime prevention.

Each topical search contains 30 single-page summaries, with bibliographic information, of crime prevention <u>programs</u> and <u>research</u> designed to assist practitioners in finding answers to crime prevention problems. Topical searches are available on the following subjects:

- o Arson
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- o Business Crime
- o Campus Crime and Police
- o Community Crime Prevention
- o Crime and the Elderly
- o Crime Prevention Through Environmental Design
- o Employee Theft
- o Evaluation of Crime Prevention Programs
- o Intrusion Alarms
- o Private Police

Many of the documents described in topical searches are available from publishers (addresses are provided), or on free microfiche from the National Institute of Justice/NCJRS. In addition, all may be borrowed on interlibrary loan from NCJRS.

The cost of a topical search is \$5.00 for each title. Send your order, with payment, to: National Institute of Justice/NCJRS, Attention: Dept. F, Box 6000, Rockville, MD 20850. For additional information about topical searches, other bibliographies, or additional aspects of crime prevention and security, contact NCJRS Customer Service or telephone 301/251-5500.

Topical searches are free to public criminal justice agencies. Send requests for free searches on <u>agency letterhead</u> to: National Institute of Justice/NCJRS, Attention: Customer Service, Box 6000, Rockville, MD 20850.

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The bibliography is easy to use, with both geographic and subject indexes. Many of the documents described are available from publishers (addresses are provided), or on free microfiche from NCJRS. In addition, all may be borrowed on interlibrary loan from NCJRS.

The cost of this bibliography is \$17.50. For additional information about this and other topical bibliographies or other aspects of security, contact: National Institute of Justice/NCJRS, Box 6000, Rockville, Maryland 20850; telephone 301/251-5500.

Fact Sheet

Bureau of Justice Statistics/ 115 Box 6000, Rockville, MD 20850 (301) 251-5500

"Report to the Nation on Crime and Justice" Available Free From National Criminal Justice Reference Service

How much crime is there? Who does it strike? When? Where? Who is the typical offender? What happens to him? What are the costs of justice? Who pays? These and other questions are answered in a free 112-page book published by the Bureau of Justice Statistics and now available from the National Criminal Justice Reference Service.

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United States Department of Justice

Office of Juvenile Justice and Delinquency Prevention National Advisory Committee for Juvenile Justice and Delinquency Prevention

Washington, D.C. 20531

FOR IMMEDIATE RELEASE

Contact: Michael Cromartie (202) 457-6704

April 2, 1984

NAC ANNOUNCES RECOMMENDATIONS FOR NEW FEDERAL INITIATIVE CONCERNING SERIOUS JUVENILE CRIME

The National Advisory Committee (NAC) for Juvenile Justice and Delinquency Prevention just released a report to the President and the Congress entitled, Serious Juvenile Crime:

A Redirected Federal Effort. The NAC is appointed by the President and consists of 15 nembers who advise the President on the prevention and treatment of juvenile delinquency.

The report recommends that Federal policy in the field of delinquency be reformulated to focus primarily on the serious juvenile offender. In submitting this report, Charles B. (Bud) Wilkinson, Chairman of the NAC, observed:

"Numerous studies -- and our own experience -- demonstrate that most serious juvenile crime is committed by a small core of chronic offenders. Logic suggests that a dramatic reduction in juvenile crime could be achieved if we improve our ability to identify, apprehend, prosecute and treat or incarcerate these juveniles. The NAC believes this ought to be the principal focus of a redirected Federal effort in the juvenile justice area."

The NAC report is particularly timely as the statutory authorization for the Office of Juvenile Justice and Delinquency Prevention expires in September of this year. While the report does not take a position on the issue of reauthorization of the Office, it encourages a new federal initiative targeted on the small core of youth who are responsible for much of the serious and violent crime that plaque our nation.

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