Arson Analysis Newsletter

CONTENTS

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March 1983

NUMBER 2

MASS FRAGMENTOGRAPHY IN ARSON ANALYSIS

by

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and
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A familiar technique in arson analysis is gas chromatography, which produces the separation of a mixture of chemicals. Another technique with increasing popularity in arson work is mass spectrometry coupled with gas chromatography: the mass spectrometer creates fragment ions from the compounds in the chromatographic effluent as it emerges from the column. The chromatographic patterns of fragment ions may be used for specific identification. In the method of mass spectrometric detection known as mass fragmentography a limited number of masses are selected for detection, consequently enhancing the sensitivity of measurement. The acquired data is stored in a computer and an "ion chromatogram" is generated by the computer for each selected mass fragment number. Each chromatogram represents the chemicals which contain the selected mass fragment number. These mass fragmentography chromatograms are then compared to known mass chromatograms for identification.

Mass fragmentography has been applied in biological identifications(1)(2), drug research(3), geochemistry(4)(5), toxicology(6), and pollutant identifications(7). Now it is being used in arson analysis(8)(9).

Mass fragmentography is used in our laboratory as an aid in identifying questionable samples and for the detection of lead

additives in gasoline (Figure 1). A mass chromatogram of a sample can be obtained in ten minutes, and can sometimes help "clean up" the chromatogram of an unknown sample. For example, pyrolysis products of materials, such as rubber-backed carpets, can mask a residue of gasoline or even kerosene in a gas chromatogram and make it difficult to identify the accelerant. Since each of the "ion chromatograms" reflects a different aspect of the chemical nature of the injected specimen, they frequently permit easier and more certain identification or exclusion of accelerants (Figures 4, 5 and 6).

The mass numbers 91, 105, and 119 ($C_7H_7^+$, $C_8H_9^+$ and $C_9H_{11}^+$) were selected for ion monitoring of gas chromatographic effluents because these are the weights of ions characteristic of the electron impact fragmentation of alkyl benzenes. Thus, chromatograms based on the abundance of these ions enhance the relative response to these important constituents of gasoline and similar petroleum distillates. Similarly, mass 71 ($C_5H_{11}^+$) was selected to monitor the occurence of alkanes in the chromatogram, enhancing the response to diesel fuel or kerosene and other saturate-rich petroleum distillates. Chromatograms based on these ions reveal at a glance whether a petroleum distillate is rich in aromatic hydrocarbons or in alkanes and tends to reduce interference from non-hydrocarbon compounds in the chromatographic effluent (Figures 2, 4). Mass numbers 206, 208, 237 and 252 (^{206}Pb, ^{208}Pb, $^{208}PbC_2H_5^+$, $^{208}PbC_3H_8^+$) were selected for the identification of lead additives in gasoline. (Figure 3). We have experimented with masses 93 and 136 (characteristic ions from the fragmentation of terpenes)(10)(11)(Figures 7, 8) and masses 128 and 142 (characteristic of naphthalenes)(12). These appear

in mass spectra of petroleum products (Figures 9, 10, 11), but not residue from burned carpet (Figure 12) or linoleum (Figure 13).

The GC/MS used is a Finnigan 3200 gas chromatograph/mass spectrometer with a Finnigan 6100 data system. The instrument conditions are:

	HYDROCARBONS	LEAD ADDITIVES
Mass range:	(71, 91, 105, 119)	(206, 208, 237, 252)
Integration time:	M	M
Scans/sec:	1	1
Threshold:	1	1
Max run time:	30	30
Pre amp. sensitivity:	10^{-7} range	10^{-8} range
Oven temp:	50°C for 1 min.	50°C for 30 sec.
Temp program:	20/min to 245°C	20/min to 245°C
Injector temp:	250°	250°

Column: glass, 5 foot, 3% OV-1

Our utilization of mass spectrometry on accelerants needs further development. More suitable columns, an expanded data library of known accelerants and control samples, and further exploration of the use of other ion masses to characterize additional classes of compounds of interest are needed.

We have used an OV-1 column thus far simply because it is usually the one in the GC/MS, having been selected for drug analysis. Our arson accelerant specimens are first subjected to temperature programmed gas chromatography on a 12 ft. column packed with 10% SP-2100, the effluent being detected by flame ionization. A column of that sort or another with equal or better resolving power would be expected to provide improvement in the application of mass fragmentography to the analysis of accelerants.

1. Hammar, C.G., Holmstedt, B., Ryhage, R., Analytical Biochemistry, Vol. 25, 1968, pp. 532-547.

2. Eyem, J., Advances in Mass Spectrometry, Vol. 78, 1978, pp. 1534-1539.

3. Strong, J., Atkinson, A., Finnegan Spectra, Vol. 4, No. 3, 1974

4. Philip, R., Spectra, Vol. 6, Sept. 1982.

5. Hites, R., Biemann, K., Analytical Chemistry, Vol. 42, 1970, p. 855.

6. Zoro, J., Hadhy, K., Journal of The Forensic Science Society, Vol. 16, 1976, p. 103.

7. Van Vacck, L., Van Cauwenberghe, K., Advances in Mass Spectrometry, Vol. 8B, 1980, p. 1436.

8. Smith, R., Analytical Chemistry, Vol. 54, 1982, p. 1399A.

9. Trimpe, M., Dean, W., Arson Analysis Newsletter, Vol. 5, 1981 p. 94.

10. Hamming, M., Foster, N., Interpretation of Mass Spectra of Organic Compounds, Academic Press, New York, 1972.

11. Lowry, W., Stone, I., Lamonte, J., "Scientific Assistance in Arson Investigation", a report prepared for the American Society of Crime Laboratory Directors, June 1977.

12. Harris, H., "Application of Gas Chromatography-Mass Spectroscopy to Suspicious Burning cases", presented at the American Academy of Forensic Sciences meeting, Feb. 1982.

Figure 1.

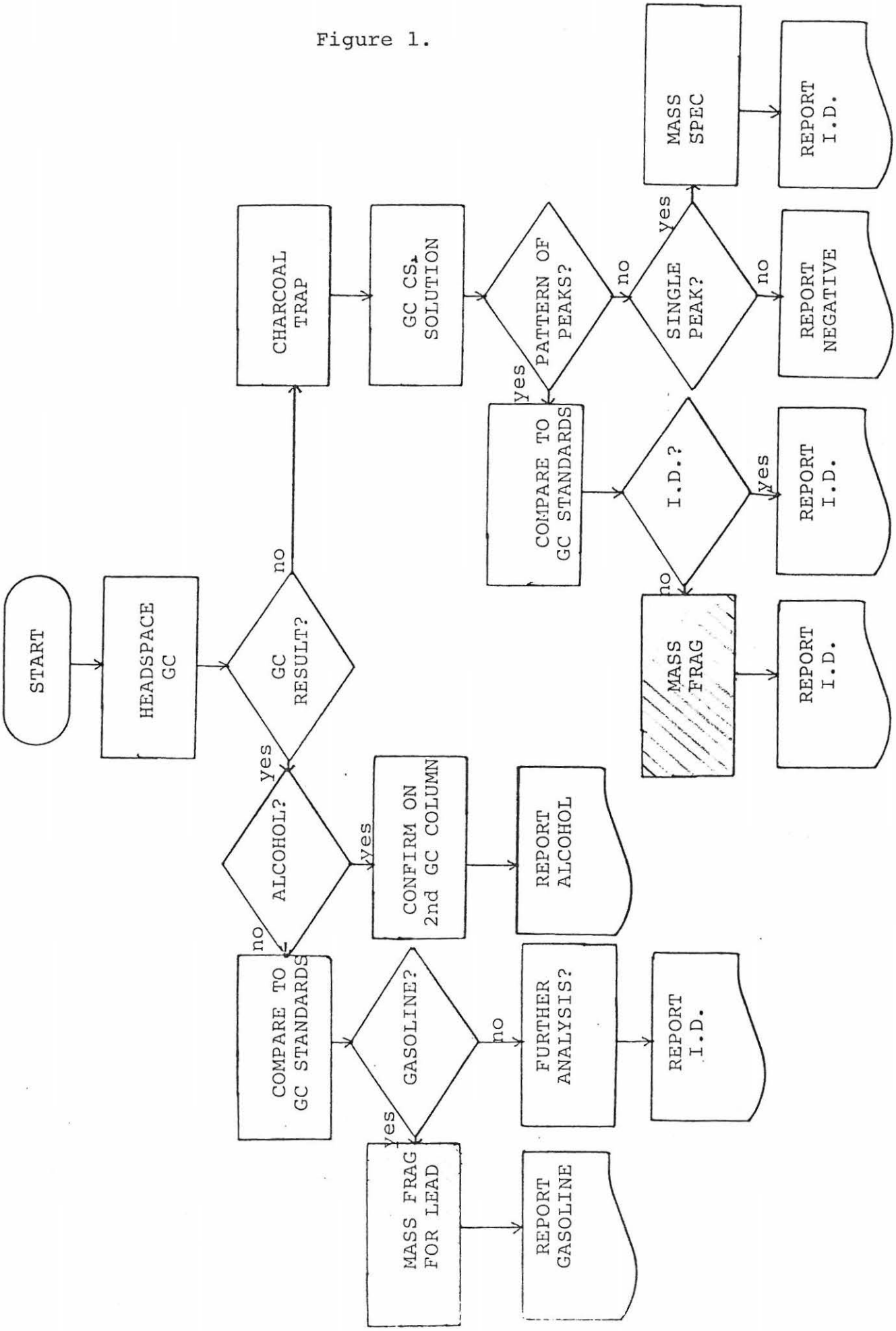

START
HEADSPACE GC
GC RESULT?
no
yes
CHARCOAL TRAP
GC CS. SOLUTION
PATTERN OF PEAKS?
yes
no
SINGLE PEAK?
yes
no
MASS SPEC
REPORT I.D.
REPORT NEGATIVE
COMPARE TO GC STANDARDS
I.D.?
yes
no
REPORT I.D.
MASS FRAG
REPORT I.D.
ALCOHOL?
yes
no
CONFIRM ON 2nd GC COLUMN
REPORT ALCOHOL
COMPARE TO GC STANDARDS
GASOLINE?
yes
no
MASS FRAG FOR LEAD
REPORT GASOLINE
FURTHER ANALYSIS?
REPORT I.D.

Figure 2.

MT3
HYDROCARBON MIXTUR
(71, 91, 105, 119)
M
1
1
30

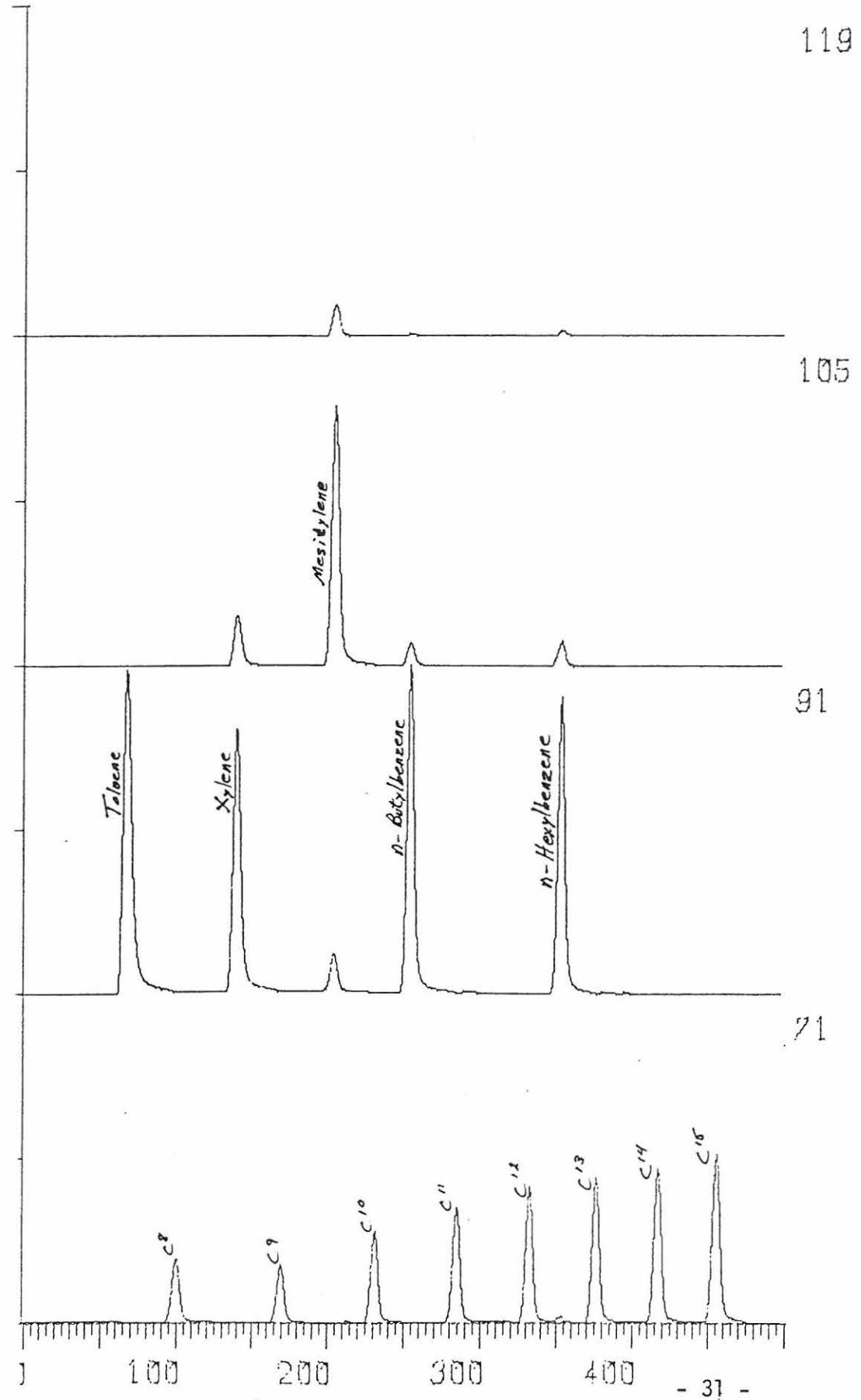


Figure 3.

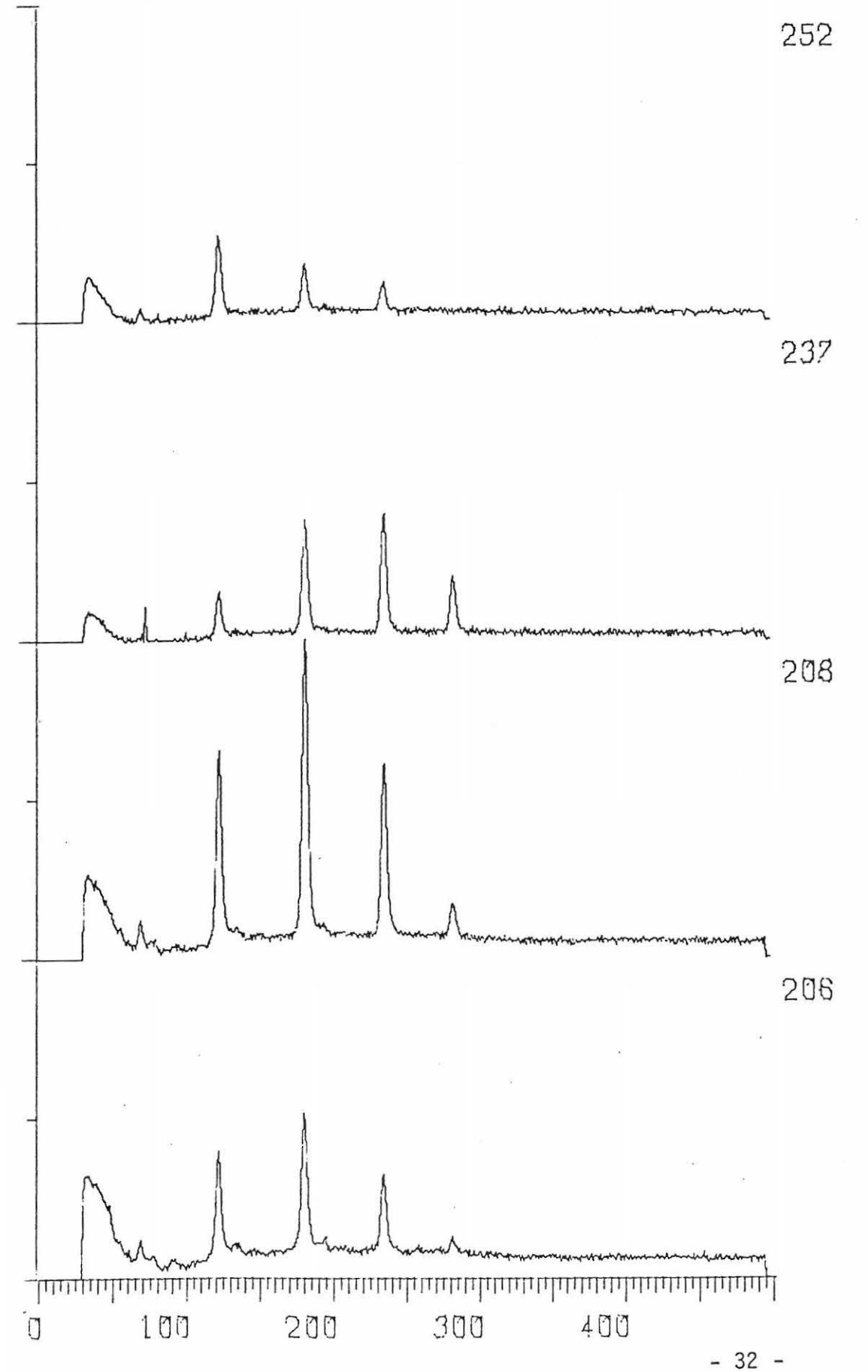

MT2
TEXACO REGULAR GASOLINE LEAD ADDITIVES
252
237
208
206
0
100
200
300
400

Figure 4.

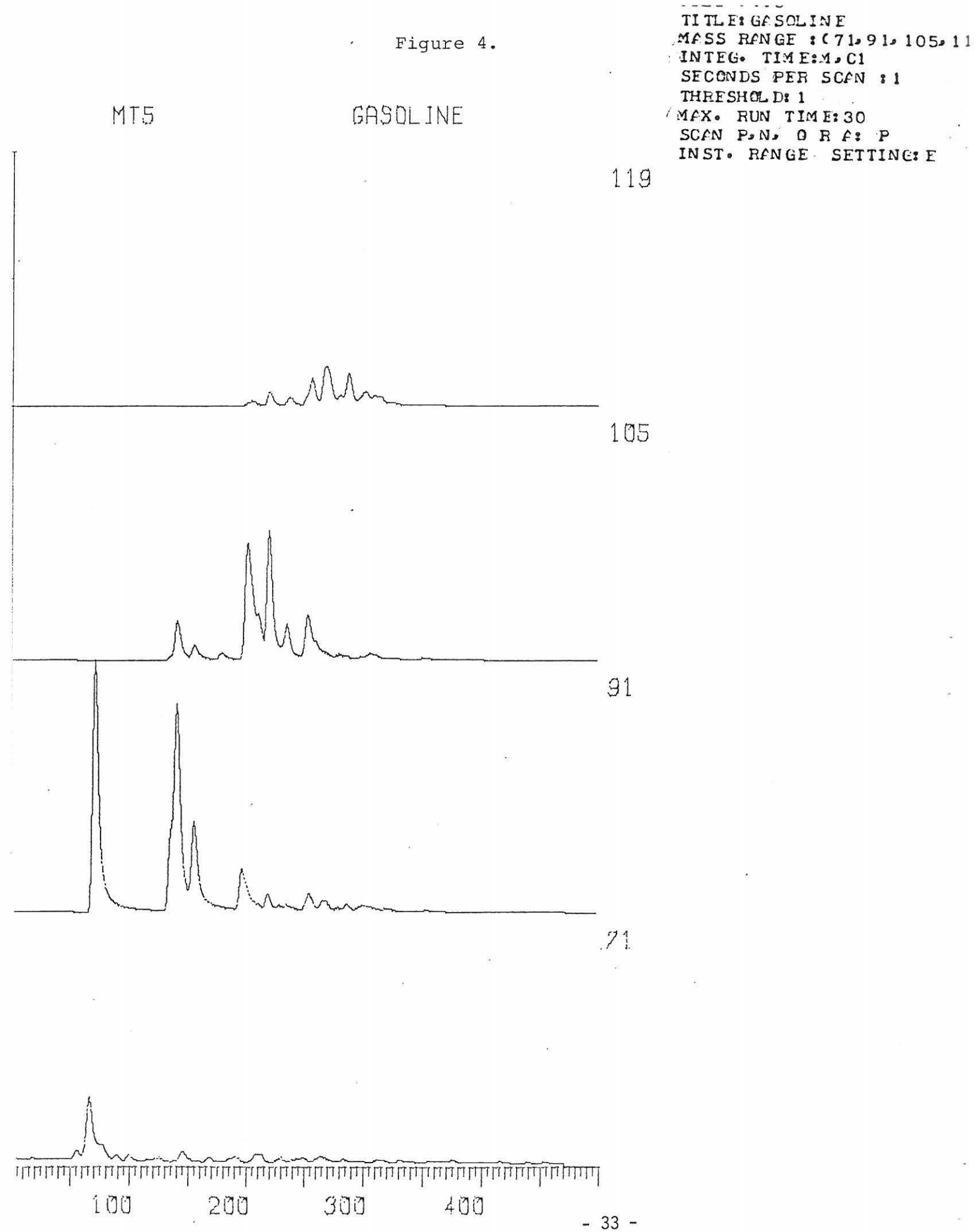

TITLE: GASOLINE
MASS RANGE : (71, 91, 105, 11
INTEG. TIME: M, C1
SECONDS PER SCAN : 1
THRESHOLD: 1
MAX. RUN TIME: 30
SCAN P, N, O R A: P
INST. RANGE SETTING: E
MT5
GASOLINE
119
105
91
71
100
200
300
400

Figure 5.

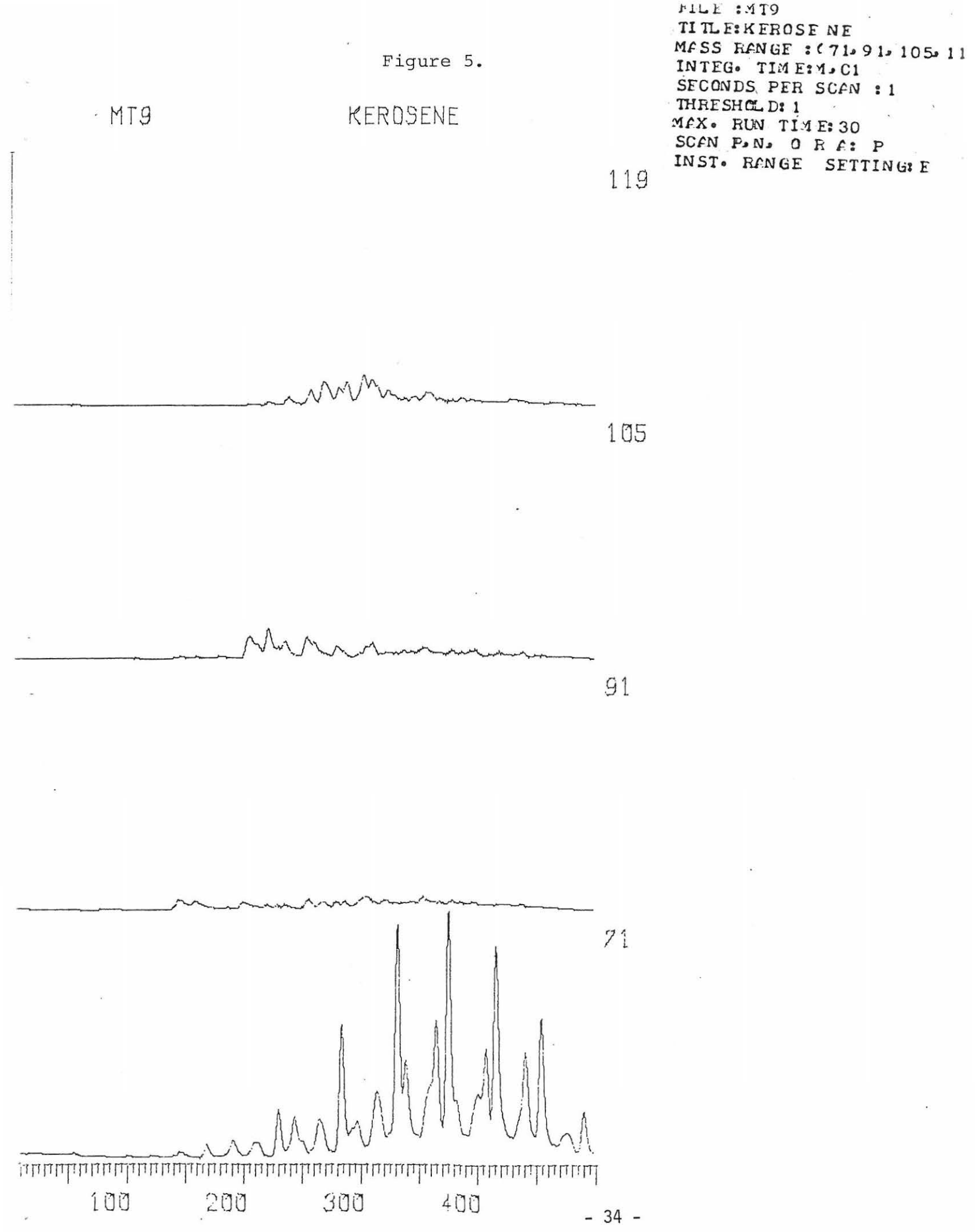

MT9
KEROSENE
FILE :MT9
TITLE:KEROSENE
MASS RANGE :(71, 91, 105, 11
INTEG. TIME:M,C1
SECONDS PER SCAN : 1
THRESHOLD: 1
MAX. RUN TIME:30
SCAN P.N. 0 R A: P
INST. RANGE SETTING: E
119
105
91
71
100
200
300
400

Figure 6.

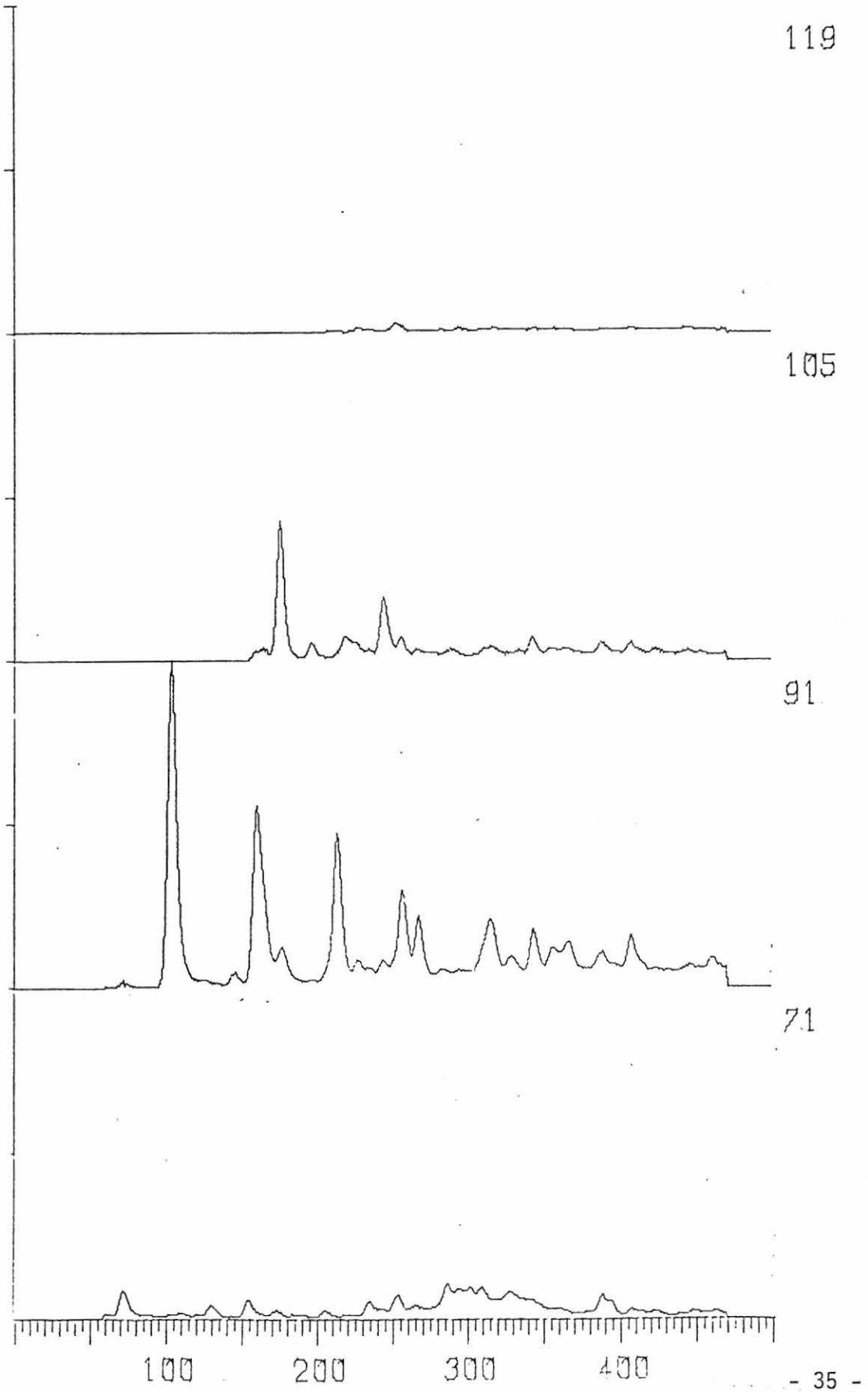


Figure 7.

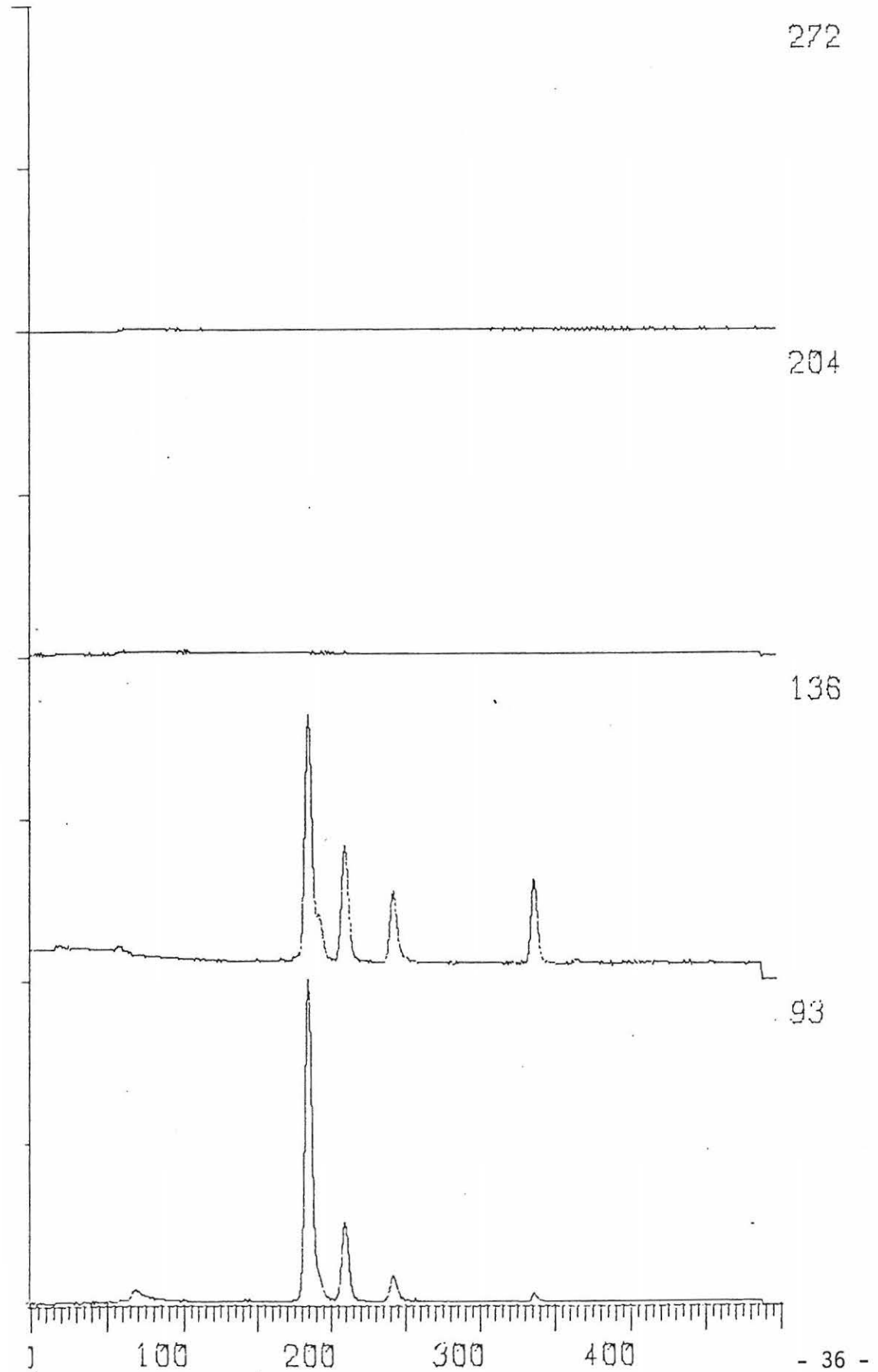


Figure 8.

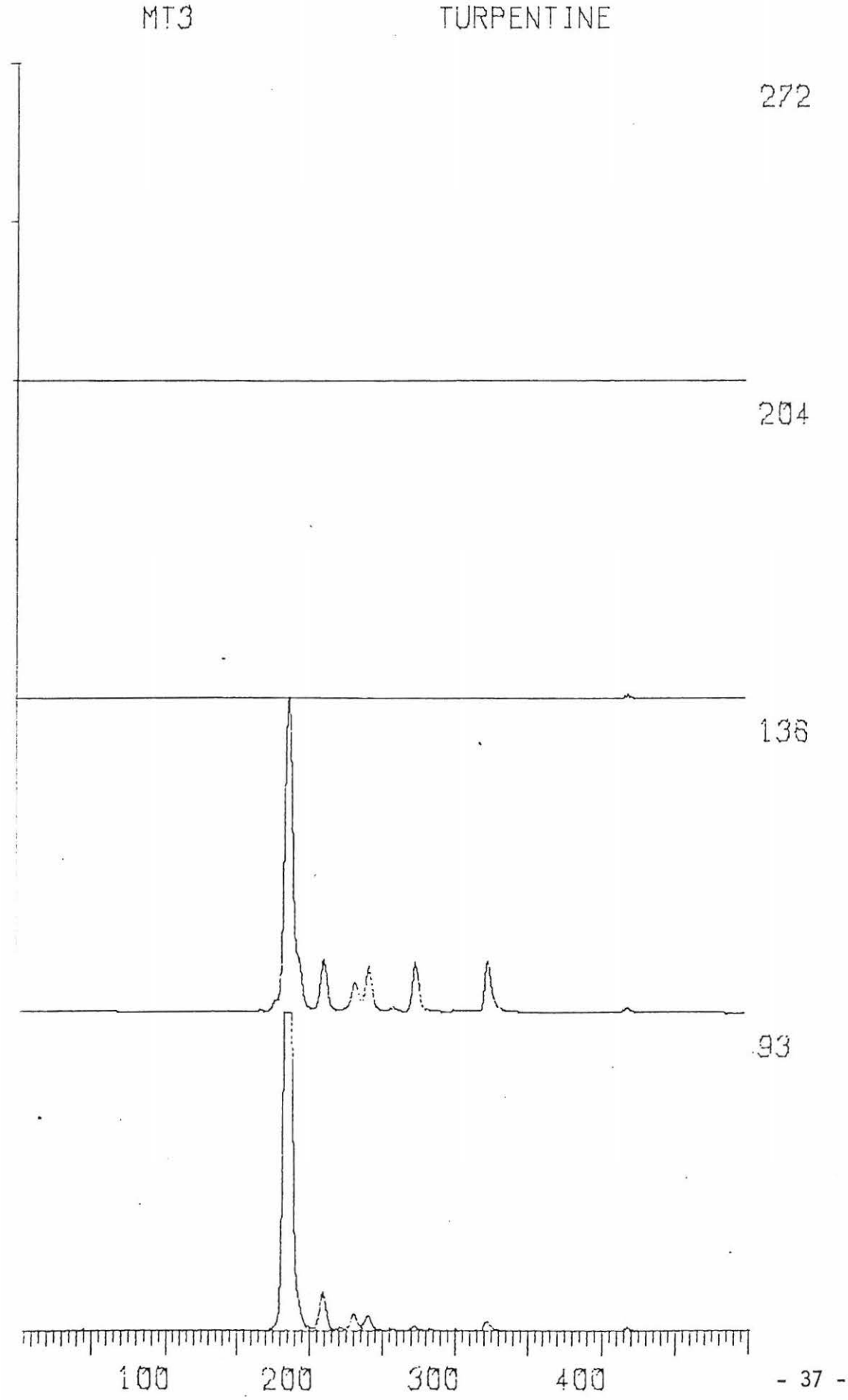


Figure 9.

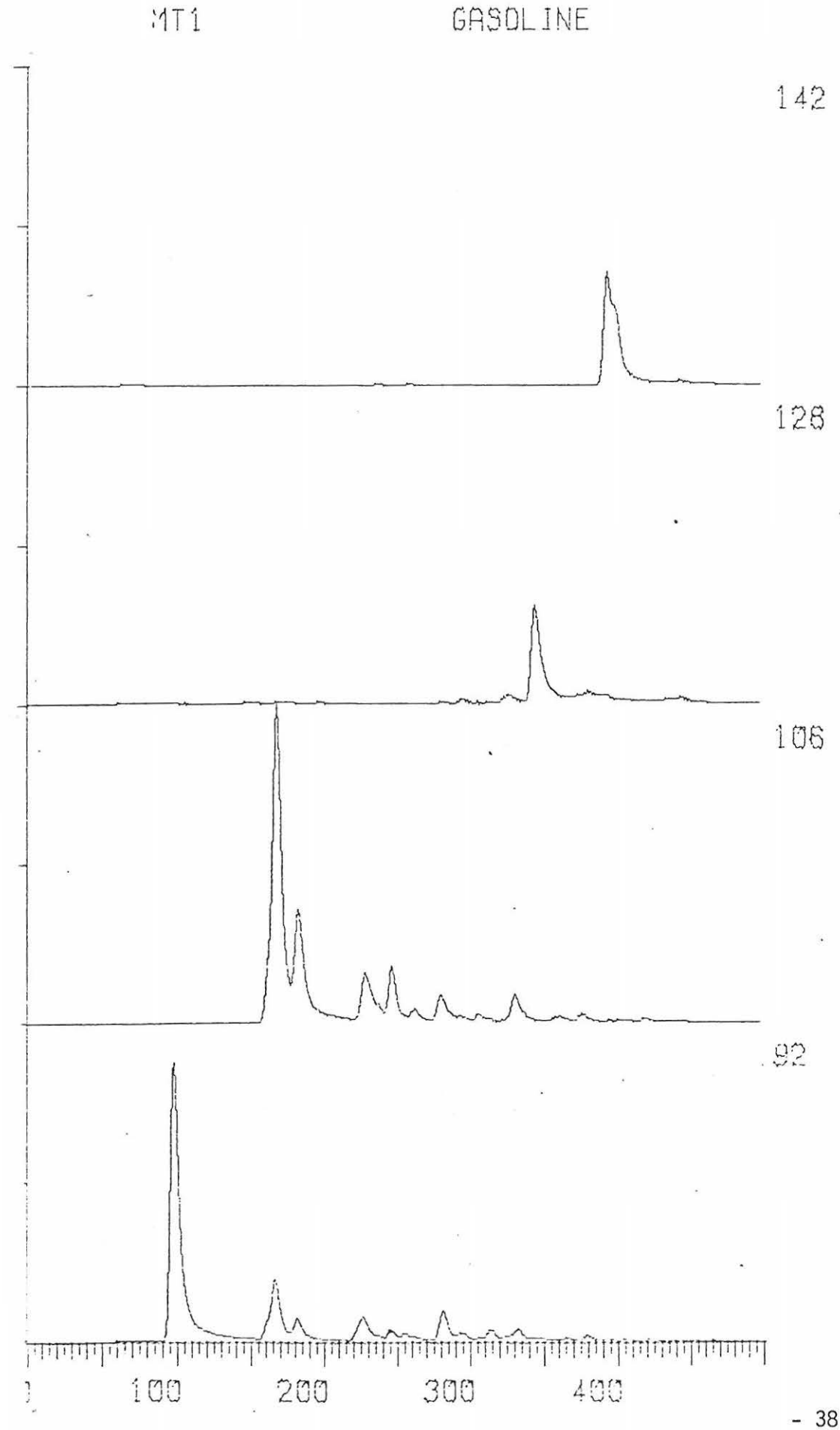


Figure 10.

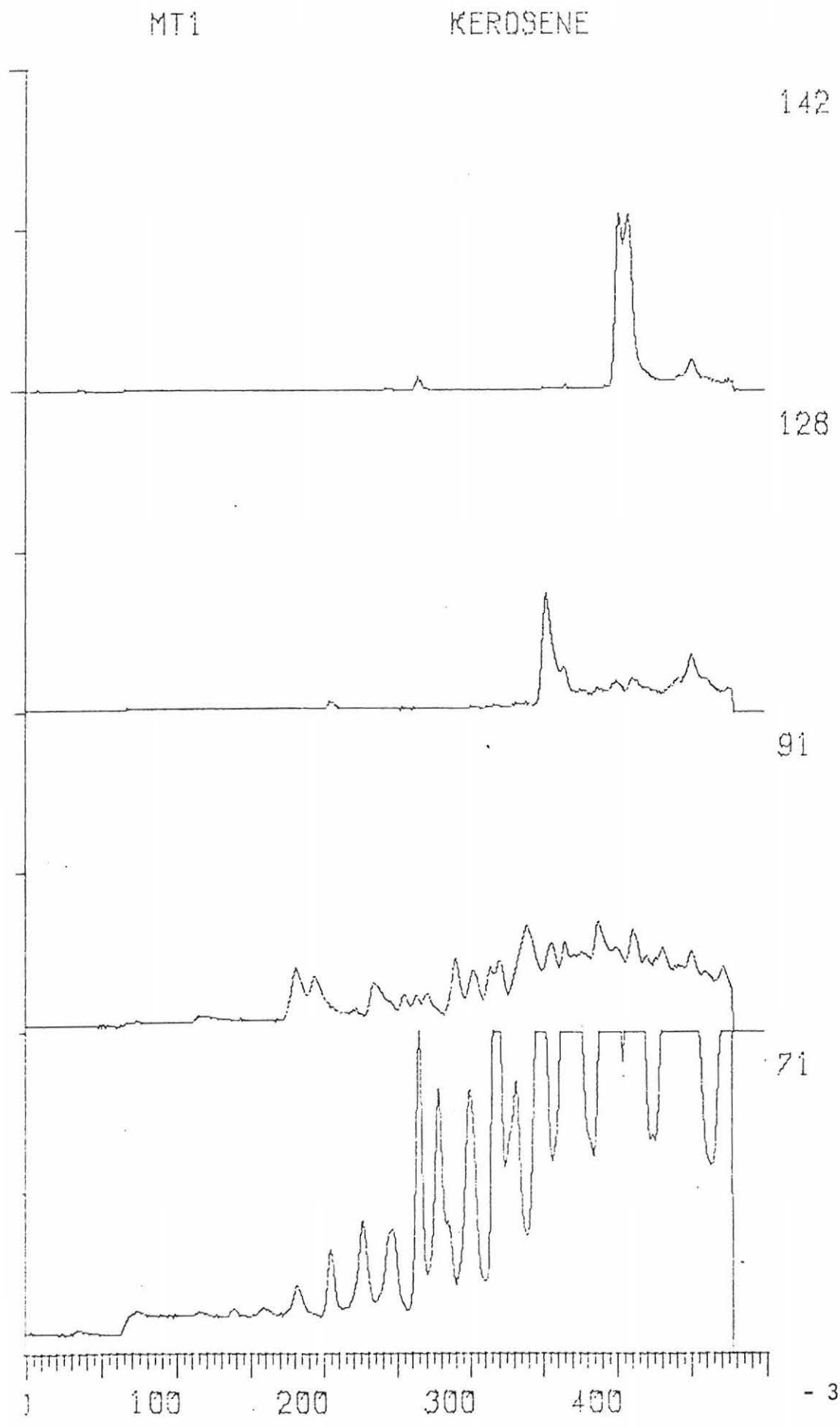


Figure 11.

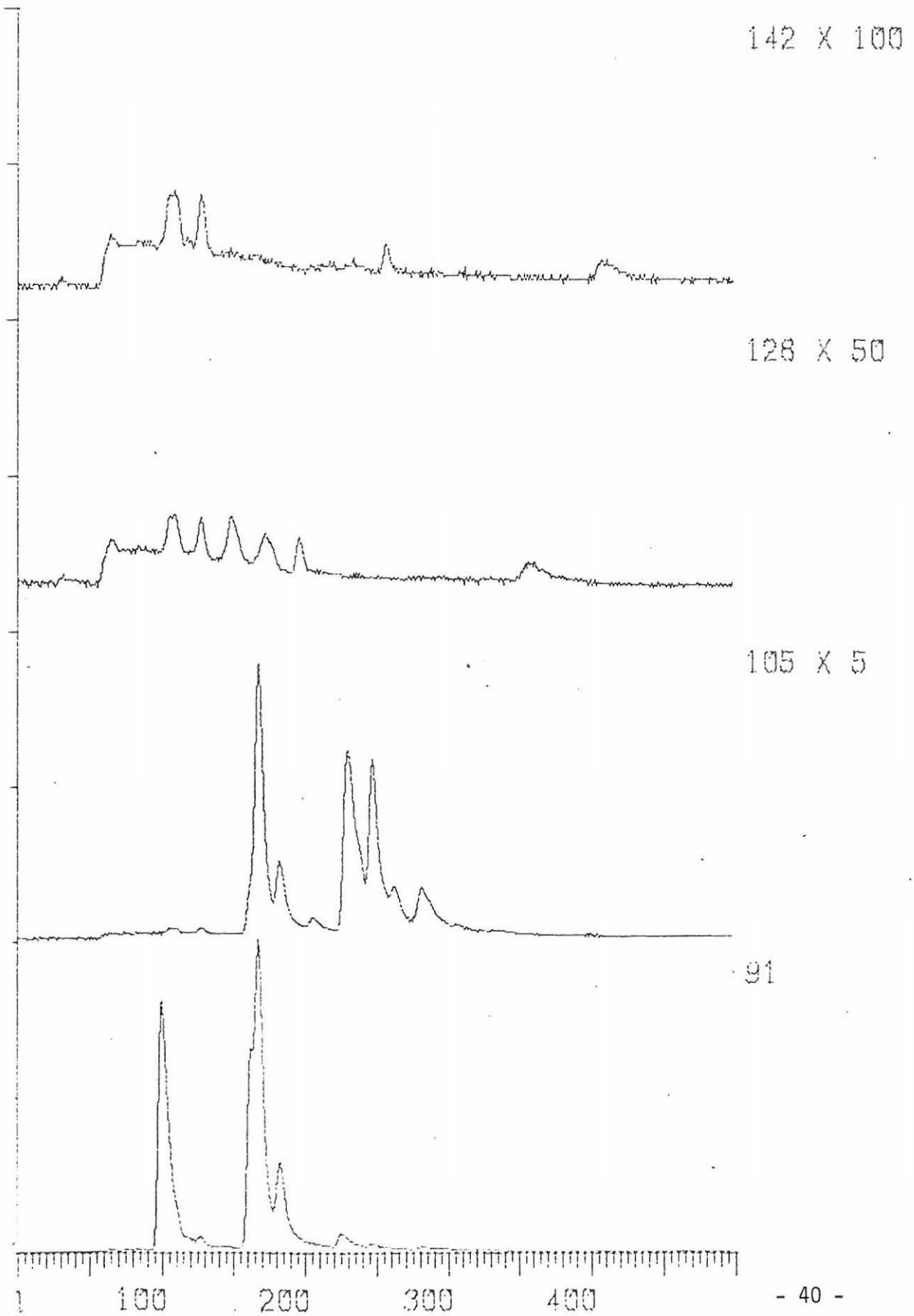


Figure 12.

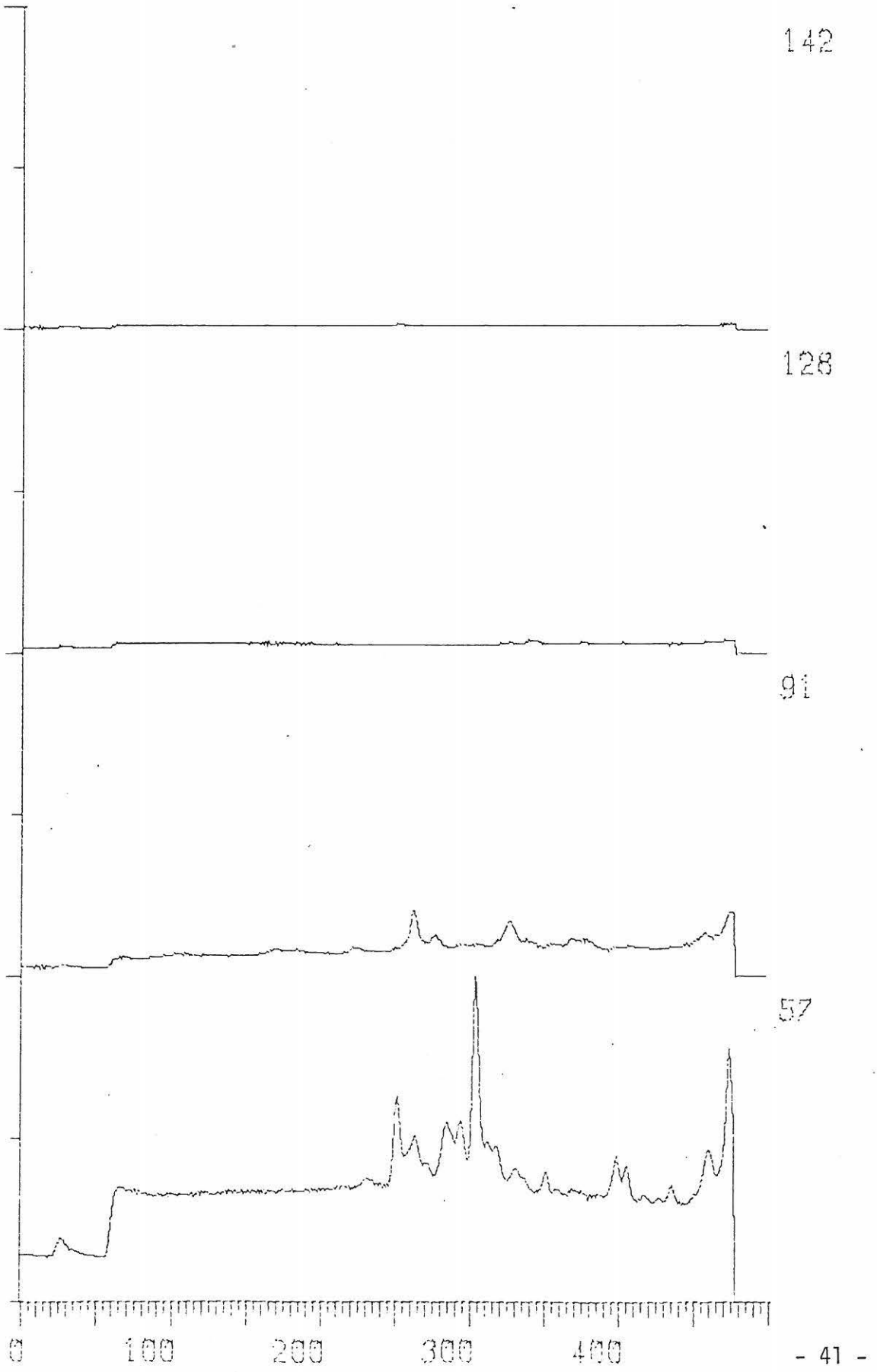
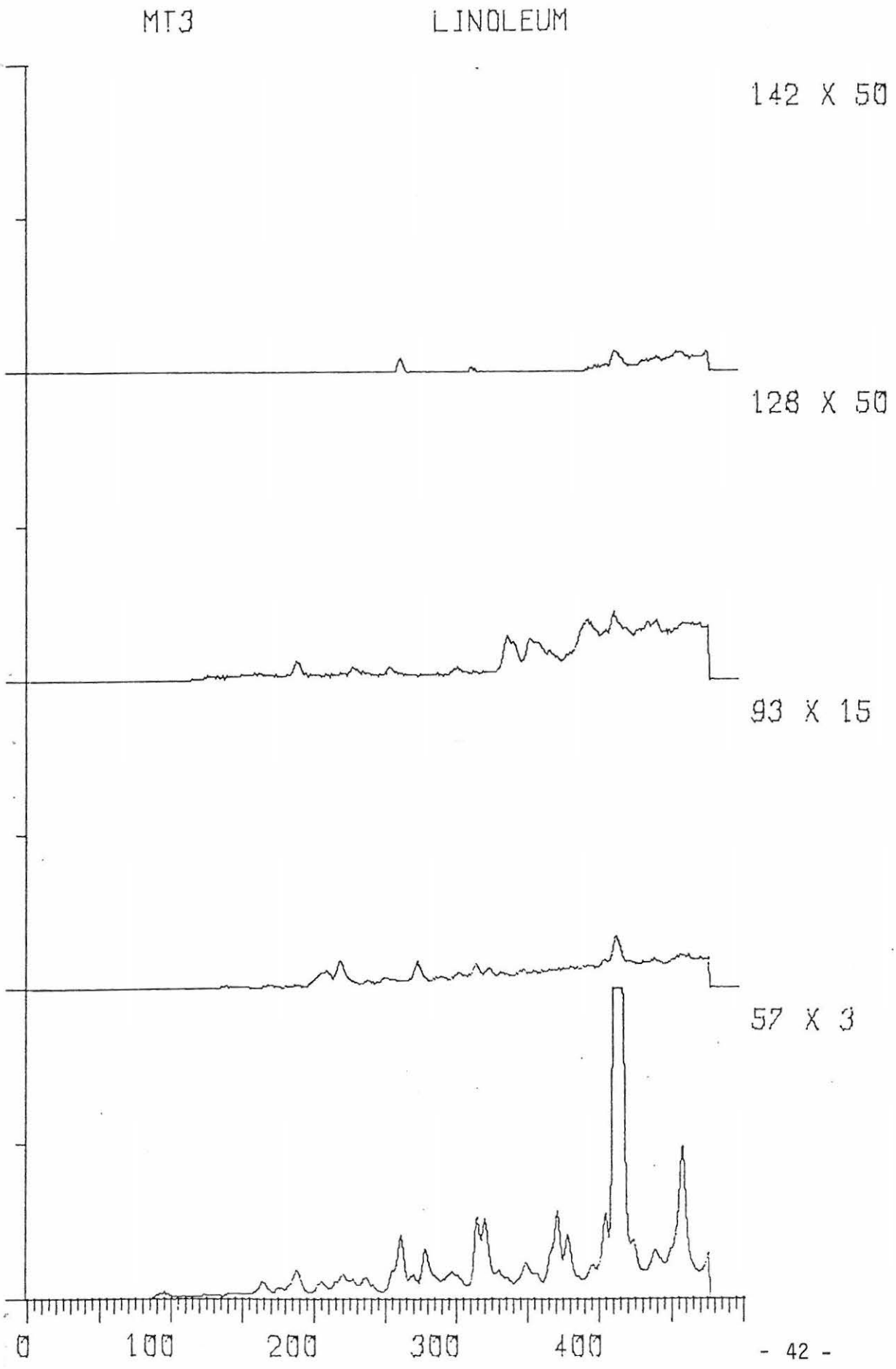


Figure 13.



SIMPLIFIED TECHNIQUE FOR CHARCOAL ABSORPTION/ELUTION OF ARSON SAMPLES*

by

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Springfield, Illinois 62703

INTRODUCTION

This article is a report on two general topics. First, a modification of the charcoal absorption/elution (CAE) technique for collection of volatiles from arson samples; and second, general comments on the analysis of arson samples. What is presented in relation to the first is not a new technique, but rather a simplification of an existing one. The general comments are not the result of specific research, but are based on limited experimentation and observations made as a result of reviewing publications and conducting arson analyses.

Recovery techniques. All have certain advantages and disadvantages associated with them, so it is a question of determining what technique best fits the situation. Listed below are some of the more obvious problems encountered with common techniques. CAE is discussed separately.

A. Steam or vacuum distillation
   1. Cumbersome except for small samples; selective sampling required by limited sample size could lead to erroneous results.
   2. Dependent upon physical separation of a liquid sample, resulting in relatively low sensitivity, adding to the small sample problem.
   3. Requires sample transfer from the original container and considerable cleanup to avoid contamination.

B. Solvent extraction
   1. Possibility of selective extraction causing an unrepresentative sample.
   2. Difficult with large or porous items, also see A1.
   3. Loss of volatiles while concentrating.
   4. Expensive, if pure solvents are used.
   5. Requires extensive cleanup of apparatus.
   6. Possible extraction of substrates such as glues or resins from plywood, etc.

C. Headspace
   1. Risk of sample loss while heating.
   2. Provides incomplete sample for injection either with unheated or heated samples; possibly resulting in identification problems.
   3. No control over the amount of volatiles included in the sample injected into the GC, resulting in lack of reproducibility with repeat injections.
   4. Lack of correlation between GC headspace and liquid GC injections.

* Presented at the Spring, 1982 MAFS meeting in St. Louis

Ideal recovery technique. Such a technique would have certain characteristics, although some may not be attainable. The most desirable characteristics would be:

A. Recovery of the complete, or at least a representative, sample of the volatiles present.
B. Separation of the added volatiles from the pyrolysis products.
C. Ability to utilize all of the available sample for the extraction.
D. Recovery of sufficient homogeneous sample to allow for more than one analysis.
E. Apparatus allows for convenient and complete cleanup.
F. Minimum processing time needed.
G. Low cost.
H. Ability to recover small amounts of volatiles.

PROCEDURE

Charcoal absorption/elution. This technique has been promoted as being the most satisfactory recovery method. The most frequent problem is reported to be contamination of the collection apparatus causing carry-over to subsequent samples. An adapted can lid was not available and others have demonstrated this to be a prime cause of contamination, so a vacuum oven was tried using a collection tube in the outlet. This was found to have significant problems with carry-over. Consequently, a setup was sought which would provide for heating of various sized samples without making sample transfers and avoiding carry-over by direct insertion of the collection tube into the original container. This would also provide for extraction of the complete sample.

An ice pick was being utilized to facilitate headspace extractions and since this provided an opening which was adequate for insertion of a collection tube, the only problem was to provide a seal between the tube and the container. This was accomplished by using black plastic electrical tape over the hole and inserting the tube through the tape. Different types of tape have been used by others with apparently equal success. When inserting the tube, it will occasionally punch out a circle of the tape blocking the end of the tube, so the system must be checked for free air flow.

Extraction. After making two holes in the lid and covering them with tape, the can is placed in a heating mantle and the tubes inserted. Two tubes are used, one to filter the air going into the can and the other as the collection tube. A vacuum line is attached to the collection tube and the extraction done. A water aspirator was used, but other sources of vacuum could also be utilized.

Tube preparation. This was done in the usual manner, using 50/200 mesh charcoal in Pasteur pipettes. Initially glass wool was used, but it was found that only fine, short fiber wool worked well. Because of the problems inherent in handling glass wool, the packing was changed to absorbent cotton. A problem was noted with wet (with water) samples in that as the cotton becomes wet, it tends to slide in the tube because of the air flow. In some instances it has gone past the tube restriction. To prevent loss of sample, a trap (Figure I) is used. This trap will hold the cotton in the tube, preventing the loss of the charcoal into the vacuum system.

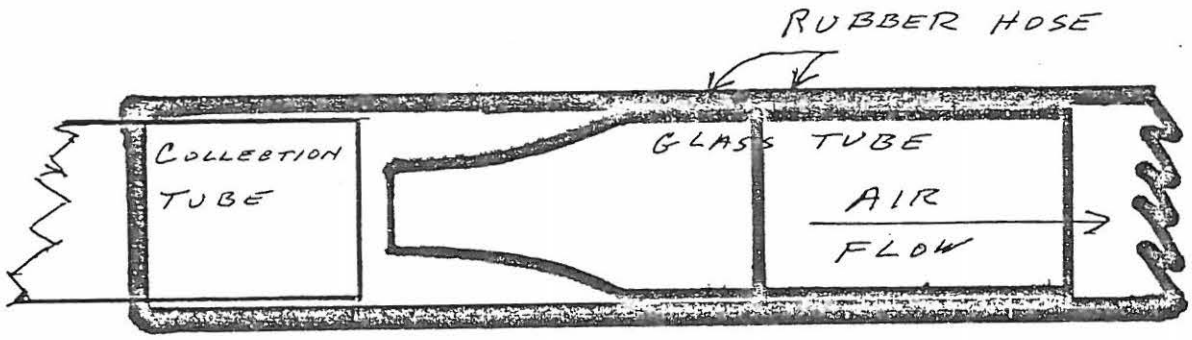

FIGURE I

Elution. Performed with 0.5 to 1 ml of CS_2. Care must be taken not to bring the CS_2 in contact with anything other than glass or metal as contaminants may be introduced. The collection container must be stoppered if the GC run is not to be made within a short time.

COMMENTS

The described technique has been used on several hundred arson samples with no problems other than those noted and corrected. The technique is rapid, clean, inexpensive and the vacuum seal between the tubes and the can is quite complete. In instances where the inlet tube has been clogged, the seal is sufficient to cause the vacuum to collapse the can. It is important to check the flow of air by checking the inlet tube to avoid this.

The following general comments are offered for information and to generate discussion and further study. They have not been researched to the point of being offered as conclusions.

Containers. Paint cans are the most nearly ideal container, although coffee cans with plastic lids are adequate and the same sampling technique will work. Isolating the contents from the plastic lid with aluminum foil is recommended. Interior coated paint cans have been used with no problems noted; negatives have been obtained with samples in coated cans and no significant background has been noted.

Pyrolysis products. In some articles, reduction of pyrolysis products has been cited when using CAE, but the opposite seems to be the case. This could be due to the high sensitivity of the procedure. Care must be taken not to overheat the sample (even in small spots) as this can generate pyrolysis products.

Temperature measurement. This has not been used and its necessity is questioned as it is impossible to accurately measure the temperature throughout the container or even be sure of an "average" temperature due to non-homogeneous samples, also it will not avoid spot overheating. Water addition would be one means of obtaining more uniform heating, but this seems to reduce the recovery of volatiles.

Stripping of high volatiles. Overheating of the collection tube has been cited as a cause for loss of high volatiles; this seems to be more related to overloading of the tube in the presence of large amounts of volatiles. Even when this occurred, it did not seem to interfere with the identification of the volatile material. Using the described procedure, the tubes do not become very warm, but a relatively large volume (400-600 ml/min.) of air was passed through the collection tubes.

Extract cooling. Some procedures call for the immediate cooling of the extracts; this has not been found to be necessary as long as complete evaporation of the CS_2 is prevented. Air evaporation of the CS_2 will cause loss of hydrocarbons up to at least C-10 to 12.

Recovery of high boilers. No problem has been noted with recovering the less volatile portions of #2 fuel oil; in the presence of gasoline in the same sample, this may become a problem.

GC conditions. In determining the most ideal conditions, the purpose of the analysis should be kept in mind. If the goal is to make a detailed comparison, greater separations and longer runs may be advantageous. On the other hand, if the goal is to characterize the volatiles present, the greater resolution may not be needed and only serve to increase the analytical time. A three foot Dexsil column will differentiate between common accelerants and allows for a complete run of #2 fuel oil in ten minutes. This column combined with the described CAE technique will allow about three samples per hour to be run.

SUMMARY

Direct insertion of charcoal absorption tubes into the sample containers allows for extraction of the complete debris sample and avoids the contamination problems resulting from more complicated set-ups. Comments, suggestions and/or questions are invited, either directly to the address listed below or via communications to the Newsletter.

AA Notes

* From the Journals:

  ** A good article detailing the GC/FTIR analysis of terpenes recently appeared in the Journal of Chromatographic Science, Volume 21, May 1983. Small quantities of α-pinene, β-pinene, p-cymene, fenchane, d-carvone, camphor, 1,8-cineole, limonene, α-terpinene, γ-terpinene, pulegane and thymal were analyzed by a Nicolet 7199 and Varian 3700 GC.

* A forensics program devoted to fire debris analysis was recently held at the 8th Annual Spring Workshop of the Association of Official Analytical Chemists in Indianapolis, Indiana on April 19-21, 1983. The program consisted of the following speakers:

  ** Mohamed M. Gohar (Ohio Arson Crime Laboratory) - Quality Assurance as an Important Parameter in Fire Debris Analysis.

  ** Phil L. Wineman, Ph.D. (Bureua of Alcohol, Tobacco and Firearms) - National Bureau of Standards Round Robin Test Program Results.

  ** R. Martin Smith, Ph.D. (Division of Law Enforcement Services Crime Laboratory, Wisc.) - Mass Chromatographic Analysis of Arson Residues.

  ** Ronald N. Thaman (Systems Engineering Associates) - Automated Analysis of Fire Debris Samples.

International Society of Crime Prevention Practitioners, Inc.

May 19, 1983

Dear Editor:

The International Society of Crime Prevention Practitioners will be conducting its Sixth Annual Conference in Columbus, Ohio on Friday, November 18, 1983 through Monday, November 21, 1983. The theme of the conference is "Partners in Prevention - Capture the Spirit."

The Society would appreciate any consideration you might give to informing your readers of the Conference.

If you have any questions or concerns regarding the Society and/or the Conference, please feel free to contact me at (313) 224-4030.

Thanking you in advance for your consideration.

Sincerely,

James Knittel

JAMES KNITTEL
President
ISCPP

JK:jb

Attachment

INTERNATIONAL SOCIETY OF CRIME PREVENTION PRACTITIONERS

6th Annual Conference

Friday, November 18, 1983 - Monday, November 21, 1983

Hyatt Regency-Columbus at Ohio Center

"Partners in Prevention-Capture the Spirit"

In partnership with - -

American Association of Retired Persons (AARP)
American Society of Industrial Security (ASIS)
Federal Bureau of Investigation (FBI)
Columbus Division of Police (C.P.)
Franklin County Sheriffs Office
National Criminal Justice Association (NCJA)
National Crime Prevention Council (NCPC)
National Crime Prevention Institute (NCPI)
National Rural Crime Prevention Center (NRCPC)
Ohio Attorney General's Office
Ohio Crime Prevention Association
Ohio State Highway Patrol
Ohio Office of Criminal Justice Services, Crime Prevention, State of Ohio
Texas Crime Prevention Institute
U. S. Postal Inspection Service
Buckeye State Sheriffs Association (BSSA)
Ohio Association of Chiefs of Police (OACP)

Who Should Attend:

Crime Prevention Practitioners, the business community, civic leaders, firefighters, security progessionals, military officials, volunteers organizations, everyone has a vested interest in reducing the level of crime.

THE CONCEPT

This years theme, "Partners in Prevention-Capture the Spirit", highlights the basic concept of crime prevention, people working together to fight crime. Each day of the Conference seminars will be held concurrently with topics attracting various audiences with a vested interest in crime prevention. The seminars will average 2-3 hours in length. This will maximize the amount of information presented and better equip participants with tools to aide them in their crime prevention efforts.

The location - -

The entire program will be held at the Hyatt Regency-Columbus at Ohio Center. The Hyatt is located on the North edge of downtown Columbus, 15 minutes from Port Columbus, International Airport, within walking distance are all major corporate office complexes, the State Capitol, the Columbus Art Gallery, the Center of Science and Industry, two historical theatres and other points of interest.

The cost:

Full registration (includes one-year ISCPP membership $25.00 value)	$105.00
Full time student (must have paid fee card)	$50.00
One day registration	
ISCPP members	$25.00
Non-members	$35.00
Special one-day registration (non-members) (includes one-year ISCPP membership-$25.00)	$50.00
Monday, November 21, 1983	
Awards banquet, speaker and dance	$25.00

Programs Highlights - -

Training seminars will be offered on the following topics.:

Arson Prevention
Crime Prevention and Private Sector
Security Lighting
Neighborhood Watch
Crime Prevention and the Military
Crime Prevention and the Media
Crime Analysis
Volunteer Programs
Rural Crime
Security Hardware and Electronics
Dispute Mediation
Commercial Crime Prevention
Executive Protection
International Terrorism
Youth Crime Prevention Programs
Construction Crime Prevention
Cost-effective ways & means of developing audio visual aides for Programs
Crime Prevention in the Corporation
Computer Crime Prevention
Drug & Alcohol Prevention
and more.....

UNITED STATES DEPARTMENT OF
COMMERCE
NEWS
WASHINGTON, D.C. 20234

NATIONAL BUREAU
OF
STANDARDS

FOR IMMEDIATE RELEASE:
April 29, 1983

Jan Kosko
301/921-3181

TN-5434

NBS FIRE RESEARCH CONFERENCE

TO HONOR NOTED PROFESSOR

Fire modeling, flame phenomena and spread, fire plumes, and fire suppression and extinction are among the topics to be featured August 23-25, 1983, at a conference on fire research at the Commerce Department's National Bureau of Standards (NBS) in Gaithersburg, Md.

The format for the conference will differ from that of the past few years in which grantees of the NBS Center for Fire Research (CFR) met with CFR staff and others in the fire research field. This year's conference will focus around the areas of fire research in which Professor Howard Emmons, who retires this year from Harvard University, has made significant contributions.

A publication describing research conducted at CFR, the federal government's principal fire research laboratory, and grants and contracts sponsored by CFR during fiscal year 1983 will be available at the conference.

For further information and registration details, contact Sonya Cherry, Polymers Building B260, National Bureau of Standards, Washington, D.C. 20234, 301/921-3845.

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News

March 14, 1983

CONTACT: Robin Maydeck
or
Jack M. Berkman
BERKMAN & DANIELS
Marketing Communications
(619) 234-6151

UNIQUE NATIONAL CONFERENCE ON THE FUTURE OF CRIMINAL JUSTICE SET FOR JUNE IN LOS ANGELES

LOS ANGELES, CA -- Gene Roddenberry, TV and film producer and creator of "Star Trek," and noted criminologist Jerome Skolnick, will be keynote speakers at the first "National Institute on the Criminal Justice System; The Future," a unique forum on future directions of the U.S. criminal justice system, to be held here at the California Polytechnic University Pomona campus June 1 - 3, 1983. Co-sponsors are the National Conference of Christians and Jews (NCCJ), the Claremont College Graduate School and the California Commission on Peace Officer Standards and Training.

"Rapid change is altering the fabric of our society and straining our social institutions," states Glen Poling, program director of NCCJ, creator and coordinator of the institute. "At the center of these buffeting circumstances is the criminal justice system, which has the distinction of bearing the brunt of the chasm between social realities and social expectations."

The purpose of the Institute, said Poling, is to provide criminal justice professionals across the nation with the opportunity to come to grips, through the insights of experts, with the trends affecting several areas of

society that, according to a national survey of professionals, impact on the criminal justice system and their jobs. The Institute has been endorsed by the U.S. Justice Department's Community Relations Service.

The eight key areas that the institute will explore are

- - The Economy of the Future
- - Technology of the Future
- - Demographics of the Future
- - Community Values
- - Budgeting in the Future
- - Productivity
- - Social Issues
- - Community Relations

There will also be two synthesis workshops in the areas of Productivity and Budget, and Social Issues and Community.

Keynoting with Roddenberry, who will be speaking on "How to Look at the Future" and Skolnick, whose topic will be "Managing the Future," will be the U.S. Justice Department's Stanley E. Morris, associate deputy attorney general, who will address the issue of "Criminal Justice System Coordination."

There will be a technological display provided by IBM and a computer link-up to the National Institute of Justice in Washington, D.C.

The registration fee will be $250, which includes room and board. There are discount arrangements available to local and state governments that send interagency teams.

Further information on the National Criminal Justice Institute is available through Glen Poling or Richard Vega of the National Conference of Christians and Jews, 635 S. Harvard, Los Angeles, CA 90010 or by calling (213) 385-0491.

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