

# CASE TEACHING NOTES

for

## “Burning Down the House: A Case Study in Forensic Instrumental Analysis”

by

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### INTRODUCTION

Gas chromatography-mass spectrometry (GC-MS) analysis is a commonly used technique in organic chemistry, quantitative analysis, and instrumental methods courses. Case studies are an effective way to introduce students to GC-MS analysis. This case study was designed for an instrumental methods course, but could also be adapted for a non-science major course.

In reading the case, students find themselves in the middle of a scenario in which they must determine both the nature of a fire and any criminal accusations which may be raised as a result of their findings. Students come to understand that a fire investigator is being accused of burning down her ex-husband's house in a fit of rage, and it is up to them to validate or refute the suspicions of police investigators by analyzing ash samples collected from the scene. In addition, they must analyze a clothing sample collected from the suspect. Once they have determined whether or not the fire was arson, they must then determine if the allegations leveled against the fire investigator are credible. Students are presented with ash samples and cloth samples collected from the scene, and are asked to compare what they find within the samples to accelerant standards whose spectra are already known.

Use of this case depends greatly on the level of experience of the students involved. In an introductory class, this case is best utilized to introduce the principles of GC-MS analysis. In a more advanced course in which students are already familiar with GC-MS analysis, this case is a useful exercise in interpreting mass spectrometry. In any scenario, headspace analysis and the principles of forensic investigation—particularly as they relate to arson investigation—can be introduced and cross disciplinary learning emphasized.

### *Objectives*

Students will:

- Introduce GC-MS analysis.
- Introduce the principles of static headspace analysis.
- Introduce forensic analysis.
- Stimulate interest in chemistry.

### CLASSROOM MANAGEMENT / BLOCKS OF ANALYSIS

#### *Overview*

##### **Pre-Lab**

Depending on the students' experiences with GC-MS analysis, the instructor may want to review its principles in class, prepare an overview handout, or use the review provided in the Supplemental Material for this case. Students should read the case for homework and come prepared to discuss

proposed methodologies which can address the problems presented in it. The students should also have researched and prepared answers for the questions at the end of the case.

If the size of the class is large, the instructor may want to divide the lab into smaller groups, which will allow the students to brainstorm ideas together. For this experiment, these groups may also serve as lab partners, an efficient way of organizing the students' work given that most schools have limited access to GC-MS instruments and the amount of time it takes to complete one run.

After the students have been given about 15 minutes to discuss their ideas on how the case can best be investigated, the instructor should ask groups what ideas they have for analyzing the charred fire samples and cloth sample. The instructor should guide the groups towards the preferred methodology—using static headspace analysis coupled with GC-MS. The instructor should also discuss the advantages and disadvantages of different extraction techniques, including dynamic, passive, and static headspace analysis (see Supplemental Material). Steam distillation is not the preferred method for collecting volatile materials from charred debris because it can be time consuming and it is difficult to collect enough liquid to analyze. Solvent extraction, while useful, is destructive in nature, which may be a disadvantage with limited physical evidence.

At its core, static headspace analysis takes advantage of the high vapor pressure of accelerants to induce them into the gas phase, rendering them available to be withdrawn with an airtight syringe and subjected to GC-MS analysis. Because the analyte being analyzed is in the vapor phase, there are a few important procedural considerations that the instructor should stress in order to ensure a good result:

1. It is necessary to aspirate the syringe between samples to avoid any contamination.
2. To ensure a sufficient vapor phase analyte concentration, students should place vials containing charred samples in a warm water bath for several minutes before attempting headspace analysis. This will allow time for gas phase accelerant concentration to increase.
3. In our procedures we divided the pine wood very finely so that the pieces could fit inside 4 mL amber screw top vials, which are often used in GC-MS analysis.
4. The syringe plunger should be held down for one minute after injecting the sample into GC. Because accelerants have high vapor pressures, the vapor which is extracted from the vials containing the charred samples may have a tendency to push the plunger of the syringe up, thus allowing the gaseous analytes to escape.

To conclude the discussion, the instructor may wish to give some statistics about the widespread nature of arson. According to the United States Fire Administration, 37,500 intentionally set fires resulted in 305 civilian deaths and \$692 million in property damages in 2003 (see <http://www.usfa.fema.gov/>). The ability to characterize arson and convict those who commit it remains an important and widely used skill.

### **Lab**

After groups have determined a correct methodology, the students should then proceed to the analysis. Groups may take turns performing headspace analysis on either standards or unknowns with each run lasting about 15 minutes. Detailed methods are outlined in Figure 1. After all of the standards and charred samples have been run, students may then pool their chromatograms so that each group has access to all of the chromatograms. Based on their findings, groups develop conclusions about the possibility of arson and the guilt or innocence of the suspect in question. They should complete lab reports in accordance with the instructor's directions.

The entire experiment can be accomplished in one laboratory period if each group injects one unknown sample and one standard sample. The groups can then pool their chromatograms to reach individual conclusions.

### **Laboratory Preparation**

For this lab, the instructor will need to create three charred samples and one cloth sample to present to students for analysis. To prepare the charred samples, the instructor should obtain pine wood chips or a piece of pine wood from a local hardware store. The pine board can then be finely divided into pieces small enough to fit inside 125 mL Erlenmeyer flasks. After about 10 small pieces of pine have been added to the flask, the instructor should add three different accelerants to each of the three Erlenmeyer flasks. For the purposes of this lab, gasoline, lighter fluid, isopropyl alcohol, and lamp oil are ideal accelerants to use because all would be easily accessible to potential arsonists and yet, if found within the proper environment of a home, the presence of each could be logically explained. Detailed explanations for the preparation and ignition of the charred samples and the preparation of the cloth sample can be found in [Figure 1](#). The preparation of the charred samples is modeled after the procedure given by Eldred et al. (1996) and Sodeman et al. (2001). The total preparation time is two hours.

### **Results**

GC chromatograms and mass spectra obtained from two standards and charred unknowns are presented in Figures 2 through 4. All of the accelerants used had a distinctive chromatogram which makes identification of unknowns an easy task. The major difference between the chromatograms obtained from the standard accelerants and those obtained from the charred samples is the relative intensity of the peaks—yet, owing to their unique character, each is easily matched.

- [Figure 2](#)—Total Ion Current (TIC) Chromatograms of Ronsonol Lighter Fluid
- [Figure 3](#)—Mass Spectrums of Ronsonol Lighter Fluid
- [Figure 4](#)—Total Ion Current (TIC) Chromatogram of Gasoline Standard and Headspace Sample of Charred Wood Treated with Gasoline

### **Constructing a Solution**

The analysis of the charred samples should be planned to reveal whether or not the mock fire was the result of arson. To lend a greater sense of realism to the experiment, and to aid students in their elucidation of the truth, we have included a crime scene schematic in the case showing the precise locations from which the charred samples were collected; this schematic can be modified by other instructors to serve their own purposes regarding the guilt or innocence of Marie Stanforth.

The instructor may wish to remind students that the detection of an accelerant from any charred sample does not necessarily constitute arson, due to the fact that there could be a reasonable explanation for the presence of many accelerants in the common household. For upper division classes, the instructor may wish to allow students to make this conclusion on their own.

For example, in the crime scene schematic provided, the detection of isopropyl alcohol in sample 2 would not lend much weight to an arson case because it is possible that isopropyl alcohol would have been stored in the bathroom of the victim's house. Likewise, the detection of lighter fluid in sample 1 would not clearly prove arson, because it is not uncommon for lighter fluid to be stored in garages. In order to make a strong case for arson, students will need to show that an unnatural accelerant can be detected near the point of

origin. For our purposes, we chose sample 1 to correspond to lighter fluid, sample 2 to correspond to isopropyl alcohol, and sample 3 to correspond to gasoline.

Armed with the information that the detection of accelerants at or near the “point of origin” is a strong case for arson, students should pay particular attention to unknown #3, or whichever sample the instructor chooses to correspond to the point of origin. In the provided crime scene schematic, the detection of gasoline in unknown #3 makes a strong case for arson because there are no real reasonable explanations for the presence of gasoline in the victim’s living room.

In order to make a conclusion about the guilt or innocence of Dr. Marie Stanforth, students should be provided with a fourth unknown which should be introduced as a swatch of clothing recovered from a glove found in the trunk of Dr. Stanforth’s automobile on the day of the crime. Again, the lab is sufficiently flexible that the instructor has the option of spiking the clothing any number of different ways. To give the impression that Dr. Stanforth is guilty, you might choose to spike the clothing sample with the same accelerant you chose for the point of origin. Or, you might choose not to spike the clothing with anything at all. An interesting cross disciplinary discussion might arise if you chose gasoline as your “point of origin” accelerant and as your clothing accelerant. Though this correlation might clearly seem to indicate Dr. Stanforth’s guilt, some students may recognize that gasoline is sometimes stored by motorists in the trunks of their automobiles. Some students may argue, then, that the presence of gasoline on the swatch of clothing recovered from Dr. Stanforth may not adequately establish her complicity in any arson charges, and consequently, may not be enough to charge Dr. Stanforth with arson.

The overall flexibility of the experiment makes it appropriate for use in many different settings. For a higher level chemistry analysis class, the instructor may choose not to provide the students with standards but instead require them to respond based on their knowledge of mass spectrometry and their familiarity with chemical literature to uncover the composition of the accelerants. With the standards in place and the analysis qualitative, the experiment can also be tailored for an introductory course in forensic analysis or a forensic analysis class for non-science majors.

## ANSWER KEY

Answers to the questions posed in the case study are provided in a separate answer key to the case. Those answers are password-protected. To access the answers for this case, go to [the key](#). You will be prompted for a username and password. If you have not yet registered with us, you can see whether you are eligible for an account by reviewing our [password policy and then apply online](#) or write to [answerkey@sciencecases.org](mailto:answerkey@sciencecases.org).

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Sodeman, D. A., and S.J. Lillard. 2001. *Journal of Chemical Education*. 78: 1228–1230.

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## Figure 1—Detailed Methods

### Lab Preparation

#### *Preparation of Charred Samples*

1. Pine wood should be cut into small pieces approximately 3 cm long and 1 cm wide. A piece of craft board which is ¼ " thick by 3 " wide and 3 " long easily yields enough wood pieces for several large lab sections.
2. About ten pieces of the finely divided pine wood should then be placed into four different 125 mL Erlenmeyer flasks.
3. Using a disposable Pasteur Pipette, deliver 10 mL of accelerant to its corresponding flask. Enough accelerant should be added so that the wood chips are able to soak up the accelerant, but accelerants (particularly gasoline) should not be added in great excess. After adding accelerant, stopper each flask with rubber septa.
4. Leave the wood chips to soak for 2–6 hours.
5. After the wood chips have soaked for several hours, pour the excess accelerants out of each Erlenmeyer flask, and discard the excess accelerant in the appropriate waste container.
6. Either in a hood, or outdoors, place the wood chips on a large watch glass and ignite the chips with a match. Take care not to drop the match in the wood chips and allow it to burn with the chips.
7. After the chips are mostly charred, extinguish the fire by blowing out the flame, and allow the chips to stop smoking. Immediately after the wood chips have stopped smoking, take small pieces of the chips and place them in a small labeled container.

#### *Preparation of Clothing Swatch*

1. Soak the clothing in the accelerant of your choice and allow it to dry. Place inside a labeled vial as unknown #4.

#### *Preparation of Standards*

1. Using a disposable Pasteur pipette, add 10-20 mL of each standard accelerant into labeled vials and secure lid.

#### Materials

1. Samples of gasoline, isopropyl alcohol, Ronsonol lighter fluid, and Lamplight lamp oil were purchased from local vendors and used as received. Pine wood was obtained from a local vendor and cut into small pieces according to specifications listed above.
2. A 100 micro Liter airtight syringe, with a Teflon tip plunger, from Agilent Technologies (no. 5183-2058).

### Running the Experiment

An HP mass detector 5973 and HP GC system 6890 was used in this experiment. The autosampler was removed to allow for manual injections.

## GC-MS Settings

Column:	HP-1MS (30m x 0.25mm, 0.25µm film thickness)
Temperature Program:	Initial 50°C for 1 minute, ramp 15°C per minute to 270°C, 270°C for 1 minute.
Injector Temperature:	260°C
Flow Rate:	He, 1 mL/min
Split Ratio:	20:1

## Running Samples

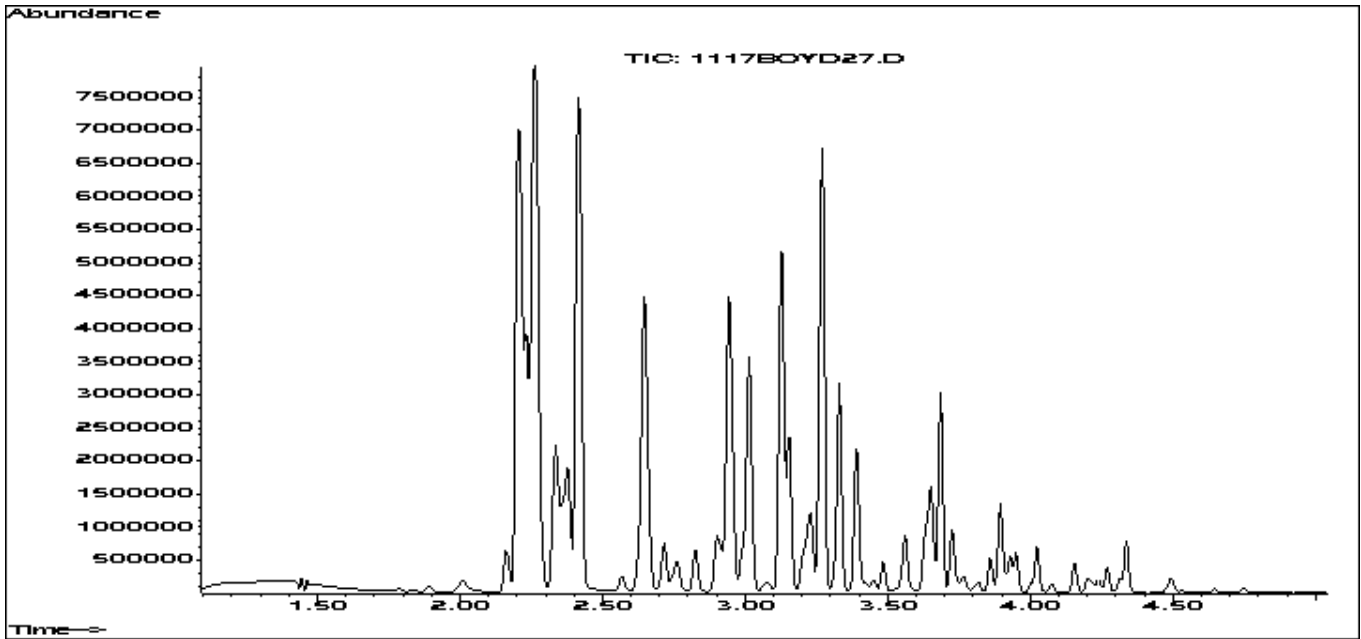
1. Amber vials containing standard accelerants should be placed in a 100°C hot water bath and allowed to warm for five minutes.
2. After the vial has been warmed, students should use the static headspace analysis technique to collect 100 µL of gas just above the liquid surface of the accelerant using an air tight 100 µL syringe.
3. The vapor should then be injected into GC-MS. In the case of manual injection, the student should take care to hold the plunger of the syringe down for one minute after the sample has been injected. This is necessary because the high vapor pressure of some accelerants will cause the gaseous analyte to escape if not forced onto the GC column.
4. Allow the GC-MS to complete run.
5. Between samples, students should continually aspirate the syringe to ensure that previous accelerant vapor does not contaminate successive standard runs.
6. After the chromatograms for the standard accelerants have been collected, students should collect chromatograms for the four unknowns, repeating steps 2–6 after warming each unknown vial in a 100°C warm water bath for five minutes.
7. Software integrated with the GC-MS allows for total ion current chromatograms to be analyzed almost immediately after the mass spectra are stored in the computer. Once a peak is obtained it is possible to pull up the mass spectra at any retention time on the chromatogram. A library search can then be performed against that mass spectrum to give insight as to the chemical composition of the injected sample.

## Hazards

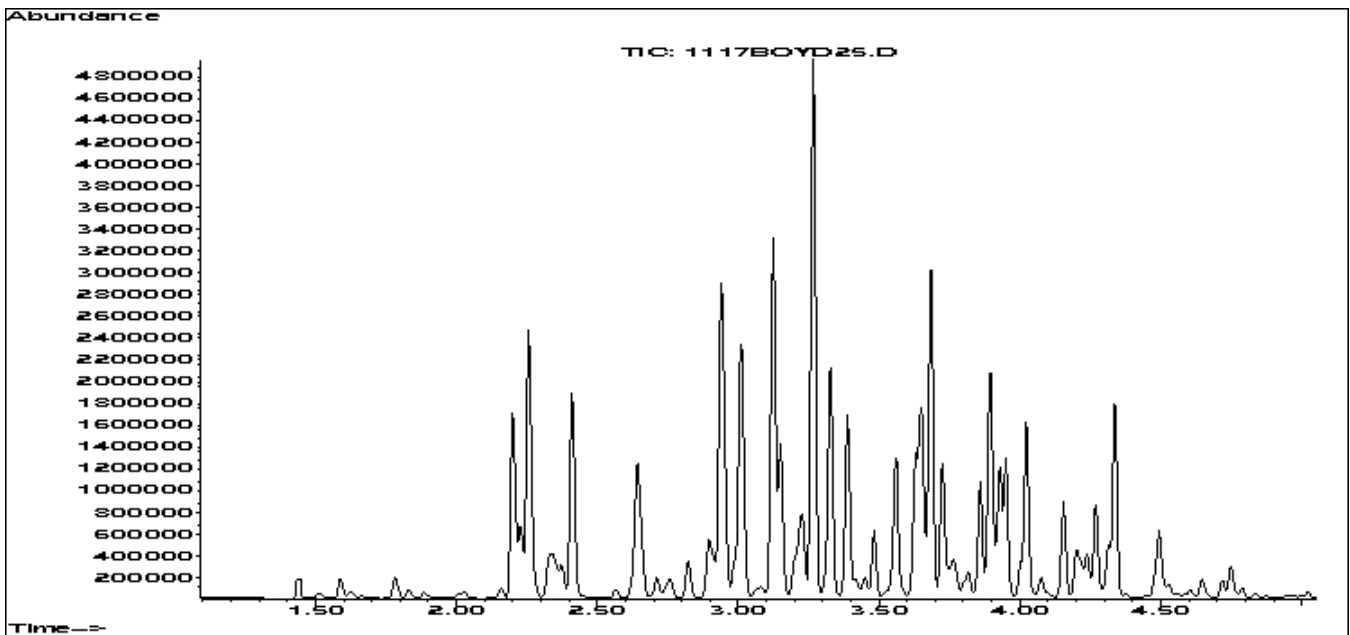
1. Caution must be used when handling flammable accelerants. It is especially important to remove all excess accelerant from wood chips before ignition.
2. Proper eyewear, gloves, and a lab coat should be worn while in lab.

**Figure 2—Total Ion Current (TIC) Chromatograms of Ronsonol Lighter Fluid**

**Standard Sample**

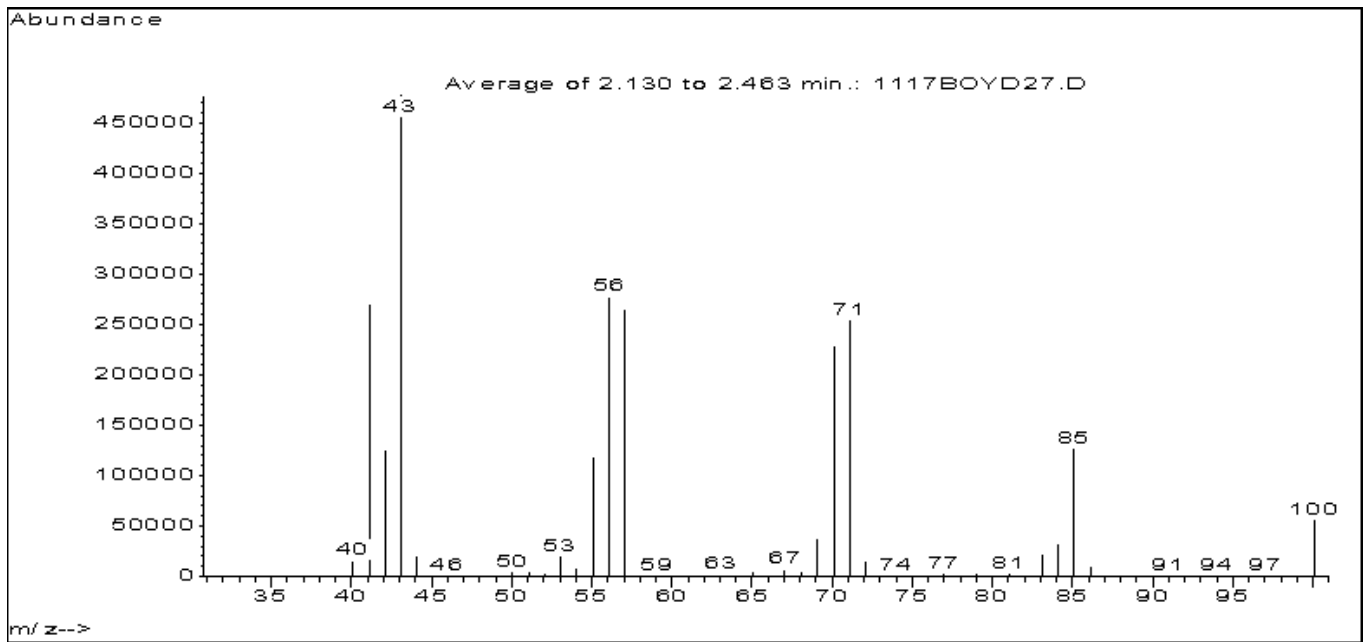


**Charred Sample**

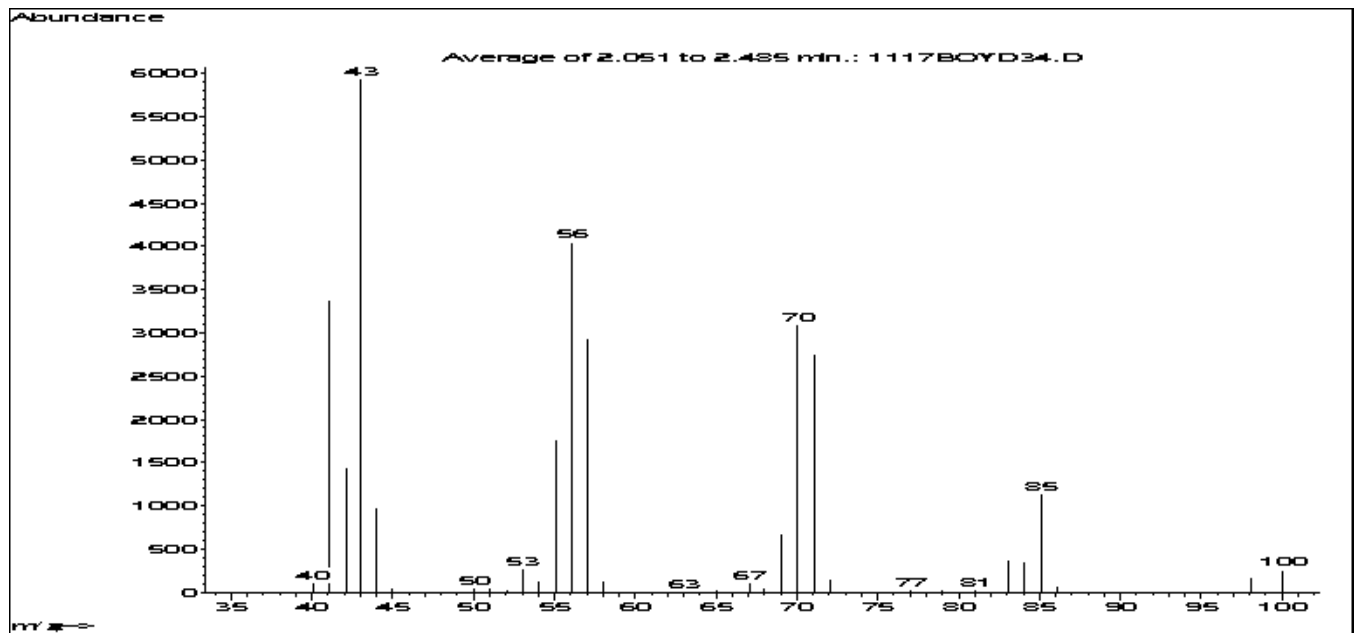


**Figure 3—Mass Spectrums of Ronsonol Lighter Fluid**

**Standard Sample**

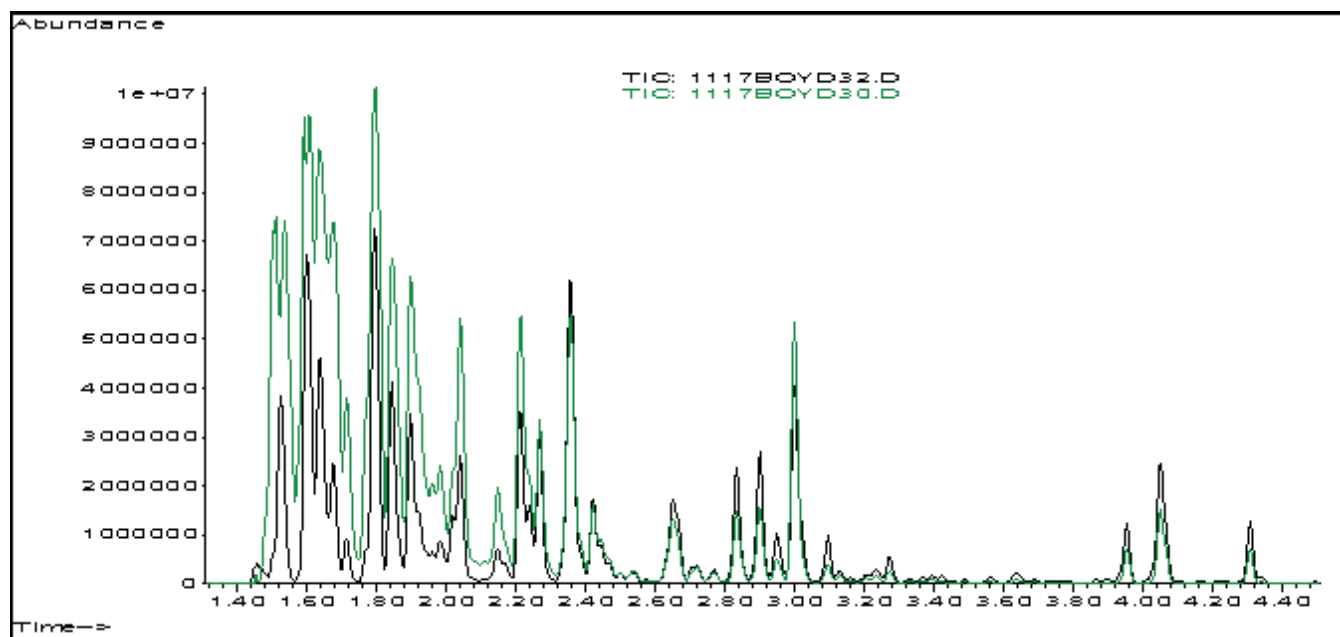


**Charred Sample**



## Figure 4

Total Ion Current (TIC) Chromatogram of Gasoline Standard (Green Line) and Headspace Sample of Charred Wood Treated with Gasoline (Black Line)



## SUPPLEMENTAL MATERIAL

### *Introduction to GC-MS*

In gas chromatography (GC), a sample is transported through a chromatography column by a gas mobile phase, called the carrier gas. When a volatile or semi-volatile liquid sample is injected into the heated injection port of the GC, the sample volatilizes into a gas and is swept out of the injection port and through the column via the carrier gas. Because the components of a mixture move through the column at different rates based on their affinity to the column material, they are said to have different retention times. These retention times can be used to help identify components of the sample.

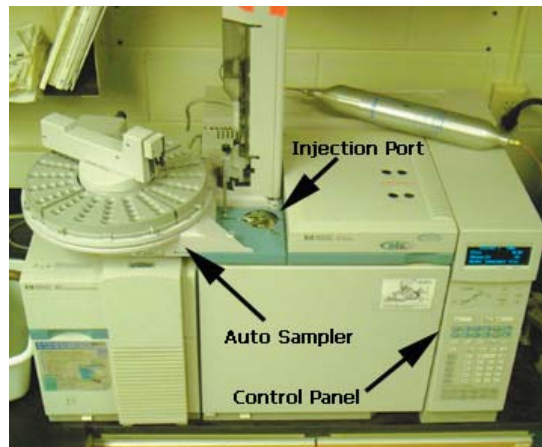
Mass spectrometry (MS) is an analytical technique in which atoms or molecules from a sample are ionized, separated according to their mass to charge ratio, and then recorded. In mass spectrometry, molecules are bombarded with high energy electrons. When an electron with a sufficient amount of energy collides with a sample molecule, it dislodges an electron from the sample molecule and creates a molecular ion called a cation ion radical. The cation radical is positively charged and has an odd number of electrons but has basically the same mass as the uncharged parent compound because the mass of an electron is negligible. The positively charged ions are then directed into an analyzer tube surrounded by a magnet. Given that different fragments will be formed, and they will have different sizes, particular types of cationic species will have characteristic mass-to-charge ratios ( $m/z$ ). A mass spectrometer scans all the  $m/z$  values and gives a distribution of positive ions, called a mass spectrum, which is characteristic of a certain compound. The most intense peak in the mass spectrum is called the base peak and is assigned a relative intensity of 100.

When these two methods are coupled together into the same machine, we perform gas chromatography / mass spectrometry or GC-MS analysis. Gas chromatography is used to separate a mixture and mass spectrometry is used to analyze it. If we can selectively monitor specific ions we can easily measure one component, for example an accelerant, in a complex chromatogram of poorly separated compounds.

### *Introduction to Headspace Analysis*

To recover and identify accelerants from charred debris, forensic chemists typically use either headspace analysis or steam distillation. In headspace analysis, a portion of the matrix (which contains the analyte) is placed in a glass tube, which is covered with a rubber septum. When the tube is heated, volatile residue present in the debris will vaporize and will be enclosed in the air space above the sample in the tube. The vapor can be removed with a syringe and subsequently analyzed by gas chromatography. There are three different types of headspace analysis (Grob, 1995):

- Static Headspace
  - Sample is heated to increase vapor pressure of analyte and gaseous sample collected with syringe and analyzed via gas chromatography
  - (+) Detection limit: 5–10 microliters of petroleum accelerant in gallon can; ease of application



A typical GC-MS. An HP 5970 was used in this experiment. Although this GC-MS is shown with an auto sampler, injections were performed manually, at the injection port shown.

- Passive Headspace
  - Accelerant vapors are diffused from sample onto an absorbent placed inside the container of fire debris.
  - Accelerants are desorbed with carbon disulfide
  - SPME (Solid-Phase Microextraction) An alternative passive headspace sampling technique for gasoline in fire debris. SPME is a simple, solventless extraction procedure in which a phase coated fused silica fiber is exposed to the headspace above the fire debris packaged in a closed container. SPME has been able to detect .04 microliters of gasoline in a quart can.
  - (+) multiple samples can be taken from one sample matrix, Teflon strips can be frozen for later analysis
  - (-) carbon disulfide can interfere with FID (Flame Ionization Detector)
- Dynamic Headspace
  - Air or inert gas is passed over the sample (purge and trap technique if sample is liquid) and adsorbed onto a substance such as activated carbon. After the accelerant has been adsorbed onto the carbon, it is often extracted with carbon disulfide. Thermal desorption is not practical because of the high temperature needed to remove hydrocarbons from charcoal (950°C). In one version of this technique, chemists purged a can containing fire debris with heated nitrogen. The nitrogen then passed out a hole in the lid of the container and through a Pasteur pipette packed with activated charcoal. The accelerant was then desorbed from the charcoal with carbon disulfide and subjected to GC analysis.
  - (+) more sensitive than static headspace, and not as cumbersome as distillation methods
  - (-) does not give good recoveries of high petroleum distillates, such as diesel fuel

### ***Other Procedural Considerations***

In the given scheme, the students are tasked with developing a methodology, determining if the fire was arson, and determining the innocence or guilt of Dr. Marie Stanforth. Depending on how the instructor wishes to arrange the experiment, Dr. Stanforth may be guilty or she may be innocent. Yet, both in planning the experiment, and allowing students to conduct their investigation, the instructor should keep in mind that some accelerants may have a logical explanation for their presence other than arson. The detection of isopropyl alcohol in the bathroom, for example, would not prove arson because it is not uncommon for people to store this product in the bathroom.

The instructor may want to remind students of this fact, or may want to hint at it, so that the detection of one accelerant does not force the students into automatically assuming the fire was arson or that the suspect in question is guilty. For more advanced classes, the instructor may want students to make this conclusion on their own.

Consider the environment from which the sample was taken—if an analyte is detected, is there any other logical explanation for its presence other than arson?

Inspect clothing for traces of accelerant.

Students should use headspace analysis on the clothing sample in the same fashion as the charred samples provided to them. Their conclusions about the guilt or innocence of Dr. Marie Stanforth should evolve from the correlation of any accelerants found on the clothing swatch recovered from Marie and the accelerant (if any) found near the point of origin (which would indicate the fire to be the result of arson).

### ***Sources of Additional Information***

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