

Arson Investigation: A Gas Chromatography Laboratory Experience for General Chemistry

Angela C. Orf, Meghan Morris, and James Chapman*

Rockhurst University, Kansas City, MO 64110, James.Chapman@rockhurst.edu

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Abstract: We have developed an arson investigation experiment for the introduction of gas chromatography analysis and forensic sample preparation into the general chemistry laboratory. Working with information provided to us from the Kansas City Crime Lab, we modified their procedures to yield a less expensive method and more suitable time frame to complement our laboratory schedule. The experiment is carried out over a three-week period with a total time commitment of 2 to 3 hours. The laboratory is designed to be an add-on to our schedule without taking the place of any existing experiments. The students are introduced to the concepts of gas chromatography (single and multi-vial sampling procedures), sample preparation, data acquisition, and data analysis.

Introduction

In hopes of creating an educational and entertaining laboratory experience for a general chemistry laboratory, we decided to develop a mock arson investigation. This laboratory was designed to fit into an already existing syllabus and to be done in conjunction with other laboratories. Recently published experiments in the *Journal of Chemical Education* and *The Chemical Educator* use a mock arson investigation to teach gas chromatography techniques. We reviewed these sources and contacted the Kansas City Crime Laboratory before designing our own procedure. Two of the experiments previously published conducted headspace sampling with the aid of a gas-tight syringe [1, 3] while the third utilized passive headspace sampling with activated charcoal [4]. The fourth analyzed a neat liquid using GC/MS [2]. The two similar methods employed long accelerant soak times (4 to 6 hours) prior to ignition and this procedure does not reflect how an actual arsonist would set a fire. The third uses accelerant samples passively adsorbed to charcoal for investigation that are neat liquids and not obtained from charred fire debris. All of the methods analyze the accelerants by gas chromatography with either a flame ionization detector (FID) or mass spectrometry detector (MSD). The Kansas City Crime Lab uses activated charcoal strips (Albrayco) suspended in the headspace of a sealed and heated can to sample the fire debris [6]. The crime lab heats the fire debris samples in an oven for 2 to 24 hours. Because our laboratory period meets for four hours once a week, it would be difficult for the students to set up the passive sampling and remove their samples later in the same laboratory session. Also, based on the number of students that would be conducting this experiment, it seemed to be a hazard to have that many sealed containers of accelerants in an oven for a week. Based on this concern, the sealed sample containers were allowed to sit at room temperature until the next laboratory session (one week) before removing the charcoal. The accelerants are desorbed from the charcoal with a solvent and this solution is injected into a GC and analyzed by FID [5] or MSD [7]. The price of the strips was determined to be too expensive for the number of students that would be

conducting the experiment. The strips cost \$3 each if purchased in bulk and we would need as many as 80 for our general chemistry laboratories. Because of this expense, we decided to investigate other forms of charcoal. Nuchar (Meadwestvaco, Covington, VA) and aquarium charcoal were available for testing. The Nuchar and aquarium charcoal were both capable of absorbing the accelerant vapors although the absorptive properties of the aquarium charcoal were reduced in comparison.

Experimental

Equipment. A Varian 3400 GC equipped with an 8200 autosampler and a PC-Chrom A/D (H&A Scientific) acquisition system was utilized to separate and record the sample profiles. The column was a RTX-200 (trifluoropropylmethyl polysiloxane, 30m × 0.32 mm ID, 0.50 μm DF) from Restek. The initial column temperature was set at 50 °C and held for 2 minutes. The temperature was increased at a rate of 30 °C per minute to a final temperature of 220 °C with a hold time of 2 minutes. The injector temperature was set at 250 °C and the detector at 300 °C.

Reagents. Six common accelerants were selected for the experiment. Gasoline, charcoal lighter fluid, Goof Off (trademark industrial cleaner), isopropyl alcohol [67-63-0], perfume, and paint thinner. All of the accelerants were commercial grade except for the isopropyl alcohol. These six samples were also mixed to create more possibilities for the students with no more than two accelerants applied to the fire debris samples. Mixtures of accelerants can be made to increase variability, but care should be taken to eliminate confusion. Certain accelerants contain similar components and it can be difficult to detect differences. For example, the perfume or cologne samples contain isopropyl alcohol, so it is helpful to the student not to make a mixture of perfume or cologne and isopropyl alcohol. Reagent grade carbon disulfide [75-15-0] was utilized for the desorption process. Diethyl ether, pentane, or another suitable solvent that will not interfere with the GC analysis can also be used.

Procedure I. The wood samples were burned one week prior to the laboratory session. Wood pieces were cut into approximately 0.2 g pieces. The appropriate number of pieces needed for each accelerant was placed on a heat resistant watch glass. Two milliliters of the accelerant were poured over the wood samples and allowed to soak for 5 minutes. When mixtures were prepared 1 mL of each accelerant was poured onto the wood and allowed to stand. At the end of this

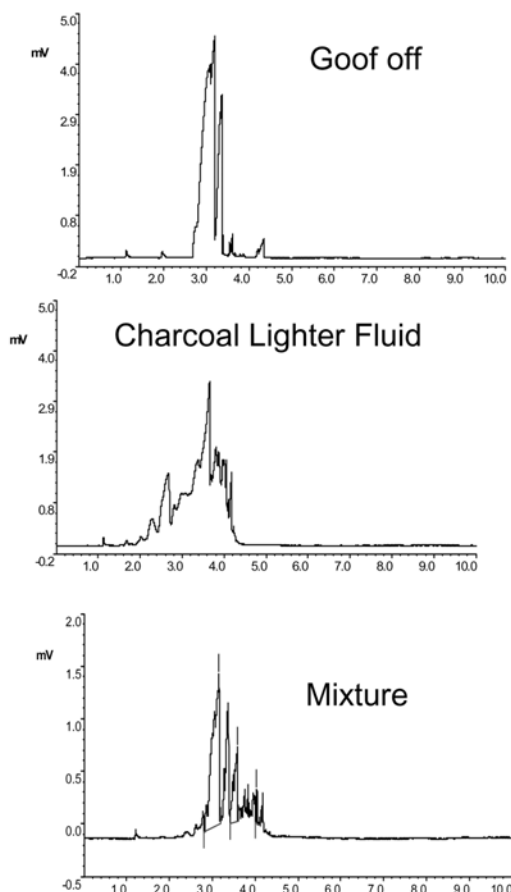


Figure 1. GC chromatograms of two accelerants and the resultant mixture.



Figure 2. Crucible positioned over fire debris sample in wire triangle. Side view, top view and placement in paint can.

time the accelerant was ignited and extinguished after 1 minute, if flames were still visible. The pieces were stored in a capped and sealed scintillation vial to minimize the loss of fumes. The fire debris samples were stored at room temperature until needed for the

designated laboratory period. Immediately prior to the laboratory period the wood samples were distributed among numbered test tubes and a rubber stopper inserted.

A paint-can holding chamber was assembled from available materials based on modifications to ASTM 1412-00 [6]. A quart size paint can was used for the holding chamber. The three “legs” of a wire triangle were bent it to form a stand similar to that found in a pizza box. A Gooch crucible was placed inside the opening of the stand. The Nuchar charcoal (0.1 g) was placed in the crucible for the absorption process. The accelerant or wood chip was placed on a small watch glass and set in the bottom of the paint can (Figure 2). The can was sealed and labeled with its contents.

Procedure II. Week 1: Prior to the laboratory period, the students were given a prelaboratory assignment to complete. They were also given access to the gas chromatograms of the six accelerants used. In the laboratory, students were paired in groups and given a burned wood sample. They constructed the paint can holding chamber described above and placed the wood pieces inside to sit for a week.

Week 2: The groups unsealed their paint cans and removed the charcoal. The charcoal is easily introduced into a GC vial by first pouring it into a scoopula. The instructor added 1mL of carbon disulfide to the GC vial. The autosampler needle sampling depth was chosen so that it would not disturb the charcoal and the autosample was set to withdraw 2 μ L for each injection. The students were allowed to observe one or two runs as they were introduced to the components of the GC instrument. The GC and autosampler were set up to run the samples and the results were given to the students in their regular class times the following day.

Week 3: The students used their printed GC results in conjunction with the standard chromatograms to determine which accelerant was used to start the fire. They typed a written laboratory report discussing their results.

Hazards. The carbon disulfide used should be used only in a ventilated hood. In our laboratory it was only handled by the instructor because of the risk when dealing with a large number of students. Precaution should be taken as well when burning the wood samples. The accelerants are highly flammable and should be handled carefully.

Results

Some of the mixtures were misinterpreted because of their similar overall profiles. Because of this problem, retention time should be emphasized to the students. Looking closely at this factor can help them better determine the difference between two mixtures.

Also, after the completion of this laboratory, we decided to experiment with aquarium charcoal for an even more cost-efficient method. It was found that the aquarium charcoal worked just as well. The only noticeable difference between the Nuchar and aquarium charcoal was the intensity of the samples, presumably because the aquarium charcoal did not absorb as well as the Nuchar.

Conclusion

This laboratory experience was well received overall. The students seemed to enjoy the procedure and had a high success rate. The students enjoyed the laboratory during each week’s activities. When these students encountered the GC again in their organic chemistry or other chemistry classes, they were able to recall some of the techniques they utilized in this experience, which was one of the objectives we hoped to accomplish.

Supporting Materials. Sources for materials and a student handout are included in the supporting materials (<http://dx.doi.org/10.333/s0089712182a>)

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References and Notes

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