TECHNICAL NOTE

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Automated Sampling and Computer-Assisted Identification of Hydrocarbon Accelerants

ABSTRACT: A method is described for rapidly screening samples in arson cases and making a tentative identification of the accelerant present.

KEYWORDS: criminalistics, arson, accelerants, computer-assisted identification

As the arson case load has increased in laboratories, there have been numerous efforts to reduce analysis time per sample. Most of this work has concentrated on reducing time on the gas chromatograph. [1,2].[2] This paper discusses the two other analytical steps in arson analysis—sample preparation and chromatographic interpretation—and minimizing examiner time per sample during these steps.

Sample Preparation

A comparison of sample preparation techniques in use today has shown that an adsorption/elution technique has several advantages, including minimum examiner time and greatest sensitivity of methods compared [3]. With multiple sampling setups (Fig. 1), numerous samples can be collected in a short period of time. The only time the examiner spends on the sample preparation portion of the analysis is to prepare an adsorption tube, place the sample container in the heating mantle, and, after sufficient sampling time (1 h maximum), rinse the adsorption tube (charcoal tube) with carbon disulfide.
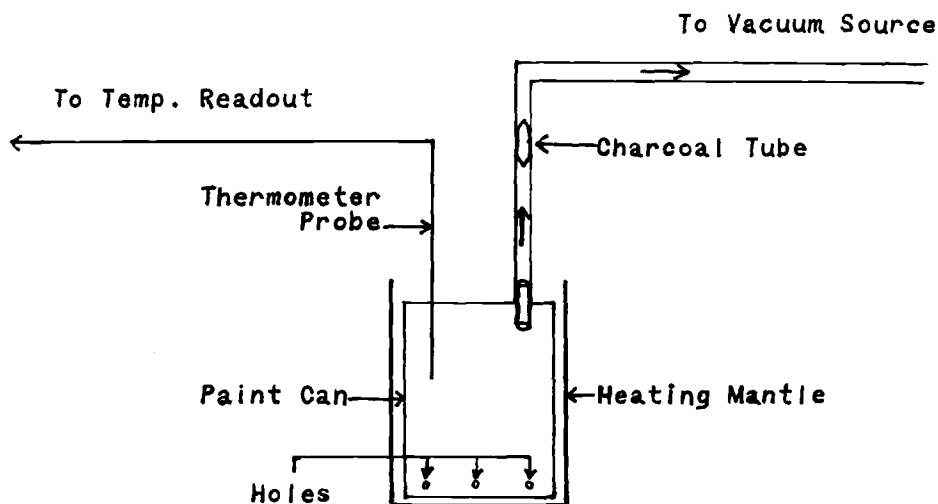
Charcoal tubes are commercially available[3] but are unnecessarily sophisticated for arson laboratory use. With the materials and procedures described below, satisfactory tubes can be easily and quickly prepared at a cost of a few cents apiece for materials.

To Vacuum Source

To Temp. Readout

Charcoal Tube

Thermometer
Probe

Paint Can

Heating Mantle

Holes

PARAMETERS :   1. DRAW VACUUM (0.5 ATM.) THROUGHOUT SAMPLING RUN.

2. STOP RUN WHEN SAMPLE TEMP. REACHES 100C.

FIG. 1—*Sorption/elution sampling setup. The paint can's friction lid is replaced by a lid fitted with a septum for the thermometer probe and a fitting, soldered in place, for the sampling tube. Holes are punched in the bottom of the evidence container to allow heated air to flush the debris.*

Materials

The materials needed are pipets, charcoal, and cotton. The pipets should be disposable glass Pasteur pipets, 146 mm (5 ¾ in.) long (for example, Kimble 72000). The charcoal should be activated coconut charcoal, 50–200 mesh (for example, Fisher 5-690). As a precautionary measure to assure that the charcoal used for tube loading is clean and active, the as-received material may be heated overnight at $250 \pm 25°C$ and then cooled and stored in a desiccator. The cotton should be absorbent, first aid–type cotton.

Procedure

1. Insert a piece of cotton into a Pasteur pipet until it begins to enter the tip, then tap lightly. A suitable quantity of cotton forms a plug approximately 6 to 9.5 mm (¼ to ⅜ in.) long.
2. With a short length of flexible tubing, connect the stem of a small glass funnel to the mouth of the pipet. Load the pipet with charcoal, introduced through the funnel, to a depth of 63.5 mm (2.5 in.).
3. After removing the funnel and connector from the pipet, introduce a second piece of cotton and tap it lightly into place against the top of the charcoal column.
4. The tube is now ready for use. If it is not to be used immediately, or if a stock of tubes is being assembled, storage in a clean vapor-tight container is strongly advised.

The heating mantles are available from Glas-Col Apparatus Co. of Terre Haute, IN, in a variety of sizes.[4]

[4]Heating mantles to fit 1-qt and 1-pt paint cans are available as stock items. A mantel with inner diameter of 171.5 mm (6 ¾ in.) may be custom-made for 1-gal cans.

The adsorption/elution technique is currently the most sensitive sample preparation technique; examiner time and attention is kept to a minimum. The technique also produces a low-volume liquid sample (less than 0.5 mL), which facilitates efficient use of an automatic sampler.

Chromatogram Interpretation

The automatic sampler, coupled with computer-assisted chromatogram interpretation, can further reduce operator attention and improve the accuracy of results. The equipment used throughout this work was a Perkin-Elmer Sigma I Analyzer and a Sigma 10 Data Station with printer/plotter and 4000-word memory capacity.

The computer capabilities of this equipment allow us to define attenuation windows by changing attenuation several times in an analytical run. An attenuation window is a time segment over which a chromatogram has a given attenuation.

A standard accelerant mixture (SAM) (see Fig. 2) of equal parts gasoline, kerosine, and diesel fuel was used to simulate the range of acclerants normally encountered in arson cases. Figure 3 shows the attenuation windows applied to SAM. Windows 1,4,6, and 8 are the windows of interest and should be set at your instrument's most sensitive attenuation setting. The attenuation in Windows 2,3,5,7,9, and 10 is set at increasing increments on either side of your windows of interest. Their functions are best illustrated by example.

If your sample contains "weathered" gasoline, the less volatile components such as 1,2,4-trimethylbenzene will be found in Window 4. The components of midrange petroleum products, such as paint thinners and charcoal starters, also will be found there. The major
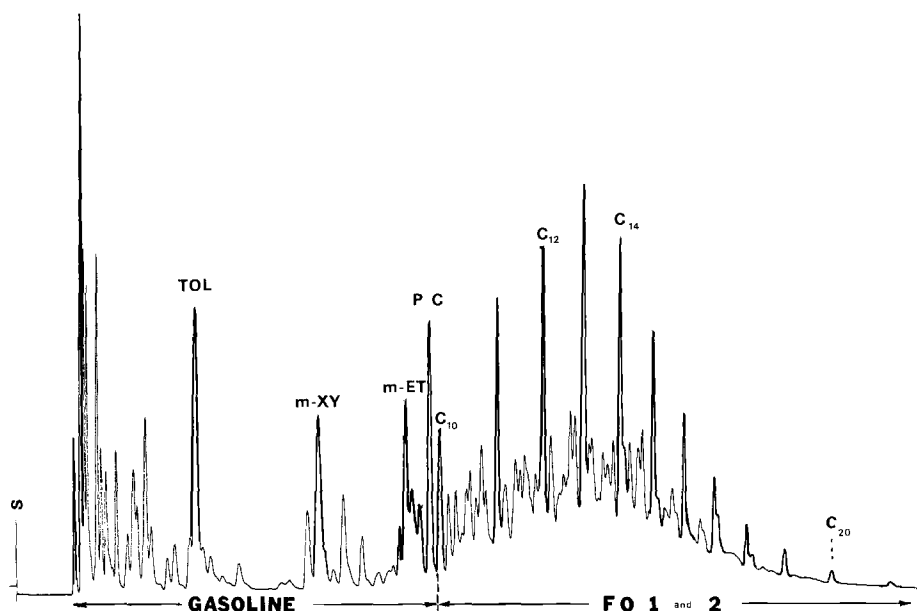

FIG. 2—*Chromatogram of the SAM. TOL = toluene, m-XY = meta-xylene, m-ET = metaethyltoluene, PC = pseudocumene (1,2,4-trimethylbenzene), and FO 1 and 2 = fuel oils No. 1 and 2. Chromatographic conditions: 3-mm by 6.1-m ($^1\!/_8$ in. by 20 ft) stainless steel column, 3% SP-2100 on 80-100 Supelcoport; flow rate of 25 cm^3/min nitrogen; injector temperator 250°C; 300°C flame ionization detector. Program: 6 min initial hold at 50°C, 12°/min ramp to 280°C; 6 min final hold.*
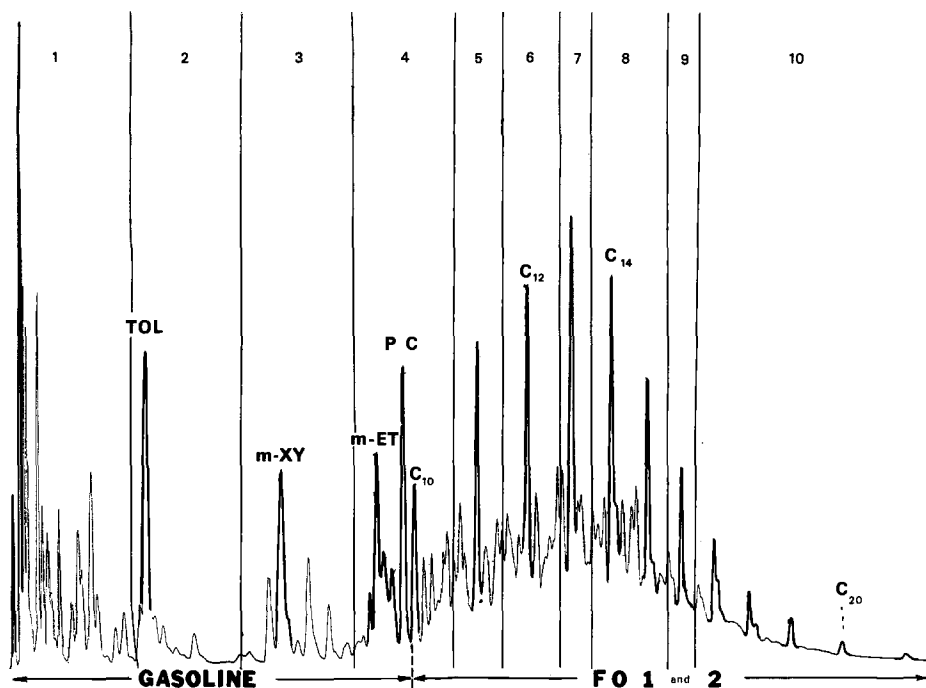
FIG. 3—*Chromatogram of the SAM with the attenuation windows applied. Abbreviations, chromatographic conditions, and program are the same as in Fig. 2.*

component, C_{12}, of No. 1 fuel oil will be found in Window 6; the major components of No. 2 fuel oil, C_{14} and C_{15}, will be found in Window 8. The high sensitivity in Window 1 will allow you to detect more volatile accelerants such as pocket lighter fluid and rubber solvents. In short, the windows of interest have been chosen so that any class of petroleum accelerant will be detected. If no peaks exist within the windows of interest at maximum sensitivity setting, then it can be concluded that no accelerant could be detected.

Another useful application of this procedure comes into play when the questioned sample is a mixture of hydrocarbon products, for example gasoline and fuel oil. When the fuel oil amounts to 10% or less of the mixture, a chromatogram run at the proper attenuation to keep gasoline on the chart may well miss the heavier fuel oil. This screening program eliminates that problem.

Normally the examiner makes an educated guess at the appropriate sample size and attenuation and must constantly monitor the instrument to keep the chromatogram on scale since identification is usually made by pattern recognition. By progressively decreasing the sensitivity in Windows 2,3,5,7,9, and 10, the examiner can now determine which attenuation will give the best pattern over the whole range of the questioned sample.

This computer assistance allows an automatic-sampler to run unattended and still assure chromatograms with interpretable results. If peaks are off-scale on the printer/plotter the computer still "sees" all the peaks and will tentatively identify those off-scale peaks by retention time. When the examiner has to have a chromatogram entirely on-scale for identification, it is easy to determine the proper attenuation by looking at the attenuations used in the windows. A significant advantage to this approach to chromatogram interpretation is that it can be applied to any chromatographic system that allows automatic attenuation changes.

Conclusion

This work has discussed two analytical steps used in arson analysis and how to reduce examiner time per sample in each phase of the analysis. To date, the computer-assisted interpretation has been used on a limited number of cases, but based on the expected analysis of 1500 samples per year this procedure will save approximately one analyst-year in the laboratory.

Other procedures can be developed to reduce actual time on the instrument by modifying the analytical conditions. By using a short capillary column with high flow and ramp rate it is possible to produce a chromatogram in less than 6 min with resolution comparable to our present chromatogram, which now takes 32 min to run. If your gas chromatograph has BASIC computer language capabilities, it is possible to program the instrument to automatically rerun any chromatogram that goes off-scale. Key peaks are selected and a maximum count predetermined. If this area count is exceeded in your run, the BASIC program will rerun your sample. Future work will address both of these time-saving ideas.

References

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