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Improved Charcoal Packaging for Accelerant Recovery by Passive Diffusion

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ABSTRACT: Two simple procedures for trapping volatile accelerant vapors are presented as alternatives to other techniques which utilize passive diffusion. These procedures take advantage of the simple diffusion of volatiles into the headspace of a closed container and trap the vapors onto charcoal which is either encased in a porous pouch or impregnated into a flexible membrane. Data demonstrating the abilities of these procedures to collect accelerant vapors with respect to changes in adsorption time, temperature, and sample concentration are presented.

KEYWORDS: forensic science, accelerants, charcoal, passive diffusion

The most widely known and simplest technique presently being used for the recovery of accelerant vapors from fire debris is headspace analysis. This technique employs sampling a small portion of vapor (heated or ambient) from within a closed container of debris and injecting that sampled vapor into a gas chromatograph for analysis [1]. Because it is a relatively clean and rapid recovery technique, it is quite often used as a simple screening procedure before using other recovery techniques such as purge-and-trap, steam distillation, or solvent extraction. Since the technique relies on the relatively high vapor pressure of hydrocarbons to produce their separation from the debris, limitations are imposed by the low vapor pressures of high-molecular-weight hydrocarbon compounds in accelerants. In addition, the accelerant concentration within a given aliquot of debris vapor can be very low and even vary significantly between samples, causing complications in the interpretation of resulting chromatograms.

Several other techniques have been developed and modified over the years in an attempt to overcome the problems of dilute accelerant concentrations. These methods include charcoal-coated ferromagnetic wires and Curie point apparatus [2,3]; charcoal-coated copper wires and Plexiglas beads [4]; and purge-and-trap charcoal tubes [5]. While these techniques have achieved sensitivities one hundred times greater than headspace analysis [6], they also possess some inherent disadvantages worthy of correction. The coated wires and beads have been inherently fragile, with charcoal occasionally falling from the devices during analysis [7]. Purge-and-trap techniques have effectively retained

Certain commercial equipment, instruments, or materials are identified in this paper to specify the experimental procedure. Such identifications do not imply recommendation or endorsement by the Bureau of Alcohol, Tobacco and Firearms, nor do they imply that the materials or equipment identified are necessarily the best available for the purpose. Received for publication 13 Nov. 1989; revised manuscript received 24 Jan. 1990; accepted for publication 1 Feb. 1990.

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the adsorbing charcoal, but, when the process is not carefully monitored to prevent excessive heating and purging, the increased time of migration through the charcoal tube can enhance breakthrough of the more volatile hydrocarbons [8]. This technique is also prone to contamination by the introduction of outside air or gases during the purging process.

This paper presents two mechanisms by which accelerant vapors may be recovered from fire debris, suspect clothing, and even water samples. The mechanisms basically combine the simplicity of headspace analysis with the effectiveness of purge-and-trap methods by using the process of passive diffusion within a closed evidence container (metal cans, glass jars, and even plastic bags). The one device, which has been called the "C-bag" [7], uses granular activated charcoal encapsulated within an envelope of porous paper. The other device, which has been referred to as the "charcoal strip," was originally taken from a commercial organic vapor detection badge. As a result of this research, the strips are now sold separately without a plastic badge. One major advantage of these devices is that they function by in-situ trapping. This helps avoid the loss of accelerants after they have been collected onto the charcoal and prevents the collection of contaminants from outside the container. Another advantage of these devices is the simplicity with which they can be made and handled for analysis. Because these devices enable the adsorption of vapors with or without heating, they can effectively save time when the adsorption is conducted outside laboratory work time (for example, overnight, while stored in a vault prior to analysis, or during transmission to the lab).

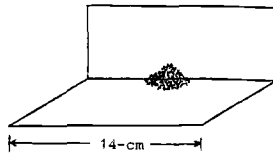
Equipment and Materials

The C-bags were prepared by encapsulating 0.2 g of activated cocoanut charcoal (50 to 200 mesh) within a folded 11 by 14-cm sheet of high-strength, lightweight, and high-porosity tissue paper commonly used for making tea bags. Two grades of paper were supplied by D. H. Dexter Division, Windsor Locks, Connecticut, for testing. No significant differences were found in the performance of the two grades (1234T and 8400) tested. The charcoal was activated prior to assembling the C-bags by heating 4 to 5 g in a platinum dish for 1 h at 400°C. The C-bags were folded into the shape of a common tea bag to hold the charcoal with maximum surface exposure yet enable it to be placed into the mouth of a 250-mL Erlenmeyer flask for extraction. A 15-cm length of string was stapled to the top of the C-bag to allow it to hang freely inside an evidence container. Instructions for the construction of the C-bags are given in Fig. 1.

The charcoal strips were prepared from charcoal membranes contained in organic vapor badges manufactured by Pro-Tek® Systems, Inc., Middletown, New York. The membranes were cut into 32 by 4-mm sections, weighing approximately 0.08 g each. Gas chromatographic analysis of the charcoal strips revealed no contamination components and thus required no additional activation. A 15-cm length of string was tied to a clean paper clip. The outside leg of the paper clip was inserted through the charcoal strip, allowing it to hang freely inside an evidence container.

A standard accelerant mixture (SAM) was prepared by mixing together equal portions of gasoline, kerosene, and diesel fuel No. 2 (Fig. 2). Various quantities of this mixture were placed onto sheets of Kimwipes® inside sealed 1-gal (3.8 L) metal paint cans in order to test the adsorption ability of the C-bags and charcoal strips. Carbon disulfide was used to elute adsorbed SAM from the charcoal. The carbon disulfide extracts were stored in 12 by 32-mm glass vials with crimp tops.

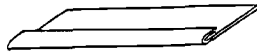
Analyses of the extracts were performed on a Perkin-Elmer Sigma 2000 gas chromatograph (GC) with a flame ionization detector (FID) and a Model 3600 data station. The capillary column used was a 15-m J&W Scientific, DB-1, measuring 0.25 mm in diameter, with a film thickness of 0.25 μm . The carrier gas was helium, with a linear gas velocity



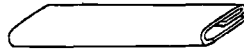
1. Cut paper into a 11-cm by 14-cm rectangle.
2. Fold paper in half lengthwise (14-cm edges together).
3. Place activated charcoal on the center fold.



4. Bring the 14-cm edges together. This is the "open edge".



5. Fold the open edge back 1/3 of the distance toward the center fold.



6. Fold the bottom edge once more to meet the center fold. The paper will measure approximately 2-cm by 14-cm.



7. Fold the 2-cm edges together. The bulk of the paper will be on the interior of this fold. The charcoal will be visible from the outside.



8. Fold the 2-cm edge back 1-cm.
9. Lay the end of string across this fold.
10. Staple string in place on this fold.

FIG. 1—Construction of C-bag.

of 21.4 cm/s and a split ratio of 100:1. The temperature program began at 40°C for 1 min with a ramp rate of 30°C/min and reached a final temperature of 300°C for 3 min.

Experimental Procedures

Although substantial testing has been conducted since 1984 to study the effects of time, temperature, and sample concentration on the adsorption capabilities of these devices, this paper will present only a representative sampling of those tests. However, the tests and results presented here accurately present the various capabilities of these devices.

To illustrate the C-bag's adsorption performance with respect to the factors of time and temperature, three identical samples were prepared by placing 0.2 μ L of SAM onto two Kimwipes placed inside 1-gal (3.8-L) paint cans. A C-bag was suspended halfway

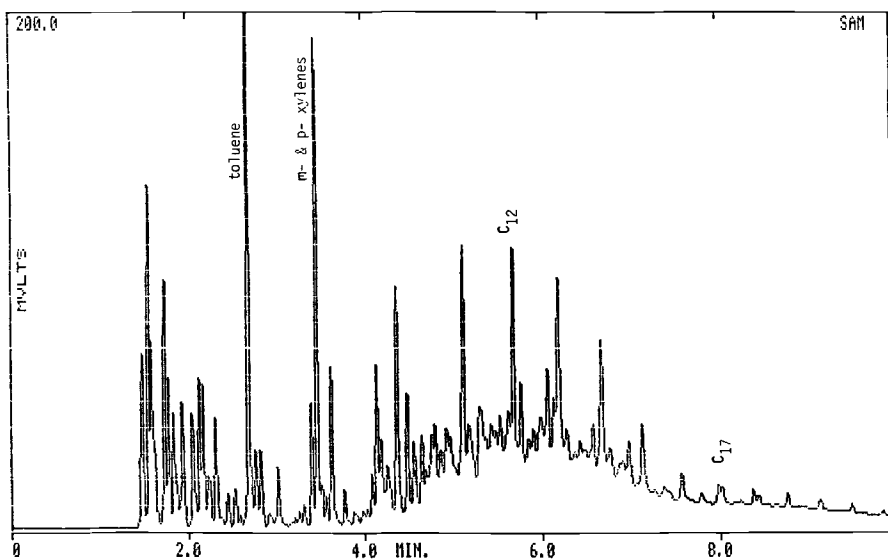


FIG. 2—Chromatogram of 0.04 μL SAM.

down the inside of each can by extending the attached string over the mouth of the can. The lids were firmly placed on the cans to ensure an airtight seal.

The first sample sat at ambient room temperature for 24 h after the C-bag was suspended inside. The C-bag was then transferred to a 250-mL ground-glass stoppered Erlenmeyer flask. The flask was tilted to one side by placing a Pasteur pipet underneath a portion of its base. After 2 mL of carbon disulfide were added to the flask, the stopper was inserted and the C-bag was allowed to soak in the pooled carbon disulfide. After 2 min, the carbon disulfide extract was removed with a disposable Pasteur pipet, placed into a glass sample vial, and capped tightly.

The second sample sat at ambient room temperature for only 4 h before the C-bag was removed and extracted as before.

The third sample was heated for 1 h at 90°C after the C-bag was suspended in the can. After the can cooled for approximately 1 h, the C-bag was removed and also extracted as before. A 3- μL aliquot of each extract was analyzed by GC; the relative quantities of the SAM recovered from samples 1, 2, and 3 are shown in Figs. 3, 4, and 5, respectively.

The effects of varying the concentration levels of SAM were studied, as were those of varying the time and temperature factors for adsorption. Three 1-gal (3.8-L) paint cans were prepared by placing two Kimwipes inside each. To each of these cans, 2.4, 0.5, and 0.2 μL of the SAM was added, respectively. A C-bag was suspended in each can. After sitting for 24 h at ambient temperature, the cans were heated for 1 h at 90°C. The C-bags from these three cans were extracted in the same manner as described previously. The results of the GC analyses are shown in Fig. 6.

While the C-bags were effective and relatively inexpensive to make, the attraction of using a charcoal strip instead of a C-bag arose from doubts about the efficiency of the C-bag's extraction procedure. Some of the SAM could be lost during this process by simple evaporation with the carbon disulfide while in the flask or by poor recovery of the carbon disulfide from the flask and C-bag itself. Loss of the SAM could be significantly reduced by placing a charcoal strip directly into a sample vial after the adsorption process and then adding the carbon disulfide so the extraction takes place inside the sample vial.

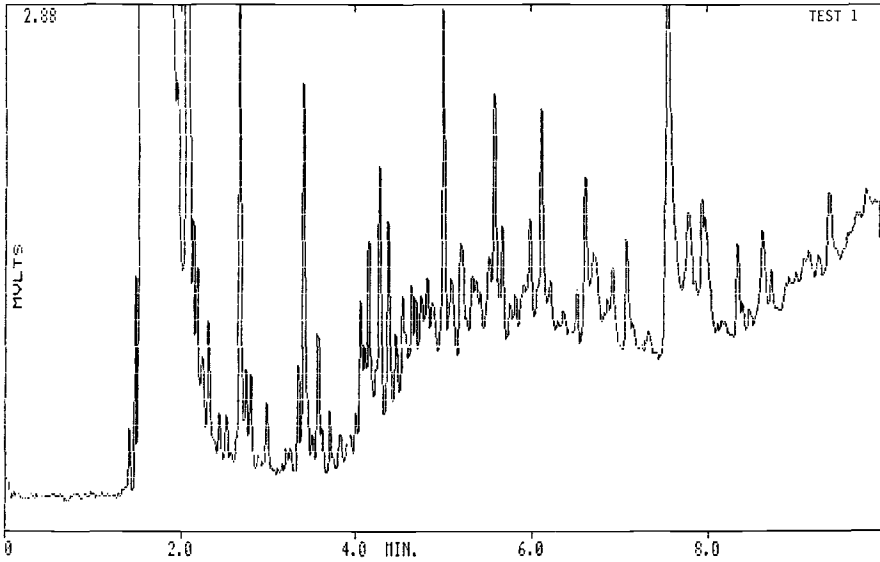


FIG. 3—C-bag's adsorption from 0.2 μ L SAM at ambient temperature for 24 h.

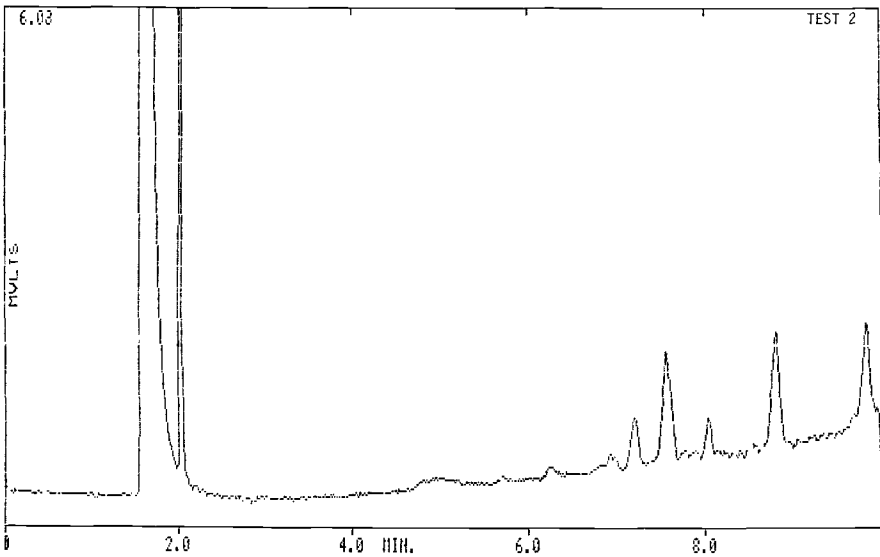


FIG. 4—C-bag's adsorption from 0.2 μ L SAM at ambient temperature for 4 h.

However, the charcoal strip had to adsorb the SAM as well as or better than the C-bags. To test the adsorption capabilities of the charcoal strip and compare them with those of the C-bag, a 0.08-g portion of the charcoal strip was suspended in a sealed 1-gal (3.8-L) paint can containing 0.2 μ L of SAM on two Kimwipes. The can sat at ambient temperature for 24 h before being heated 1 h at 90°C. After cooling to room temperature, the charcoal strip was removed from the paper clip using clean tweezers. The charcoal strip was folded

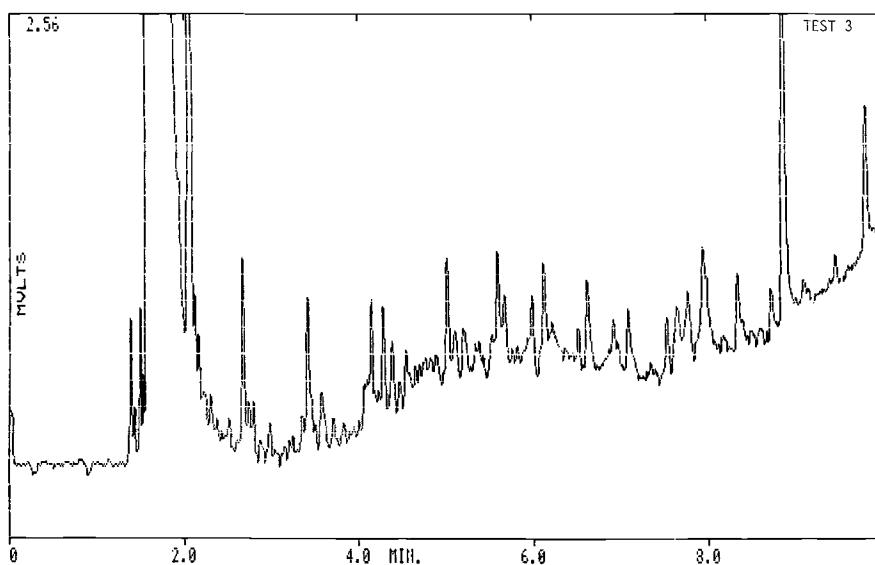


FIG. 5—C-bag's adsorption from 0.2 μL SAM at 90°C for 1 h, followed by cooling to ambient temperature for 1 h.

and placed into a glass sample vial. Then 1 mL of carbon disulfide was added to the vial and capped tightly. After a 2-min period in which the vial was slightly agitated by hand, a 3- μL aliquot of the extract was injected into the GC. The results of this test are shown in Fig. 7.

Water is frequently present in fire debris samples. Therefore, the effects which water vapor have on the adsorption ability of the C-bags and charcoal strips were tested. Original testing of this effect was conducted by subjecting both devices to identically designed samples. Each sample consisted of a 1-gal (3.8-L) paint can in which two Kimwipes had been placed, containing 0.2 μL of the SAM and 2 mL of tap water. The cans sat at ambient temperature for 24 h before being heated for 1 h at 90°C. Each device was extracted as described previously. The chromatograms resulting from these tests are shown in Figs. 8 and 9.

A more recent study of the effects of water vapor on the charcoal strip's adsorption ability came through a proficiency test prepared by the Northwest Association of Forensic Scientists. In that testing, a stock solution was prepared by mixing (for 24 h) 200 μL of gasoline with 2 L of water. The first sample of this test consisted of a 1-pt (0.475-L) metal can containing 1 mL of stock solution diluted to approximately 50 mL with tap water (approximately 0.1 μL gasoline in the sample). The second sample of this test consisted of another 1-pt (0.475-L) metal can, but containing 10 mL of stock solution diluted to approximately 50 mL with tap water. A charcoal strip was suspended in each of these cans for a period of 72 h at ambient temperature. The samples were then heated for 1 h at 50°C. The charcoal strips were then extracted with 0.5 mL of carbon disulfide. The chromatograms resulting from these two tests are shown in Figs. 10 and 11.

Results and Discussion

The C-bags and charcoal strips are capable of adsorbing as little as 0.2 μL of the SAM in a 1-gal (3.8-L) can. These levels of the SAM can be adsorbed either by heating for

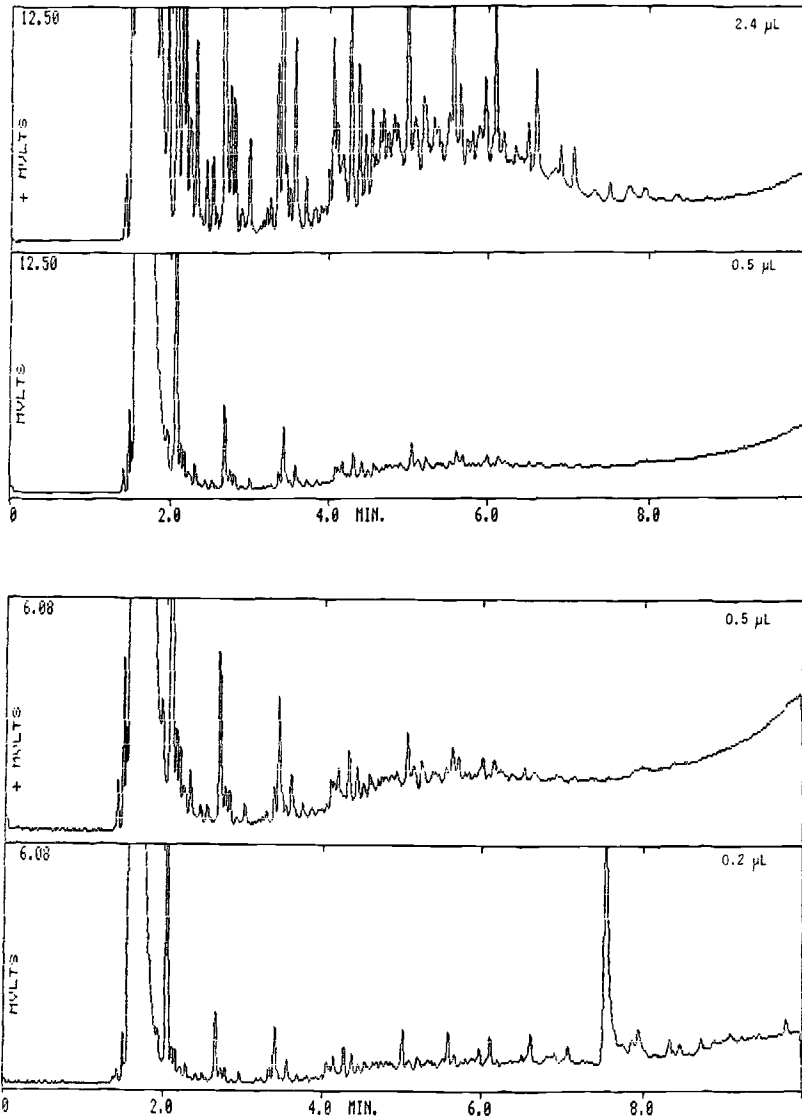


FIG. 6—C-bag's adsorption from 2.4 μL SAM, 0.5 μL SAM, and 0.2 μL SAM after 24 h at ambient temperature plus 1 h at 90°C.

short periods of time (1 h) or by standing at ambient temperature for longer periods of time (24 h). This would offer increased flexibility to the analysis of accelerants. It would allow the opportunity to have a C-bag or charcoal strip placed into an evidence container at the time the evidence is collected in the field or when it is received in the laboratory in order to have it adsorbing long before any actual laboratory analysis time would begin. If the nature of the sample (for example, a suspect's clothing, shoes, or items to be examined for latent prints) would prohibit it from being heated, the adsorption capability of the C-bag or charcoal strip at ambient temperature would still allow an analysis of volatile vapors to be conducted. If the nature of the debris would permit it to be heated,

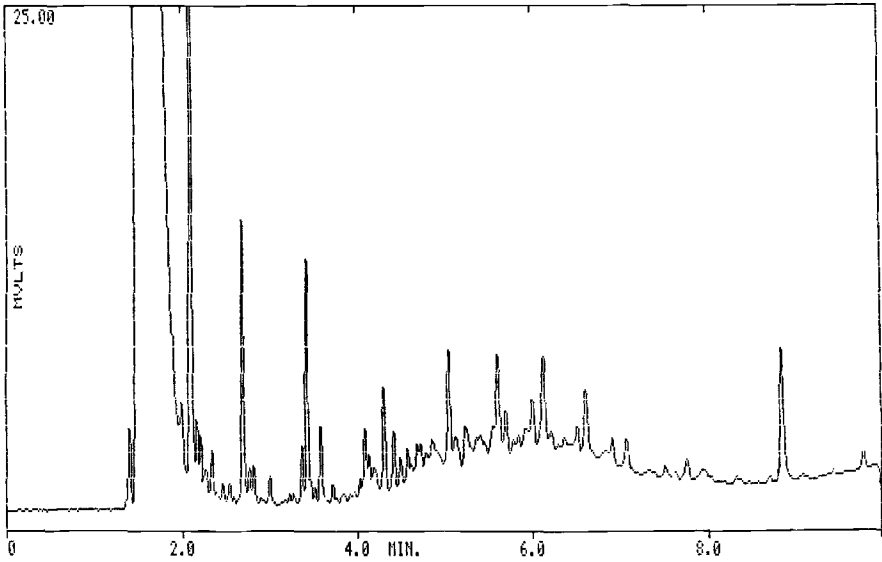


FIG. 7—Charcoal strip's adsorption from 0.2 μ L SAM after 24 h at ambient temperature plus 1 h at 90°C.

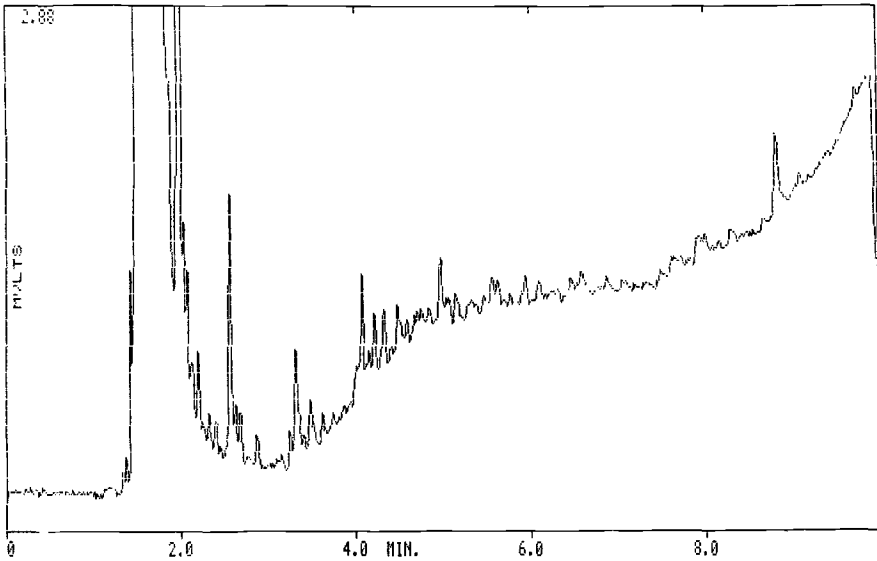


FIG. 8—C-bag's adsorption from 0.2 μ L SAM with 2 mL water after 24 h at ambient temperature plus 1 h at 90°C.

then a minimal heating time (1 h or less) would be needed, since a majority of the compounds would have already been adsorbed as a result of the diffusion within the evidence container while it was in transit to the laboratory or being stored prior to examination.

Since the C-bags and charcoal strips are so easy to work with, two or more may be placed into an evidence container at the same time. One advantage of this is that it would

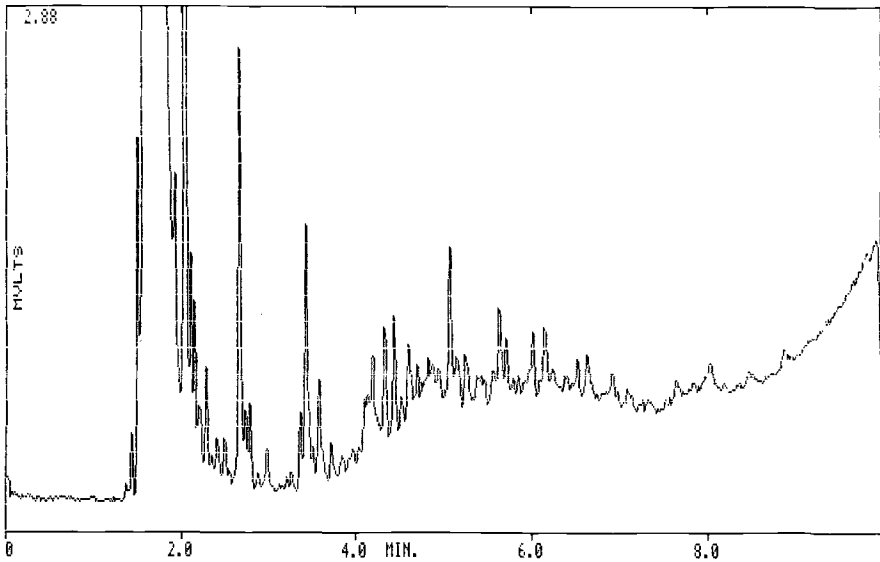


FIG. 9—Charcoal strip's adsorption from 0.2 μL SAM with 2 mL water after 24 h at ambient temperature plus 1 h at 90°C.

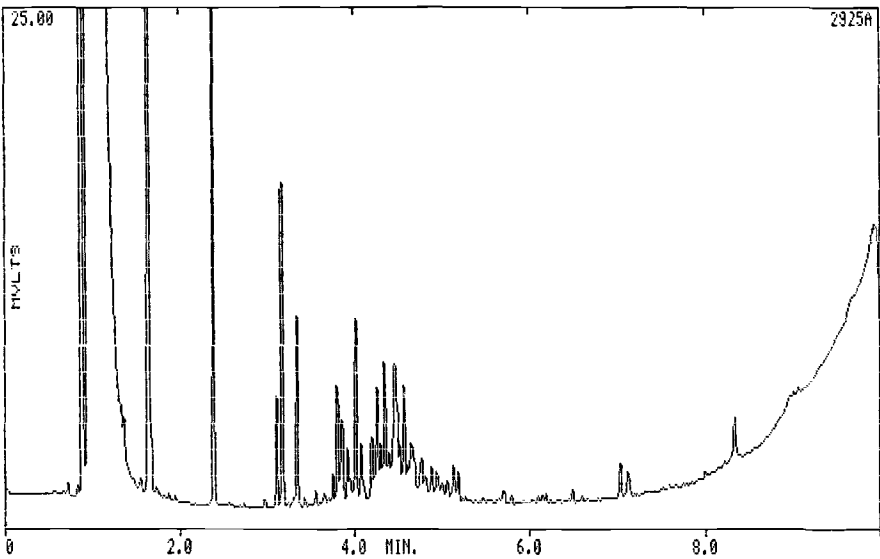


FIG. 10—Charcoal strip's adsorption from 0.1 μL gasoline in 50 mL water after 72 h at ambient temperature plus 1 h at 90°C.

allow periodic sampling of the adsorbed vapors over time. A second advantage would be that additional C-bags or charcoal strips not used for immediate instrumental analysis could be sealed and preserved for future examinations by other analysts (such as review chemists or defense experts).

The presence of water produced little or no effect of the recovery of the SAM when the charcoal strip was used, but reduced recovery somewhat when the C-bag was used.

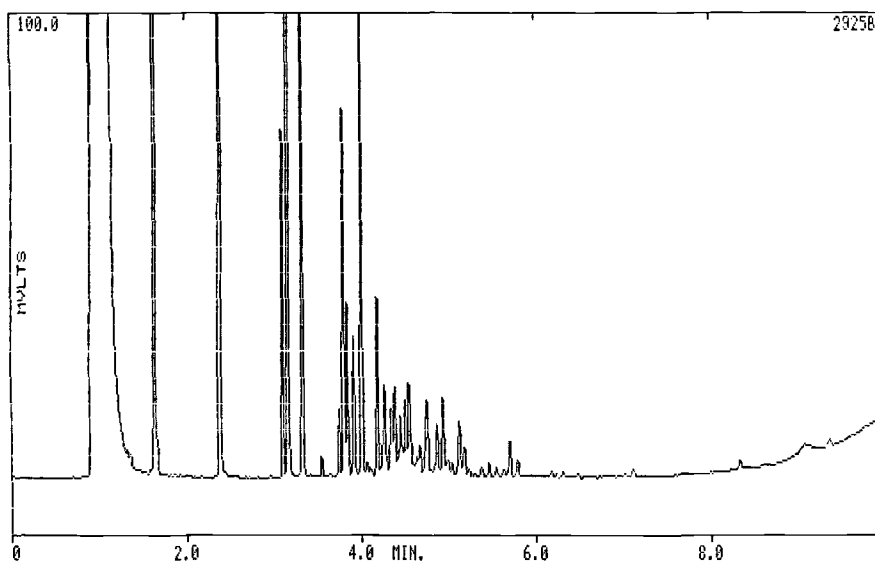


FIG. 11—Charcoal strip's adsorption from 1.0 μ L gasoline in 50 mL water after 72 h at ambient temperature plus 1 h at 50°C.

The latter result may be due to the water's attachment on the paper and the reduction in available surface area for adsorption on the charcoal. A similar effect has been reported when using charcoal-filled glass tubes for purge-and-trap accelerant recovery [9].

Acknowledgments

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