

The Use of Activated Charcoal Strips for Fire Debris Extractions by Passive Diffusion. Part 1: The Effects of Time, Temperature, Strip Size, and Sample Concentration

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ABSTRACT: The introduction of commercially produced activated charcoal strips into fire, debris analysis has provided an easy, efficient and cost effective method for accelerant extraction. Several parameters associated with passive diffusion extraction of fire debris using activated charcoal require consideration in order to obtain a truly representative sample of the accelerant. This study investigated the effects of time, temperature, charcoal strip size and sample concentration on the adsorption of common accelerants. Notable displacement of specific volatile components, similar to the breakthrough noted in dynamic systems, occurred under certain extreme conditions. An analysis protocol was developed to minimize these effects.

KEYWORDS: forensic science, criminalistics, fire investigation, arson, fire debris, arson accelerant, activated charcoal, adsorption, gas chromatography

One of the most popular extraction techniques in fire-debris analysis is passive diffusion adsorption using commercially produced activated charcoal strips (c-strips). Introduced in 1985 (1,2) this method provides for a simple, clean and sensitive method of accelerant extraction. Charcoal trapping of volatiles is not a new technique. Several dynamic and passive adsorption systems have been proposed and are currently used (3-8). The use of commercially produced activated charcoal strips improves these methods by minimizing preparation and handling time, eliminating many possible sources of contamination, and decreasing analyst work time. While the application of dynamic adsorption systems in fire-debris extractions using activated charcoal have been studied extensively (9-11) passive systems have not. This study, initially designed to investigate the effects of the adsorption time, adsorption temperature, charcoal strip size, and sample concentration on the adsorption of common accelerants, has led to further investigation into the dynamics of passive diffusion adsorption with activated charcoal.

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This study will be presented in two articles. The first addresses the effects of the aforementioned parameters on the adsorption of common accelerants with the intent of optimizing the analysis procedure. The second article will examine the dynamics of passive diffusion adsorption in detail in order to explain the process and its effects.

Materials and Instrumentation

The activated charcoal strips used in this study were purchased from Pro-Tek Systems, Inc., Portland, CT. The carbon disulfide was obtained from Fisher Scientific.

The instrumentation consisted of a Hewlett Packard 5890 Gas Chromatograph (GC) with a Flame Ionization Detector (FID) A 12 meter J & W Scientific DB-1 (methyl silicone) column with 0.25 mm ID and 0.25 μm film thickness was installed. The parameters were as follows:

Injector Temperature:	275°C
Detector Temperature:	280°C
Linear velocity:	25.6 cm/s
Split Ratio:	25:1
Carrier Gas	Helium
Temperature Program:	
Initial Temperature:	60°C
Initial Time:	1.00 min
Rate:	25.0°C/min
Final Temperature	300°C
Final Time	5.00 min

Apparatus

The extractions were achieved using sampling devices constructed by tying a length of unwaxed dental floss to a small clean metal paper clip. The outside leg of the paper clip was bent outward and then inserted through the activated charcoal strip. The device was suspended inside a paint can, using the sealed lid to secure it in place (Fig. 1).

Experimental

Time and Temperature Comparison

Charcoal strips were cut into $\frac{3}{8}$ -in. (9.5-mm) diameter discs using a hole punch to ensure uniform size. Quart size paint cans containing Kimwipes were spiked with 3 μL of heat Standard

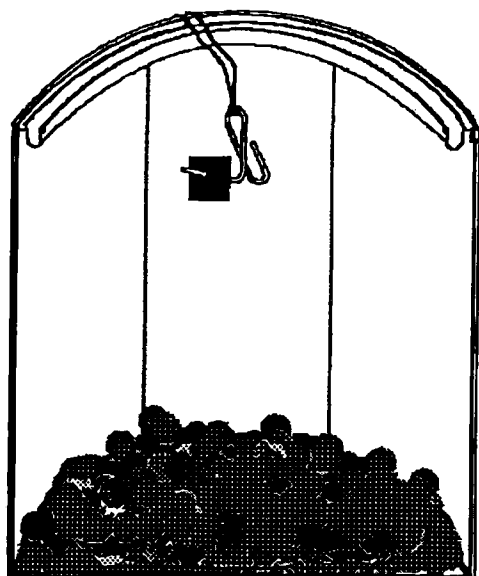


FIG. 1—Extraction apparatus (not to scale).

Accelerant Mixture "SAM" (1:1:1 50% Evaporated Gasoline: Kerosene: Diesel Fuel) using a disposable micropipet (Fisher Scientific). The adsorption was conducted at ambient temperature, 60°C, 90°C and 120°C for periods of 2, 8, 16, 24 and 48 h. After adsorption, the heated cans were allowed to cool for approximately 30 min before the adsorbed strips were placed into screw cap glass vials. The strips were eluted with 100 μ L of carbon disulfide, vortexed and analyzed by GC-FID. The minimum volumes of carbon disulfide, which allowed for complete elution of the strips with maximum sensitivity were used throughout this study (12).

Strip Size Comparison

Activated charcoal strips were cut into four sizes (4 mm \times 4 mm, 4 mm \times 8 mm, 8 mm \times 8 mm, and 8 mm \times 16 mm). Sets of four strips, one of each size, were suspended into gallon size paint cans containing Kimwipes. The following accelerants were added with pipets:

- Can 1: 3 μ L 50% Evaporated Gasoline
- Can 2: 25 μ L 50% Evaporated Gasoline
- Can 3: 3 μ L SAM
- Can 4: 25 μ L SAM

The samples were placed in the oven to adsorb for 16 h at 60°C. After cooling to room temperature, the strips were removed and placed into screw cap glass vials. Since the larger strips would require larger volumes of carbon disulfide, the samples were "normalized" by cutting 4 mm \times 4 mm sections from each adsorbed strip. Each section was eluted with 50 μ L of carbon disulfide, vortexed, and then analyzed by GC-FID.

Concentration Comparison

Samples were prepared by suspending $3/8$ -in. (9.5-mm) diameter activated charcoal discs into quart size paint cans. The following volumes of SAM were added to the containers with syringes and pipets.

Can 1	0.3 μ L
Can 2	1.0 μ L
Can 3	5.0 μ L
Can 4	10.0 μ L
Can 5	20.0 μ L

The samples were heated in an oven at 60°C for 16 h and then allowed to cool to room temperature. The adsorbed strips were placed into screw cap glass vials, eluted with 100 μ L of carbon disulfide, vortexed, and analyzed by GC-FID.

Results and Discussion

Time and Temperature Comparisons

At lower temperatures, the higher molecular weight compounds were not sufficiently volatilized for adsorption as illustrated by the comparison of ambient temperature adsorptions (Fig. 2). A reasonable representation of the components up to C14 (tetradecane) was achieved. However, even with an adsorption time of 48 hours, the components above C15 (pentadecane) were not sufficiently sampled.

At higher temperatures, the higher molecular weight components were volatilized and sufficiently adsorbed, however, a distortion occurred in the lighter end of the volatile range (Fig. 3). There appeared to be a displacement of the lighter components in the sample, not unlike the effects of breakthrough noted in dynamic systems (13,14). Over time and with increasing temperature, the distortion was much more pronounced, with the emphasis of the chromatograms shifted to the higher molecular weight components. Under extreme conditions, the relative concentration of the lighter components was drastically diminished (Fig. 4).

Strip Size Comparison

The amount of activated charcoal did not appear to be a factor in displacement when lower concentrations (3.0 μ L) of accelerants were used (Fig. 5). However, at the higher concentrations (25.0 μ L), displacement was again evident, the degree of displacement increased as the strip size decreased. This indicates that displacement is, at least in part, a function of the capacity of the strip (Fig. 6).

Concentration Comparison

The dependence on strip capacity was further illustrated by the comparison of accelerant concentration. As the concentration of the accelerant in the sample increased, the amount of displacement also increased (Fig. 7).

Conclusion and Recommendations

An adsorption process that would provide a reasonable sampling of volatiles components in the range of C5-C20 would be ideal. This study shows that in order to obtain a representative sample, all the parameters associated with the extraction must be considered. There are a number of time, temperature, strip size and concentration combinations that could be used, however, other factors, including efficiency, cost effectiveness, and practicality should also be considered. Based on the results of this study and actual case sample analysis, the authors propose an analysis scheme for extraction of fire debris (Fig. 8).

While the concentration of ignitable liquids present in fire-debris samples submitted for analysis is not by any means controllable, it can be notable. Samples that contain high levels of light petroleum products, gasoline and/or medium range petroleum products are prone to displacement, and therefore require shorter adsorption times, lower temperatures or larger charcoal strips to minimize the effect. Preliminary screening of samples prior to or during the

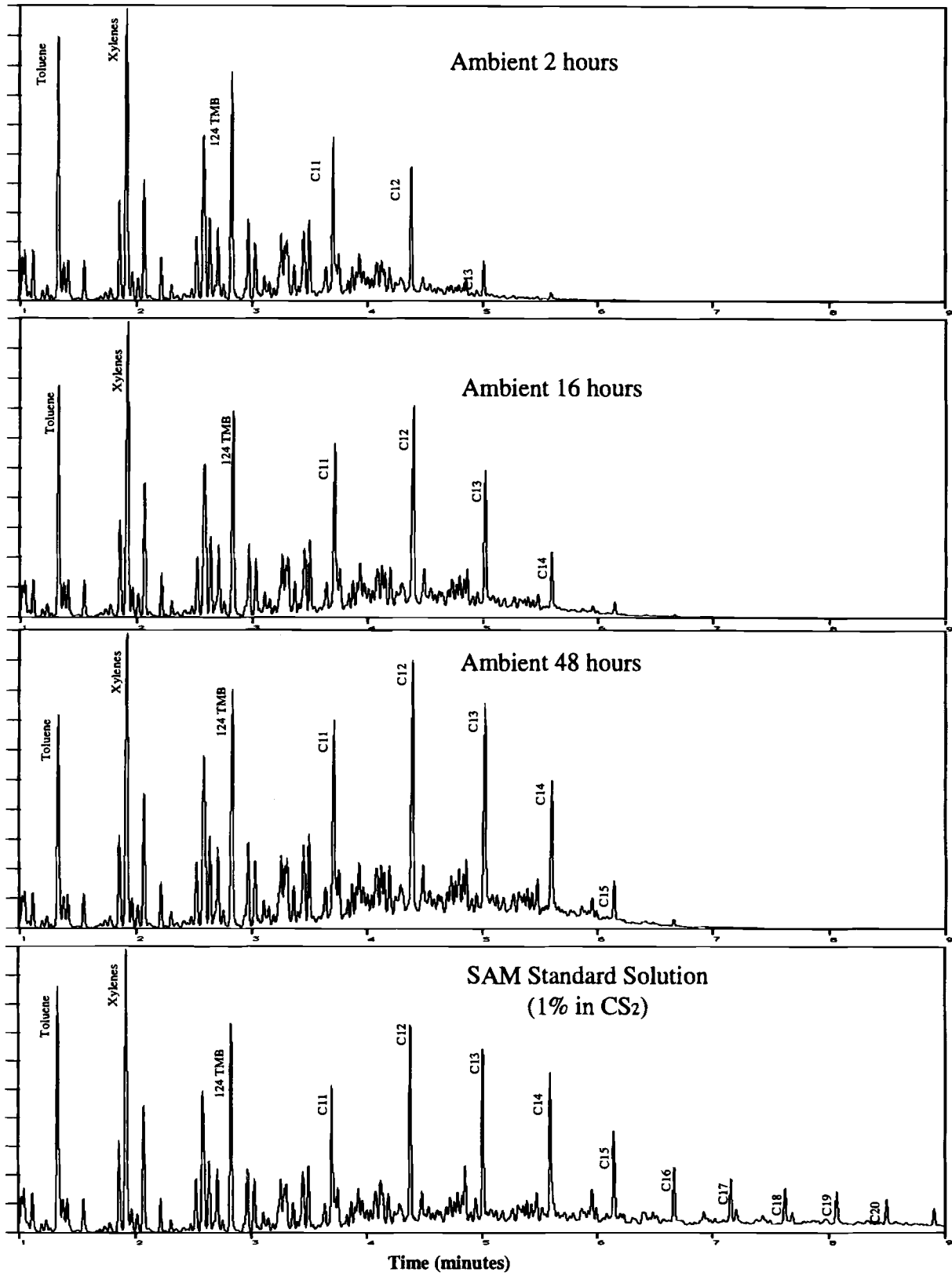


FIG. 2—Comparison of chromatograms obtained by adsorption of 3 μ L of SAM on $\frac{3}{8}$ " activated charcoal discs at ambient temperatures for various lengths of time. Chromatograms abbreviated (1–9 minutes) to enhance detail.

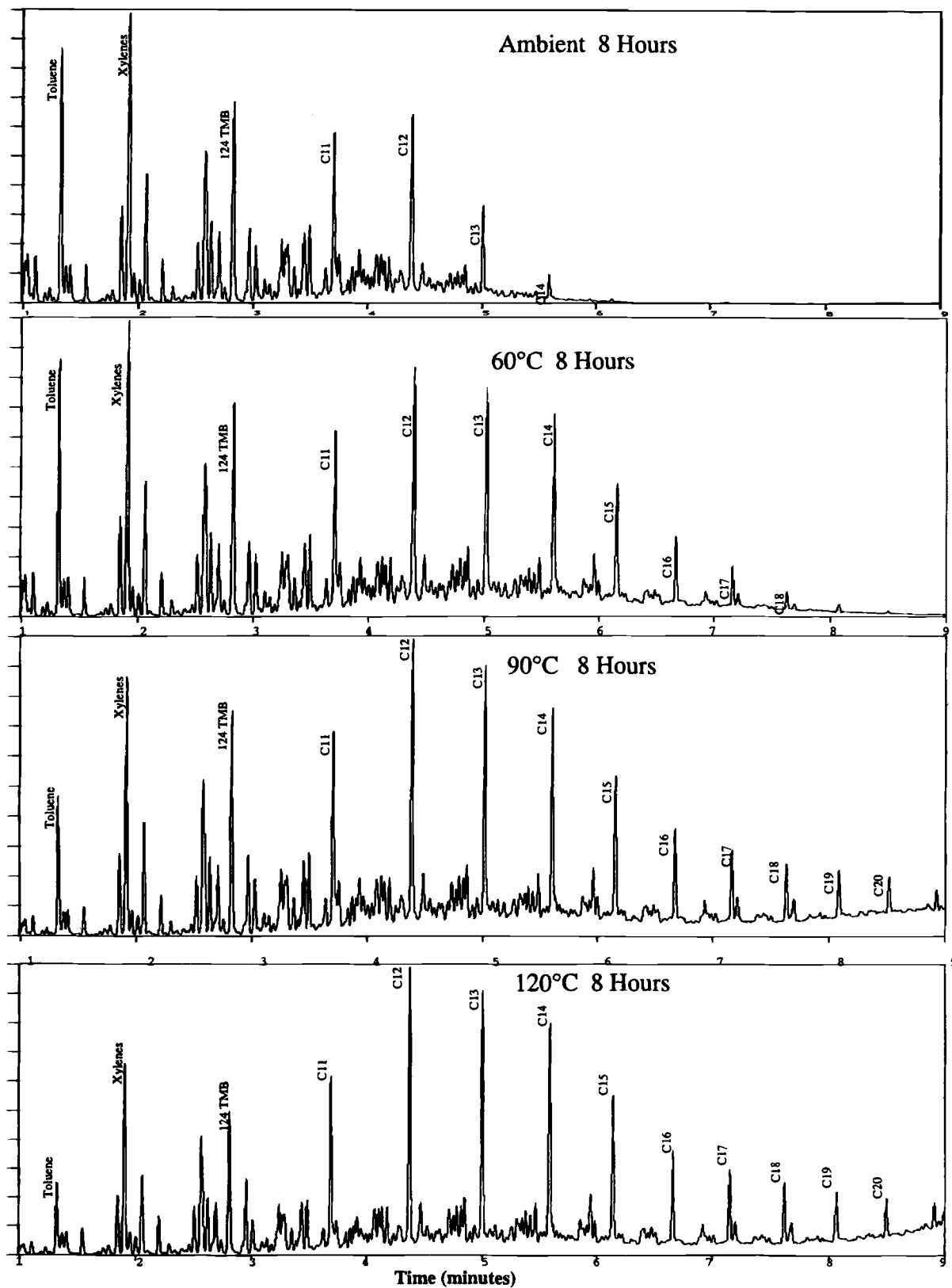


FIG. 3—Comparison of chromatograms obtained by adsorption of 3 μ L of SAM on $3/16$ " activated charcoal discs at various temperatures for a period of eight hours. Chromatograms abbreviated (1–9 minutes) to enhance detail.

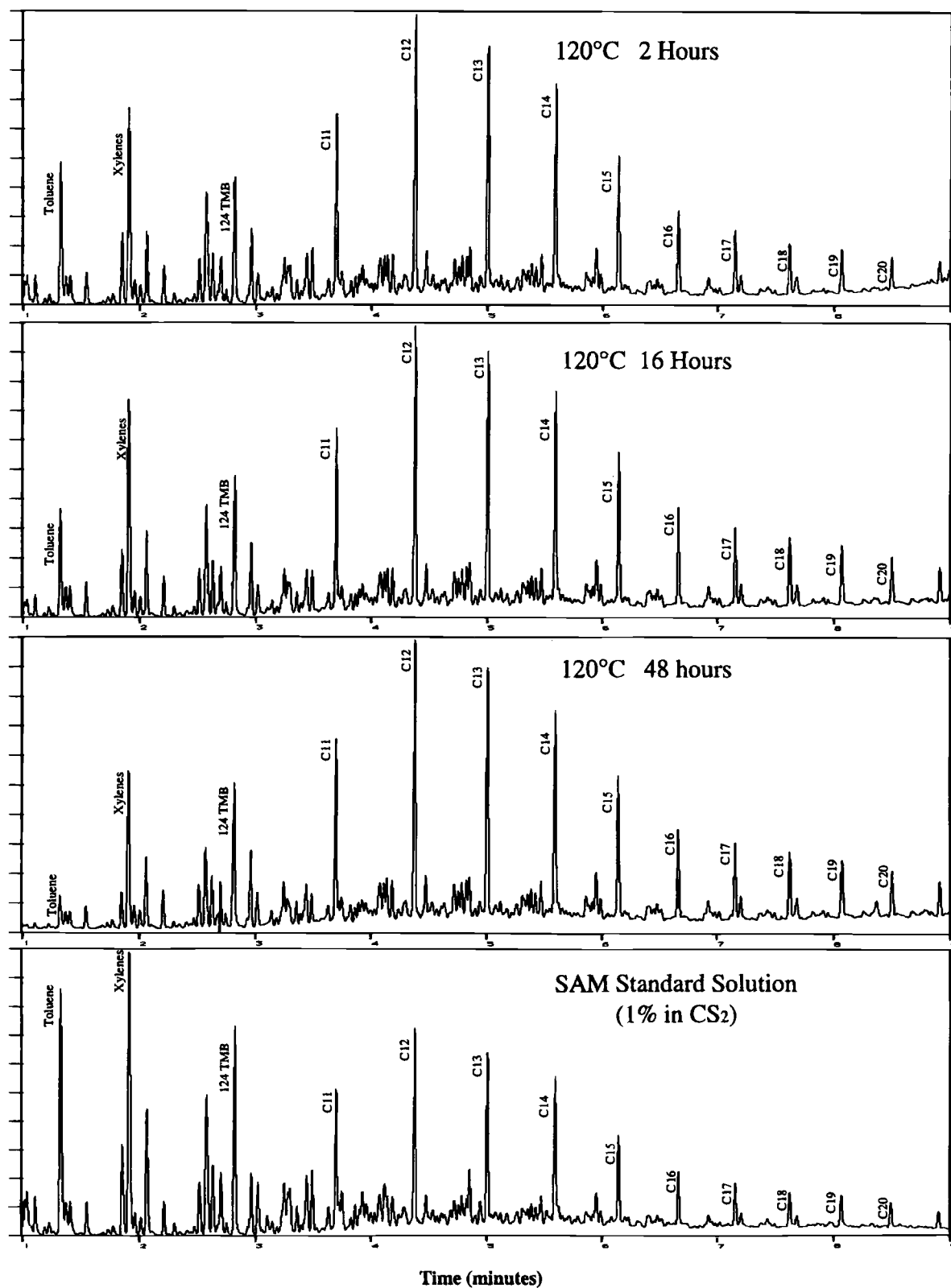


FIG. 4—Comparison of chromatograms obtained from adsorption of 3 μ L SAM on $3/8$ " activated charcoal discs at 120°C with varied adsorption times. Chromatograms abbreviated (1–9 minutes) to enhance detail.

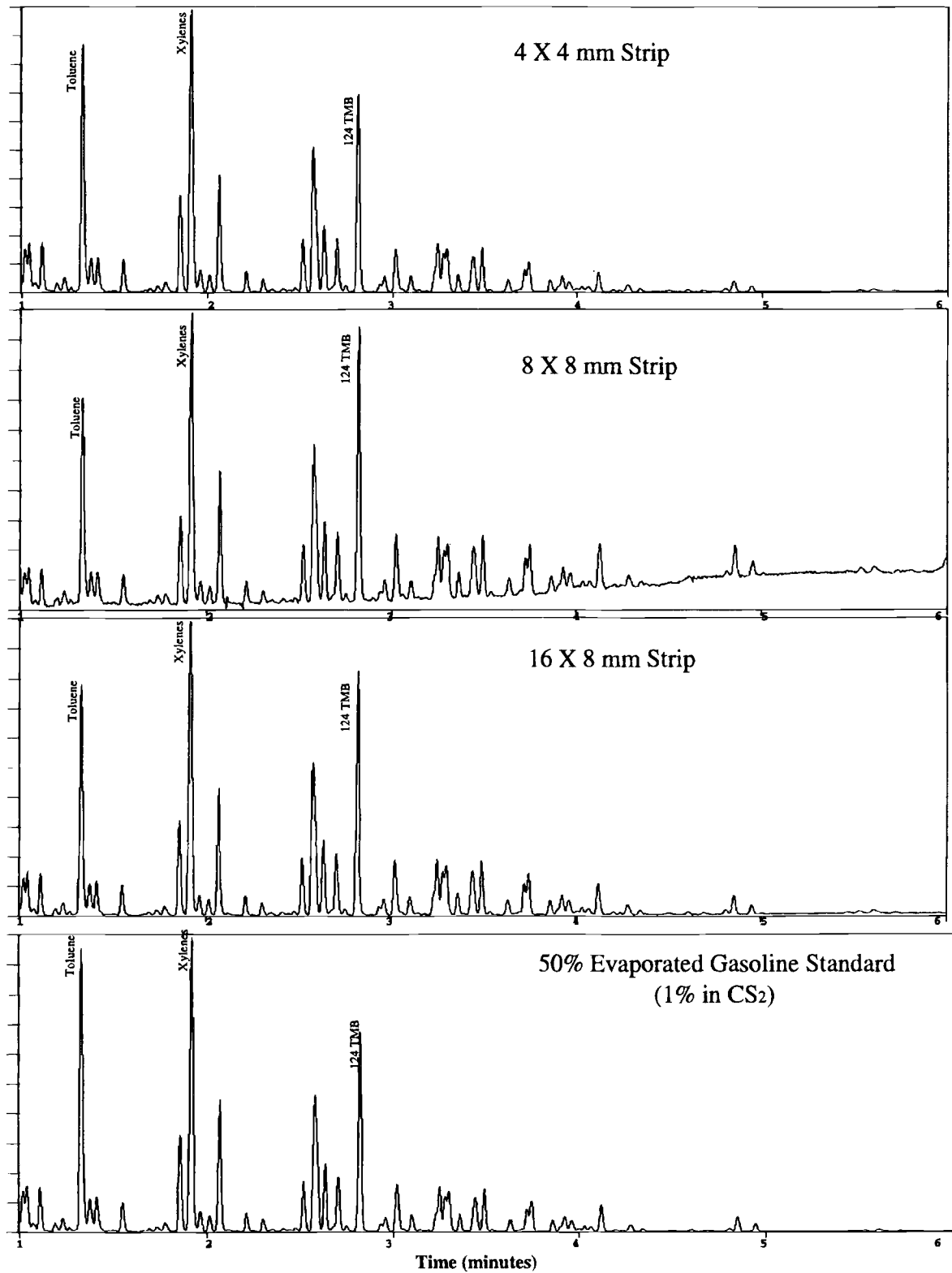


FIG. 5—Comparison of chromatograms obtained from adsorption of 3 μL of 50% evaporated gasoline at 60°C for 16 h on varied sizes of activated charcoal strips. Chromatograms abbreviated (1–6 minutes) to enhance detail.

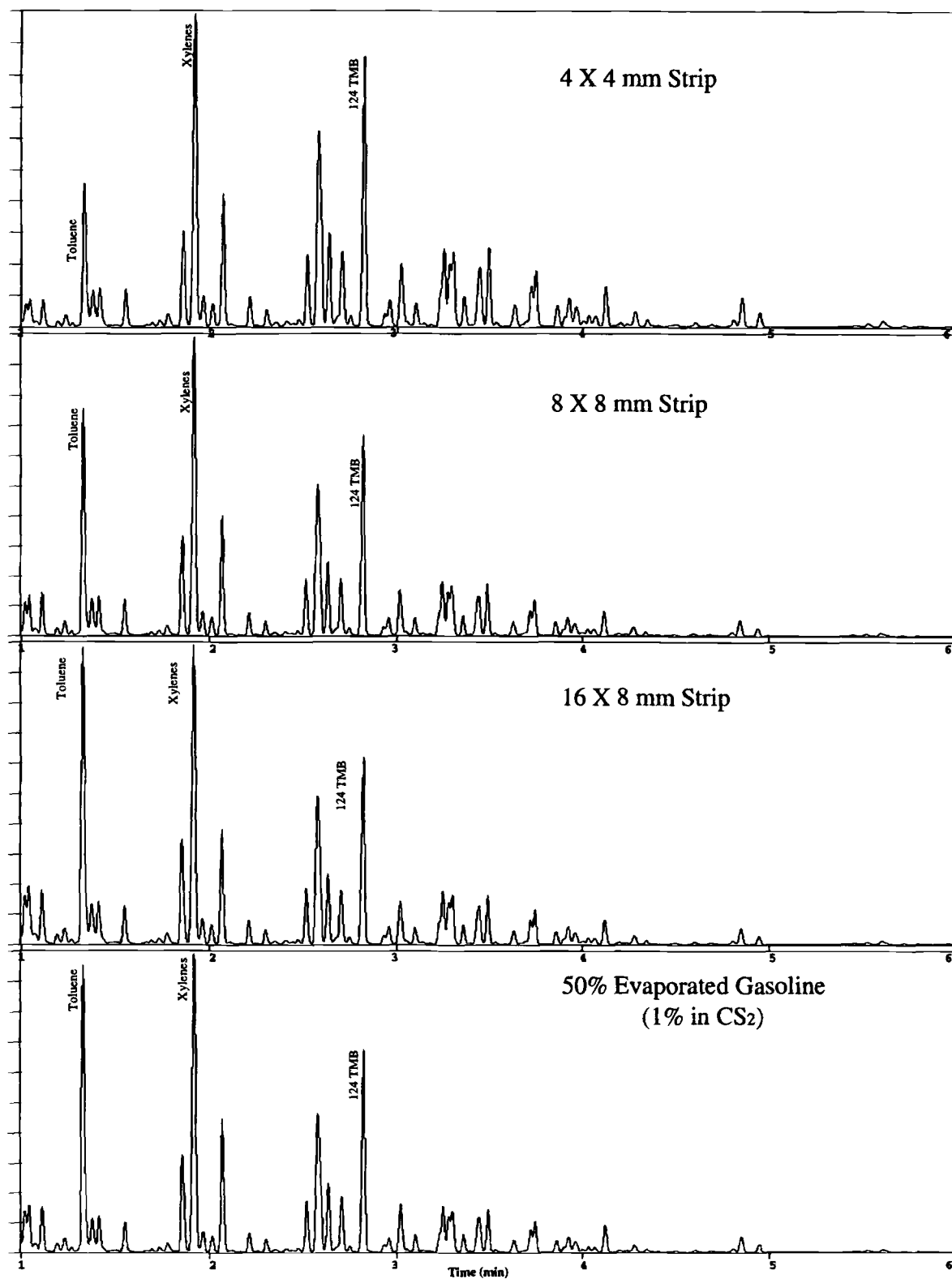


FIG. 6—A comparison of adsorption of 25 μ L 50% evaporated gasoline with varying sizes of activated charcoal strips at 60°C for 16 h. Chromatogram abbreviated (1–6 minutes) to enhance detail.

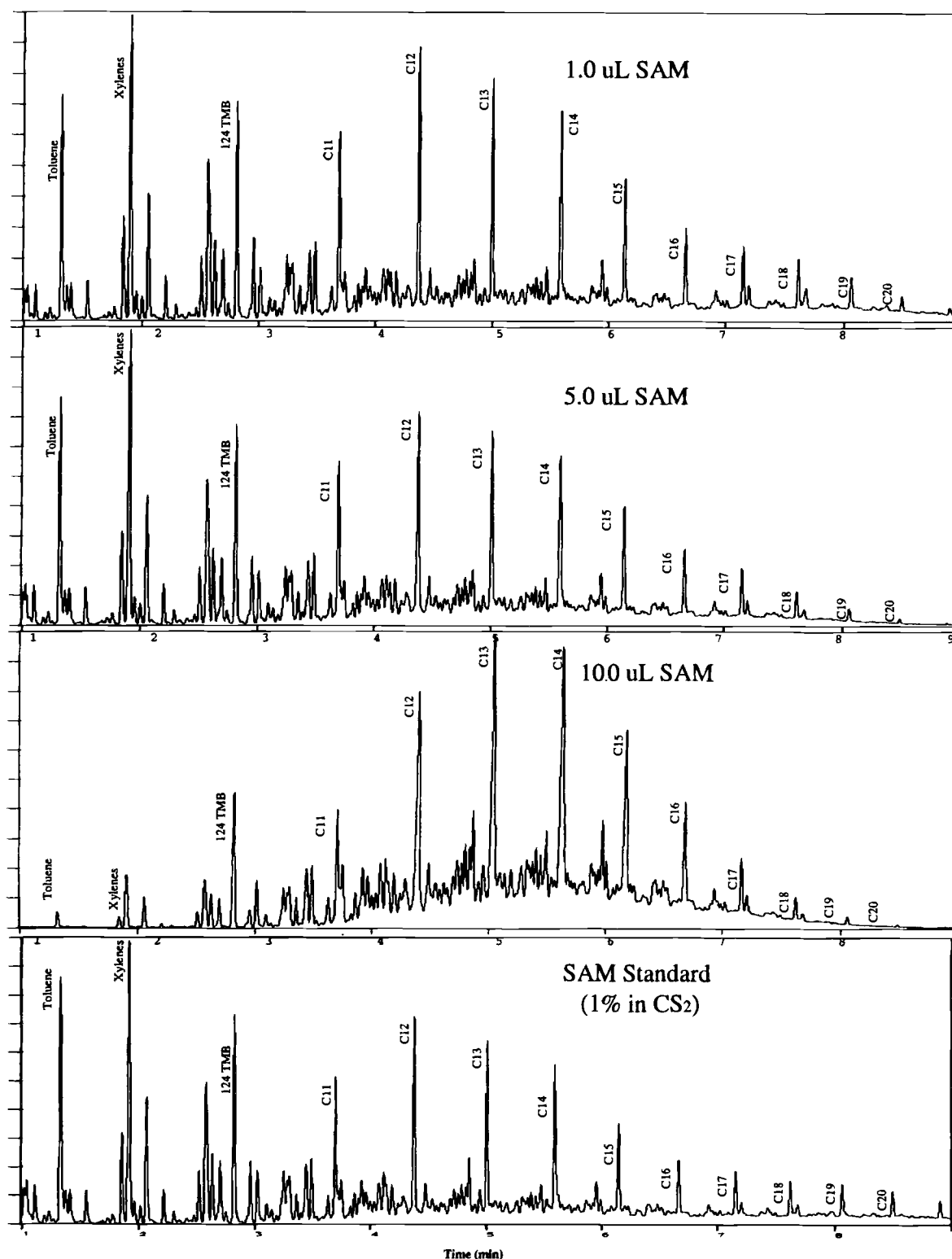


FIG. 7—Comparison chromatograms obtained from adsorption of various amounts of accelerant (SAM) adsorbed on $\frac{3}{16}$ " activated charcoal discs for 16 h at 60°C. Chromatograms abbreviated (1–9 minutes) to enhance detail.

adsorption process allows the analyst to determine the proper combination of adsorption parameters.

Samples may be screened by either olfactory appraisal or direct headspace sampling. Many flammable and combustible liquids have distinctive odors, their relative concentration may be subjectively determined by the analyst from the strength of their odor.

Since many analysts are uncomfortable with inhaling volatile chemicals, direct headspace sampling of the debris prior to or during the c-strip adsorption is an alternative method for determining the relative concentration (15). The results of the headspace analysis or the strength of ignitable liquid odors are an important first step in the analysis scheme.

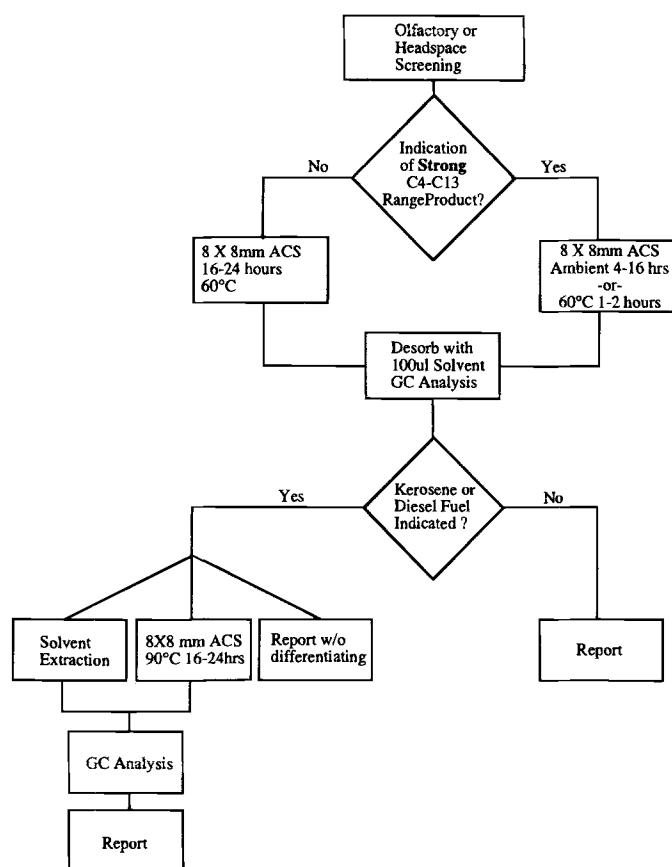


FIG. 8—An analysis scheme for extraction of fire debris.

The combination of time and temperature for the adsorption process should allow for the adsorption of heavier components without significantly contributing to the displacement of the lighter components. Temperatures below 60°C are not adequate to volatilize the heavier components of products in the diesel fuel range making it difficult to differentiate between kerosene and diesel-fuel products. Temperatures above 60°C increase the rate of displacement in light petroleum products, gasoline, and medium range products (16). Higher temperatures (over 90°C) are not recommended because they promote the degradation and pyrolysis of substrates, further complicating the separation and identification processes.

The optimum adsorption time is dependent on the adsorption temperature as well as the concentration and composition of any flammable or combustible liquids present. Lower temperatures require more time to volatilize and adsorb the higher molecular weight compounds. At higher temperatures, the adsorption time must be abbreviated to discourage displacement of the lower molecular weight components. At moderate temperatures (50°C–70°C), the adsorption time can effectively range from 12–24 h for general screening of most samples. In terms of practicality and efficiency, the duration of adsorption in this analysis scheme was limited to 16–24 h to conform with work schedule of analysts. Samples prepared at the end of the day and adsorbed overnight (approximately 16 h) are ready for extraction and analysis the following morning.

The size of the activated charcoal strip used in the analysis has a significant bearing on the efficiency of the extractions and cost of the analysis. Larger strip sizes are not only more expensive, but require more solvent for desorption, increasing the volume

of potentially toxic and hazardous solvent being used. However, smaller strip sizes saturate quickly and thus begin displacement at lower concentrations. An 8 mm × 8 mm strip (roughly 1/4 of a Pro-Tek Strip) is recommended as the minimum amount of charcoal for analysis of debris in quart or gallon size containers.

Finally, the composition of the substrate should be considered. It can, in some circumstances, be as significant as the concentration of the accelerant. With some common substrates, most notably, burned carpet and pad, the heavier components of diesel fuel may not be sufficiently volatilized or adsorbed (17). In samples where kerosene or diesel fuel are indicated, the analyst has several options. Many laboratories elect to report samples as heavy petroleum products, heavy petroleum distillates, or fuel oils without differentiating between the kerosene and diesel-fuel classes. Others elect to perform additional extractions to aid in the classification. Solvent extraction is one option, re-extraction with activated charcoal at a higher temperature is another.

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