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The Collection and Detection of Accelerant Vapors Using Porous Polymers and Curie Point Pyrolysis Wires Coated with Active Carbon

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ABSTRACT: The sampling of organic vapors from arson residues using different types of adsorbents is studied. Sampling tubes packed with porous polymers (Porapak Q and Tenax GC) as well as Curie point pyrolysis wires coated with finely divided or granular active carbon are used. The adsorption and desorption of several common accelerants carried out by these techniques is compared. The optimum conditions for applying the described techniques to the forensic science examination of fire debris are studied. Some comparisons with conventional headspace analysis of accelerant vapors are discussed.

KEYWORDS: forensic science, accelerants, vapors, fire accelerants, vapor analysis, porous polymers, activated charcoal, Curie point pyrolysis, thermal desorption, fire residues

In our laboratory two methods are used for the routine analysis of accelerant residues: headspace analysis of vapors and steam distillation of residues. Other methods for the detection or isolation or both of volatile fire accelerants from fire debris are described in literature [1].

Several years ago, a new method for detection of accelerant vapors was developed by Twibell and Home [2]. This method is based on the Curie point pyrolysis system. The Curie pyrolysis wires are coated with finely divided activated charcoal. The accelerant vapors are adsorbed on the charcoal and desorbed in the usual manner in the pyrolysis unit. This method is more sensitive than the methods mentioned above, particularly when the desorbed accelerant is analyzed by capillary gas chromatography [3-5].

The method that is well established for the adsorption of organic vapors in air pollution studies uses adsorption tubes packed with porous polymers. Vapors are adsorbed by drawing a certain amount of air through the adsorbent tubes. By heating the tubes the adsorbed organic compounds are desorbed and analyzed by gas chromatography. Various experimental arrangements for adsorption and desorption of vapors have been described in literature [6-9]. Good results achieved by this technique in detection and analysis of accelerant vapors were recently reported [10].

In this work the adsorption and analysis of accelerant vapors by different types of adsorbents and using the two adsorption techniques described above were studied. Several types of

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porous polymers were used as adsorbents and also several types of adsorption wires coated with active carbon.

Sensitivity, advantages, and disadvantages of these techniques are compared. Some comparisons are made with conventional headspace analysis. We have also tried to reach optimal conditions for sampling of vapors from arson residues when the type of accelerant is not known.

Several types of common accelerants with different boiling ranges and thus different volatilities were employed in this study. Before all, petrol, paraffin (kerosene), and diesel fuel were studied. Some experiments were performed using volatile alcohol and ketone mixtures, representing accelerants often encountered in Sweden.

Materials and Methods

Materials

Porous Polymers—Porapak Q (80–100 mesh) was purchased from Waters Associates and Tenax GC (60–80 mesh) and Chromosorb 102 (80–100 mesh) were gifts from Institut för Vatten och Luftanalys (IVL), Göteborg, Sweden. The polymers were washed with toluene and methanol and conditioned overnight at 330°C (Tenax GC) or 245°C (Porapak and Chromosorb).

Active Carbon—Granular active carbon (type NIOSH) was obtained from commercially available sampling tubes; powdered active carbon (Norit, passing 240 mesh) was a gift from Home Office Central Research Establishment (HOCRE), Aldermaston, England.

Methods

Sampling of Accelerant Vapors with Porous Polymers—The experimental arrangement for sampling of accelerant vapors on porous polymers is shown in Fig. 1. It is a very simple arrangement. Glass liners (10 cm long and 2.5 mm inner diameter) from the injector of a Perkin-Elmer Sigma 2 gas chromatograph were packed for a length of 3 to 4 cm with Tenax GC, Porapak Q, or Chromosorb 102. The amount of Tenax was about 35 mg, that of the other two polymers about 50 to 60 mg. The adsorbent was held in position by plugs of glass wool.

Accelerant residues or filter paper impregnated with pure accelerant were placed on the bottom of a paint can and the vapor phase from the can was drawn through two adsorbent tubes (for double sampling or for example, comparison between two different adsorbents). The can was heated on a hot plate. The sampled volumes were usually between five to ten times the volume of the can. After the collection of vapors, the tubes were transferred to the heated injector of Sigma 2 gas chromatograph. The thermal desorption of adsorbed samples was carried out at 170 to 200°C when using Porapak Q or Chromosorb 102 and at 250°C with Tenax GC.

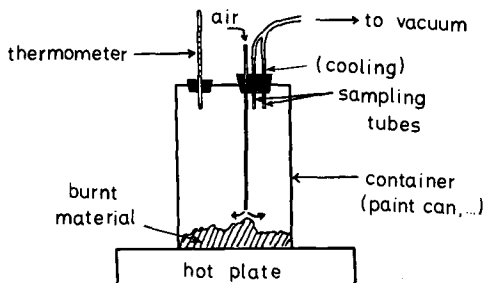


FIG. 1—Accelerant vapor collection system with adsorbent tubes. Cooling of the adsorbent, for example, by air jet, is possible.

Sampling of Accelerant Vapors with Adsorption Wires—The adsorption wires proposed by Twibell and Home [2] are prepared by bonding finely divided active carbon (passing 240 mesh) with inert adhesive (cement binder—water glass [sodium silicate]) to pyrolysis wires. The coated wires are simply suspended in the atmosphere containing accelerant vapors. The desorption of the sample is made in a Curie point instrument in the normal manner. The pyrolysis temperature used in the study mentioned above was 358°C. We have used 510°C Ci point pyrolysis wires coated in three different ways (Fig. 2).

The first wire was coated by powdered active carbon [2]. The end of the second wire was flattened and looped to form an eye. The eye was immersed into water glass for a little while. Afterwards, several (two to four) granules of NIOSH active carbon were placed in the loop and the wire allowed to dry at room temperature. The third wire in the figure is a combination of the first and the second wire.

A piece of filter paper impregnated with an accelerant was placed in a 250-mL glass flask. The sampling was carried out with the adsorbent wire stuck in the cork sitting in the neck of the flask.

Before sampling several 10-s pyrolytical pulses were applied to the adsorbent wires to remove impurities and adsorbed compounds.

Headspace Analysis—Headspace analysis was performed by taking 2- to 5-mL aliquots of the vapor phase. For comparison reasons the sampling was carried out from cans or glass flasks of the same volume as those employed in the adsorbent studied. The temperature was room temperature if not otherwise stated.

Gas Chromatography—The analysis of samples adsorbed on porous polymers was performed on a Perkin-Elmer Sigma 2 gas chromatograph. Glass columns were packed with 5% OV-17 on Gas Chrom Q. The length of the column was 2.7 m, nitrogen gas flowed at a rate of 40 mL/min, the temperature program was 25 to 10°C/min—300°C for all the samples except petrol where the program 25 to 6°C/min—200°C was used.

The desorption of samples from activated carbon was carried out in a Curie point pyrolysis unit attached to a Pye-Unicam Series 104 gas chromatograph. Glass columns, 1.7 m long, were packed with 6% OV-101 on Gas Chrom Q. The nitrogen flow was 25 mL/min. The temperature program used was 60 to 10°C/min—300°C for most of the samples and 50 to 7°C/min—200°C for petrol samples. Alcohol and ketone mixtures were analyzed using 1.7-m-long glass columns packed with Porapak Q (80-100 mesh). The temperature program was 140 to 7°C/min—200°C.

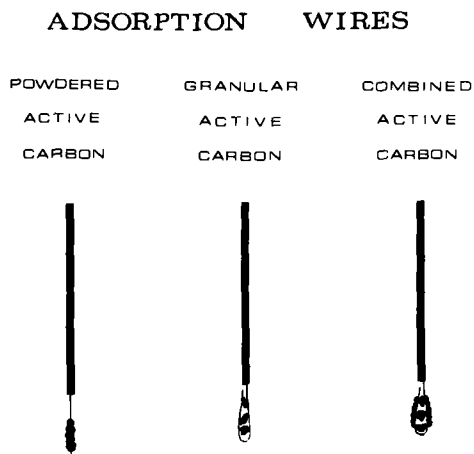


FIG. 2—Three types of adsorbent wires used in this study.

The desorption of the adsorbed samples was performed by two consecutive 10-s pyrolytical pulses in experiments with powdered active carbon, otherwise by two 15-s pulses.

Results

The headspace technique, that is, the direct injection of vapor aliquots, is a well established technique for vapor analysis and often can indicate the type of accelerant used. Its sensitivity is satisfactory for the most volatile compounds but decreases rapidly with decreasing volatility of analyzed substances.

Porous Polymers

Because of their hydrophobic nature, the porous polymers used in this study have the ability of adsorbing, reversibly, most organic compounds while not retaining water and lighter permanent gases. Since various porous polymers have different physical characteristics, several types of polymers were employed in this study. The surface area of Porapak Q is 550 to 650 m²/g, Chromosorb 102 is 300 to 400 m²/g, but that of Tenax GC is only 19 m²/g [11]. Thus, Porapak Q and Chromosorb 102 are stronger adsorbents than Tenax. We found that the adsorption capacities of Porapak Q and Chromosorb 102 for hydrocarbons were almost identical, and therefore only the results obtained with Porapak Q and Tenax GC are shown. Porapak Q retains volatile compounds like *n*-hexane to a much higher degree than Tenax. This can be seen in Fig. 3 showing the adsorption properties of Tenax and Porapak Q using an approximately equimolar test mixture of *n*-alkanes (from C₆ to C₂₂) at room temperature. The difference between headspace analysis and the analysis employing porous polymers is also illustrated. The sensitivity of the headspace analysis is acceptable only for the most volatile components of the test mixture.

At room temperature, the volatility of hydrocarbons higher than C₁₆ is very low. Nevertheless, only the C₂₂ hydrocarbon was not detected with porous polymers in these experiments. If the sampling is carried out at elevated temperatures, a very broad range of volatilities of the analyzed compounds will be covered, provided that the sampling tubes are kept cool or at temperatures close to room temperature. An analysis of a similar mixture of *n*-alkanes sampled at elevated temperatures is shown in Fig. 4. The sample was heated slowly on a hot plate, starting at room temperature and interrupting when the temperature at the level of the lowest part of the sampling tubes reached approximately 70°C. Figure 4 demonstrates the greater capacity of Porapak Q compared with Tenax to retain volatile hydrocarbons (C₆-C₈) for sampling at elevated temperatures. The detection of hydrocarbons heavier than C₁₆ is clearly improved.

The adsorption on porous polymers is the most sensitive method described here. The accelerant may be transferred almost completely to the adsorbent and the thermal desorption of the compounds studied here appears to be complete. This high sensitivity is evident from the values of attenuation (att.) in the figures.

Figure 5 illustrates the comparison between headspace analysis and the desorption of paraffin vapors from Porapak Q and Tenax. In addition to much higher sensitivity, the analysis with porous polymers results in chromatograms that are in good agreement with the original accelerant. The relative proportions between various hydrocarbon isomers (for example, smaller peaks between two *n*-alkanes) is maintained particularly well using Porapak Q.

The disadvantage of weak adsorbents turns into an advantage when the adsorbed sample must be thermally desorbed. The rate of desorption from Tenax is much higher than that from Porapak Q (at the same temperature). Tenax is also thermally more stable—does not decompose below 375°C [11]. The temperature limit for Porapak Q is only 250°C. These facts together with the higher chemical stability of Tenax against oxidation make it a more suitable adsorption material when larger adsorbent tubes are used or in connection with capillary gas

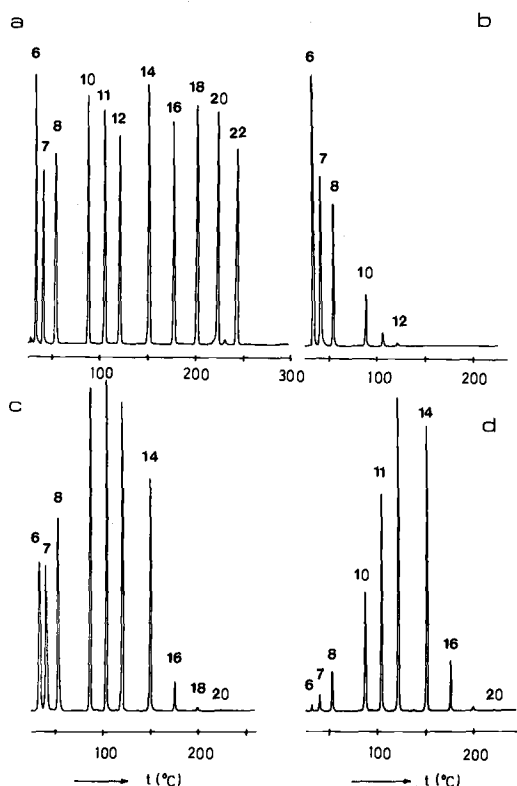


FIG. 3—The analysis of a mixture of n-alkanes ($C_6 - C_{22}$). The sampling was performed at room temperature and Tenax GC and Porapak Q tubes were used in a parallel manner for 1- μ L test mixture. Since the gas flow through the two adsorbent tubes was different the sampling volumes for Porapak and Tenax are not the same. (a) 0.15 μ L of the test mixture injected, att. 12 800. (b) 2.5 mL of headspace injected, att. 320, 20 μ L of the mixture in a 250-mL can. (c) The sample desorbed from Porapak Q, att. 12 800, sample volume 1.5 L. (d) The sample desorbed from Tenax GC, att. 18 200, sample volume 3.4 L.

chromatography [10]. In our experimental arrangement—thin adsorption tubes, small amount of adsorbent—Porapak Q is preferred. The slower desorption from Porapak results in poorer resolution in the chromatograms, but this was observed only for the most volatile compounds in petrol (Fig. 6).

Figure 7 shows an analysis of burnt petrol in wood. A small piece of wood was impregnated with petrol and ignited. After the fire was extinguished, the residue was placed in a can and the vapor phase analyzed using Porapak adsorbent tube. Note the sensitivity of the analysis—the desorbed amount corresponds to only a few nanolitres of petrol.

The hydrophobic nature of porous polymers is an important property in the forensic science examination of fire debris. The debris may often contain considerable amounts of water. Since water vapors pass the chromatographic columns packed with Tenax or Porapak Q between the ethane and propane peaks [12] they will not be retained in small adsorbent tubes at ambient temperature. Small quantities of water possibly present in the adsorbent tube do not disturb the analysis of hydrocarbons. We have used adsorbent tubes packed with porous polymers in connection with electron capture detection for the analysis of vapors from explosives without interferences from water.

Relatively common types of accelerants in Sweden are mixtures of alcohols and ketones, for

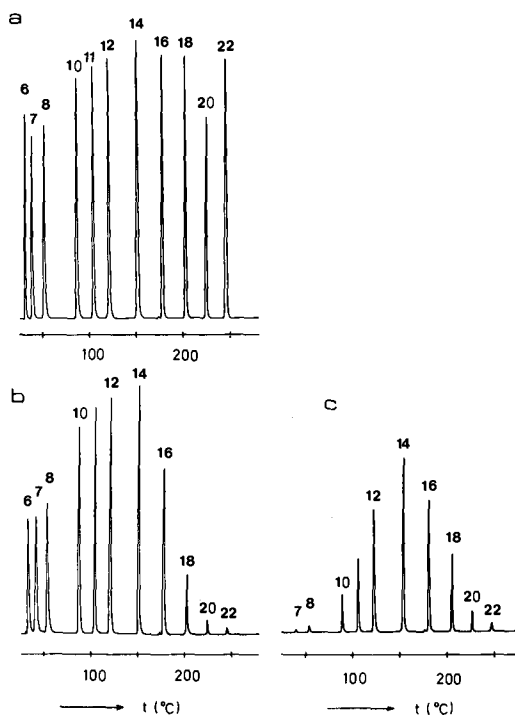


FIG. 4—The analysis of a mixture of *n*-alkanes sampled at elevated temperature. Tenax GC and Porapak Q tubes used in a parallel manner for 1 μ L of test mixture. The sampling temperature increased from 25 to 70°C measured at the lowest part of the sampling tubes. (a) 0.15 μ L of the test mixture injected, att. 12 800. (b) The desorption from Porapak Q, att. 12 800, sample volume 1.5 L. (c) The desorption from Tenax GC, att. 25 600, sample volume 3.4 L.

example, methanol, ethanol, methyl ethyl ketone, and so on. Porapak Q, the strongest adsorbent for alcohols among the porous polymers used in this study, was not able to retain methanol and ethanol vapors under the experimental conditions used here. In the homologous mixture of volatile alcohols, *n*-butanol was the first compound retained on Porapak Q to a significant extent. Cooling of the adsorbent would be necessary to detect the more volatile alcohols.

Adsorption Wires

The use of adsorption wires coated with active carbon is a very simple method. The adsorption is a "passive" diffusion process. The sensitivity is lower compared with that of porous polymers because of the small amount of adsorbent used and the limited time available for the adsorption of vapors. In this work we used several types of adsorption wires because of the differences in their adsorption and desorption properties.

Granular active carbon was found to retain compounds of very high volatility to a much higher degree than powdered carbon. Figure 8 shows the analysis of a test mixture of alcohols, ketones, and benzene. The sampling temperature was +9°C, thus below room temperature; the volume of the glass flask was 250 mL and the sampling time was 30 min. The amount of ethanol adsorbed on granular active carbon corresponds to about 40-mL headspace, that of methanol was lower—about 25-mL headspace. Powdered active carbon does not retain the

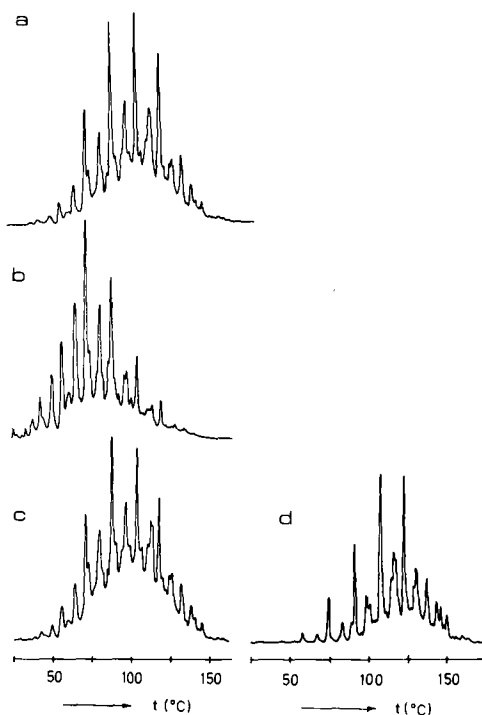


FIG. 5—The analysis of paraffin vapors with Tenax GC, Porapak Q, and by conventional headspace analysis at room temperature where (a) 0.4 μ L of paraffin oil injected, att. 12 800; (b) 2.5 mL of headspace injected, att. 640, 50- μ L sample; (c) the sample desorbed from Porapak Q, 1- μ L paraffin oil in a 250-mL can, sample volume 1.5 L, att. 25 600; and (d) the sample desorbed from Tenax GC, 1 μ L of sample, sample volume 3.4 L, att. 25 600.

volatile alcohols to the same extent. The adsorbed amount is, of course, dependent on temperature and sampling time, as well as the amount and kind of adsorbent.

The kinetics of the adsorption process for granular active carbon is shown in Fig. 9. It can be seen that the adsorbent is saturated by methanol already after 20 min of sampling at room temperature. The less volatile specimens are enriched with prolonged sampling time. The difference between powdered and granular active carbon in adsorption capacities for volatile compounds might depend predominately on differences in the amount of these adsorbents. The weight of three to four carbon granules was approximately 0.5 mg, while the weight of powdered carbon attached to the wires by adhesive was not known, but must be considerably less.

The presence of water glass as an adhesive influenced the adsorption characteristics of granular active carbon. The amount of ethanol and methanol adsorbed by the same amount of granular carbon without the adhesive was much lower than that in the presence of adhesive. There is reason to believe that this adhesive affects the adsorption characteristics of powdered carbon as well.

Using powdered active carbon the less volatile compounds will be adsorbed and thermally desorbed easier than using granular active carbon. By contrast, granular active carbon adsorbs the more volatile compounds better. In order to cover a wide range of volatilities, two sample collections with two different wires should be done. In that case, the first sampling is performed with powdered carbon wire followed by a second sampling with granular carbon wire which adsorbs the remaining volatile specimens in the sample. We have carried out some

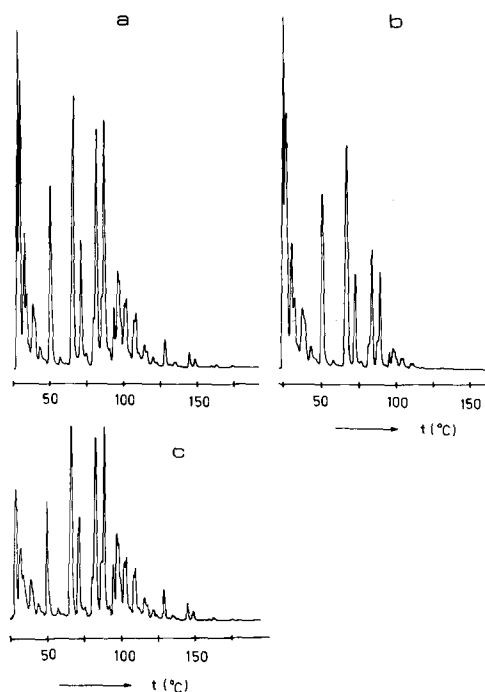


FIG. 6—The analysis of petrol vapors at room temperature where (a) $0.35 \mu\text{L}$ of petrol injected, att. 12 800; (b) 2 mL of headspace injected, att. 6 400, $50 \mu\text{L}$ of sample; and (c) the sample desorbed from Porapak Q, att. 12 800, $0.4 \mu\text{L}$ of sample, sample volume 0.9 L.

experiments of that kind—first sampling with powdered carbon at elevated temperatures followed by sampling with granular carbon at low temperatures (8 to 10°C). The sensitivity and the range of volatilities of these experiments were high but the method was rather time-consuming. To simplify the sampling procedure we also used single sampling with a combined adsorption wire (Fig. 2). The wire was found suitable for a wide range of volatilities and the resulting chromatograms resembled very much those of the original samples.

Figure 10 illustrates the analysis of petrol vapors using the three types of adsorption wires. The chromatogram obtained with the combined adsorption wire seems to be a superposition of those with powdered and granular active carbon. Depending on the amount of powdered and granular active carbon in the combined wire, the results varied from wire to wire to some extent, being somewhere between those for powdered and granular carbon wire, respectively. In order to adsorb and analyze less volatile hydrocarbons (C_{20} and higher) the accelerant vapors must be collected at higher temperatures. However, as the sampling temperature increases, the adsorption capacity of active carbon decreases quite significantly. Thus, it is desirable to find optimal conditions for sampling of unknown fire accelerant debris. It seems that a temperature of 100 to 110°C and a sampling time of 2 to 3 h give satisfactory results in most cases. At lower temperatures, for example, 80 to 90°C , the overall sensitivity is higher but the sampling time has to be increased to detect higher hydrocarbons. This is in good agreement with the results originally reported [4, 5], where 110°C and a sampling time of 2 h were found optimal for powdered carbon wires.

The analyses of vapors from diesel fuel and paraffin sampled at elevated temperatures ($\sim 105^\circ\text{C}$) are shown in Figs. 11 and 12. The maxima in the chromatograms obtained with powdered carbon wire are shifted to higher hydrocarbons in comparison with the original

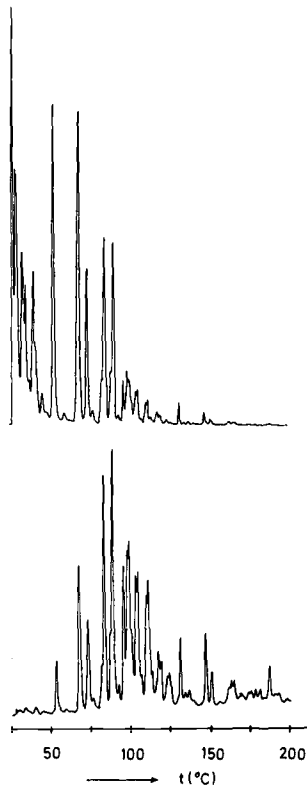


FIG. 7—The detection of burnt petrol in wood using adsorbent tubes packed with Porapak Q where the upper chromatogram is 0.25 μ L of petrol injected, att. 6 400 and lower chromatogram is burnt petrol desorbed from Porapak Q, att. 320, sample volume 1 L.

liquids. Also this agrees with the literature data [3,4]. Some small peaks, presumably representing pyrolysis products of the hydrocarbons, appear shortly after the injection. These peaks are the strongest for powdered carbon and the weakest for granular carbon.

The sensitivity of the analysis of petrol vapors sampled at elevated temperatures is decreased. However, the most volatile hydrocarbons in petrol may still be detected.

The presence of water in debris does not seem to affect the adsorption capacity of active carbon to a significant degree. Since the adhesive used for preparing the adsorption wires is water soluble, large quantities of water might destroy the wires mechanically, particularly those with powder active carbon. The presence of water vapors had some influence on the adsorption capacities of activated carbon for various compounds, but this phenomenon was not studied more closely in this work.

Discussion

The analysis of volatile hydrocarbons in fire debris using adsorbent tubes packed with porous polymers is a very sensitive method. The experimental arrangement described here is very simple; other arrangements have appeared in the literature [6-9]. The detection limits depend on several factors such as the type of accelerant, the material in the debris, the sampling temperature, the amount of adsorbent, and so forth, but may be of the order of 1 nL of accelerant if the accelerant is completely evaporated as in our laboratory experiments. In a

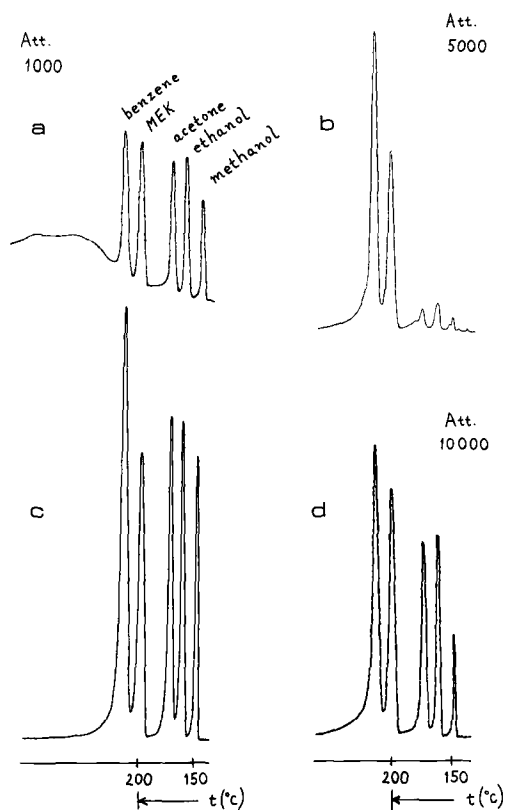


FIG. 8—Adsorption wire analysis of 0.8- μ L test mixture of alcohols, ketones, and benzene. Sampling time 30 min at 8°C where (a) 2.5 mL of headspace; (b) the sample desorbed from powdered active carbon; (c) original liquid (att. 50 000); and (d) the sample desorbed from granular active carbon.

real case, the detection limit is expected to be below 1 μ L of accelerant. Since the type and amount of accelerant encountered in casework is unknown, the signals from a gas chromatograph output should not be carried out using conventional recorders necessitating attenuation changes. Integrators of the type Hewlett-Packard 5880A Series Terminal or magnetic tape recorders [10] should be used for this purpose. With 30 to 60 mg of porous polymers as used in this study, the chromatographic detector may be overloaded if the amount of accelerant is large. The solution to this problem was proposed by Russell [10] who used small amounts of adsorbent (1.5 mg) and limited sampling volumes.

In our opinion, the analysis of fire accelerants in the forensic science laboratory should be started by using conventional headspace analysis. In a case when no or very weak signals are detected by this analysis, the debris can be analyzed as described in this text. Otherwise, only a fraction of the debris should be sampled, or tubes with small amounts of adsorbent and low sampling volumes should be employed.

There are some common disadvantages associated with porous polymers as adsorbents:

1. These materials are not absolutely stable against oxidation. Some degradation products thus may appear as additional peaks in the chromatograms, particularly at low attenuation values. This problem is more serious with Porapak Q than with Tenax. The adsorbent tubes must be stored in a refrigerator when not used.

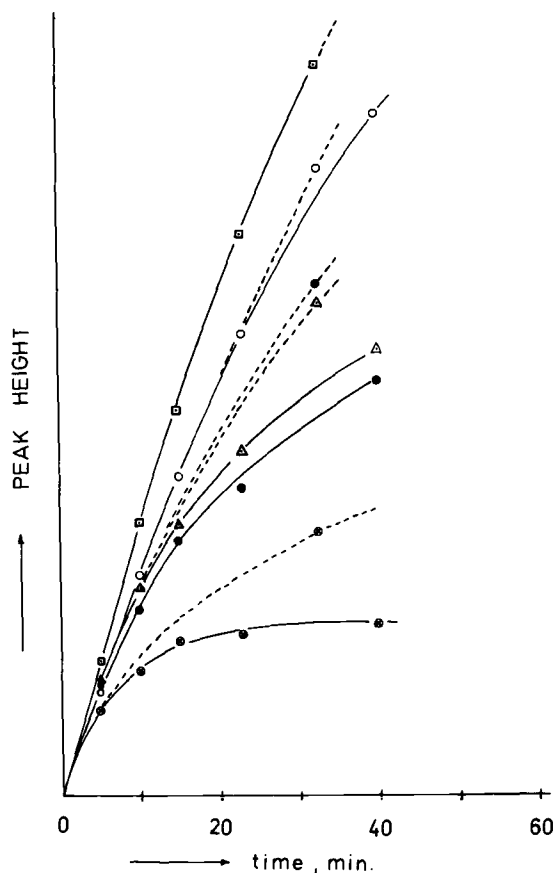


FIG. 9—The kinetics of the adsorption of the test mixture in Fig. 8. An adsorption wire with granular active carbon was used. Sampling at room temperature is a solid line and at 9°C is a dashed line. The latter sampling is based on only two sampling times thus the dashed lines are only meant for a rough comparison. (⊗)—methanol, (●)—ethanol, (Δ)—acetone, (○)—methyl ethyl ketone, (□)—benzene.

2. Porous polymers are not suitable for sampling of alcohol vapors that are too volatile and polar to be retained.

The use of adsorption wires is a very simple method suitable for forensic science laboratories who own a Curie point pyrolysis unit. The desorption of vapors from active carbon is surprisingly fast resulting in a high resolution [3, 4]. This technique always leaves some accelerant in the debris which generally implies lower sensitivity compared with porous polymer method. Nevertheless, the sensitivity is sufficiently large to detect accelerant quantities down to 1 μ L in such strong adsorbing medium as 100 g of charcoal [5]. Adsorption wires prepared by binding active carbon to Curie point pyrolysis wires can be used repeatedly for many analyses in similar manner as adsorbent tubes.

Alcohol vapors are detectable, but a realistic procedure implies two separate samplings—one at elevated temperatures followed, if necessary, by a second sampling with granular active carbon at low temperatures for the detection of, for example, alcohols. The container of course must be tight.

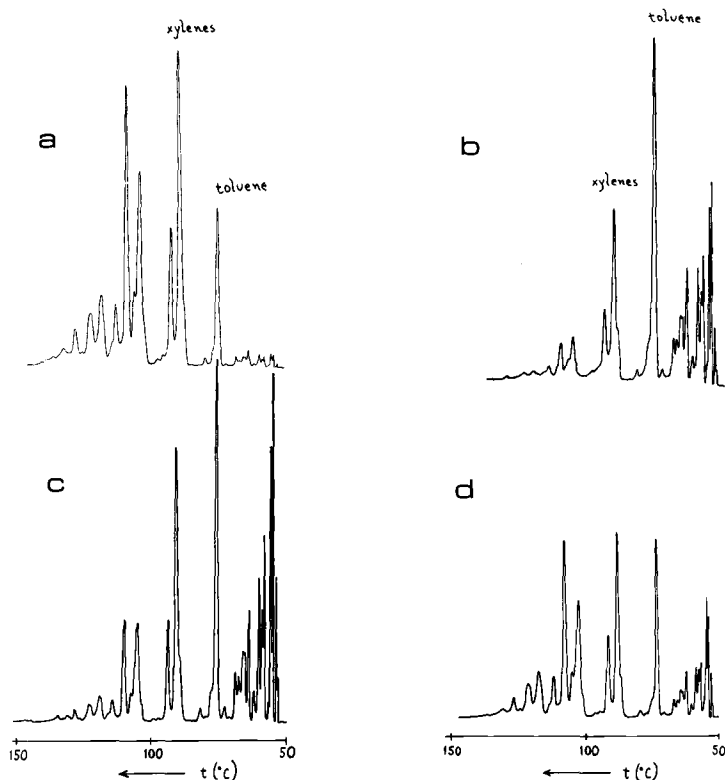


FIG. 10—The analysis of petrol vapors using the three types of adsorption wires. Filter paper impregnated with $0.5 \mu\text{L}$ of petrol was placed in a 250-mL glass flask. The sampling time was 1.5 h at room temperature where (a) powdered adsorption wire (att. 10 000); (b) granular adsorption wire (att. 20 000); (c) original liquid, $0.3 \mu\text{L}$ injected (att. 100 000); and (d) combined adsorption wire (att. 20 000).

Headspace analysis, in our opinion, is not necessary when adsorption wires are used. The risk for overloading the column is minimal, since the amount of adsorbent and the sampling capacity at elevated temperatures are low.

An important advantage of adsorption wires is the absence of oxidation and degradation products of the adsorbent. There is, however, some risk for pyrolytical degradation of the adsorbed sample, particularly for samples of very low volatility.

The mechanical properties of adsorption wires are not ideal. The wires are not robust and care must be taken not to destroy the sorbent while inserting and removing the wire from the pyrolysis unit and also in sampling. We had some problems with wires coated with powdered carbon—the sorbent sometimes flaked off during the first pyrolysis as a result of dehydration of the adhesive. The chromatographical column should be protected from dropped powder of activated carbon.

Thermal desorption from powdered active carbon is very rapid, a single 10-s pyrolytical pulse is sufficient to desorb most hydrocarbons to 95 to 99%. One 15-s pulse applied on granular carbon however desorbs only 90% or less of the higher hydrocarbons. Actually, we used two consecutive 10-s pulses for powdered and two consecutive 15-s pulses for granular and combined active carbon in this study. The compounds possibly not desorbed completely

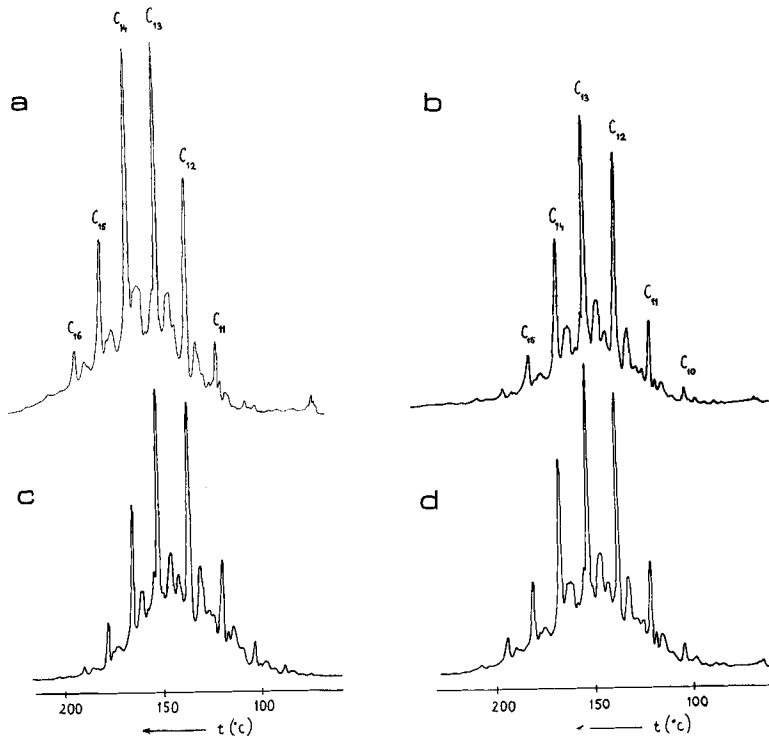


FIG. 11—The analysis of paraffin vapors using the three types of adsorption wires. The sampling time was 2.5 h at 105 to 110°C and the amount of paraffin was 0.5 μ L where (a) powdered wire (att. 5 000); (b) granular wire (att. 5 000); (b) granular wire (att. 20 000); (c) original liquid, 0.45 μ L injected (att. 100 000); and (d) combined wire (att. 10 000).

during this initial “pyrolysis” must be desorbed by applying several pyrolytical pulses after the completion of the analysis and before using the wire again.

It is advisable to store adsorption wires over freshly activated charcoal [2] but the wires must still be conditioned by several pyrolytical pulses before each analysis. The analysis of vapors adsorbed on adsorption wires is carried out immediately after sampling.

Adsorbent tubes packed with porous polymers are stored simply in a refrigerator (to minimize oxidation). After sampling, the tubes may be stored in a refrigerator again and analyzed on a later occasion. Adsorbent tubes may be mailed from one place to another without damage to the adsorbed sample. Thus, the sampling may be performed directly at the place of the fire.

Acknowledgment

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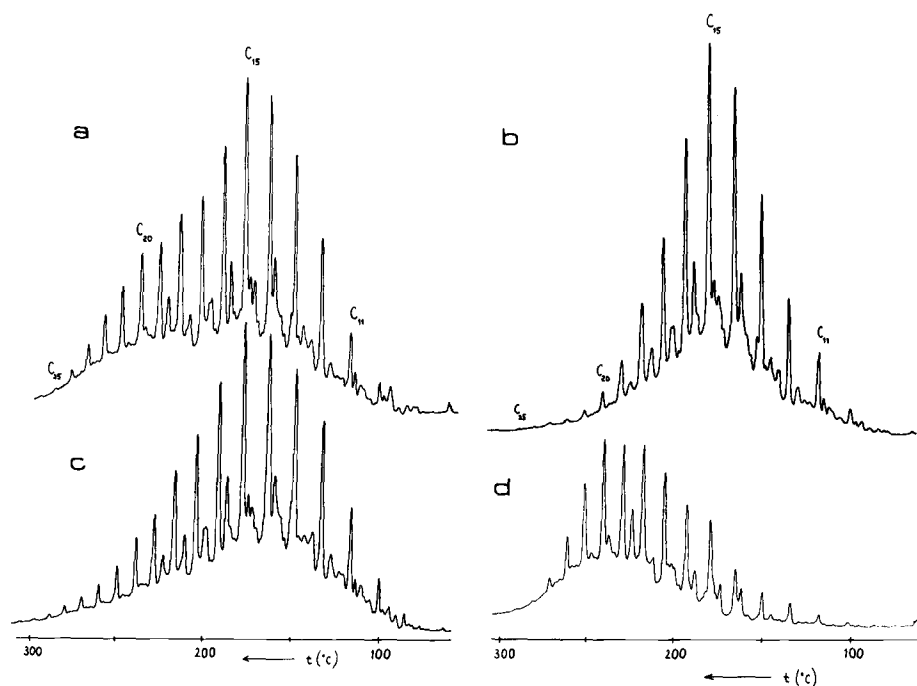


FIG. 12—The analysis of diesel fuel vapors using adsorption wires. The sampling time 2.5 h at 105 to 110°C with 1 μ L of diesel fuel where (a) combined wire (att. 5 000); (b) granular wire (att. 10 000); (c) original liquid, 0.5 μ L injected (att. 50 000); and (d) powdered wire (att. 5 000).

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