

Looking into some basic properties of accelerants in fire residues for higher performance in arson analysis

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Received: November 21, 2002/ Received in revised from: February 21, 2003/ Accepted: March 6, 2003

ABSTRACT

The success of an arson analysis relies largely on a sound sampling procedure. In addition to many technical aspects on the sample preparation itself, it is also of primary importance that fire scene investigators and arson analysts have a full understanding of the basic behaviors of exogenous flammable fluids so that a better sampling and hence a more successful analysis of the arson evidence is achieved. In this report, some previously unexplored basic physical aspects of accelerants in fire residues and their effects on the detectability of the added fuels are discussed based on a few simple experiments conducted on various accelerants and matrices.

Keywords: Arson Analysis, Accelerant, Fire Residue, Continuous Steam Micro-Distillation, Headspace Gas Chromatography

Introduction

For many years forensic scientists have found it very useful to identify exogenous fuels in fire residues to prove any incendiary element of arson. Because, very often, only trace quantities of accelerant are available for analysis and some complex matrix effects are involved in the analysis, it is necessary to perform pre-treatment or pre-concentration procedure prior to instrumental analysis.

Although a variety of sample preparation techniques, in an effort to easily and effectively separate accelerants from fire debris, have been extensively studied and promoted [1-3], few reports in the literature discuss the equally important basic properties of either the accelerant or the matrix. While engaged in arson-analysis related research work, we have often got the chance to participate in some fire scene investigations. In addition to the tremendous amount of care and the many skills required on the part of the arson investigator, several questions have often arisen from our mind. For instance,

if some exogenous flammable fluids have actually been added to start the fire, then what would be the marginal conditions under which the accelerants can still be detected? That is, in the sense of unambiguously detecting any concealed accelerants, how severe is a burning allowed to be? How long is a burning allowed to last for? And how long a time is allowed for a fire scene not searched or for samples not collected since the fire was put out? Moreover, should fire debris always be collected from dry areas or not at all should even totally aqueous specimens be ignored? All of these are extremely crucial to arson analysis, yet rarely scrutinized. Prompted by the saying of Dr. Henry C. Lee, an internationally recognized forensic scientist: "a competent criminalist has got to enrich his or her common sense, and that sometimes necessitates some simple experiments be conducted", we feel it informative to deduce via a short project some general rules or guides so that better sampling at the fire scene and hence better arson analysis in the laboratory can be performed.

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Experimental

Materials

Paraffins *n*-C6 through *n*-C26 (with purities ranging from 97 to 99.9%) used as the tested accelerant components and as reference standards for retention indices calculation were obtained from Merck Co. in Taiwan. For the latter use, a 0.2- μ L aliquot of CS₂ solution of the serial alkane homologues (ca. 0.001 v/v with respect to each alkane component) was routinely co-injected with the formal sample for the gas chromatography (GC) analysis. Carbon disulfide and ammonium sulfate (as a salting-out agent) were also purchased from Merck Co. in Taiwan, and were used without further purification. The 95 unleaded gasoline, kerosene and diesel used in this study were all from the Chinese petroleum Co.

Equipments

1. Continuous steam micro-distillation

The set-up used for the modified steam distillation is a variation on Dean-and-Stark apparatus and is mostly the same as those described by Stone et al. [4] and Twibell et al. [5]. It has also been previously presented in an international forum by us [3] except in this study a small metal can instead of a glass distilling flask was used to hold the sample and all the dimensions of the set-up were reduced (see below). The lid of the paint can was so punctured that it could be fitted with a bored-through union (i.e., Swagelok tube fitting), which, in turn, was connected through a segment of PTFE tube to a 24/40 ground-glass joint for performing the continuous steam micro-distillation. The bored-through union, ground-glass joint, micro-condenser (10 cm long), micro-distillation adapter (with the distillate collector column being 5 cm long), and even the cover of the paint can are all reusable after a standardized cleaning process.

2. Purge and trap/thermal desorption (P&T/TD)

A Supelco 1000/110 Dynamic Thermal Stripper fitted with a dry gas assisted carbotrap 300 adsorbent tube (packed with graphitized carbon black) was used for the purge-and-trap sample enrichment, and a Supelco 890 Thermal Tube Desorber interfaced with a gas chromatograph (described below) for the thermal-desorption sample introduction [6,7]. The latter step was performed without cryogenic focusing. The detailed operational procedure and conditions were the same as those previ-

ously described [8-10].

3. Gas chromatography

As has been previously described [3,8-13], an HNU Model 421 GC equipped with dual wide-bore capillary columns (SPB-1 and SPB-20, 30m \times 0.53mm ID, 1.5 μ m film) and dual FIDs, which offered two-fold retention data, was used for the detection and quasi-confirmation of the tested accelerants. The two chromatograms from the two columns were displayed simultaneously on a personal computer terminal. The software used for the direction of the analysis and the calculation of the retention indices (only for analytes more retained than *n*-hexane) was obtained from Shiunn Hua Co. Ltd., Taipei, Taiwan. Three temperature programs were used. PGM I (for volatile hydrophilic accelerants): inj., 250 °C; FID, 290 °C; oven, 35 °C (5 min) to 50 °C at 5 °C/min, 50 °C to 200 °C at 30 °C/min, hold 2 min. PGM II (for lower-boiling hydrophobic accelerants): inj., 280 °C; FID, 290 °C; oven, 35 °C (10 min) to 100 °C at 2 °C/min, hold 100 °C for 30 sec, 5 °C/min to 250 °C, hold 250 °C for 30 sec, 30 °C/min to 260 °C, hold 260 °C for 2.5 min. PGM III (for higher-boiling hydrophobic accelerants): inj., 300 °C; FID, 290 °C; oven, 70 °C (3 min) to 280 °C at 3 °C/min, hold 2 min. Carrier gas (N₂) in each column: ca. 10 mL/min; splitless injection. A dBase III Plus program was used to compile a retention indices data bank for more than 100 accelerant exemplars, fresh or weathered, pure organic solvent or mixture, petroleum-based or natural products. This program was also able to undertake searching for any matching target accelerant.

Volatility of hydrophobic accelerants

A 100- μ L portion each of authentic *n*-heptane through *n*-dodecane, fresh 95 unleaded gasoline, kerosene, diesel and turpentine, respectively, was put in a 3.7-mL vial (ca. 1.5 mm deep) and exposed to the open air at ambient temperatures. The remaining amount of the liquid after an exposure of 1 hr, 2 hrs, 3 hrs, 1 day, 2 days, etc, was visually checked or, when the residue was invisible, checked through CS₂ extraction followed by GC-FID analysis (PGM II; for detecting single-component normal alkanes, no paraffin references were co-injected).

Osmosis/adsorption/absorption/diffusion behaviors of hydrophobic accelerants in/on wood and concrete

A 10-mL portion each of fresh kerosene and diesel, respectively, was poured onto a polished surface of crushed-stone-concrete floor, the interior of which had been thought of to be more difficult than ordinary concrete floor for higher-boiling and hence more viscous fuels to run into. The osmosis action was closely observed. On the other hand, a 100- μL portion each of fresh kerosene and diesel, respectively, was dropped onto the surface of a piece of dry pinewood of ca. $5 \times 3 \times 3 \text{ cm}^3$. After the fuel had completely diffused into the interior of the pinewood (it took a few seconds only), the fuel-fortified pinewood was transferred into a 500-mL beaker containing 200 mL of water and pressed down below the water level. The water surface was then visually examined for any multicolor thin film that should be discernible even if only a trace amount of diesel or kerosene was spilled over the water surface. If no colorful film was seen, the whole thing was continuously shaken and examined again on the next day. The pinewood procedure was repeated using a 10- μL portion of gasoline and a small concrete debris (ca. 50 cm^3) instead. However, due to the less visibility of gasoline film, the resulting water and concrete debris this time were subjected to continuous steam micro-distillation followed by GC-FID analysis (PGM II) respectively.

The capabilities of continuous steam micro-distillation in recovering high-boiling hydrophobic accelerants

A 200- μL portion each of authentic normal decane and $n\text{-C}_{26}\text{H}_{54}$ (a relatively high-boiling component of diesel and the highest-boiling normal alkane as an authentic standard commercially available in Taiwan), respectively, was dropped onto the surface of a concrete debris of appropriate size. After the tested fluids had completely diffused into the interior of the debris, the fortified debris was transferred into a small new paint can and subjected to 0.5 hr (counted from the first drop of distillates) of continuous steam micro-distillation. The 1hr-recoveries of the tested fluids were calculated through the relevant GC-FID responses (PGM II for $n\text{-C}_{10}\text{H}_{22}$ and PGM III for $n\text{-C}_{26}\text{H}_{54}$ with no paraffin references co-injected).

The capabilities of shaken-headspace GC-FID in detecting volatile hydrophilic accelerants in water

The serial calibrator solutions used in this part of study contained 10, 5, 3, 2, 1, 0.5, 0.3, 0.2, 0.1, 0.05, 0.

03, 0.02, and 0.01 ppm of methanol/ethanol/acetone trinary analytes in 100 mL of water. A 250-mL Erlenmeyer flask containing an appropriate aqueous calibrator was stoppered with a serum stopper and vigorously shaken for 1 min. A 5- μL portion of the headspace (HS) vapor was then injected onto the dual-column GC-FID (PGM I) without cryogenic focusing. The limit of detection (LOD) was defined as the analyte concentration giving a peak in the GC-FID chromatogram with peak height equal to the mean + $N \times$ standard deviation, where $N = 3$. The mean is the measured average of noises taken from a baseline region located far away from the analyte peak using a 1-ppm fortified sample. Accordingly, the standard deviation is the measured fluctuations of the noises. The chromatographic peak shapes and resolution of the analytes were also evaluated.

Simulated tests directed towards the foregoing properties

A 50-mL portion of gasoline was poured into a 7-cm thick concrete bowl self-made from appropriate amounts of Portland Type I cement and fine aggregate. The gasoline was allowed for 2 min to diffuse into the interior of the concrete. A fire was then started and allowed to burn until it naturally went out. The hot concrete bowl was placed in an unsealed furnace and further heated at 1000°C for 30 min. Having been sufficiently flushed with water, the cooled concrete bowl with the residual water in it was exposed to the open air at ambient temperatures for 3 days. Two identical portions (ca. 50 cm^3 each) of the weathered concrete were then taken from the fortified spot and subjected to continuous steam micro-distillation GC-FID and P&T/TD GC-FID analyses (PGM II), respectively. The same procedure was repeated except that 50 mL of ethanol instead of gasoline was used and that two ca. 50-cm^3 portions of the resulting concrete together with the recovered water were submitted to the continuous steam micro-distillation GC-FID and P&T/TD GC-FID analyses (PGM I), respectively.

Results and Discussion

Volatility of hydrophobic accelerants

With an initial depth of 1.5 mm, pure n -heptane (bp 98°C) and n -octane (bp 126°C) evaporated to dryness

within a day in the open air at ambient temperatures. Pure *n*-nonane (bp 151°C) under the same conditions took 2 days to evaporate; *n*-decane (bp 174°C) took 8 days; *n*-undecane (bp 196°C) took 23 days. In contrast, the tested pure *n*-dodecane (bp 216°C) remained visually discernible after 2 months' exposure and instrumentally (GC-FID) detectable after 3 months'. As for gasoline, kerosene, diesel and turpentine, it was generally safe to say that all but none remained detectable after at least 1 week's exposure to the open air at ambient temperatures. In fact, the reduction in the amounts of kerosene and diesel under these conditions could not be visually appreciated. As a parallel test, 50 mL of fresh diesel was also submitted to a rotatory evaporator and evaporated at 90°C and 5 mm-Hg for 3 hrs; its amount remained almost unchanged. Moreover, our experience from the preparation of 50% evaporated kerosene and 50% evaporated diesel exemplars by vigorously stirring 100 mL each of the fuels in a ventilation hood indicated that the preparation of the former took ca. 3 days and the latter ca. 6 days. In sharp contrast, when 10 µL of gasoline was exposed to the open air at ambient temperatures for just 3 hrs, only a few originally unseen or weak peaks stemming from the higher-boiling components, particularly biannular aromatics such as naphthalene, 2-methylnaphthalene, etc., were left in the GC-FID chromatogram with increased relative peak heights. Besides, the preparation of 95% evaporated gasoline in a ventilation hood took only about 16 hrs. A valuable semi-empirical rule that should be pointed out collaterally here states that aromatic hydrocarbons are usually more heat-resistant than their aliphatic counterparts. The appropriate dual-column GC chromatograms with retention indices labeled have been previously reported for fresh gasoline, kerosene, diesel and turpentine [3], and those for the partially evaporated counterparts will be reported elsewhere.

Since most of the hydrophobic accelerant components (either petroleum-based or non-petroleum-based) do not evaporate so rapidly as was supposed to and a major part of them may somehow be protected from the fire and heat by the collapsed structural materials benefiting from their extensive diffusion prior to and during the fire as will be demonstrated below, the arson analyst consistently holds a high chance to identify them even though the fire is terribly severe and even though the sampling time is relatively late so long as a sensitive means (such as employing a well trained canine) can be used to pinpoint the exact spot where any accelerants

have been intentionally added by the arsonist.

Osmosis/adsorption/absorption/diffusion behaviors of hydrophobic accelerants in/on wood and concrete

Although diesel and kerosene have been demonstrated to evaporate quite slowly under natural conditions, the pouring of 10 mL of each of them onto a polished crushed-stone-concrete surface resulted in only obscure trace within a minute. This implied most of the fuel had diffused into the interior of the polished floor which had been thought of to be more difficult than ordinary concrete floor for higher-boiling and hence more viscous fuels to run into. While pinewood itself was subject to the buoyancy of water, no fuels that had run into the interior of the pinewood had been appreciated to escape from the forcefully drowned and shaken pinewood and float up again. As the tested pinewood was replaced by concrete, most of the fuel would still remain absorbed in the interior of the concrete, although a little of it would float up. Theoretically, the above stated osmosis/adsorption/absorption/diffusion behaviors should hold true for all types of hydrophobic accelerants but only partially true for hydrophilic accelerants, the latter being also influenced by solvation (hydration) effect. Thus, it becomes apparent that, provided such water-compatible sample preparation methods as continuous steam micro-distillation and various dynamic or static adsorption/desorption techniques using water-compatible adsorbents are employed [1,6-10], water not only will not interfere with the following instrumental analysis, but also will retard the evaporation of the accelerants (either hydrophobic or hydrophilic) under or in it prior to fire scene processing. It strongly suggests the arson investigator not ignore any moistured area when collecting the evidence at the fire scene. What is equally crucial is how to sensitively detect any presence of water-shielded or water-dissolved accelerants at the scene.

The capabilities of continuous steam micro-distillation in recovering high-boiling hydrophobic accelerants

Batch steam distillation has long been dismissed from the arson analysis community as cumbersome and lengthy [1]. In our laboratory, however, a modified continuous steam distillation apparatus operated in micro- or semimicro-scale has proved efficient, reliable, robust, convenient and economical, particularly when

the volume of samples is large [3]. Starting with 200 μL of $n\text{-C}_{10}\text{H}_{22}$ spiked in a concrete debris, the proposed continuous steam micro-distillation afforded an averaged recovery of $75 \pm 3\%$ ($N = 3$) after 0.5 hr of steaming. More encouragingly, 200 μL of the higher-boiling $n\text{-C}_{26}\text{H}_{54}$ achieved an averaged 0.5-hr recovery of $61 \pm 4\%$ ($N = 3$). The losses of 25% and 39% were mostly attributed to the adherence of the distillates to the distillation line. In our experience, several favorable comments can be made on this modified steam distillation: 1) while easy to operate, it is not so time consuming as supposed, even for a high-boiling hydrocarbon analyte and even without the appeal for a higher-boiling co-distillation solvent such as ethylene glycol; 2) unlike some adsorption/desorption techniques that may suffer from such varying factors as adsorption/desorption efficiencies and moisture interference, the present method always achieves reproducibly fair 0.5-hr recoveries for high-boiling hydrocarbons; 3) the absolute detection limit for diesel (20 μL , obtained by taking up the upper-layered distillates with 1 mL of pentane followed by concentration to 20 μL and then a 1- μL injection onto GC-FID) may be a little poorer than those achieved by active adsorption techniques (said of the total of all components; actually, "better" for high-boiling hydrocarbons), but is better than or comparable with those obtained using passive adsorption techniques; 4) most of the time, the distillates are visible and collectable, and can be presented in court as real evidence.

In practice, arson analysts are frequently faced with interference from the sample matrix. An extensive study on the capabilities of retention-index-comparison assisted P&T dual-capillary-column GC in surviving various matrix interferences has been conducted in our laboratory [9], and another study on the continuous steam micro-distillation counterpart is almost through. Taking pinewood, one of the most well known interfering matrices, as an example, without further refinement process it will produce only tolerable amounts of background material after 0.5 hr of the modified steam distillation, and this background material will not interfere with the following GC-based instrumental analysis seriously provided a specific profiling method such as target compound GC-MS [14-16] or dual-column GC assisted with retention-indices comparison [3,8-13] is employed.

It should also be pointed out that the proper use of joints and/or fittings can make the continuous steam

micro-distillation adaptable to different types of distillation vessels (e.g., paint can, distilling flask, etc.), compatible with other methods chosen for the screening test (e.g., cold or hot headspace GC; in this case, the bored-through union should be replaced by a septum fitting) or sample preparation (e.g., dynamic or static adsorption/desorption), and versatile to various forms of specimens (e.g., solid and/or aqueous; hydrophilic and/or hydrophobic).

The capabilities of shaken-headspace GC-FID in detecting volatile hydrophilic accelerants in water

Despite its relatively lower sensitivity, static headspace GC (HS-GC) has long been recognized as a convenient technique in screening arson suspect specimens. However, this is said of an essentially incomplete arson analysis, i.e., an analysis directed towards only hydrophobic accelerants and solid fire debris. As a matter of fact, the possibility for an arsonist to employ such volatile hydrophilic accelerants as methanol, ethanol and acetone can never be eliminated. Moreover, the post-fire scene may be moistured or flooded due to the fire-fighting process. To make the analytical scheme as general, versatile and systematic as possible, a comparative study was conducted to see how HS-GC would work with volatile hydrophilic accelerants in water. Listed in table 1 are the limits of detection (LODs) resulting from the serial analyses of methanol/ethanol/acetone trinary analytes in 100 mL of water using: 1) shaken HS-GC-FID (5- μL injection); 2) direct injection of aqueous aliquot onto GC-FID [17,18] (1- μL injection); 3) P&T/TD GC-FID (total-amount analysis). Although P&T/TD GC-FID achieved the lowest LODs benefiting by a virtually total-amount analysis, it was far more costly and complicated than the other two approaches. The direct injection of aqueous aliquot onto GC-FID not only led to the poorest sensitivities but also resulted in the most serious peak-tailing no matter what kinds of columns were used. In this study, the use of non-polar SPB-1 and intermediate-polarity SPB-20 capillary columns would even see intolerable peak-broadening if more than 1 μL of aqueous aliquot was directly injected. It was the shaken HS-GC-FID approach that, while simple, rapid, and inexpensive, would afford acceptable LODs and chromatographic resolution as well. Besides the benefit from a larger injection capacity (5- μL headspace vapor vs. 1- μL aqueous aliquot), the addition of an excess amount of ammonium sulfate (as a salting-out

agent) and the shaking of the sampling flask should have facilitated the release of analytes from the aqueous matrix to the headspace, thereby making the headspace supersaturated and giving enhanced sensitivities compared to the situations in directly injecting aqueous aliquots. Moreover, the corresponding peaks were adequately resolved with only inappreciable peak-tailing: on SPB-1 column, the retention times of ethanol, ethanol and acetone (their retention indices were unavailable as they were even less retained than the least retained paraffin

reference, n-hexane) were 1.982, 2.326 and 2.484 min, respectively, and those on SPB-20 column, 2.098, 2.453 and 2.544 min, respectively. The order of elution and resolution of peaks were in agreement with those previously proposed by other researchers; namely, highly polar organic compounds like alcohols can be effectively separated using non-polar SPB-1 and intermediate-polarity SPB-20 capillary columns, where the analytes' relative retention was mainly determined by the boiling points [18].

Table 1 Limits of detection (LODs; in ppm) for the GC-FID analyses of methanol, ethanol and acetone in 100 mL of water using three sample preparation methods.

Accelerant	Shaken HS-GC-FID (5- μ L injection)	Direct injection of aqueous aliquot (1 μ L) onto GC-FID	P&T/TD GC-FID (total analysis)
Methanol	0.3	3	0.02
Ethanol	0.2	2	0.03
Acetone	0.5	1	0.05

In a supplementary experiment, a comparison was made between the peak heights obtained upon the shaken HS-GC-FID analyses of 1) 5 μ L of vapor taken from the HS of an undistilled spike containing 1 ppm each of methanol, ethanol and acetone in 100 mL of water, and 2) 5 μ L of vapor taken from the HS of a 2-mL aliquot of lower-layered aqueous distillates after 0.5 hr of continuous steam micro-distillation of the above spike. No significant differences in the concentrations of the relevant analytes were found caused by the continuous steam micro-distillation. This finding implied that, in order to make their HS-GC screening more compatible with the whole analytical scheme and their identification more trivial (i.e., less cross-interference between hydrophilic and hydrophobic accelerants), any hydrophilic accelerant containing suspect might as well be handled by firstly running a continuous steam micro-distillation followed by the shaken HS or P&T analysis of the aqueous distillates.

Simulated tests directed towards the foregoing properties

Putting aside the equally important issue of matrix interference with GC analysis, the emphasis of this part of study was placed on the previously unvalidated heat-resistant potential of accelerants co-contributed by the foregoing properties. In this context, only an oversimplified concrete matrix fortified with gasoline, the most common and relatively volatile multi-component accelerant, was under test. Nevertheless, the whole course of the thermal exposure of the gasoline-fortified concrete bowl has been assessed to be realistic enough or even more severe than that at a real fire scene. The outcomes have been encouraging. Both the continuous steam micro-distillation and P&T/TD approaches resulted in GC chromatograms whose respective dual-column retention indices and overall chromatographic patterns were well matched with those of our previously

established 97 % evaporated gasoline standard [8]. Displayed in Fig.1. is the SPB-20 chromatogram resulting from the P&T/TD GC-FID analysis showing an all-matched retention index set and a typical pattern of 97 % evaporated gasoline. It should be noted that the P&T/TD approach have caused the theoretically equal-height paraffin reference peaks to step down progressively in the higher-boiling region, whereas the continuous steam

micro-distillation has not. After all, this simulated test comes to an optimistic conclusion that a high chance always exists for any exogenous petroleum-based accelerants to be detected after a severe fire. Unfortunately, the same thermal exposure conditions tested with 50 mL of ethanol did not lead to positive results, presumably because ethanol was too volatile.

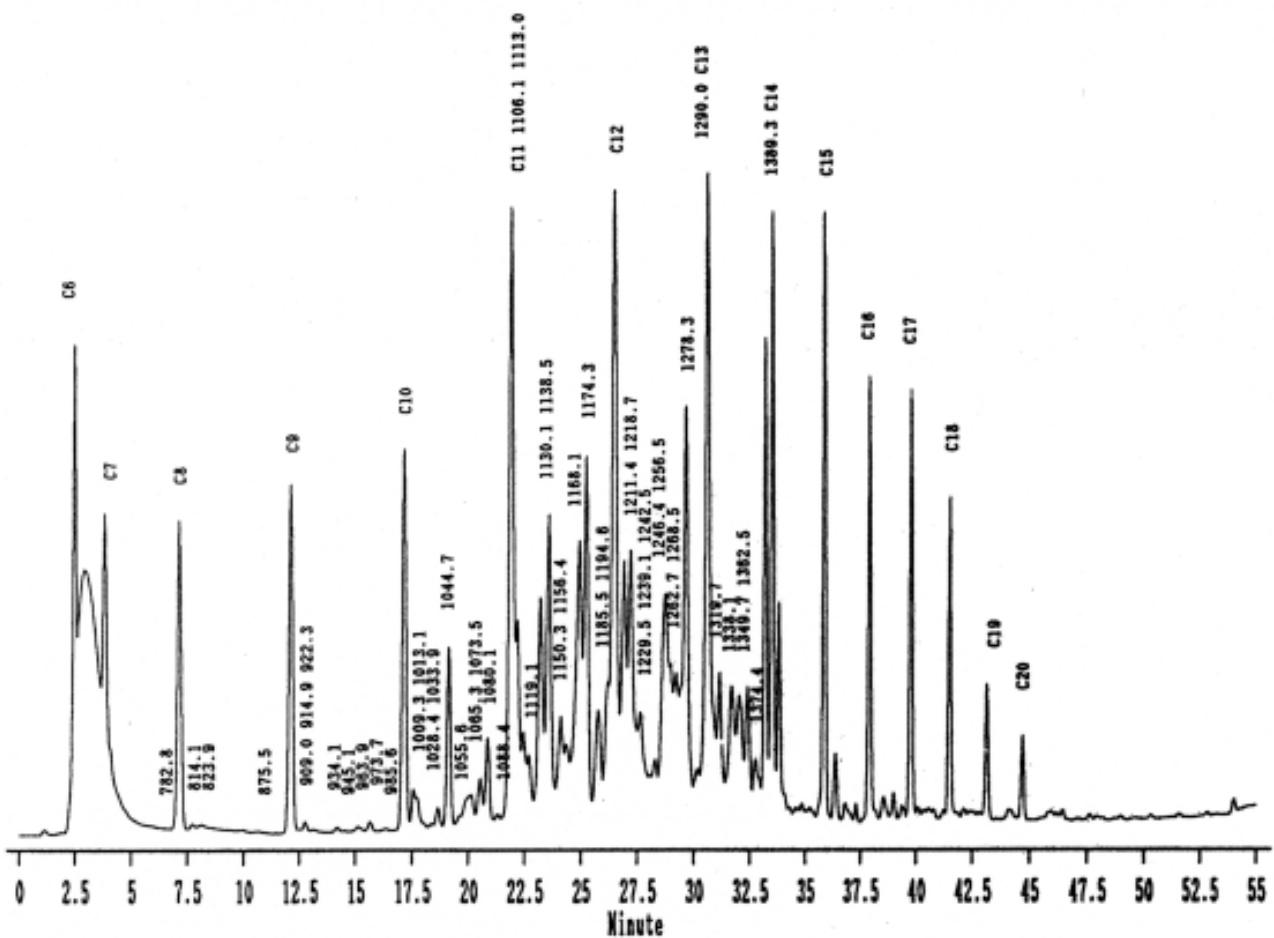


Fig.1 The SPB-20 column chromatogram resulting from a severe simulated heating of a gasoline-fortified concrete bowl. Also designated are the retention indices for the reference paraffins, i.e., Cn, and those well matched with 97% evaporated gasoline standard. Sample preparation: P&T/TD (see text). Dual-column GC conditions: inj., 280°C; dual FIDs, 290°C; oven, 35°C (10 min) to 100°C at 2°C/min, hold 100°C for 30 sec, 5°C/min to 250°C, hold 250°C for 30 sec, 30°C/min to 260°C, hold 260°C for 2.5 min. Carrier gas (N₂) in each column: 10 mL/min; splitless injection.

Conclusions

The results of this study have demonstrated that the analysis of incendiary liquids in complex matrices after a fierce fire is generally optimistic. A full understanding of the above explored properties or issues should be helpful for the arson investigator, while confident, to perform better sampling at the fire scene and better analysis in the laboratory.

Acknowledgment

The authors wish to thank the Super Chroma Enterprise Ltd., Taipei, Taiwan, ROC, for its kind assistance in performing various fire debris preparation techniques, which made this study possible. A financial support under the grant No. NSC-89-2113-M-015-001 from the National Science Council of the Republic of China is gratefully acknowledged.

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