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A Study of the Effects of a Micelle Encapsulator Fire Suppression Agent on Dynamic Headspace Analysis of Fire Debris Samples*

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ABSTRACT: The effects of a Micelle Encapsulator Fire Suppression Agent (F-500, Hazard Control Technologies Inc., Fayetteville, Georgia) on the routine analysis of fire debris samples by Gas Chromatography (GC) were studied. When mixed with water the product can be used in the suppression of Class A and Class B fires. Laboratory tests were performed to determine whether or not the product has any effect on the analysis for ignitable liquids by GC, in particular for gasoline, medium petroleum distillates, and heavy petroleum distillates. Test burns were suppressed using either the micelle encapsulator or water and samples collected from these burns were analyzed. The results of analysis show that use of the micelle encapsulator at a fire scene may affect the chromatographic data obtained from samples collected by the investigator. However, the effect does not prevent the identification of common ignitable liquids in fire debris samples.

KEYWORDS: forensic science, fire debris, ignitable liquids, gas chromatography-mass spectrometry, fire suppression agent, F-500, micelle encapsulator

It is well recognized in the field of fire investigation that suppression and the suppression agents used by the fire service may affect the subsequent fire scene investigation and the associated laboratory analyses (1,2). Although widespread use of water in fire suppression does not adversely affect typical fire debris analytical schemes, it is important to remain aware of new developments in fire suppression agents and the possible effects of such agents on the routine analysis of fire debris samples in the forensic laboratory.

The identification of ignitable liquids using pattern recognition following separation of the components by gas chromatography is well established (3). Ignitable liquids produce characteristic patterns under fixed conditions that permit their classification (4). However this identification process has become more complex due to the variety of commercial specialty solvents that are now available to the consumer (5). These solvents produce different, often less complex, patterns than gasoline or the traditional distillates, making them more difficult to interpret in the presence of extraneous peaks.

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Burned samples collected at fire scenes present further difficulties for the analyst in the forensic laboratory. Exposure to the heat of a fire can lead to evaporation of ignitable liquids or weathering may occur over time. The chromatogram generated from an evaporated or weathered ignitable liquid may be very different from that of the original liquid. Interference peaks from the sample matrix contribute to the chromatographic results. Burned substrate materials found at fire scenes can produce some of the same volatile compounds as those found in ignitable liquids.

Another source of potential interference that must be considered is the type of suppression agent used to extinguish the fire. Specific casework at this laboratory has required the evaluation of some commercially marketed fire suppression products, such as foams and wetting agents, to determine whether they might affect data interpretation in fire debris analysis. Most recently, a product called F-500, described as a "Micelle Encapsulator Fire Suppression Agent," has been finding use in Ontario due to its claims of overall speed and effectiveness of suppression as well as its cooling properties, low toxicity, and ability to reduce the likelihood of re-ignition. Its mechanism of action is described as differing from conventional wetting agents and foams in that it forms micelles around the fuel "in both the liquid and vapor state rendering it nonflammable and inert" (6).

Recommended concentrations of F-500 in water are 1% for Class A fires and 3% or 6% for Class B fires (7). The product is added directly to the booster tank of the fire truck or introduced into the handline using a standard in-line eductor. Due to its claimed mechanism of action, it is reasonable to explore whether this encapsulation of liquid and gaseous fuel would be of particular relevance to dynamic headspace analysis of fire debris samples for the presence of volatile ignitable liquids.

This study was undertaken to determine whether the use of F-500 as a suppression agent would result in a decrease of ignitable liquid vapors from fire debris samples, and whether it would interfere with the associated ignitable liquid patterns.

Laboratory tests were undertaken to compare the detection of headspace vapors at room temperature from three common ignitable liquids, gasoline, medium petroleum distillate (MPD), and heavy petroleum distillate (HPD), in F-500 solution and in water. An examination of the effect of increasing volumes of F-500 on the available headspace vapors of gasoline standards was performed.

Field tests were conducted using F-500 or water as the suppression agent on small-scale and large-scale fires, which were set either with or without an ignitable liquid accelerant. Debris samples from these tests were subjected to the routine analytical scheme employed at this laboratory.

Experimental

The analysis of fire debris samples at the Centre of Forensic Sciences involves dynamic headspace sampling using tubes packed with Tenax adsorbent (8).

Samples are subjected to a two-stage thermal desorption process using a Perkin-Elmer Automated Thermal Desorption Unit (ATD-400) as a means of introducing the sample onto the analytical column. Tenax is a hydrophobic porous polymer that preferentially adsorbs organic vapors. Therefore, the widespread use of water in fire suppression does not have a significant negative impact on fire debris analysis using this adsorbent.

Instrumentation

Headspace samples were adsorbed onto 80mg of Tenax TA packed in stainless steel sampling tubes which were then subjected to a two-stage thermal desorption process using an Automated Thermal Desorption Unit (ATD-400, Perkin-Elmer, Norwalk, CT). Sample desorption was carried out at 300°C for 25 min. Sample components were swept onto a cold trap packed with Tenax TA, which was held at −30°C. The cold trap was then flash heated to 300°C, to transfer the components to the analytical column. The fused silica transfer line was held at 200°C.

The ATD-400 was coupled to an Auto System Gas Chromatograph fitted with a Flame Ionization Detector (Perkin-Elmer, Norwalk, CT). Mass spectral data was obtained using an ATD-400 coupled to a Saturn 2000 Ion Trap Mass Spectrometer (Varian Analytical Instruments, Walnut Creek, CA).

Chromatographic separations were achieved using DB-1 and DB-1MS capillary columns, 30 m, 0.25 mm ID, and 0.25 μm film thickness (J & W Scientific, Folsom, CA).

The gas chromatograph (GC) oven was programmed with an initial temperature of 50°C, which was maintained for 8 min. The temperature was then increased to 280°C at a rate of 10°C/min and held for 2 min. Helium was used as the carrier gas at a flow rate of 1 mL/min. The flame ionization detector temperature was set at 300°C. The mass spectrometer was operated in Electron Ionization mode using Automatic Gain Control. Spectra were collected in full scan mode m/z 40 to 350 at a trap temperature of 140°C.

Instrument performance was checked for all sample runs using a test mixture including n-alkanes (C6–C20) as specified for the separation requirements of the ASTM Standards. The mass spectrometer was tuned and calibrated using Perfluorotributylamine (PFTBA, FC-43).

Samples and Sampling Procedures

The supply of F-500 used for this study was obtained from MICELL Fire Systems Ltd., Markham, Ontario, Canada (Lot numbers 0220,0229, 0469). A 1% concentration of F-500 in water was used throughout the study.

Test burn material was allowed to become fully involved before extinguishment with water or 1% F-500. Samples were then collected and stored in 1-liter mason jars with rubber sealed metal lids, the recommended packaging for fire scene debris samples submitted to this laboratory.

All standards were prepared in 1-liter glass mason jars. Gasoline, MPD (Varsol®), HPD (Diesel Fuel) and isoparaffin product (Royal Oak® Charcoal Lighter) were purchased from local commercial outlets and used as standards in the laboratory tests or as accelerants in the test burns.

Headspace samples were taken at room temperature by punching a hole in the jar lid and drawing vapors through the sample tube using a 50cc plastic syringe. Heated headspace vapors were sampled after oven heating the jars at 130°C for a minimum of 30 min. Fire debris samples are heated at this laboratory to increase the headspace vapor concentration of any ignitable liquid that may be present (9). Identification of ignitable liquid patterns was made using the criteria set out in the ASTM Standard Test Methods E 1387-95 and E1618–97 (4).

Laboratory Tests

Lab Test A—To investigate the possibility that F-500, as a "micelle encapsulator fire suppression agent", may reduce hydrocarbon vapors in the sample headspace 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8 and 1.0 microliters of gasoline, MPD and HPD standards were added to 10 mL of water or 1% F-500 in water. Duplicate sets of these ignitable liquid standards, and blanks containing only 10 mL of water or 10mL of 1% F-500, were prepared and analyzed at room temperature (Table 1).

Lab Test B—The effect of increasing the volume of 1% F-500 on the detection of gasoline was studied. Duplicate standards containing 1.0 μL and 10.0 μL of gasoline were prepared in 20, 50, and 100 mL volumes of water and 1% F-500 and analyzed at room temperature.

Fire Test Burns

Five separate test burns (A to E) were performed at various venues over a seven-month period. From these burns, 59 debris samples were collected from tests in which an ignitable liquid was used to accelerate the fire and 30 samples were taken from tests in which no ignitable liquid was used. Samples were collected and analyzed in the same manner as casework items.

Fire Test A—This test involved burning straw and wood without the use of an ignitable liquid, followed by suppression with 1% F-500. Seven samples, including comparison samples of wood and straw, were collected and analyzed.

Fire Test B—A large two-storied house was burned without the use of an ignitable liquid. Two separate upstairs bedroom fires

TABLE 1—*Lab Test A: Comparison of the effects of 1% F-500 versus water on detection of ignitable liquid standards prepared in the laboratory.*

Standard Volume (μL)	Gasoline Pattern Identified		Medium Petroleum Distillate Pattern Identified		Heavy Petroleum Distillate Pattern Identified	
	Water	1% F-500	Water	1% F-500	Water	1% F-500
0.1	yes	yes	yes	yes	no	no
0.2	yes	yes	yes	yes	no	no
0.3	yes	yes	yes	yes	yes	yes
0.4	yes	yes	yes	yes	yes	yes
0.5	yes	yes	yes	yes	yes	yes
0.6	yes	yes	yes	yes	yes	yes
0.8	yes	yes	yes	yes	yes	yes
1.0	yes	yes	yes	yes	yes	yes

were suppressed using 1% F-500. Twenty-three samples were collected and analyzed.

Fire Test C—These accelerated burns involved straw and wood and suppression with 1% F-500. Three samples from a gasoline burn and three samples from a HPD burn were collected and analyzed.

Fire Test D—This series of six test burns was designed to compare the effects of water versus 1% F-500 when used in suppression at a fire scene. To prevent cross-contamination the burns were carried out in different rooms in a Fire Training Tower. In each burn, four-liter volumes of ignitable liquid (gasoline, MPD or HPD) were poured over bales of straw on wood pallets. The use of four liters of accelerant ensured that a range of positive results would be obtained from a representative number of samples. A total of 36 samples, six from each burn, were collected and analyzed.

Fire Test E—This test compared the effects of water versus 1% F-500. Two burns of straw on wood skids were performed using one liter of an isoparaffin product as an accelerant. One fire was extinguished using water and a comparison fire was extinguished using 1% F-500. Six samples from each burn were collected and analyzed.

Fire Test F—Two vehicles were burned and the fires suppressed using 1% F-500. Gasoline was used in one burn and an isoparaffin in the other. Five samples were collected for analysis.

Results and Discussion

Four major peaks were observed in the chromatogram generated by the F-500 standard (Fig.1). These were identified by retention time and mass spectra as n-octanol, 2-ethylhexanoic acid, dodecane, and n-decanol in order of elution. The F-500 peaks elute between n-C10 and n-C14 and, as such, may interfere with common ignitable liquid patterns spanning this region, e.g., gasoline, MPD, and HPD. The intensity of the F-500 peaks increased after heating in a similar manner to those of ignitable liquids.

Laboratory Tests

Lab Test A—No significant differences in patterns or peak intensities were observed in chromatograms obtained from room temperature headspace samples for the three classes of ignitable liquids tested when mixed with 1% F-500 as compared to those mixed with water. The F-500 peaks were strong in all the F-500 standard patterns and were the dominant peaks in the chromatograms of the 0.1 to 0.4 μL range of standards. Figure 2 illustrates typical patterns obtained for gasoline and MPD standards in water and in F-500.

Lab Test B—There were no appreciable differences observed in the pattern intensities of gasoline standards prepared in 20, 50, or 100mL volumes of 1% F-500 or water.

Fire Test Burns

Fire Test A: *Wood and Straw, No Ignitable Liquids, Suppression with 1% F-500*—F-500 components were detected in all five debris samples when analyzed at room temperature. The F-500 peaks were more intense than the background substrate peaks in two of the five samples and of similar intensity in the remaining three. A sample positive for F-500 at room temperature was heated and analyzed. This resulted in an approximately fifteen-fold increase in the F-500 pattern intensity without a corresponding increase in the intensity of the background peaks.

Fire Test B: House Fire, No Ignitable Liquids, Suppression with 1% F-500—No F-500 components were detected in the room temperature analysis of the 23 bedroom debris samples; however, F-500 components were detected in twelve of the samples after heating. A representative sample chromatogram is shown in Fig. 3. The F-500 peaks were dominant in the patterns of three of the twelve heated samples and were of similar intensity to the background peaks in the other nine samples. F-500 components were detected in approximately 60% of the combined samples analyzed from the Fire Tests A and B when no ignitable liquid was used.

Fire Test C: Wood and Straw, Gasoline or Heavy Petroleum Distillate, Suppression with 1% F-500—Gasoline was identified in all three samples from the gasoline accelerated fire and no F-500 peaks were detected. The HPD was identified in two of the three debris samples from the HPD accelerated fires. F-500 peaks were present at a background level in the negative HPD sample.

Fire Test D: Wood and Straw, Gasoline or Medium Petroleum Distillate or Heavy Petroleum Distillate, Suppression with 1% F-500 or Water—A total of 36 debris samples were collected from six separate burns. Gasoline was identified in five of six water suppression samples and in five of six F-500 suppression samples. F-500 components were not detected. The MPD was identified in all six water suppression samples and in all six F-500 suppression samples. F-500 peaks were detected in two of the six F-500 suppression samples. The HPD was identified in all six water samples and in four of six F-500 samples. F-500 peaks were detected in one of these six samples.

Ignitable liquids were identified in approximately 94% of the water suppression samples and in approximately 83% of the F-500 suppression samples. F-500 peaks were detected in approximately 17% of the F-500 suppression samples. The number of positive results obtained from the ignitable liquid burn samples was similar irrespective of which suppression agent was used.

Fire Test E: Wood and Straw, Isoparaffin, Suppression with 1% F-500 or Water—The isoparaffin product was identified in four of the six water suppression samples and in four of the six F-500 suppression samples. F-500 was not detected. The use of F-500 as a suppression agent compared to water did not affect the number of positive findings of the ignitable liquid.

Fire Test F: Car Fires, Gasoline or Isoparaffin, Suppression with 1% F-500—Two samples were taken from a gasoline accelerated car fire. Gasoline was identified in one of the two samples analyzed. A background level of F-500 was detected in the negative sample. Three samples were collected from a car fire accelerated with an isoparaffin. The isoparaffin was identified in all three samples in the presence of F-500 components. F-500 was identified in four of five car fire samples. The presence of F-500 in the debris samples did not preclude the detection or identification of the isoparaffin product. Figure 4 illustrates the increase in intensity of F-500 peaks in the presence of an ignitable liquid upon sample heating.

The majority of debris samples analyzed (26 of 30 suppressed with F-500) from the combined ignitable liquid fire test burns were

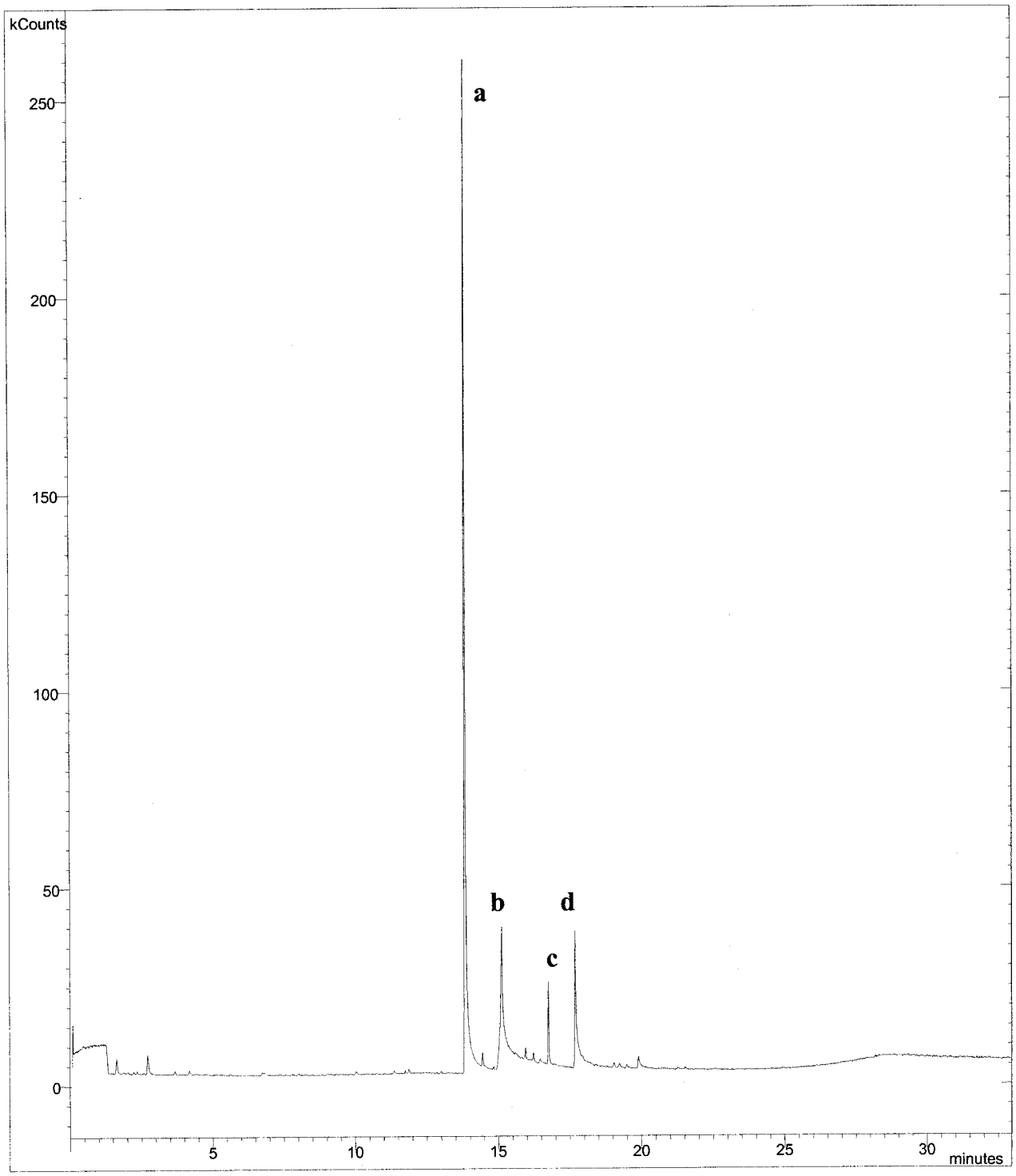

FIG. 1—*F-500: a) n-octanol; b) 2-ethylhexanoic acid; c) dodecane; d) n-decanol.*

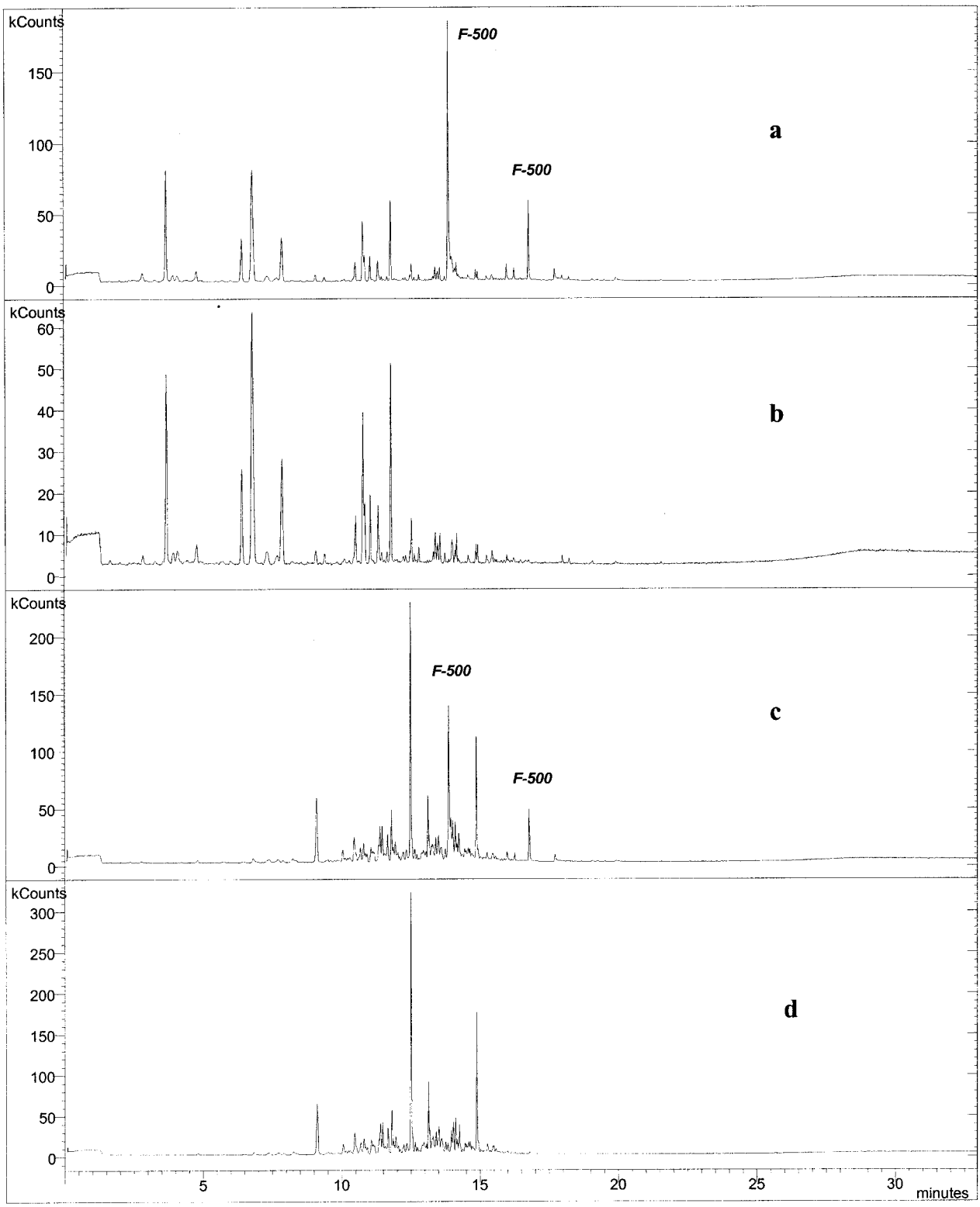

FIG. 2—*a) 0.4μL gasoline in 10 mL 1% F-500; b) 0.4μL gasoline in 10 mL water; c) 0.4μL MPD in 10 mL 1% F-500; d) 0.4μL MPD in 10 mL water.*

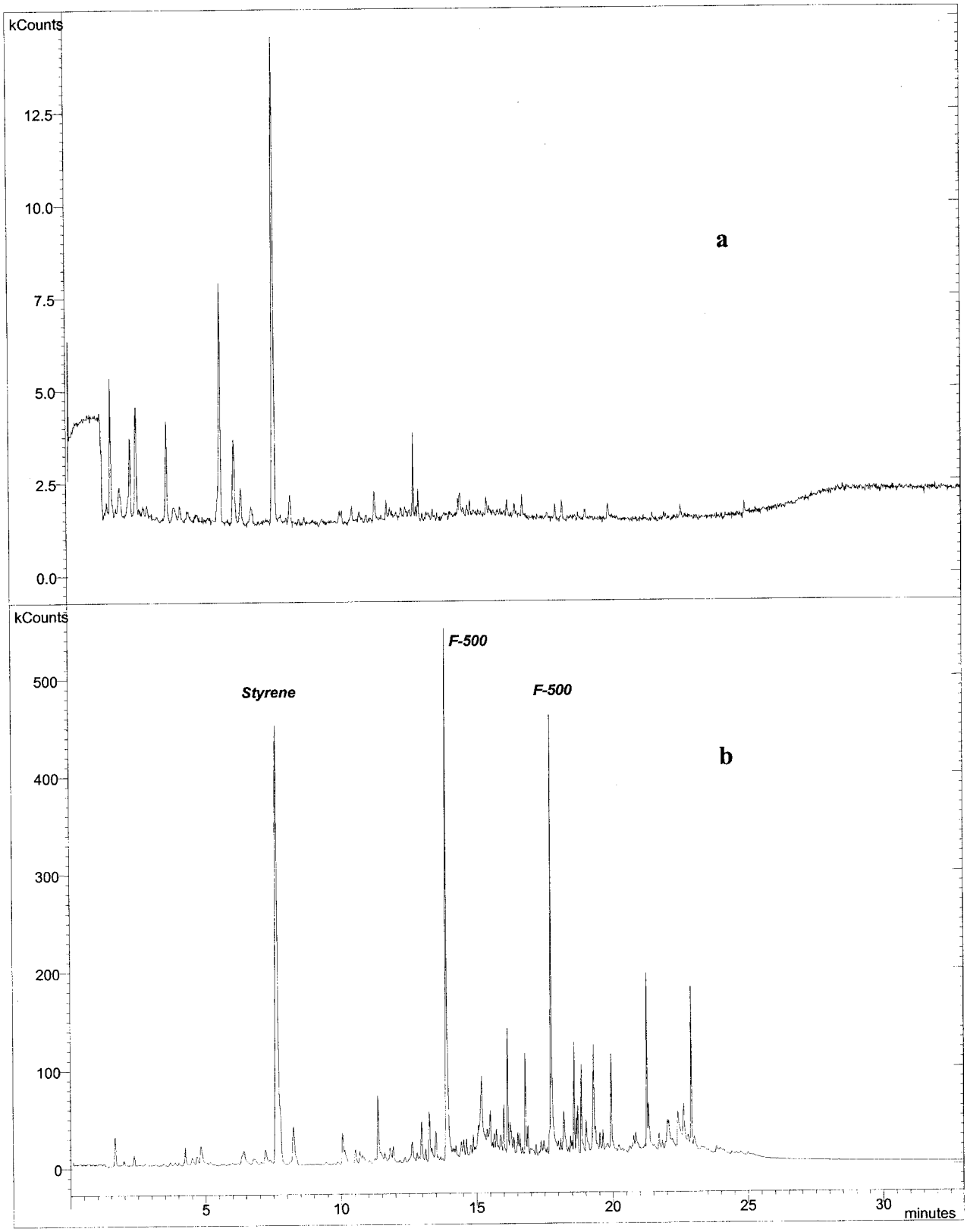

FIG. 3—*A carpet sample collected from a house fire in which no ignitable liquid was used: a) room temperature headspace analysis; b) heated headspace analysis.*

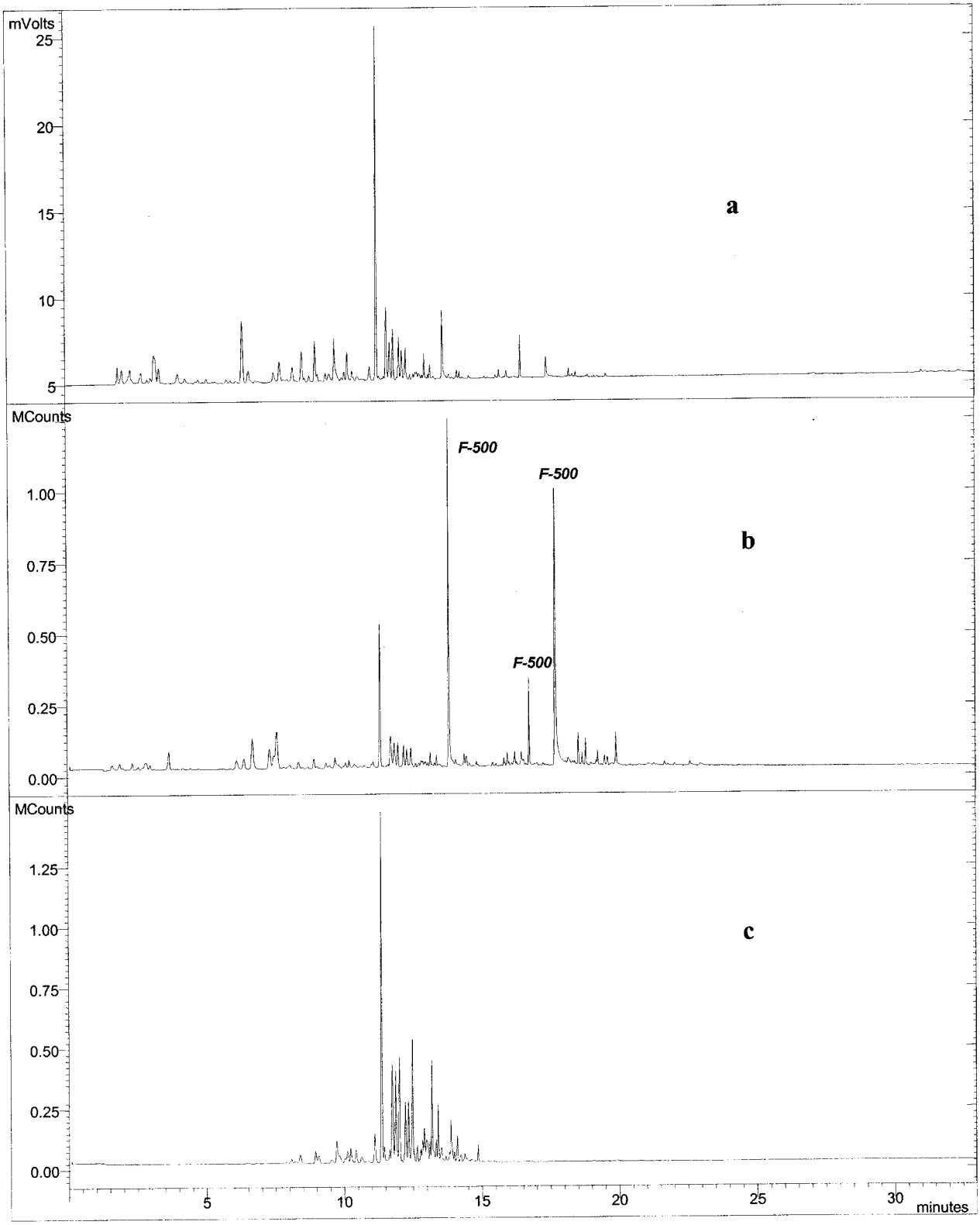

FIG. 4—*Car fire accelerated with isoparaffin; a) room temperature headspace analysis of debris sample; b) heated headspace analysis of debris sample; c) heated headspace analysis of isoparaffin standard.*

negative for the presence of F-500. One of five samples collected from accelerated car fires was negative for the presence of F-500.

F-500 was not detected in any of the house fire samples at room temperature, but was detected in a significant number of samples after heating. The background peaks associated with substrate materials do not normally increase in intensity as much as ignitable liquid peaks with sample heating. F-500 responds to heating in a manner similar to ignitable liquids.

Conclusion

This study has shown that the use of F-500, a micelle encapsulator fire suppression agent, does not preclude the detection or identification of common ignitable liquids in fire scene debris samples. If present in a sample, F-500 may generate a series of chromatographic peaks that interfere with patterns of ignitable liquids. It is important to note, however that the majority of fire debris samples collected and analyzed during this study did not show background interference from F-500. The level of interference will depend on many factors including the volume of F-500 used in suppression, the fire scene dynamics, the number and types of samples collected and the laboratory analysis scheme. F-500 peaks have been identified in casework samples at this laboratory as a result of this study. The fire debris analyst should become familiar with the range of components generated by this product and its contribution to background interference.

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