

## TECHNICAL NOTE

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# Extraction of Alternative Fuels from Fire Debris Samples\*

**ABSTRACT:** Alternative fuels, specifically biodiesel, biodiesel blends, and E85 fuel, have been gaining a market share over the past few years. With the emergence of these fuels, fire debris analysts should be able to recognize their characteristics since these fuels may be encountered in case-work. In this study, pure biodiesel (B100) and a 20% blend of pure biodiesel with petroleum diesel (B20) are examined as liquids and are extracted from debris samples using both passive headspace concentration and solvent extraction. Typical fire debris instrumental conditions are used to analyze these samples. Components of B100 and B20 may be observed in debris samples extracted using the passive headspace concentration method, but the chromatographic patterns are different from the pure liquid samples. When solvent extraction is used as a secondary extraction method on debris samples, the resulting patterns are consistent with the pure liquids of B100 and B20. E85 fuel, a blend of 85% ethanol and 15% gasoline, can be extracted using a typical fire debris extraction technique but requires slight modifications to typical fire debris instrumental conditions. E85 is shown at various stages of evaporation to demonstrate the resiliency of the ethanol. Additionally, samples of E85 were placed on carpet, burned and extinguished to demonstrate the effects of the suppression medium on the retention of ethanol.

**KEYWORDS:** forensic science, alternative fuels, biodiesel, E85, fire debris

In 1992, Congress passed the Energy Policy Act (EPAct) in order to reduce the United States' dependence on foreign petroleum. This act, officially known as Public Law 102-486, defines alternative fuels and establishes regulatory approaches to encourage an alternative fuel market in the United States (1). Pure biodiesel (B100) and E85 (a blend of 85% ethanol and 15% gasoline) are two fuels that are specifically designated by the EPAct as alternative fuels. A blend of 20% biodiesel and 80% petroleum diesel (B20), is not specifically defined as an alternative fuel, but qualifies for a biodiesel fuel use credit according to a modification of the EPAct (2). For purposes of discussion in this study, B20 will be considered as an alternative fuel.

Many government and corporate fleet vehicles use alternative fuels instead of traditional petroleum fuels. In addition, private vehicle owners are also turning to alternative fuels because of the financial and environmental benefits these fuels provide over traditional fuels. According to the United States Department of Energy, there are currently over 770 outlets for biodiesel and approximately 1,250 outlets for E85 fuel (3). The National Biodiesel Board estimates that biodiesel production in the United States is 30 times greater today than 5 years ago. There are 85 biodiesel production plants in the United States and 65 additional plants under construction (4). The consumption of E85 was approximately 38,074,000 GGEs (gasoline gallon equivalents) in 2005. This is a substantial increase from 1992 when E85 consumption was approximately 22,000 GGEs (5). These statistics indicate the increased availability and use of alternative fuels.

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\*This paper was presented, in part, at the 59th Annual American Academy of Forensic Sciences Meeting in San Antonio, TX, February 19-24, 2007.

Received 21 May 2007; and in revised form 12 July 2007; accepted 22 Dec. 2007.

A good overview of biodiesel including its history, production, and composition is given by Stauffer and Byron (6). Additionally, these authors showed the chromatographic features of B100 and B20 liquid samples and the chromatographic features of each liquid extracted from quart cans using passive headspace concentration (ASTM E 1412) (7). However, they only discussed the theoretical extraction of B100 and B20 from fire debris samples.

Traditional fire debris instrumental conditions that detect hydrocarbons to at least *n*-eicosane (C<sub>20</sub>) allow the analyst to see the pertinent components of biodiesel. Identification of E85, however, may require slight modifications to those instrumental conditions to detect the ethanol if a traditional solvent delay is used. Phelps, Chasteen and Render showed that ethanol and other volatile oxygenated compounds could be extracted and identified using passive headspace concentration (8). To date, limited work has been done in the forensic field regarding E85. Cornett et al. characterized E85 fuel using solid phase microextraction (9). No work discussing the extraction of E85 samples from fire debris has been published in the forensic literature.

Given the growing availability of alternative fuels, fire debris analysts may unknowingly encounter these fuels more often in their casework. Many fire debris analysts, however, are not familiar with B100, B20, and E85. Therefore, these fuels were evaluated both as neat liquid samples and extracted from debris using typical fire debris analysis techniques in order to introduce and familiarize the fire debris analyst with the chromatographic features of these fuels. Additionally, the effects of the extinguishment medium on the retention of E85 in fire debris were studied.

## Methods and Materials

A sample of B100 was obtained from Taylor Oil Southeast, Incorporated of Baltimore, Maryland. A sample of B20 was obtained from Major Brand Gas in Austin, Texas. The E85 fuel

was obtained from a Montgomery County, Maryland, municipal fueling station.

### Biodiesel Samples

Liquid samples of B100 and B20 were diluted with carbon disulfide (certified, Fisher, Fair Lawn, NJ). Various substrates including a single sheet of Kimwipes® (Kimberly-Clark, Roswell, GA), a 1-inch square of burned carpet, and a 2-inch square of burned wood were placed in quart cans. A 20  $\mu$ L aliquot of B100 was added to each substrate. A second set of substrates was created with a 20  $\mu$ L aliquot of B20 added to each substrate. These samples were then extracted using the passive headspace concentration method in accordance with ASTM E 1412 (7) using an activated charcoal strip (Albrayco Technologies, Cromwell, CT). These samples were heated at 65°C for 16 h and then extracted with approximately 300  $\mu$ L of carbon disulfide. This process was then repeated on burned carpet and burned wood using 200  $\mu$ L aliquots of B100 and B20. These samples were also subjected to solvent extraction in accordance with ASTM E 1386 (10). Each sample was extracted with approximately 20 mL of pentane (HPLC grade, Fisher). The extracted samples were then evaporated down to approximately 2 mL under a stream of nitrogen gas. All of the biodiesel samples were analyzed using the gas chromatograph-mass spectrometer (GC-MS), the column, and Method A listed in Table 1.

### E85 Samples

Samples of neat E85 fuel and evaporated E85 fuel were diluted in carbon disulfide and analyzed using the GC-MS, the column, and Method B listed in Table 1. Samples of either 5 mL or 10 mL of E85 fuel were placed on one-square foot carpet samples and burned. The modes of extinguishment included: allowing the sample to burn out (self-extinguishment), water, and a CO<sub>2</sub> fire extinguisher. The burned carpet samples were collected in their entirety and placed in gallon cans. Additionally, a 20  $\mu$ L sample of E85 was placed on a single sheet of Kimwipes® in a quart can. All samples were extracted using the passive headspace concentration

TABLE 1—Instrumental conditions for the analysis of biodiesel (Method A) and E85 (Method B).

	Method A	Method B
Initial oven temperature (hold time)	60°C (3 min)	40°C (2 min)
Temperature ramp	5°C/min	5°C/min
Temperature (hold time)	120°C (0 min)	120°C (0 min)
Temperature ramp	12°C/min	12°C/min
Final oven temperature (hold time)	300°C (5 min)	300°C (5 min)
Scan conditions	0–2.80 min: MS off > 2.80 min: 33–300 amu	0–1.91 min: 15–100 amu 1.91–2.60 min: MS off > 2.60 min: 33–300 amu

Instrument: Hewlett-Packard 6890 Series Gas Chromatograph Equipped with a 5972 Mass Selective Detector.

Column: Hewlett-Packard HP-1 (polydimethylsiloxane); 25 m; 0.20 mm I.D.; 0.5  $\mu$ m film thickness.

Carrier Gas: Helium, constant flow 0.5 mL/min.

Injector: Hewlett-Packard Automatic Liquid Sampler.

Injector temperature: 250°C.

Split ratio: 20:1.

Interface temperature: 280°C.

method (7) with an activated charcoal strip. These samples were heated at 65°C for 16 h and then extracted with approximately 300  $\mu$ L of carbon disulfide. The extracted samples were also analyzed by using the GC-MS, the column, and Method B listed in Table 1.

## Results and Discussion

### Biodiesel Samples

The total ion chromatogram (TIC) of a liquid sample of B100, obtained using a typical fire debris capillary column and GC-MS temperature program, is shown in Fig. 1a. Four main peaks, corresponding to various fatty acid methyl esters (FAMES), are observed in the TIC and identified by their mass spectra. These components are methyl hexadecanoate (C16:0), methyl octadecanoate (C18:0), methyl octadecenoate (C18:1), and methyl octadecadienoate (C18:2), each of which is labeled in Fig. 1a. The presence of these components corresponds well to the composition list provided by Taylor Oil, which shows that these four FAMES make up most of the biodiesel sample. It should be noted that methyl octadecatrienoate (C18:3) is not resolved from the C18:1 peak. Even though the separation of the FAMES is not ideal, the nonpolar column and the gas chromatographic conditions listed in Method A of Table 1 do allow for adequate detection and resolution of the components of B100. The fire debris analyst should recognize and realize the significance of this pattern in an unknown sample. Several ions characteristic of FAMES may also be extracted from the TIC to

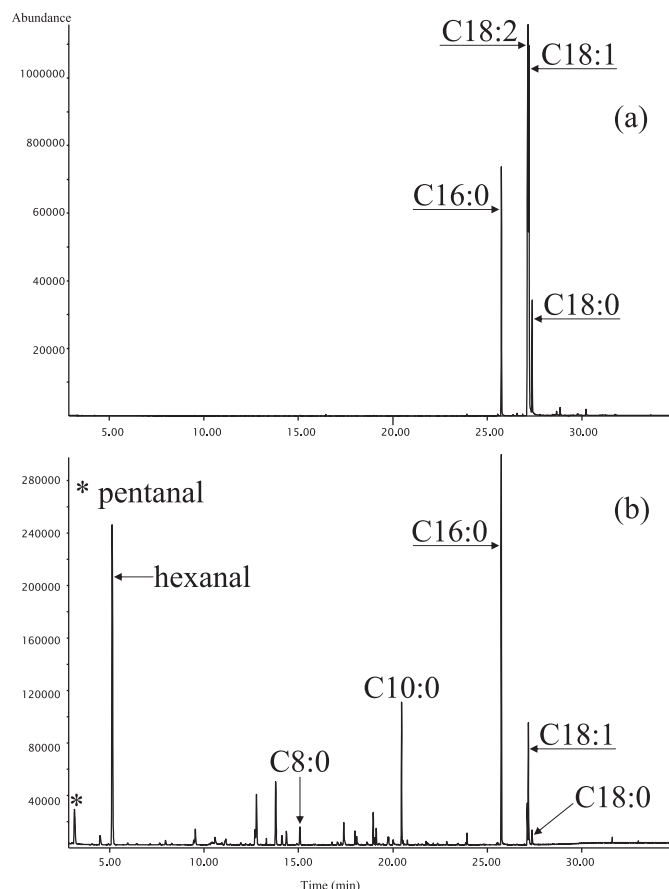


FIG. 1—TICs of (a) liquid B100 sample and (b) 20  $\mu$ L of B100 extracted from a clean substrate using passive headspace concentration.

TABLE 2—Characteristic ions of FAMES that can be used for extracted ion profiling.

<i>m/z</i>	FAME
74	Methyl ester fragment ( $\text{CH}_3\text{OCHO}^+\text{HCH}_2$ )
270	Methyl hexadecanoate (C16:0)
294	Methyl octadecadienoate (C18:2)
296	Methyl octadecenoate (C18:1)
298	Methyl octadecanoate (C18:0)

help with the interpretation of the data. These ions are listed in Table 2. If an analyst wants or needs to achieve better resolution of the FAMES, a column with a more polar stationary phase such as 95% cyanopropyl 5% phenyl polysiloxane could be used (11). Fig. 2a shows the TIC for liquid B20. A heavy petroleum distillate (diesel) pattern is readily apparent, but the biodiesel FAMES dominate the TIC. These chromatograms for B100 and B20 are consistent with those shown by Stauffer and Byron for liquid biodiesel samples (6).

The TICs of the B20 and B100 extracted from a sheet of Kimwipes<sup>®</sup>, which served as a clean substrate, look distinctly different from their respective liquid TICs. Fig. 1b shows the TIC of 20  $\mu\text{L}$  of B100 extracted from a clean substrate using passive headspace concentration. The C18:0, C18:1, C18:2, and C16:0 are still present, but their relative ratios have changed. This is due to the reduced efficiency of the extraction technique for heavier components, which results from the more volatile compounds being more prevalent in the headspace. In addition, Fig. 1b shows several other

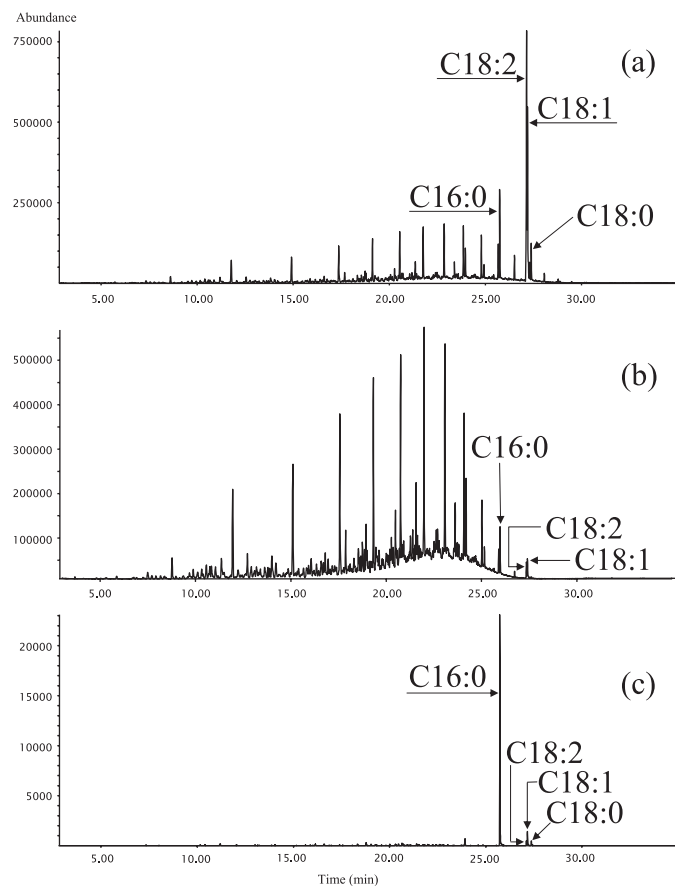


FIG. 2—(a) TIC of liquid B20 sample. (b) TIC of 20  $\mu\text{L}$  of B20 extracted from a clean substrate using passive headspace concentration. (c) FAME EIP of 20  $\mu\text{L}$  of B20 extracted from a clean substrate using passive headspace concentration.

lighter oxygenated compounds including shorter chain FAMES (C10:0 and C8:0) and aldehydes (hexanal and pentanal) as identified by their mass spectra. There are also several other oxygenated compounds which could not be specifically identified by their mass spectra. The presence of these compounds is most likely because of the oxidation of the unsaturated FAMES. The presence of these additional non-FAME peaks was not observed by Stauffer and Byron (6) in the TIC of a neat B100 sample extracted from a quart can. On the other hand, their B100 chromatogram displays a better recovery of the FAMES, and the peak ratios are more consistent with the liquid B100 sample. This is because of the higher temperature (80°C) they used for the extraction process. The TIC of a 20  $\mu\text{L}$  sample of B20 extracted from a clean substrate using passive headspace concentration is shown in Fig. 2b. The FAME compounds have a much lower abundance than the liquid B20 sample. In fact, the C18:0 peak is not observed. The overall skewing of the petroleum diesel pattern shown in Fig. 2b is consistent with what is normally seen when diesel fuel is extracted using passive headspace concentration. The reduced efficiency of the passive headspace concentration method for extracting heavier components is responsible for the skewing of the petroleum pattern and is responsible for the reduced abundance of the biodiesel FAMES in the extracted B20 sample. As shown by Stauffer and Byron (6), a higher extraction temperature will enhance the recovery of the FAMES. Another factor which may play a role in the reduced abundance of the FAMES in the B20 sample is the displacement of the FAME components by the petroleum hydrocarbons. Because of this reduction of the FAMES it is important for the fire debris analyst to identify extraneous peaks in a heavy petroleum distillate pattern. This can be done relatively easily using the mass spectrometer and a computerized library search. The analyst may also use the ions listed in Table 2 to assist in the interpretation of the data by producing an extracted ion profile (EIP) as shown in Fig. 2c. If FAMES are present, the analyst should suspect the presence of a biodiesel blend and should obtain samples of B100 and B20 for comparison purposes and to determine the retention time of the FAMES.

B100 and B20 samples were each placed on substrates commonly encountered in fire debris analysis. Figure 3a shows the TIC for 20  $\mu\text{L}$  of B100 on burned wood extracted using passive headspace concentration. The C16:0 peak is clearly observed in the TIC as are some of the lighter aldehyde and fatty acid methyl ester components. The C18:1 peak is also observed, but the C18:2 peak is hardly discernible and the C18:0 peak is absent. A 20  $\mu\text{L}$  aliquot of B100 was also placed on burned carpet. However, no peaks attributable to the B100 were noted in the TIC of the spiked carpet sample. This may be due to a competitive adsorption process with the burned carpet, the reduced efficiency of extracting heavier components intrinsic to the extraction method, or a combination of both processes. When the volume of B100 was increased to 200  $\mu\text{L}$  on burned wood, all of the major peaks attributable to the B100 are observed. Figure 3b shows this chromatogram with the primary FAMES labeled and other peaks attributed to the B100 (as seen in Fig. 1b) marked with asterisks. The C18:0 peak is not labeled since it is barely discernible after the C18:1 peak. An EIP developed using all five ions listed in Table 2 is shown in Fig. 3c. This EIP clearly shows the C16:0 and the C18 FAMES. Similarly, by increasing the volume of B100 on burned carpet to 200  $\mu\text{L}$ , peaks attributable to the B100 are observed, but none of the C18 FAMES are present.

Burned wood and burned carpet were also spiked with 20  $\mu\text{L}$  aliquots of B20. The petroleum diesel portion of B20 is clearly seen in both the wood and carpet samples; however, the biodiesel

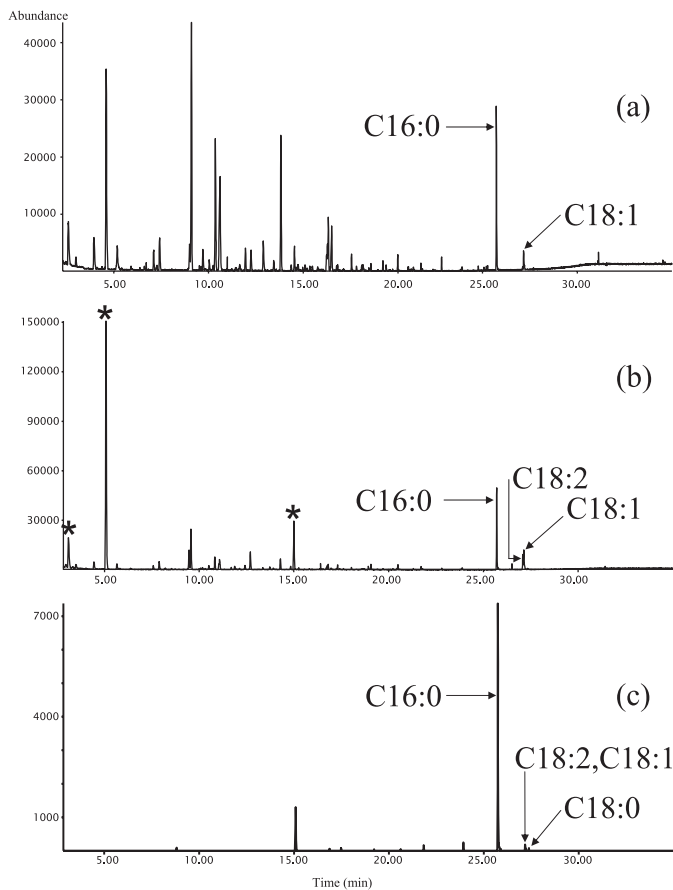


FIG. 3—(a) TIC of 20  $\mu\text{L}$  of B100 on burned wood extracted using passive headspace concentration. (b) TIC of 200  $\mu\text{L}$  of B100 on burned wood extracted using passive headspace concentration. Asterisks indicate additional components attributable to B100. (c) FAME EIP of 200  $\mu\text{L}$  of B100 on burned wood extracted using passive headspace concentration.

portion is not observed in either sample. This result is not really surprising given the reduced proportion of the biodiesel components in the sample of B20 extracted from a clean substrate. When the volume of B20 was increased to 200  $\mu\text{L}$ , there was a slight indication of the C16:0 peak following the nonadecane ( $\text{C}_{19}$ ) peak. The TIC for 200  $\mu\text{L}$  of B20 on burned wood is shown in Fig. 4a, and the TIC for 200  $\mu\text{L}$  of B20 on burned carpet is shown in Fig. 4b. Since the FAMEs are not extracted efficiently, even the use of an EIP would not help elucidate them. There are a variety of possible reasons why the FAME components of B20 are not extracted efficiently. One possibility, as mentioned previously, is the reduced efficiency of the passive headspace concentration method for extracting heavier compounds. The hydrocarbons from the petroleum diesel may also be displacing the FAMEs. A final possibility is that a competitive adsorption process is occurring with the burned substrates. Most likely, a combination of these effects is taking place.

Given that passive headspace concentration of burned samples with B100 and B20 displayed some chromatographic features different from the liquid samples, the 200  $\mu\text{L}$  spiked samples of burned wood and burned carpet were subjected to solvent extraction after passive headspace concentration sampling. Solvent extraction is a valuable technique for recovering heavier components (above  $\text{C}_{17}$ ) from fire debris and can be used to show difference between a kerosene-type and a diesel fuel-type of heavy petroleum distillate. This method is infrequently used because of

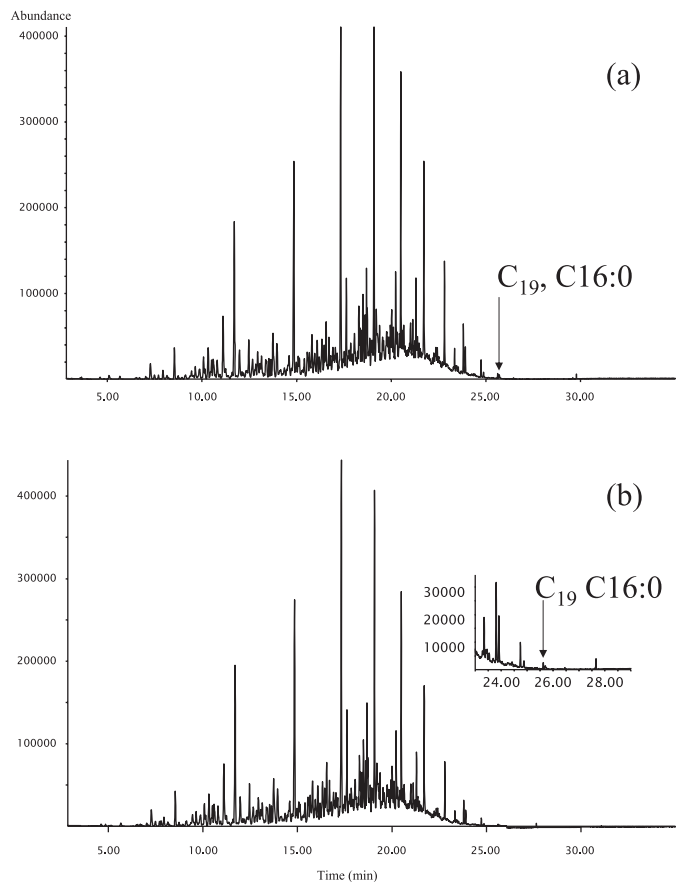


FIG. 4—TICs of (a) 200  $\mu\text{L}$  of B20 on burned wood extracted using passive headspace concentration and (b) 200  $\mu\text{L}$  of B20 on burned carpet extracted using passive headspace concentration.

its destructive nature, the volume of solvent needed and the subsequent need for evaporation of the solvent. However, a solvent extraction with pentane proves to be effective in extracting the FAMEs of B100 from the burned wood and the burned carpet samples even after samples have been previously extracted by passive headspace concentration. The C16:0 and the C18 FAMEs are easily detected and identified and the pattern is virtually identical to the liquid sample of B100 shown in Fig. 1a. The observed patterns are in stark contrast to the patterns shown in Fig. 3 where only the C16:0 and some of the lighter peaks are observed after using the passive headspace concentration method. Both the burned wood and the burned carpet samples extracted with pentane do display some of the lighter FAMEs and aldehydes that are seen in samples extracted by passive headspace concentration, but their contribution to the chromatographic pattern is relatively minor.

As shown in Fig. 4, the FAMEs in samples spiked with 200  $\mu\text{L}$  of B20 and extracted with passive headspace concentration are nearly non-existent. When a solvent wash with pentane was used after passive headspace concentration on the samples of burned wood and burned carpet, all of the FAME components were readily extracted from the substrates. The ratios of the FAME components to the petroleum diesel are consistent with the liquid B20 sample shown in Fig. 2a. These results clearly demonstrate the benefit of using a solvent wash if a fire debris sample extracted by passive headspace concentration contains FAMEs or if the debris is suspected to contain biodiesel.

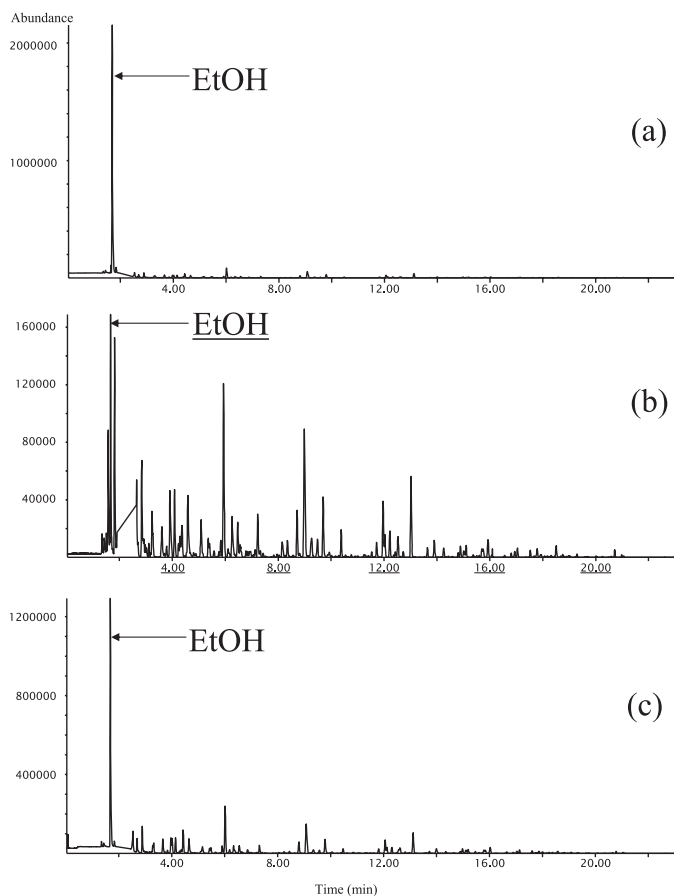


FIG. 5—TICs of (a) E85, (b) gasoline commonly sold in the United States with up to 10% ethanol, and (c) 20  $\mu$ L of E85 extracted from a clean substrate using passive headspace concentration.

### E85 Samples

Method A in Table 1, typical of the method used in many fire debris analysis laboratories, begins data collection after a solvent delay. This method's solvent delay does not allow for the detection of the ethanol in E85; therefore, the resulting chromatogram is indistinguishable from a traditional gasoline pattern. When the instrumental conditions are adjusted to detect light oxygenated compounds, as in Method B in Table 1, ethanol is readily observed. Figure 5a shows the TIC of E85; ethanol dominates the chromatogram, but a traditional gasoline pattern is also visible. This TIC pattern and all other TICs for the E85 samples are expanded to better show the area of chromatographic interest.

Gasoline now commonly sold in the United States contains up to 10% ethanol, and its TIC is shown in Fig. 5b. Even though ethanol is observed, it is at a much lower level than the ethanol in E85. Fire debris analysts are familiar with the changes that occur in the gasoline pattern because of evaporation. Therefore, samples of E85 were evaporated to various levels to determine if the same trends were observed with this alternative fuel. The TICs of E85 samples evaporated to 50%, 90%, and 98% are shown in Figs. 6a, 6b, and 6c, respectively. It is important to note that ethanol remains prominent throughout the various stages of evaporation while the gasoline portion evaporates as expected. There is not a substantial decrease in the ethanol content until the evaporation of E85 reaches 98%. The resilience of the ethanol in evaporated E85 may allow for the identification of E85 and in some circumstances could be a valuable investigative lead.

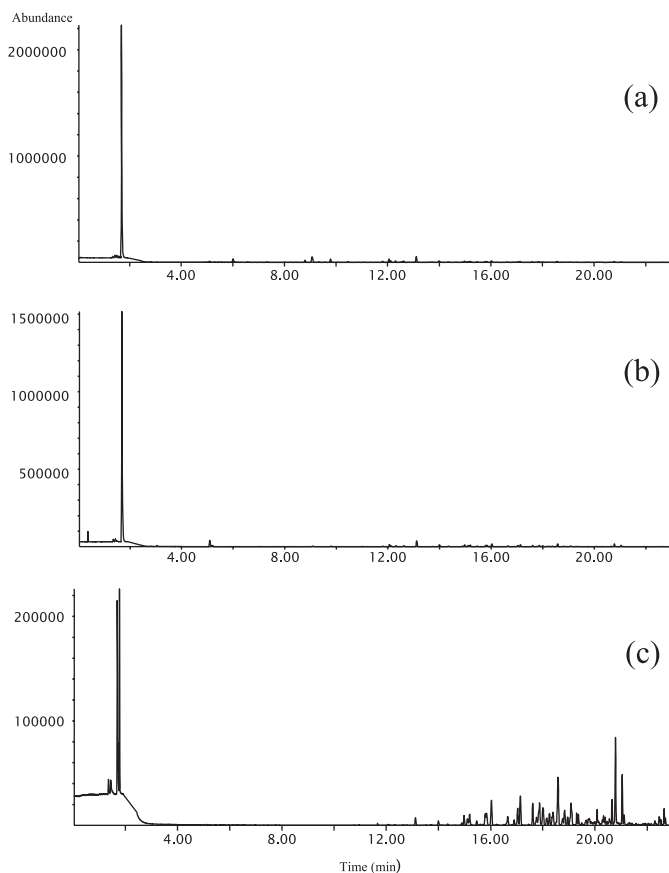


FIG. 6—TICs of evaporated E85. (a) 50%, (b) 90%, and (c) 98%.

To determine the effect of passive headspace concentration on E85, a 20  $\mu$ L sample of E85 was placed on a clean substrate and extracted with carbon disulfide. The resulting TIC is shown in Fig. 5c. Ethanol is still the predominant peak, but the gasoline portion is slightly more pronounced when compared to the TIC from the liquid E85 sample. This is likely because of the gasoline hydrocarbons preferentially adsorbing to the charcoal strip and displacing the ethanol.

Since ethanol is water soluble, fire suppression activities may have an effect on the presence of ethanol in fire debris samples. To examine this aspect, E85 was placed on carpet samples, set on fire, and extinguished in various ways. When a 5 mL sample of E85 was placed on carpet and allowed to burn, it self-extinguished in approximately 1 min and 15 sec. The TIC of this sample, which was extracted by passive headspace concentration, is shown in Fig. 7a. An ethanol peak is present, but at a much lower level than the gasoline pattern. The observed ratio of ethanol to gasoline in Fig. 7a is much lower than the ratio of a comparably evaporated E85 sample (Fig. 6a). In fact, the ethanol peak is barely detectable and may not meet the identification criteria for an oxygenated compound according to ASTM E 1618 (12), especially in an unknown fire debris sample. Since the ethanol is at such a low level, the E85 sample cannot be differentiated from a gasoline sample containing up to 10% ethanol.

Once the self-extinguishment time was determined, 5 mL samples of E85 were placed on separate pieces of carpet and burned. These samples were extinguished at approximately one-half of the self-extinguishment time. Some samples were extinguished with water while the other samples were extinguished with a carbon dioxide ( $\text{CO}_2$ ) fire extinguisher. A sample extinguished with water,

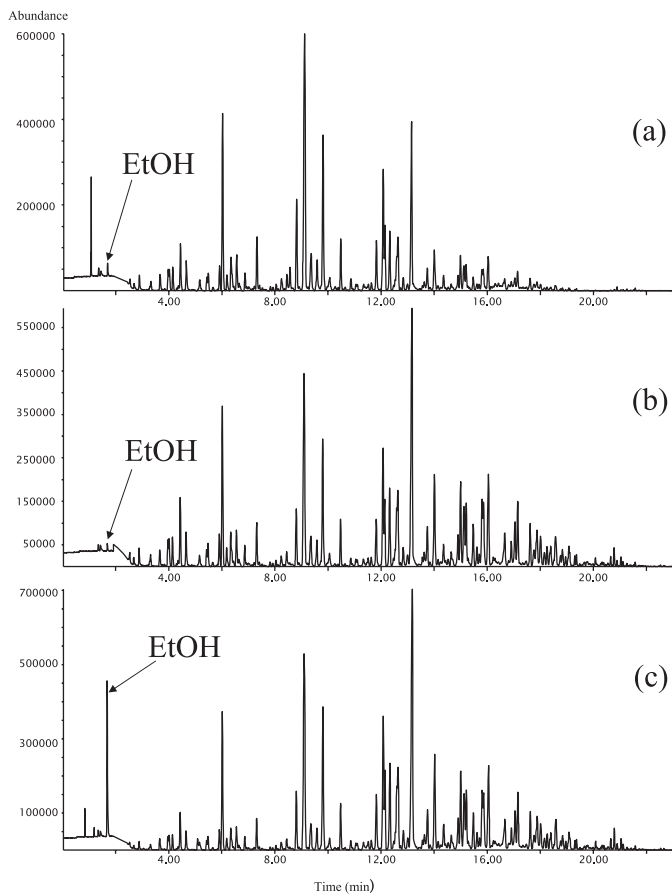


FIG. 7—TICs of (a) 5 mL of E85 burned on carpet and allowed to self-extinguish (extracted using passive headspace concentration), (b) 5 mL of E85 burned on carpet and extinguished with water at one-half of the self-extinguishment time of 5 mL of E85 on carpet (extracted using passive headspace concentration), and (c) 5 mL of E85 burned on carpet and extinguished with a CO<sub>2</sub> fire extinguisher at one-half of the self-extinguishment time of E85 on carpet (extracted using passive headspace concentration).

whose TIC is shown in Fig. 7b, displays a very small ethanol peak, comparable to the self-extinguished sample. Figure 7c shows the TIC of a sample that was extinguished with CO<sub>2</sub>. This chromatogram displays a distinct and identifiable ethanol peak. When the burning process was repeated with 10 mL of E85 at the 5 mL self-extinguishment time (double the volume and double the extinguishment time), the resulting TIC for the sample extinguished with water was generally the same as the one observed with the previous test. When the sample was extinguished with CO<sub>2</sub>, there was a greater than expected increase in the ethanol content relative to the previous CO<sub>2</sub> test. These tests demonstrate that, on a small scale, water does have a detrimental effect on the presence of ethanol in E85 samples in fire debris. Since water is the most common mode of extinguishment at fire scenes, it is expected that the ethanol in E85 would be drastically reduced in samples collected for fire debris analysis. However, if a sample is collected from an area with a large pour or from a protected area, the presence of ethanol may be detected at a sufficient level to identify it and allow the fire debris analyst to distinguish an E85 sample from regular gasoline containing 10% ethanol.

## Conclusions

Since alternative fuels are gaining a niche in the automotive fuel marketplace and may be encountered in fire debris submitted to the

laboratory, analysts need to become familiar with the chromatographic features of these fuels as neat liquids and in debris samples. Pure biodiesel (B100) and 20% biodiesel mixed with 80% petroleum diesel (B20) contain various FAMES that can be observed and identified using a typical fire debris column and GC-MS temperature program; however, the analyst may need to specifically look for them. The extraction of B100 and B20 from fire debris using passive headspace concentration is not optimal because of its reduced efficiency for extracting heavier components and the phenomena of competitive adsorption. However, a subsequent solvent wash with pentane of a sample suspected to contain biodiesel allows for the clear detection and identification of FAMES present in burned substrates.

It is recommended that fire debris analysts obtain samples of B100 and B20 to add to their ignitable liquid reference collections. The analysis of these liquids will show the presence of the FAMES and provide retention time and mass spectral information that can be used to evaluate samples suspected to contain biodiesel. If investigative information suggests the presence of biodiesel, or if the analyst observes indications of FAMES in a debris sample, a solvent wash should be used to extract the FAMES from the debris. Additionally, ions characteristic of the FAMES in biodiesel may also be used to help determine the presence of FAMES. Even though typical fire debris instrumental conditions can be used to detect biodiesel, a GC column more suitable for FAME analysis may be used at the examiner's discretion to help elucidate the biodiesel components in a complex fire debris sample.

E85, a blend of 85% ethanol with 15% gasoline, is readily extracted from debris using passive headspace concentration and is easily recognized using a fire debris GC-MS method configured to detect light oxygenated compounds. Although the ethanol peak dominates the TIC, the gasoline pattern is still readily observed. Upon evaporation of E85, the ethanol remains the primary component in the TIC while the gasoline evaporates in the expected manner. In addition, the mode of extinguishment plays a substantial role in the retention of ethanol in a fire debris sample and may affect the analyst's ability to determine the presence of an E85 fuel used as an ignitable liquid.

## Acknowledgment

The authors would like to thank Meghan Miller for her assistance in preparing some of the samples used in this study.

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