

Rapid Detection of Accelerants in Fire Debris Using a Field Portable Mid-Infrared Quantum Cascade Laser Based Analyzer

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Abstract

Arson presents a challenging crime scene for fire investigators worldwide. Key to the investigation of suspected arson cases is the analysis of fire debris for the presence of accelerants or ignitable liquids. This study has investigated the application and method development of vapor phase mid-Infrared (mid-IR) spectroscopy using a field portable quantum cascade laser (QCL) based system for the detection and identification of accelerant residues such as gasoline, diesel, and ethanol in fire debris. A searchable spectral library of various ignitable fluids and fuel components measured in the vapor phase was constructed that allowed for real-time identification of accelerants present in samples using software developed in-house. Measurement of vapors collected from paper material that had been doused with an accelerant followed by controlled burning and then extinguished with water showed that positive identification could be achieved for gasoline, diesel, and ethanol. This vapor phase mid-IR QCL method is rapid, easy to use, and has the sensitivity and discrimination capability that make it well suited for non-destructive crime scene sample analysis. Sampling and measurement can be performed in minutes with this 7.5 kg instrument. This vibrational spectroscopic method required no time-consuming sample pretreatment or complicated solvent extraction procedure. The results of this initial feasibility study demonstrate that this portable fire debris analyzer would greatly benefit arson investigators performing analysis on-site.

Keywords

Quantum Cascade Laser (QCL), Mid-Infrared Spectroscopy, Fire Debris

1. Introduction

Fires are a major concern for both public safety and economic reasons. Arson, the deliberate setting of fires to destroy property, to take human life or to obscure a crime scene is of particular concern and continues to be a problem worldwide [1]. Arson scenes are complicated and highly challenging for an investigator as much of the evidence is damaged by the fire in addition to any subsequent extinguishing efforts. Arsonist typically uses cheap, easily obtainable common ignitable liquids to accelerate combustion. According to statistics most arson cases involve gasoline, diesel, or high-concentration alcohol as an accelerant [2] [3] [4]. Therefore, for the investigator, the identification of accelerants and ignitable liquids are important for determining the nature and cause of the fire. Fortunately, such ignitable liquids used can be identified from traces that are left in the fire debris or residues that remain by incomplete combustion.

The current gold standard method for analyzing fire debris to determine if an accelerant is present is gas chromatography-mass spectrometry (GC-MS) [5] [6]. However, this instrumentation is expensive and requires samples to be brought back to the lab for analysis by a skilled technician. It is time consuming as the samples must be pretreated to extract possible accelerants using solid-phase micro extraction techniques and other complicated sample preparation methods [6] [7]. A variety of spectroscopic techniques have been reported for analyzing petroleum products in fire debris samples [8]. This includes vapor phase UV-vis [9], electronic aroma detection, [10], laser induced break down [11], and vibrational spectroscopy such as near-IR, mid-IR, and Raman [8] [12] [13] [14]. Most of these spectroscopic studies involved sample pretreatment and complicated extraction methods. The vibrational spectroscopic methods provide a non-destructive technique that has gained wide use in many application areas in both analyzing neat ignitable liquids and accelerants. However, despite advantages over traditional techniques, both IR and Raman spectroscopy have been limited to mostly studies analyzing ignitable liquid samples, or decomposed polymers and cotton materials found in fire debris or have required extensive pretreatment and extraction to detect accelerants such as gasoline [13]. Sensitivity, size, speed, and ease of sampling have been an issue with many such systems.

Of importance, quantum cascade lasers (QCL), which are semiconductor lasers emitting in the mid-IR, have found wide use in various spectroscopic sensing applications over the past decade [15] [16]. The fast response time, low power consumption and wide tuning range capabilities of these lasers have allowed for development of highly sensitive, faster, and more compact gas analyzers that are replacing larger and slower FT-IR systems. These lasers are ideally suited as mid-IR light sources and therefore such QCL-based gas sensing systems have

been used in applications such as industrial process and environmental monitoring [16]. This Mid-IR technique has advantages in that it is sensitive and can discriminate between different compounds of interest covering a wide spectral range useful for identification purposes. Furthermore, it is a nondestructive technique well suited for analyzing gases as well as vapors of volatile liquids. This is important as the fire debris can be directly analyzed without any pretreatment, simply measure vapors collected above the burned sample material.

The purpose of this study is to evaluate the ability to rapidly detect accelerants in fire debris samples requiring no sample pretreatment using a mid-IR based vapor analyzer. Here the mid-IR technique is used to analyze fire debris samples for presence of gasoline, diesel, and ethanol residues. The mid-IR QCL based analyzer presented in this study is field portable and incorporates quantum cascade lasers that provide a unique compact design. This is the first time as far as we can tell that such a mid-IR QCL system has been used and reported to detect and identify gasoline and other accelerants directly from vapor measurement of a fire debris sample. This feasibility study is important as it demonstrates that the field portable mid-IR QCL system presented here can provide fire investigators with an important tool to help determine on-site the nature of accelerants used in the case of arson.

2. Materials and Methods

2.1. Experimental Setup

The study presented here was conducted at Micro Optical Instruments in Shenzhen China and at Shanghai Fire Research Institute of MEM in Shanghai China. All measurements were made with MOI's portable mid-IR system (model QCL-BXFDA203) incorporating two pulsed quantum cascade lasers sources operating at room temperature that offer a gap-free tuning range from 6.1 μm to 10.4 μm (1635 cm^{-1} - 960 cm^{-1}) with a spectral resolution of 5 cm^{-1} . We use two lasers to expand our spectral coverage range. Laser 1 covers the region from 6.1 μm to 7.4 μm and laser 2 provides overlapping coverage from 7.4 μm to 10.4 μm . The lasers provided an average optical power of 5 mW. Sampling is achieved via a built-in pump that draws air/vapors into an internal gas cell. A valve controls the flow of vapor into and out of the cell. The sample to be measured is drawn through an external intake tube into the gas cell and is expunged through the output tube. This cell is designed to produce multiple reflections such that a pathlength of 1.5 m is obtained. This allows for exceptional sensitivity within a compact portable system design. The schematic of the experimental setup is shown in **Figure 1** for this field-portable analyzer. This system is integrated into a hardened case equipped with wheels for greater mobility. The system is battery operated and can also be plugged directly into an electric socket for continuous operation. In addition, ports allow for direct connection to a laptop when needed.

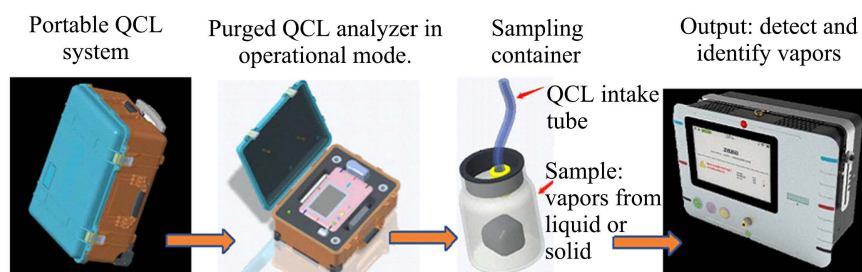


Figure 1. Experimental setup for mid-IR QCL analysis. The portable mid-IR (QCL) system is first purged using built-in pump housed inside the instrument, air background is measured then sample gas/vapors are drawn into the internal gas cell through the intake tubing (30-s) and is flowed out through the output tubing (purging the cell between measurements with air takes about 1 minute). Output: detect and identify accelerants.

2.2. Sample Preparation and Measurements

Common accelerants used in arson were purchased at a local Shenzhen China gas station (92-octane gasoline, kerosine, and diesel). These liquids were transferred to 20 mL amber vials with Teflon cap liners and maintained at 4°C until ready to use. Other ignitable liquids (ethanol, banana oil, cleaning solvent) and fuel component/additive chemicals xylene and methyl tert-butyl ether (MTBE) were purchased from Aladdin (Shanghai, China). Ethanol vapor at 98.7 parts-per-million (ppm) in an 8-liter gas cylinder was purchased from Guangdong Huate Gas Limited Company (Shenzhen, China) and used as a standard in the preliminary performance measurements.

A spectral library was constructed from measurement of each of these liquids. First a background is measured by flowing ambient air through the system gas cell for a minute. The valve is closed and then the background spectrum is collected with 4-s integration time and stored. The gas cell is then purged with air for 1 minute. Vapor in the headspace above the liquid in the half-filled vial was sampled by opening the vial and allowing the gas to be drawn into the purged gas cell for 30-s. The data collected is stored as an excel csv file. In this format the y-axis spectral intensity is collected as transmittance and the x-axis as wave-number. Our in-house collection software automatically corrects the sample spectrum for any background contribution present. The corrected reference sample spectrum was stored in the library. This procedure was repeated for each of the above-listed reference liquids. The stored spectral data provides the means to detect and identify potential accelerants and ignitable liquids investigated in this fire debris study. The spectral identification and analysis software (described below in the Data Analysis section) was developed in-house.

Figure 2 illustrates the procedure and steps used to make fire debris measurements. In this feasibility study, crumpled up paper was soaked with 92-octane gasoline and then ignited. The controlled fire was allowed to burn for 3 minutes, and then it was extinguished with water. The fire debris was then collected and placed into a bag and closed. An ambient air background was obtained in the fire control area. After several minutes the bag was opened, and it



Figure 2. fire debris sample measurement, (a) material is doused with an accelerant and ignited and then extinguished with water, (b) an air background is collected from the burn area, and (c) the vapors from fire debris sample enclosed in a bag is opened and measured.

was sampled by drawing vapors into the analyzer. The output results shown on the display screen identifies if an accelerant was present. Separate fire debris experiments were also performed on diesel and ethanol.

2.3. Data Analysis

In this study the analysis and presentation of spectroscopic data was accomplished by the following: The spectral data collected for the background and sample measured were provided in a Microsoft Excel document (as a csv file). The raw data is given as transmittance (T). All spectra presented here in this study were manually converted to absorbance in Excel by the equation $-\text{Log}_{10}(T)$. Both background and sample measurements could be individually converted in this way. To obtain the background corrected sample spectrum in absorbance simply use $-\text{Log}_{10}(\text{uncorrected sample data}/\text{air background data})$. The absorbance spectra were plotted in Origin Pro 9.0.

The spectral identification and analysis software was developed in-house with python. The algorithm used here to match spectra stored in the library has been discussed in a previously published paper [17]. The estimated limit of detection (LOD) was calculated by a simple approach (following ICH guidelines) based on the signal-to-noise ratio of 3, where $\text{LOD} = (C)/([S/N]/3)$, C is the concentration in parts-per-million (ppm) or parts-per-billion (ppb), S is the signal baseline-corrected peak intensity, N is root mean square (RMS) noise, and where the cut-off peak signal is assigned as three times the background-signal or blank [18].

3. Results and Discussion

3.1. Reference Spectra and Spectral Library

Spectroscopic reference data were measured for various pure samples (vapor phase) of accelerants and ignitable fluids that can potentially be used for arson. The spectra were collected on pure reference samples with our portable QCL system that was continuously tuned from 960 cm^{-1} to 1635 cm^{-1} ($10.4\text{ }\mu\text{m}$ - $6.1\text{ }\mu\text{m}$) as described above in the Materials and Methods section. This reference data was stored in the QCL analyzer as background corrected transmittance

spectra in the library for spectral matching and identification purposes. All mid-Infrared spectra presented in this paper were manually converted from transmittance to absorbance as described in the above experimental data analysis section. **Figure 3** shows the spectra of the common fuel accelerants gasoline, kerosine and diesel and these spectra are consistent with previously reported mid-IR studies of reference samples [16] [19] [20] [21]. The 92-octane gasoline spectrum in **Figure 3(a)** is dominated by absorption peaks at 1095 cm^{-1} (singlet), $1204/1215\text{ cm}^{-1}$ (doublet), $1364/1371/1380\text{ cm}^{-1}$ (triplet) and an additional feature with discernible peaks at 1459 , 1464 and 1475 cm^{-1} . The spectra for kerosine (**Figure 3(b)**) and diesel (**Figure 3(c)**) are very similar to each other and are characterized by two doublets at $1364/1382\text{ cm}^{-1}$ and $1457/1471\text{ cm}^{-1}$ (kerosine), and $1365/1382\text{ cm}^{-1}$ and $1459/1471\text{ cm}^{-1}$ (diesel). However, there are spectral differences that allow for discriminating between diesel and kerosine. For example, diesel shows an additional discernible peak at 1371 cm^{-1} that is unique, and kerosine shows a small doublet centered around 1350 cm^{-1} that is absent in diesel. The spectral software can analyze the full spectrum and identify both diesel and kerosine samples. It is worth noting that diesel contains kerosine (from 80:20 up to 50:50 v/v ratio), which is added to improve diesel fuel performance, especially for cold weather. The spectrum of gasoline is clearly distinguishable from that of kerosine and diesel. Gasoline detection is important as it is the most common accelerant used in reported arson cases [2].

Figure 4 illustrates as an example the removal of the air background contribution for the gasoline reference sample. As described in the methods section (data analysis) an ambient air background is collected prior to each sample measured and is automatically removed from the sample spectrum by the software. **Figure 4(a)** shows the uncorrected absorbance spectrum of the 92-octane gasoline. **Figure 4(b)** shows the background corrected absorbance spectrum of this gasoline sample. **Figure 4(c)** provides the absorbance spectrum of the air background from 960 cm^{-1} to 1635 cm^{-1} . The corrected spectrum result is the

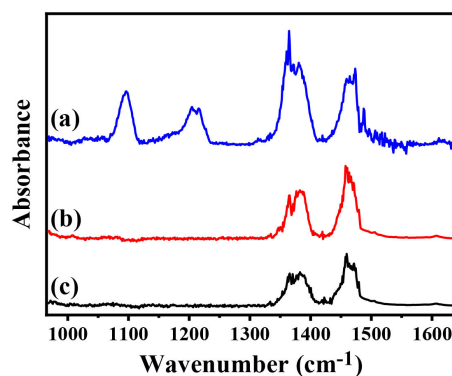


Figure 3. The absorbance spectra of fuel accelerant reference samples included in the spectral library. (a) 92-octane gasoline, (b) kerosine, and (c) diesel. Conditions as described in the material and methods section. All single measurement spectra were background corrected.

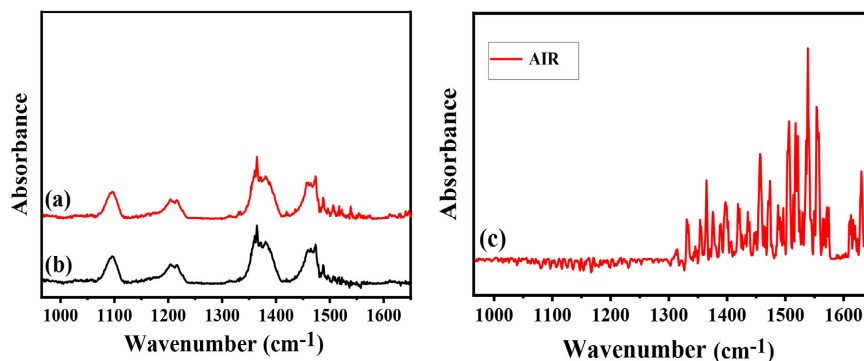


Figure 4. The absorbance spectra of (a) uncorrected and (b) background corrected 92-octane gasoline reference sample. (c) shows the air background spectrum. Conditions as in **Figure 3**.

same as shown above in **Figure 3(a)**. It is worth noting that the air background is dominated by water moisture, and it is well known that mid-IR is sensitive to such water vapor. Although the air background is not intense it does complicate the spectral region above 1300 cm^{-1} , particularly above 1475 cm^{-1} when sample absorbance intensity is weak. We are investigating new algorithms for more effective removal of this weak inherent background. Furthermore, in most cases regarding fire and suspected arson, debris samples will be collected after water is used to extinguish the flames. For this reason, we are looking at various physical means (in-line drying tube filled with desiccant, drying agent, or molecular sieves) to absorb any moisture content from the vapor being sampled from fire debris.

Figure 5 below shows additional library reference spectra including ignitable fluids such as ethanol (**Figure 5(a)**), a flammable cleaning solvent (**Figure 5(b)**) and banana oil (**Figure 5(c)**). Ethanol shows two characteristic absorbance features in the spectrum, a triplet centered at 1065 cm^{-1} , and a multippeak feature centered around 1382 cm^{-1} . The cleaning solvent shows two strong features at 1554 and 1591 cm^{-1} . Banana oil is dominated by three peaks at 1066 , 1144 and 1233 cm^{-1} . Most important is that gasoline with a strong unique signature peak at 1095 cm^{-1} can be discriminated against these other ignitable liquids that can be used to start fires. A fuel additive (MTBE in **Figure 5(d)**) and a major fuel component (Xylene in **Figure 5(e)**) were also measured and included in the spectral library and are consistent with reported spectra [21] [22].

Sensitivity as a performance metric for this Mid-IR QCL system can be estimated by calculating the limit of detection (LOD) as described in the data analysis section. In this case we have measured a sample of 98.7 ppm EtOH gas standard (see sample preparation and measurements section). The estimated LOD for 98.7 ppm EtOH using the dominant peak signal at 1065 cm^{-1} was calculated as follows: $\text{LOD} = (C)/([\text{Signal}/\text{Noise}]/3) = (98.7\text{ ppm})/([0.046/0.000014]/3) = 0.090\text{ ppm} = 90\text{ ppb}$. This indicates that the current system has the capability to detect trace amounts of accelerants (at ppb levels) in fire debris.

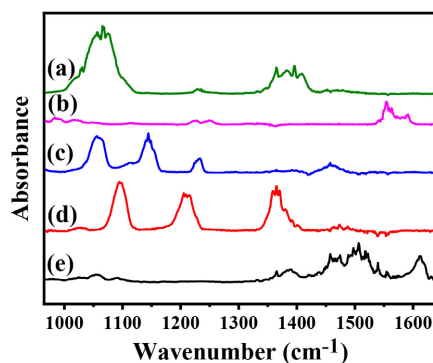


Figure 5. The absorbance spectra of additional accelerants and fuel components included in the library. (a) ethanol, (b) cleaning solvent, (c) banana oil, (d) MTBE and (e) xylene. Conditions as in **Figure 3**.

3.2. Fire Debris Spectra

The focus of this current study has been directed towards demonstrating the feasibility of using our portable QCL analyzer in identifying volatile accelerants in fire debris samples. Fire debris samples were prepared and measured as described in the materials and methods section. **Figure 6** presents the absorbance spectra of the ambient background collected in the surrounding area used to burn samples (**Figure 6(a)**) and that of the resulting paper sample presoaked with 92-octane gasoline, burned for several minutes then extinguished with water, placed in a bag, followed by measurement of accumulated vapors drawn from the bag (**Figure 6(b)**). The methodology for collecting and performing the fire debris measurements was described in the materials and methods section and illustrated in **Figure 2**. **Figure 6(b)** has not been background corrected. Clearly there is a background contribution from the fire burning area in addition to the inherent air/moisture background. **Figure 6(c)** presents the background corrected fire debris sample. For comparison, **Figure 7** provides the stacked plot of this background corrected absorbance spectrum of the fire debris sample (**Figure 7(a)**) with that of the 92-octane gasoline reference spectrum previously shown above in **Figure 3(a)** and **Figure 4(b)** (background corrected). The software correctly identified the gasoline accelerant in the fire debris sample. These results clearly demonstrate that gasoline could be detected on a fire debris sample. Of importance, this is the first time as far as we can tell that gasoline could be detected directly on an actual fire debris sample without any sample pre-treatment using a field-portable mid-IR (QCL) analyzer.

Additional fire debris measurements were performed on other samples containing potential accelerants that could be used to commit arson. **Figure 8** shows the results for diesel fuel. The same methodology as used in the above gasoline fire debris measurements was next applied to diesel fuel soaked on paper. **Figure 8(a)** shows the background corrected results of this fire debris measurement using diesel. Although the signal is weak, the diesel fuel accelerant could still be identified as shown by comparison to the diesel reference spectrum in **Figure 8(b)**.

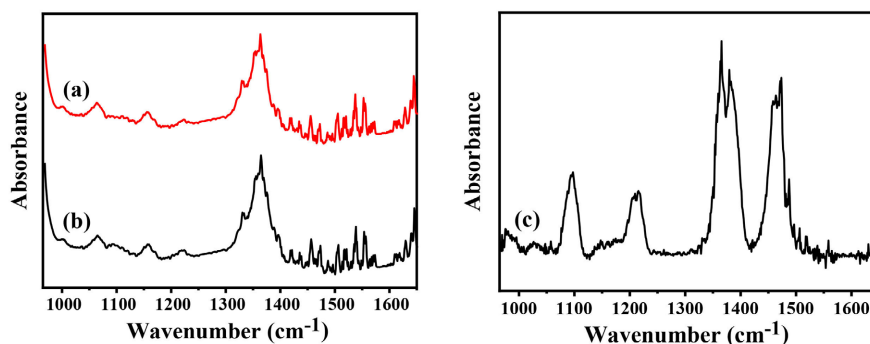


Figure 6. The absorbance spectra of (a) ambient air background collected in fire debris area, (b) of fire debris sample with 92-octane gasoline used as accelerant, and (c) is the resultant spectrum after background removed (as described in data analysis section). Measurement conditions as in **Figure 3**. The methodology used to make these fire debris measurements was illustrated in **Figure 2**.

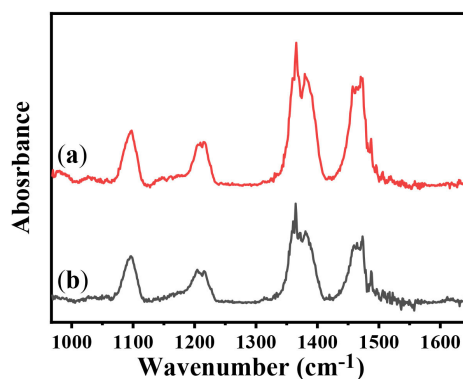


Figure 7. Comparison of absorbance spectra, (a) background corrected fire debris gasoline sample from **Figure 6(c)** above, and (b) background corrected gasoline reference spectrum shown in **Figure 3(a)** and **Figure 4(b)**.

We are considering two methods to help us improve overall sensitivity in the fire debris measurements. We are in the process of making a sample chamber that can safely heat the enclosed fire debris (between 60°C and 90°C). Heating fire debris samples have been previously discussed in reference to established GC-MS methodology [6]. This heating component when integrated into our analyzer will increase the vapor concentration of an accelerant present. We can also improve sensitivity by increasing the current 1.5 m pathlength, which according to Beer's law will allow the light to interact with a greater number of molecules as it traverses the longer distance.

As a final measurement for demonstrating feasibility of our portable QCL (mid-IR) system to identify potential accelerants in fire debris samples, we next measured ethanol as shown in **Figure 9**. The same methodology as used in the above gasoline fire debris measurements were similarly applied to ethanol soaked on paper. **Figure 9(a)** shows the background corrected results of this fire debris measurement using ethanol. Comparison to the ethanol reference spectrum in **Figure 9(b)** supports ethanol as the accelerant in this fire debris sample.

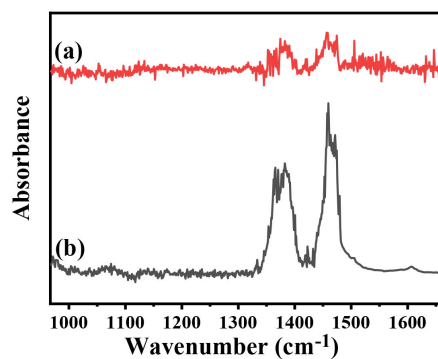


Figure 8. Comparison of absorbance spectra, (a) background corrected fire debris diesel sample, and (b) background corrected diesel reference spectrum shown in **Figure 3(c)**.

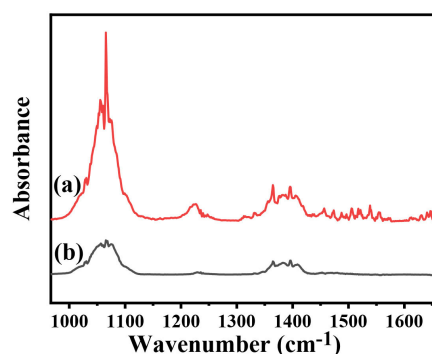


Figure 9. Comparison of absorbance spectra, (a) background corrected fire debris ethanol sample, and (b) background corrected ethanol reference spectrum shown in **Figure 5(a)**.

4. Conclusions

The results obtained in this work showed that the portable mid-IR QCL based system is well suited for detecting the presence of accelerants and ignitable liquids in fire debris samples. Here gasoline, diesel and ethanol were used to accelerate the combustion of paper and their vapors were successfully detected on the resulting fire debris samples that were burned and extinguished with water in a controlled laboratory setting. No complicated sample preparation and extraction were required.

Future studies will focus on implementation of components to increase sensitivity (increase path length of cell, and provide sample heating chamber), and a device (drying tube) to aid in removal of water vapor from the sample. In addition, the spectral library will be expanded, and different fire debris materials will be investigated (for example, wood, rug, soil, containers). A rigorous figures-of-merit study including quantitation, statistical analysis and reproducibility will also be carried out. Finally, we will collaborate with fire investigators to make measurements on actual samples involved in arson cases.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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