ADVANCEMENTS IN COLLECTION AND ANALYSIS OF FIRE DEBRIS-IMPROVED FIELD COLLECTION KITS WITH FAST GAS CHROMATOGRAPHY

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Abstract: Gas chromatography with mass spectrometry (GC-MS) is the primary tool used in fire debris analysis by forensic scientists. Improving the efficiency of this analysis through fast GC was the first aim of this work, and it utilized commercially available software to translate the current method and provide a starting point. The method has previously been used in increasing GC speed for pesticide analysis and is applicable if only translatable parameters are exchanged. The new fast GC method utilized a shorter GC column with a narrower bore, and adjustment of the injection volume and temperature ramp yielded an improved GC/MS method, decreasing the run time of the method by approximately half without a change in resolution or retention order. After new method development, the fast GC method was used in the development of a new collection technique, specifically an activated carbon strip field kit for use on ignitable gasoline. It was also applied to archived activated carbon strip case samples dating back to 2000. Currently, investigators at fire scenes collect liquid samples directly or with cotton swabs, creating evidence to maintain and house in fire debris cabinets. The developed field kit uses activated carbon strips directly, eliminating the need for maintaining cotton swab evidence after analysis, allowing efficient analysis of activated charcoal strips directly from the field with an improved GC/MS method.

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CHAPTER I

INTRODUCTION TO FIRE DEBRIS ANALYSIS

Background

In a South African cave over one million years ago, campfires flickered, and the controlled use of fire began.[1] Fire's earliest uses were for warmth, food preparation, and protection from predators, but as humans evolved so did the use of fire. The Aboriginal Australians used fire more than 100,000 years ago to regenerate soil and drive animals out of their habitats for hunting. Unfortunately, manmade fires not only enabled the advancement of mankind but eventually turned towards arson.[2] Fire, known to have the ability to destroy land and homes, instigated a high level of fear in landowners. In the 1700's in Britain, arson became a form of rural protest. Peasants who had limited access to resources would burn wealthy enclosed properties especially during seasons of high unemployment.[2] This rebellious practice continued through the centuries and has been custom in America even as recent as the George Floyd protests in 2020.[3] Although some incendiarism draws on this idea of using fires in protest, not all arson is conducted as an expression of grievances, but whatever the reason fire setting requires very few tools and little effort.[4]

Fire is an exothermic oxidation reaction, known as combustion, that occurs at a quick enough rate to produce heat and light. For a fire to occur, there are specific conditions which must be met. Two models exist to describe fire: the fire triangle and the fire tetrahedron (Figure 1). The fire triangle model illustrates that for a fire to occur three conditions must exist: heat, oxygen (an oxidizer), and fuel. If any one of these components are consumed or removed, the fire will be extinguished. The fire tetrahedron model adds a fourth condition that explains the sustainability of the fire, an uninhibited chain reaction.

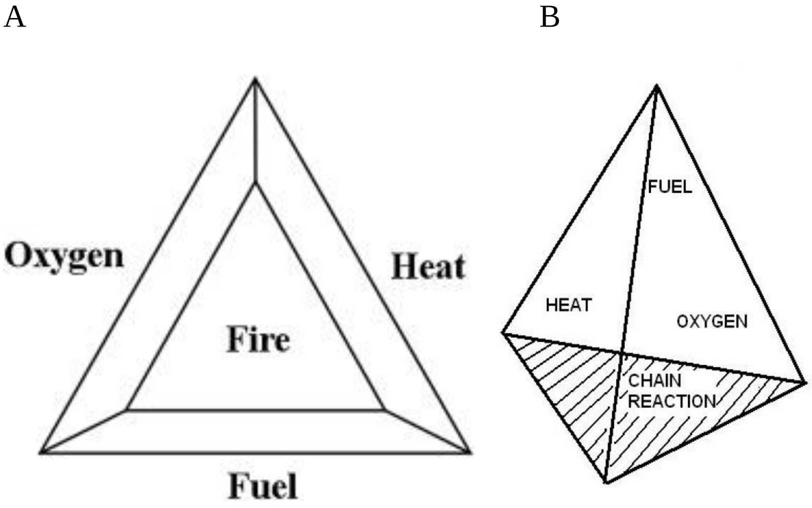


Figure 1. The two fire models (A) fire triangle (B) fire tetrahedron

The uninhibited chain reaction is a process by which the fuel is broken down into smaller units so it can combine with oxygen and become its own self-sustaining cycle. Fuel types include organic substances, inorganic substances, waxes, fats, and oils. In cases of arson, the most common fuel type is organic, typically wood, plastics, and gasoline. For liquid fuels such as gasoline to ignite, they must change from the liquid to gas phase and then be heated above their flash point (the lowest temperature at which a fuel gives off enough vapor to create a momentary flame). The point at which a liquid goes into the vapor phase is dependent on its vapor pressure and boiling point. For a solid fuel to be ignited, it too must be changed into its vapor phase. The transition of the fuel sources into their vapor phase only accounts for the physical transformation occurring prior to burning; chemical transformations are also taking place. Pyrolysis, the chemical transformation process necessary for combustion of solids, involves the breaking of the bonds of the compound through thermal degradation into smaller more volatile molecules. Pyrolysis products contribute to the chemicals recovered during analysis of fire debris samples.[5]

Although not included in the two models, substances can be used during arson or suspicious fires to accelerate the chemical reaction. An accelerant is a substance that has been deliberately introduced to a fire scene for the sole purpose of enhancing the fire. Accelerants do not make the fire burn hotter, but instead cause a more rapid increase in temperature, thereby facilitating the spread of the fire.[5] Solid accelerants exist: candles, powder mixtures, and flares, but are not as commonly used as liquid accelerants for criminal intent.[6] The American Society for Testing and Materials (ASTM) provides a classification system for ignitable liquids (Table 1).[7]

Table 1: ASTM Classification System

	Examples		
Classification	Light (C4-C9)	Medium (C8-C13)	Heavy (C9-C20+)
Gasoline, including E85	Fresh gasoline is typically in the range C4-C12		
Petroleum Distillates	Petroleum ether Cigarette lighter fluids Camping fuels	Charcoal starters *B* Paint thinners Dry cleaning solvents Mineral spirits Automotive parts cleaners Spray lubricants Lamp oils Deck sealers Varnishes Kerosene Insecticides	Kerosene Diesel fuels Charcoal starters Aviation fuels Insecticides Fuel additives Lamp oils Automotive parts cleaners

Table 1 (cont..)	**Examples**		
Classification	Light (C4-C9)	Medium (C8-C13)	Heavy (C9-C20+)
Isoparaffinic Products	Aviation gasoline Lighter fluids Charcoal starters	Charcoal starters Paint thinners Copier toners Mineral spirits Solvent cleaners Kerosene Lamp oils Gun oils	Spot cleaners Penetrating oils Insecticides
Naphthenic- Paraffinic Products		Charcoal starters Insecticide vehicles Lamp oils Automotive part cleaners Mineral spirits	Insecticides Lamp oils
Aromatic Products	Paint and Varnish removers Automotive parts cleaners Degreasing solvents Adhesives and adhesive removers	Automotive parts cleaners Degreasing solvents Specialty Cleaning solvents Insecticide vehicles Brush cleaners	Insecticides Adhesives
Normal-Alkane Products		Lamp oils Copier toners Wax cleaners	Lamp oils Carbonless paper forms Copier toners
Oxygenated Products	Solvents (for example, Alcohols, Ketones) Denatured alcohols or spirits		
Petroleum Products	Adhesive removers Lacquer thinners Enamel reducers	Paint thinners Mineral spirits Automotive parts cleaners Surface preparation materials	Automotive parts cleaners Fuel additives Specialty solvents Insecticides Paint thinners
Oil and Fat-based Products		Flooring Treatments Charcoal starter fluids Torch fuels	Olive oil, bacon grease, linseed oil, sunflower oil, canola oil, massage oils B100 Biodiesel
Single Compounds	Acetone, Ethanol, Hexane	Limonene, 2-butoxyethanol	
Mixtures	Enamel reducer Paint vehicles Lacquer thinners	Adhesive removers Roof sealants Mineral spirits	Lamp oils Insecticides

Although other ignitable liquids are listed on the classification system list (alcohols, ketones, turpentines), petroleum distillates are the most common accelerants.[6] Petroleum or crude oil is a hydrocarbon derivative that occurs naturally in the earth and is derived from animal and vegetable deposits. The elemental composition of crude oil varies depending on geographic location but is typically 83-87% carbon and 10-14% hydrogen with other elements present in low levels. Although the elemental composition of crude oil shows little variation, the types and proportions of compounds formed from these elements vary and influence the properties of crude oil. Varying proportions of paraffinic hydrocarbons, monocyclic and polycyclic naphthenes and aromatics exist naturally in the oil, therefore refining of the crude oil is necessary to render it marketable.[5]

Basic refinery operations are divided into four major steps: pretreatment, separation, size change, and configuration change. Pretreatment involves the removal of contaminants that can cause damage to the refinery structure, after which separation occurs. Separation is based upon boiling points of the fractions and occurs by conducting fractional distillation at atmospheric pressure and under vacuum. Once separated into boiling point fractions, chemical conversions and configuration changes are used to change sizes of fractions or rearrange molecules into the most desirable boiling point range products. These desirable products, within that boiling point range, coincide with the petroleum distillate ignitable liquids.[5] Detection, collection, and identification of these ignitable liquids in fire scenes are a valuable tool in forensic science.

Detection and Collection of Ignitable Liquids on Fire Scenes

Detection and collection of ignitable liquids begins on the fire scene and is handled by trained investigators. While distinctive physical markers such as pour patterns and localized burning are indicators of ignitable liquids, investigators use other tools including their senses, canines, and portable instruments for the detection of ignitable liquids on the scene. The human nose is a remarkable tool and can distinguish the smells associated with ignitable liquids; however, there are some downfalls. Smell is often subjective, weather dependent, and fatigues with over exposure to a scent or throughout the course of the day. Additionally, there is an inhalation risk to investigators in smelling unknown substances. The canine nose is more selective and sensitive, which has led to canine accelerant detection teams being commonly used. Canines can be trained over a large array of ignitable liquids, but training and upkeep can be costly and like a human's sense of smell, fatigue can set in for the canine. Furthermore, canines are not trained to distinguish between a petroleum distillate smell from an accelerant and pyrolysis products. A third resource for distinguishing smells at a fire scene is the use of a portable "sniffer" detector or "electronic nose", which is both reliable and inexpensive.[6] These electronic noses use a series of sensors to analyze the chemical compounds within an odor. These devices are more sensitive than human noses and are not affected by sense fatigue. However, they can give false positives of pyrolysis products and can be susceptible to humidity.[5] Other portable instruments such as gas chromatographs (GC) or mass spectrometers (MS) can be useful on fire scenes for rapid and discriminate information on ignitable liquids but can be bulky and expensive. Whichever method for detection is utilized, without confirmatory laboratory analysis, the testimony in court can be easily disputed.[6]

For the laboratory to analyze a sample from a scene, it must be collected effectively by the fire scene investigator. All items collected for accelerant analysis must be secured in a clean airtight container that does not contain any interfering compounds. The type of container is dependent on the sample collected and includes unused metal cans with a friction lid, or glass vials (Figure 2). Samples typically collected from a fire scene include carpet, wood, soil, and other building materials. The ideal samples are porous and either adsorbent or absorbent with a large surface area so that the most accelerant residue is obtained. As well as the suspected sample with accelerant, a comparison sample from another area, consisting of the same material, should be collected, and placed in a separate container.[6]

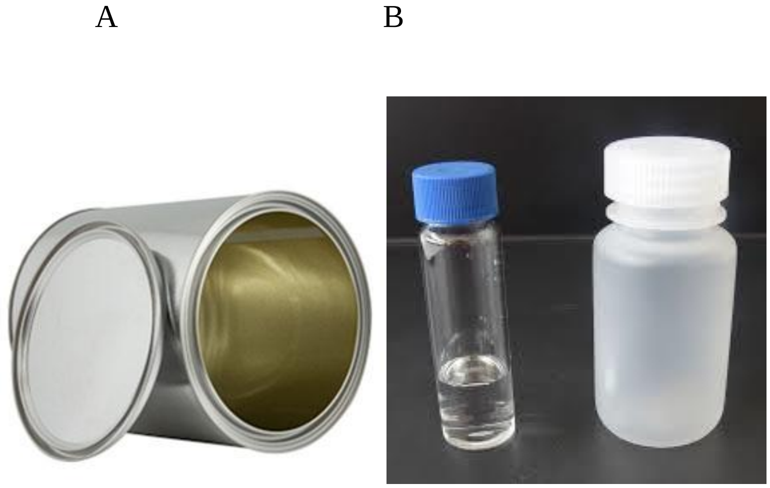


Figure 2. Sample fire debris collection containers (A) unused can (B) glass vial

Collection of Ignitable Liquids in the Laboratory

Once accepted into the laboratory for analysis, the volatiles of the ignitable liquid need to be extracted from the fire debris sample that was collected. This pre-treatment of samples is typically conducted with solvent or headspace-based extractions.[8] When considering an extraction method and sample preparation, care must be taken to reduce sample loss and to concentrate the analytes to increase sensitivity. The traditional extraction method is solvent extraction. This method involves the rinsing of a sample with a solvent to extract the ignitable liquid and often uses highly toxic organic solvents. Although it is still utilized in instances of non-porous samples (glass) and for higher molecular weight and lower volatile molecules, this technique has largely been replaced with headspace techniques which produce lower interference from background components of the sample.[6,9] The preferred method of extraction uses dynamic headspace sampling or passive headspace concentration.

Dynamic Headspace sampling, sometimes referred to as “purge and trap,” involves purging the heated headspace of the sample with an inert gas to remove the volatile components. The volatile components, now a gas stream, are then drawn through a tube (trap) packed with an adsorbent which traps the volatile compounds. These can then be desorbed by solvent extraction or thermal desorption. Although this method can be automated, it is still labor intensive, prone to contamination in the supply lines, and destroys the original sample.[6,5] Passive headspace concentration is the favored method of extraction due to its nondestructive nature and simplicity compared to dynamic headspace sampling.[6] In passive headspace concentration an adsorbent strip is suspended above the headspace of the sample and the sample is heated in an oven to 60^{o}C (Figure 3). The simplicity of the technique allows for different variations of sample containers and suspected ignitable liquids. Another advantage of this technique is the nondestructive aspect. Samples are left in their original state and can be reanalyzed if needed. A portion of the adsorbent strip can also be archived for further testing.[6]

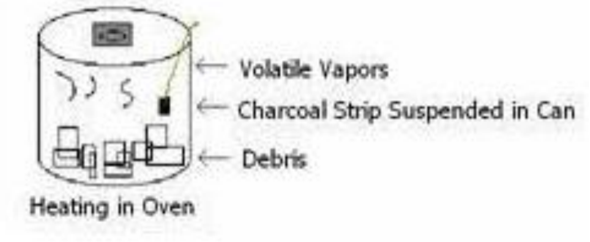


Figure 3. A schematic of a passive headspace concentration

The adsorbent strip most commonly utilized for passive diffusion in the fire analysis community is the activated charcoal strip (C-strip).[6] These strips are composed of a homogeneous mixture of activated charcoal and Teflon which binds the carbon into convenient strips.[9] The C-strip has an affinity for hydrocarbons, while not absorbing water or nitrogen. However, C-strips do not discriminate between target analytes and background pyrolysis products, leading to competitive adsorption.[6,9]

Once the C-strip is suspended into the container's headspace and heated, analytes release from the sample to the headspace and onto the adsorbent strip. Although the C-strip has micropores to increase surface area and adsorption, there is still a finite amount of adsorption sites. Thus, the C-strip size and heating time within the headspace of the sample are important considerations.[10]

Once the analytes are collected onto the adsorbent material, the analytes must be extracted (desorbed) for analysis. Not considering cost and safety, an ideal solvent for fire debris extraction has a low boiling point, does not interfere with the spectra of the desired analytes, could not potentially be used as an accelerant, has a high extraction efficiency for the analytes but not the sample (indirect solvent extraction), and is nonpolar in nature (unless looking for alcohol). Solvents historically considered were pentane, hexane, diethyl ether, tetrachloroethylene, and carbon disulfide (CS_2). [5] Tetrachloroethylene is said to be efficient when desorbing light petroleum distillates off adsorbents, and dichloromethane is considered a useful alternative as a less toxic solvent. Despite the research expressing its toxicity, CS_2 has been established as the most efficient solvent for desorption.[5] The efficiency of CS_2 is not based on the volatile substances (analytes) of interest being soluble in the solvent as much as its high adsorptivity for charcoal. The CS_2 displaces the analytes on the adsorbent strip and thus, releases the analytes from the absorptive strip.[5] Once separation of the analytes and extraction from the adsorbent strip occurs, confirmatory instrumental analysis must take place. The ASTM offers standards on the instrumental analysis of fire debris samples, mainly gas chromatograph-mass spectrometry (GC-MS) that the analyst can use as a reference when interpreting the unknown sample data.[7]

Identification of Ignitable Liquids in the Laboratory

In 1907, JJ Thompson at the University of Cambridge developed a device that created an electric arc within a container with gas. The device created positive ions in a range of masses that could be accelerated and manipulated in an electric field. Later, his assistant, Francis Astir, continued Thompson’s previous work and created the first mass spectrometer (MS) in 1922.[12] Within any MS, under identical conditions, compounds will always produce the same pattern of ions, that are unique for that compound. Unfortunately, if a mixture of compounds is introduced to the MS a spectrum of the combined ions is produced and difficult to interpret.[11]

Not until the early 1950's did James and Martin at the National Institute for medical Research introduce the gas chromatograph (GC) that allowed for the separation of mixtures into individual compounds based on the retention time.[11] The combination of the GC and the MS was thought to be a beneficial union that played off the strengths and weaknesses of each instrument. Original GC instruments utilized a packed column that were comprised of particles coated with a nonvolatile oil or wax. The mixture to be separated would be injected into a carrier gas, volatized, and then separated based on their affinity for the oil or wax that was on the packed column.[13] A GC utilizing a packed column operates under high pressure, because of the flow rate needed to push the carrier gas through the column, while the MS operates under vacuum, therefore a method to interface the two instruments had to be determined.[12] Packed columns were also not extremely selective, often compounds could not be separated from each other and produced one unresolved peak. Although they existed earlier, the alternative, capillary columns, did not become popular until the late 1970's. Capillary columns are open tube columns, typically 100-500 µm in diameter and 10-50 m long, coated with a stationary phase. The design of these columns allowed for lower flow rates therefore no need for an interface into the MS system and they offered better resolution of compounds.[13] Differences in chemical properties of compounds, typically boiling point, allow for the compounds to be separated as they travel the length of the column.[12] Capillary columns of 30 meters coated with 5% phenyl methylpolysiloxane with a 0.25 µm diameter are now standard use in fire debris analysis.[7] Pairing the separation power of the GC with the identification power of the MS allows for the separation of multiple analytes from a potentially mixed sample, then the identification of unique mass ions of each analyte within the sample[12] (Figure 4).

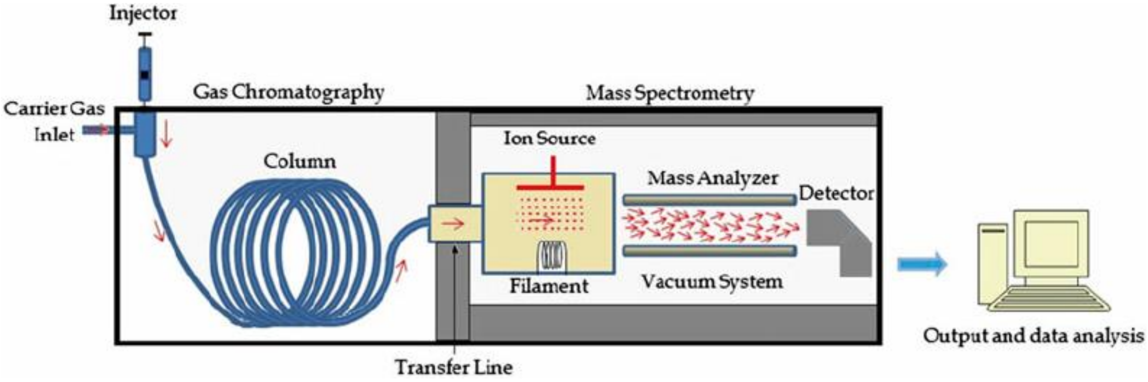


Figure 4. A schematic of a gas chromatograph-mass spectrometer[14]

Interpretation of Data

The GC data of retention time (the time it takes the compound to travel the length of the column) depends on the dimensions of the column (length, diameter, and thickness) and properties of the stationary phase coating.[12] Retention times of compounds of interest are available in spectral libraries and by analyzing reference standard materials within individual laboratories. Mass spectral data gives a mass to charge ratio of ions produced by individual compounds as they are separated by the GC. Compounds produce the same unique pattern of ions every time they are analyzed. These ions aid in the elucidation of the structure of the compound. Mass spectral data allows for the display of common ions shared by compounds with similar structural features. Key extracted ions (Table 2) are used in fire debris analysis to observe the alkane, alkene, aromatic, indanes, cycloalkanes, and fused ring compounds common to ignitable liquids used as accelerants.

Table 2: Mass Spectral Extraction Ions for Data Interpretation in Fire Debris Analysis

Compound Type	**Ions (m/z)**
Alkanes	57, 71, 85, 99
Aromatics	91, 105, 119, 133
Indanes	117, 131, 145
Cycloalkanes	55, 69, 83
Naphthalenes	128, 142, 156

Fast Gas Chromatography-Mass Spectrometry

With the advent of capillary columns, scientists in the early 60's invested time into researching faster GC techniques. At the 1960 International Symposium on GC, Desty and Goldup presented their theoretical ideas on speed- related aspects of separation.[15] Even with the research on faster GC methods, practical use of these techniques did not translate over into establishing routine methods utilized in analysis such as fire debris analysis.[15] This was due to the limited use of capillary columns at that time. The rigid glass made the columns difficult to install, they had limited availability, and no consistent method of production of the capillary columns existed. Moreover, changing over from a packed column to a capillary column required modification of the GC.[15] Once capillary column production, availability, and GC instruments improved, faster GC sample processing was accessible, but methods had already been developed without speed as a primary goal.[15] These slower methods are still in use in fire debris analysis and although they meet an analytical need may not be optimal for the laboratory. Fast GC becomes an interesting topic to discuss for its financial gains as forensic laboratories seek to decrease cost per analysis. Faster GC can lead to increased throughput, better utilization of equipment, and decreased time per case.[15] Also interest in quick field portable applications on crime scenes has increased. Implementation of fast GC applications could be an opportunity for a “quick look” at evidence on scene.

What are the best means by which to convert slower GC methods to their faster counterparts? Seven possible modifications exist to potentially increase the speed of GC analysis: increase the carrier gas flow rate, increase the temperature program heating rates, use a faster carrier gas, reduce the column length, reduce the column diameter, reduce the thickness of the stationary phase, and/or use a detector that operates at a lower outlet pressure.[15] Optimization of GC speed, though, comes at a cost. A balance must be obtained between speed, sample capacity, and separation. Any increase in one will cause minimization in the other two.

Changing a temperature program rate or carrier gas flow rate are simple changes within a method, but both have an optimal point of which the speed of the GC method cannot be increased any further without substantial loss of peak separation.[15] If the carrier gas flow rate is too high there is limited interaction with the stationary phase by analytes, causing poor peak separation and inversely if it is too low the analyte will spend too much time on the stationary phase causing peak broadening.[16] Both parameters are typically already optimized for a given method within laboratories if they are using a standard approved method for analysis.[7]

The carrier gas and the detector can influence speed. Relative speed of analysis for some common GC gases show hydrogen, then helium, followed by nitrogen then argon to be the order of fastest to slowest.[15] Hydrogen is often considered a safety concern and for this reason many laboratories utilize the next best option, helium. Converting from helium to hydrogen is not necessarily a simple or cost-effective means to increase GC speed. Also, completely changing a detector from one that is currently in use is not a cost or time effective means especially when standards are in place for the interpretation of forensic data that is based on specific detectors.

After the options are narrowed down, the dimensions and aspects of the column in use are left to increase GC speed. A narrower internal diameter column which also lends itself to a shorter column length can reduce the analysis time.

Research Overview

This research proposed to reduce the analysis time of fire debris samples from 30 minutes to 15 minutes by utilizing a shorter length and narrower internal diameter column, without changes in retention time or resolution of peaks within the data. To adapt the existing method, to a new method, for use with the shorter column, method translation software was used. Before method translation software, there was a concern that shortening the column, even though it might speed up the GC, could cause greater separation in some components, loss of separation in others, or even peak reversal in the chromatogram. With method translation software, these uncertainties were reduced or eliminated.[15] Wool and Decker have previously shown this to be an effective method in pesticide analysis, which like fire debris analysis, has numerous compounds within a sample.[16]

After validation of the new method a field kit for the collection of liquid gasoline on fire scenes was explored. Currently investigators at fire scenes collect liquid samples directly or with cotton swabs creating evidence to maintain and house in fire debris cabinets. The proposed field kit uses activated C-strips directly, eliminating the need for maintaining evidence after analysis and the use of passive headspace extraction for the extraction procedure in the laboratory. Removing the passive headspace analysis step from the analyst workflow shortens analysis time, since no heating is required. Finally, archived C-strip samples from case work ranging in years 2000-2020 were reanalyzed using the fast GC method with the 20 m, narrower bore column. This further validated the 20 m method and investigated the durability of archived C-strips and the conditions they have been stored under.

CHAPTER II

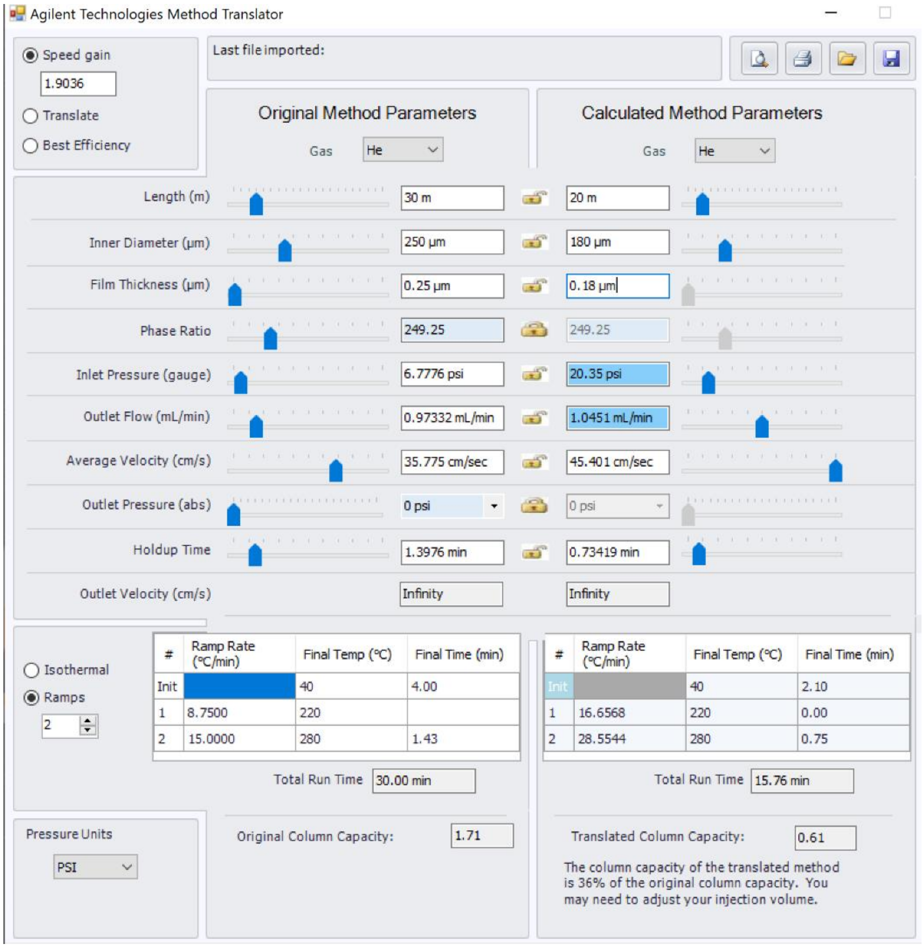
FAST GAS CHROMATOGRAPHY AND METHOD TRANSLATION FOR USE IN FIRE DEBRIS ANALYSIS

Introduction to Method Translation

In fast GC development a method can be developed from the beginning, or an existing method can be modified. Agilent Technologies has made available an online method translation tool that can translate GC methods for different parameters.[17] The method translation obtained yields a scaled version of the original method. If there are difficulties with an existing method other than speed, starting from the beginning might be warranted, but if not, translating the existing method is advantageous. Method translation is based on two chromatographic conditions: translatable changes (column dimension, carrier gas type, carrier gas flow rate, proportional changes in heating rates) and non- translatable changes (stationary phase of the column, phase ratio, initial and final temperature of the temperature program). Two methods will only be mutually translatable if both share the same non-translatable components and have a normalized temperature program. They will then also share the same peak elution order.[15]

The Agilent software takes all this into consideration and translates methods while attempting to keep the relative retention and selectivity the same as the original method.[16]

Initially, the analyst enters the original and new conditions of the method into the software to include column dimensions and carrier gas type. Three translation modes are available on the software; speed gain, translate, and best efficiency. Translate and best efficiency lock the ability to change inlet pressure, outlet flow, average velocity, and hold up time conditions on the software. The speed gain option keeps all items unlocked and allows for an analyst to type in the amount of speed gain desired. The translate option keeps the properties of the original and newly translated method as closely linked as possible. The best efficiency and speed gain options vary in the way carrier gas velocity is calculated. The best efficiency mode calculates carrier gas velocity by using the optimum linear velocity. This gives the maximum number of theoretical plates for maximum resolution but increases the analysis time as compared to the speed gain mode. The speed gain mode calculates the carrier gas velocity as maximum efficiency per unit of time causing some loss in resolution but a larger reduction in retention time therefore a faster analysis time.[16] For the original and new methods with varying column dimensions to be translatable they must share the same non-translatable components so even if the speed gain option is used the phase ratio must be kept the same and locked. After inputting the information, the original method is translated. A speed gain method translation was performed for a change in carrier gas flow rate without changing the column and then a final method translation was performed with the intent of cutting the analysis time in half. Smaller diameter, shorter GC column dimensions were used with the speed gain application to yield the final method translation (Figure 5).



● Speed gain 1.9036
○ Translate
○ Best Efficiency

	Original Method Parameters	Calculated Method Parameters
Gas	He	He
Length (m)	30 m	20 m
Inner Diameter (µm)	250 µm	180 µm
Film Thickness (µm)	0.25 µm	0.18 µm
Phase Ratio	249.25	249.25
Inlet Pressure (gauge)	6.7776 psi	20.35 psi
Outlet Flow (mL/min)	0.97332 mL/min	1.0451 mL/min
Average Velocity (cm/s)	35.775 cm/sec	45.401 cm/sec
Outlet Pressure (abs)	0 psi	0 psi
Holdup Time	1.3976 min	0.73419 min
Outlet Velocity (cm/s)	Infinity	Infinity

○ Isothermal
● Ramps 2

#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		40	4.00
1	8.7500	220	
2	15.0000	280	1.43

Total Run Time 30.00 min

#	Ramp Rate (°C/min)	Final Temp (°C)	Final Time (min)
Init		40	2.10
1	16.6568	220	0.00
2	28.5544	280	0.75

Total Run Time 15.76 min

Pressure Units PSI

Original Column Capacity: 1.71

Translated Column Capacity: 0.61

The column capacity of the translated method is 36% of the original column capacity. You may need to adjust your injection volume.

Figure 5. Final method translation with a shorter column using the speed gain function.

Materials

Standards obtained from Restek Chromatography include E1618, 25% weathered gasoline, 50% weathered gasoline, and 50% weathered diesel all in carbon disulfide (CS_2). Other substances analyzed were purchased at ACE Hardware and include Jasco Xylene, Klean Strip Adhesive Remover, Coleman Camp Fuel, Jasco VM8P Naptha, Klean Strip Paint Varnish Stripper, Jasco Turpentine, Tiki ready to Light Torch fuel, Ronsonol Lighter Fluid, Ultra-Pure Paraffin lamp oil soot less, smokeless, odorless, and kerosene. Activated charcoal strips (C-strip) from Albrayco technologies were used for extraction.

Treated lumber, carpet, carpet padding, paneling, siding, and shingles were all collected as scraps from a construction site. The 100% cotton cloth was obtained at a local Dollar General.

Method

All Restek standards were purchased ready to analyze. All other liquid substances were analyzed by placing approximately two drops of the liquid onto an inert substrate (Kimwipes) in a quart size unlined can. The extraction method was passive headspace with activated charcoal strips, suspended inside the can by paper clip and magnet and heated at 65 °C for 16 hours.[8] After 16 hours the C-strips were removed and eluted with CS_2. Approximately one half of the C-strip was analyzed on the original method. The other half was archived by placing inside a gas chromatograph-mass spectrometry (GC-MS) vial and sealed with a crimp top. These archived C-strips were later analyzed on new translated method. Kerosene was also examined by simply placing two drops into a GC-MS vial with CS_2. For all experiments a system blank using the same techniques and materials was analyzed.

Materials obtained for unburned and burned comparison were cut into approximately 1 inch x 1-inch pieces and placed into a quart size unlined can for passive headspace analysis, as described previously. Two cans were made for each sample type, one to run unburned and the other to run burned. As well an empty can was analyzed burned and unburned as a system blank. To simulate combustion, the following procedure was used (Figure 6):[18]

1. Place samples of the material in a can.
2. Place a lid tightly on the can and place three half centimeter holes (about the size of a standard size screwdriver) through the lid.
3. Place the can on a ring stand and heat the bottom with a propane/butane torch until smoke begins to evolve from the holes.
4. After 2 minutes of smoke evolution turn off the torch
5. Without waiting for the can to cool, rapidly remove the lid with holes and replace it with a solid lid.
6. Wait for the can to cool to room temperature.
7. Extract using passive headspace with activated charcoal strips.

Figure 6. Picture of a sample simulated combustion process.

As before, one half of the C-strip was analyzed on the original method and the other half was archived and later analyzed on the new translated method. All samples were analyzed once on the original method and the new translated method, except for shingles and carpet padding, which were ran a second time on the new translated method. Also, the new translated method was repeated on shingles with either a drop of gasoline or a drop of kerosene and on carpet padding with a drop of gasoline. Comparison of data was done using known retention times and similarities of extracted ion profiles. This method is qualitative only and therefore abundance of ions is relative to the sample.

Instrumentation

All substances were analyzed using an Agilent GC-MS method designed for ignitable liquids. The first method utilizes a 30-meter 5% phenyl methylpolysiloxane column and is an established column type within the field.[19] The second method utilizes a 20-meter 5% phenyl methylpolysiloxane column and was translated from the original method using the Agilent method translation program. These methods will be referred to as original method and 20 m method, respectively (Table 3).

Table 3: GC-MS Methods

Agilent 6890 Gas Chromatograph- Agilent 5973 Mass Spectrometer

		Original Method	20 m Method
Column	Type	J&W VF-5ms	J&W DB-5
	Dimensions	30 m x 0.25 mm x 0.25 µm	20 m x 0.180 mm x 0.18 µm
Mobile Phase	Carrier Gas	Helium	Helium
	Flow rate	1.0 mL/min	1.0 mL/min
Injection	Type	Liquid/Autosampler	Liquid/Autosampler
	Volume	1 µL	0.2 µL
	Split ratio	14:1	14:1
Temperatures	Injector	250 °C	250 °C
	Column	40 °C for 4 min 8.75 °C/min to 220 °C for 0 min 15 °C/min to 280 °C for 1.43 min	40 °C for 2.10 min 16.65 °C/min to 220 °C for 0 min 28.55 °C/min to 280 °C for 0.75 min
	Transfer Line	280 °C	280 °C
	Quadrupole	150 °C	150 °C
	Source	230 °C	230 °C
Mass Spectrometer	Scan range	41-354 amu	41-354 amu
Total Run Time		30 min	15.76 min

Carrier Gas Flow Rate

Keeping all other parameters shown in Table 3 the same, including the column dimensions, a method translation was performed changing only the carrier gas flow rate to determine how much this parameter alone would change the analysis time of the method. The subsequent temperature ramp and total run time change is shown in Table 4.

Table 4: GC-MS Method with Change in Carrier Gas Flow Rate
Agilent 6890 Gas Chromatograph- Agilent 5973 Mass Spectrometer

		Original Method	20 m Method
Mobile Phase	Carrier Gas Flow rate	Helium 1.0 mL/min	Helium 2.0 mL/min
Temperatures	Injector	250 ^{o}C	250 ^{o}C
	Column	40 ^{o}C for 4 min 8.75 ^{o}C/min to 220 ^{o}C for 0 min 15 ^{o}C/min to 280 ^{o}C for 1.43 min	40 ^{o}C for 2.44 min 14.32 ^{o}C/min to 220 ^{o}C for 0 min 24.55 ^{o}C/min to 280 ^{o}C for 0.87 min
	Transfer Line Quadrupole Source	280 ^{o}C 150 ^{o}C 230 ^{o}C	280 ^{o}C 150 ^{o}C 230 ^{o}C
Total Run Time		30 min	18.32 min

The carrier gas flow rate was increased from 1.0 mL/min to 2.0 mL/min based on the speed optimized flow calculations performed that are dependent on the column length and inner diameter.[15] The research on fast GC shows that increasing the flow rate will lead to a decrease in run time, but at a cost of efficiency greater than changing the column length and inner diameter.[15] To showcase this phenomenon a sample of 50% weathered gasoline and E1618 standard were analyzed on the original method and then on the translated 2.0 mL/min carrier gas flow rate method. The 2.0 mL/min flow rate method showed a decrease in efficiency for both samples and a loss of any resolution between the 1,2,4 trimethyl benzene and the decane peak (Figures 7 and 8). Subsequently all experiments were performed at a 1.0 mL/min carrier gas flow rate.

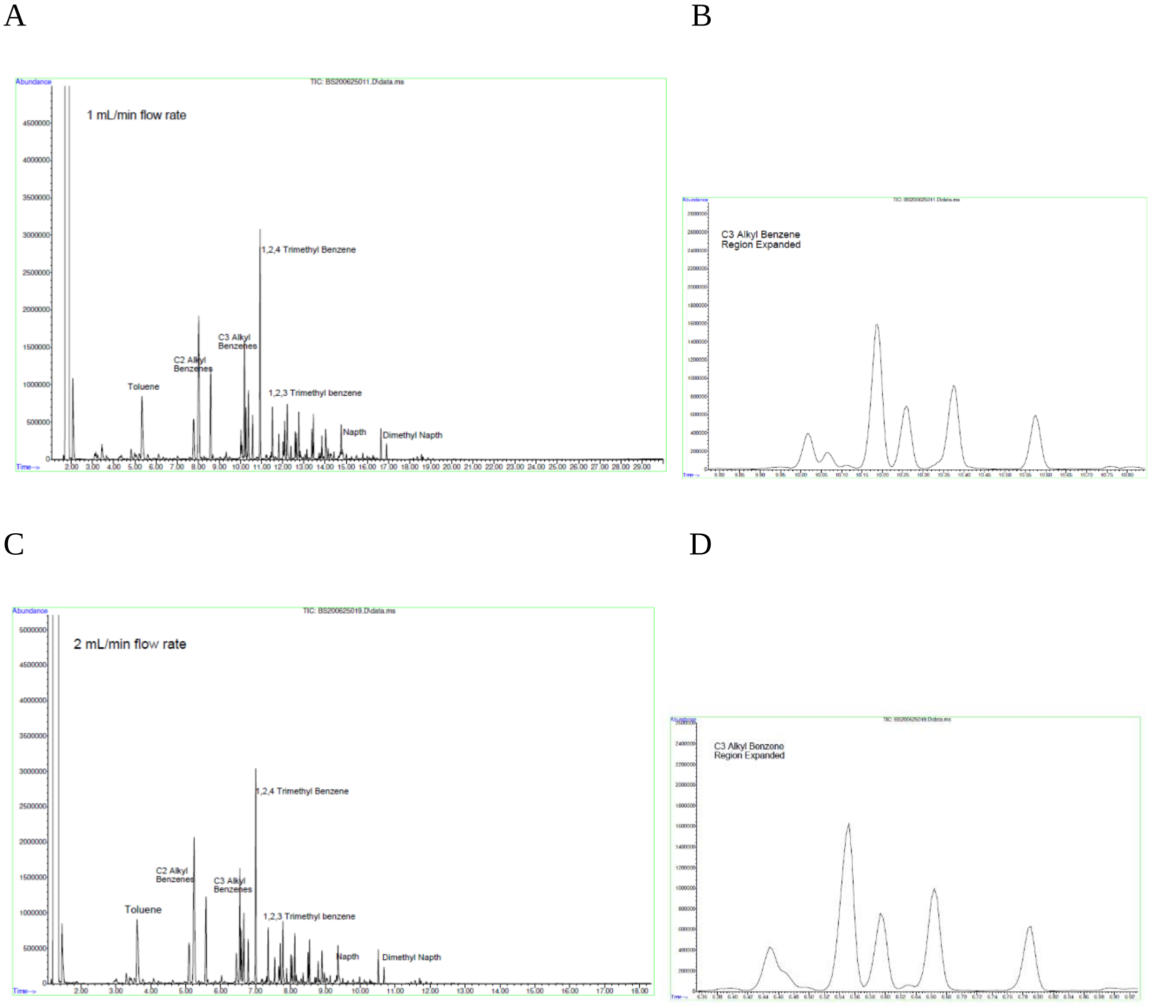


Figure 7. Comparison of carrier gas flow rates on 50% weathered gasoline (A) TIC at 1.0 mL/min (B) Expanded C3 alkyl benzene region at 1 mL/min (C) TIC at 2 mL/min (D) Expanded C3 alkyl benzene region at 2.0 mL/min.

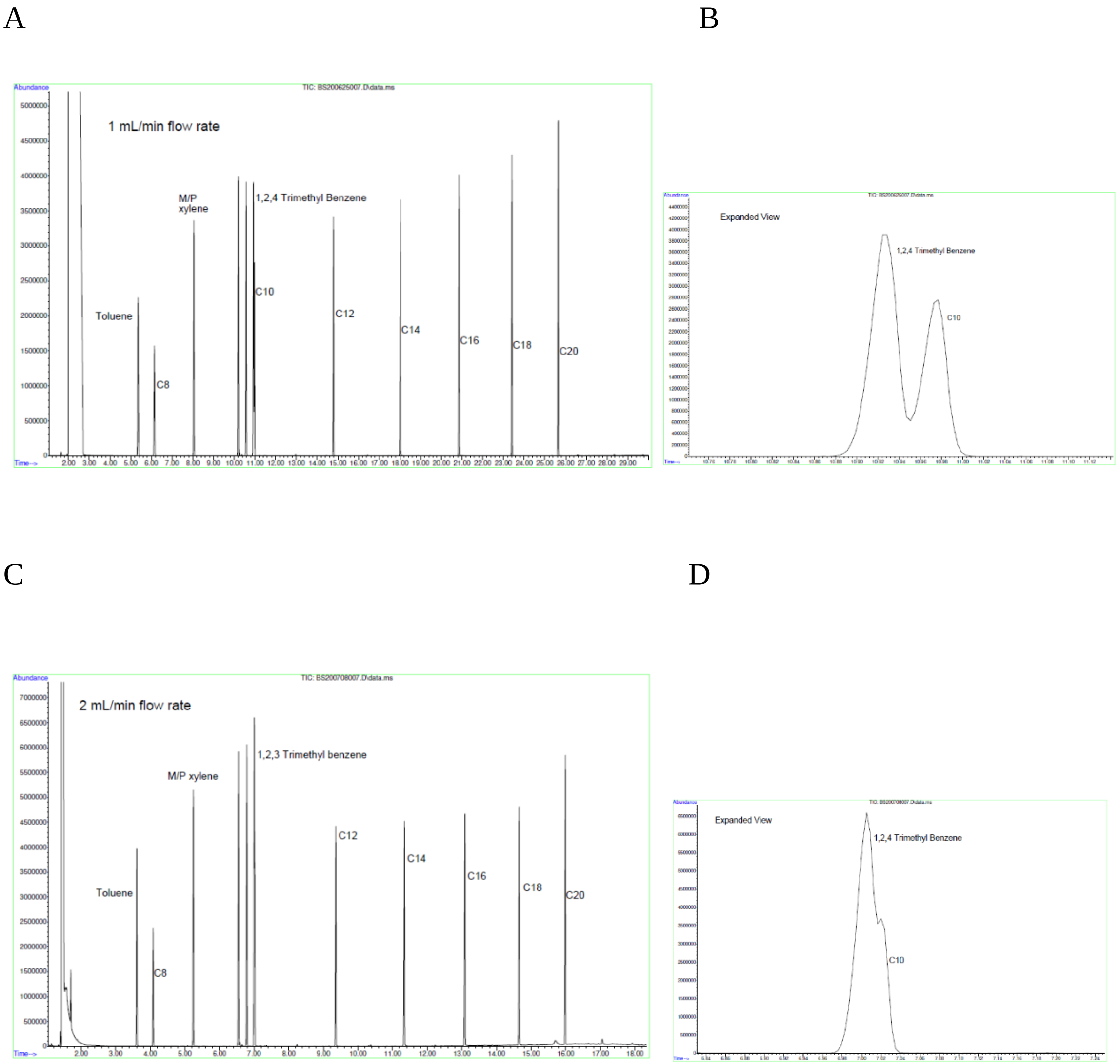


Figure 8. Comparison of carrier gas flow rates on an E1618 standard (A) TIC at 1.0 mL/min (B) Expanded 1,2,4 trimethylbenzene/C10 region at 1 mL/min (C) TIC at 2 mL/min (D) Expanded 1,2,4 trimethylbenzene/C10 region at 2.0 mL/min.

Injection Volume

The compromise of moving to a shorter column length with a smaller diameter is the sample capacity. If the amount of solute reaching the column is not reduced proportionately then the peaks become distorted. The drawback is that injecting less gives a greater signal to noise because there is less solute to be detected.[15] For fire debris cases in which the solutes are concentrated onto an activated charcoal strip for analysis, signal to noise is not an issue because they are concentrated on the C-strip. After method translation an injection volume of 1 µL was attempted using a 50% weathered gasoline standard. Peaks within the data were broad and not well defined (Figure 9); therefore, an injection volume of 0.2µL was attempted and utilized for all other experiments.

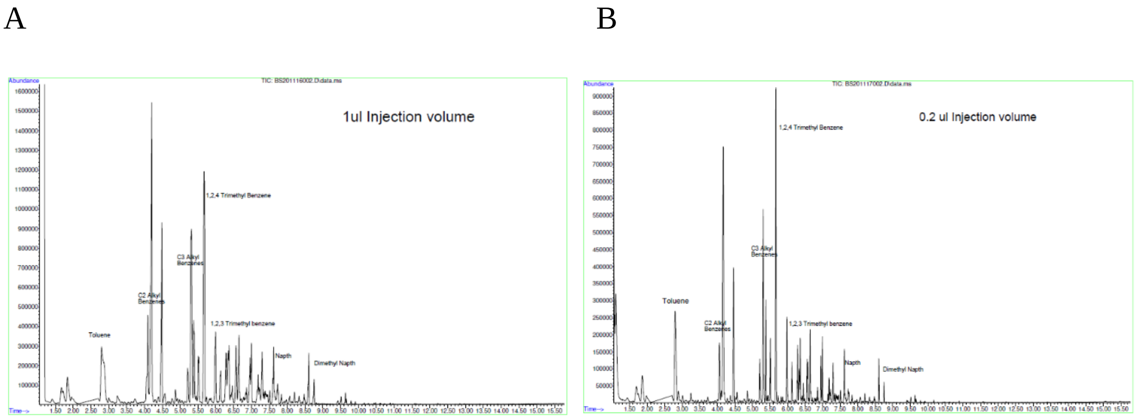


Figure 9. Comparison of injection volumes using the translated method on 50% weathered gasoline (A) 1µL injection (B) 0.2 µL injection.

Results and Discussion

To validate the method based on qualitative analysis, the following were evaluated: accuracy (degree of exactness), intermediate precision (over different days), repeatability (precision over short periods of time), and selectivity (ability to discriminate a particular analyte in the presence of interferences).

To evaluate the accuracy of the method to determine the presence of an ignitable liquid under the accepted ASTM classifications,[7] all above listed substances were analyzed on the original method then subsequently analyzed under the new conditions with the 20 m method. All substances analyzed were consistent between methods and are shown in appendix A. Although consistent between methods, one difference in the E1618 spectra was better separation between the 1,2,4 trimethylbenzene and the C10 peaks when analyzed on the 20m column (Figure 10). A longer column in general should increase separation due to the increase in theoretical plates but because of the longer run time, peak broadening occurs. On the 20m column, the narrower peaks allow for the separation of the 1,2,4 trimethylbenzene from the C10.[15]

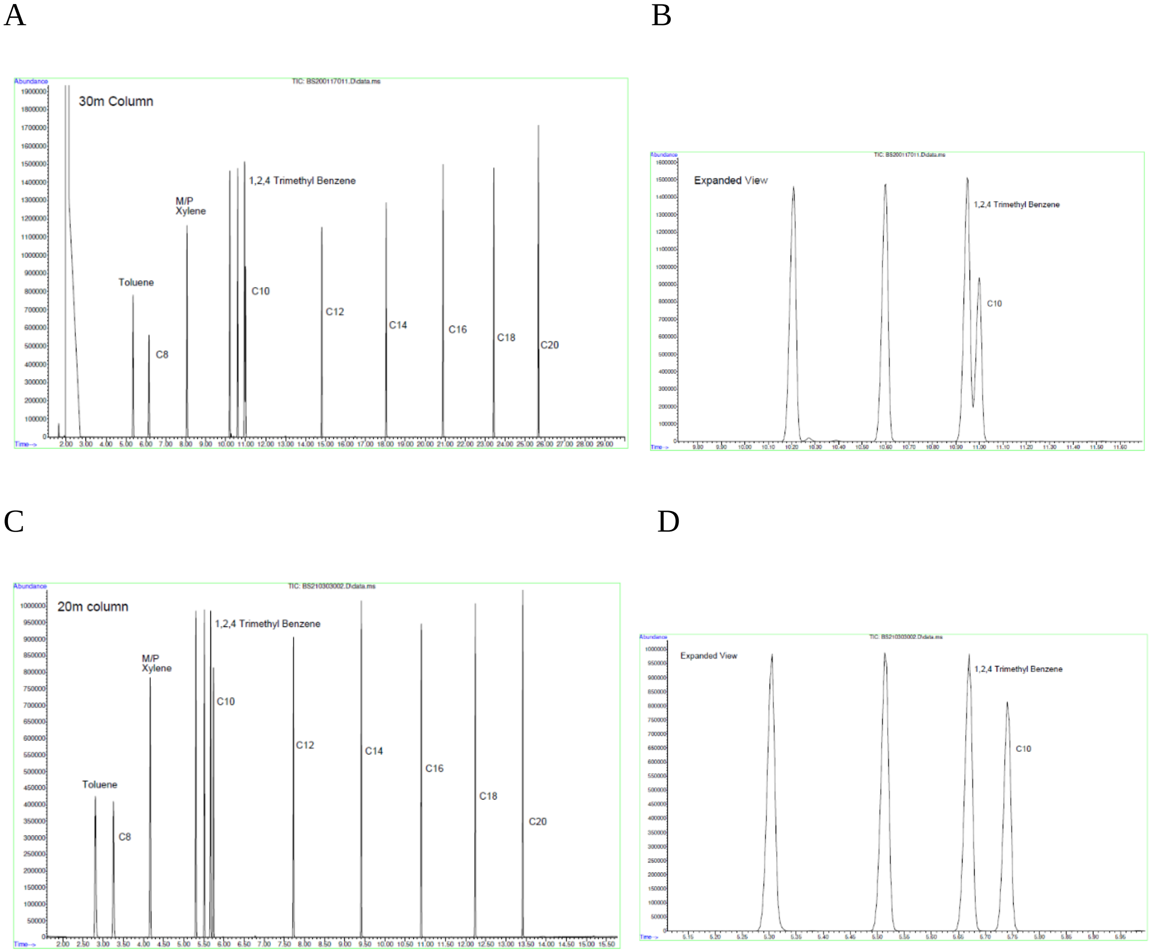


Figure 10. Comparison of the GC spectra of an E1618 standard on the 30m column versus the 20 m column method (A) TIC on 30m column (B) expanded view of the 1,2,4 trimethylbenzene/C10 region on the 30m column (C) TIC on the 20m column (D) expanded view of the 1,2,4 trimethylbenzene/C10 region on the 20m column.

To evaluate the intermediate precision and repeatability of the 20 m method, 50% weathered gasoline, 25% weathered diesel fuel, kerosene, and E1618 were analyzed at least three times within the same day and at least three times over different days with the same results. These standards were chosen due to availability and the frequency in which they are seen in actual case samples. Two GC spectra on different days of the E1618 standard can be seen in Figure 11.

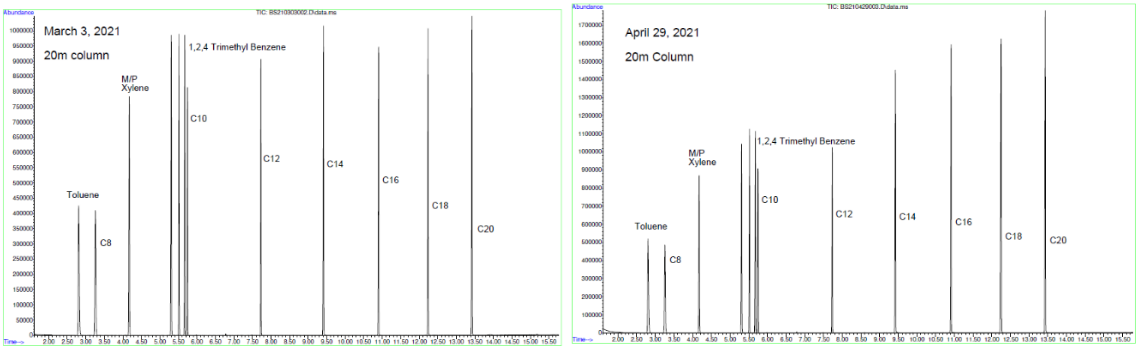


Figure 11. Two GC spectra on different days of the E1618 standard on the 20m column

The selectivity or the ability of the method to discriminate between the analytes of interest and interferences were evaluated by analyzing common materials found in fire debris samples. The materials commonly found in household fires such as wood, carpet, and shingles undergo pyrolysis when heated and produce substrates that can possibly interfere with common analytes in accelerant data. Treated lumber, carpet, carpet padding, paneling, siding, and shingles were analyzed unburned and burned, then compared to the original method. Interference does exist in the GC spectra of some materials, but this is well documented for the original method, and pattern burning attributed to shingles has been well documented.[18,20] The same interferences were observed for the 20 m method as had been previously seen in literature (Table 5).[18,20] No additional interferences occurred. Of note is the overlap of styrene with ortho xylene seen for both column length methods in siding, carpet, and carpet padding, and the presence of pinene in treated wood. The GC spectra and extracted ions for burned carpet padding and burned carpet padding spiked with gas are shown in Figure 12 as a representation of the overlap of styrene with ortho xylene. Although styrene can be seen in the GC spectra, it does not contain ion 91 of the extracted ions utilized in fire debris analysis, and therefore can easily be discounted as ortho xylene.[21] Figure 13 shows the aromatic extracted ion profile of burned carpet padding with no styrene peak in the ion 91 profile at the 4.45 retention time. Pinene does not overlap with any key compounds of gasoline but does show up in the aromatic extracted ions so should be considered when looking at compound ratios (Figure 14). All data for materials burned and unburned were consistent between methods and is shown in appendix B.

Table 5: Unburned Products and Pyrolysis Products of Note in Some Common Household Construction Items on the 20 m Method

Item	Unburned	Burned
Carpet	NA	Styrene and Benzaldehyde
Carpet Padding	Toluene, ethyl, meta/para, ortho xylene, C3alkyl benzenes	Addition of styrene and naphthalene
Siding	NA	Styrene and Benzaldehyde
Treated wood	Pinene	Pinene
Paneling	Pinene	Addition of phenols which look like an alkane pattern
Shingles	distillate pattern	distillate pattern
100% cotton cloth (new)	NA	NA

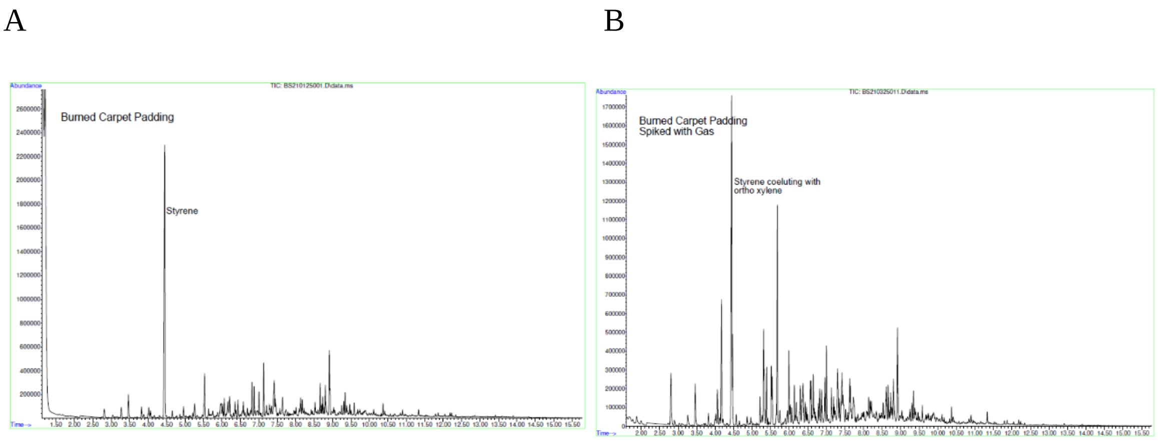


Figure 12. Co-elution of styrene with ortho xylene on the 20m method in burnt carpet padding (A) burnt carpet padding only (B) burnt carpet padding spiked with gasoline.

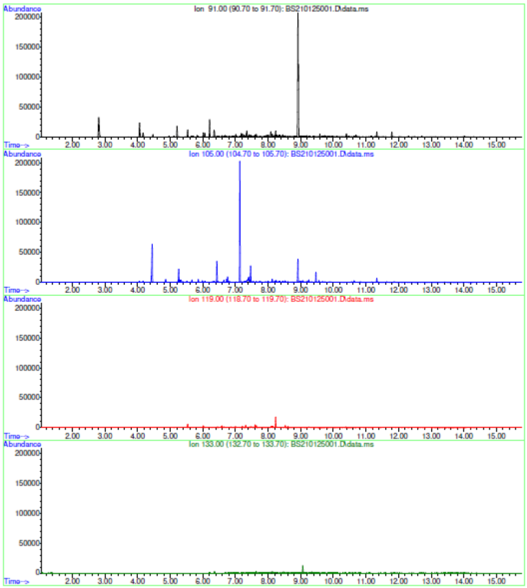


Figure 13. The extracted ion profile of burnt carpet padding.

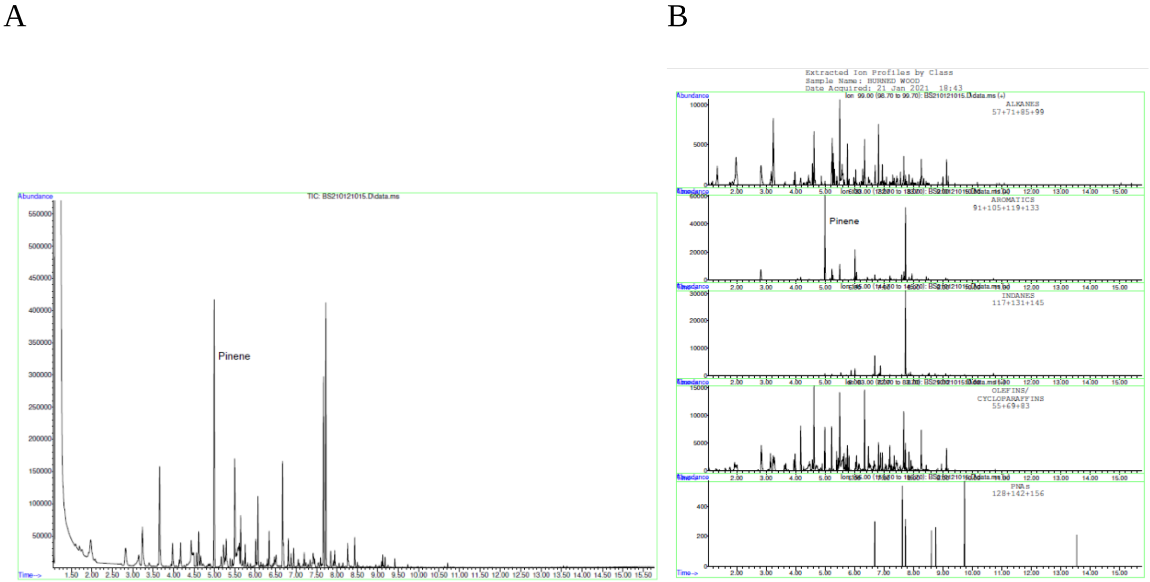


Figure 14. GC-MS data showing the presence of pinene in treated wood on the 20m method (A) TIC (B) extracted ion profile.

As a final validation step, actual case work was analyzed on the original method and the new 20 m column method with consistent results. One example of this case work consisted of a piece of charred wood from a burnt house submitted to the lab for analysis. The sample was submitted in a sealed airtight can as a K9 alert. The sample was extracted with the passive headspace concentration method and one half of the C-strip was ran on the original method while the archived other half was analyzed on the 20 m method. For both methods styrene co-eluted with ortho xylene, and pinenes were present as pyrolysis products from the wood. The heavy petroleum distillate pattern coeluted under the pyrolysis products but was easily visible when the extracted ion profiles were investigated (Figures 15 and 16). Since installation of the 20m column, over 50 case samples have been completed on the 20 m method.

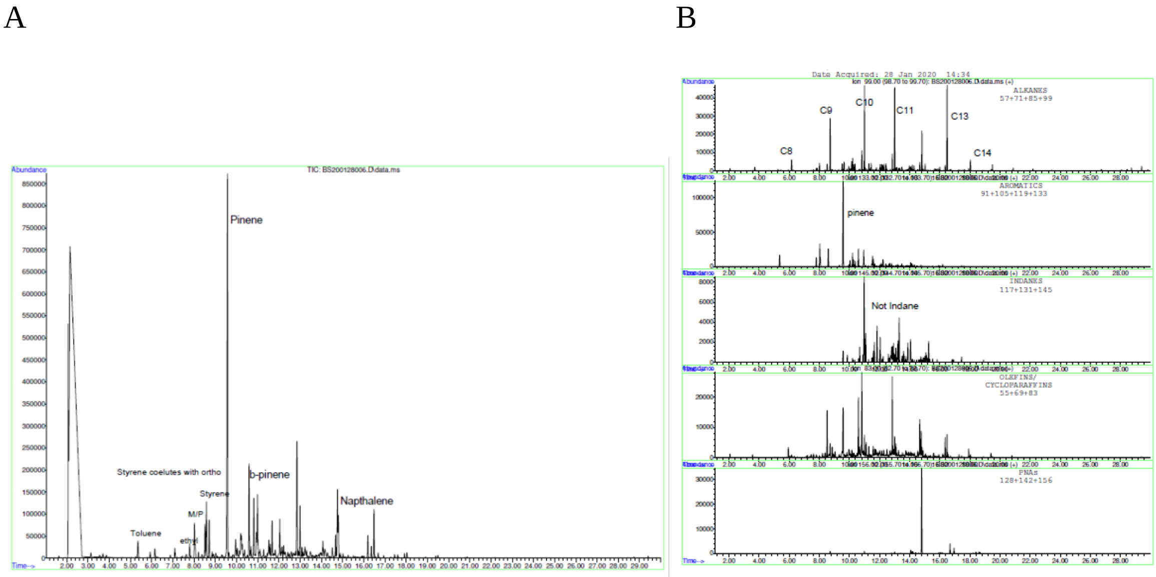


Figure 15. GC-MS data of a case sample of burnt wood with a heavy petroleum distillate on the original method (A) TIC (B) extracted ion profile.

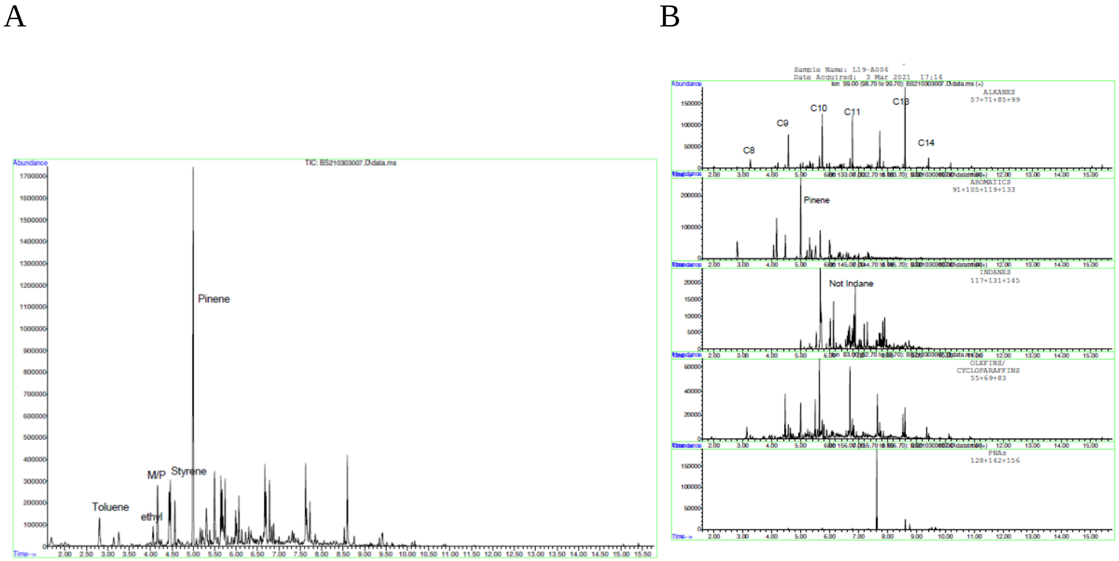


Figure 16. GC-MS data of a case sample of burnt wood with a heavy petroleum distillate on the 20m method (A) TIC (B) extracted ion profile.

Conclusion

Utilization of a 20-meter column is a valid means by which to speed up the analysis time of fire debris samples on the GC-MS instrumentation. The readily available and easily understandable Agilent method translator provides the analyst an excellent starting point to convert a standard temperature program from a 30-meter column to a 20-meter column equivalent, essentially cutting the analysis time on the GC-MS in half.

CHAPTER III

ACTIVATED CHARCOAL STRIPS AS A POSSIBLE FIELD KIT FOR THE COLLECTION OF LIQUID GASOLINE ON FIRE SCENES

Introduction

The two most common means by which to collect liquid accelerant on fire scenes are soaking up the liquid with cotton gauze then placing in a vapor tight unlined paint can or placing the liquid in a glass vial.[22] Inherent in these two collection methods are larger items of evidence that need to be transported in investigator vehicles (cans), and the production of a piece of evidence that must be maintained even after analysis. In the case of actual liquid in a glass vial, the evidence must be maintained in a fireproof cabinet and over-packed so that the glass will not break.

When liquid samples of an accelerant are submitted to a laboratory for analysis, traditionally a passive headspace extraction is performed followed by elution with a solvent. Passive headspace extraction entails the use of an adsorbent material, activated carbon, suspended over the sample, heated in an oven, and then collected for elution by a solvent.[8] The heating portion of the procedure is the most time consuming at approximately 16 hours. The field kit proposed for investigators in this research would eliminate the need for the passive headspace extraction and allow the analyst to simply elute the activated carbon strip (C-strip) submitted by the investigator and analyze the sample instantly. This research will discuss a possible field kit to be given to investigators comprised of C-strips that can directly adsorb ignitable liquids, namely gasoline, and then be directly analyzed upon receipt by the Fire Debris Analyst.

Methods and Materials

Phase 1: Laboratory Testing: Time and Size Trials

A 10 mL test tube was filled approximately one third full of 50% evaporated gasoline, closed, and allowed to reach ambient temperature of the laboratory (70°F). C-strips from Albrayco technologies were suspended by wire inside the test tube right above the liquid level (in the vapor) without touching the liquid. Time of exposure of the C-strip was investigated at 5 minutes, 90 seconds, 60 seconds, 30 seconds, 15 seconds, and 10 seconds. Except for the 5-minute time, which was only attempted once, all other times were examined for three consecutive days. On the first day each time was also replicated three times in a row. For the time trial phase of the experiments the C-strips were folded and cut in half and only one half was suspended over the gasoline in the test tube. Size of the C-strip was examined at lengths of ⅛ inch, ¼ inch, ½ inch, and ¾ inch. The C-strips come packaged as ¾ inch x ¼ inch strips and were measured and cut to the desired lengths in the laboratory. At each size the C-strip was suspended by wire for 60 seconds and replicated twice over the course of two days. During each experiment a C-strip utilized as a control was placed beside the test tube of 50% evaporated gasoline then analyzed, to ensure immediate contamination of the C-strip did not occur.

Immediately after exposure the C-strip was placed into a gas chromatograph mass spectrometer (GC-MS) vial with a 0.4 mL liner and extracted with carbon disulfide (CS_2). Experiments were run on multiple days over the course of two weeks to determine the optimal collection and testing parameters before sending field kits out with investigators.

During the experiments a C-strip was hung near the lab bench out in the open and another C-strip was hung sealed in a Kapak bag to determine susceptibility to contaminants at room temperature. The Kapak bags were ProAmpac brand bought from VWR International and in all instances of use were cut to just larger than the size of the C-strip. At the end of two weeks, when gasoline had been opened within the lab for at least seven of the 14 days, the C-strips that were hung near the lab bench were analyzed. C-strips were also placed in the oven heated at 60^0C for 16 hours, with sample cases of fire debris containing gasoline. One C-strip was sealed in a Kapak bag, and the other was left loose.

Phase 2: Initial Field Testing

An initial field kit sealed inside a Kapak bag and consisting of a larger ½ inch C-strip and smaller ¼ inch C-strip, copper wire, and two labeled ziploc bags (sample and blank) were given to three different investigators from two different agencies along with these directions:

Directions:

Please keep this sealed kit in your vehicle where you would normally keep your arson investigative tools for approximately a month. After a month at your convenience:

1. Open the sealed package.
2. Place the smaller C strip into the ziploc labeled blank.
3. Take the larger C strip and put it on the copper wire so it will not fall off.
4. Hold the larger C strip into the headspace of liquid gasoline in a container (this would correspond to holding the C strip right above the liquid level without touching the liquid) for approximately 60 seconds.
5. Please note the container the gasoline was in, if you conducted this indoors or outside, the date you did this and the approx. temperature at the time.
6. Place the sample C strip into the ziploc labeled sample.
7. Please deliver the items to me.

Thank you for participating. I would like to hear your feedback of this on-site gasoline sampling technique versus others you have used such as soaking a cotton strip or taking an actual sample of the liquid. Please call me with any questions.

Phase 3: Final Field Testing

The final field kit (Figure 17) was comprised of the same size C-strip pieces previously used but sealed separately this time in small individual Kapak bags labeled blank and sample. The sample piece was then placed in a glass vial then inside a plastic over pack bottle while the blank piece was placed inside the plastic bottle. The final field kit was given to six different investigators from two different agencies along with these directions. One field kit was also left in the laboratory environment:

Directions:

Please keep this sealed kit in your vehicle where you would normally keep your arson investigative tools for approximately a month. After a month at your convenience:

1. Inside the plastic bottle you will find a glass vial that contains two sealed packages each containing a C-strip.
2. Put on fresh gloves and remove the sealed package labeled blank. Place to the side-DO NOT OPEN- this is the control sample.
3. Take out the second sealed package, open, and hold into the headspace of the liquid gasoline in a container (this would correspond to holding the C-strip right above the liquid level without touching the liquid) for approximately 60 seconds.
4. Please note the container the gasoline was in, if you conducted this indoors or outside, the date you did this and the approx. temperature at the time.
5. Place the sample C-strip back into the glass vial then place the glass vial back into the plastic bottle. Leave the blank sealed C-strip separate, DO NOT place the blank back in the vial with the sample. DO NOT touch the blank with dirty gloves.
6. Please deliver the items to me.

Thank you for participating. I would like to hear your feedback of this on-site gasoline sampling technique versus others you have used such as soaking a cotton strip or taking an actual sample of the liquid. Please call me with any questions.

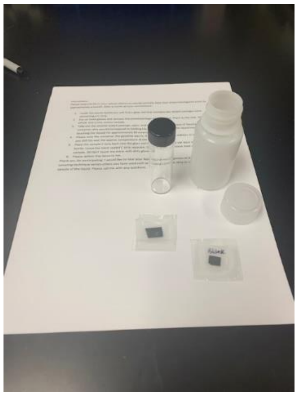


Figure 17. Final field-testing kit distributed to Investigators.

Instrumentation

All samples were analyzed using the GC-MS method designed for ignitable liquids on a 20-m column (Table 6).

Table 6: GC-MS Method
Agilent 6890 Gas Chromatograph- Agilent 5973 Mass Spectrometer

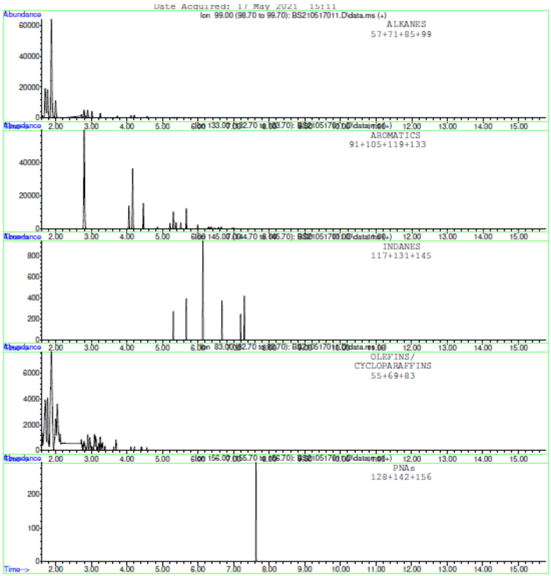
Column	Type	J&W DB-5
	Dimensions	20 m x 0.180 mm x 0.18 um
Mobile Phase	Carrier Gas	Helium
	Flow rate	1.0 mL/min
Injection	Type	Liquid/Autosampler
	Volume	0.2 µL
	Split ratio	14:1
Temperatures	Injector	250 °C
	Column	40 °C for 2.10 min 16.65 °C/min to 220 °C for 0 min 28.55 °C/min to 280 °C for 0.75 min
	Transfer Line	280 °C
	Quadrupole	150 °C
	Source	230 °C
Mass Spectrometer	Scan range	41-354 amu
Total Run Time		15.76 min

Results and Discussion

Phase 1: Laboratory Testing: Time and Size Trials

The first part of phase 1 consisted of determining the optimal amount of time to suspend the C-strip over the gasoline. 10, 15, 30 60, 90 seconds and 5 minutes were the times investigated. Extracted ion profiles for each time were examined (Figure 18). For all times the lighter molecules from gasoline including toluene, ethyl, meta/para, and ortho xylene, the C3 alkyl benzene group, 1,2 4 trimethylbenzene, 1,2,3 trimethylbenzene, indane, and naphthalene were present. The higher molecular weight methyl naphthalenes were only present in times greater than 15 seconds. The higher molecular weight compounds 1,2,4,5 tetramethyl benzene, 1,2,3,5 tetramethylbenzene, and the dimethyl naphthalenes were never present in any of the spectra. As expected, a longer suspension time yielded an increased abundance of compounds, but the spectra were always skewed toward lighter molecular weight compounds with toluene being the largest in each spectrum. A suspension time of five minutes was investigated to determine if a longer suspension time would allow the higher molecular weight molecules to be adsorbed onto the C-strip. The spectra still showed the same lighter molecular weight molecules and did not have any higher molecular weight molecules even after suspension of the C-strip for 5 minutes. Ultimately an optimal suspension (collection) time of 60 seconds was determined to be suitable to capture all compounds that could be obtained while not having the investigator hold the C-strip over the liquid for a long length of time (Figure 19).

A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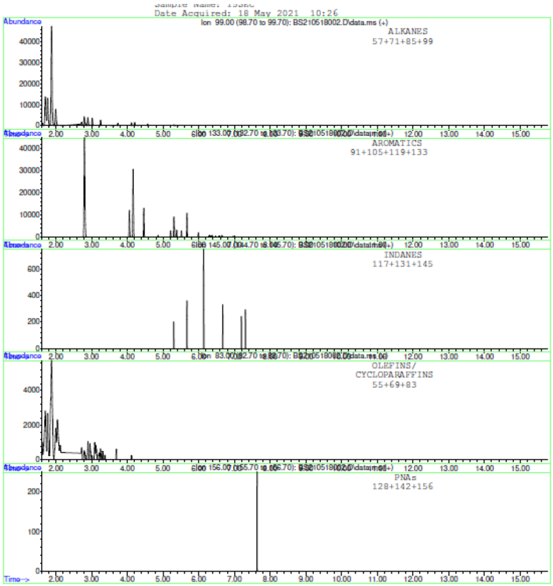
PNAs
128+142+156
Abundance
Time-->

B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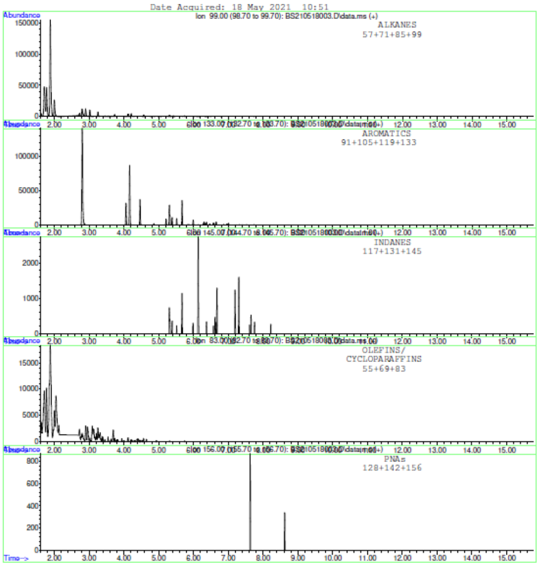
C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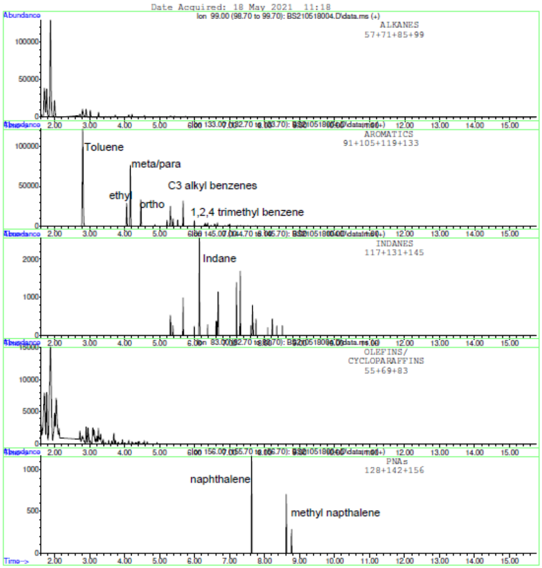
D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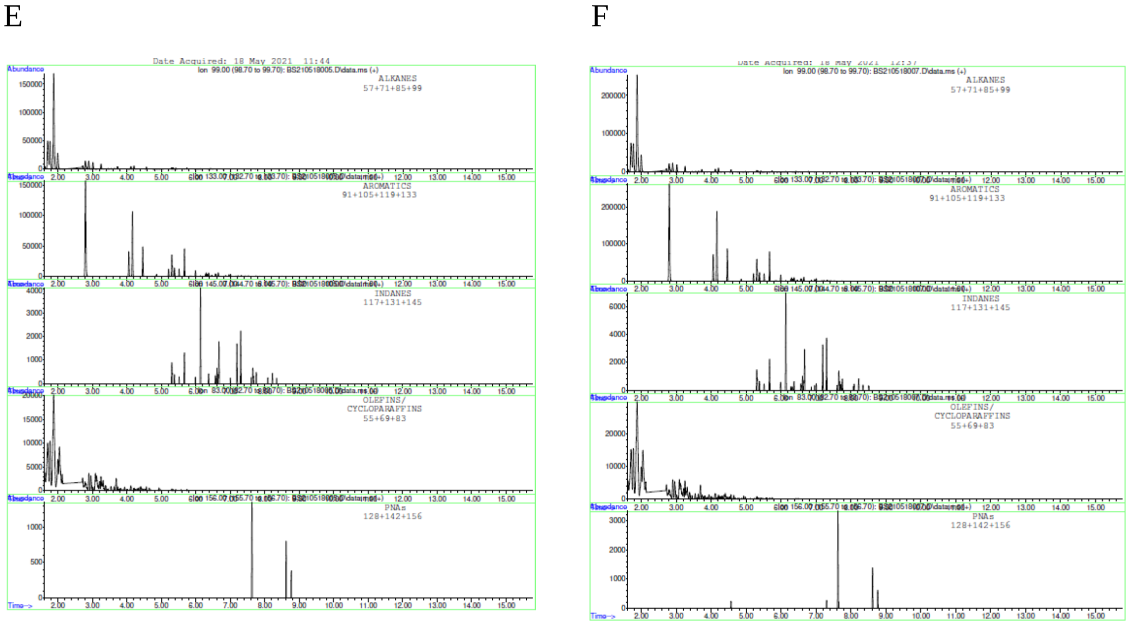

Figure 18. Time trial extracted ion profiles of gasoline at (A) 10 seconds (B) 15 seconds (C) 30seconds (D) 60 seconds (E) 90 seconds, and (F) 5 Minutes.

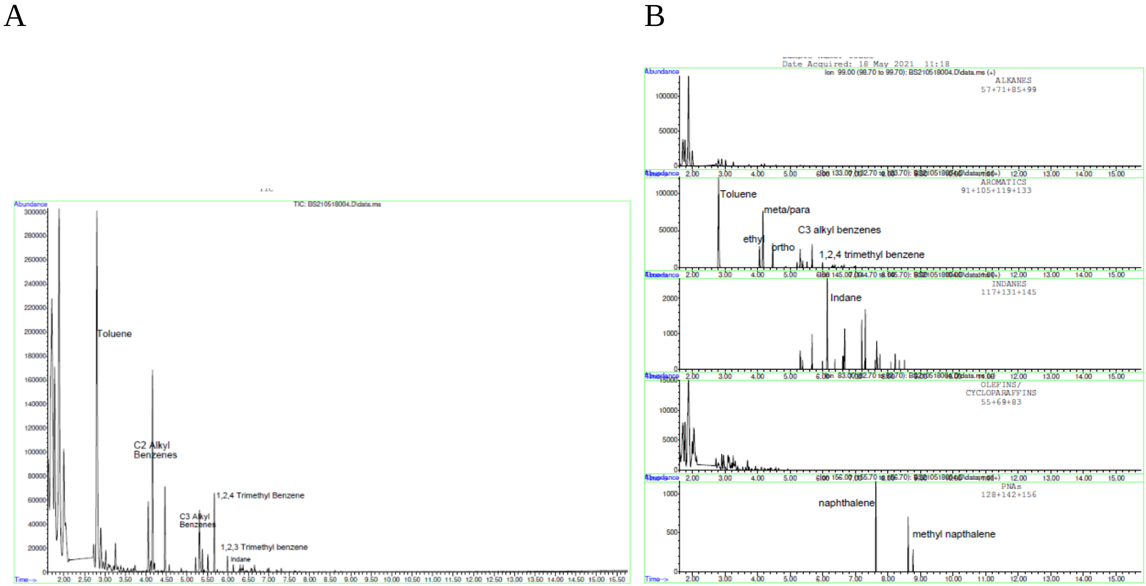

Figure 19. GC-MS data of gasoline at a 60 second C-strip exposure time (A) TIC (B) extracted ion profile.

After 60 seconds was established as an optimal collection time, the size of the C-strip versus the abundance of molecules was examined. Although exact abundances of molecules could never be reproduced in subsequent experiments when using the same size C-strip, the general trend that the larger the C-strip, the more abundant the molecules held true throughout all trials (Figure 20).

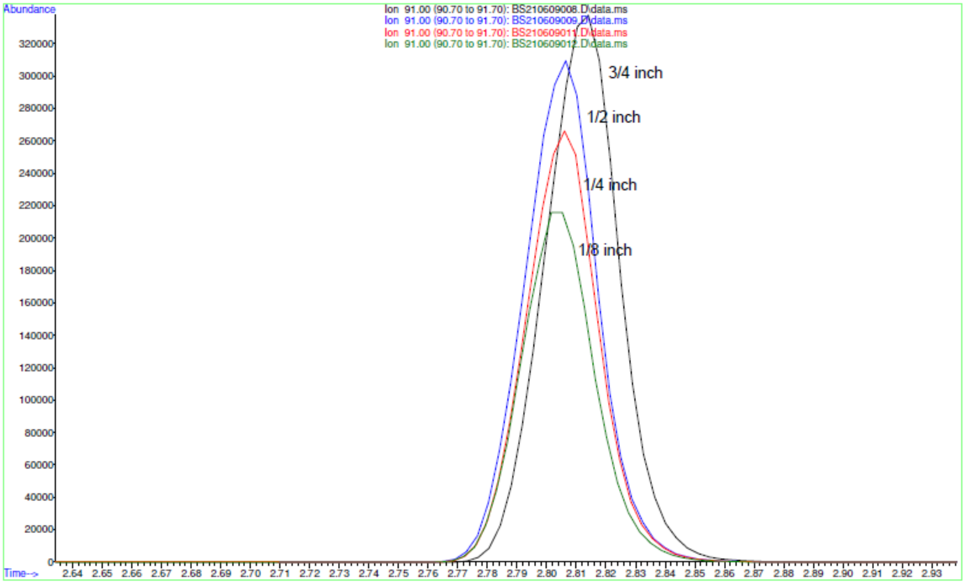

Figure 20. Extracted ion abundances of toluene at different C-strip sizes.

The second part of phase 1 consisted of determining the optimal C-strip size for ease of use. C-strips arrive precut in ¾ inch x ¼ inch strips. For ease of measurement the lengths ¾ inch, ½ inch, ¼ inch, and ⅛ inch were examined (Figure 21). For a chemist the ¾ inch strip is more difficult to work with compared to smaller strip sizes and did not increase the abundance of compounds significantly over the ½ inch strip. The ¾ inch strip was large and had to be torn in half before placing both pieces in the liner within the GC-MS vial. Inversely the ⅛ inch strip was difficult to handle as an investigator. The small size made holding it over the gasoline difficult and the fear of dropping and losing it was present. With the largest and smallest strips negated, the ½ inch strip was placed in the field kit as the sample strip while the remaining ¼ inch of the strip was placed in the field kit as a blank. This also conveniently utilizes only one entire C-strip per field kit.

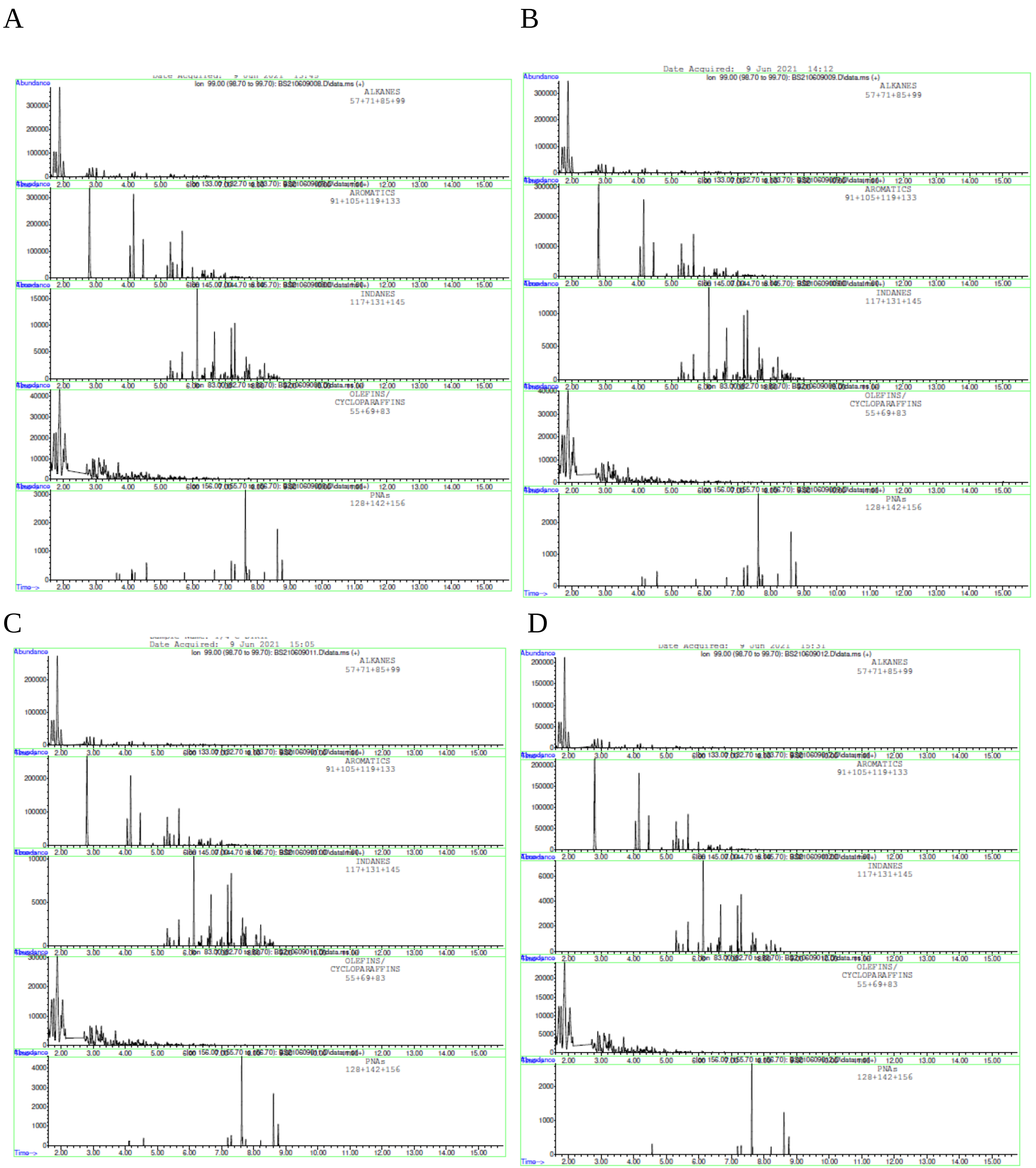

Figure 21. Extracted ion profiles of gasoline on C-strip sizes (A) ¾ inch (B) ½ inch (C) ¼ inch (D) 1/8 inch.

Analysis of the room temperature blank C-strip left exposed to the laboratory environment and the C-strip sealed in a Kapak bag were conducted after two weeks and at least seven exposures to open gasoline in the laboratory. Extracted ion profiles of the two C-strips showed contamination of the one open to the environment and some contamination from the sealed C-strip (Figure 22). The C-strip open to the laboratory environment showed an alkane pattern as seen in distillates and aromatics that are commonly seen in gasoline. The sealed C-strip, although having some unidentifiable peaks, did not have any alkanes or aromatics commonly seen in fire debris samples. At room temperature even a sealed C-strip with repeated exposure to gasoline will not give a completely blank extracted ion profile, though.

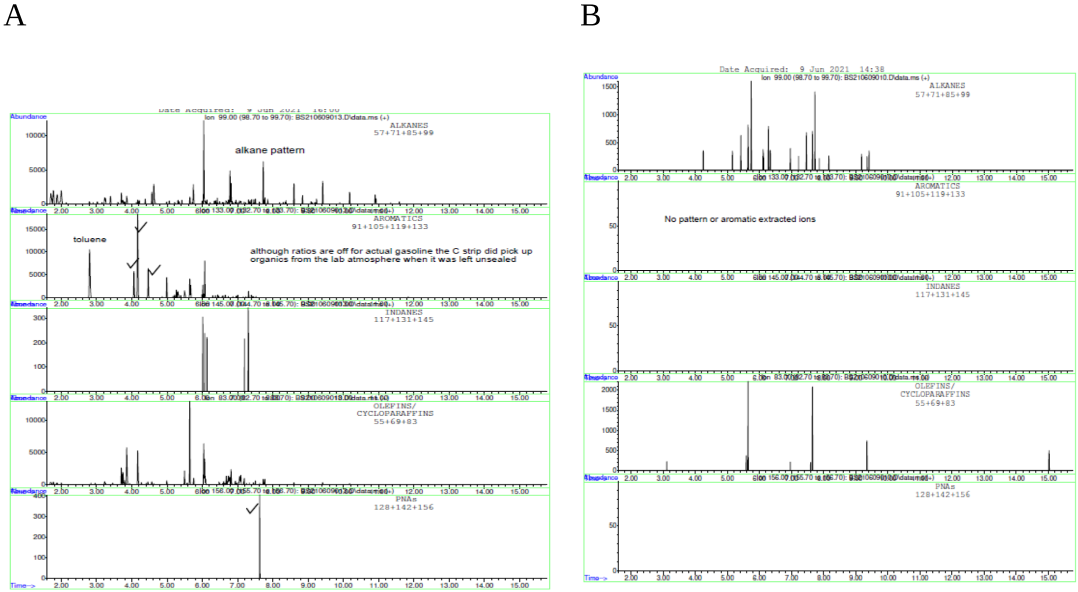

Figure 22. Extracted ion profile comparisons of control C-strips (A) left open to the environment (B) sealed in a Kapak bag.

Phase 2: Initial Field Testing

All investigators received the initial field kit on June 14, 2021 and returned them at their own leisure but at least a month later (Table 7). All three samples received from the investigators were consistent with the results obtained in the lab atmosphere during testing. The sample C-strips showed gasoline with the abundance shifted toward the lighter toluene molecule but with all compounds present to confidently call gasoline. The surprising factor was the blanks received from the investigators, even though sealed, all had an alkane profile, which was only seen in the C-strip left open to the lab environment during the first phase of the project (Figure 23). The alkane profile for the blanks does not affect the determination for gasoline but would pose a problem if these field kits were to be expanded for use for distillates.

Table 7: Collection Information for Field Kits Phase 2

	Years of experience	Sample Taken	C-strips Tested	Container	Atmosphere	Comments
Investigator 1	15	7-28-21	8-11-21	Metal Roughneck 30 Gallon	Outdoors/84^0F	"This way of collecting is nice."
Investigator 2	8	8-18-21	8-20-21	Vehicle Gas Tank	Outdoors/88^0F	
Investigator 3	10	8-19-21	8-20-21	Glass Jar	Indoors/73^0F	*

* Investigator 3 had numerous suggestions: "Instructions on putting on new latex gloves immediately prior to sampling may prevent the control from being contaminated." "The strips are difficult to see in low light, so ample lighting is helpful." "The testing process was not difficult, and it certainly takes up less room to store." "I would recommend keeping the sealed kit(s) in their own small hard plastic container for storage in a vehicle to prevent abrasion and unintentional opening of the sealed bag." [23]

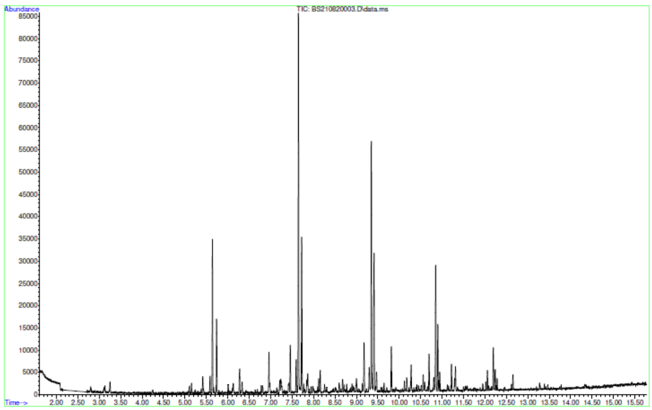

Figure 23. GC spectra showed a contaminated blank C-strip from the initial field kit from Investigator 2. Contamination was an alkane pattern.

To ensure the contamination of the blanks was not from the Kapak bag, a C-strip was placed in a Kapak bag, and a loose C-strip was placed in an oven for 16 hours alongside a case that was positive for gasoline. The loose C-strip in the oven showed a gasoline extracted ion profile up to Indane while the C-strip secured in the Kapak bag was clean of contaminants (Figure 24).

Phase 3: Final Field testing

Concerned by the contaminated blanks returned in the initial field kit, a second field kit was attempted to alleviate this problem. The second field kit was comprised of the same size C-strip pieces, but over-packed in sealed Kapak bags, a glass vial, and a plastic bottle. One investigator commented “In reference to pre-sampling storage the two C-strips contained in a glass vial packaged in a plastic bottle were convenient and compact for storage. While convenience is subjective, it was found that with this form of sample collection it was slightly easier to store the components.” [23]

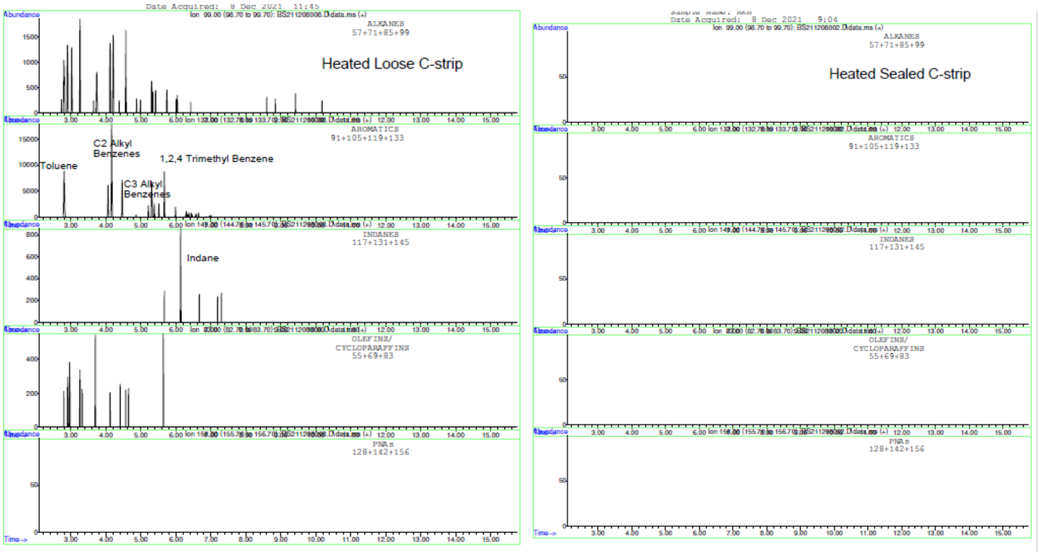

Figure 24. Extracted ion profile comparisons of heated control C-strips (A) left open to the environment (B) sealed in a Kapak bag.

There are important items of note concerning the field kit directions and dispersal. Namely, no training was given to the investigators for sampling procedures, only the paper directions. Also, the mention of fresh gloves was added to the directions in phase 3 after the suggestion was made by Investigator 3. Finally, Investigator 3 also pointed out, after all samples were turned in, that there was a lack of directions on where to store the blank package when turning in the sample. Investigators all ended up leaving the Kapak with the blank in the plastic over pack bottle but not within the glass vial. Investigators received the field kit at least a month before sampling and returned it at their own leisure (Table 8). The second field kit was initially given to the same three investigators as the first field kit and are listed again as Investigator 1, 2, and 3 respectively. When the first samples distributed were tested, Investigator 2 had a contaminated blank while Investigator 1 and 3 did not (Figure 25). Although the contaminated blank from Investigator 2 is easily explainable, the Kapak with the blank was unsealed upon receipt, additional field kits were distributed. The additional three kits tested later did not have any unsealed Kapak packages or contaminated blanks.

Table 8: Collection Information for Field Kits Phase 3

	Years of experience	Field kit distributed	Sample Taken	C-strips Tested	Container	Atmosphere	Comments
Investigator 1	15	12-9-21	1-12-22	2-8-22	Gas Can	Outdoors/ 84^{0}F	“I like this”
Investigator 2	8	12-9-21	1-13-22	2-8-22	Vehicle Gas Tank	Indoors	
Investigator 3	10	12-9-21	3-21-22	4-5-22	Glass Jar	Outdoors/ 70^{0}F	“I didn’t know what to do with the blank”
Investigator 4	15	2-24-22	3-29-22	4-5-22		Indoors	
Investigator 5	<1	2-24-22	4-6-22	5-6-22	Gas Can	Outdoors	“I thought this was easy”
Investigator 6	15	2-24-22	5-3-22	5-6-22	Gas Can	Outdoors	
Laboratory sample	NA	2-24-22	4-4-22	4-5-22	Glass vial	Indoors	NA

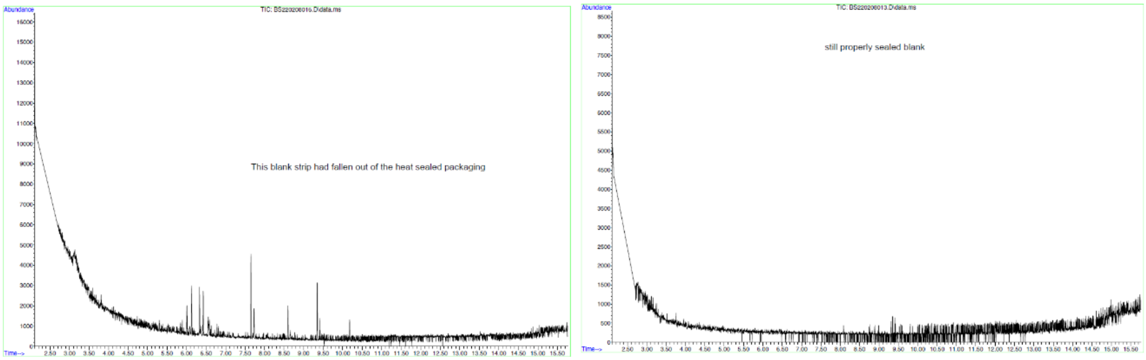

Figure 25. GC data of sample blanks from the final field-testing kit (A) unsealed from the Kapak bag (B) still sealed in the Kapak bag.

For the first time, when the sample from the field kit of Investigator 2 was analyzed a typical gas profile was not readily evident, but only a large abundance of toluene (Figure 26). This type of data did not occur for any other sample and can be attributed to sampling error of the Investigator. Investigator 2 later indicated that he sampled a tank with racing fuel instead of normal gasoline. Racing fuel has additives to increase octane ratings that normal gasoline does not, one of which can be toluene.[24] Sampling error due to inexperience might have caused a concern but Investigator 5 has the least amount of training or experience of any of the Investigators and the data for his sample was an easily identifiable gas profile, as seen previously, with an uncontaminated blank.

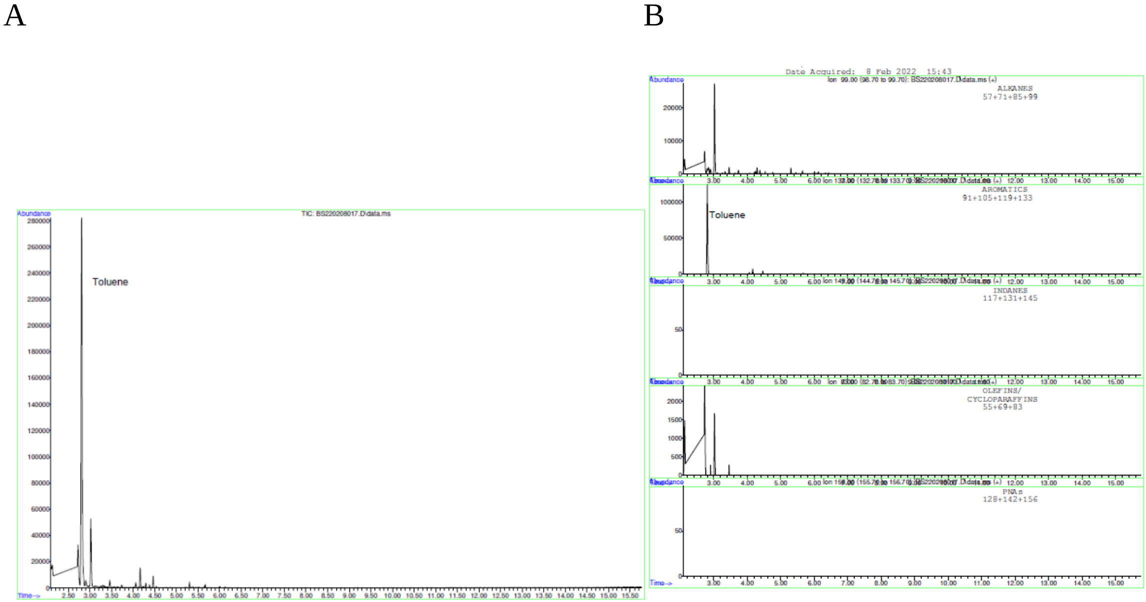

Figure 26. GC-MS data from the field kit of Investigator 2 (A) TIC (B) extracted ion profile.

Conclusion:

The Field Kits developed are a more compact unit, taking up less space than additional cans for the collection of gasoline. Also, no storage issues exist with these field kits such as maintaining the liquid gasoline or the can with the cotton gauze within an agency's evidence facility. Simplicity of analysis also exists for the receiving chemist. The C-strips only need to be placed into a GC-MS vial and extracted with CS_2 before analysis. Advantages to using C-strips as the extraction medium in these field kits are that it is a well-known medium commonly used by fire debris chemists, once the C-strip is exposed to gasoline the gasoline is retained until extracted by CS_2, C-strips are low in cost, and positive feedback was received from the participating Investigators. Disadvantages include the solvent extraction involved, that they are non-reusable, collection errors by an Investigator, and the ease in which they may be misplaced in transport back to the laboratory.

CHAPTER IV

COMPARISON OF ARCHIVED ACTIVATED CHARCOAL STRIP DATA WITH ORIGINAL CASE DATA

In 2020 a total of 54 archived C-strip samples were reanalyzed ranging from years 2000-2020 (20 to 0 years archived), and the GC and MS data compared to the original data. Comparisons were based on the total ion chromatogram and mass spectral extracted ion data of the original versus the new data. The comparison of data has a two-pronged purpose; to further illustrate the validity of the 20 m method but also to investigate the durability of archived C-strips and the conditions they have been stored under.

Method and Materials

Archived samples originated from actual case work at the Spartanburg County Sheriff’s Office Forensic laboratory analyzed between the years 2000-2020. The samples tested could have been analyzed by four different chemists during this time frame (2000-2020), but no note was made of the testing analyst for this research. At the original time of analysis, the data would have been peer reviewed before the finalization of the report.

For all archived samples, the extraction method was passive headspace with C-strips suspended inside a can by a cork and copper wire or by paper clip and magnet (prior to 2015) and heated at 65 °C for 16 hours (Figure 27).[8] After 16 hours the C-strips were removed and cut approximately in half. One half of the C-strip would have been eluted with carbon disulfide (CS_2) and then analyzed with the original 30-meter GC-MS method. The other half of the C-strip would have been archived by placing inside a GC-MS vial and sealed with an 11 mm crimp cap, except in noted instances in 2006 and 2007 where GC-MS vials were found to be closed with an 11 mm snap-it seal cap. The archived samples would have been labeled with the assigned laboratory number then stored in boxes in the laboratory in a cabinet underneath the hood system. (Figure 28). Activated charcoal strips (C-strip) from Albrayco technologies were used for extraction.

Figure 27. A picture of a C-strip suspended by a paper clip for passive headspace extraction.

Figure 28. A picture of the storage cabinet where archived C-strips are housed.

Since there were numerous archived samples over the course of 2000-2020 criteria was created to decide from each year what archived sample to reanalyze:

Criteria:

1. Reanalyze if the fire debris sample contained an ignitable liquid but also a comparison sample with no ignitable liquid found.
2. Reanalyze if a “Gasoline found” and “No Ignitable Liquid Found” within the same year.
3. Reanalyze if the fire debris was an interesting substrate or contained an ignitable liquid not seen as often (ex. Turpentine, Nike Shoe).
4. For the years 2004-2006, two samples from each year were picked at random, reanalyzed and the data interpreted blind (before looking at the old data).
5. Reanalyze at least one archived sample from each year unless otherwise noted.

Prior to 2011, data was printed and stored in files in a record keeping room. Although archived samples for 2008 and 2009 were available, no data could be found in the records room for these years. No samples were retested for the years 2008 and 2009. After 2011, The laboratory management system was installed and utilized and is still in use in the laboratory. All data since then has been stored electronically and was easily retrievable.

Once archived samples were chosen and located in the cabinet, the cap was taken off the GC-MS vial, the C-strip was removed with tweezers, placed into a 0.4 mL flat bottom insert, that was then placed back into the same GC-MS vial and secured with an 11mm snap- it cap. Each sample was ensured to have a cap on it before the next sample was uncapped. The C-strips were eluted with CS_2, the same solvent utilized in all years previous, and analyzed on the 20-meter GC-MS method. A new C-strip was left on the counter in the lab during the opening and CS_2 extraction of the archived samples and used as a system blank to ensure no contamination occurred during transfer. Also, in cases where the archived data was dissimilar to the newly acquired data, the data of the original system blanks were checked to ensure no contamination had occurred originally. There were no contaminated system blanks. A total of 54 archived samples over the course of January to May of 2021 were reanalyzed.

Results and Discussion

Collection of data began with the most recent cases in 2020, because the archived data and C-strips were easy to locate and ended with the collection of data of archived samples from 2000 (Table 9). Until a 2018 case with a Nike shoe, all new data had been consistent with the original data. The archived sample involving the Nike show was the first sample where the archived GC-MS vial had a snap-it cap instead of a crimp cap (Figure 29). After this discovery, archived samples with snap caps were located and reanalyzed. In the years 2006 and 2007 an additional 7 archived samples were found to have snap caps instead of crimp caps, including an archived system blank. (Figure 30) In total, seven out of the eight samples reanalyzed (87.5%) that had a snap cap instead of a crimp cap were contaminated. One snap cap sample (L07-A008) also had an archive sample with a crimp cap. Both were reanalyzed and both were contaminated with gasoline. (Figure 31) This crimp cap sample information was included in future statistical studies.

A logistic regression analysis with 46 sample data was conducted to determine whether the amount of time fire debris sample C-strips were archived could predict the likelihood that the archived sample data matched or did not match the original C-strip data. Comparisons were based on the total ion chromatogram and mass spectral extracted ion data. After comparison the data was marked as the same or not the same as the original data. An analysis that resulted in a different ignitable liquid classification was considered not the same, as was any difference in the data, to include addition of peaks or disappearance of peaks even if the classification remained the same.

Table 9: Comparison of Archived Samples Original and New Data

Snap top data marked with ** was excluded from further statistical data analysis.

Year	Item #	Sample Type	Original Results	20M Column Results	Additional Information
2000					
L00-A007	1	Soil	HPD	HPD	
2001					
L01-A012	1	Debris	NI	NI	
L01-A034	1	Carpet	Gas	Gas	
2002					
L02-A011	1	Soil	NI	Gas	Looks like fresh gas pattern.
L02-A011	2	Container	MPD	Gas	Gas in this one as well. This is the same case as above.
L02-A034	2	Soil	Gas	Gas	
2003					
L03-A007	1	Wood	NI	NI	
L03-A007	2	Liquid	HPD	HPD	
L03-A025	2	Wood	Gas	Gas	
2004					Picked at random
L04-A009	1	Wood	HPD	HPD	
L04-A023	2	Wood	NI	NI	
2005					Picked at random
L05-A013	2	Unknown	NI	NI	
L05-A035	1	Cloths from body	NI	NI	Spectra the same
2006					Picked at random
**L06-A032	3	Wood	NI	HPD	Snap top-The alkane extracted ions for my sample and the original are different even though the pinenes and limonenes are present in both
**L06-A043	1	Carpet	Heavy N-alkanes	Heavy N alkanes Gas	Snap top - The gas Is not present in the original spectra. Is obviously once again a contamination in an older C strip
2007					
L07-A008	2	Carpet	NI	Gas	2 archive samples- this one had crimp top- has gas pattern in aromatics
**L07-A008	2	Carpet	NI	Gas	This one had snap top-The gas pattern is not in the original

Year	Item #	Sample Type	Original Results	20M Column Results	Additional Information
**L07-A036	1	Carpet and shirt	HPD	HPD	Snap top- do not see extra peaks
**L07-A039	2	flooring	NI	HPD	Snap top- no extracted ion spectra available from the original but gained a huge toluene peak and I would call HPD- very contaminated
**Archived CS2 system blank			blank		Snap top- has toluene and some alkanes C10-C14- is contaminated
2010					
**L10-A026	1	Clothing	NI		Snap top- picked up toluene
2011					
L11-A006	2.	Yard debris, grass, soil	Gas	Gas	The small amount of toluene and ethyl benzene that was in the original spectra is gone
L11-A010	1.	Clothing from victim	NI	NI	Has aldehyde/ no aldehyde on 20M column sample
2012					
L12-A002	1.	Wood trim/plastic	Gas	Gas	
L12-A013	1.	Cotton gauze	HPP	HPP	See Toluene and early aromatics not in original data
2013					
L13-A010	1.	carpet	Gas	Gas	
	3.	carpet	NI	NI	Comparison sample
2014					
L14-A015	2.	Mattress/linen	Gas	Gas	
L14-A019	1.	Nike shoe	HNAP	HNAP	
2015					
L15-A001	1.	Wood Flooring	NI	NI	Comparison sample
	2.	Wood flooring	HPP	HPP	
2016					
L16-A005	1.	Soil	Gas	Gas	
	2.	Soil	NI	NI	Comparison sample
L16-A007	1.	Mop head	Terpenes	Terpenes	
L16-A013	1.	Carpet/Padding	Gas	Gas	

Year	**Item #**	**Sample Type**	**Original Results**	**20M Column Results**	**Additional Information**
	2.	Carpet	NI	NI	Comparison sample
L16-A020	2.	Deck Railing	NPP	NPP	
2017					
L17-A017	3.	Wall	Gas	Gas	
	4.	Wall	NI	HPD	Comparison sample
2018					
L18-A009	1.	Carpet/Padding	NI	NI	
L18-A021	1.	Cardboard	LAP	LAP	
	2.	Cardboard	NI	NI	Comparison sample
**L18-A022	1.	Nike Shoe	Gas	NI	No comparison sample. Had snap top. Looks nothing like the original.
2019					
L19-A001	1.	Newspaper	HPD	HPD	
L19-A003	1.	Carpet Padding	NPP	NPP	
	2.	Shirt	MPD	MPD	
L19-A005	1.	Mulch	NI	NI	
L19-A011	3.	Cotton Gauze	Isopropyl OH	Isopropyl OH	Isopropyl much less prominent on archived C-strip
2020					
L20-A004	1.	Wood/Particle Board	HPD	HPD	
L20-A007	1.	Carpet Foam	NI	NI	
L20-A010	1.	Cotton Gauze	MPD	MPD	
L20-A016	1.	Wood	Gas	Gas	
	2.	Wood	NI	NI	Comparison sample

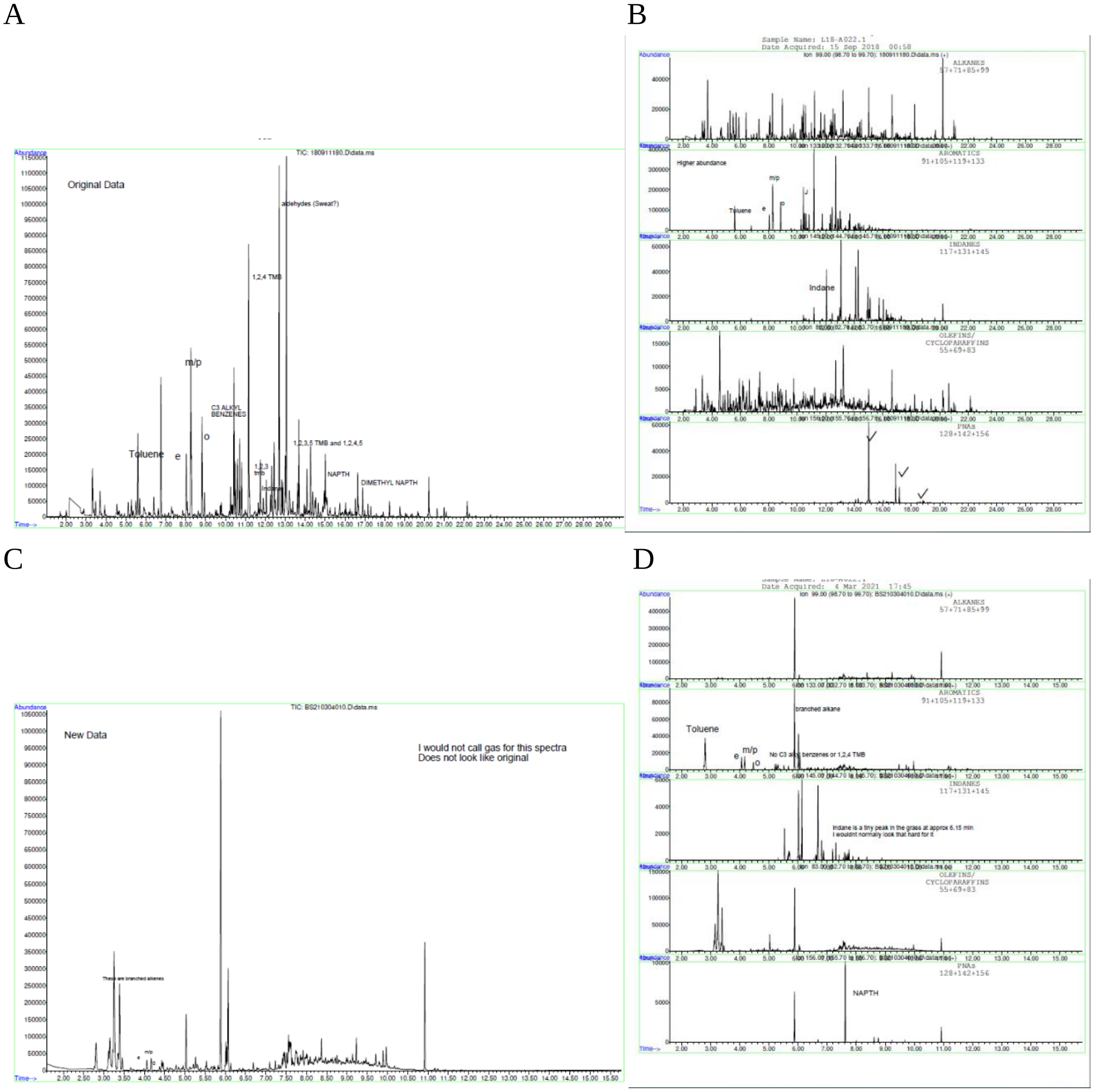

Figure 29. GC-MS data of a Nike shoe on the original method in 2018 (A) TIC (B) extracted ion profile, and of the archived sample C-strip on the 20m method in 2020 (C) TIC (D) extracted ion profile.

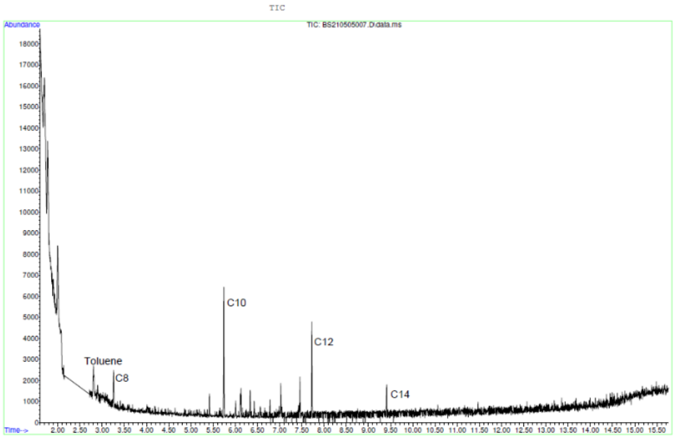

Figure 30. GC data of a contaminated archived blank C-strip from 2007 that was housed in a GC vial with a snap cap

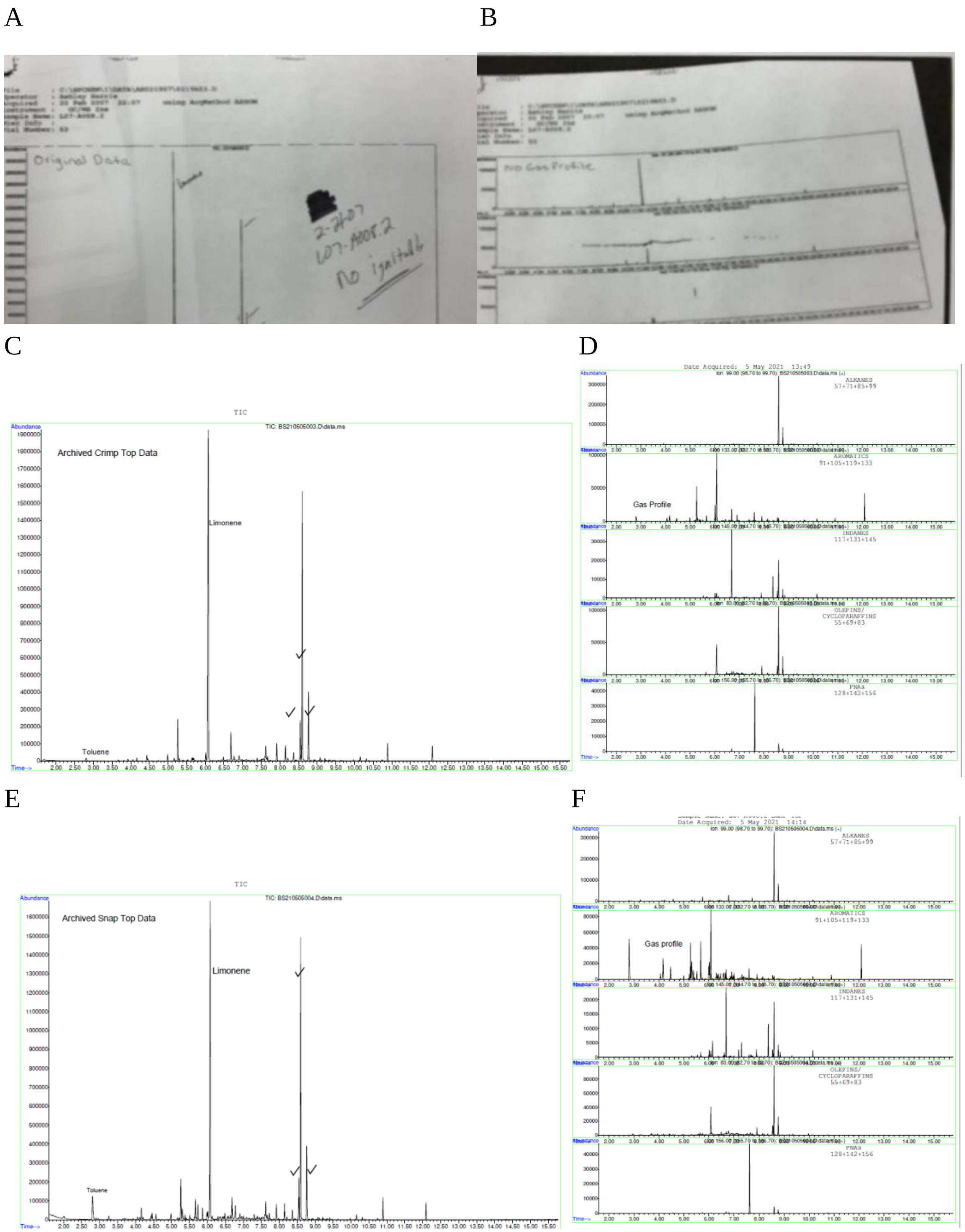

Figure 31. GC-MS data for original and archived C-strip of case L07-A008 (A) original method TIC (B) original method extracted ion profile (C) crimp cap TIC (D) crimp cap extracted ion profile (E) snap cap TIC and (F) snap cap extracted ion profile.

Good model fit was evidenced by nonstatistically significant results on the Homer-Lemeshow test, χ^2 (n = 46) = 9.686, df = 6, p = .139 and a Press Q value (22.26) greater than the critical value at 1 degree of freedom and alpha of .05. The assumption of linearity based on the Tidwell Transformation test was met (*p*=0.084) and the assumption of independence was met with all but two points falling within the absolute value of 2.0. The number of years archived was found to be not statistically significant in determining the correctness of archived C-strip data (Wald = 2.136, df = 1, *p* = .144). The odds ratio suggests that for every one-point increase in years the chance of the data being not the same versus the same only increases by 1%. Overall, the logistic regression model classified 84.8% of the cases correctly. The cases where 20m new data was the same as the original were classified correctly 100% of the time, while the cases where the original and 20m new data were not the same were classified correctly 0% of the time. Two outliers were indicated in the logistic regression model, L12-A013 and L17-A017.4. L17-A017.4 was a comparison sample that originally had no ignitable liquids identified but gained a heavy petroleum distillate pattern during archive. L12-A013 would have been concluded as the same classification of heavy petroleum product but was considered not the same statistically because of the addition of early aromatic peaks such as toluene.

Conclusion

Contamination is almost assured when using a snap-it cap. Seven out of the eight samples reanalyzed that had a snap cap instead of a crimp cap were contaminated. Excluding the data from samples with a snap cap only, 7 archived samples out of 46 tested were contaminated. The logistic regression model suggested that time archived is not a predictor of data consistency. Basically, the model suggests that 84.8% of the time if an archived sample, regardless of the year, is called the same as the original sample, the determination would be accurate. This research indicates that specimens should be stored with a crimp top vial.

CHAPTER V

CONCLUSION

Fast Gas Chromatography

The accuracy, intermediate precision, repeatability, and selectivity of a new faster gas chromatography method coupled with mass spectrometry was examined for fire debris analysis. An existing method utilizing a 30-meter 5% phenyl methylpolysiloxane column was translated into a method using a 20-meter 5% phenyl methylpolysiloxane column thereby shortening the analysis time by 15 minutes. The change in column length and diameter did shorten the run time of the analysis without any change in the order of retention times of compounds or resolution of peaks within the data.

Implications

Shorter analysis time within laboratories leads to faster turnaround time and therefore less back-log of cases. In smaller laboratories, where only one GC-MS is in operation, shorter analysis time increases instrument availability for other types of analyses to occur (i.e., drug analysis). Overall, there would be a decreased, time per item, analysis and with the advent of smaller instrumentation an increase in options for fast portable on-scene instruments.

Limitations

Extensive online searchable databases with the National Center for Forensic Science (NCFS) exist that utilize a 30 m column for GC-MS analysis of ignitable liquids.[25] These databases give exhaustive GC and MS data for different types and brands of ignitable liquids as well as different ignitable liquids on different substrates. Ten ignitable liquids from ACE Hardware were analyzed in this research, which is far from exhaustive. A similar database that exists for the 30 m column would need to be set up for the 20 m column GC-MS method.

Further Research

Other avenues to decrease the analysis time of ignitable liquids on GC also exist. Wool and Decker successfully decreased their analysis time of pesticides not only by changing the column length and diameter but by changing the carrier gas from helium to hydrogen.[16] Changing the carrier gas would entail a change in lenses and the source within the MS to a hydro inert source, but once the conversion was done all validation procedures could be performed again. Another, simpler way, to explore to decrease analysis time is by decreasing the time it takes for the GC oven to cool between injections. Time between injections during this research was approximately 8-10 minutes. Shortening the cool down time by installing a GC oven insert, thereby decreasing the volume of the oven, would again speed up the analysis time per sample.

Field Kit

A final field kit consisting of a ½ inch by ¼ inch C-strip (to obtain the sample) and a ¼ inch by ¼ inch C-strip (blank) over-packed in sealed Kapak bags, a glass vial, and a plastic bottle, was sent out to 6 investigators with instructions to let the kit sit in their car for approximately 30 days. The kit was then to be used for sampling of the headspace of gasoline from any container. The submitted field kit was analyzed using the new 20 m GC-MS fire debris method then the data examined for the determination of gasoline and the peaks typically associated with gasoline during fire debris analysis. Gasoline was successfully reported from the C-strip field kits.

Implications

C-strips are a well-known, cost-effective adsorbent medium for ignitable liquids.[6] Within 60 seconds on scene, investigators can obtain an ignitable liquid sample without spillage of liquids, pipette usage, or directly encountering the liquid (the C-strip is held in the headspace). Transport of the collected evidence is simple, requires limited space, and will not potentially spill out into the investigator's vehicle. Within the laboratory, standard passive headspace methods utilize C-strips in the analysis of fire debris samples, therefore analysis can directly flow from on scene collection to the laboratory.[8] Field collection of accelerants directly onto the C-strips, on scene by investigators, leads to eliminating a lengthy step within the laboratory analysis (the passive headspace portion that includes heating for 16 hours) cutting analysis time. Also, no large can or liquid evidence remains for storage in property rooms.

Limitations

The main element in eliminating contamination of the blank or sampling C-strips is proper sealing of the Kapak pouches. If any portion of the Kapak packaging is unsealed before sampling the field kit would need to be discarded due to possible contamination. Investigators would need to be diligent in their sampling so that no cross contamination with other ignitable liquids of the sample C-strip occurred. Analysts would need to have trust in their investigator to properly sample and secure the C-strips. The field kits are small as are the C-strips inside, so care would need to be taken not to lose any portion of the field kit or the evidence after collection or during transport to the lab. Within the lab, CS_2 would still be used as the solvent for extraction of the C-strips, as this is a hazardous chemical, care would need to be taken. Finally, the C-strips are non-reusable after analysis.

Further Research

Although gasoline is a common accelerant in fire debris analysis it is not the only one that is found in liquid form on fire scenes. Research into the expansion of the field kit to look at other ignitable liquids such as distillates and alcohols would further advance the installation of this kit as a tool on scenes. The destructive nature of the laboratory technique on the C-strip does not allow for reanalysis of the sample, but archiving a portion of the field kit sample C-strip would eliminate this difficulty. In this research a ½ in piece of C-strip was used to collect sample, determinations of what portion of this would be an accurate amount to archive would be beneficial.

Archived C-strip Analysis

A binary logistic regression analysis of archived C-strip data was conducted to determine whether the amount of time fire debris sample C-strips were archived could predict the likelihood that the archived sample data matched or did not match the original C-strip data. Reanalyzation of the archived C-strips occurred on the new 20 m column GC-MS method. In 2020 a total of 54 (46 used in the logistic regression) archived C-strip samples were reanalyzed ranging from years 2000-2020 (20 to 0 years archived) and the GC and MS data compared to the original data.

Implications

Although, this research suggests time is not a predictor of differences in spectra between original and archived C-strip data, there are instances where GC-MS data between the two is different. These differences could be as major as completely changing the ignitable liquid that would be reported or simply the addition or deletion of compounds within the spectra. Within this sample set 84.8% of the time, assuming the archived sample is the same as the original sample, would be an accurate assumption.

Limitations

During this study only 54 archived samples were reanalyzed and of these only 46 were used in binary logistic regression. Within the 20-year time span examined, the years 2008 and 2009 samples could not be reanalyzed due to lack of original data, and the 2007 data was excluded due to the archived samples being closed with snap-it seal caps. Larger sampling size and more samples within the earlier years (2000-2010) could be beneficial.

Further Research

This research suggests that time is not a predictor of impurities or loss of compounds in archived fire debris samples. To know what may or may not cause contamination a long-term study (10 years+) would need to be conducted where known concentrations of accelerants on C-strips were analyzed then archived under varying conditions. Examples of these varying conditions could be exploring alternate packaging or housing conditions of the archived C-strip

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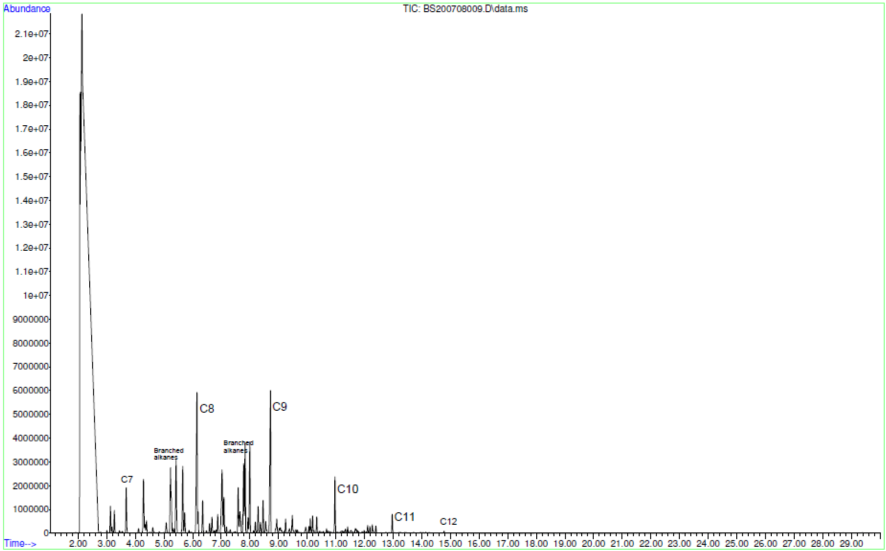
APPENDICES

APPENDIX A: IGNITABLE LIQUID REFERENCE STANDARDS

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708009.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  13:06
SAMPLE NAME:        COLEMAN CAMP FUEL
MISC. INFO:
```

TIC



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124004.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  10:16
SAMPLE NAME:        COLEMAN CAMP FUEL
MISC. INFO:
```

TIC

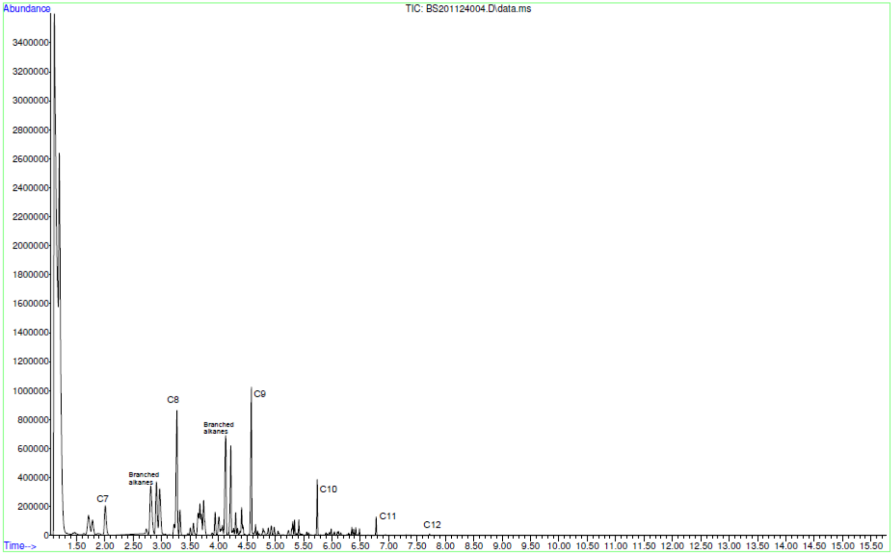
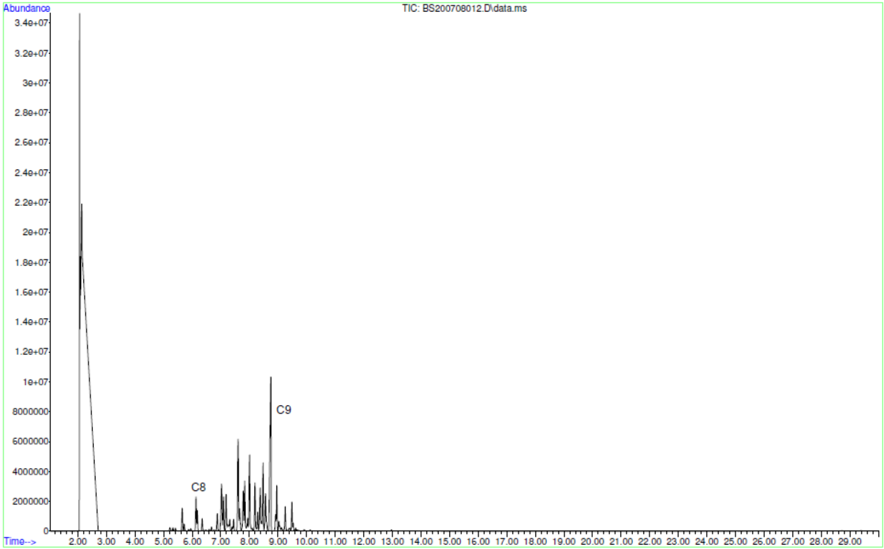


Figure 32. Coleman Camp Fuel

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708012.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  15:10
SAMPLE NAME:        JASCO VM8P NAPTHA
MISC. INFO:

                                             TIC
```



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124008.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  12:00
SAMPLE NAME:        JASCO VM8P NAPTHA
MISC. INFO:

                                             TIC
```

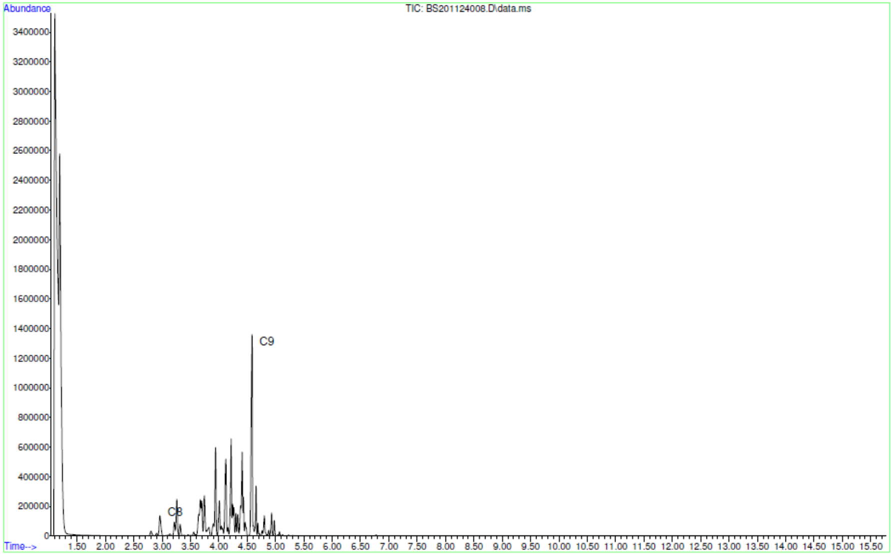
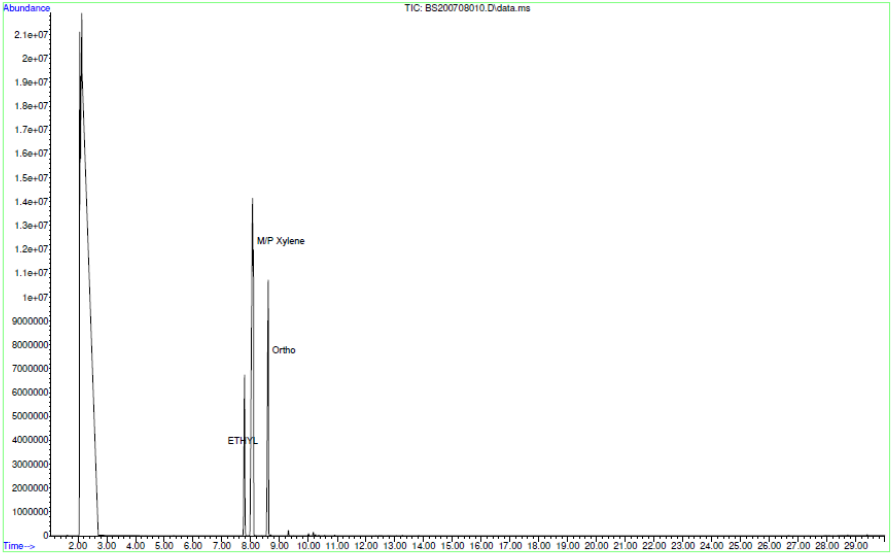


Figure 33. Jasco VM8P Naptha

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708010.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  13:47
SAMPLE NAME:        JASCO XYLENE
MISC. INFO:
```

TIC



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124006.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  11:08
SAMPLE NAME:        JASCO XYLENE
MISC. INFO:
```

TIC

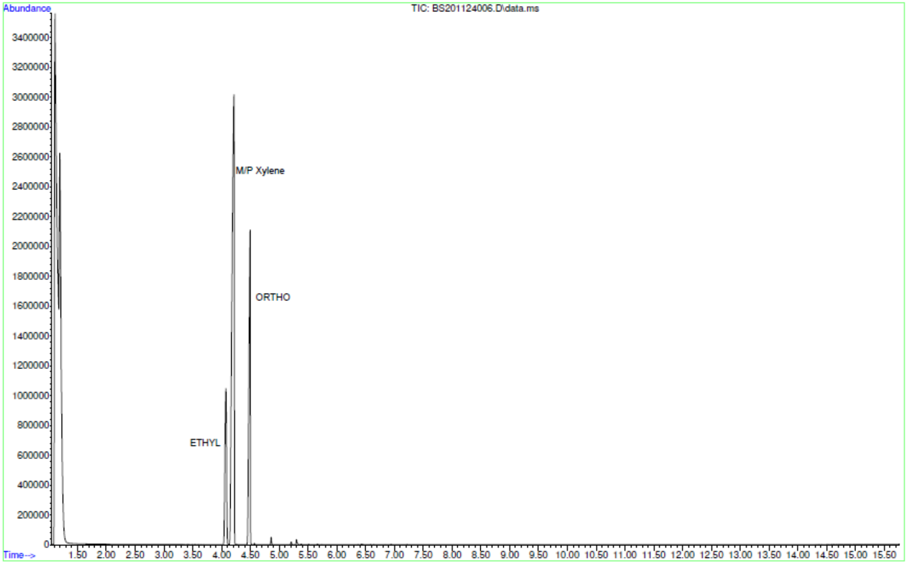
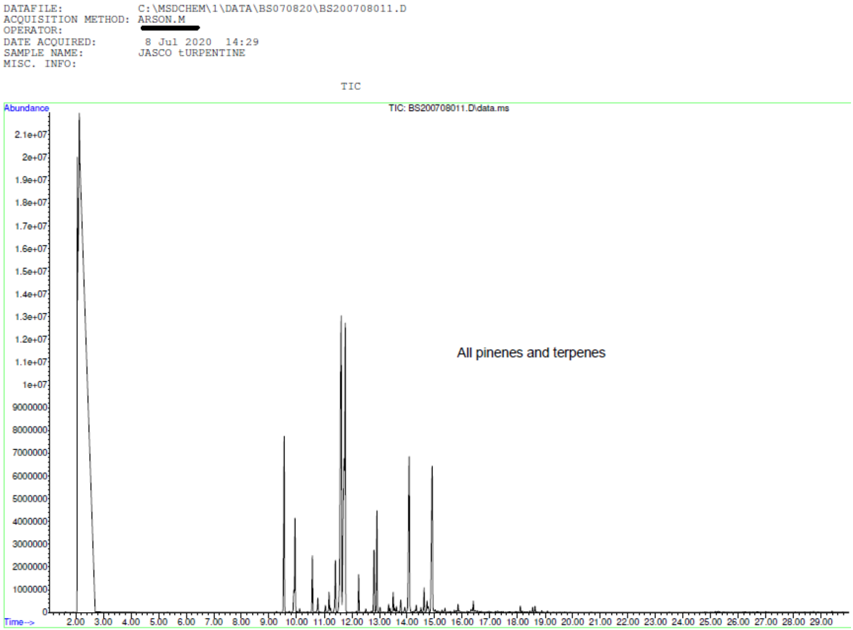
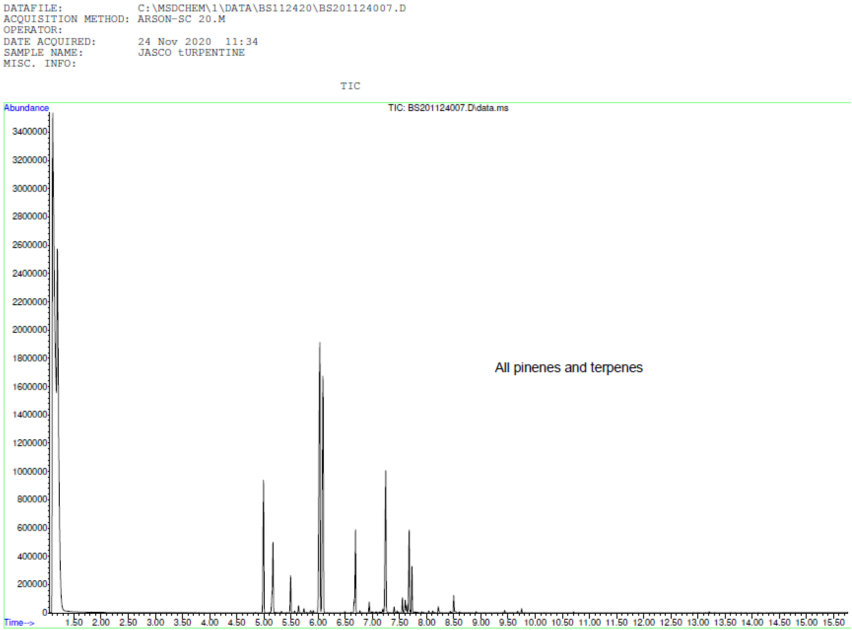


Figure 34. Jasco Xylene

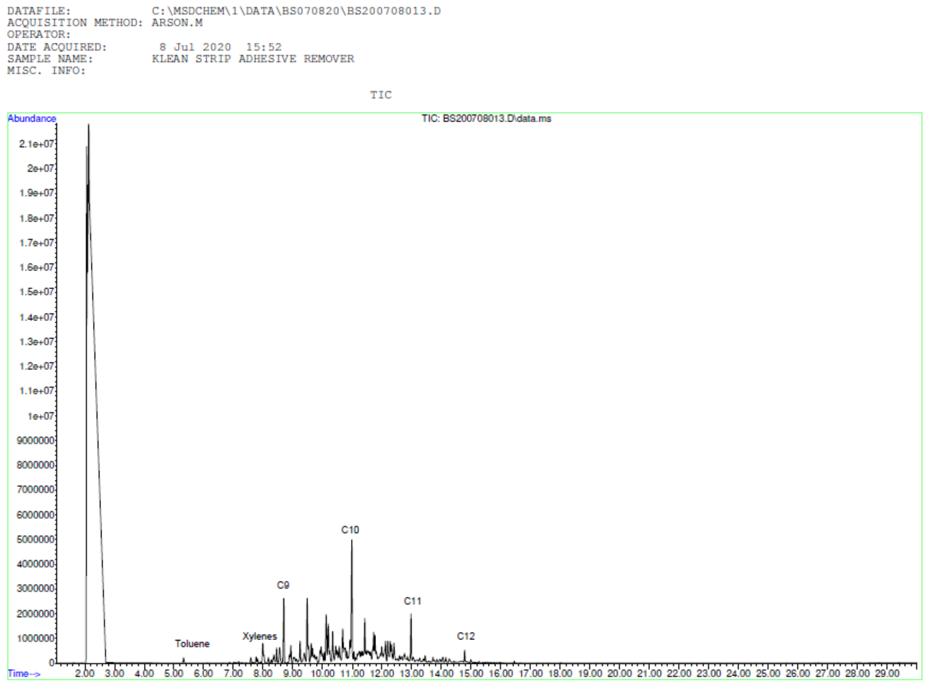


```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708011.D
ACQUISITION METHOD: ARSON.M
OPERATOR:           [redacted]
DATE ACQUIRED:       8 Jul 2020  14:29
SAMPLE NAME:        JASCO tURPENTINE
MISC. INFO:
```

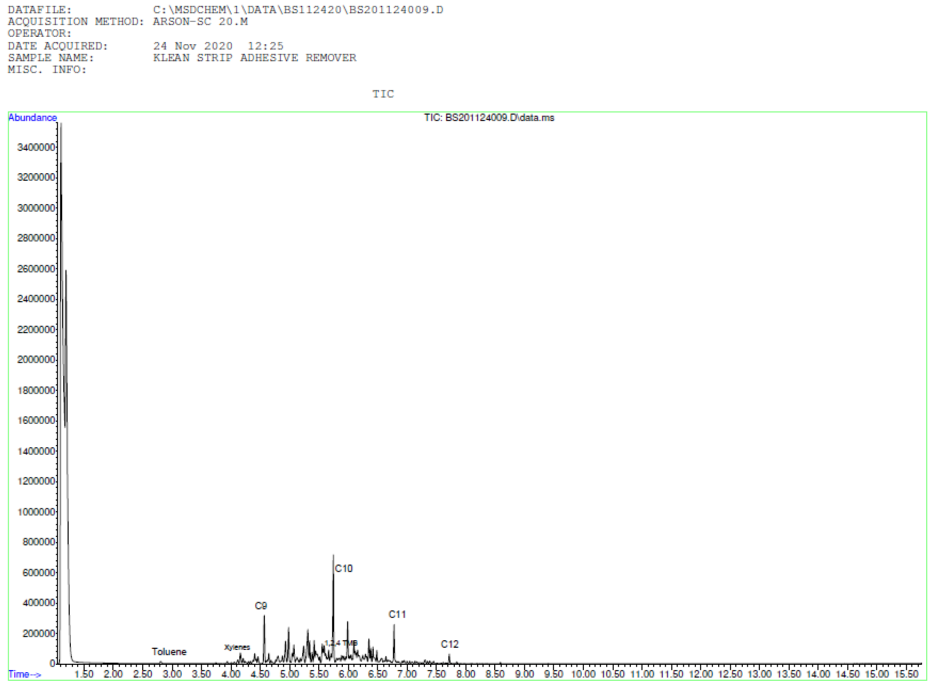


```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124007.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  11:34
SAMPLE NAME:        JASCO tURPENTINE
MISC. INFO:
```

Figure 35. Jasco Turpentine



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708013.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  15:52
SAMPLE NAME:        KLEAN STRIP ADHESIVE REMOVER
MISC. INFO:
```

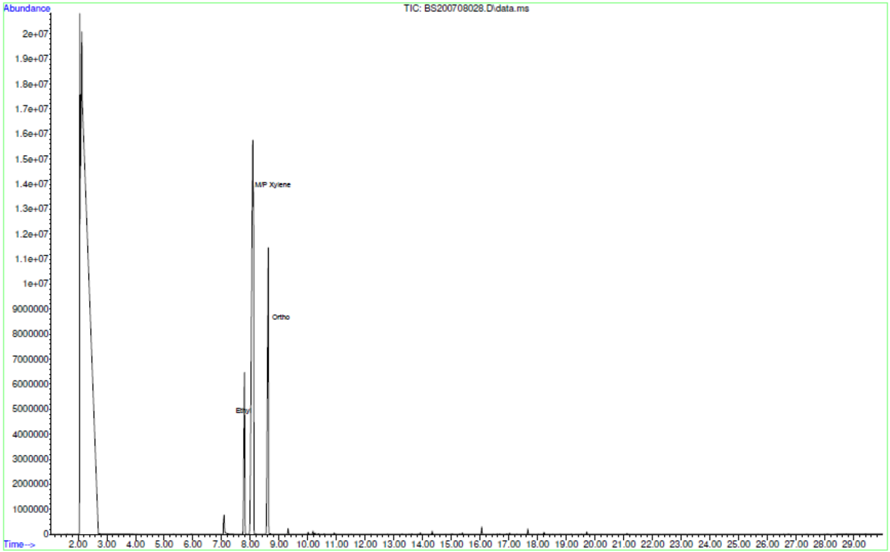


```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124009.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  12:25
SAMPLE NAME:        KLEAN STRIP ADHESIVE REMOVER
MISC. INFO:
```

Figure 36. Klean Strip Adhesive Remover

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708028.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  23:56
SAMPLE NAME:        KLEAN STRIP PAINT VARNISH STRIPPER
MISC. INFO:

                                        TIC
```



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124013.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  14:10
SAMPLE NAME:        KLEAN STRIP PAINT VARNISH STRIPPER
MISC. INFO:

                                        TIC
```

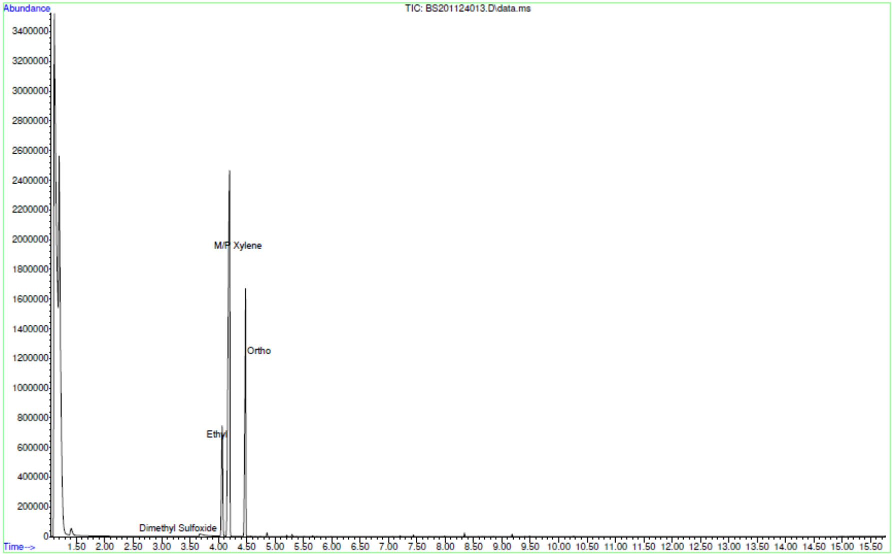
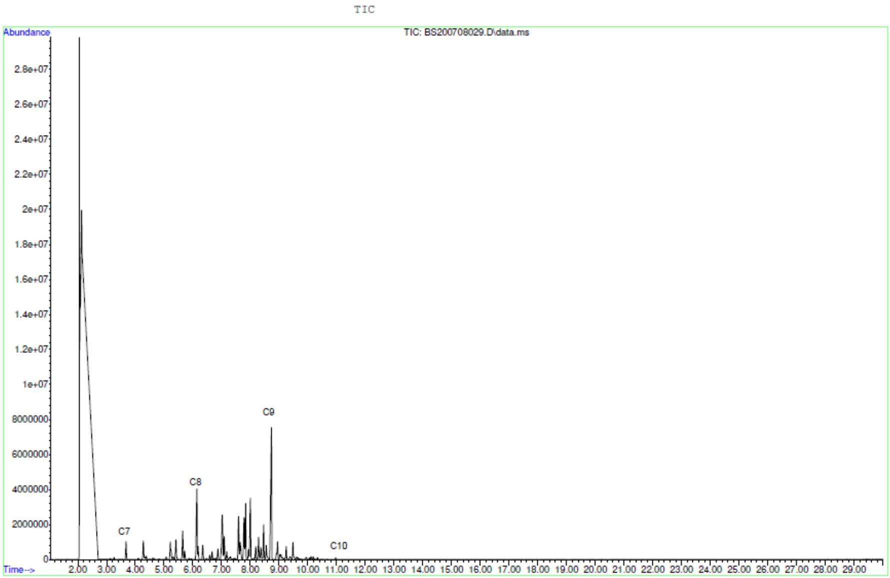


Figure 37. Klean Strip Paint Varnish Stripper

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708029.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       9 Jul 2020  00:37
SAMPLE NAME:        RONSONOL LIGHTER FLUID
MISC. INFO:
```



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124014.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  14:36
SAMPLE NAME:        RONSONOL LIGHTER FLUID
MISC. INFO:
```

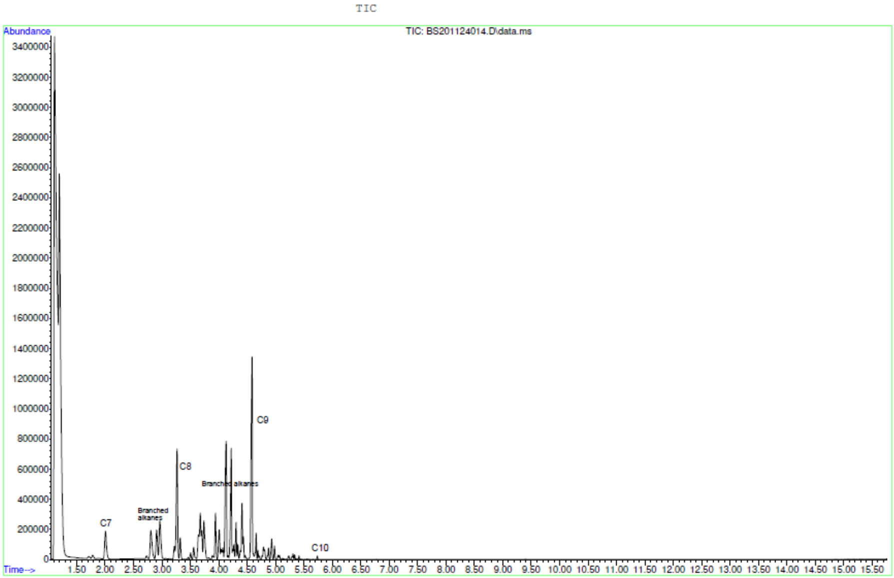
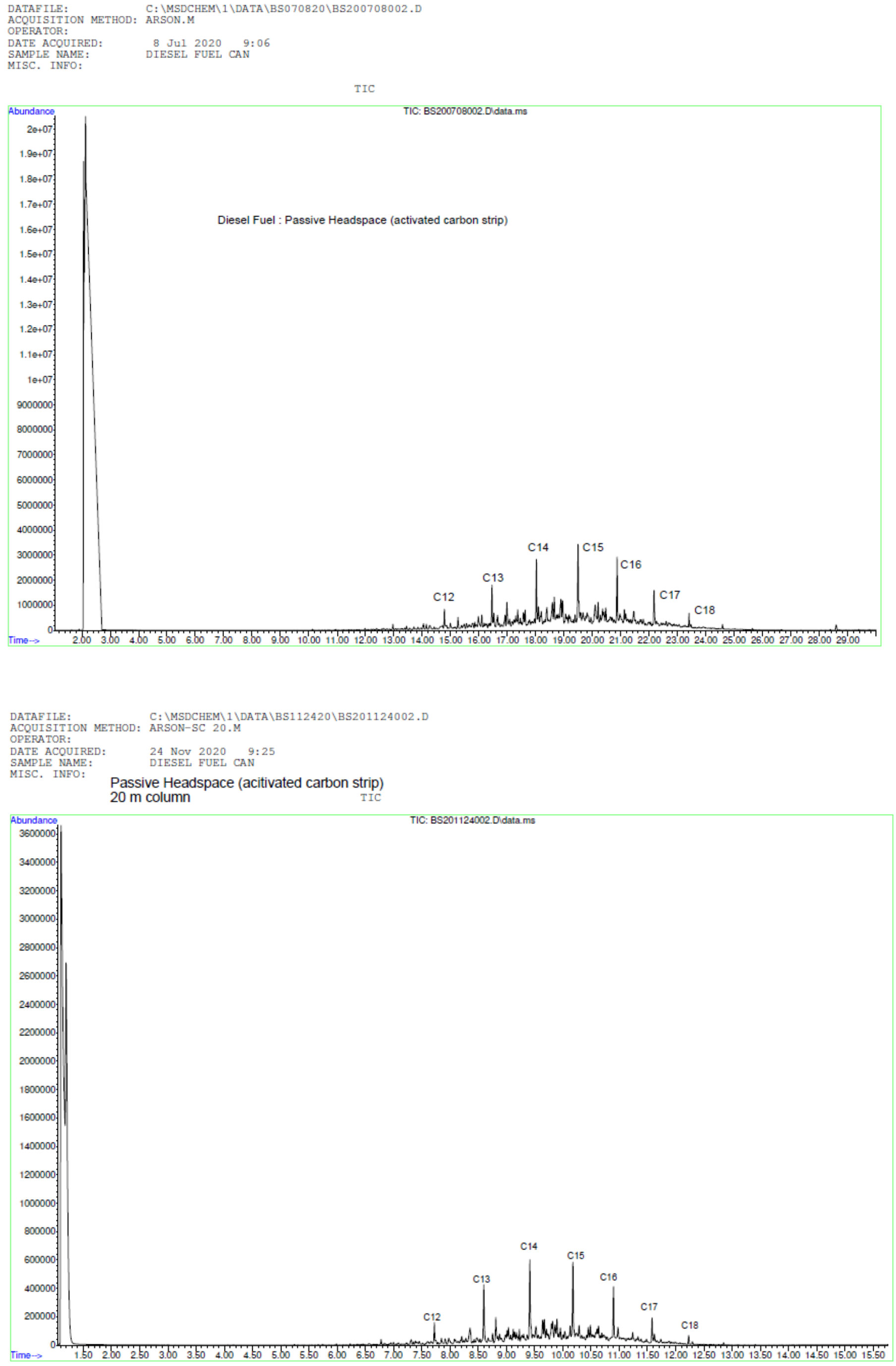


Figure 38. Ronsonol Lighter Fluid



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708002.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020   9:06
SAMPLE NAME:        DIESEL FUEL CAN
MISC. INFO:
```

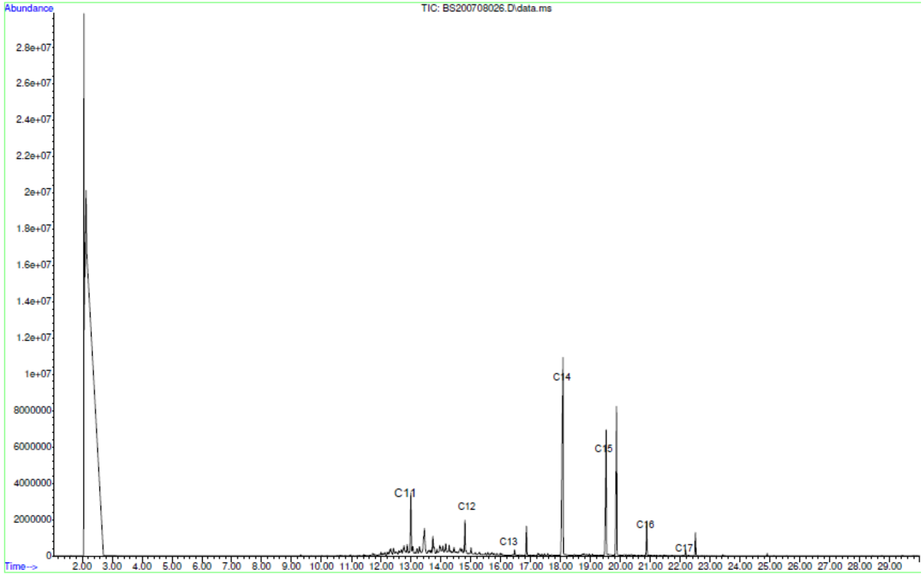
```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124002.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020   9:25
SAMPLE NAME:        DIESEL FUEL CAN
MISC. INFO:
```

Passive Headspace (acitivated carbon strip)
20 m column

Figure 39. Diesel Fuel (50% Weathered)

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708026.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  22:34
SAMPLE NAME:        TIKI READY TO LIGHT TORCH FUEL
MISC. INFO:
```

TIC



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124011.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  13:18
SAMPLE NAME:        TIKI READY TO LIGHT TORCH FUEL
MISC. INFO:
```

TIC

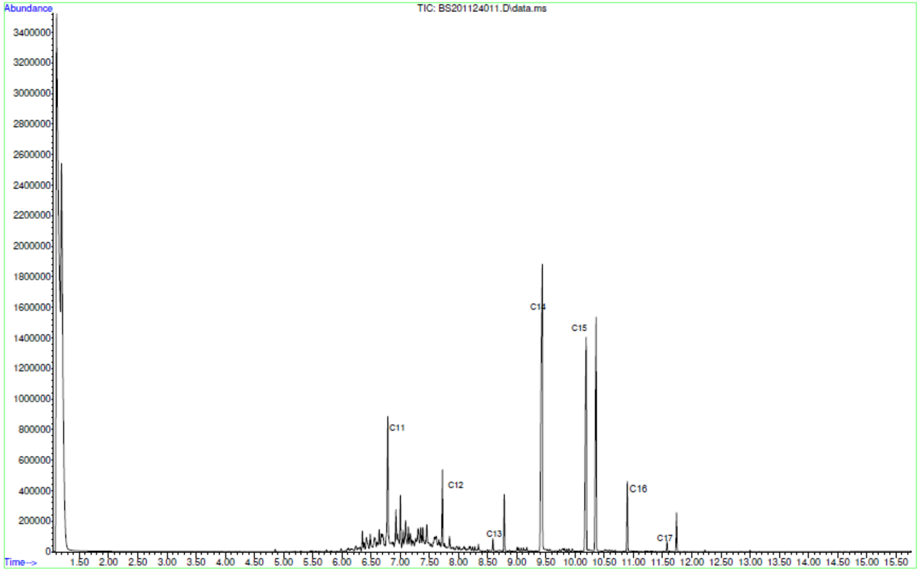
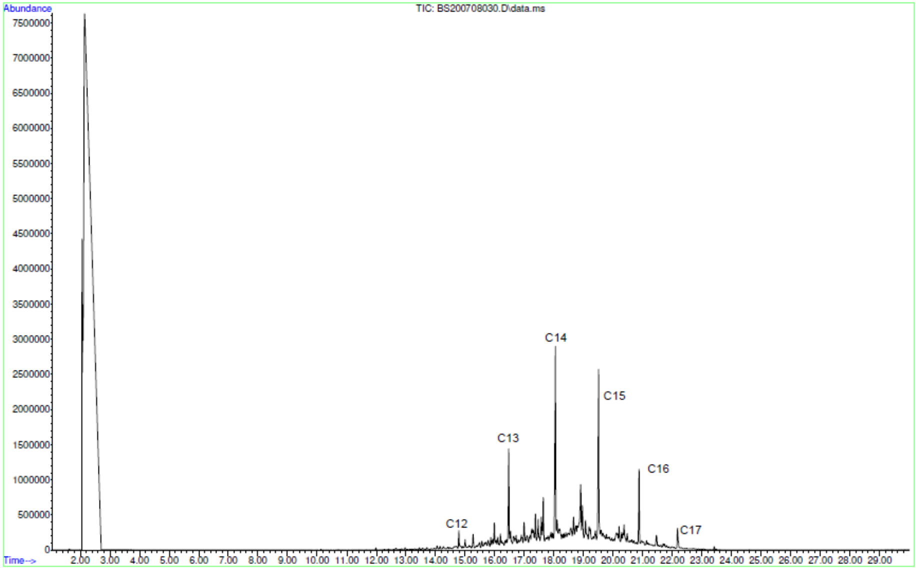


Figure 40. Tiki Ready to Light Torch Fuel

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708030.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:      14 Jul 2020   8:56
SAMPLE NAME:        KEROSENE (CAN)
MISC. INFO:
```

TIC



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124016.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  15:29
SAMPLE NAME:        KEROSENE
MISC. INFO:
```

Passive Headspace (acitivated carbon strip)
20 m column

TIC

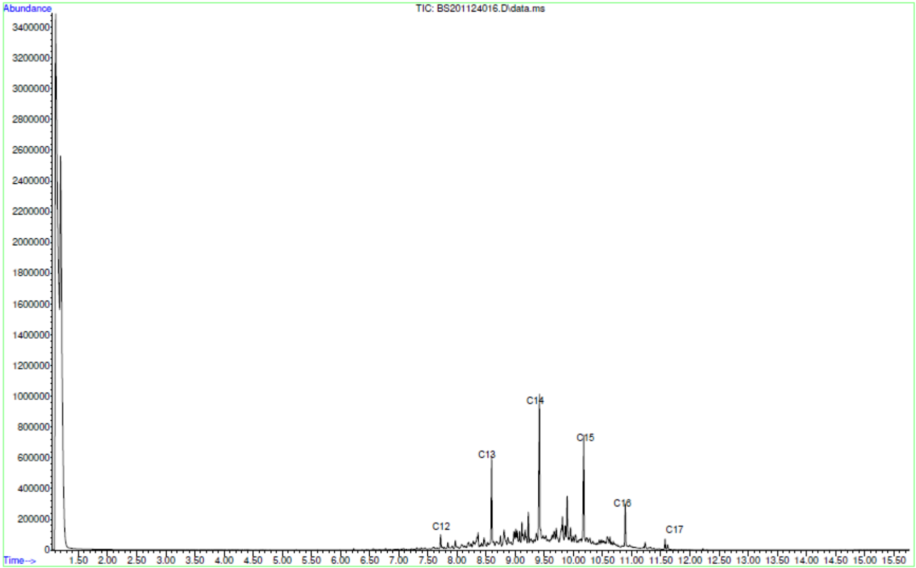
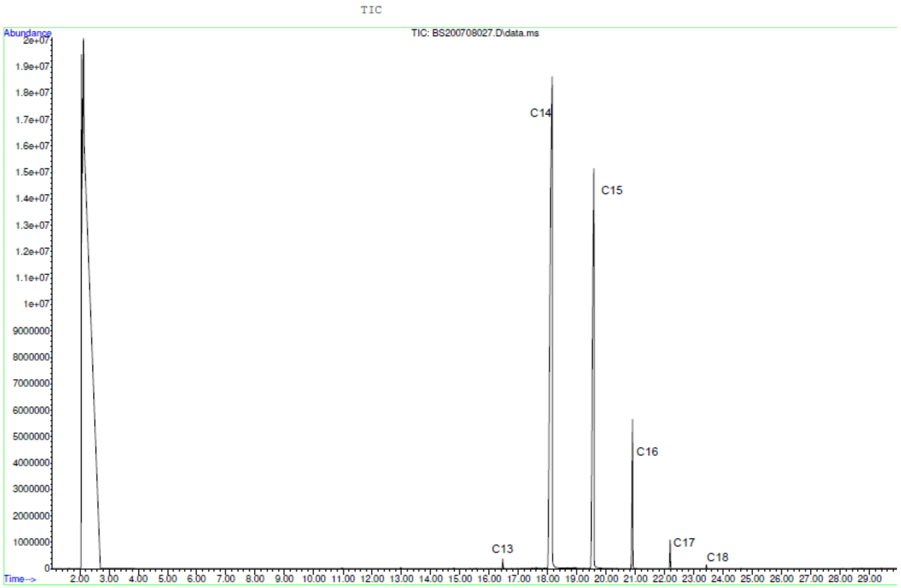


Figure 41. Kerosene

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS070820\BS200708027.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:       8 Jul 2020  23:15
SAMPLE NAME:        ULTRAPURE PARAFFIN OIL
MISC. INFO:
```



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112420\BS201124012.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      24 Nov 2020  13:43
SAMPLE NAME:        ULTRAPURE PARAFFIN OIL
MISC. INFO:
```

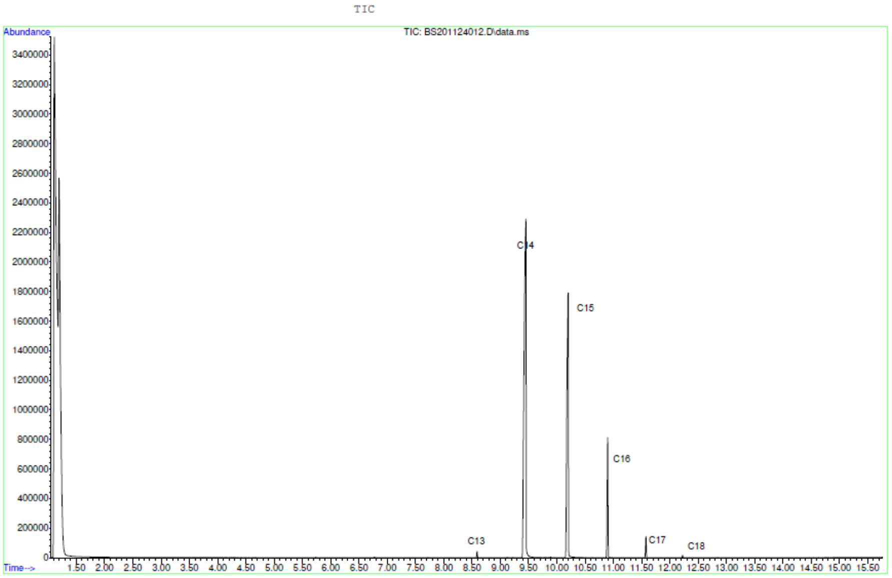
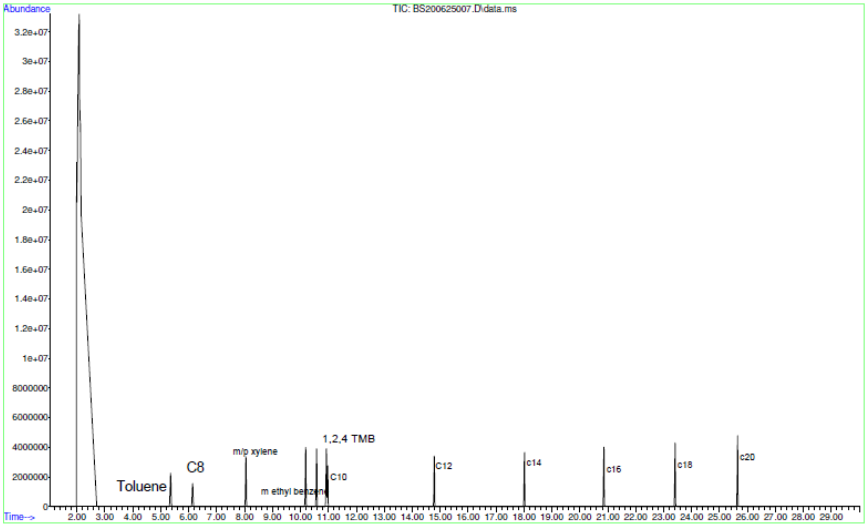


Figure 42. Ultra-Pure Paraffin Lamp Oil Soot Less, Smokeless, Odorless

```
File       :C:\MSDCHEM\1\DATA\BS062520\BS200625007.D
Operator   :
Acquired   : 25 Jun 2020  12:12     using AcqMethod ARSON.M
Instrument :   5973
Sample Name: E16-18 STD
Misc Info  :
Vial Number: 6
```



```
DATAFILE:            C:\MSDCHEM\1\DATA\BS111620\BS201116003.D
ACQUISITION METHOD:  ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:       16 Nov 2020  11:57
SAMPLE NAME:         E16-18
MISC. INFO:
```

TIC

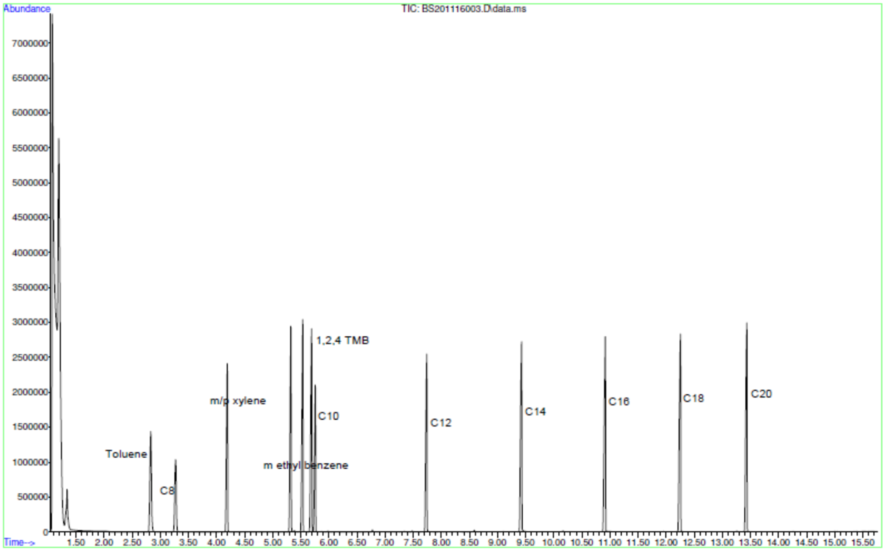
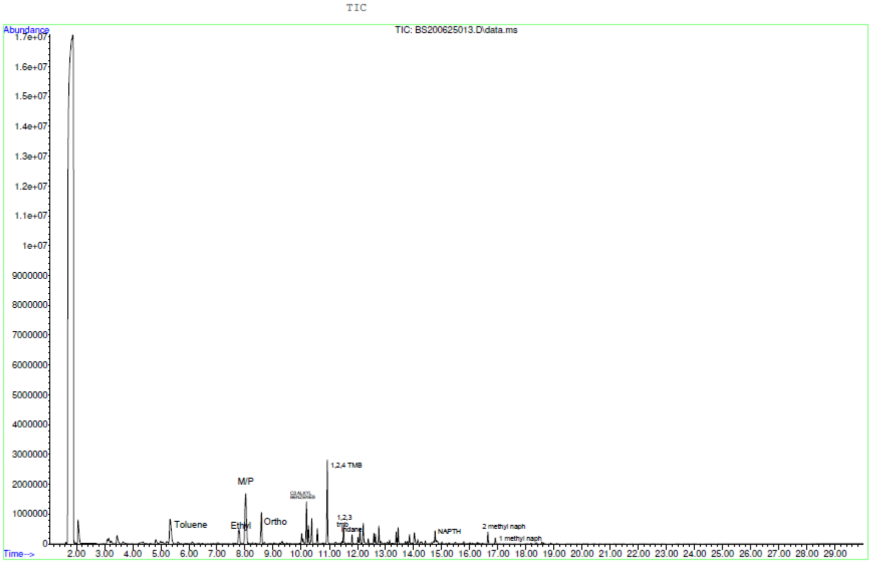


Figure 43. E1618 Standard

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS062520\BS200625013.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:      25 Jun 2020  16:19
SAMPLE NAME:        25% EVAP GAS STD
MISC. INFO:
```



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS112320\BS201123008.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      23 Nov 2020  11:35
SAMPLE NAME:        25% GAS
MISC. INFO:
```

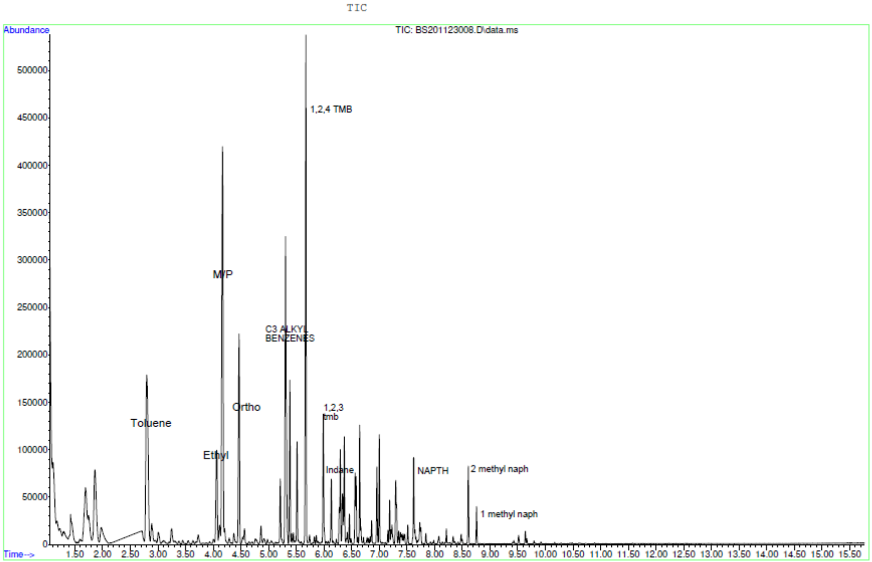
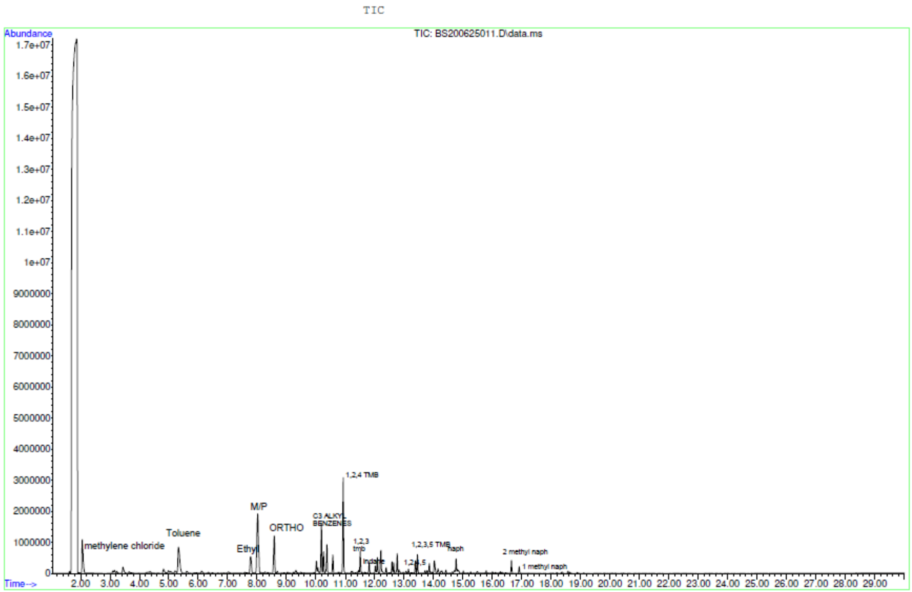


Figure 44. Gasoline (25% Weathered)

```
DATAFILE:             C:\MSDCHEM\1\DATA\BS062520\BS200625011.D
ACQUISITION METHOD:  ARSON.M
OPERATOR:
DATE ACQUIRED:        25 Jun 2020  14:56
SAMPLE NAME:          50% EVAP GAS STD
MISC. INFO:
```



```
DATAFILE:             C:\MSDCHEM\1\DATA\BS111720\BS201117002.D
ACQUISITION METHOD:  ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:        17 Nov 2020   7:40
SAMPLE NAME:          50% EVAP GASOLINE
MISC. INFO:
```

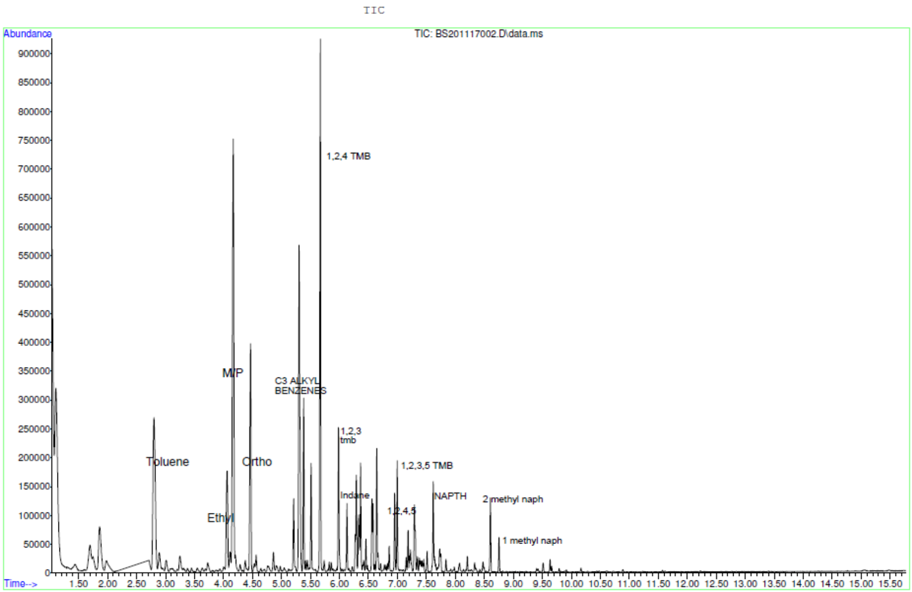
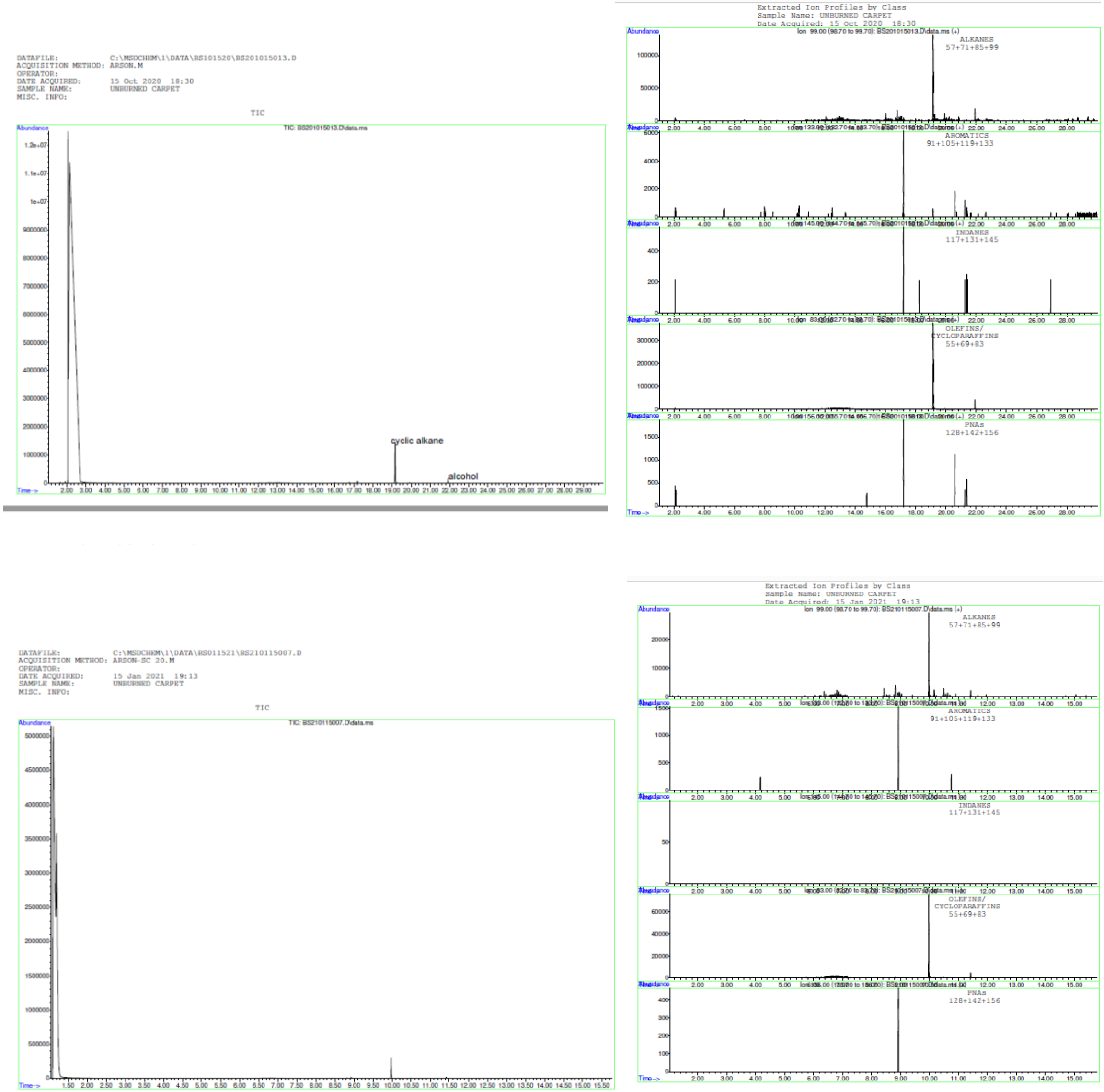


Figure 45. Gasoline (50% Weathered)

APPENDIX B: BURNED AND UNBURNED DATA OF MATERIALS COMMONLY FOUND IN HOUSEHOLD FIRES



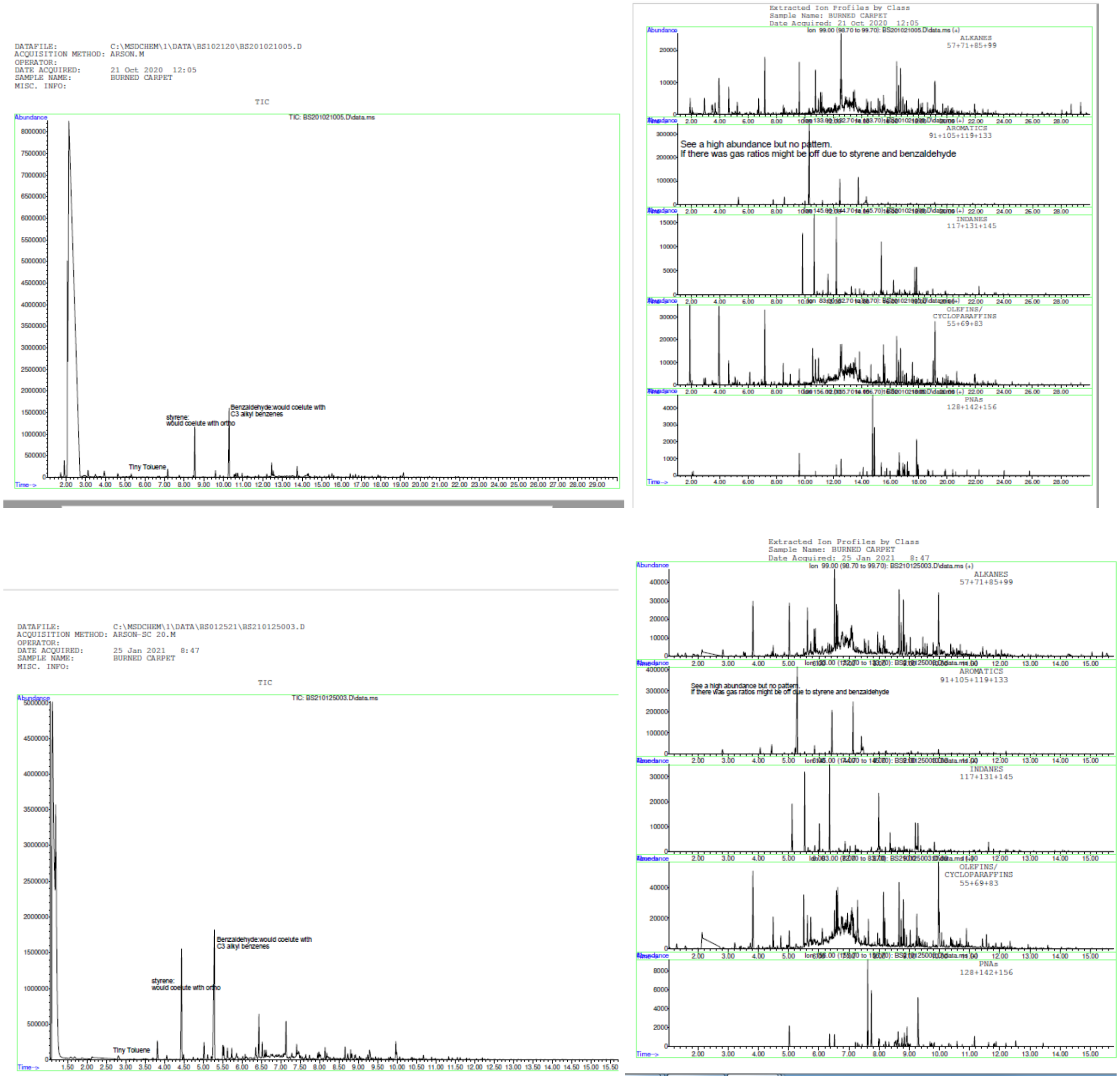
DATAFILE: C:\MSDCHEM\1\DATA\BS101520\BS201015013.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 15 Oct 2020 18:30
SAMPLE NAME: UNBURNED CARPET
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED CARPET
Date Acquired: 15 Oct 2020 18:30

DATAFILE: C:\MSDCHEM\1\DATA\BS011521\BS210115007.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 15 Jan 2021 19:13
SAMPLE NAME: UNBURNED CARPET
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED CARPET
Date Acquired: 15 Jan 2021 19:13

Figure 46. Unburned Carpet



DATAFILE: C:\MSDCHEM\1\DATA\BS102120\BS201021005.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 21 Oct 2020 12:05
SAMPLE NAME: BURNED CARPET
MISC. INFO:

TIC

Tiny Toluene

styrene:
would coelute with ortho

Benzaldehyde:would coelute with
C3 alkyl benzenes

Extracted Ion Profiles by Class
Sample Name: BURNED CARPET
Date Acquired: 21 Oct 2020 12:05

See a high abundance but no pattern.
If there was gas ratios might be off due to styrene and benzaldehyde

DATAFILE: C:\MSDCHEM\1\DATA\BS012521\BS210125003.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 25 Jan 2021 8:47
SAMPLE NAME: BURNED CARPET
MISC. INFO:

TIC

Tiny Toluene

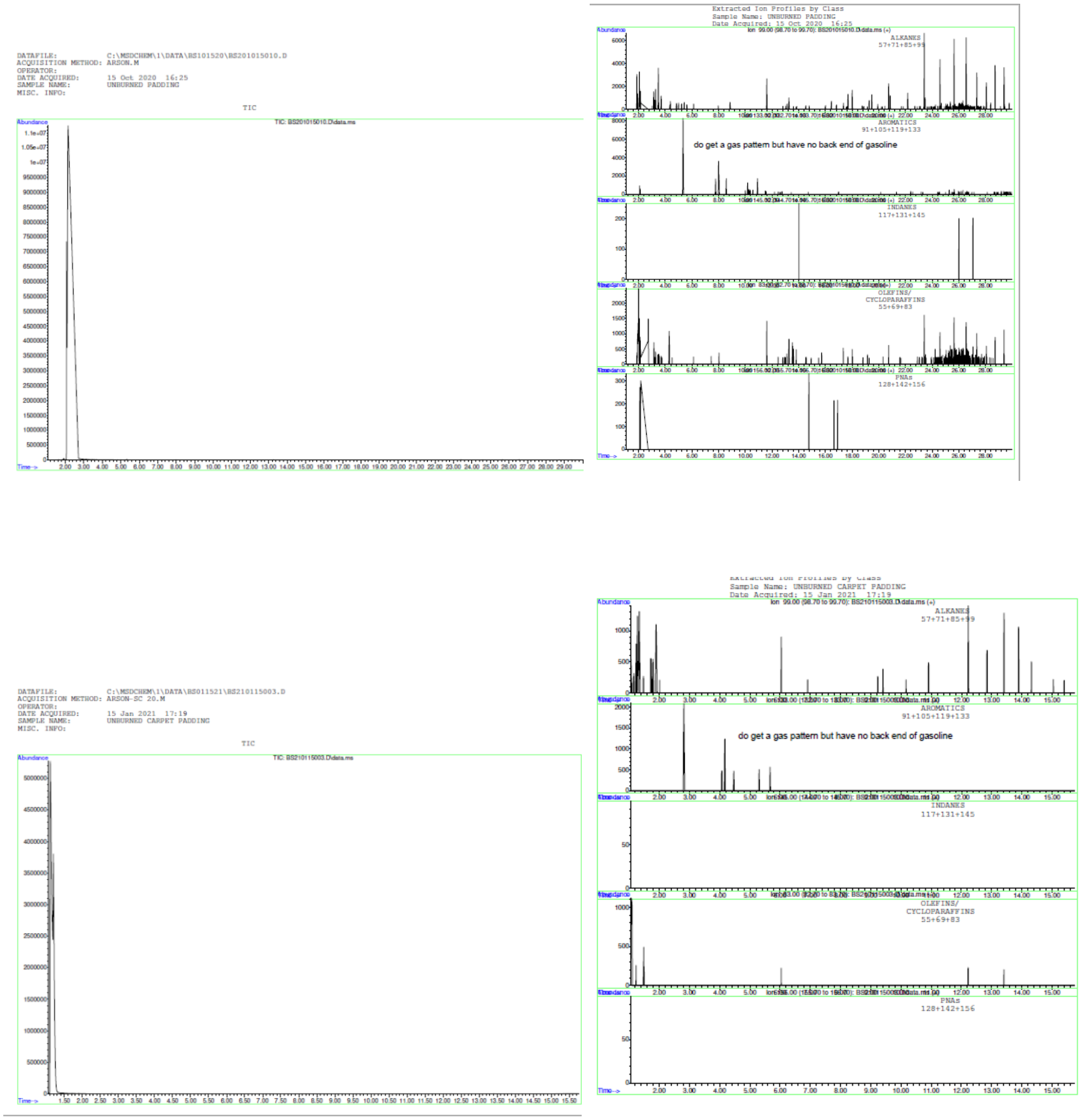
styrene:
would coelute with ortho

Benzaldehyde:would coelute with
C3 alkyl benzenes

Extracted Ion Profiles by Class
Sample Name: BURNED CARPET
Date Acquired: 25 Jan 2021 8:47

See a high abundance but no pattern.
If there was gas ratios might be off due to styrene and benzaldehyde

Figure 47. Burned Carpet



DATAFILE: C:\MSDCHEM\1\DATA\BS101520\BS201015010.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 15 Oct 2020 16:25
SAMPLE NAME: UNBURNED PADDING
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED PADDING
Date Acquired: 15 Oct 2020 16:25

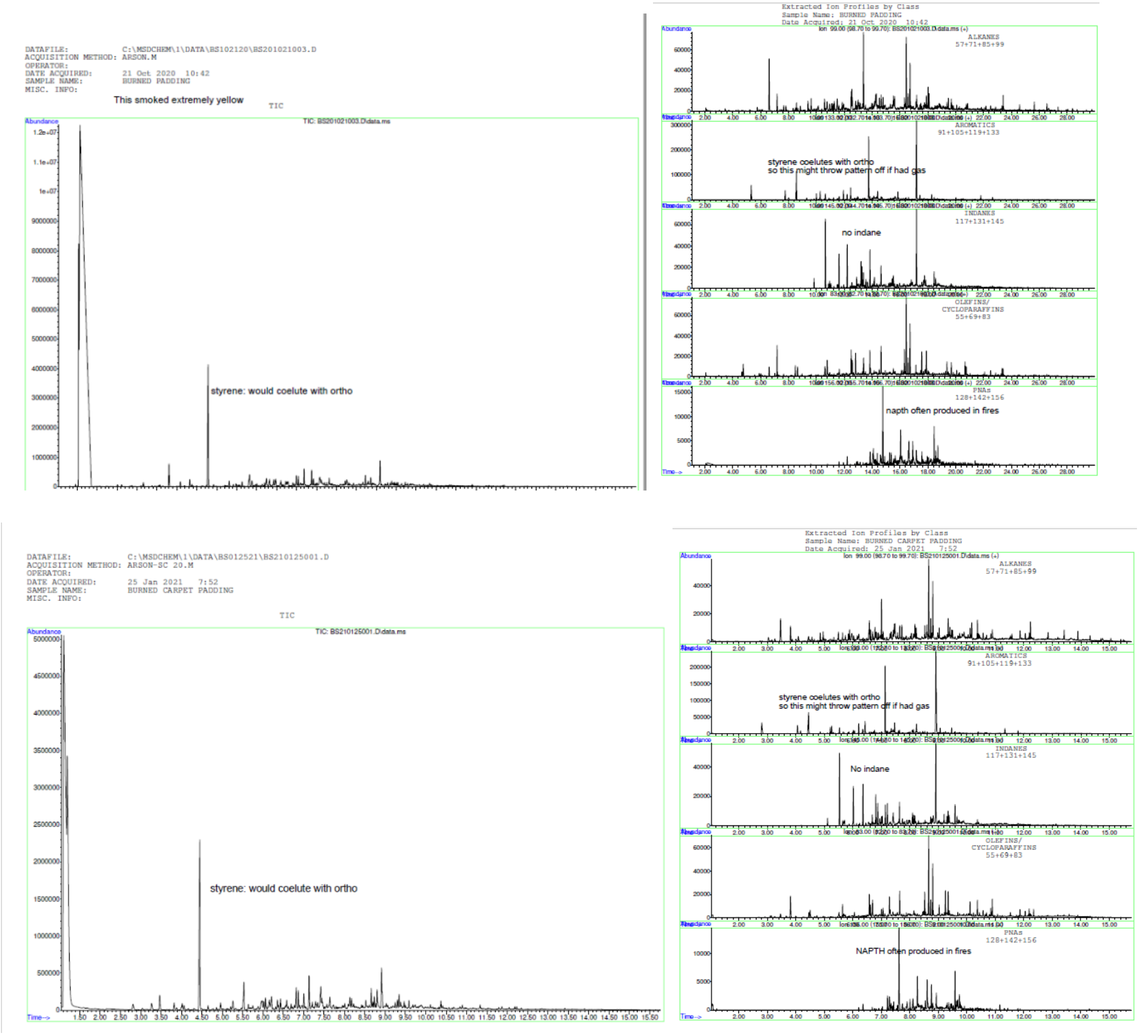
do get a gas pattern but have no back end of gasoline

DATAFILE: C:\MSDCHEM\1\DATA\BS011521\BS210115003.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 15 Jan 2021 17:19
SAMPLE NAME: UNBURNED CARPET PADDING
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED CARPET PADDING
Date Acquired: 15 Jan 2021 17:19

do get a gas pattern but have no back end of gasoline

Figure 48. Unburned Carpet padding



DATAFILE: C:\MSDCHEM\1\DATA\BS102120\BS201021003.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 21 Oct 2020 10:42
SAMPLE NAME: BURNED PADDING
MISC. INFO:

This smoked extremely yellow

styrene: would coelute with ortho

Extracted Ion Profiles by Class
Sample Name: BURNED PADDING
Date Acquired: 21 Oct 2020 10:42

styrene coelutes with ortho
so this might throw pattern off if had gas

no indane

napth often produced in fires

DATAFILE: C:\MSDCHEM\1\DATA\BS012521\BS210125001.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 25 Jan 2021 7:52
SAMPLE NAME: BURNED CARPET PADDING
MISC. INFO:

styrene: would coelute with ortho

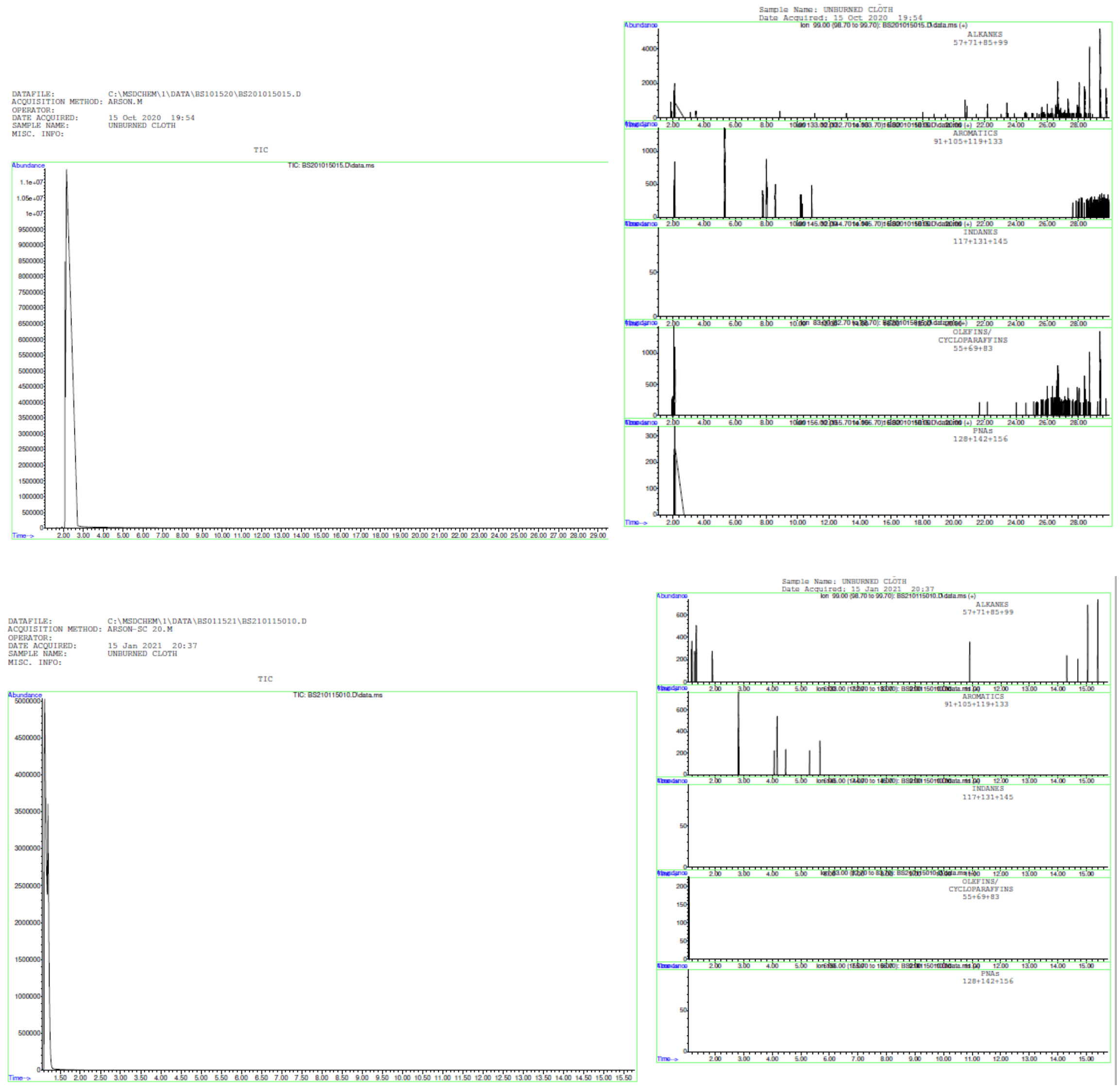
Extracted Ion Profiles by Class
Sample Name: BURNED CARPET PADDING
Date Acquired: 25 Jan 2021 7:52

styrene coelutes with ortho
so this might throw pattern off if had gas

No indane

NAPTH often produced in fires

Figure 49. Burned Carpet Padding



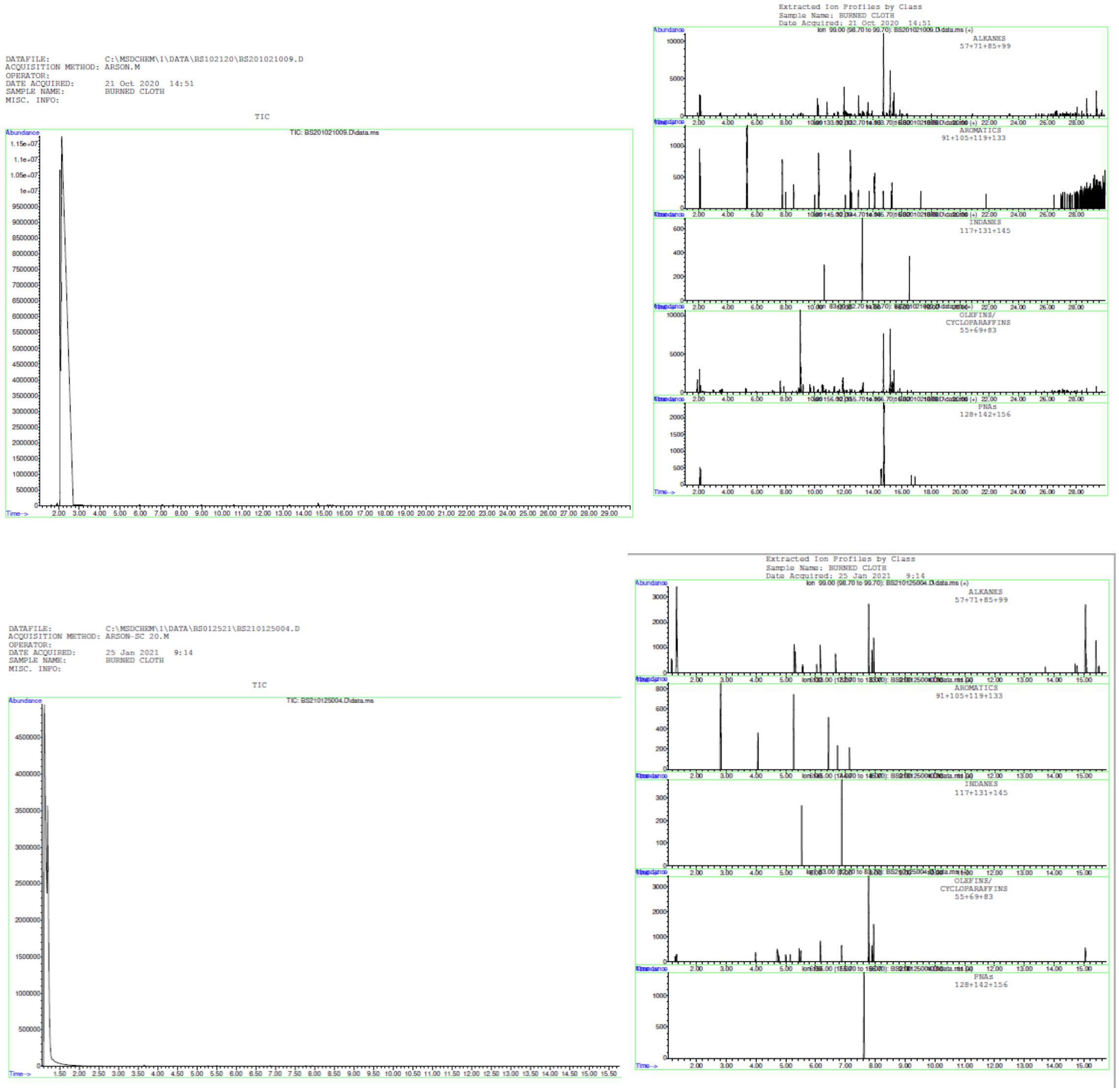
DATAFILE: C:\MSDCHEM\1\DATA\BS101520\BS201015015.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 15 Oct 2020 19:54
SAMPLE NAME: UNBURNED CLOTH
MISC. INFO:

Sample Name: UNBURNED CLOTH
Date Acquired: 15 Oct 2020 19:54

DATAFILE: C:\MSDCHEM\1\DATA\BS011521\BS210115010.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 15 Jan 2021 20:37
SAMPLE NAME: UNBURNED CLOTH
MISC. INFO:

Sample Name: UNBURNED CLOTH
Date Acquired: 15 Jan 2021 20:37

Figure 50. Unburned 100% Cotton Cloth (new)



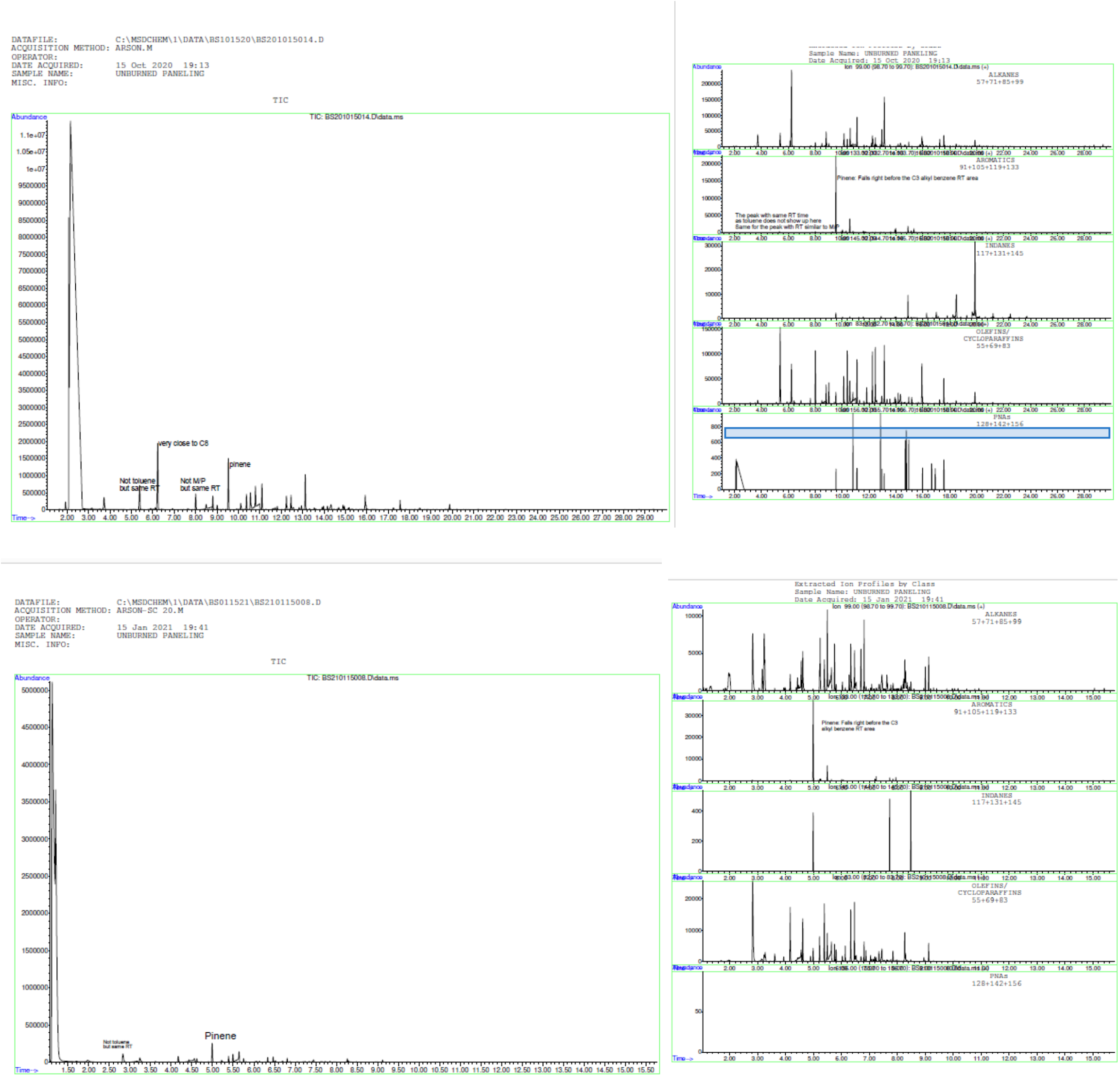
DATAFILE: C:\MSDCHEM\1\DATA\BS102120\BS201021009.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 21 Oct 2020 14:51
SAMPLE NAME: BURNED CLOTH
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: BURNED CLOTH
Date Acquired: 21 Oct 2020 14:51

DATAFILE: C:\MSDCHEM\1\DATA\BS012521\BS210125004.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 25 Jan 2021 9:14
SAMPLE NAME: BURNED CLOTH
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: BURNED CLOTH
Date Acquired: 25 Jan 2021 9:14

Figure 51. Burned 100% Cotton Cloth (new)



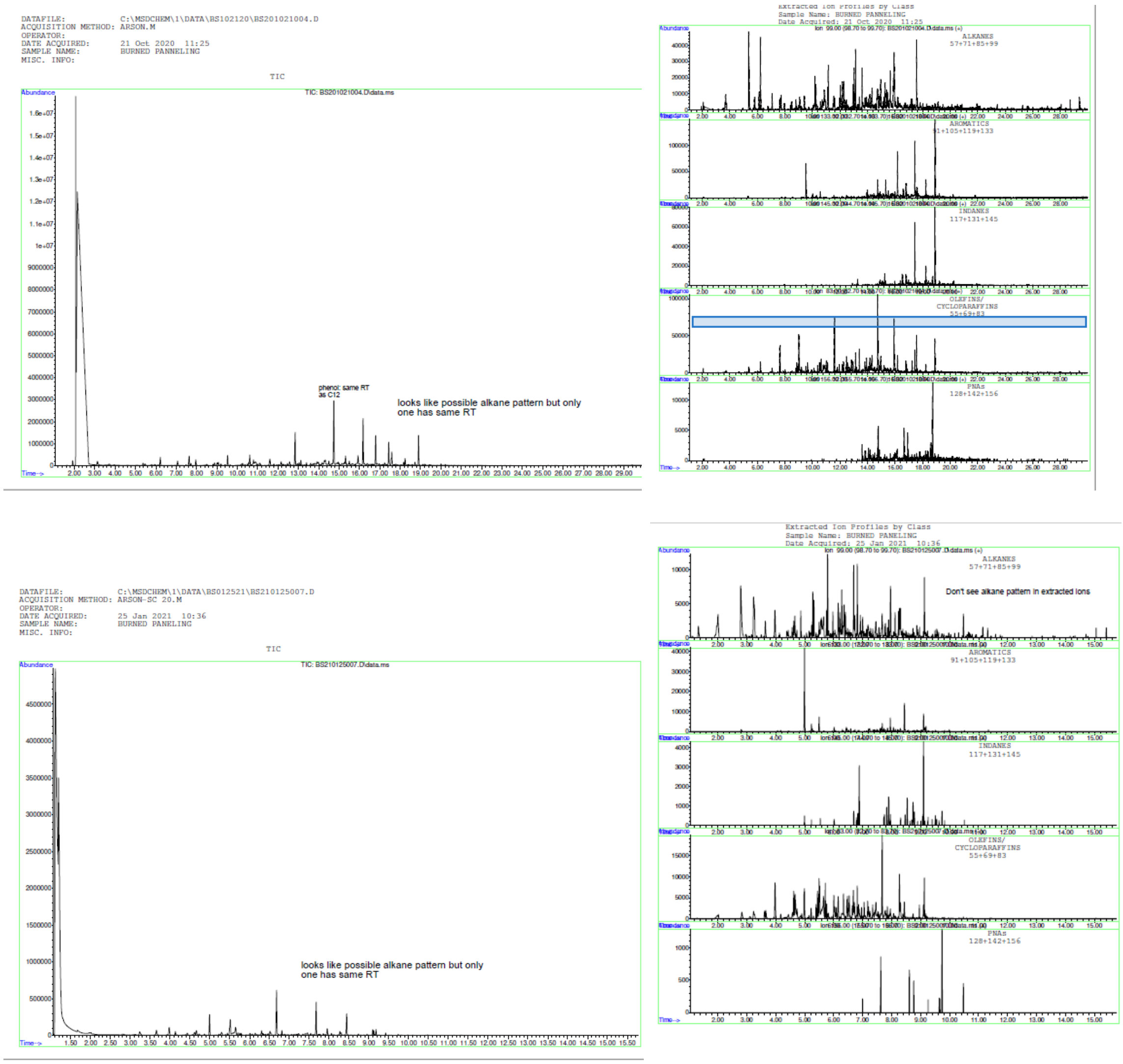
DATAFILE: C:\MSDCHEM\1\DATA\BS101520\BS201015014.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 15 Oct 2020 19:13
SAMPLE NAME: UNBURNED PANELING
MISC. INFO:

Sample Name: UNBURNED PANELING
Date Acquired: 15 Oct 2020 19:13

DATAFILE: C:\MSDCHEM\1\DATA\BS011521\BS210115008.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 15 Jan 2021 19:41
SAMPLE NAME: UNBURNED PANELING
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED PANELING
Date Acquired: 15 Jan 2021 19:41

Figure 52. Unburned Paneling



DATAFILE: C:\MSDCHEM\1\DATA\BS102120\BS201021004.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 21 Oct 2020 11:25
SAMPLE NAME: BURNED PANNELING
MISC. INFO:

phenol: same RT as C12

looks like possible alkane pattern but only one has same RT

Extracted Ion Profiles by Class
Sample Name: BURNED PANNELING
Date Acquired: 21 Oct 2020 11:25

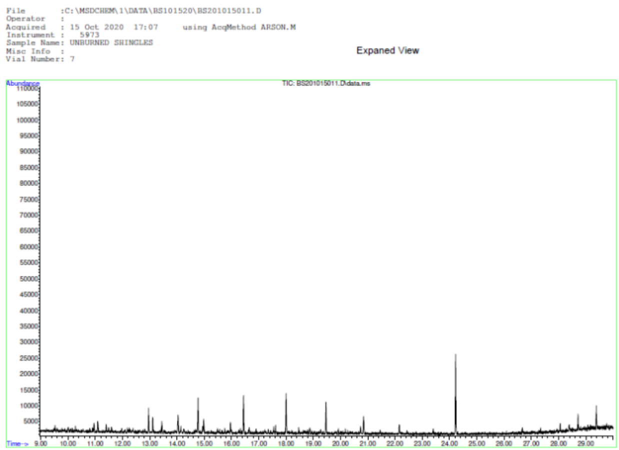
DATAFILE: C:\MSDCHEM\1\DATA\BS012521\BS210125007.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 25 Jan 2021 10:36
SAMPLE NAME: BURNED PANELING
MISC. INFO:

looks like possible alkane pattern but only one has same RT

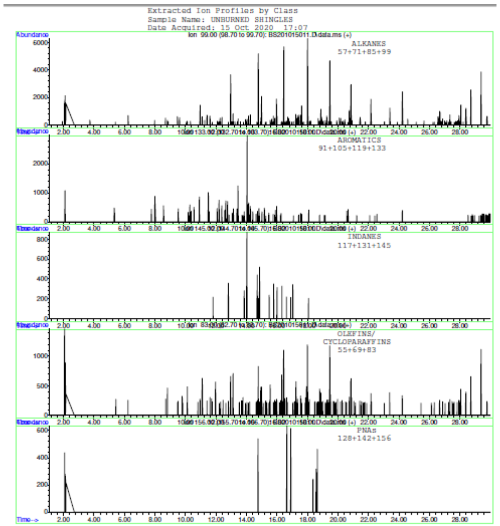
Extracted Ion Profiles by Class
Sample Name: BURNED PANELING
Date Acquired: 25 Jan 2021 10:36

Don't see alkane pattern in extracted ions

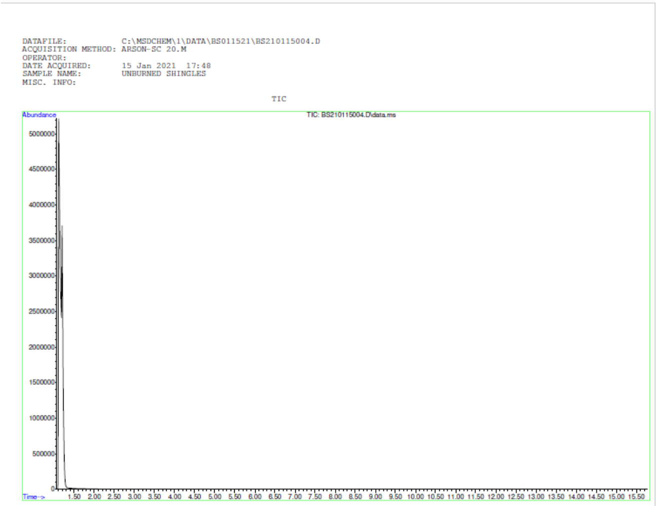
Figure 53. Burned Paneling



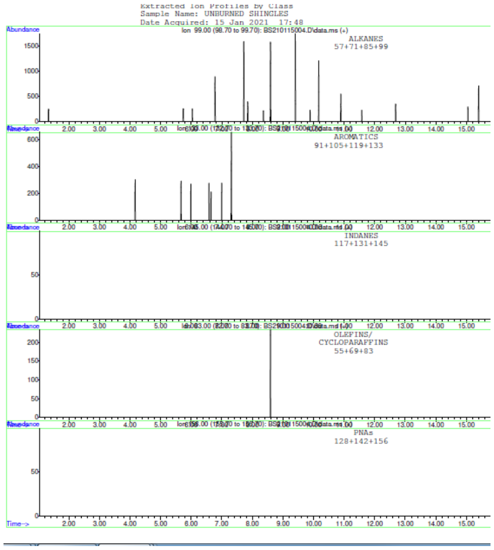
```
File        :C:\MSDCHEM\1\DATA\BS101520\BS201015011.D
Operator    :
Acquired    : 15 Oct 2020  17:07     using AcqMethod ARSON.M
Instrument  :   5973
Sample Name: UNBURNED SHINGLES
Misc Info   :
Vial Number: 7
```



```
Extracted Ion Profiles by Class
Sample Name: UNBURNED SHINGLES
Date Acquired: 15 Oct 2020  17:07
```

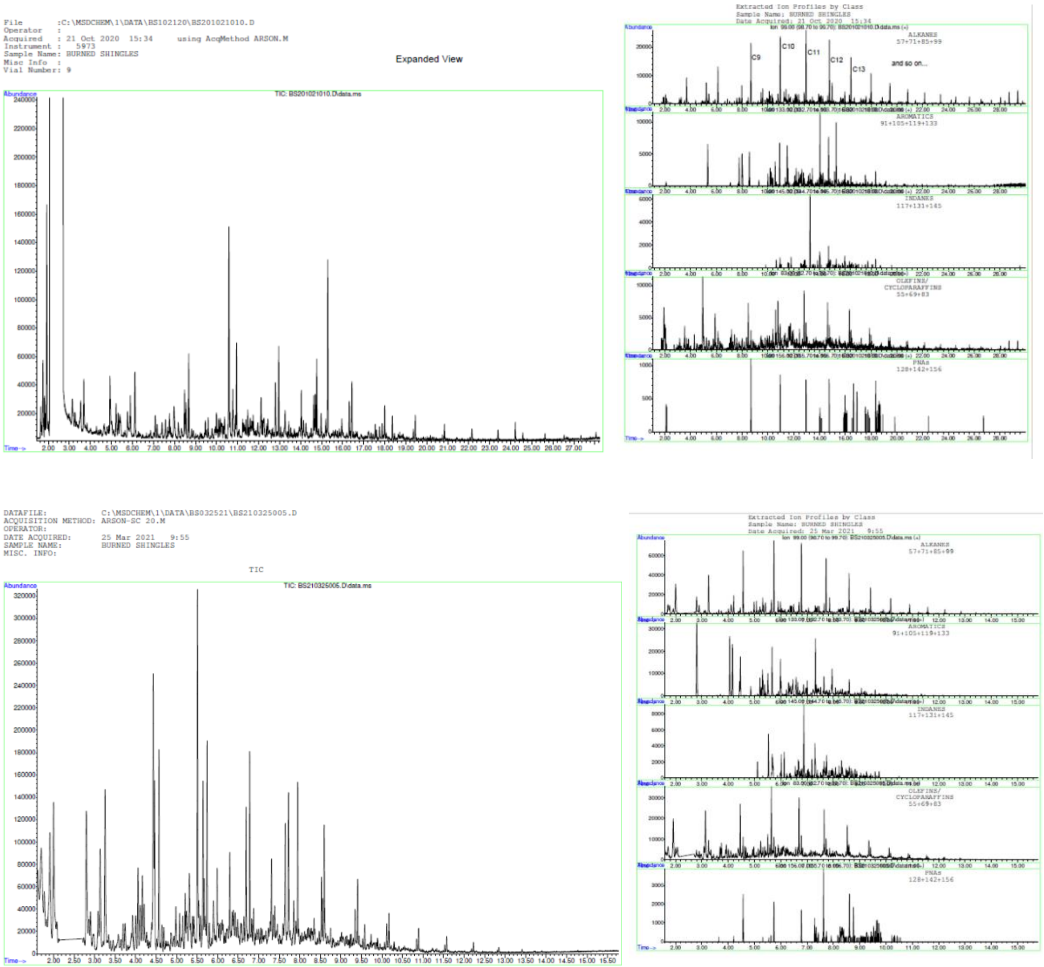


```
DATAFILE:            C:\MSDCHEM\1\DATA\BS011521\BS210115004.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:       15 Jan 2021  17:48
SAMPLE NAME:         UNBURNED SHINGLES
MISC. INFO:
```



```
Extracted Ion Profiles by Class
Sample Name: UNBURNED SHINGLES
Date Acquired: 15 Jan 2021  17:48
```

Figure 54. Unburned Shingles



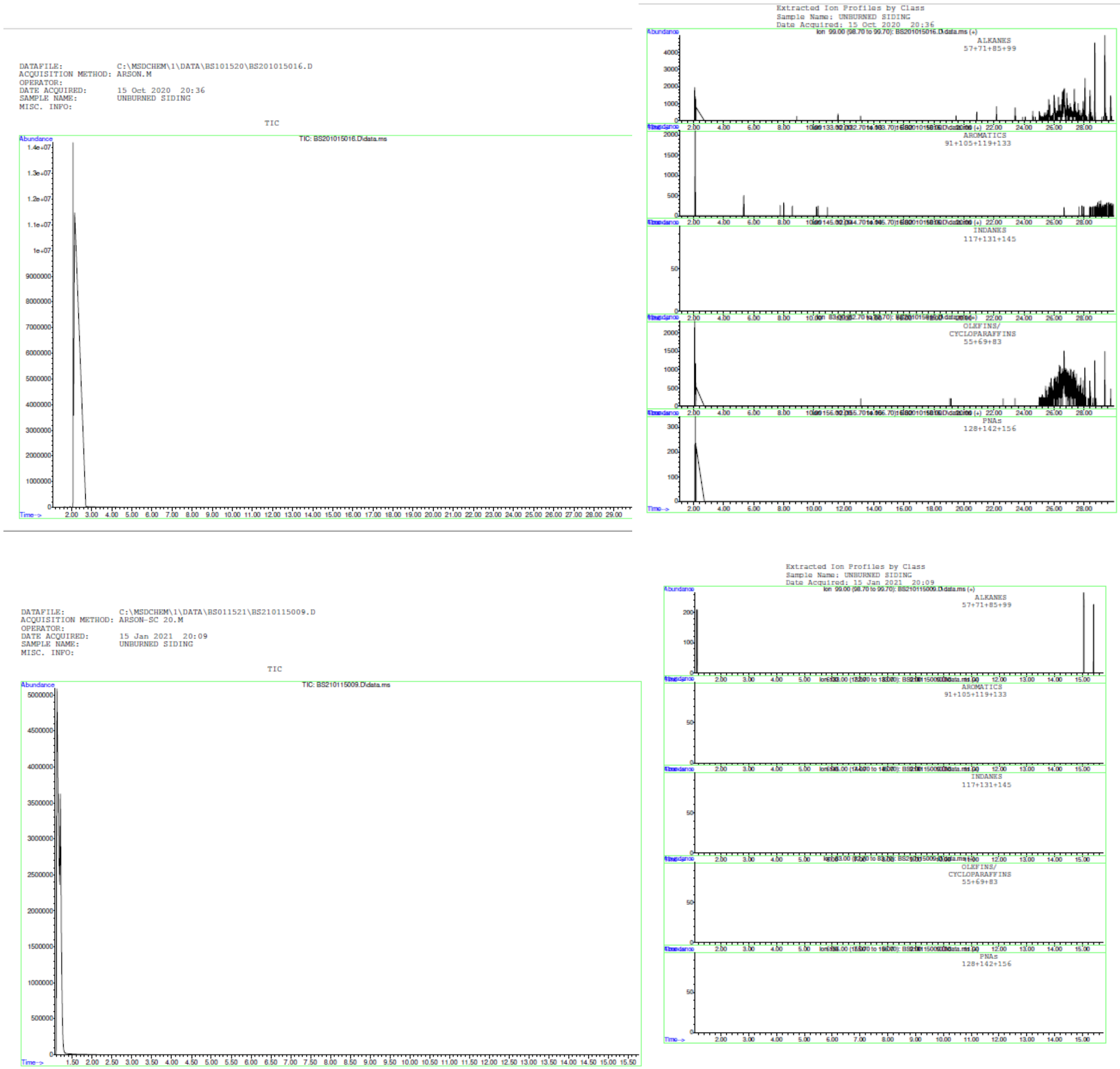
```
File        :C:\MSDCHEM\1\DATA\BS102120\BS201021010.D
Operator    :
Acquired    : 21 Oct 2020  15:34     using AcqMethod ARSON.M
Instrument  :   5973
Sample Name: BURNED SHINGLES
Misc Info   :
Vial Number: 9
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED SHINGLES
Date Acquired: 21 Oct 2020  15:34
```

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS032521\BS210325005.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      25 Mar 2021   9:55
SAMPLE NAME:        BURNED SHINGLES
MISC. INFO:
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED SHINGLES
Date Acquired: 25 Mar 2021   9:55
```

Figure 55. Burned Shingles



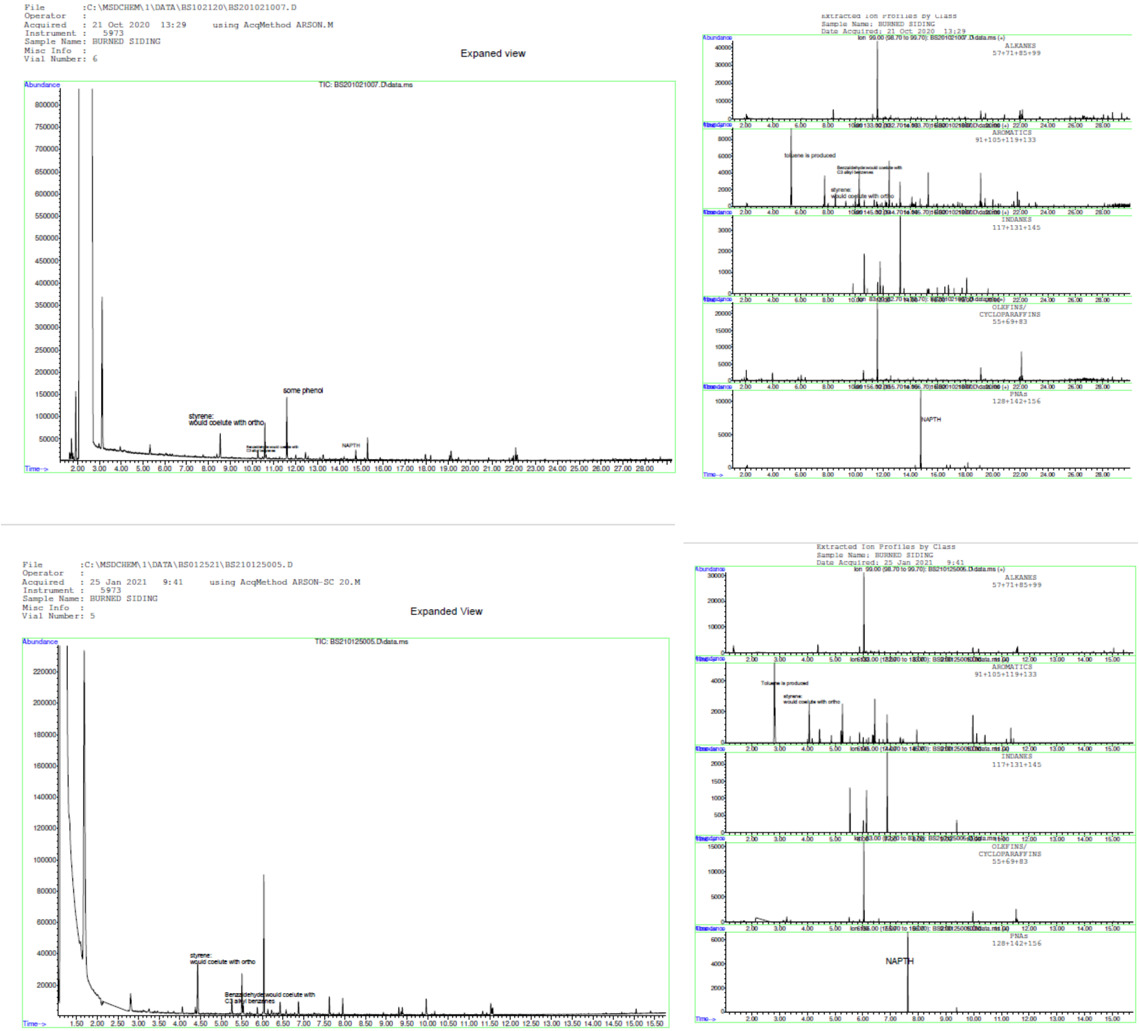
```
DATAFILE:           C:\MSDCHEM\1\DATA\BS101520\BS201015016.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:      15 Oct 2020  20:36
SAMPLE NAME:        UNBURNED SIDING
MISC. INFO:
```

```
Extracted Ion Profiles by Class
Sample Name: UNBURNED SIDING
Date Acquired: 15 Oct 2020  20:36
```

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS011521\BS210115009.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      15 Jan 2021  20:09
SAMPLE NAME:        UNBURNED SIDING
MISC. INFO:
```

```
Extracted Ion Profiles by Class
Sample Name: UNBURNED SIDING
Date Acquired: 15 Jan 2021  20:09
```

Figure 56. Unburned Siding



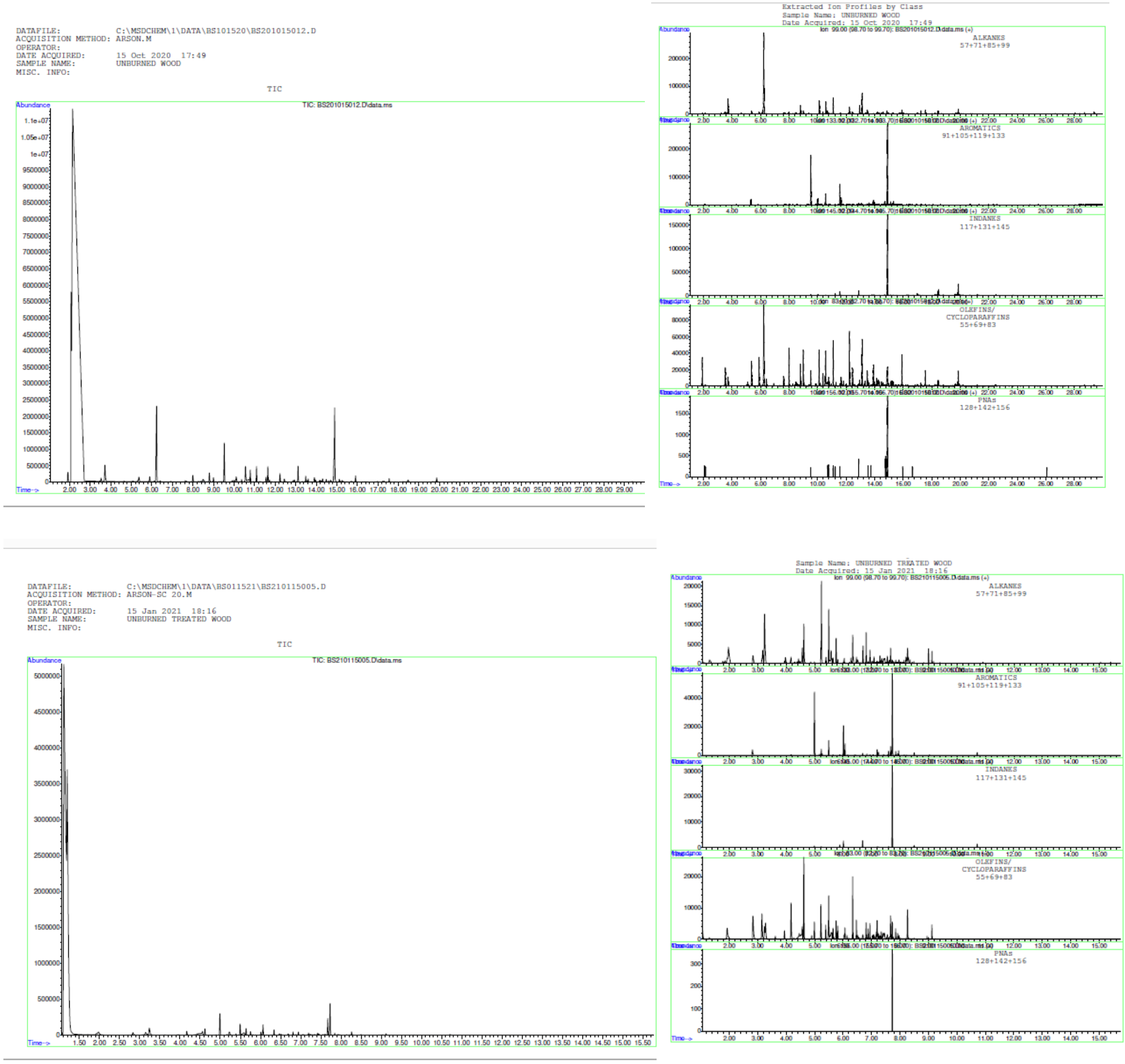
```
File       :C:\MSDCHEM\1\DATA\BS102120\BS201021007.D
Operator   :
Acquired   : 21 Oct 2020  13:29     using AcqMethod ARSON.M
Instrument :   5973
Sample Name: BURNED SIDING
Misc Info  :
Vial Number: 6
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED SIDING
Date Acquired: 21 Oct 2020  13:29
```

```
File       :C:\MSDCHEM\1\DATA\BS012521\BS210125005.D
Operator   :
Acquired   : 25 Jan 2021   9:41     using AcqMethod ARSON-SC 20.M
Instrument :   5973
Sample Name: BURNED SIDING
Misc Info  :
Vial Number: 5
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED SIDING
Date Acquired: 25 Jan 2021   9:41
```

Figure 57. Burned Siding



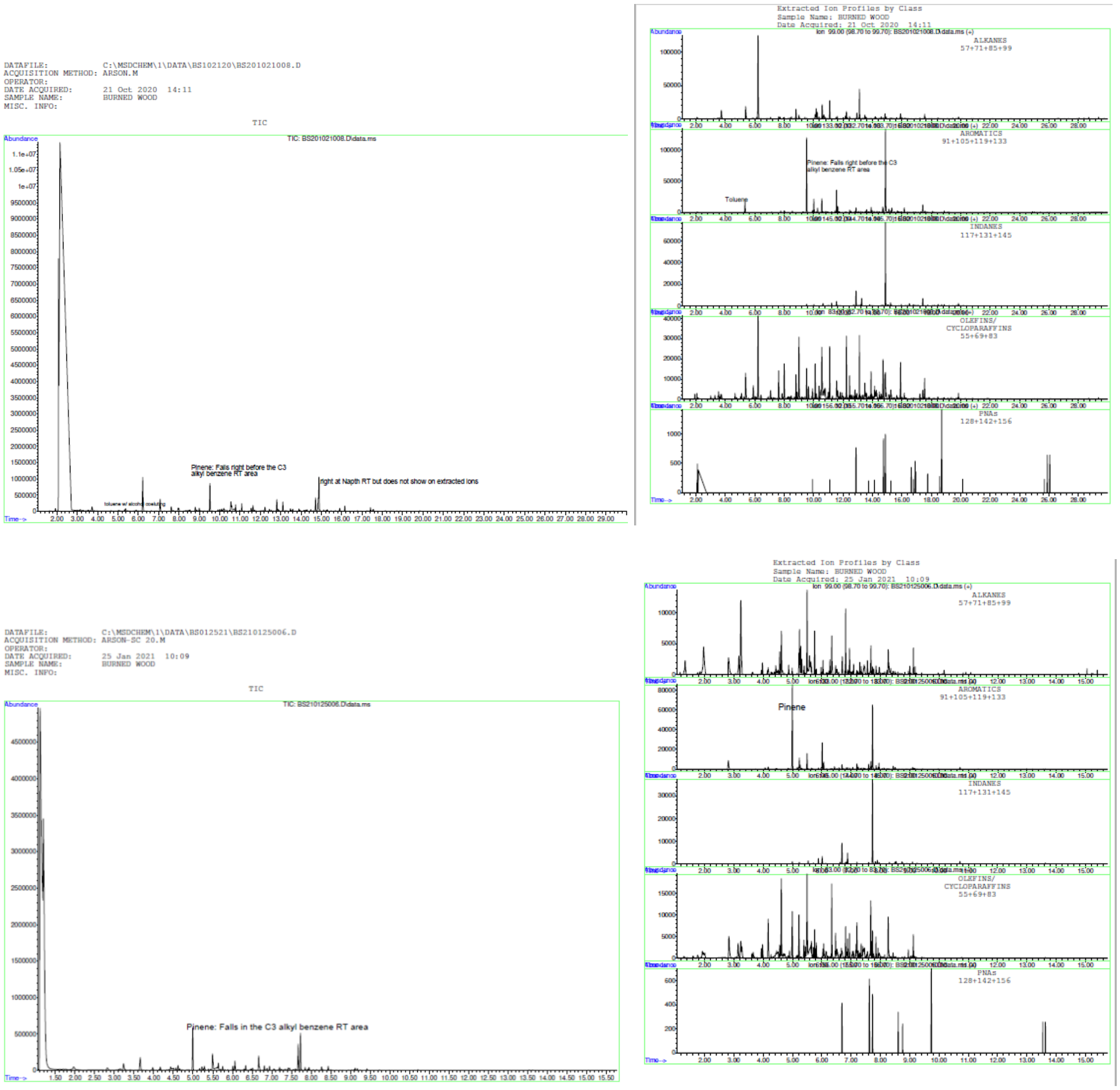
DATAFILE: C:\MSDCHEM\1\DATA\BS101520\BS201015012.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED: 15 Oct 2020 17:49
SAMPLE NAME: UNBURNED WOOD
MISC. INFO:

Extracted Ion Profiles by Class
Sample Name: UNBURNED WOOD
Date Acquired: 15 Oct 2020 17:49

DATAFILE: C:\MSDCHEM\1\DATA\BS011521\BS210115005.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED: 15 Jan 2021 18:16
SAMPLE NAME: UNBURNED TREATED WOOD
MISC. INFO:

Sample Name: UNBURNED TREATED WOOD
Date Acquired: 15 Jan 2021 18:16

Figure 58. Unburned Treated Wood



```
DATAFILE:           C:\MSDCHEM\1\DATA\BS102120\BS201021008.D
ACQUISITION METHOD: ARSON.M
OPERATOR:
DATE ACQUIRED:      21 Oct 2020  14:11
SAMPLE NAME:        BURNED WOOD
MISC. INFO:
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED WOOD
Date Acquired: 21 Oct 2020  14:11
```

toluene w/ alcohol coeluting

Pinene: Falls right before the C3 alkyl benzene RT area

right at Napth RT but does not show on extracted Ions

```
DATAFILE:           C:\MSDCHEM\1\DATA\BS012521\BS210125006.D
ACQUISITION METHOD: ARSON-SC 20.M
OPERATOR:
DATE ACQUIRED:      25 Jan 2021  10:09
SAMPLE NAME:        BURNED WOOD
MISC. INFO:
```

```
Extracted Ion Profiles by Class
Sample Name: BURNED WOOD
Date Acquired: 25 Jan 2021  10:09
```

Pinene: Falls in the C3 alkyl benzene RT area

Figure 59. Burned Treated Wood

VITA

Mary E. Stuart

Candidate for the Degree of

Doctor of Philosophy

Dissertation: ADVANCEMENTS IN COLLECTION AND ANALYSIS OF FIRE DEBRIS-IMPROVED FIELD COLLECTION KITS WITH FAST GAS CHROMATOGRAPHY

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Completed the requirements for the Doctor of Philosophy in Forensic Sciences at Oklahoma State University, Stillwater, Oklahoma in May, 2023.

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Completed the requirements for the Bachelor of Science in Chemistry and Biochemistry at College of Charleston, Charleston, South Carolina in 1999.

Experience:

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Professional Memberships:

Clandestine Laboratory Investigating Chemists (2004-present)