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Eastern Kentucky University

Oil Dries Used in Arson Scenes: A New Absorbent Material

Honors Thesis Submitted In Partial Fulfillment Of The Requirement of HON 420 Fall 2016

> By Jessica Dove

Faculty Mentor Mrs. Barbara Wheeler Department of Chemistry, Forensic Science Program

#### Oil Dries Used in Arson Scenes: New Absorbent Material

#### Jessica Dove

#### Mrs. Barbra Wheeler, Department of Chemistry; Forensic Science Program

Abstract: In many fire investigation cases, it is hard to find evidence that will lead to the cause or person that started the fire, especially within arson scenes. Because of this, the identification of ignitable liquids is critical to an arson investigation. Obtaining enough evidential information from the scene is key if arson is suspected. Many scenes involve ignitable liquids that have been poured onto furniture or carpets, allowing the ignitable liquid to easily be collected. However, in some investigations ignitable liquids are poured onto semi-porous surfaces such as concrete. Collection of these materials is difficult. Oil dries are absorbents commonly used to clean up spills, at scenes, involving hazardous materials. Since many hazardous material spills involve petroleum products, it is suspected that the oil dries might also absorb ignitable liquids. Studies done in the past on creating a solid absorbent have not been successful, while some household absorbent have been somewhat beneficial.<sup>1</sup> This research will concentrate on the potential use of different solid oil dry absorbents to be used at a fire scene to assist in the collection of questioned arson samples. All analysis of samples will be conducted using a technique commonly used in forensic laboratories, gas chromatography with flame ionization detection.

*Keyword and phrases:* Arson Analysis, Forensics Science, Arson Investigation, Hazardous Material Clean Up, Oil Dry, Gas Chromatography with Flame Ionization Detector.

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#### **Introduction**

In 2015 1,345,500 fires were reported within the United States of America.<sup>2</sup> Fire is a rapid and sustained oxidation process with the evolution of light and heat at varying intensities. Of the fires in 2015, 3,280 civilian deaths occurred, 15,700 civilians were injured, and approximately 14.3 billion dollars in property damage.<sup>2</sup> Four things are required for fire; an oxidizing agent (air), a fuel, an ignition source and a chain reaction. For most fires, the oxygen in the air is the oxidizing agent. Fuels can be a variety of compounds; solids such as charcoal, gases such as propane or liquids such as alcohol or petroleum products. Ignition sources can be natural, accidental or intentional. Combustion is the chemical reaction between the oxidizer and a fuel that has been ignited. This feeds a fire more heat and allows it to continue while the supply of the oxidizer and fuel remain.

When either property damage or injury/death of a person happens from a fire, an investigation is brought forth. The investigation helps determine the ignition source and/or fuel and who possibly started the fire. During a fire investigation, one of the very first questions asked is where is the source of the fire? This is a critical question within the investigation because close examination of the fires origin could help the investigator determine whether the fire was intentional or not. Indications of whether a fire was intentional or not are usually given by examining the ignition source and whether or not any additional fuels are present. As stated previously, an ignition source can be natural, accidental or intentional. Examples of natural ignition sources would include lighting strikes or high winds breaking power lines both of which start a fire. An accidental ignition source would include electrical short or a kerosene heater/gasoline generator

malfunction that would start a fire. The last ignition source is intentional. Intentional ignition is when someone sets a fire on purpose. This may be to start a fire i.e., a campfire or a fire set to harm or damage property. If it is an intentional source, information concerning the fuels present become very important. When ignitable liquids are identified at a fire, arson is suspected.

The crime of arson is when a fire is intentionally set to hurt or damage someone or property. This could be someone trying to get insurance money, or trying to cover up another crime such as a rape or murder. The fire scene becomes a crime scene when the ignition source is intentional and it is thought to be arson. From 2007-2011, 282,600 intentional fires were set. Of these fires, 420 civilian deaths occurred, 1,360 civilians were injured and an estimated 1.3 billion dollars was lost in property damage.<sup>2</sup>

To determine the source at a fire scene presents a challenge to investigators because fire can destroy almost everything. Deeply charred areas and burn patterns can give an indication of the origin. A closer examination may give details of the ignition source and fuel. In the case of arson, the fuel usually involves the addition of ignitable liquids. Common ignitable liquids include petroleum products, such as gasoline, diesel, kerosene, etc. These liquids are most commonly placed onto items such as a chair, couch, or carpet. Once ignited, the furnishings and ignitable liquid adds fuel to the fire. The identification of the ignitable liquids at a scene help prove the intentional crime of arson.

The identification of ignitable liquids requires the collection and analysis of fire debris from the fire scene. The collection phase is important, since the samples must be obtained and preserved so that analysis can be conducted. A variety of collection containers can be used to preserve the sample, but lined "paint" cans are the most common choice. Since common ignitable liquids are volatile, samples must be packaged so that evaporation doesn't occur. The cans give an air tight seal and will not interfere with testing. Glass jars can also be used however a protective layer must be inserted (usually foil) since the rubber rings within of jar lids can produce an interference problem during analysis. While choosing a container for preserving the sample is fairly easy, obtaining the sample at times can be a challenge.

There are different types of surfaces that can samples can be obtained from during an investigation; porous, semi-porous and non-porous. Porous surfaces are upholstered furniture, carpeting, curtains, and table cloths etc. These types of surfaces soak up the ignitable liquid very easily. Semi-porous surfaces will allow liquids to absorb, but not as easily. Wood and concrete are good examples of a semi-porous surface. Non-porous surfaces don't allow the liquid to penetrate the surface. Most porcelain tile is an example of non-porous surfaces. In addition to the surface itself, the sample may also be hard to extract from the scene. Heavy timber and concrete are typically hard to remove and package in a container for analysis. While they are "porous and absorbent material".<sup>3</sup> The investigator must use jack hammers, saws and other tools to break apart the surface to be able to put it into the proper evidence container. The investigators will have to "collect pieces of spalled concrete, chip the concrete away, or drill core samples" using long and hard man hours.<sup>3</sup> This can cause an issue for an investigation due to long hours and heavy man power needed to retrieve possible evidence. This is currently what it takes to obtain ignitable liquid.

#### **Background**

The presence of an ignitable liquid helps to prove arson in most cases. For porous substrates, recovery is fairly simple. However as stated above, semi-porous and nonporous can prove much more difficult. For substrates like this, the possibility of a solid absorbent material may make retrieval of ignitable liquids much easier. To be able to solve the issue of obtaining ignitable liquids from semi-porous surfaces, this paper looks into a solid absorbent material that might be able to obtain the liquids without the heavy, long man hours and power. There has been a study done with calcium carbonate and allpurpose flour and they seem to be good absorbents.<sup>3</sup> These compounds have shown to work well in the collection and detection of gasoline and diesel. However, the downfall of these absorbents is that they might not be easily accessible to the investigator. Another possible material is an oil dry. Oil dries, are absorbent materials used to clean up spills. There are usually granular and are composed of a variety of compounds. However, the question is, will they be able to soak up ignitable liquids from fire scenes for reliable, accurate detection and identification. This paper focus on oil dries, a material which is already being used by emergency response personnel. Therefore, the material would be readily accessible. Hopefully this paper will show that the oil dry is a "divided powder with greater absorptivity than the concrete" allowing the recovery of the ignitable liquids.<sup>3</sup>

Oil dries are typically used during hazardous material scenes to clean up oil, diesels, or other liquids on the roads or dirt. A good example is when a semi-tractor trailer crash happens and all of the fuel from the truck is on the roadway. The oil dry is placed on top of the roadway to soak up the hazardous liquid so the road can be safely used again. There are many different types of oil dries available. This paper looks specifically at six common oil dries to see if they can be used at arson scenes. The six oil dries include Instazorb, Diversified Absorbent, Super Sorbent, COA Chem Oil-Away, Safe T Oil Absorbent, and Universal Organic Absorbent. These six oil dries range in different chemical makeup that could or could not interfere with the isolation and identification within the laboratory. Instazorb is an amorphous aluminum silicate. Diversified Absorbent is a melt blown polyolefin polymer. Super Sorbent is attapulgite, a magnesium aluminium phyllosilicate. COA Chem Oil-Away is volcanic ash. Safe T Oil Absorbent is a montmorillonite clay. Universal Organic Absorbent is a Coir pith fiber and water. Most oil dries are treated with surfactants that lower the surface tension between the absorbent and the liquid which increases its absorbency.

#### **Methods**

Once samples are collected from a fire scene, there are many different methods that can be used for isolation of the ignitable liquid. These include steam distillation, solvent extraction, direct headspace, passive headspace with activated charcoal strip, dynamic headspace concentration, and passive headspace concentration with solid phase microextraction (SPME).<sup>4</sup> Steam distillation uses heat to separate mixtures based on the volatility of a boiling liquid. Samples are heated creating vapors which are collected by a cooled condensing column. Vapors will rise, and separation occurs as they pass through a cooled condenser. These separations can be collected for further analysis. Solvent extraction is where a particular solvent is applied to the fire debris sample. The solvent is then collected for analysis. Direct headspace uses the volatiles from the debris which rise

to the top of the container. A syringe is placed into the container to extract the gas from the container for analysis. Dynamic headspace uses inert gas to purge the sample container. Volatiles are trapped on a solid sorbent material. The solid sorbent material is then placed in an organic solvent to elute the volatiles. The most common method however is the passive headspace. There are two forms of passive headspace one that uses an activated charcoal strip, and the other uses the solid phase micro-extraction (SPME). With passive headspace, volatiles are trapped onto a solid sorbent substance. With the most common form of passive headspace, the solid sorbent is an activated charcoal strip. With SPME, the solid sorbent is a fiber. There are several different types of fibers which can be used for hydrocarbons. There is also a difference between the analytical process. With SPME the fire debris is heated for 10 minutes, and the solid phase micro-extraction fiber is placed into the container to trap the vapors. The SPME fiber is placed directly into the instrument for analysis without an elution process. When the activated charcoal strip is used, it is suspended over the fire debris in the container and heated for approximately sixteen hours. Once heated the carbon strip will trap any volatiles that can be eluted by an organic solvent. This eluted solvent can be used for further analysis.

Just like there are variations of the isolation method, there are also two instruments that can be used to identify ignitable liquids; Gas Chromatography with Flame Ionization Detector (GC-FID) or Gas Chromatography with Mass Spectrometry (GC-MS). GC-MS uses the ion spectra of the fragmented sample to identify possible petroleum distillates present.<sup>5</sup> GC-FID uses the retention times of peaks to help identify possible petroleum distillates present. The overall peak pattern of the chromatograph is related to a specific petroleum distillate. This paper focuses on isolation by passive headspace with activated charcoal strip and identification by GC-FID.

Samples are isolated by passive headspace sampling using an DFLEX activated charcoal strip. Activated charcoal is carbon that has been treated with oxygen to create a highly porous charcoal substrate. The porous surface of an activated charcoal strip allows vapors to pass through the charcoal and chemically bind with the exposed carbon. The charcoal strip can be used with any ignitable liquid. The strips "do not absorb water or nitrogen but have a high affinity for hydrocarbons and being resistant to oxidation."<sup>6</sup> When using this isolation technique, a charcoal strip is attached to a paper clip which is then suspended by string over the solid absorbent within the jar. Aluminum foil is placed over the top of the jar then secured with the jar lid. The aluminum foil prevents the rubber seal of the lid from being exposed to the charcoal strip. Since rubber is composed of hydrocarbons, if exposed to the charcoal strip, interference may be seen in any resulting data. The jar is then placed into a 60°C oven for 16 hours. This lets the sample heat up for an extended time, allowing any ignitable liquids to be vaporized and trapped on the charcoal strip.

After isolation of the sample with passive headspace, it is extracted with carbon disulfide. The extracted samples are then analyzed by GC-FID. The gas chromatograph separates the sample with detection of the various components by flame ionization. Separation of compounds is accomplished in gas chromatography by the use of a mobile phase (carrier gas) and a stationary phase (column). Depending on a samples chemical and physical properties, the interaction with a specific column causes each compound of the sample to elute at different times. A flame ionization detector uses an electrode placed adjacent to a flame which is near the end of the column. The formation of cations and electrons (by pyrolysis of compounds at the flame) creates a current between the electrodes which can be translated into a peak on the chromatogram.

Experimental methods:

Isolation: (A): Isolation was initially performed with oil dry samples only.

Approximately 100 cm<sup>3</sup> of an oil dry sample was placed in a clean mason jar. A DFLEX activated charcoal strip was placed in between a paper clip and attached to a string. This was hung over the oil dry. Foil is placed over the jar and the lid is sealed. The jar was then placed in a 60°C oven for about 16 hours. After allowing the jar to cool, the charcoal strip was removed and placed into a small glass vial. 0.5 mL of carbon disulfide was added to the vial and sealed tightly. This was allowed to desorb for 30 minutes.

(B): Isolation was then done with a petroleum product added.

Approximately 0.5 mL of petroleum product (gasoline, kerosene, diesel) was added to the oil dry sample. Additional steps as described above were then done. Analysis: Hewlett Packard HP 6890 Series GC-FID instrument.

The column was HP-5 5% Phenyl Methyl Siloxane with measurements of 30.0m x 250 $\mu$ m x 0.50  $\mu$ m. The method parameters are as follows: initial oven temperature of 50°C. The temperature was increased 25°C per minute to 160°C. Final temperature was held for 1 minute. 1  $\mu$ L of the prepared sample was injected.

Several factors come into play when an examination procedure is being modified, as in this case with the proposed oil dry use. This involves consideration of several possible "contamination" concerns. Before the testing could begin, it was necessary to make sure that these were addressed so there was no unforeseen interference. Interference would cause problems when interpreting data results.

The initial contamination concern was addressed by using personal protection equipment and rinsed syringes while performing all steps of analysis. The activated charcoal strip was only touched by gloved hands, preventing the addition of samples onto the charcoal strips. Syringes used for injection of samples into the GC-FID where rinsed multiple times prior to injections.

The second contamination concern was addressed by running blanks in-between samples. This assured that there was no sample carryover, causing problems with interpretation of data.

The final concern involved the sample itself. If an oil dry exhibited peaks which would interfere with petroleum distillate interpretations, it could not be used. For instance, if the oil dry sample separates and creates peaks in the same location as a petroleum product, the peaks cause by any petroleum product present may "be hidden" in the resulting chromatogram.

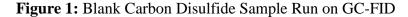
#### Intended Analytical Research Scheme

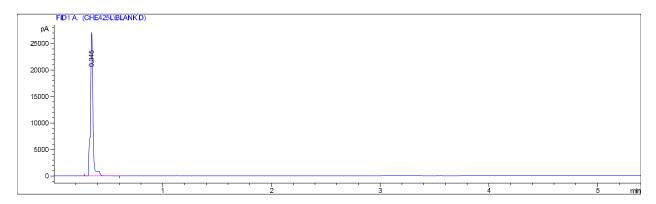
1. Initial runs to see if oil dries would have background interface.

- Initial runs to see if oil dries will absorb petroleum liquids; kerosene, diesel, and gasoline.
- 3. Initial runs to see if oil dries will absorb petroleum liquids from a semi-pours surface.

## **Results**

A blank sample of carbon disulfide is shown in **Figure 1**. The blank sample run shows that there will be no interference from the instrument or in other words, that all resulting data is actually from the sample being tested. In the blank sample run, there is one peak at the very beginning of the chromatogram. This shows that the carbon disulfide does not separate to produce peaks, therefore giving no interference.

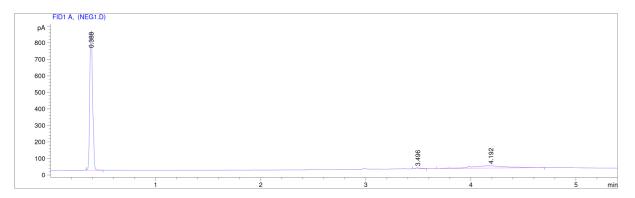




After a blank sample was run the sample name was changed to the oil dry being tested and then that sample was run as previously described. These chromatograms are referred to as "negative control runs" since the sample being tested is the oil dry product without any petroleum product added. All resulting chromatograms were printed and saved as a pdf file. This process was used for all of the initial runs for the solid absorbents. This determined whether or not the oil dry product would interfere with

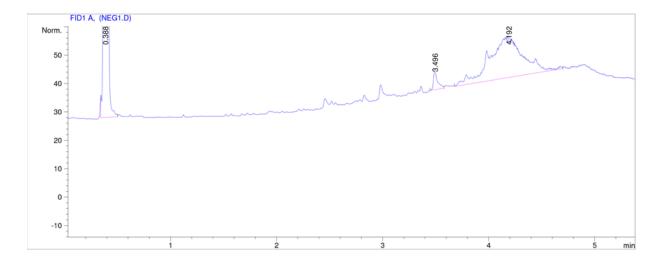
further testing. Figures 2, 4, 5, 6, 8, and 9 show the initial results of the oil dries. Figures 3 and 7 have been integrated.

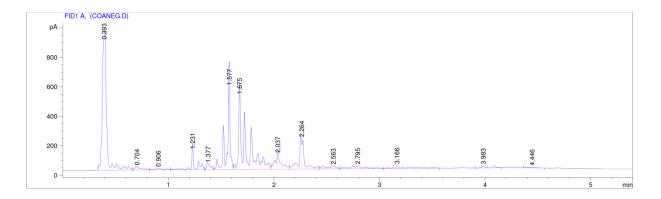
Figure 2: Instazorb Negative Control Run on GC-FID



As shown above, there is no separation from the Instazorb oil dry. There is a little peak just before the 4 minute mark, but when zoomed in shown in **Figure 3** it is well within the background range of the instrument. This means that these marked peaks did not come from the oil dry, but from the normal operating background of the instrument.

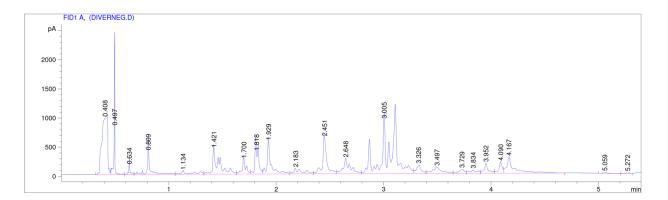
Figure 3: Instazorb Negative Control Run on GC-FID Zoomed In



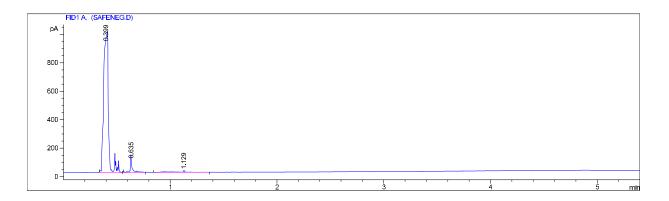


This chromatograph is a negative control run for Chem Oil Away. This chromatograph produces peaks where you would expect to find light petroleum distillates. Due to the fact that no light petroleum products were added, this could make detection of any petroleum products difficult if this oil dry was used. Therefore, Chem Oil Away will not be used for further testing.

Figure 5: Diversified Absorbent Negative Control Run on GC-FID

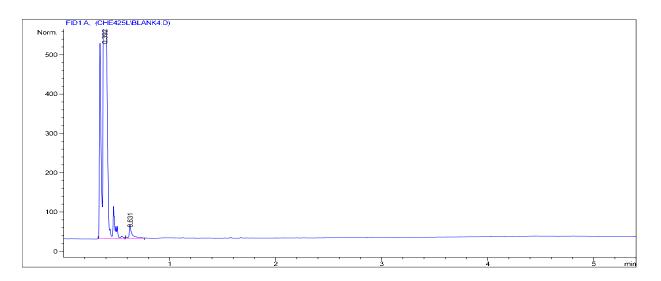


This chromatograph of Diversified Absorbent produces peaks during the light and medium petroleum distillate range. This again would make it nearly impossible to detect the correct petroleum distillate due to the interference. Therefore, Diversified Absorbent will not be used for further testing. Figure 6: Safe T Oil Negative Control Run on GC-FID



The Safe T Oil chromatograph looks very promising, but due to the small peaks at the beginning of the spectrum it cannot be used. This is within the light petroleum distillate range. The integrated view of the Safe T Oil control run is in **Figure 7**.

Figure 7: Safe T Oil Negative Control Run on GC-FID Zoomed In



The peaks are greater than the accepted background of 50 pA therefore it comes from the oil dry and not the background as seen with the Instazorb oil dry negative run. Therefore, Safe T Oil will not be used for further testing.

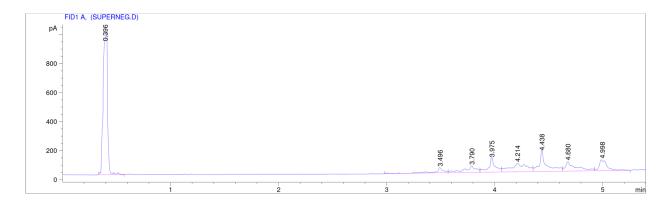


Figure 8: Super Sorbent Negative Control Run on GC-FID

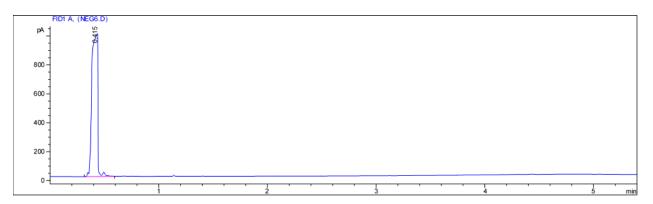
Super Sorbent negative control has peaks within the end of the chromatograph.

This is where heavy petroleum distillates are found. Therefore, it cannot be used because

it would once again be hard to distinguish between the oil dry and any petroleum product.

Therefore, Super Sorbent will not be used for further testing.

Figure 9: Universal Organic Absorbent Negative Control Run on GC-FID



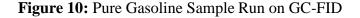
The Universal Organic Absorbent shows no interference. This oil dry does not separate therefore leaving no interference peaks within the chromatograph.

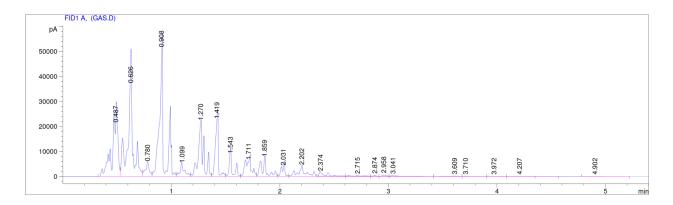
Since Chem Oil Away, Diversified Absorbent, Safe T Oil, and Super Sorbent all produced notable peaks above the background range, they should not be used for this research. The oil dries produce interference which could be mistaken for a possible ignitable liquid.

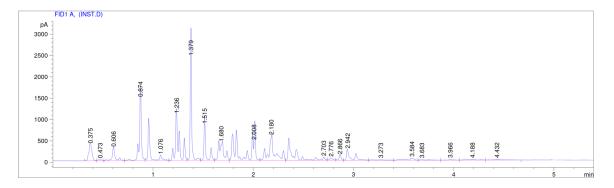
Since both the Instazorb and Universal Organic oil dries produced no interfering peaks, these samples can be used for the research project. This can be confirmed by comparing these two chromatographs back to the blank run. To address a possible contamination issue, control blank runs will be performed after each sample. Also each sample will also be run in duplicate to ensure reproducibility.

Instazorb and Universal Organic Absorbent were used in step 2 of the analytical scheme. In this portion, know standards were added to the oil dry samples and then tested. This would determine whether or not petroleum products could be isolated and recovered from the oil dry. Kerosene, diesel, and gasoline were used.

The same initial procedure was followed; however, 0.5 mL of a petroleum product was added into the mason jar with the oil dry. The same instrumental method was used to analyze the samples. A gasoline standard is shown in **Figure 10**. **Figure 11** and **12** show the results for the Instazorb and Universal Organic with gasoline.

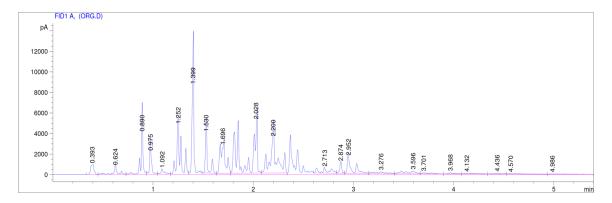






In the Instazorb spiked with gasoline chromatograph, there are peaks missing from the beginning on the chromatograph when compared to the pure gasoline standard chromatograph. This is due to the weathering of gasoline. Gasoline is weathered "as a result of evaporation or partial burning, which results in the loss of low-boiling compounds."<sup>7</sup> This is why some of the peaks at the beginning of the Instazorb and Universal Organic oil dries are missing.

Figure 12: Universal Organic Spiked with Gasoline Run on GC-FID



Second and third trial runs were done of gasoline with both oil dries and the results were the same. Gasoline was able to be identified by GC-FID in both the Instazorb and Universal Organic oil dries.

The first trials of kerosene and diesel were done next. 0.5 mL of each petroleum product was added into the mason jars with corresponding solid absorbent. All samples were run twice to confirm results. These are shown in **Figures 13, 14,** and **15**.



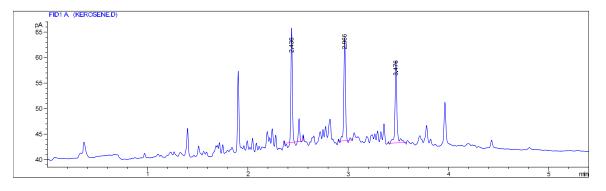


Figure 14: Instazorb spiked with Kerosene Run on GC-FID

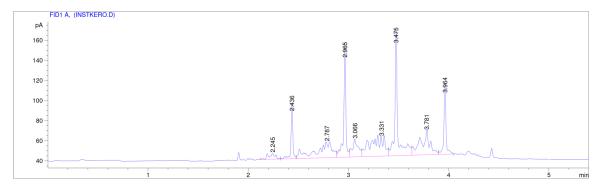
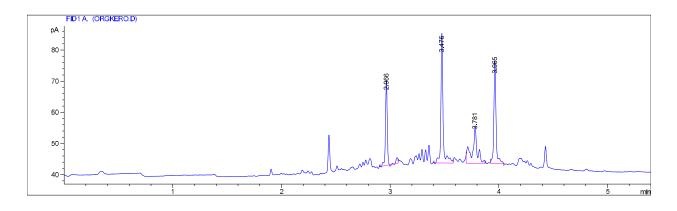
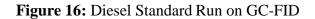


Figure 15: Universal Organic spiked with Kerosene Run on GC-FID



Kerosene shows 8 distinct, evenly spaced peaks within the middle to end of the spectrum. Both Instazorb and Universal Organic solid absorbents showed these evenly spaced peaks. These results further validate the proper absorption of the ignitable liquids.



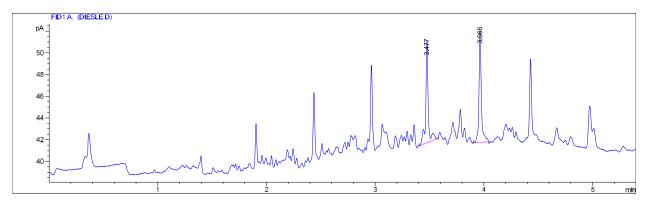


Figure 17: Instazorb spiked with Diesel Run on GC-FID

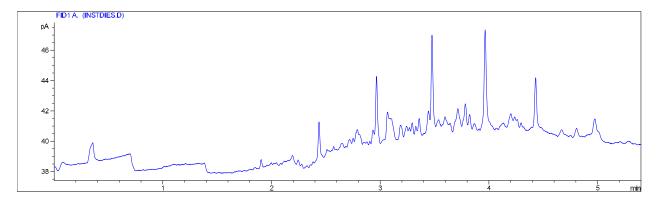
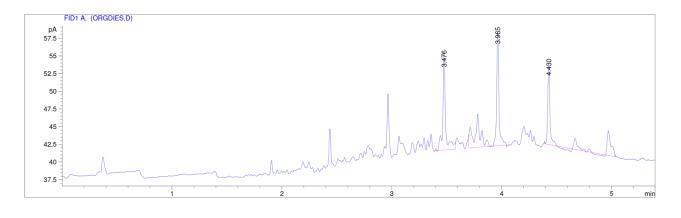


Figure 18: Organic spiked with Diesel Run on GC-FID



Diesel produces about 12 evenly spaced within the middle to the end of the spectrum very common for heavy petroleum distillates. The Instazorb and Universal Organic oil dries follow this same pattern.

#### **Discussion**

It should be noted that the chromatograms for heavy petroleum products; kerosene and diesel are somewhat different than what is expected. While the runs are similar to the standards run, additional separation and resulting peaks were not seen. To address this concern, the research project was suspended while instrumental parameters were changed. This would allow the most accurate results to be determined and reported.

With the research project temporarily suspended, the instrumentation method was adjusted. To obtain full separation of heavy hydrocarbon samples as expected, the final temperature of the oven was increased. With the increase in temperature from 160°C to 225°C, the time of a run also increased. The final temperature was held at 225°C for 2 minutes. This change allowed for all of the sample to separate as expected. The results of the method change can be seen in the diesel chromatograph below in **Figure 19**.

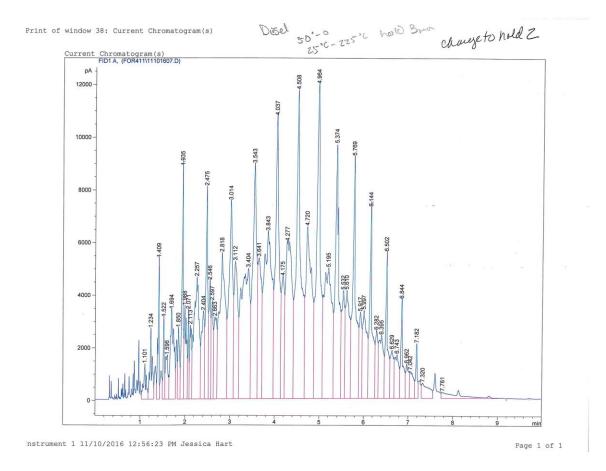


Figure 19: Method Changed Diesel Standard Run on GC-FID

As shown above, increasing the temperature was effective. It allowed for the diesel to be completely eluted from the column to produce the additional evenly spaced peaks needed for identification.

#### **Conclusion**

Once the instrument method was changed to be able to elute the full sample off of the column, the research project can continue. Initial results of the Instazorb and Universal Organic oil dries will be rerun to make sure they produce the correct peaks for gasoline, kerosene, and diesel. Once the initial findings are confirmed the research will continue. Further steps for this research project include step 3. This step includes placing the petroleum product on a porous surface, tile, and then adding the oil dry. After a period of time, the oil dry will be collected. Passive headspace isolation and analysis by GC-FID will be performed. This will determine whether or not the oil dry can actually absorb the petroleum product from a substrate for identification.

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