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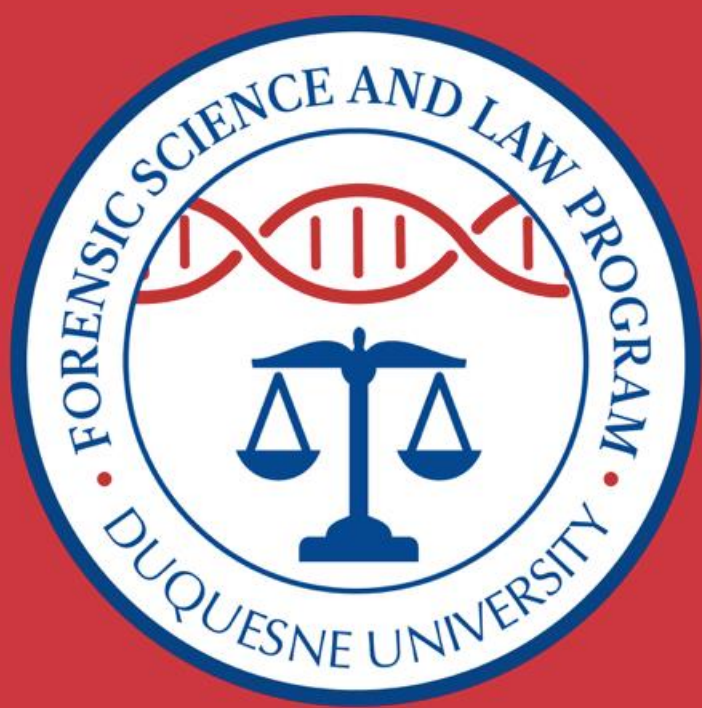


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# The Effects of Storage Conditions and Time on Extracted Ignitable Liquids Using GC-MS

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## Abstract

Arson is defined as the malicious intent to burn another person or their property.<sup>1</sup> Most often, arson is used to cover up another crime such as a murder because most physical evidence is destroyed in the fire, allowing the perpetrator to get away with the crime. Due to the lack of substantial evidence in many cases, it can be difficult to determine whether the fire was accidental or intentional. A good indication of malicious intent is the presence of an ignitable liquid.<sup>1</sup>

Three petroleum distillates (Crown® White Gas Camp Fuel, Crown® Paint Thinner, and Crown® 1-K Kerosene) classified into different sub-categories (light, medium, and heavy) were selected as samples. Samples were extracted onto activated charcoal strips using a passive headspace method. The charcoal strips were then transferred to a Sirchie® -Nylon Fire Debris Bag or PTFE-lined glass vial. Once contained, samples were stored under room temperature conditions or refrigerated at 4°C. After storing for intervals of 3 months, 6 months, and 9 months, extracted samples were subsequently analyzed using gas chromatography mass spectrometry (GC-MS) to visualize peak profiles. "Time 0" samples were also prepared in order to provide a baseline for comparison purposes. Analysis of sample data was performed through identification of target compounds according to the American Society for Testing and Materials protocol<sup>2</sup> by utilizing the National Institute of Standards and Technology Mass Spectral database. Comparisons were made between all variables noting any loss of compounds. This research may indicate a need for modification to standard protocol for the storage of extracted ignitable liquids in the future, ensuring accurate results.

## Introduction/Background

- Organization of Science Assessment Committees (OSAC) Fire Debris and Explosives<sup>3</sup>
- 2017-Indication for evaluation of storage conditions of archived fire debris extracts
  - Rank II-Major gap in the field, some current research being conducted

Variables tested in this research

- Storage vessel – Sirchie® -Nylon Fire Debris Bag and PTFE-lined glass vial
- Storage temperature – Room Temperature and Refrigerated (4°C)
- Length of storage – 3 months, 6 months, 9 months
- Ignitable liquid (petroleum distillates) – Crown® White Gas Camp Fuel, Crown® Paint Thinner, and Crown® 1-K Kerosene

Due to their availability and low cost, petroleum distillates were chosen for this study. It was hypothesized that storage in nylon fire debris bags in refrigerated conditions would best maintain analytical accuracy over time. This is because ignitable liquids volatilize more readily in warmer temperatures and nylon fire debris bags are specially manufactured for volatile evidence. It is also hypothesized that the number of compounds found within each sample will decrease over time. Ultimately, the aim of this research was to aid in optimizing a standard archiving procedure. Optimization of archival procedure is crucial in maintaining consistent analytical results, which is of importance if archived samples should need retested days, months, or years later.

## Experimental Method

### Extraction (Prepared in quintuplet)

1. Known volume of ignitable liquid applied to 10cm x 10cm 100% cotton flat sheet, in mason jar
2. 1 hour evaporation period
3. Copper ribbon cut and weighed
4. Activated charcoal (~5mg) applied to ribbon using sodium silicate, then reweighed
5. Copper ribbon hung over copper strip inserted into septum of lid
6. Lid placed on jar, heated for 1 hour at 100°C in oven
7. Removed and allowed to cool

### Storage

1. Charcoal-coated ribbon stored in vial or nylon bag, then placed in refrigerator or room temperature
2. Storage for 3 months (T3), 6 months (T6), or 9 months (T9)

### Analysis

1. Ribbon rinsed with 1mL carbon disulfide, then filtered into 1.5mL GC vial
2. Samples run on Agilent 6890N Network GC System with Agilent 5973 Network Mass Selective Detector and Agilent 7683 Series Injector, fitted with a non-polar, 5% phenylated Agilent J&W HP-5MS 30m x 0.250mm, 0.25µm column for subsequent analysis

\*\*Cotton flat sheet and solvent blank run as controls\*\*

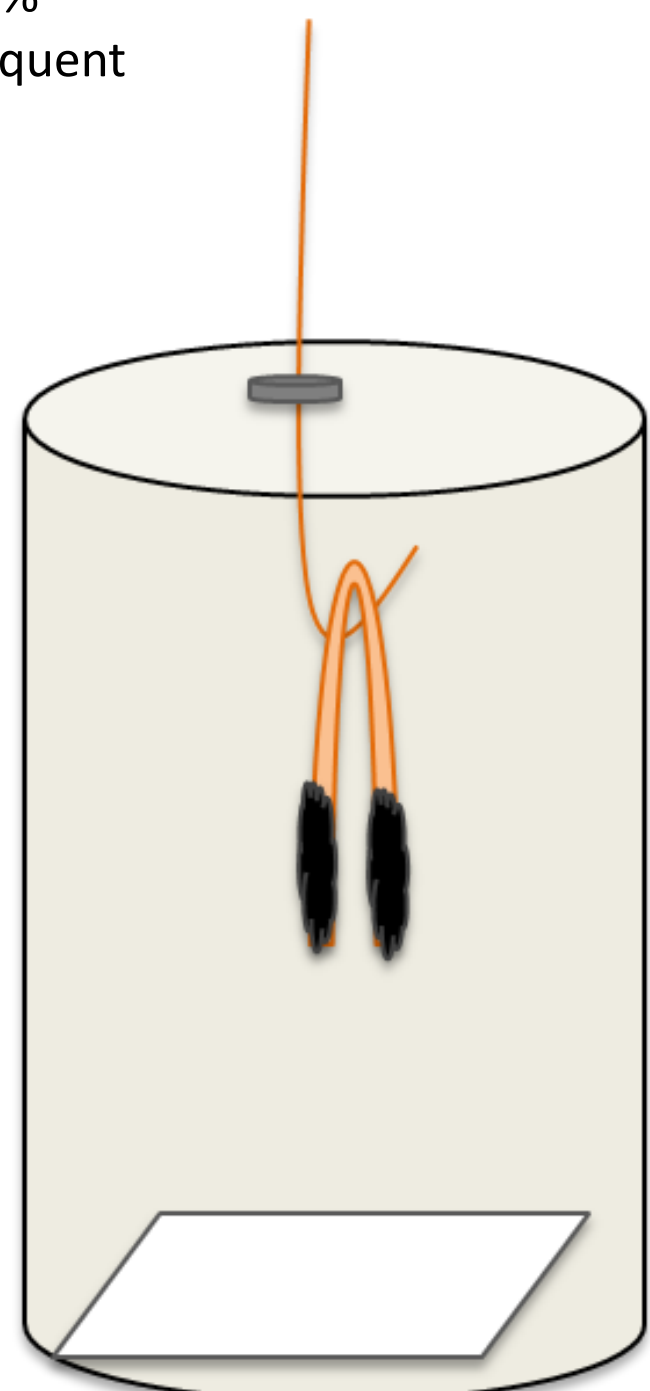


Figure 1: Representation of set up used during extraction

## Results

### Preliminary Trials

Preliminary testing was done to determine an appropriate volume of ignitable liquid to apply to the cotton swatch for storage trials, considering sample conservation. Several analysis methods were tested by alteration of GC/MS method parameters and solvent rinse choice. Volumes tested were 50 µL, 100 µL, and 150 µL. Two solvents were tested; Carbon disulfide and pentane. Two sets of method parameters were tested; one based on those created by a former graduate student at the university<sup>4</sup> and one based on those used at the Allegheny County Medical Examiner's Office laboratory.

### Preliminary Results

- Appropriate volumes: 50 µL for kerosene, 150 µL for paint thinner, and 150 µL for camp fuel
- Solvent: Carbon disulfide

Table 1: Final GC-MS method parameters

Gas Chromatography	
1 µL injection	Splitless
He carrier gas	0.8 mL/min flow rate
Initial Temp. 40°C	3 min hold
Ramp 10°C/min	
Final Temp. 300°C	5 min hold
Mass Spectrometry	
Acquisition Mode	Scan
Scanning mass range	50-350

### Storage Trials

Figure 1 illustrates a representative total ion chromatogram of the kerosene reference sample that was immediately run following extraction and did not undergo storage. This was used as a reference for comparison of stored samples. Table 2 shows the compounds that were found in the reference sample.

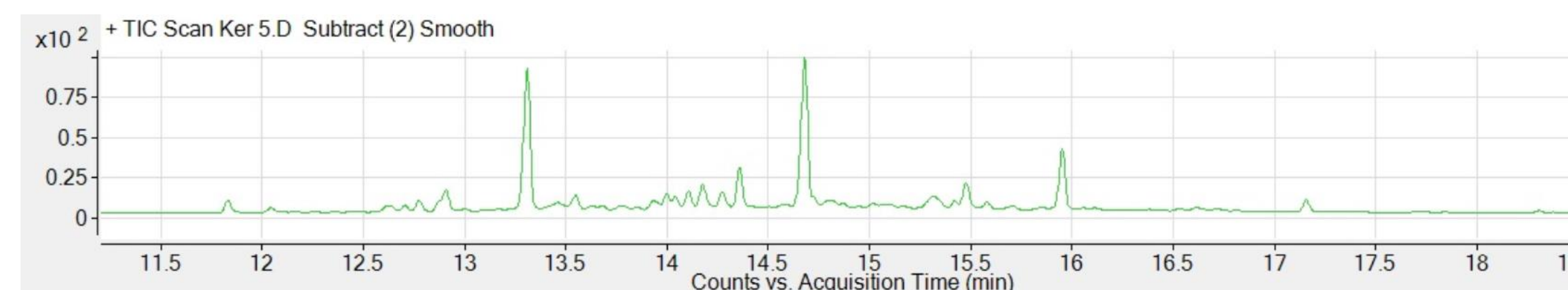


Figure 2: Kerosene reference profile (time 0)

Figures 3 through 6 illustrate representative spectra of each storage vessel/storage temperature combination at month 9. As seen, the peak profile remains constant and is the same as the reference which was not stored.

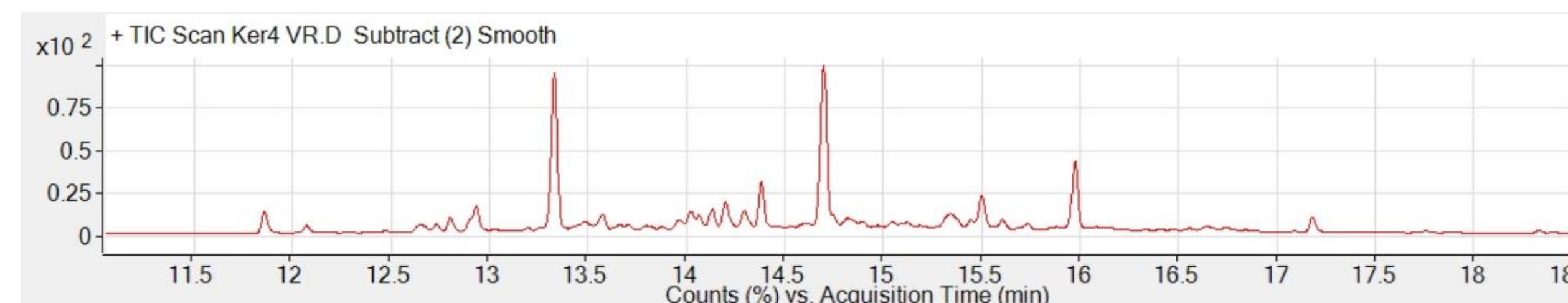


Figure 3: Kerosene in vial, room temp. (VR) at 9 months

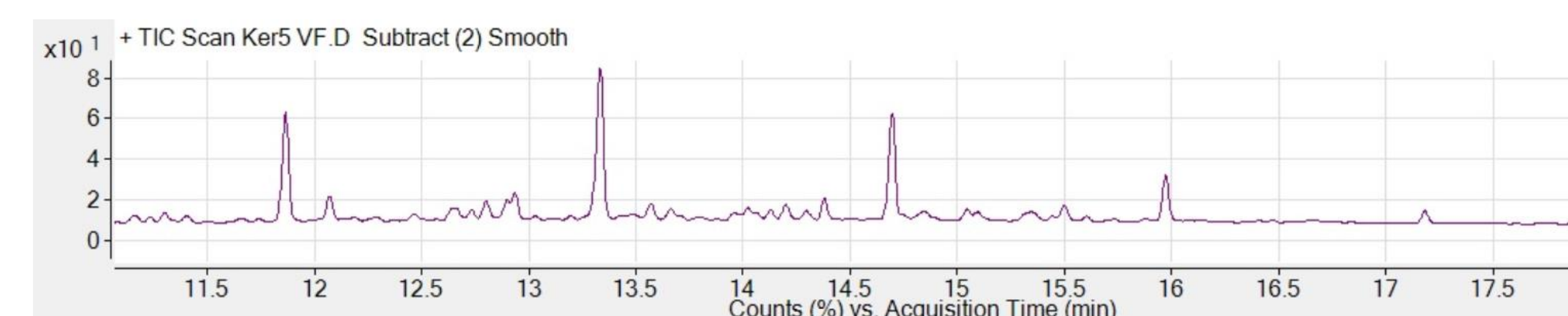


Figure 4: Kerosene in vial, refrigerated (VF) at 9 months

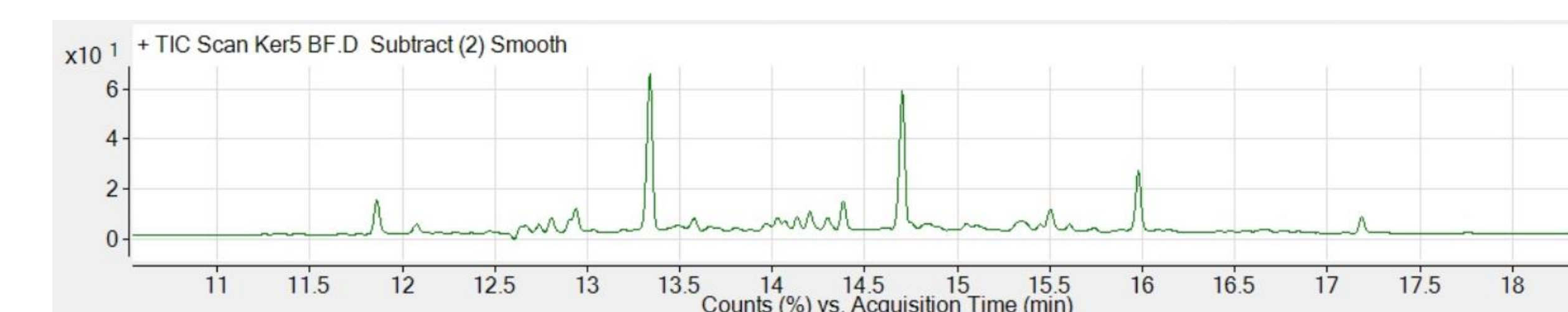


Figure 5: Kerosene in fire debris bag, refrigerated (BF) at 9 months

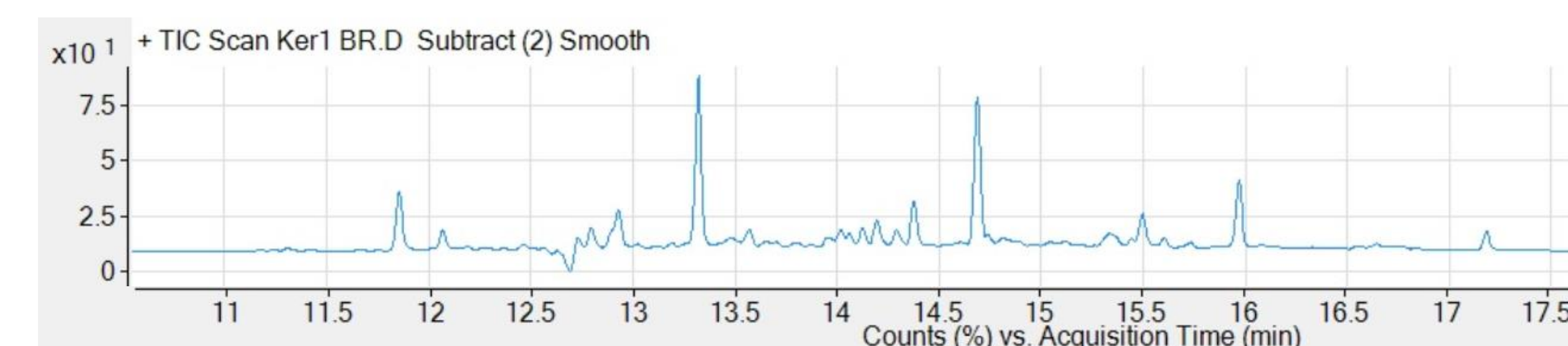


Figure 6: Kerosene in fire debris bag, room temp. (BR) at 9 months

## Discussion

Out of all the compounds found within the reference Kerosene sample, six were included on the ASTM target compound list for heavy petroleum distillates. Each component within the reference can be found in Table 2 which includes ASTM target compounds in blue. Factoring in each of the storage vessel/storage temperature combinations, there was no significant difference observed over time. Out of the sixteen compounds focused on, the minimum number seen in any of the conditions was twelve. At 3 months the refrigerated nylon bag retained the most compounds. At 6 months the room temperature vial retained the most compounds. At 9 months the room temperature vial and refrigerated bag tied for the most retained compounds. Based on this data, it cannot be said that one storage vessel or one storage temperature better retains compounds than the other. In focusing on ASTM compound loss alone the maximum number of loss was one. Again, no significant trends were found.

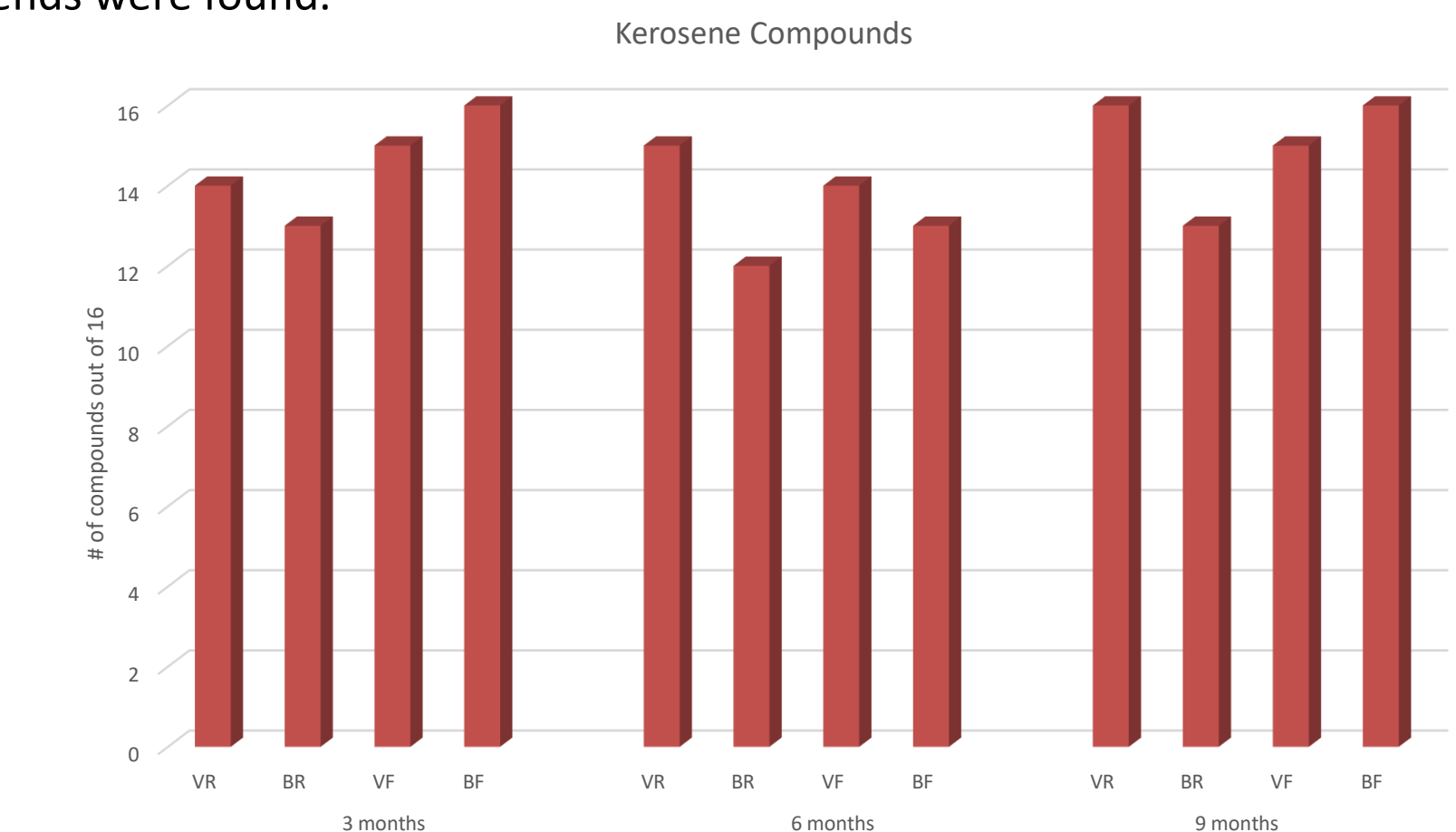


Figure 7: Avg. number of compounds found compared to kerosene reference

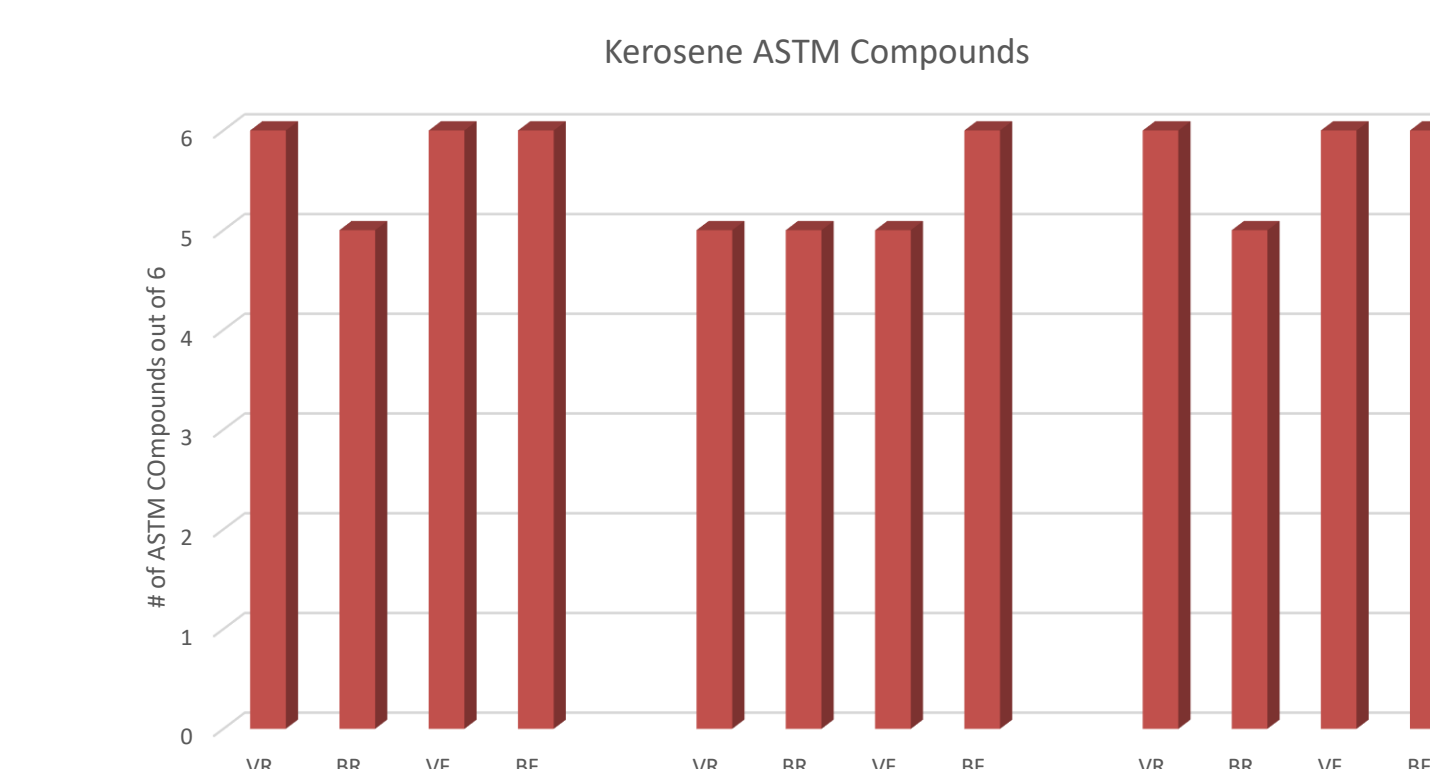


Figure 8: Avg. number of ASTM target compounds found in kerosene over time

Table 2: Kerosene Reference Compounds

Avg. Ret. Time	Compound
11.833	Dodecane
12.047	Undecane,2,6-dimethyl-
12.776	Dodecane,2-methyl-
12.907	Octane,2,3,7-trimethyl-
13.301	Tridecane
14.105	Tridecane, 4-methyl-
14.175	Tridecane, 2-methyl-
14.27	Tridecane, 3-methyl-
14.356	Dodecane, 2,6,10-trimethyl-
14.674	Tetradecane
15.315	n-tetracosanol-1
15.475	Heptadecane,2,6,10,14-tetramethyl-
15.579	Tetradecane,2,6,10-trimethyl-
15.95	Pentadecane
17.155	Hexadecane
18.307	Heptadecane

## Conclusion

In conclusion, the data collected from kerosene suggests that extracted ignitable liquid compound profiles obtained during analysis do not strongly differ whether stored at room temperature or refrigerated, in a nylon fire debris bag or PTFE-lined vial. Additionally, comparison of compound profiles over the duration of 3 months, 6 months, and 9 months also proved to have little to no effect on the compounds recovered during analysis. This trend may not hold true for paint thinner and camp fuel samples because they are much more volatile. It should also be considered that most laboratories use commercially manufactured charcoal strips rather than created their own means of charcoal adsorption strips. There may be differences observed if this study is repeated with manufactured strips. However, this research was intended to be a baseline study and a starting point for further research in this area. At its current stage, this research does not prove the need for standard procedure modification for storage of extracted ignitable liquids.

\*\*Please note that the results represented on this poster are not exhaustive. Camp fuel and paint thinner samples are currently being analyzed.\*\*

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- Bayer School of Natural and Environmental Sciences
- Duquesne University Department of Chemistry and Biochemistry

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- 1) Fisher, B. A. Chapter 11 -Arson and Explosives. *Techniques of Crime Scene Investigation*. 7th Edition. CRC Press. **2004**. 308-331.
- 2) ASTM Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry. **2014**, E1618-14, 1-15.
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