

**Investigation into the Organic Molecules Present in  
Leachate of Soils Affected by Wildfire and Fire  
Retardant Using  $^{13}\text{C}$  NMR Spectroscopy**



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T00247404  
CHEM 4480 Project  
April 30, 2024  
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# Investigation into the Organic Molecules Present in Leachate of Soils Affected by Wildfire and Fire Retardant Using $^{13}\text{C}$ NMR Spectroscopy

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Wildfire, leachate, Organic molecules, fire retardant, Soils,  $^{13}\text{C}$  NMR Spectroscopy

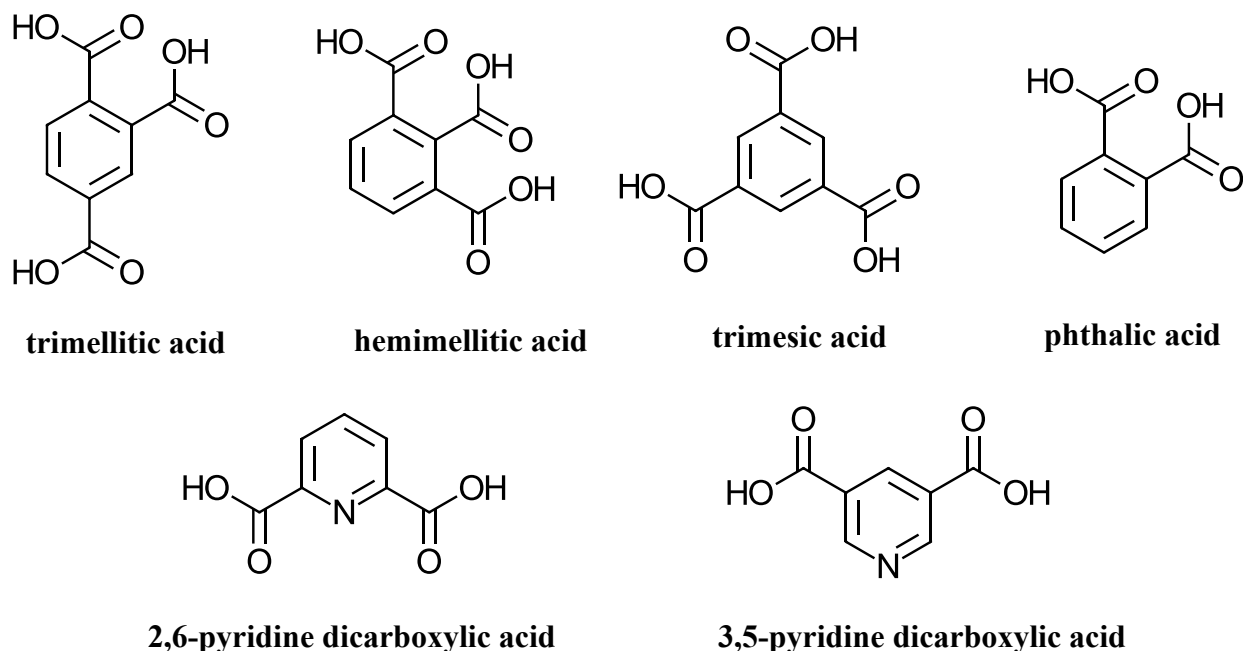
## ABSTRACT

To simulate the effects of groundwater infiltration on soils affected by wildfire and fire retardant, an *in situ* column leaching experiment was performed. Soil samples were collected from the 2023 Bush Creek East wildfire near Adams Lake within the interior of British Columbia, leached with three different solvents, and filtered. Collected leachate samples were analyzed using  $^{13}\text{C}$  nuclear magnetic resonance (NMR) to determine if there were any alterations to the organic molecules present in the soils. This study concludes that there were chemical differences in the  $^{13}\text{C}$  NMR spectra of fire-affected soil leachate between samples treated with fire retardant and those untreated. The presence of aromatic carbon signals indicated the possible presence of benzene dicarboxylic acids, tricarboxylic acids, and tetracarboxylic acid monomers, referred to as benzene polycarboxylic acids (BPCAs).

## INTRODUCTION

Wildfire activity within British Columbia and North America has significantly increased within recent years. The 2023 fire season in British Columbia officially surpassed the 2018 season as the most destructive ever recorded in terms of area burned.<sup>1</sup> Wildfires can drastically modify the vegetation, soils, and watersheds which regulate significant sources of water supplies for municipal and other uses.<sup>2</sup>

The intensity at which wildfires burn also has a significant impact on the organic molecules present post-fire.<sup>4-6</sup> Soils heated at 225-350 °C included higher proportions of condensed aromatic structures, such as black carbon and black nitrogen in their leachates.<sup>2-5</sup> There is also an increase in the formation of toxic nitrogen containing disinfection byproducts (DBPs) observed in drinking water treatment plants downstream from wildfire affected areas.<sup>2,3,6</sup> Isomers of pyridine dicarboxylic and tricarboxylic acids (PCAs) have also been identified in burned soils.<sup>5</sup> Using purchased standards, the work by Thurman *et al.*<sup>5</sup> identified three isomers of BCPAs by LC/Q-TOF-MS including trimellitic acid, hemimellitic acid, trimesic acid, and phthalic acid. Furthermore, they identified groups of PCAs including 2,6-pyridine dicarboxylic acid and 3,5-pyridine dicarboxylic acid.<sup>5</sup>



**Figure 1.** Organic molecules found in fire-affected soils.<sup>5</sup>

Fire retardants used to battle forest fires in British Columbia primarily consist of diammonium sulphate and/or ammonium sulphates, alcohol, and ether.<sup>7</sup> They are used commonly in fire suppression because of their long-lasting effects on wildfires and are applied either directly on burning fuels to extinguish a fire, or in advance of a fire to create a fire control line.<sup>7,8</sup> When the retardant contacts the heat of the fire, the reaction releases a combination of water and carbon dioxide which cools and suffocates the fire. Because the main components in fire retardants are also present agricultural fertilizers, they have been commonly considered safe to use without any long-lasting effects.<sup>8</sup> Their impacts on the environment and aquatic systems have just recently been an area of concern. There have been some studies on the impacts of fire retardant on soils,<sup>8</sup> but there remains little insight into their impact on fire-affected soils.

This study focuses on whether <sup>13</sup>C NMR spectroscopy can detect any chemical differences in organic molecules present in fire-affected soils with added treatments of fire retardant from

laboratory leaching experiments. Three different leaching solvents (water, methanol, and ethyl acetate) were used based on their different polarities, densities, and hydrogen bonding characteristics. The collected leachate samples were analyzed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy to evaluate the differences present in the soil leachate of samples treated with fire retardant and those without. Soils were analyzed using  $^{13}\text{C}$  NMR because the spectra can easily detect changes in the aromatic and carboxyl regions. The  $^{13}\text{C}$  NMR is often supplemented with other analytical techniques, but the NMR spectra has the advantage of taking a quick snapshot of the soil characteristics compared to the complicated spectra of GC-MS. Our specific objectives were to (i) develop a sampling plan and experimental procedure to detect organic molecules present in an *in situ* laboratory leaching experiment of fire-affected soils; (ii) add treatments of fire retardant to investigate changes to any organic molecules present in the leachate; (iii) determine if liquid state  $^{13}\text{C}$  NMR is an effective technique to detect changes within the organic molecules present in the leachate.

## MATERIALS AND METHODS

### **Sample Collection**

Fire-affected soil samples were collected from the 2023 Bush Creek East wildfire formerly known as the Adams Lake wildfire within the interior of British Columbia. The sample was taken on November 2, 2023, at 1:45 pm off the Scotch Creek Forest service road (50°56'30'' N 119°27'45'' W) at 420 m of elevation above sea level (Figure 2).



**Figure 1.** Sampling location (red) including area burned by the Bush Creek East Wildfire (yellow).

Conditions were cloudy with slight fog and breeze at 2 °C. Some remaining vegetation was identified as Lodgepole Pine, Douglas-Fir, Black Cottonwood, Trembling Aspen, and Western Red Cedar. A tape measure was used to measure an area of 0.3 m x 0.3 m which was marked for soil extraction (Figure 3). Overlying debris and vegetation were then removed from the sample area by sweeping by hand. A clean stainless steel garden shovel was used to collect soil within the sample area into a 20 L food grade plastic bucket. The area was excavated to 5 cm deep while continually measuring the depth with a ruler (Figure 4). Once the area was excavated to specifications, the bucket was sealed with an air proof lid for subsequent transportation.



**Figure 2.** Measured sampling location.



**Figure 3.** Depth of sampling plot measured using a ruler.

The soil sample was weighed in the bucket (4.1 kg) to determine its moisture content then immediately placed onto aluminum pans to dry to prevent any bacterial growth. The sample was left for one week to dry in a ventilated area at an average temperature of 22.5 °C. After one week, the sample was stirred using gloves to ensure even drying and to break up any large clumps present. One week after the sample was stirred, it was weighed to be 2.9 kg resulting in a moisture content calculation of 27%. The material was screened using a clean 2mm sieve and stored for further analysis. Soil was characterized as Sandy Loam (71% sand, 14% silt, 15% clay) using the procedure and soil characterization triangle by Ted Sammis.<sup>9</sup>

## **Methods**

100.0 g of sieved soil was placed separately into three 500 mL chromatography columns that were prepared for the leaching experiment. The experiment blanks were treated first with different solvents in each column (deionized water, methanol, and ethyl acetate). The blanks were each treated with their respective solvents and allowed to sit for 24 hours. Pressure was then applied to the columns to flush the leachate into separate 500 mL round bottom flasks (RBF). The leachates were concentrated using a Heidolph Hei-VAP Core under reduced pressure and the slight application of heat. The masses of the extracts were noted, and the RBFs were sealed and stored below 0 °C until further analysis. The same procedure was followed for the experimental conditions, except 10.0 mL of fire retardant was added on top of the soil before the respective solvents were added (Table 1).



**Table 1.** Column conditions for the control and experimental conditions, solvents amounts were measured with beakers while retardants amounts were measured with volumetric pipettes.

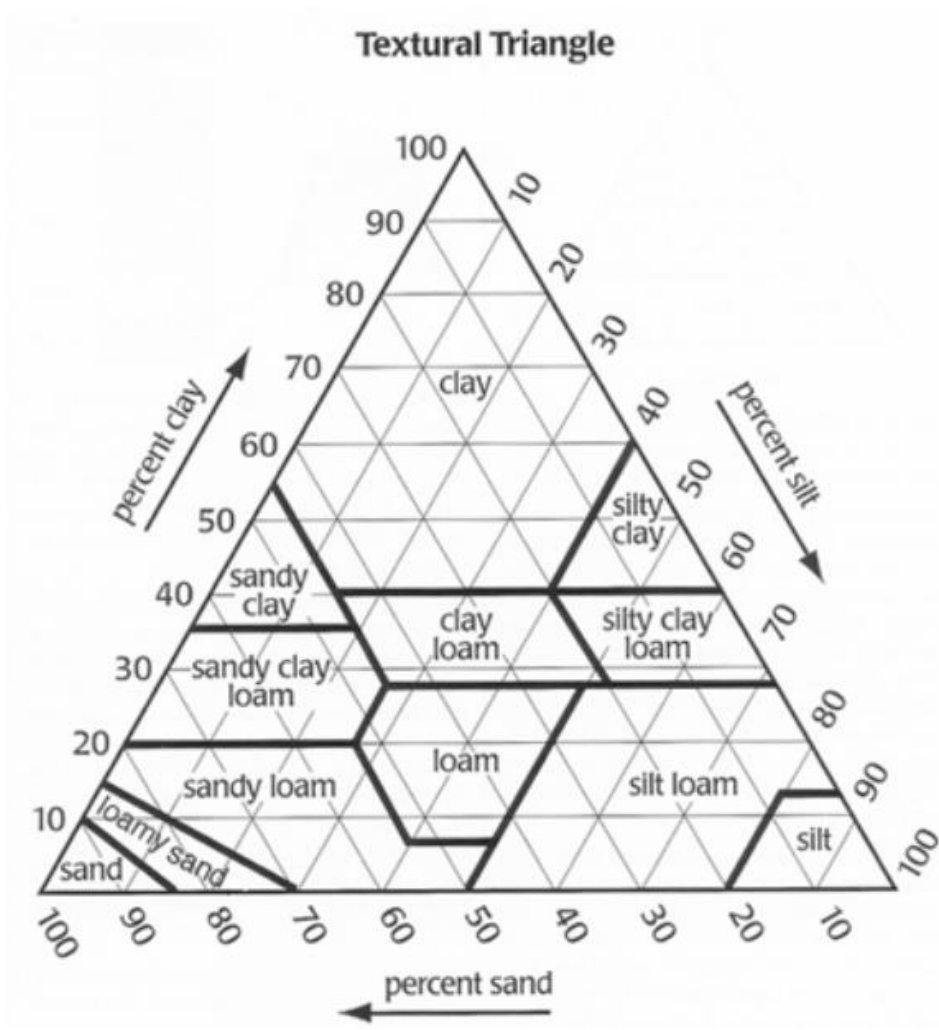
<b>Column Control Conditions</b>	<b>Column Experimental Conditions</b>	<b>Beaker Experimental Conditions</b>
100 mL water	100 mL water + 10.0 mL retardant	100 mL water + 10.0 mL retardant
100 mL methanol	100 mL methanol + 10.0 mL retardant	100 mL methanol + 10.0 mL retardant
100 mL ethyl acetate	100 mL ethyl acetate + 10.0 mL retardant	100 mL ethyl acetate + 10.0 mL retardant

An alternate trial procedure was developed after the column experimental conditions due to the difficulty of collecting the water leachate from the column. To begin, 100.0 g of sieved soil was placed into three separate 400 mL beakers. Then 10.0 mL of fire retardant was measured using a volumetric pipette and added on top of the soil followed by 100 mL of the respective solvent. The soil and liquid components were mixed and allowed to sit for 48 hours, after which time, the solvents were decanted into a Buchner funnel under suction to remove all particles. The water solvent filtrate was transferred to a 50 mL centrifuge tube and centrifuged at 6000 rpm for 10 minutes then filtered using a 0.45  $\mu\text{m}$  Luer lock filter to remove any smaller particles. All collected filtrates were concentrated using a Heidolph Hei-VAP Core under reduced pressure and slight application of heat. The masses of the extracts were noted, and the RBFs were sealed and stored below 0 °C until further analysis.

### **Classification of Soil (Texture analysis)**

The classification of the soil was followed according to the procedure by Ted Sammis, New Mexico State University.<sup>9</sup> A glass quart mason jar was obtained and filled half full of the sieved

soil. The soil was wetted to a mud consistency and tapped to settle the soil. The top of the soil was then marked on the jar. A teaspoon of dish soap was added, and the jar was filled to the top with deionized water. The jar was shaken until mixed and placed on the counter to let settle for 40 seconds. The level of the soil was then marked on the jar (sand portion of soil). After 48 hours, two more lines were marked on the jar (the silt layer lies above the sand layer then clay layer is the last to settle). Distances between the lines were measured and the percentage of each was calculated. The soil texture was then characterized using a soil triangle.



**Figure 4.** Texture triangle used to characterize the sampled soil.<sup>9</sup>

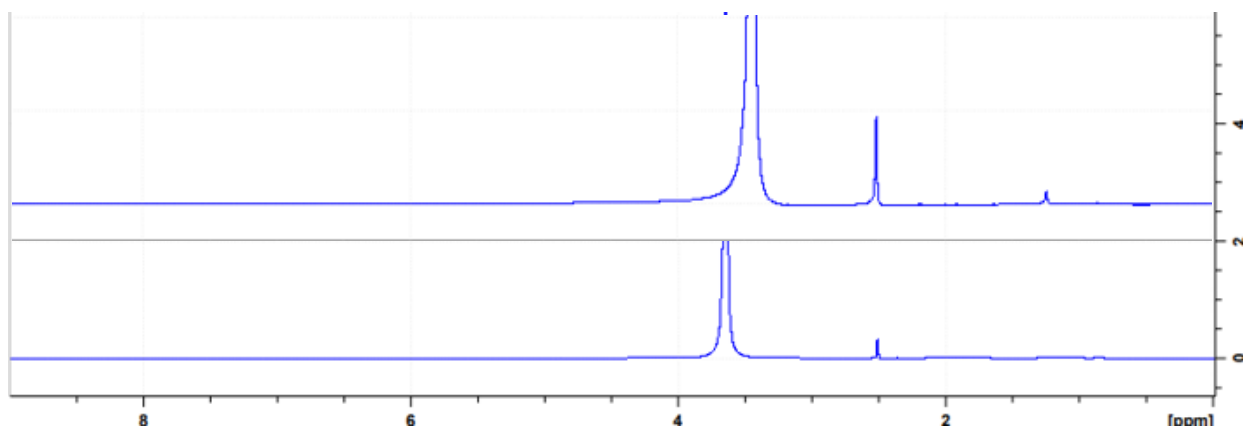
## **<sup>13</sup>C NMR Spectroscopy**

To determine if any of the organic molecules present in the soil were altered by the addition of fire retardant, samples were analyzed by liquid state <sup>13</sup>C NMR spectroscopy. The water experimental conditions in the beaker were the only beaker sample that was analyzed by NMR due to time constraints. The water experimental column conditions were not analyzed due to a minimal amount of leachate present. The <sup>13</sup>C NMR spectra were then split into four shift regions based on their chemical shift values ( $\delta$ ): alkyl-C (0-35 ppm), methoxy-C/O-alkyl-C (35-75 ppm), aromatic-C (100-150 ppm), and carboxyl-C (150-200 ppm). The methanol and ethyl acetate leachates were run using deuterated chloroform (CDCl<sub>3</sub>) as the NMR solvent, while the water leachates were run using dimethyl sulfoxide (DMSO-d<sub>6</sub>) as the NMR solvent. Each spectrum was acquired over approximately 22 hours and contained 25600 scans.

## **RESULTS**

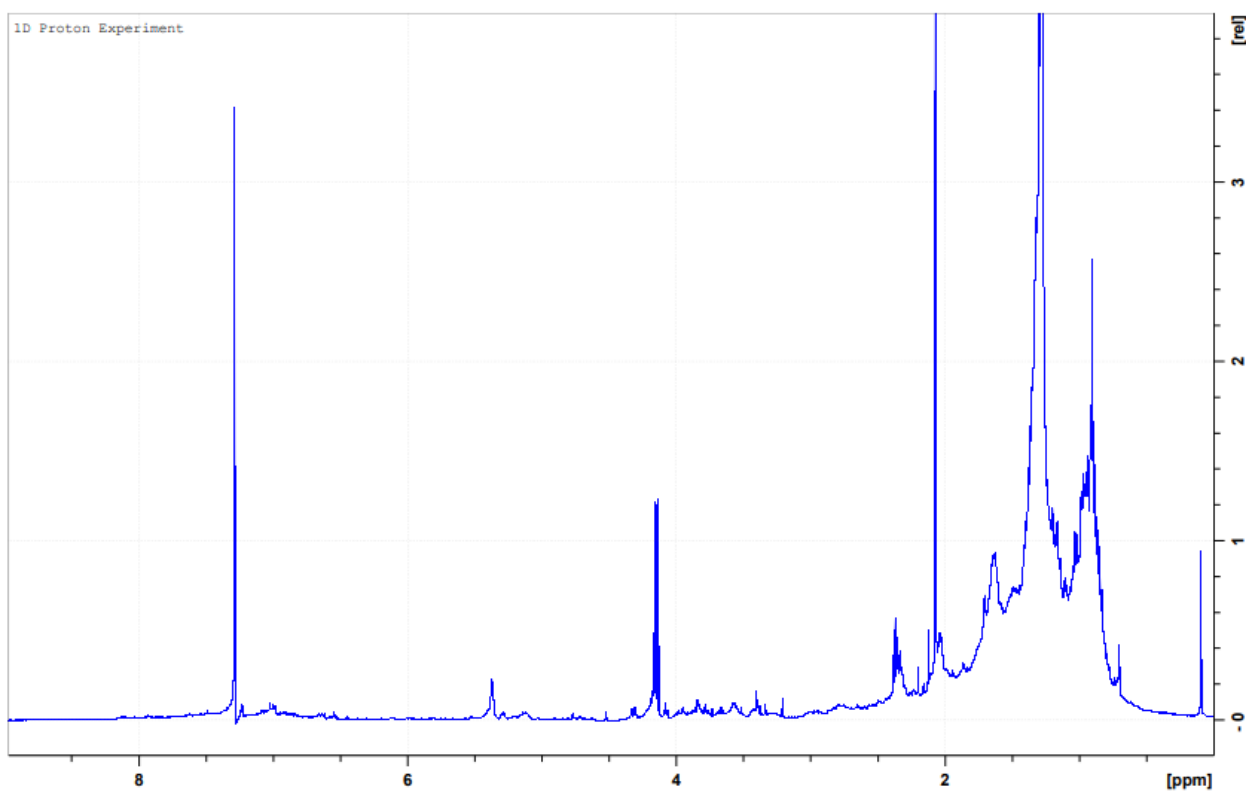
### **<sup>1</sup>H NMR Spectra**

There were no major differences noted in the spectra when using water as a solvent. One large peak at  $\delta$  3.5 ppm was noted in the spectra of the control and experimental conditions. A smaller peak was also observed in the experimental condition's spectra at  $\delta$  1.25 ppm which was not present in the control spectra.



**Figure 5.**  $^1\text{H}$  NMR spectra of water control conditions (bottom) and experimental conditions (top).

Multiple peaks were observed in the spectra when ethyl acetate was used as a solvent, although there were little differences between the control and experimental spectra. A large broad peak was present at  $\delta$  0.5-2 ppm. Multiple smaller peaks were present in the  $\delta$  4-6 ppm region. There were also multiple peaks present in the aromatic region between  $\delta$  6.5 and  $\delta$  7.5 ppm.



**Figure 6.**  $^1\text{H}$  NMR spectra of ethyl acetate experimental conditions.

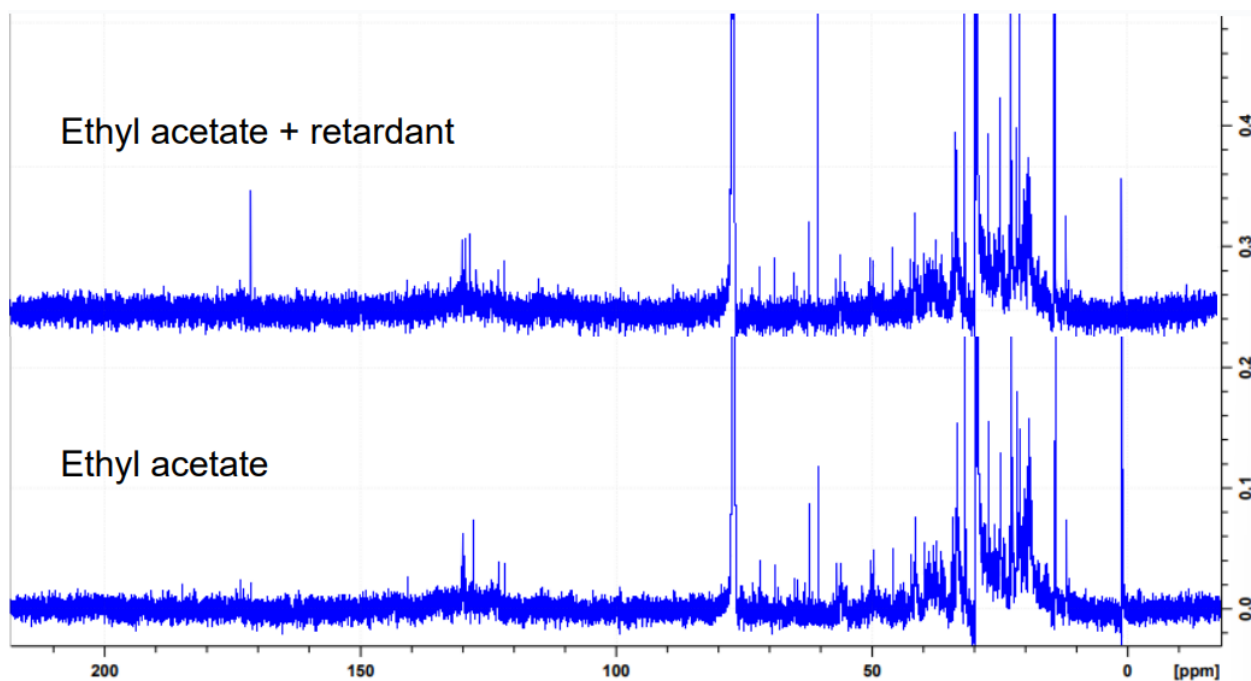
When methanol was used as a solvent, the spectra contained no discernible differences from the ethyl acetate control and experimental conditions. Although the  $^1\text{H}$  NMR spectra was able to detect the presence of aromatic hydrogens, it was not able to determine chemical differences between soils treated with fire retardant and those untreated. This result was predicted as  $^{13}\text{C}$  NMR was the primary technique being investigated for analysis and the  $^1\text{H}$  NMR spectra were going to be used as supplementary information.

### $^{13}\text{C}$ NMR Spectra

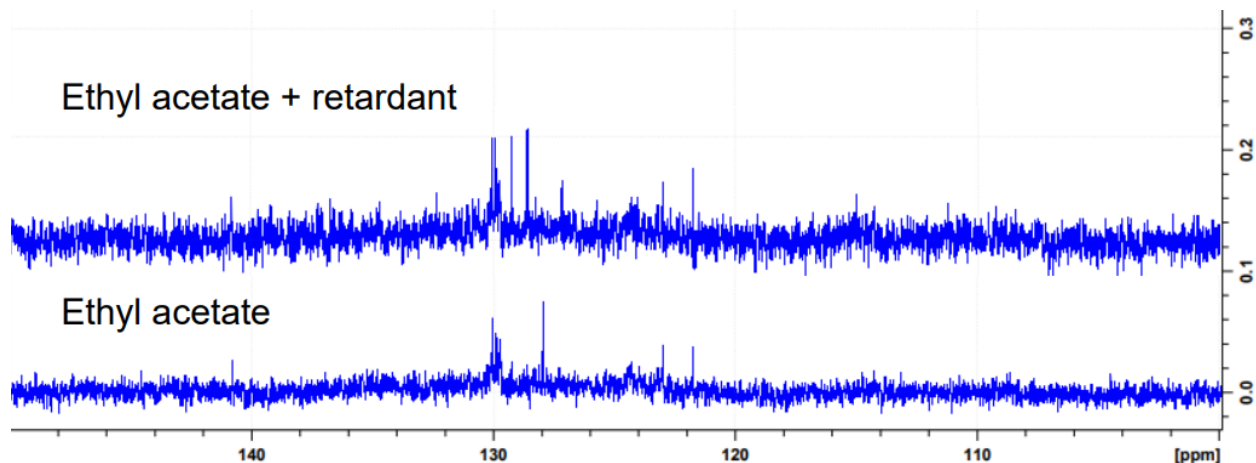
The  $^{13}\text{C}$  NMR spectra were able to show some differences in the chemical environments of signals between the control and experimental conditions. Most of the carbon nuclei peak

differences between control and experimental conditions occurred in the aromatic-C and carboxyl-C regions.

Between the ethyl acetate control and experimental conditions, there were differences in the aromatic-C region (100-150 ppm) and the carboxyl region (150-200 ppm). The alkyl-C (0-35 ppm) and methoxy-C/O-alkyl-C (35-75 ppm) regions contained no discernable chemical differences. The experimental spectra contained new smaller aromatic peaks at 155, 127, 132 ppm and a larger peak present at 129 ppm (figure 7). The appearance of another large peak was noted in the carboxyl-C region at 170 ppm (figure 6). Both the control and experimental conditions confirmed the presence of aromatic carbons within the soil leachate.



**Figure 7.**  $^{13}\text{C}$  NMR spectra of ethyl acetate leachate (bottom) and ethyl acetate + retardant leachate (top).



**Figure 8.**  $^{13}\text{C}$  NMR spectra of ethyl acetate leachate (bottom) and ethyl acetate + retardant leachate (top) focused on the aromatic region (100-150 ppm).

Between the methanol control and experimental conditions, there were differences in the spectra which can be potentially attributed to the low concentration of molecules for the methanol control conditions. Minimal peaks were detected in this spectrum which could be due to an abnormal scan from the NMR, or minimal molecules present in the leachate. The experimental conditions detected signals within the aromatic-C region.

Between the water control and experimental conditions, the spectra were very similar. There were a variety of peaks present in the alkyl-C region and two peaks present in the methoxy-C/O-alkyl-C region. All peaks appeared to be present in the control and experimental conditions, indicating no change in the chemical composition due to the different experimental conditions. The control and experimental spectra did not contain any peaks in the aromatic-C region indicating no aromatic structures present in this leachate.

## DISCUSSION

The  $^1\text{H}$  NMR spectra was not conclusive in determining chemical differences in the soil leachate when treatments of fire retardant were added. Most spectra appeared very similar and were independent of any treatment conducted. All literature topics using NMR spectroscopy as an analytical technique to detect molecules in soils used only  $^{13}\text{C}$  NMR and  $^1\text{H}$  NMR was never described in these papers.<sup>3-6,10</sup> This could be due to the large presence of hydrogen in the soil which will produce broad peaks which do not contain much useful information. Due to a large number of molecules present, the matrix produces complex splitting patterns which makes it difficult to distinguish small changes in the chemical environments which may be present. The  $^1\text{H}$  NMR spectra were able to detect aromatic hydrogens which was one source of useful information. The useful information present in the  $^1\text{H}$  NMR spectra could also be found in the  $^{13}\text{C}$  NMR spectra eliminating the need for supplementary  $^1\text{H}$  NMR analysis. Since the  $^1\text{H}$  NMR spectra was not informative, in future experiments, the  $^1\text{H}$  NMR could be exchanged for a more useful spin active nucleus such as  $^{15}\text{N}$  NMR or  $^{31}\text{P}$  NMR spectroscopy.

As discussed in the results, the ethyl acetate solvent displayed the clearest results in the  $^{13}\text{C}$  NMR spectra. The methanol trials displayed difficulties when the fire retardant was added, this could be due to a small number of molecules present in the sample. The water solvent conditions displayed some peaks but there was no change in the spectra between control and experimental conditions. This could be due to a variety of factors including difficulties extracting the water from the column and issues filtering the leachate due to very fine particulate present. There was also an issue with the solubility of the NMR solvents when attempting to transfer the dried leachate into solution. Deuterated chloroform was first used but encountered issues of solubility and no peaks were present in the scan. The NMR solvent was switched to dimethyl sulfoxide which improved the



solubility of the dried leachate but could still be an area of improvement by investigating the use of other NMR solvents.

Since this work used only liquid state NMR, the differences between solid state NMR and liquid state NMR will be discussed. In solution NMR, the spectra usually contain very sharp transitions due to averaging of anisotropic NMR interactions by rapid random tumbling. In solid state NMR, the spectra are very broad as the full effect of anisotropic interactions are observed. Since the sample is solid, these effects are much slower compared to liquid state NMR. Multiple methods have been developed to minimize large anisotropic NMR interactions between nuclei including Magic-angle spinning (MAS) and cross polarization.<sup>11</sup> Benefits of the solid state NMR technique include analyzing soils samples without the need for leachate experiments.<sup>4</sup>

The observation of different functional groups in the spectra can also reflect the temperature at which the wildfire burned. The work by Thurman *et al.*<sup>5</sup> accurately describes these changes using solid state <sup>13</sup>C NMR spectra. From temperatures of 150 to 250 °C, there was a significant loss of C-alkyl and almost complete loss of O-alkyl signals. The 250 °C trial was predominated by aromatic-C and carboxyl-C. From 350 to 450 °C, the spectra looked very similar with only aromatic-C and carboxyl-C signals present. At 500 °C, there was no observed organic carbon leaching from the soil, indicating most molecules are completely combusted to carbon ash at this temperature and beyond. In comparing these results to those from the ethyl acetate leaching conditions, we can confirm the wildfire temperature did not exceed 500 °C and was likely in the temperature range of 250 to 450 °C. These temperature estimates are consistent with the reported fast-moving nature of the wildfire. The fire grew about 20 km in 12 hours, which is among the fastest growth British Columbia has seen for a wildfire.<sup>12</sup>

## FUTURE WORK

There are many opportunities for future work on this subject since there is very little literature on the idea. Exploring other analytical instruments to detect organic molecules present in leachate of fire-affected soils could be beneficial. This project only determined changes in the chemical environments of carbon atoms present in compounds within the soils but did not characterize the identities of these molecules. The molecules present in the soils could be characterized using standards and further  $^{13}\text{C}$  NMR experiments. Using GC/MS or LC/MS could work well to determine molecules present. Many of the literature articles used a type of mass spectrometry (MS) to supplement the  $^{13}\text{C}$  NMR spectra. The types of instruments used included pyrolysis gas chromatography/mass spectrometry (Py-GC/MS),<sup>4,13</sup> liquid chromatography quadrupole-time-of-flight mass spectrometry (LC/Q-TOF-MS),<sup>5</sup> and Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR/MS).<sup>3,6</sup> One paper also supplemented direct-excitation  $^{31}\text{P}$  magic-angle-spinning NMR which could be useful in determining chemical differences since most fire retardants contain phosphates.<sup>10</sup> The settings of the NMR instrument could also be optimized for the low concentration leachate conditions. Solid state  $^{13}\text{C}$  NMR was used most used when analyzing the fire-affected soils.<sup>4-6,10,14,15</sup> The paper by Thurman *et al.*<sup>5</sup> describes their optimized solid state  $^{13}\text{C}$  NMR conditions including parameters such as spectra window, acquisition time, contact time, pulse delay, spinning rate, and line broadening. Fourier Transform Infrared spectroscopy is also a technique which can be supplemented to other instruments to indicate changes in functional groups.<sup>4</sup>

The laboratory procedure should also be adjusted to work with water as a solvent. The water conditions proved to be very difficult to collect leachate samples. Better design of column conditions could aid in eliminating this issue. The water conditions also contained very small

particulates which could not be removed by centrifuge or filtration under high vacuum. Future work should look at better procedures to eliminate these particulates. The laboratory procedure should also accommodate a sample of the fire retardant being run using  $^{13}\text{C}$  NMR to determine its composition and compare this spectrum to the spectra of experimental conditions. Further characterization studies could also be done on the fire retardant including density. Many literature articles also indicated extra clean-up steps involving hydrophobic (XAD-4) and hydrophilic (XAD-8) fractions to facilitate better separation and identification.<sup>5,6</sup> They described this step as concentrating the leachate sample and removing any unwanted interferences for instance, from humic acids.

It would also be interesting to perform the same procedure on soils unaffected by wildfire as fire retardant dropped from aircraft is most commonly deposited on these soils. Future sampling plans should also include larger sampling sizes and incorporate multiple different fire-affected areas as the soil compositions can greatly vary.

## CONCLUSION

In conclusion,  $^{13}\text{C}$  NMR can be an effective tool for detection of organic molecules present within leachate of fire-affected soils. There were chemical differences in the  $^{13}\text{C}$  NMR spectra of fire-affected soil leachate between samples treated with fire retardant and those without. The ethyl acetate solvent proved to be the best for extracting organic molecules from the fire-affected soils. The presence of aromatic carbons was detected in the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra within the soil leachate confirming the observations of literature research and the possible presence of BPCAs.

## ASSOCIATED CONTENT

**Supporting Information.** Includes sampling plan, experiment photos,  $^1\text{H}$  NMR spectra and  $^{13}\text{C}$  NMR spectra.

The following files are available free of charge.

Supplemental Information (PDF)

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### Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

### Notes

Authors declare no financial competing interests.

## ACKNOWLEDGMENT

I would like to thank Dr. Bruno Cinel for his guidance, knowledge, and commitment throughout this research project. I would also like to thank Dr. Sharon Brewer for her guidance and input as my co-supervisor. Additionally, the entire chemistry and NRS departments for their support in particular Dr. Jon Karakatsoulis, Dr. Thomas Pypker, and Dr. Darryl Carlyle-Moses

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