THE ANALYSIS OF FIRE DEBRIS USING NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

THESIS

Presented to the Graduate Council of the North Texas State University in Partial Fulfillment of the Requirements

For the Degree of

MASTER OF SCIENCE

By

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This paper describes a new technique for analyzing fire debris using nuclear magnetic resonance (NMR) spectroscopy. Petroleum distillates, which are commonly used accelerants, were weathered, burned, and steam-distilled. These, as well as virgin samples of the accelerants, were analyzed by gas chromatography and nuclear magnetic resonance spectroscopy. In addition, solvent studies and detectibility limit studies were conducted. The use of NMR is described as a valuable adjunct to the existing methods of analysis.
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CHAPTER I

INTRODUCTION

The Problem

In today's society, arson is one of the most significant criminal problems (6). "Nobody is really unconcerned about arson: they all agree that it is a problem that needs work. They just believe somebody else is working on it" (11). The crime of arson, often referred to as "the crime of the century" and "America's most malignant crime," is rapidly increasing in frequency and monetary loss, as revealed by statistics compiled by Carter (3) and detailed in Table I.

Table I

SUSPICIOUS AND INCENDIARY FIRES IN THE UNITED STATES

<table>
<thead>
<tr>
<th>Year</th>
<th>Number of Fires</th>
<th>Monetary Loss ($)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1977</td>
<td>177,000</td>
<td>1,159,000,000</td>
</tr>
<tr>
<td>1975</td>
<td>144,100</td>
<td>633,900,000</td>
</tr>
<tr>
<td>1973</td>
<td>94,300</td>
<td>320,000,000</td>
</tr>
<tr>
<td>1971</td>
<td>72,100</td>
<td>233,000,000</td>
</tr>
<tr>
<td>1969</td>
<td>56,300</td>
<td>179,000,000</td>
</tr>
<tr>
<td>1967</td>
<td>44,100</td>
<td>141,700,000</td>
</tr>
<tr>
<td>1965</td>
<td>33,900</td>
<td>74,000,000</td>
</tr>
<tr>
<td>1963</td>
<td>30,900</td>
<td>55,000,000</td>
</tr>
<tr>
<td>1961</td>
<td>21,400</td>
<td>38,400,000</td>
</tr>
<tr>
<td>1959</td>
<td>20,300</td>
<td>27,730,000</td>
</tr>
<tr>
<td>1957</td>
<td>15,000</td>
<td>26,730,000</td>
</tr>
<tr>
<td>1955</td>
<td>9,600</td>
<td>27,100,000</td>
</tr>
<tr>
<td>1953</td>
<td>7,500</td>
<td>22,000,000</td>
</tr>
<tr>
<td>1951</td>
<td>5,600</td>
<td>16,100,000</td>
</tr>
</tbody>
</table>
In keeping with the increase in suspicious fires, the number of suspected arson cases submitted to crime laboratories has also dramatically increased. Even so, only a small amount of research and development has been expended in the arson problem, its investigative techniques, and related equipment (12).

Methods of Analysis--Past and Present

Before 1952, attempts to identify petroleum distillates from fire debris were limited to observable physical properties such as refractive index, boiling range, flashpoint, and density (4). Infrared spectroscopy (IR) was also used (4, 18). In 1952, the development of gas-liquid chromatography (GLC) gave the analyst a powerful analytical procedure to use in the separation and identification of petroleum distillates (4).

Stone (18) related the history of gas chromatography (GC) analysis as it was utilized by the fire debris analyst. The use of GLC was first documented by Midkiff and Washington in 1971 (15). In 1975, Cain reported the use of capillary column GLC (2). Today, GC is the preferred method of analysis of fire debris (1, 4-6, 8-10, 12-14, 16, 17, 20, 21). GC analysis is sometimes supplemented by energy-dispersive X-ray (EDX) analysis (12, 18, 19), flashpoint determination (5), and IR analysis (5, 6, 14, 17, 19-21), although the additional
information gained from the latter technique is still debatable (7).

From the exhaustive, computer-aided literature search conducted by the author, only two articles mentioned the possible application of nuclear magnetic resonance (NMR) spectroscopy analysis of fire debris (12, 18). Although the work detailed in the two articles was preliminary in nature, it was thought that NMR could provide the fire debris analyst with another powerful analytical technique, complementing the GC methods currently in use.

**Scope of the Research**

The purpose of the research was to investigate the possibility of utilizing NMR spectroscopy as an analytical tool in the analysis of fire debris, to develop a simple technique that could be utilized in a forensic laboratory, and to introduce this technique into a scheme of analysis that would insure a consistent treatment of the fire debris submitted to a laboratory from receipt of the debris to the completion of the analysis.


CHAPTER II

MATERIALS AND METHODS

Choice of Accelerants to be Studied

The accelerants for this study were chosen on the basis of their frequency of use by arsonists. Table II lists the results of a study conducted by De Haan (2) and an unpublished study conducted by the author (1) to determine the frequency of recovery of different petroleum distillates from fire debris.

TABLE II

FREQUENCY OF PETROLEUM DISTILLATES RECOVERED FROM FIRE DEBRIS

<table>
<thead>
<tr>
<th>Petroleum Distillate</th>
<th>Percent of Accelerants Recovered</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>De Haan (%)</td>
</tr>
<tr>
<td>Gasoline</td>
<td>69</td>
</tr>
<tr>
<td>Medium Range petroleum</td>
<td>15</td>
</tr>
<tr>
<td>distillates</td>
<td></td>
</tr>
<tr>
<td>Charcoal lighter fluid</td>
<td>6</td>
</tr>
<tr>
<td>Diesel fuel</td>
<td>4</td>
</tr>
<tr>
<td>Kerosine</td>
<td>3</td>
</tr>
<tr>
<td>Cigarette lighter fluid</td>
<td>3</td>
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</tbody>
</table>

The data from this table were considered, so that the accelerants studied would represent a typical sampling of accelerants recovered and identified from fire debris analyzed in a forensic laboratory. Table III lists the accelerants used in this study.
TABLE III

ACCELERANTS STUDIED

Gasolines . . . . . . . . Texaco unleaded
Texaco regular
Exxon unleaded
Exxon regular
Shell unleaded
Shell regular

Medium range petroleum
distillates . . . . . . . . Varsol
Tru-test paint thinner
Painter's naphtha

Others . . . . . . . . . . Gulf charcoal lighter fluid
Sunnyside Kerosine
Conoco diesel

The brand names were chosen because of their availability, and they were documented to see whether brands could be distinguished by this method (3, 4). After the accelerants were obtained, they were stored in one-quart metal paint cans in a refrigerator at 2°C to reduce their volatility and to preserve their original composition (4).

Preparation of Weathered Accelerants

Samples were taken from the stock accelerants and placed in 2-mL sample vials. In addition, 40 mL of each accelerant were evaporated to 10 mL on a hotplate at 90°C, and were stored in this "weathered" condition (25% V₀) in 2-mL sample vials.
Preparation of Burned and Steam-Distilled Accelerants

Yellow pine two-by-fours were cut into 6-inch blocks, and each block was split into 2 pieces and identified by a scribed tack. One piece of the wood was used as a control and the other for the sample. Both sample and control pieces of wood were placed in a 1-quart pyrex dish. 100 mL of an accelerant was poured over the wood and the dish was then covered. The wood was allowed to soak in the accelerant for 3 minutes. Both pieces of wood were then removed and placed on a 9" x 9" pane of glass and allowed to dry for 1 minute. Next, the control was immersed in a new gallon paint can filled with 1/2 gallon of water and sealed with a lid. The sample piece of wood was then ignited with a match on the glass pane under a fume hood and allowed to burn for 1 minute. The flame was then extinguished with an air jet and the sample was immersed in a similar paint can as the control and sealed. Both pieces of wood were then steam distilled for one hour following the procedure described by Stone (3). Figure 1 illustrates the steam distillation apparatus used. The distillate was then collected, dried with sodium chloride, and placed in 2-mL sample vials.
FIG. I -- Steam distillation apparatus
Analysis

The four samples of each accelerant--virgin, weathered, steam distilled, and burned-steam distilled,--were analyzed by gas chromatography and with a Hitachi Perkin-Elmer R124A Nuclear Magnetic Resonance Spectrometer. The gas chromatograph's six-foot column was packed with 3% SP2100. The gas chromatograph was equipped with a flame ionization detector. Deuterated chloroform was used as a solvent and tetramethylsilane was used as an internal standard for the NMR analysis.

Solvent Study

A study was also conducted using several NMR solvents to see whether there was any effect on the resulting spectra. The solvents used were deuterated chloroform, carbon tetrachloride, deuterated acetone, carbon disulfide, and no solvent (neat sample). A 10% gasoline--90% solvent mixture was analyzed using nuclear magnetic resonance spectroscopy.

Detectibility Limit Study

An analysis of 5%, 3%, and 2% gasoline-carbon tetrachloride samples was performed using nuclear magnetic resonance spectroscopy to determine the detectibility limits and effects of concentration on the analysis.
CHAPTER BIBLIOGRAPHY


RESULTS AND DISCUSSION

Steam Distillation of Accelerants

Table IV lists the amounts of accelerants recovered from the burning-steam distillation procedure.

TABLE IV
DISTILLATE COLLECTED BY STEAM DISTILLATION

<table>
<thead>
<tr>
<th>Petroleum Distillate</th>
<th>Control (mL)</th>
<th>Burned (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texaco Unleaded Gasoline</td>
<td>0.6</td>
<td>0.2</td>
</tr>
<tr>
<td>Shell Unleaded Gasoline</td>
<td>0.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Varsol</td>
<td>0.6</td>
<td>0.1</td>
</tr>
<tr>
<td>Tru-Test Paint Thinner</td>
<td>0.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Gulf Charcoal Lighter Fluid</td>
<td>0.5</td>
<td>0.2</td>
</tr>
<tr>
<td>Sunnyside Kerosine</td>
<td>0.4</td>
<td>0.2</td>
</tr>
<tr>
<td>Conoco Diesel</td>
<td>0.4</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Separation of the accelerant from the substrate via steam distillation yielded enough distillate to analyze by GC and NMR spectroscopy analysis.

Interpretation of Spectra

Figure 2 shows two NMR spectra whose chemical shifts are expressed in \( \mu \) units, relative to tetramethylsilane. For accelerants, a spectrum can be divided into four major regions: aliphatic portion (A) (0-2 ppm), aliphatic moieties of aromatic components (B) (2-3 ppm), a dead
FIG. 2 -- NMR spectra of (a) benzene, toluene, and xylene mixture, and (b) Texaco unleaded gasoline.
space where no large peaks are observed (C) (3-6.5 ppm), and an aromatic region (D) (6.5-7.5 ppm). No peaks were observed farther downfield than about 7.5 ppm. Notice in the gasoline spectrum that the peaks contained in the B and D regions are due, at least in part, to the benzene; toluene, and xylene components. These components are easily identified and are present to some extent in many accelerants, especially gasoline (2). From inspection of the spectra obtained, it can be seen that the relative size and shape of the peaks in region A are strongly indicative of any particular accelerant. Therefore, by noting the relative sizes of the peaks, the accelerant can be identified. Figure 3 shows that, regardless of the condition of the accelerant, the general relative abundance of the B and D peaks remains fairly constant. It was found that an accelerant could be identified by NMR regardless of the condition of the accelerant. This identification is not always possible by GC analysis, as burning or separating the accelerant can cause a loss of components, which can complicate identification (8). Figures 4-14 show NMR spectra of the accelerants studied. Note that each accelerant has its own unique pattern and relative B and D peak shapes and abundances. This unique pattern for each accelerant varies only slightly due to
FIG. 3 -- NMR spectra of Texaco unleaded gasoline: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 4 -- NMR spectra of Shell unleaded gasoline: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 5 — NMR spectra of Varsol: (a) virgin; (b) weathered; (c) burned-steam distilled; (d) steam distilled.
FIG. 6 -- NMR spectra of Tru-Test paint thinner: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 7 -- NMR spectra of Sunnyside kerosine: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 8 -- NMR spectra of Gulf charcoal lighter fluid: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 9 -- NMR spectra of Conoco diesel: (a) virgin; (b) weathered; (c) steam distilled; (d) burned-steam distilled.
FIG. 10 -- NMR spectra of Texaco regular gasoline:
(a) virgin; (b) weathered.
FIG. 11 -- NMR spectra of Shell regular gasoline:
(a) virgin; (b) weathered.
FIG. 12 -- NMR spectra of Exxon unleaded gasoline: (a) virgin; (b) weathered.
FIG. 13 -- NMR spectra of Exxon regular gasoline: (a) virgin; (b) weathered.
FIG. 14 -- NMR spectra of painter's naphtha: (a) virgin; (b) weathered.
sample condition and brand of products, so that one is unable to distinguish between brands of a product. This conclusion is supported in the literature (4, 5).

Using the NMR technique, it is easy to distinguish among gasoline, medium range petroleum distillates (Varsol, paint thinner, naphtha, charcoal lighter fluid), and heavy petroleum distillates (kerosine, diesel), while distinction within a class can be accomplished with a well-tuned instrument.

Figures 15-24 are gas chromatograms of the accelerants studied. They have been included for documentation and for experimental control. It can be seen that evaporation of the accelerant can greatly alter its gas chromatograph.

Solvent Study

The solvent study revealed that the solvent used has no appreciable effect on the spectrum obtained. Carbon tetrachloride can be used to obtain the same quality spectrum as the noxious-smelling carbon disulfide and the more expensive deuterated chloroform. The sample can even be analyzed without the use of a solvent (neat) in those situations where very little distillate is obtained and further analysis is desired.
FIG. 15 -- Gas chromatograms of Texaco unleaded gasoline: (a) virgin; (b) weathered.
FIG. 16 -- Gas chromatograms of Texaco regular gasoline: (a) virgin; (b) weathered.
FIG. 17 -- Gas chromatograms of Exxon unleaded gasoline: (a) virgin; (b) weathered.
FIG. 18 -- Gas chromatograms of Exxon regular gasoline: (a) virgin; (b) weathered.
FIG. 19 -- Gas chromatograms of Shell unleaded gasoline: (a) virgin; (b) weathered.
FIG. 20 -- Gas chromatograms of Shell regular gasoline: (a) virgin; (b) weathered.
FIG. 21 -- Gas chromatograms of Varsol: (a) virgin; (b) weathered.
FIG. 22 — Gas chromatograms of Tru-Test paint thinner:
(a) virgin; (b) weathered.
FIG. 23 -- Gas chromatograms of Gulf charcoal lighter fluid: (a) virgin; (b) weathered.
FIG. 24 -- Gas chromatograms of Sunnyside kerosine: 
(a) virgin; (b) weathered.
Detectibility Limit Study

The detectibility limit study was encouraging, showing that, in the case of gasoline, identification can be made at a 2% level of concentration (Figure 25).

Case Studies

After the NMR method was researched, it was applied to actual cases submitted to the Southwestern Institute of Forensic Sciences in Dallas, Texas. Figure 26 shows spectra of gasoline from two cases submitted to the laboratory. Note that (b) matches the standard gasoline spectrum nicely. The (c) spectrum sample was distilled from severely burned and dehydrated debris. Upon GC analysis, it was concluded that gasoline could be present, but there were too many interfering peaks to make a positive identification. The NMR spectrum, however, reveals the presence of gasoline in the sample. Because the NMR is not as sensitive to minute quantities of a compound as GC, interfering substrate does not offer the problems with the NMR technique as it does with GC.

In Figure 27, the charcoal lighter fluid and kerosine spectra are from cases submitted to the laboratory. Similarities to the standards already presented are readily apparent. Spectrum (c) is an example of a typical non-accelerant type distillate obtained from a case sample. An interesting aspect of the NMR method is seen
FIG. 26 -- NMR spectra of gasolines: (a) gasoline; (b) case spectrum; (c) case spectrum.
FIG. 27 -- NMR spectra of case studies: (a) charcoal lighter fluid; (b) kerosine; (c) negative accelerant spectrum.
here, in that the peaks between 5.0 and 7.1 can be attributed to styrene, a common interfering species.

Merits and Limitations

The merits of the NMR technique are significant enough to be considered by the analyst for use in cases of suspected arson. It has purposely been kept simple so that it can be easily adapted to the specific needs of the analyst. The procedure has been used by the Dallas County Forensic Laboratory in cases involving fuel contamination and gasoline seepage, as well as arson. As already seen, it aids in the identification of interfering species and is not sensitive to small amounts of interfering species, which is a major problem with present methods of analysis (2, 7, 8). Another advantage of the NMR method is that spectra of standards and samples from different laboratories are easily compared, which is not necessarily true for GC, due to the diversity of columns and instrumental parameters in use today (6).

The method suggested in this paper represents an unconventional use of NMR spectroscopy. This can be partially attributed to the composition of accelerants, which are sometimes mixtures of hundreds of components (3). This makes peak splitting studies ineffective. Integration is also ineffective due to the change of peaks which occurs because of evaporation, weathering, and
distillation. More elaborate NMR techniques could have been studied, but looking for peaks in specific regions of the spectrum and comparing their relative intensities to other peaks should yield an easy, quick, and specific analysis with the advantage of simplicity.

Two drawbacks were discovered with the procedure. As previously reported, NMR is less sensitive than GC, which could present a problem if only a minute concentration of accelerant is present in the sample. Also, separation of the accelerant from the burned debris is a necessary prerequisite to analysis.

Scheme of Analysis

As stated earlier, the scope of the research included the introduction of the NMR method of analysis into to a scheme of analysis that would insure a consistent treatment of the fire debris submitted to a laboratory from receipt of the debris to the completion of the analysis. This was best accomplished by developing a flow chart similar to that illustrated in Figure 28 (1). The scheme of analysis which was developed appears as Figure 29.
FIG. 28 -- Flow chart of fire debris analysis
Sealed Container Received From Fire Marshal.
(Strong Odor?)

Yes ↓

HEADSPACE ANALYSIS
(Identification of Accelerant?)

Yes ↓

(Is Accelerant Gasoline?)

No ↓

Report Identity of Accelerant

No ↓

Yes ↓

STEAM DISTILLATION

Yes ↓

G.C., NMR, IR ANALYSIS
(Identification of Distillate?)

Yes ↓

Report "No Evidence of Accelerant Detected."

No ↓

Yes ↓

(Is Accelerant Gasoline?)

No ↓

Report "Indeterminate Results"

Yes ↓

E.D.X.
(Pb and Br Present?)

Yes ↓

Report "Leaded Gasoline"

No ↓

Report Identity of Accelerant

No ↓

STEAM DISTILLATION

(Identify Accelerant Gasoline?)

Yes ↓

Report "Unleaded Gasoline"

FIG. 29 -- Flow chart of fire debris analysis
CHAPTER BIBLIOGRAPHY


BIBLIOGRAPHY

Articles


Reports


