

Determination of the Chemical Structure of the Iraqi Oil Shale and Its Hydrocarbon Forms

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Abstract

The global impact of shale oil has revolutionized the world's energy markets, resulting in significantly lower oil prices, higher global gross domestic product, changing geopolitics and shifted business models for oil and gas companies. Further and developed research initiative is required to fill critical gaps in knowledge at the interface of shale oil development along with environmental protection, so countries can prepare better for its energy future. This paper explores the characterization of Iraqi originated oil shale using various analytical techniques, such as mass spectrometry (MS), infrared spectroscopy (IR), and Gas Chromatography Mass Spectrography (GC/MS). Based upon analytical results, it is found that the majority of chemical structure is in aliphatic hydrocarbon forms.

Keywords

Oil Shale, MS, IR, GC/MS, Characterization

1. Introduction

A few years ago, the search for alternative sources of raw materials has concentrated on oil shale deposits. As the exploitation of these raw materials is still extremely complex and expensive, analysis of oil shale samples is therefore extremely important in identifying the deposit's potential in advance to facilitate in optimizing mining methods [1]. "Oil Shale" is a term used to identify rock types found in northern Iraq and currently used within the construction industry; in particular granite tile fabrication. **Figure 1** shows a sample from this type of tiles including the type of oil shale rock which has darker colour. The rocks are solid



Figure 1. The use of Iraqi oil shale within the tiling industry. The darker elements represent the oil shale rock.

with alternating layers of carbonate and organic matter. In Iraq, nowadays, there is no viable literature found regarding these rock types. Part of the reason for the lack of research work relative to “oil shale” is due to cheap production of crude oil in Iraq. The proven massive oil reserves in the country are considered to be the world’s fifth-largest with 140 billion barrels [2]. Oil produced from shale formations costs \$50 to \$100 for a barrel to be produced, compared with \$10 to \$25 for a barrel for conventional supplies from the Middle East and North Africa. **Table 1** summarizes the results of some of Iraq’s oil technical service contracts signed by the Iraqi government with some big oil companies [3]. As the table shows, the cost of Iraqi oil extraction is relatively low. Besides, geologists and consultants have estimated that relatively unexplored territory in the western and southern deserts may contain estimated additional 45 to 100 billion barrels (bbls) of recoverable oil.

However, the production of shale oil, from oil shale, is one of the energy generation alternatives available in the world today. Oil shale is a sedimentary rock containing a complex organic substance called kerogen and its conversion to oil is considered a valuable source of energy. Therefore, an important indicator is the kerogen content which could be relative in predicting accurately the oil yield within the shale. This research focuses on the characterization of this type of oil shale (**Figure 2**) found in Iraq and some other countries in the Middle East, such as Jordan and Palestine, using different techniques as follows, solvent extraction (SE), infrared radiation spectroscopy (IR), and gas and mass chromatography (GC/MS).

2. Methodology

2.1. Samples Preparation

Oil shale samples were obtained from one factory in Baghdad, specializing in the fabrication of tiles. The raw rock material collected from a supplier in Said Sadiq Province, Al Sulaymaniah Governorate, and northern Iraq. All samples were from one deposit. Each shale sample was crushed and mixed thoroughly and then about 10 g was separated and ground to a fine powder (50 μm). Later, the

Table 1. Some of Iraq's oil technical service contract results [3].

Field/block	Company	Home country	Company type
Badra	Gazprom	Russia	State
Badra	Petronas	Malaysia	State
Badra	KOGAS	Korea	State
Badra	TPAO	Turkey	State
Garraf	Petronas	Malaysia	State
Garraf	JAPEX	Japan	Public
Halfaya	CNPC	China	State
Halfaya	Petronas	Malaysia	State
Halfaya	Total	France	Public
Majnoon	Shell	Netherlands	Public
Majnoon	Petronas	Malaysia	State
Missan G.	CNOOC	China	State
Missan G.	TPAO	Turkey	State
Najmah	Sonangol	Angola	State
Qayara	Sonangol	Angola	State
Rumaila	BP	UK	Public
Rumaila	CNPC	China	State
W.Qurna	Exxon	US	Public
Zubair	ENI	Italy	Public
Zubair	Occidental	US	Public
Zubair	KOGAS	Korea	State

**Figure 2.** Iraqi oil shale rock used in this paper.

ground oil shale samples were utilized for analysis.

2.2. Analysis Tests of Chemical Structure

1) MS: mass spectrometry is an analytical tool that uses mass to charge ratios to determine molecular weights of the chemical structure compounds in oil shale samples. This technique has been utilized previously for several studies in pyrolysis of oil shale, tar sands, and coal [4]. Full details of this technique have been

published somewhere else.

2) IR: infrared radiation spectroscopy is well-established as a useful technique for the identifying chemical characteristics of coal and kerogen [5]. The spectra were measured at 1 cm^{-1} resolution and collected in the $400 - 4000\text{ cm}^{-1}$ wave number range.

3) GC/MS: In order to more fully characterize the compounds, present in the Iraqi oil shale, since MS analysis gives a compound classification only, the gas chromatograph coupled with a mass spectrometer using electron impact ionization, is used for analyzing and separating the samples [6]. The GC was equipped with an automatic programmable-temperature system and a capillary column ($30\text{ m} \times 0.25\text{ mm I.D.}$). Helium is used as the carrier gas.

3. Results and Discussion

3.1. MS Analysis

Solvent extraction (SE) for the shale samples was undertaken using two different solvents; dichloromethane and ethanol. There were no results from the extracts with ethanol. Mass Spectrometry (MS) analysis, for the extraction performed when using dichloromethane as a solvent, indicated that there were absorptions of bands at the retention time (tR) (8.5, 8.88, 8.87, 13.15, 14.96 minutes). This result confirmed the extraction and the existence of other compounds, too.

3.2. IR Analysis

Extracted oil shale sub samples were put in a diffuse reflectance cup (10 mm diameter, 3.3 mm depth) in which the packing density was ensured by applying a constant mass (of 30 g) on the top surface of each sample. The IR spectrometer was then used for analyses. The spectra indicated CH-aliphatic at bands with $3052 - 3852\text{ cm}^{-1}$ wave number. Also, there were groups of C-S and S-H compounds at bands with $900 - 1124\text{ cm}^{-1}$ wave number. Finally, amide O-C-NH₂ appeared within the band showing a 1724 cm^{-1} wave number. The curves of different values of percentage transmittance (%T) versus the frequency of vibration, with using dichloromethane as solvent, are presented in **Figure 3** & **Figure 4** respectively.

3.3. IR for the Whole Rock Analysis

IR analysis for the whole rock showed characteristic bands at $2876 - 2980\text{ cm}^{-1}$. The bands were related to CH-aliphatic. Also, Amide O-C-NH₂, appeared with the band, had a wave number of 796 cm^{-1} . In addition, further spectra IR with band peaks at 876 cm^{-1} to 1036 cm^{-1} showed some evidence for the presence of S-H and C-S groups. The IR analysis (**Figure 5**) gives evidence of a complex chemical composition of the sample in forms of different types of groups deriving from the decomposition of various groups of the molecule. In the next section, what kinds of groups are actually existed in the oil shale sample is introduced. The figure profile shows numerous events. In fact, the number of peaks

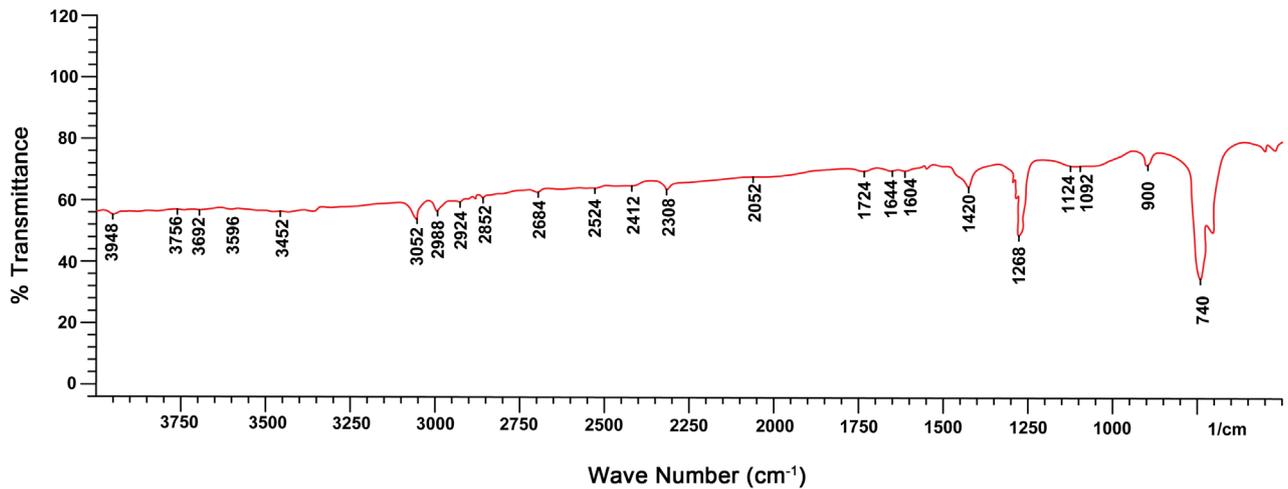


Figure 3. Curve of percentage transmittance (%T) (ranges from 55% - 65%) versus frequency of vibration (in wave numbers) for scanning of the extracted oil samples with using dichloromethane as solvent.

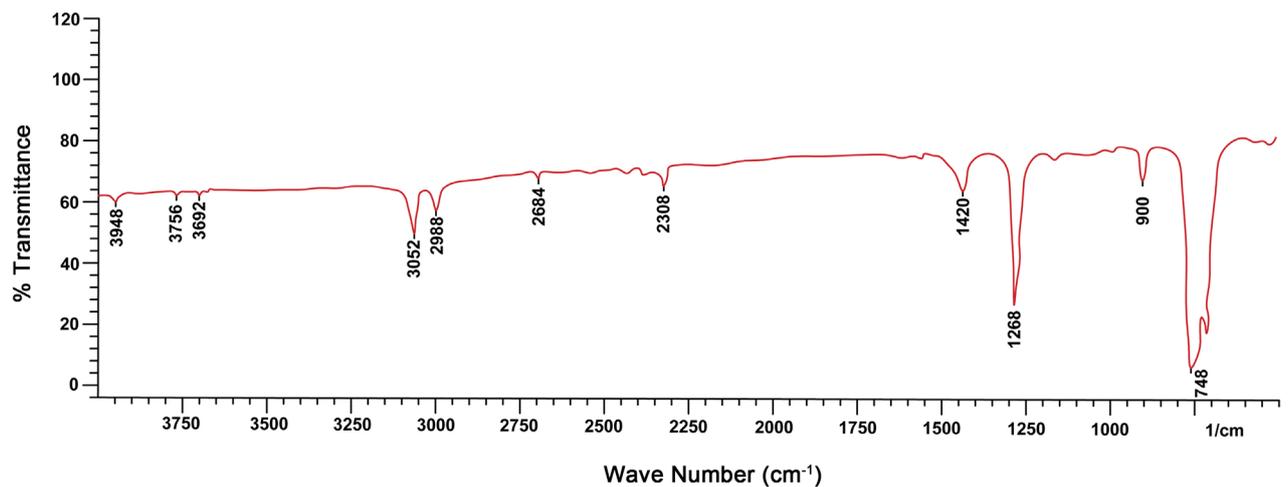


Figure 4. Curve of percentage transmittance (%T) (ranges from 65% - 75%) versus Frequency of vibration (in wave numbers) for scanning of the extracted oil samples with using dichloromethane as solvent.

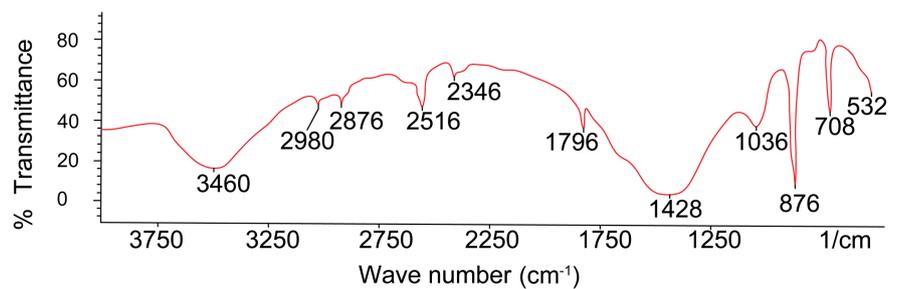


Figure 5. The IR spectra analysis of the whole oil shale rock provided identifiable peaks for the carbon bonds (near 3000 cm^{-1}), and also the oxygen carbon group (near 800 cm^{-1}).

in the chromatogram is more than number of peaks of the extracted oil samples using dichloromethane.

3.4. GC/MS Analysis

The GC separates the compounds from each other, while the mass spectrometer helps to identify them based on their fragmentation pattern. Here, methanol was used and thus considered the best solvent for extraction purposes when looking for information on the chemical structure of the sample. Following extraction, a sample extract was filtered for analysis by GC/MS system. The compounds of hydrocarbon forms were detected in a mass spectrometer (MS) interfaced to gas chromatograph (GC), and they are summarized in **Table 2**. These results are consistent with those of the MS analysis.

The elemental composition of the extracted oil was found to be C, H, and N. Full details of the GC/MS analysis are presented in **Appendix**.

4. Summary of Results

Several analytical techniques were employed to characterize the chemical composition of the oil shale. When considered alone, each technique has its own advantage, but it was unable to provide the whole picture for oil shale [7]. The chemical composition of the oil shale available in Iraq was found to be of a series of hydrocarbon forms. Hydrocarbon type analysis indicated that mono and di-cycloparaffins were the next most abundant constituents after the paraffin series.

The organic carbon content was a good indicator for the quality of organic matter in the oil shale. Depending on the previous result, it was observed that the Iraqi Oil shale contained similar chemical composition of Egyptian oil shale, which indicated in the research of Al-Alla RA, Nassef E [8].

5. Recommendation for Further Research

Chemically, oil shale consists of carbon, hydrogen, oxygen, nitrogen, and sulphur and all of these components are existed in Iraqi oil shale. The existence of these components, which has been noted, may considerably influence the results of structural investigations as reported in the literature. Further characterization of the oil shale by distinguishing between the aliphatic and aromatic hydrocarbons is needed. Also, since the challenges of economically developing unconventional shale deposits vary with the characteristics of the deposit, extraction costs,

Table 2. Hydrocarbon forms in the oil shale samples.

No.	Compound Name	Formula	Molecular weight
1	Propanesulfonylacetonitrile	C ₅ H ₉ NO ₂ S	147.195
2	Butyl Isocyanide	CH ₃ (CH ₂) ₃ NC	83.13
3	O-Allylhydroxylamine	C ₃ H ₇ NO	73.11
5	N-Hexadecanoic Acid	C ₁₆ H ₃₂ O ₂	256.4241
6	2-Propenoic Acid, Ethenyl Ester	C ₅ H ₈ O ₂	100.1158
7	Z-10-Pentadecen-1-ol	C ₁₅ H ₃₀ O	226
8	1,1-Diisobutoxy-Isobutane	C ₁₂ H ₂₆ O ₂	202.334

topography, land uses, water resource availability and climate, some of these concerns can be better understood in the light of further research. Therefore, this highlights further demand for research in a number of relevant areas, including the following:

1) Confirming the proportions of the mixture of hydrocarbon compounds found in the Iraqi oil shale, that it is Kerogen and of which type it is: alpha or beta?

2) Evaluating the potential extracted oil yield.

3) Studying the economic feasibility of extracting Kerogen from this type of oil shale, taking into consideration the oil price which is around \$100/barrel as minimum.

4) Selecting the appropriate processing technology whilst considering the reduction in extraction costs per barrel, and reducing the carbon footprint relative to the extraction process.

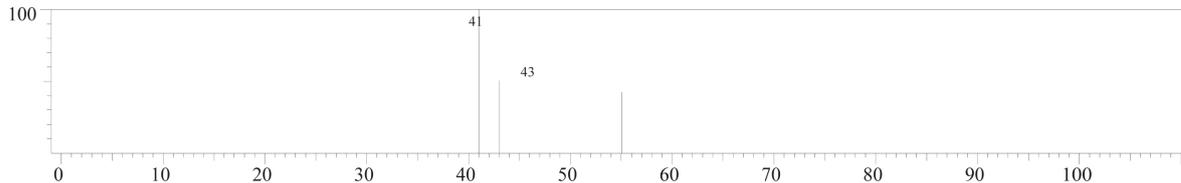
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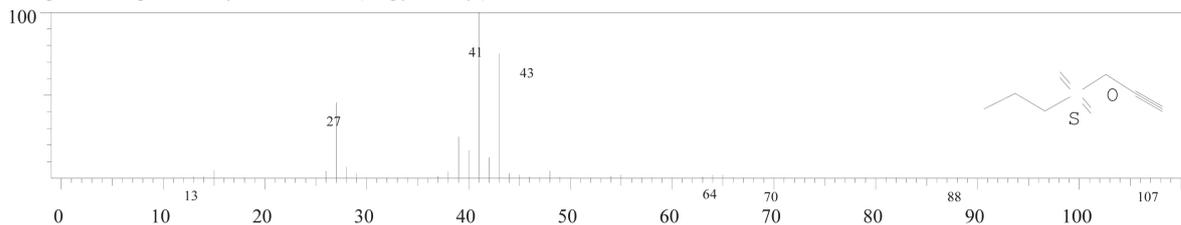
Appendix

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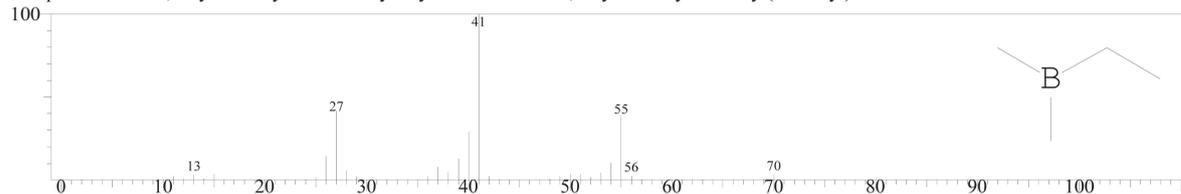
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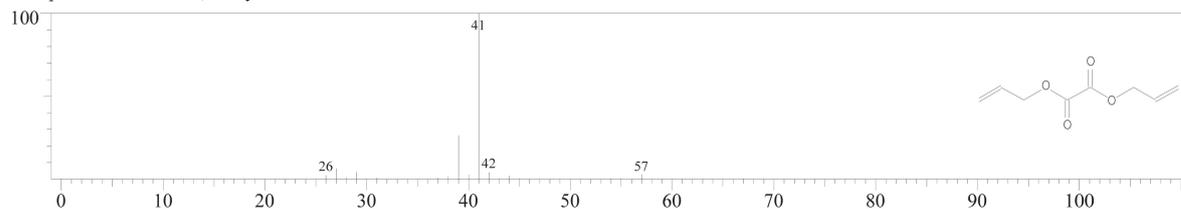
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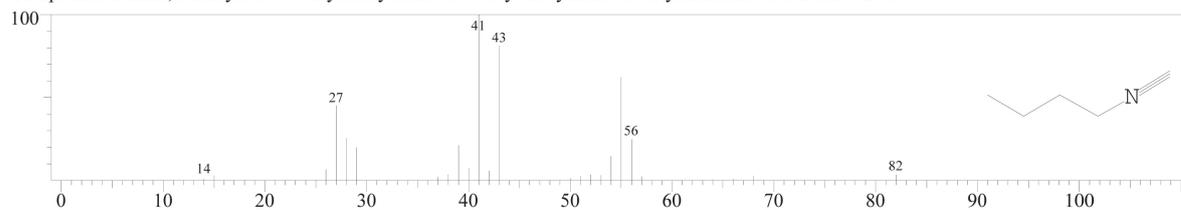
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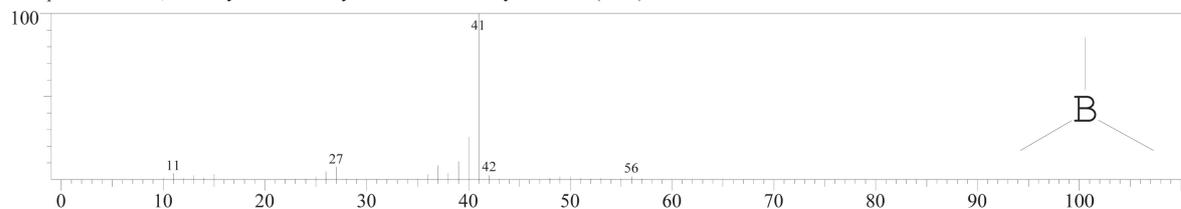
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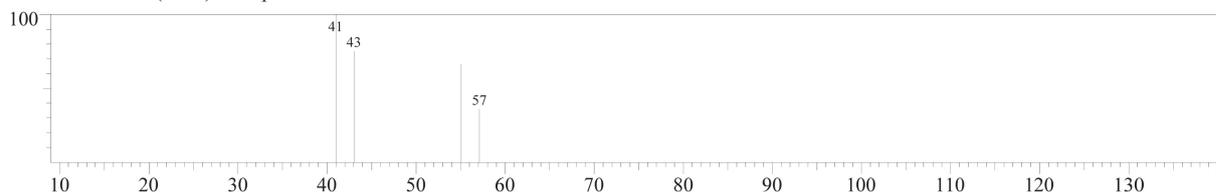


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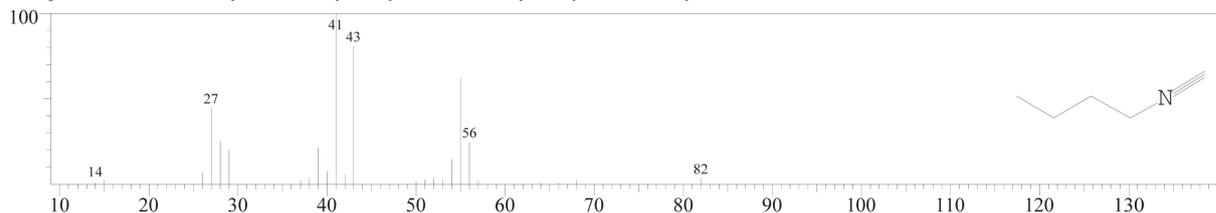


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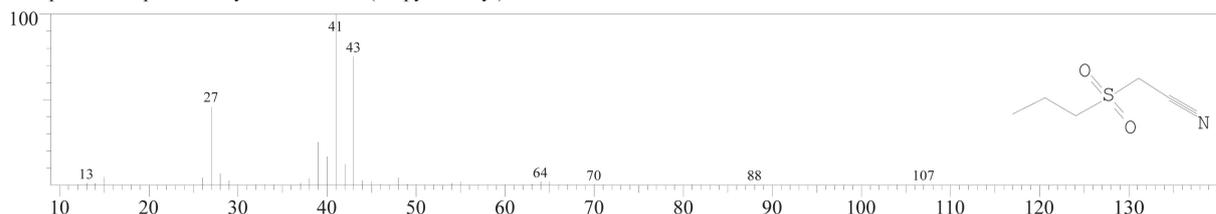
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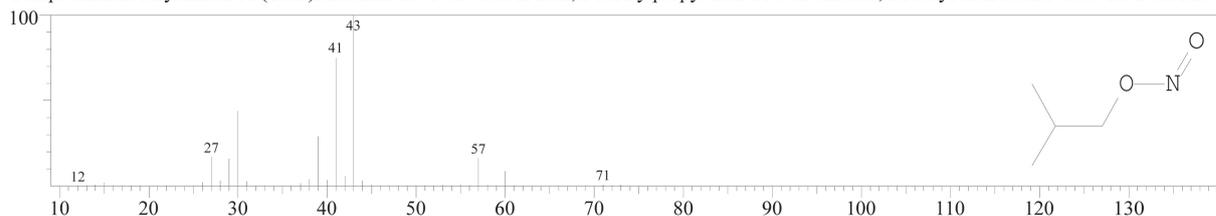
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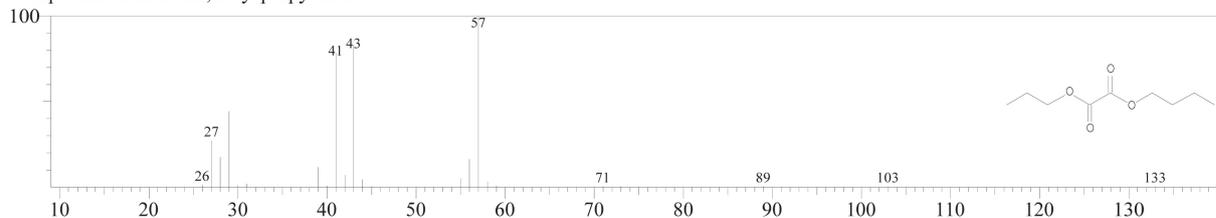
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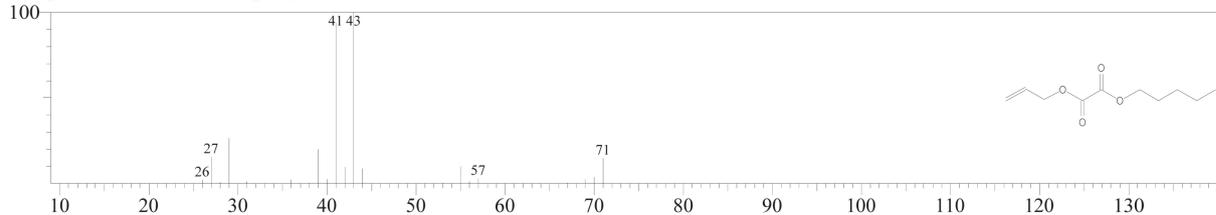
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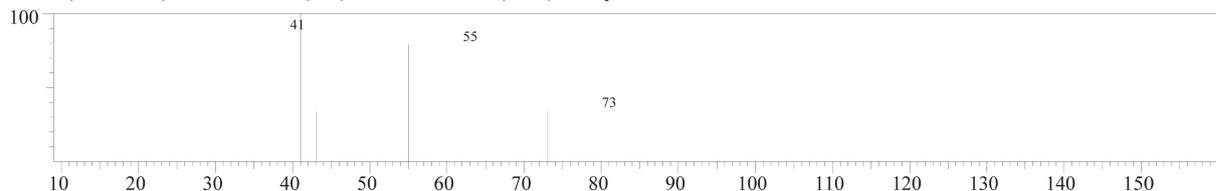


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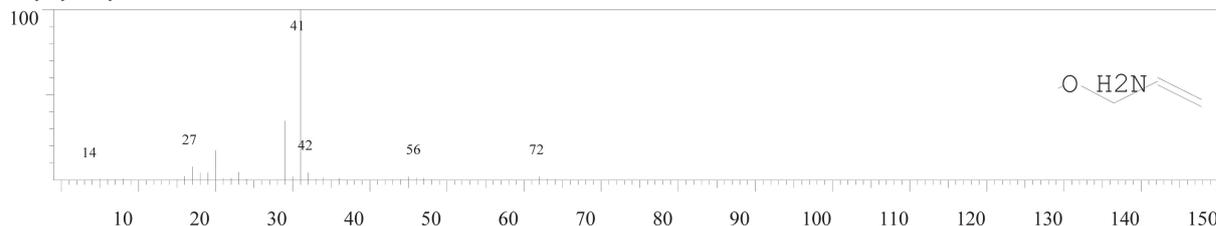
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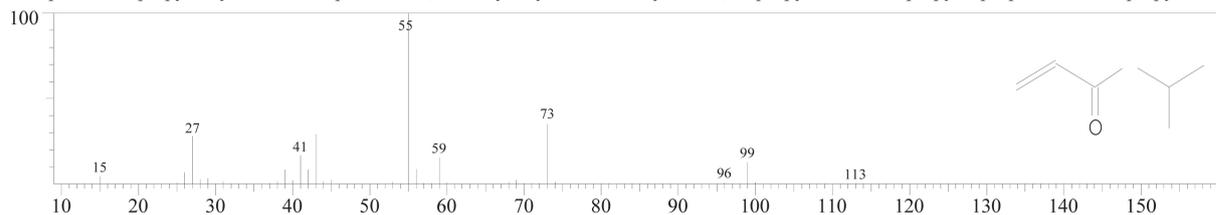
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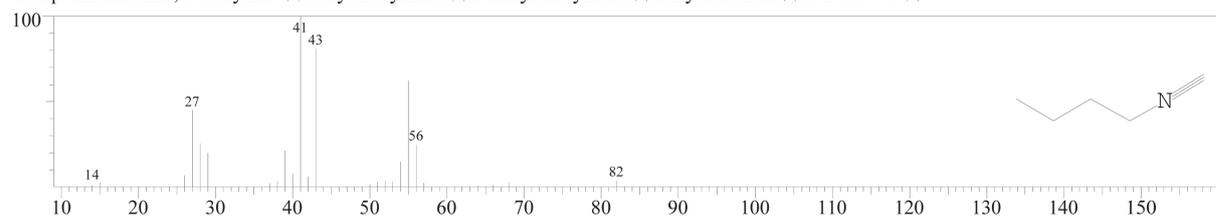
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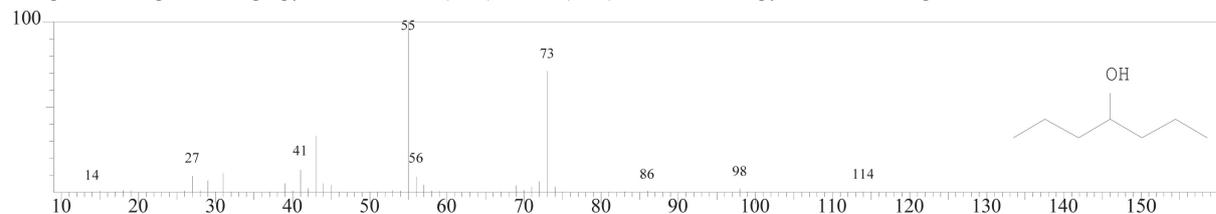
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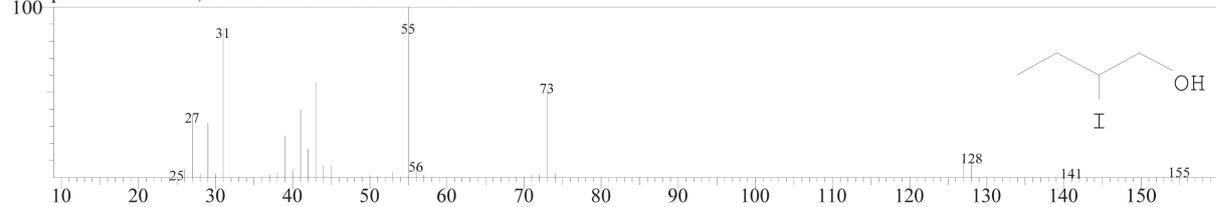
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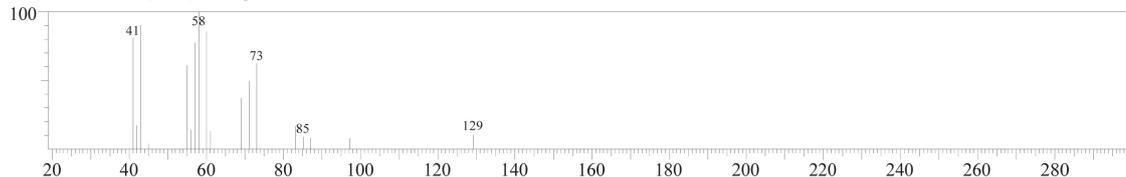
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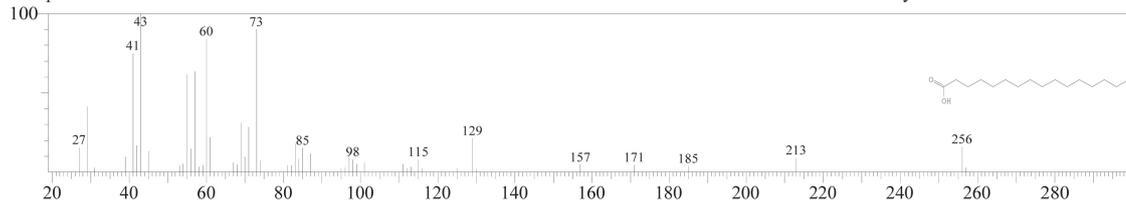


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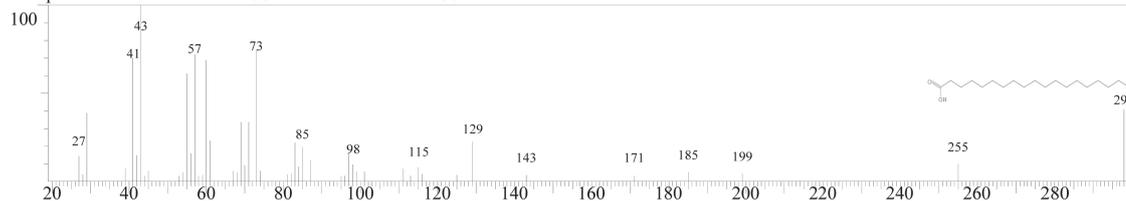
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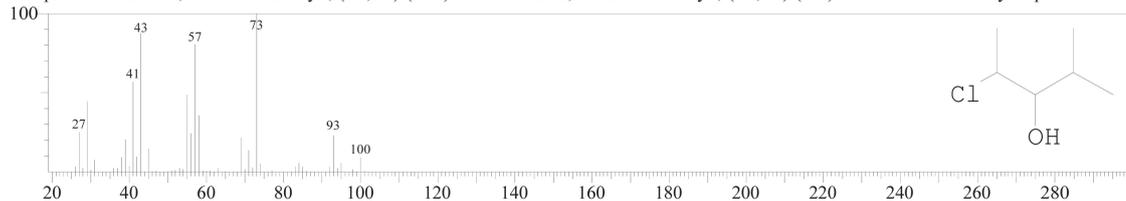
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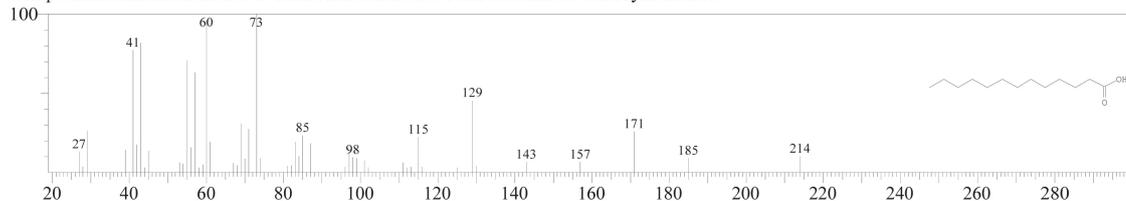
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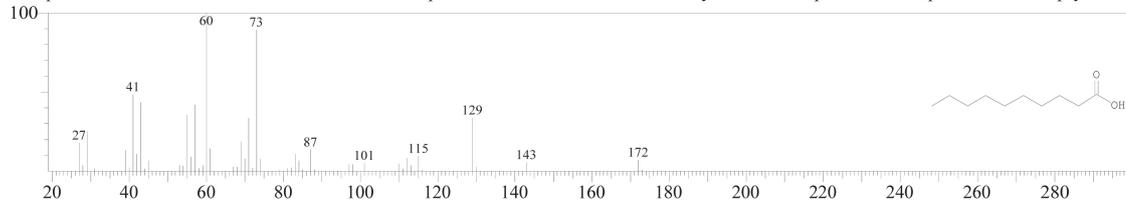
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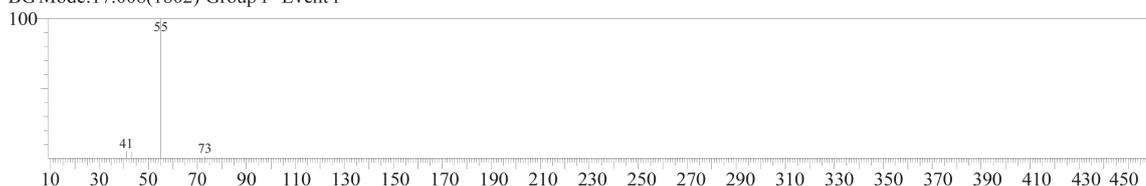


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 SI:79 Formula:C10H20O2 CAS:334-48-5 MolWeight:172 RetIndex:1372
 CompName:n-Decanoic acid\$ Decanoic acid\$ n-Capric acid\$ n-Decoic acid\$ n-Decylic acid\$ Capric acid\$ Capric acid\$ Caprylic acid\$

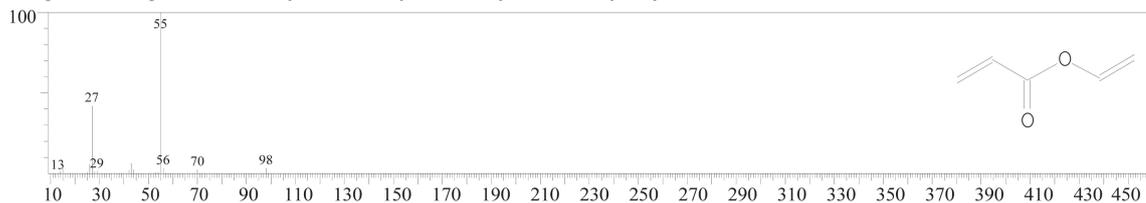


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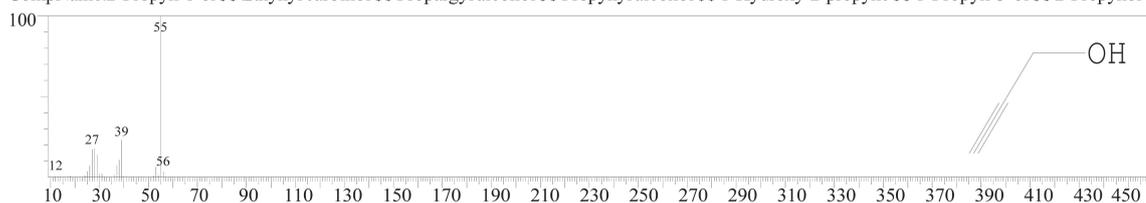
Line#:6 R.Time:16.992(Scan#:1800) MassPeaks:4
 RawMode:Averaged16.942-17.017(1794-1803)BasePeak:55.05(701)
 BG Mode:17.008(1802) Group 1 - Event 1



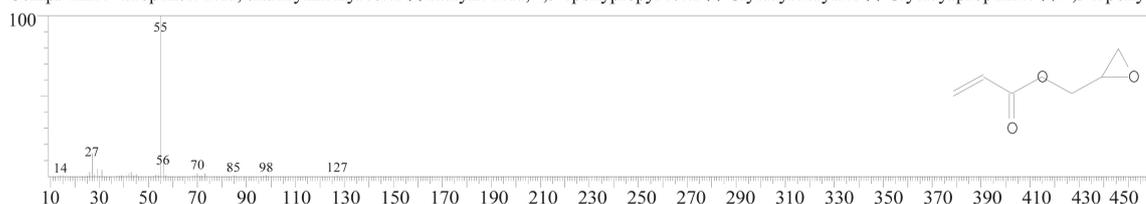
Hit#:1 Entry:1568 Library:NIST08.LIB
 SI:91 Formula:C5H6O2 CAS:2177-18-6 MolWeight:98 RetIndex:666
 CompName:2-Propenoic acid, ethenyl ester \$\$ Acrylic acid, vinyl ester \$\$ Vinyl acrylate \$\$



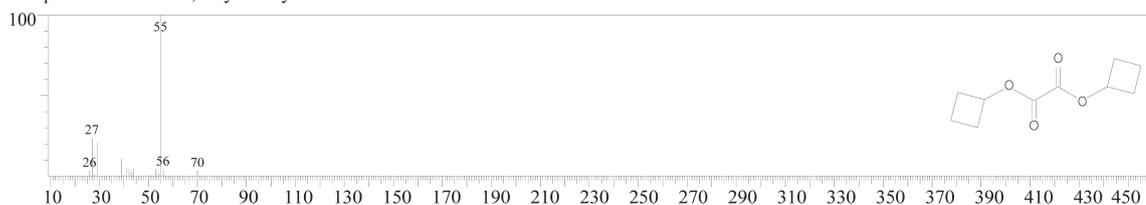
Hit#:2 Entry:89 Library:NIST08.LIB
 SI:91 Formula:C3H4O CAS:107-19-7 MolWeight:56 RetIndex:560
 CompName:2-Propyn-1-ol \$\$ Ethynyl carbinol \$\$ Propargyl alcohol \$\$ Propynyl alcohol \$\$ 1-Hydroxy-2-propyne \$\$ 1-Propyn-3-ol \$\$ 2-Propynol



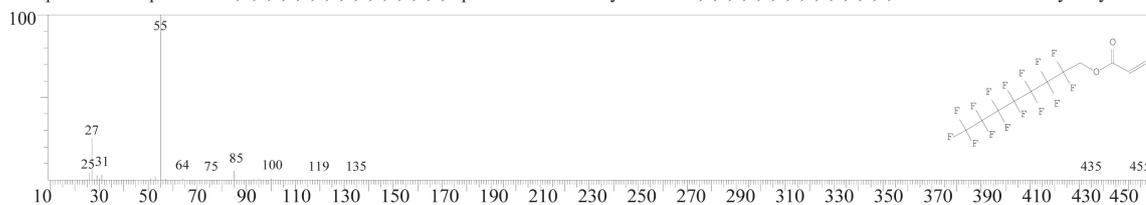
Hit#:3 Entry:7026 Library:NIST08.LIB
 SI:89 Formula:C6H8O3 CAS:106-90-1 MolWeight:128 RetIndex:865
 CompName:2-Propenoic acid, oxiranylmethyl ester \$\$ Acrylic acid, 2,3-epoxypropyl ester \$\$ Glycidyl acrylate \$\$ Glycidyl propenate \$\$ 2,3-Epoxy



Hit#:4 Entry:41447 Library:NIST08.LIB
 SI:89 Formula:C10H14O4 CAS:0-00-0 MolWeight:198 RetIndex:1395
 CompName:Oxalic acid, dicyclobutyl ester

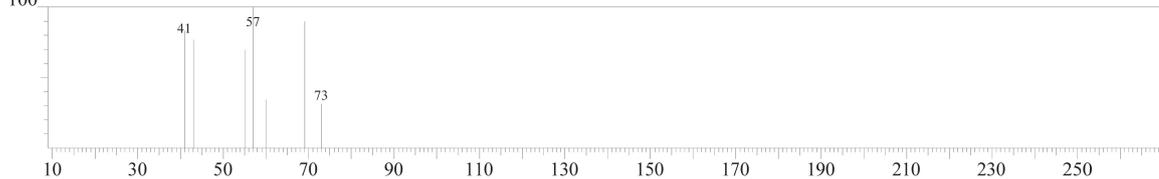


Hit#:5 Entry:179454 Library:NIST08.LIB
 SI:88 Formula:C11H5F15O2 CAS:307-98-2 MolWeight:454 RetIndex:274
 CompName:2-Propenoic acid, 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctyl ester \$\$ 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-Pentadecafluorooctyl acrylate

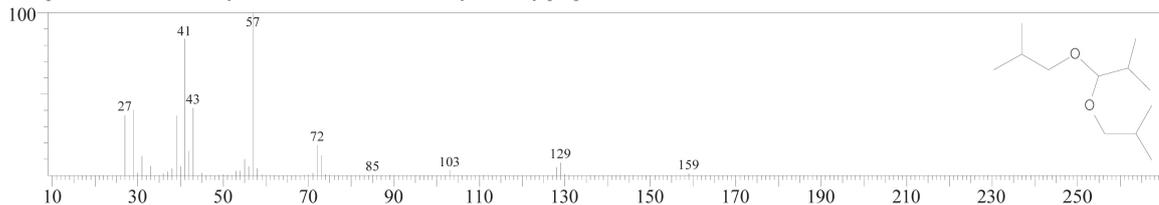


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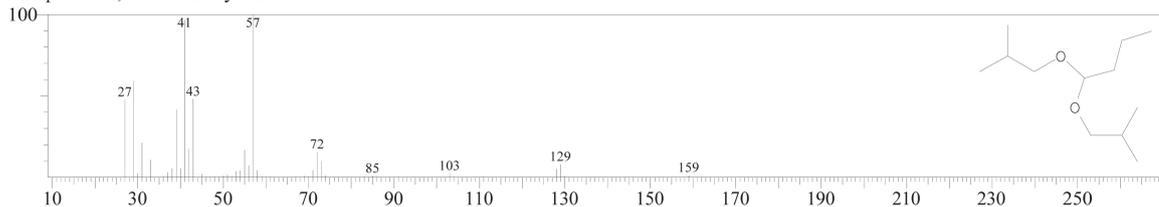
Line#:8 R.Time:18.033(Scan#:1925) MassPeaks:7
 RawMode:Averaged 18.008-18.067(1922-1929)BasePeak:57.05(829)
 BG Mode:18.083(1931) Group 1 - Event 1



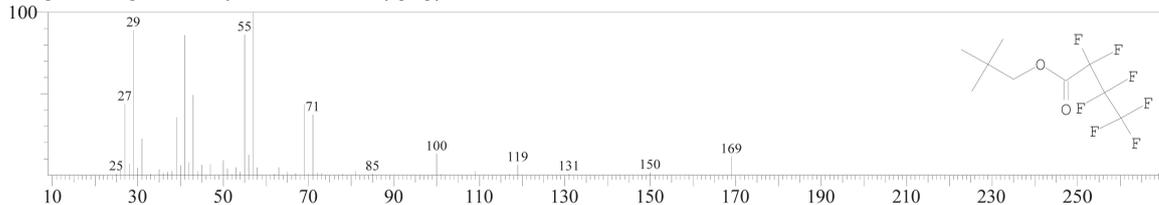
Hit#:1 Entry:44024 Library:NIST08.LIB
 SI:76 Formula:C12H26O2 CAS:13262-24-3 MolWeight:202 RetIndex:1109
 CompName:1,1-Diisobutoxy-isobutane



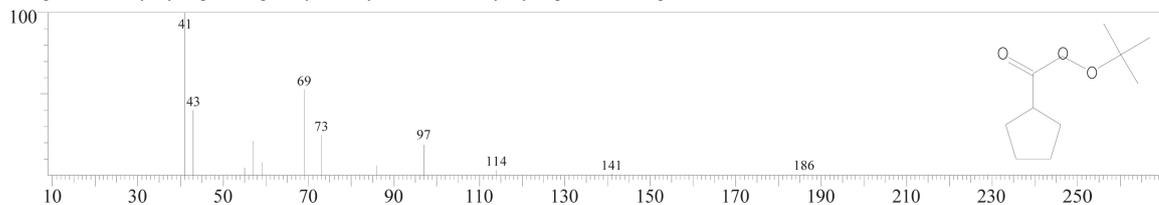
Hit#:2 Entry:44023 Library:NIST08.LIB
 SI:76 Formula:C12H26O2 CAS:13002-16-9 MolWeight:202 RetIndex:1174
 CompName:1,1-Diisobutoxy-butane



Hit#:3 Entry:99958 Library:NIST08.LIB
 SI:75 Formula:C9H11F7O2 CAS:0-00-0 MolWeight:284 RetIndex:555
 CompName:Heptafluorobutyric acid, 2,2-dimethylpropyl ester



Hit#:4 Entry:34345 Library:NIST08.LIB
 SI:75 Formula:C10H18O3 CAS:25023-14-7 MolWeight:186 RetIndex:1217
 CompName:t-Butyl cyclopentaneperoxy-carboxylate



Hit#:5 Entry:4738 Library:NIST08.LIB
 SI:75 Formula:C5H11NO2 CAS:110-46-3 MolWeight:117 RetIndex:644
 CompName:Isoamyl nitrite

