Identification of 10 Compounds by Combined Modern Analytical Techniques

NMR

MS

IR

Introduction

In each of these problems, you are given a range of information and spectra about 10 unknown compounds.

For each compound, it is recommended that you begin by using the **% composition data** to work out the **empirical formula**.

The **mass spectrum** will then allow you to determine the **molecuar formula**. Don't forget the m+1 peak! This is caused by some of the molecules (at least 1 in 100) having ^{13}C atoms in them. The more carbon atoms the molecule has, the greater the relative size of this peak to the m^+ peak. (It's a simple probability thing!)

Try to get as much from the **molecular formula** as you can! Think about levels of saturation (C to H ratio). For example, if you have a hydrocarbon with general formula C_nH_{2n+2} then you know that the compound is saturated and aliphatic. Very high carbon to hydrogen ratios (around 1:1) suggest that the compound is aromatic (an arene). Also, check to see if there are 2 oxygens present. This would suggest an ester or carboxyic acid. This isn't guaranteed but a good starting point.

You should now have some clues as to the possible functional groups that the compound could contain. The **infra-red spectrum** will help confirm the identity of some functional groups. This is especially useful for identifying the presence of **-OH** (alcohols and carboxylic acids) and **C=O** (aldehydes, ketones, carboxylic acids and amides).

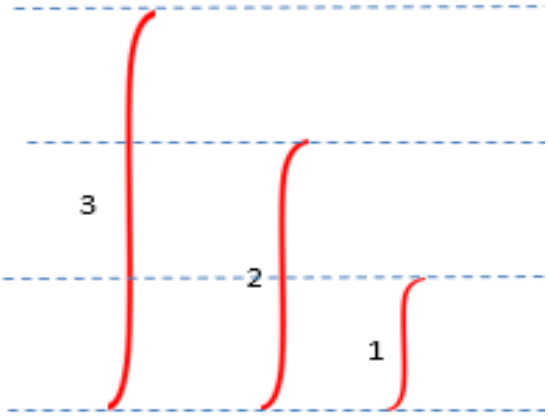
The **^1H and ^{13}C NMR spectra** should confirm the arrangement of the **H** and **C** atoms in the molecule.

Integration.

For the **^1H NMR** you will see an intergation curve for each peak or mulitplet.

The **relative heights** of these integration lines gives you the **relative number of hydrogens** represented by that peak.

Measure the heights of the red integration lines and then find the ratio of their heights.

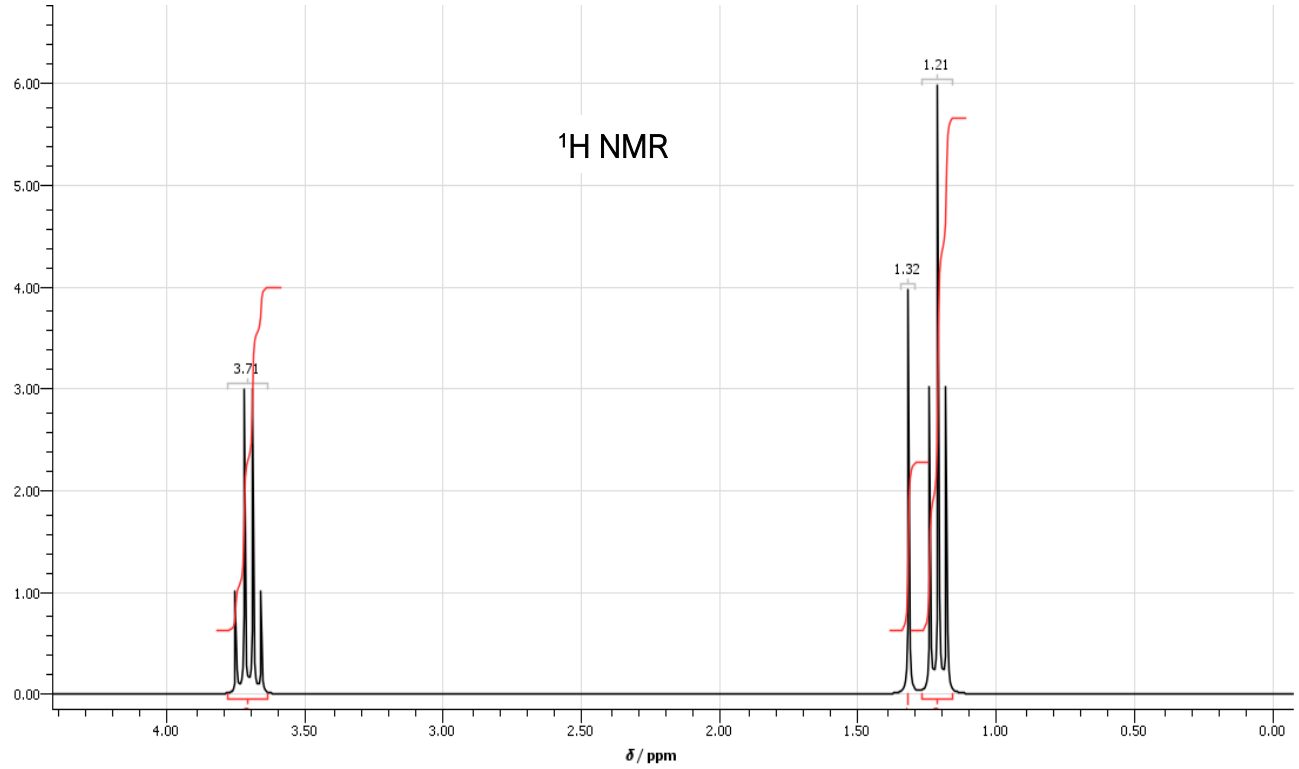


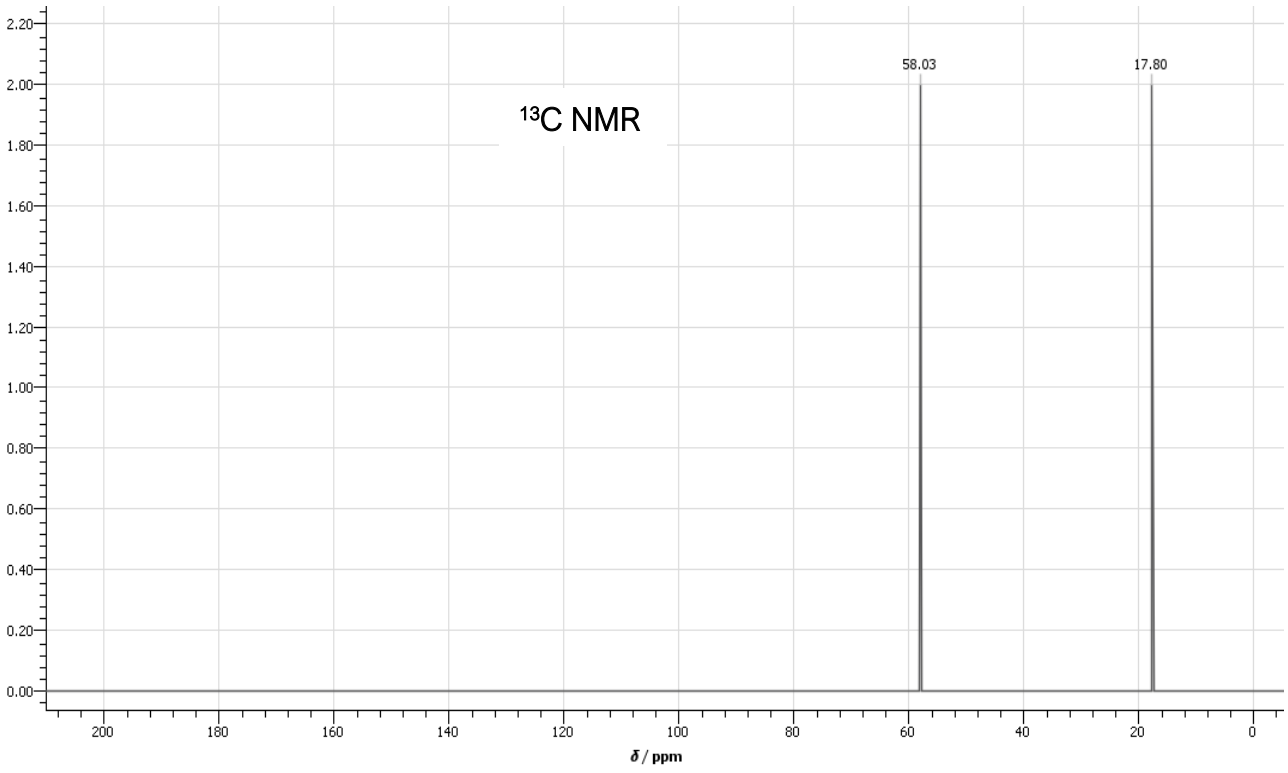
Compound 1

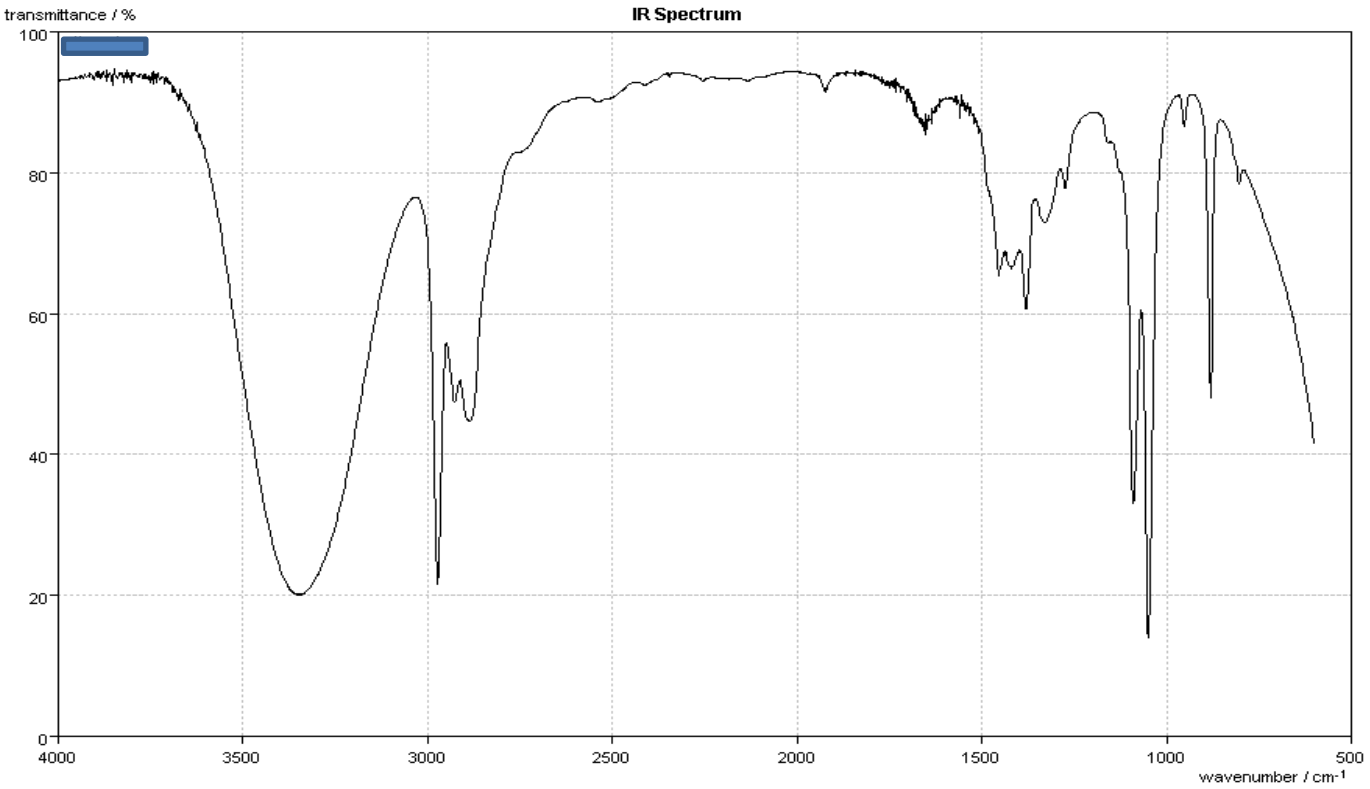
description: Colourless volatile liquid

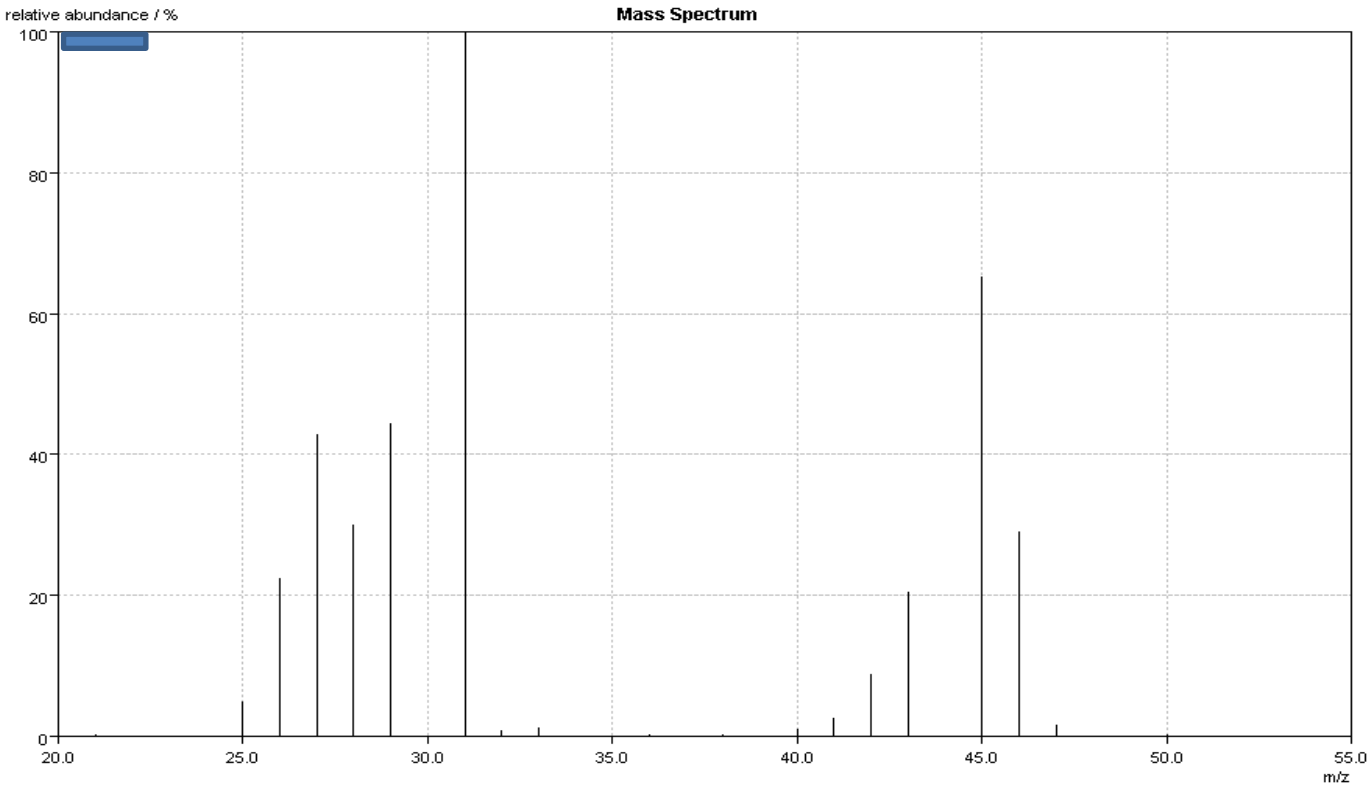
composition: C 52.17%; H 13.04%; O 34.78%

molecular formula: _____









Further Information:

Colourless liquid that mixes completely with water to form a neutral solution.

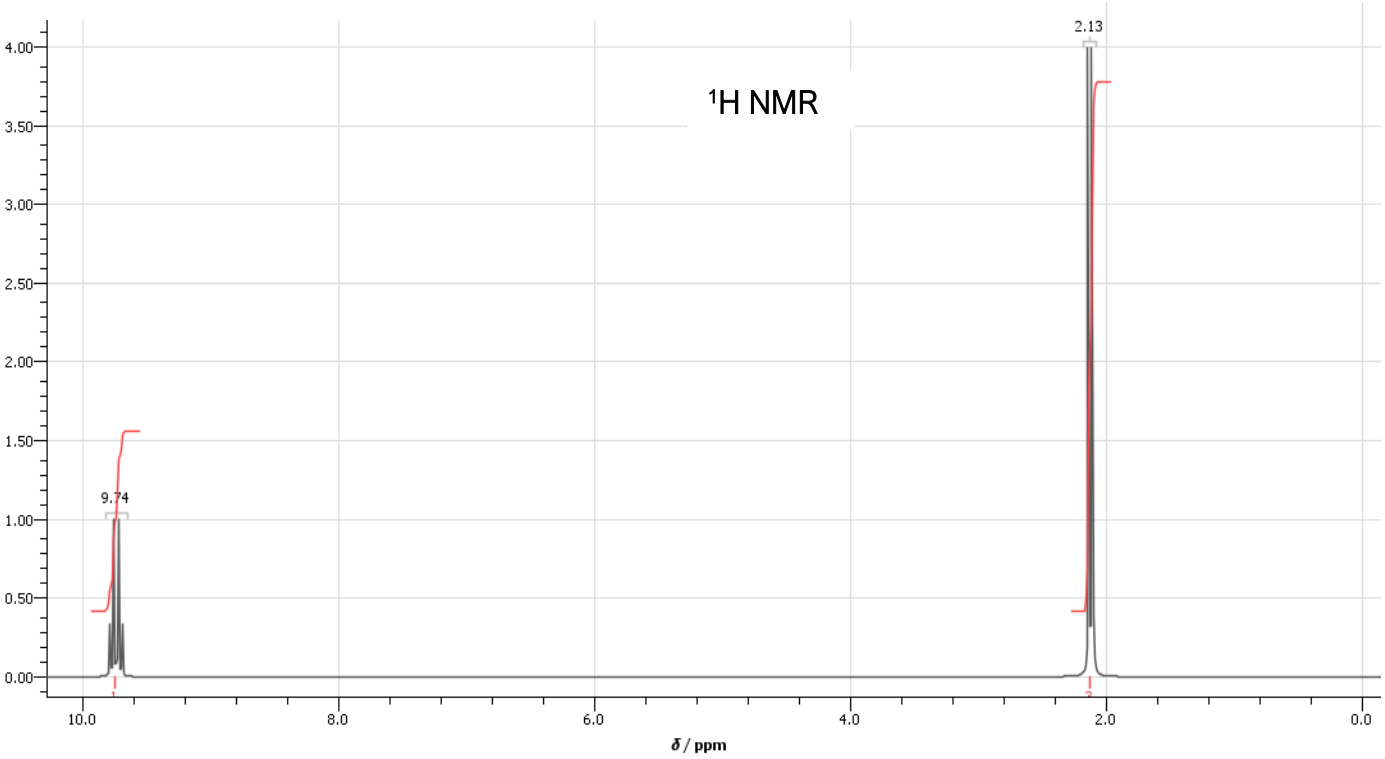
Compound reacts readily with acidified dichromate solution to give two oxidation products.

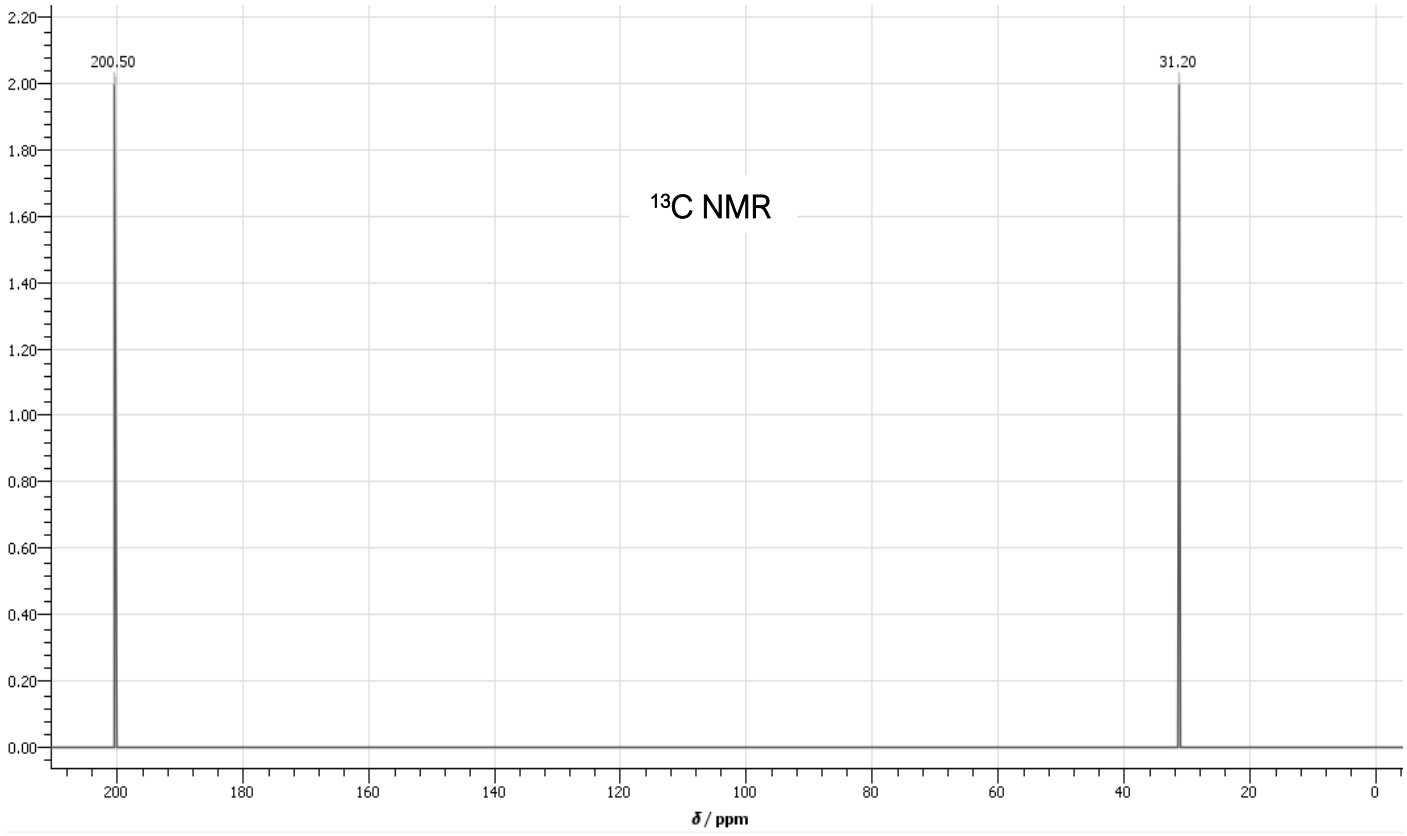
Compound 2

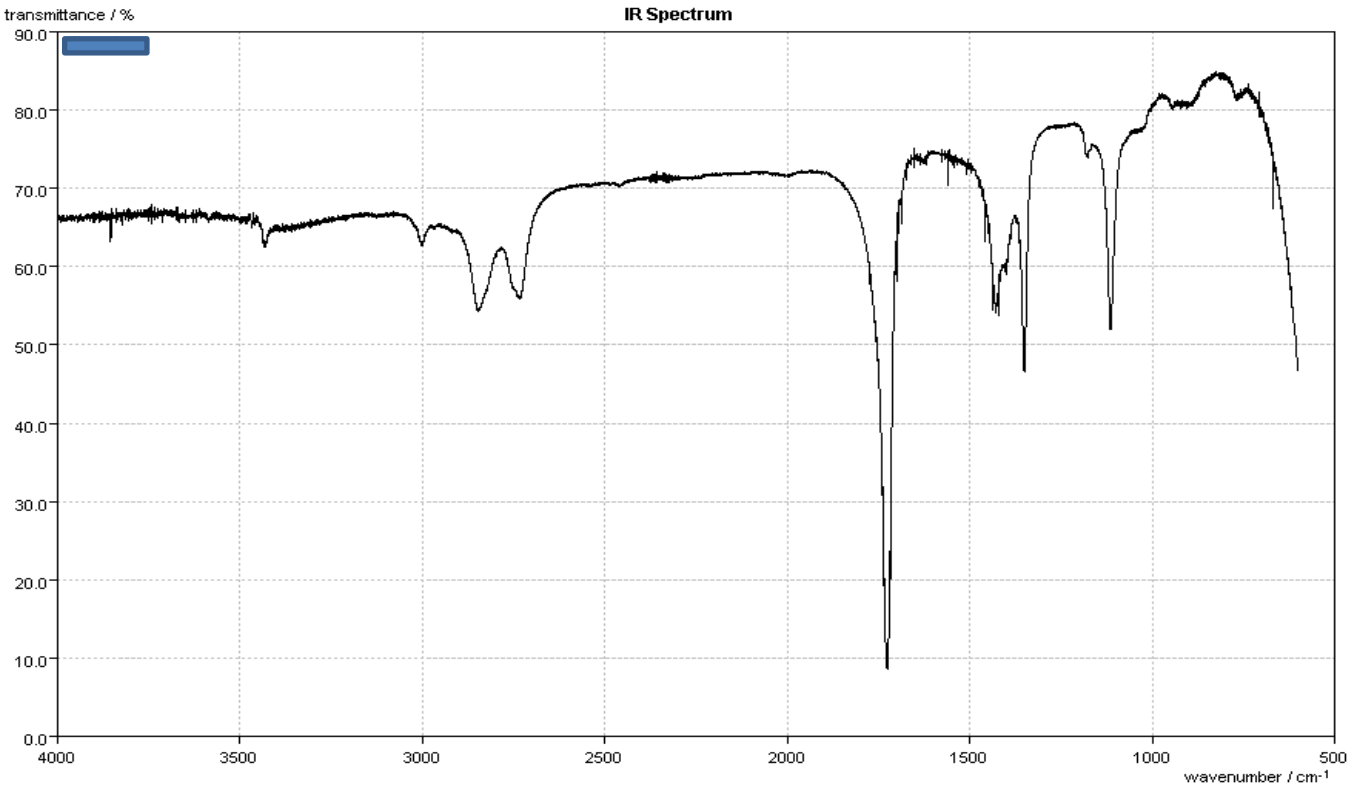
description: Colourless volatile liquid

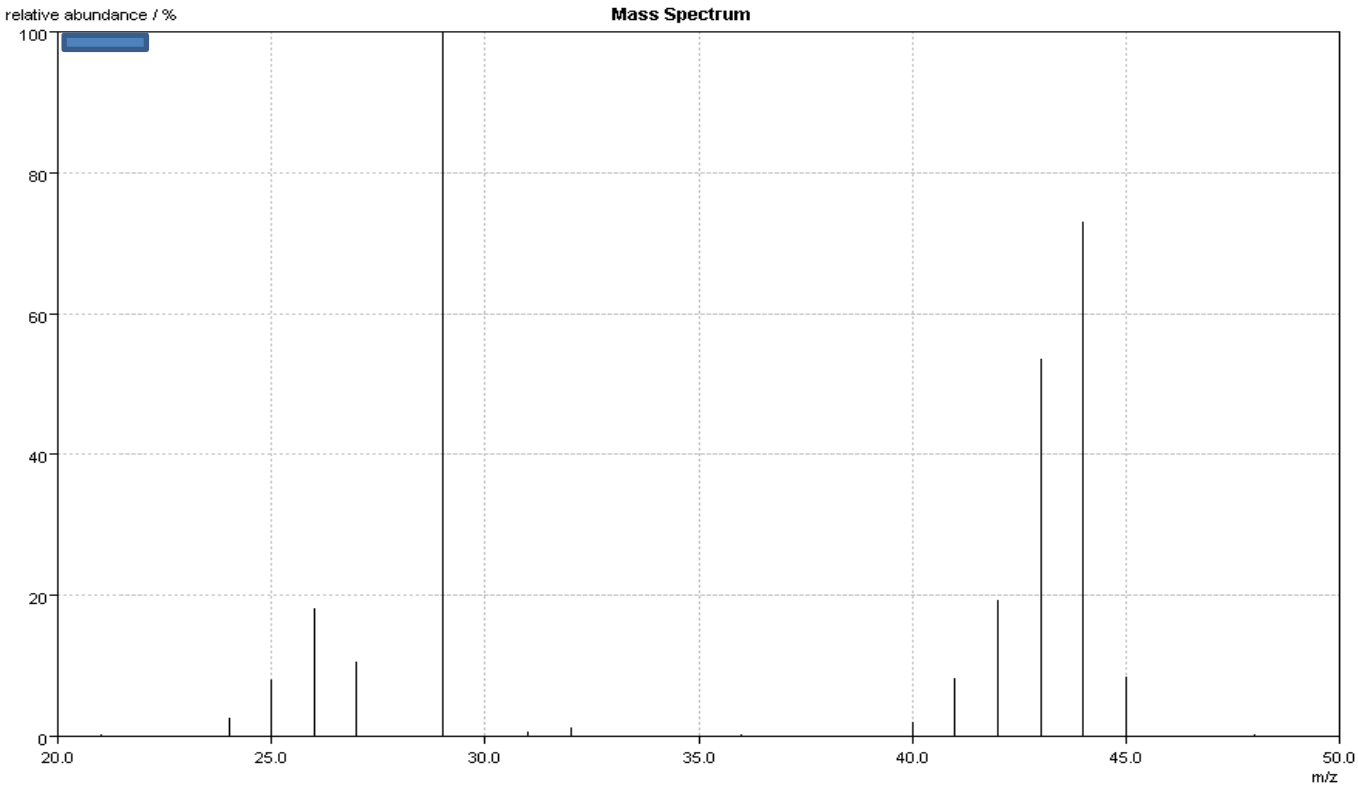
composition: C 54.55% H 9.09% O 36.36%

molecular formula: _____









Further Information:

Compound reacts with 2,4-dinitrophenylhydrazine (Brady's Reagent) to give an intensely coloured precipitate.

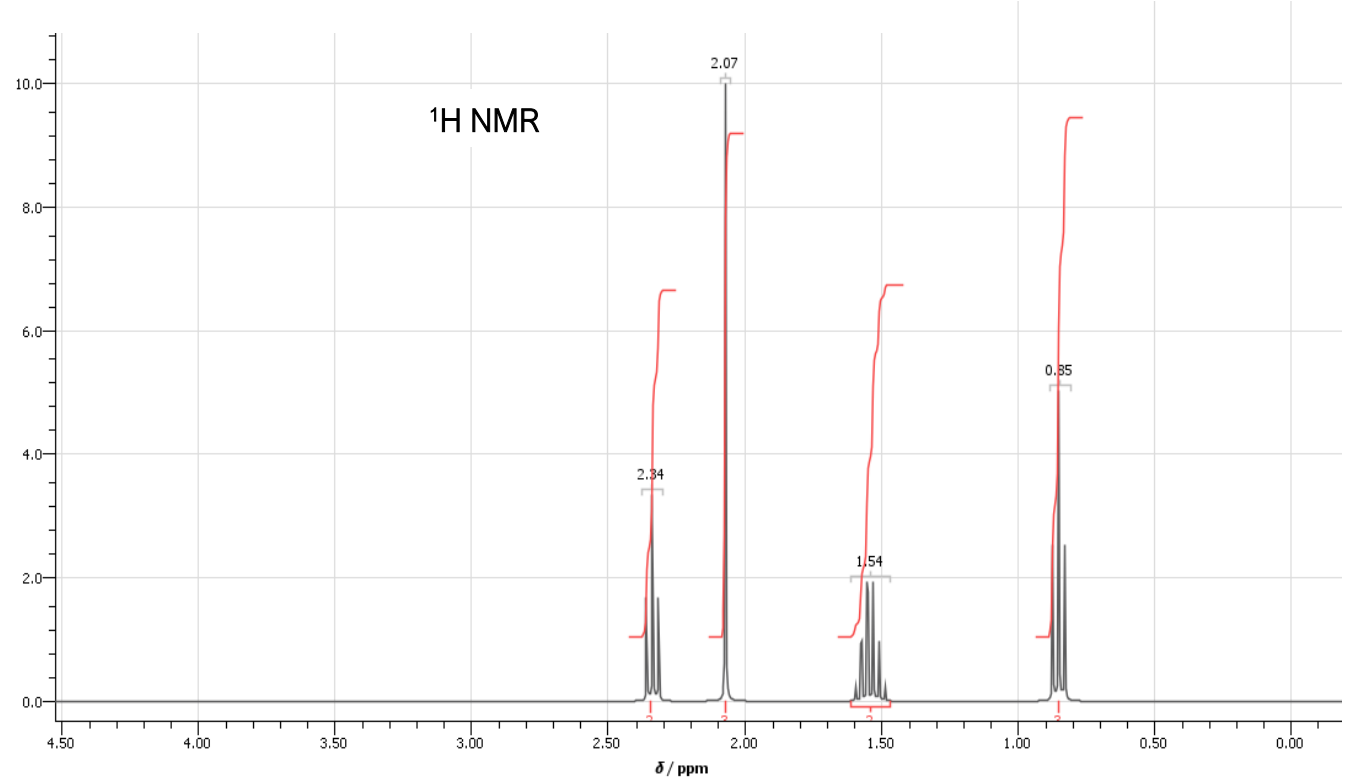
Compound reacts with Tollens' reagent to give a silver mirror.

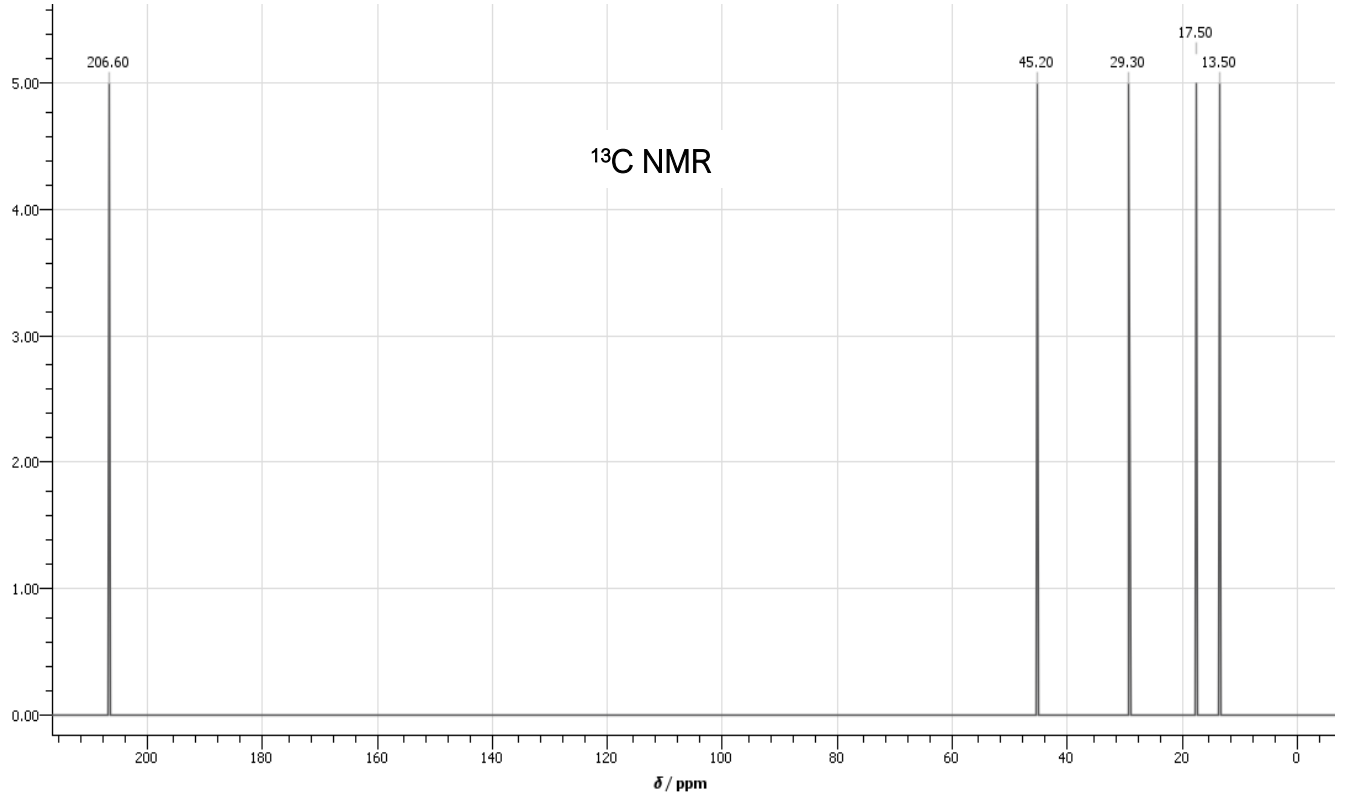
Compound 3

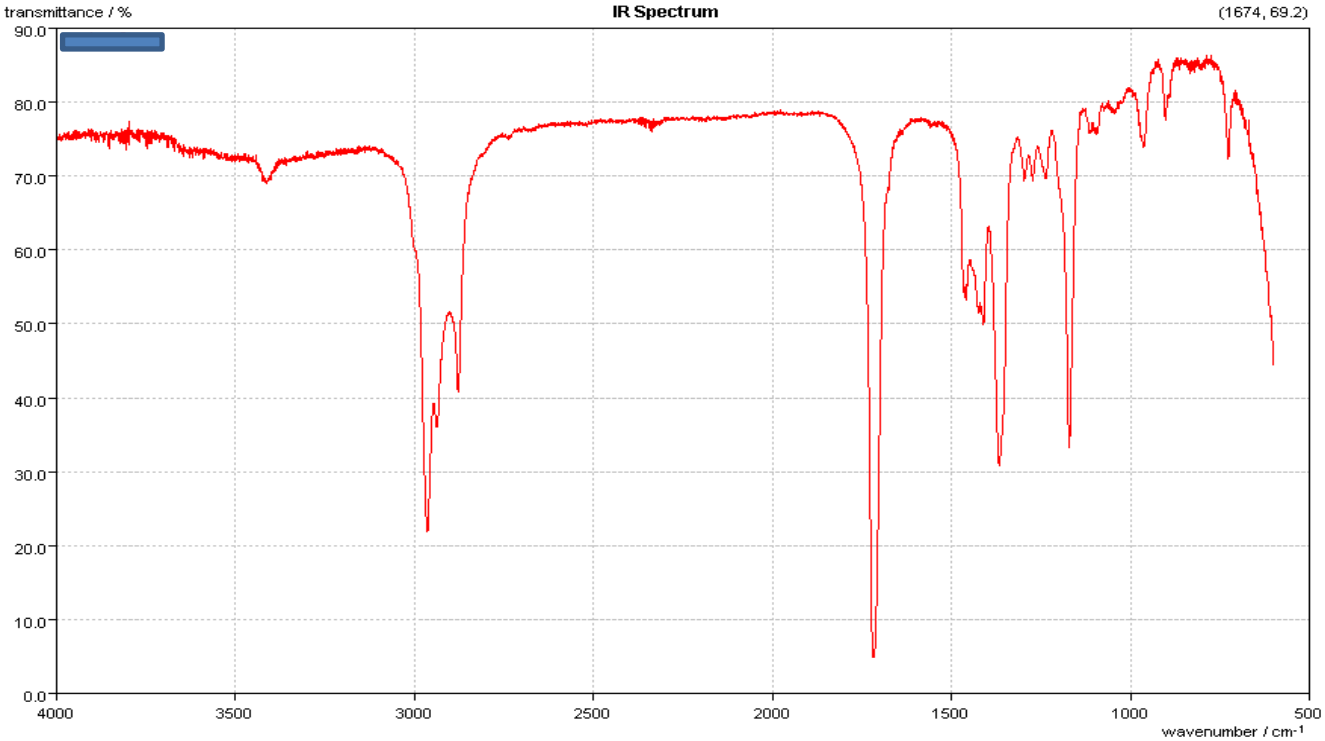
description: Colourless liquid

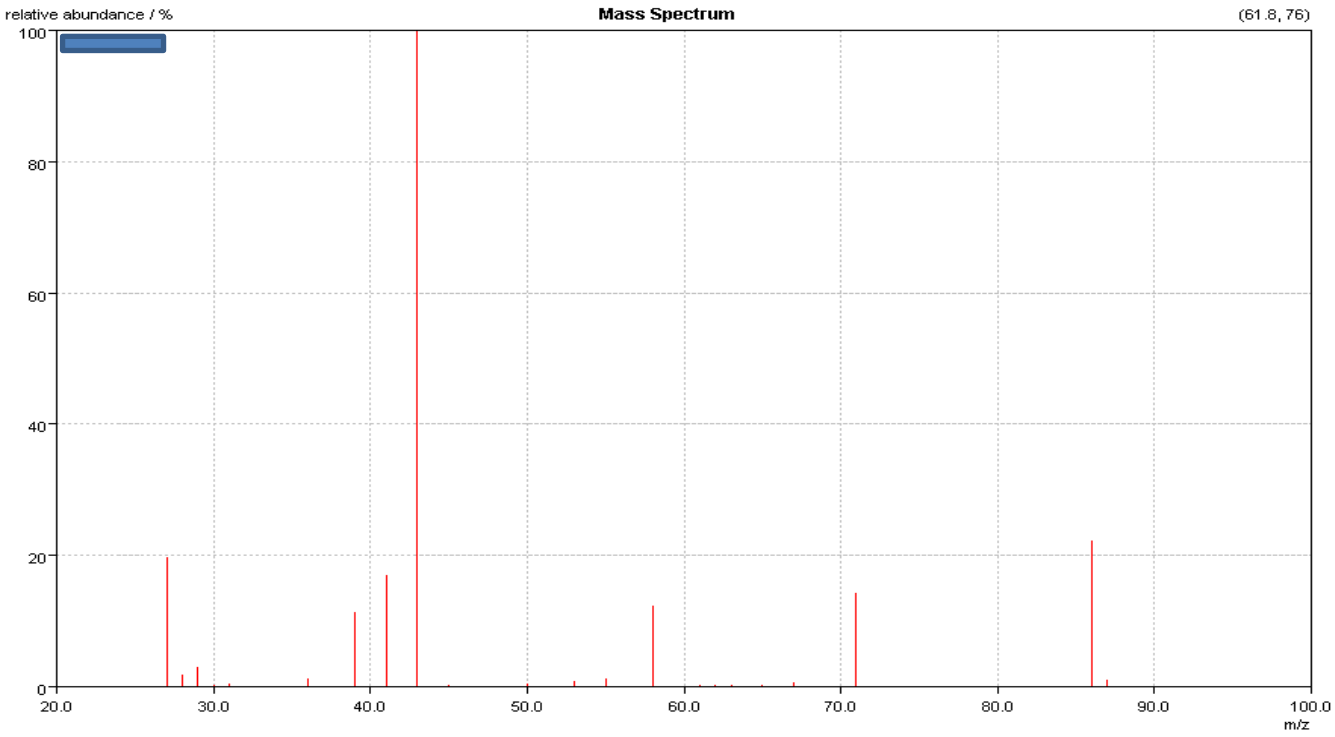
composition: C 69.76% H 11.62% O 18.60%

molecular formula: _____









Further Information: :

Compound reacts with 2,4-dinitrophenylhydrazine (Brady's Reagent) to give an intensely coloured precipitate.

Compound reacts readily with $NaBH_4$ to produce a secondary alcohol.

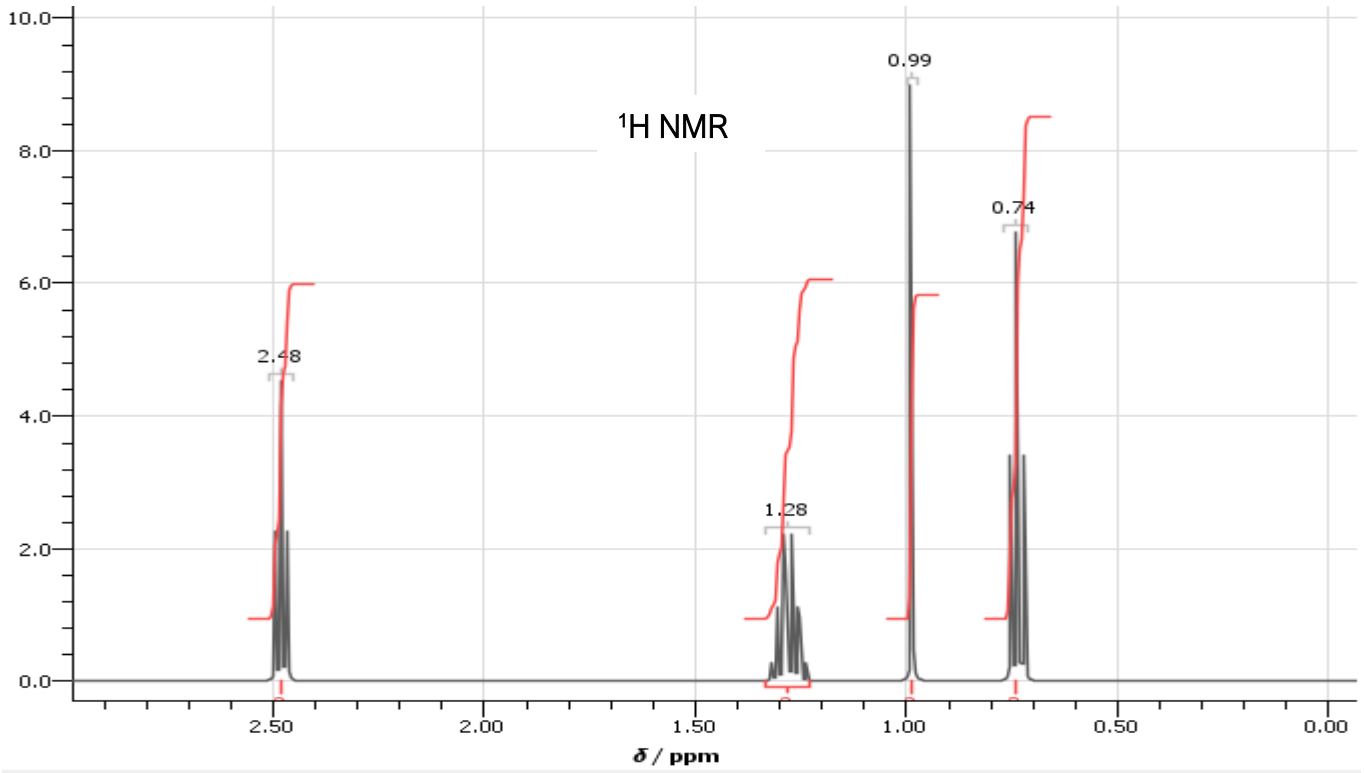
Compound does not react with Tollens' reagent.

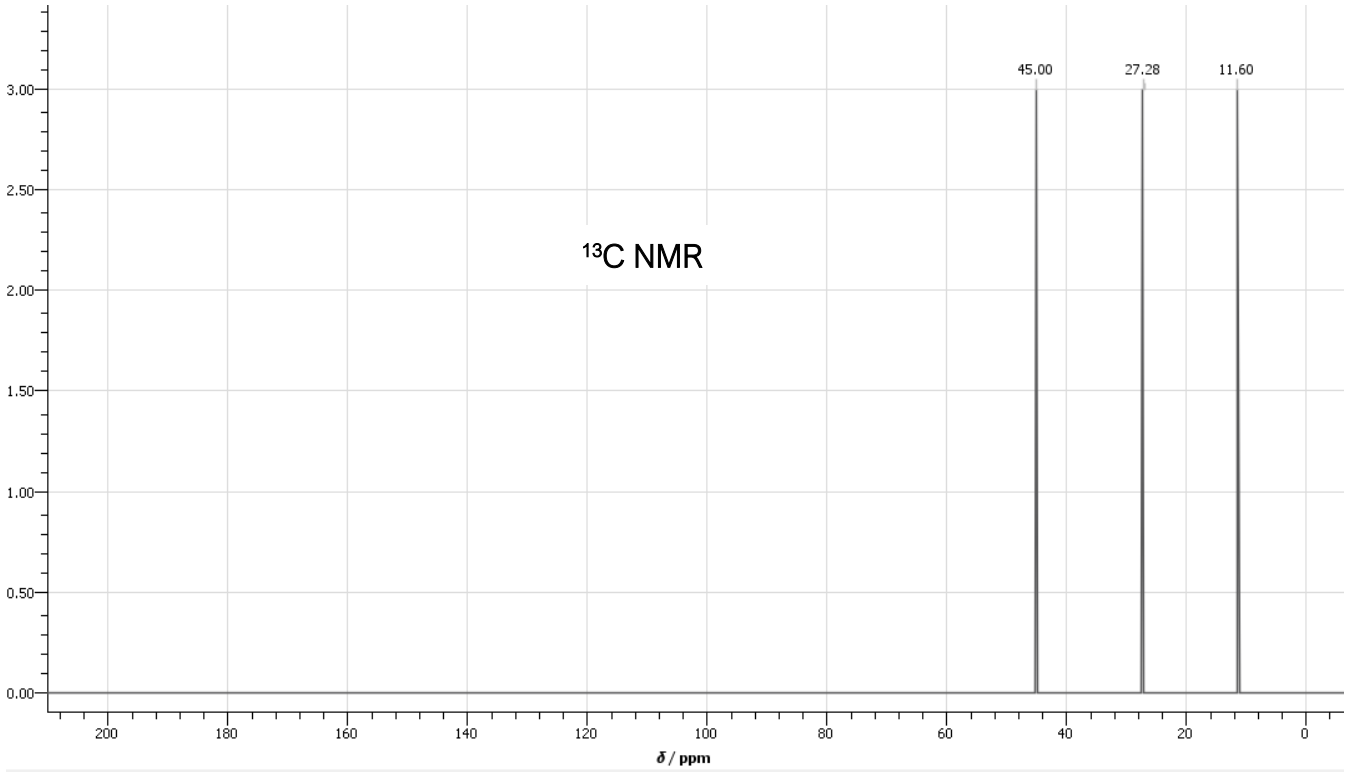
Compound 4

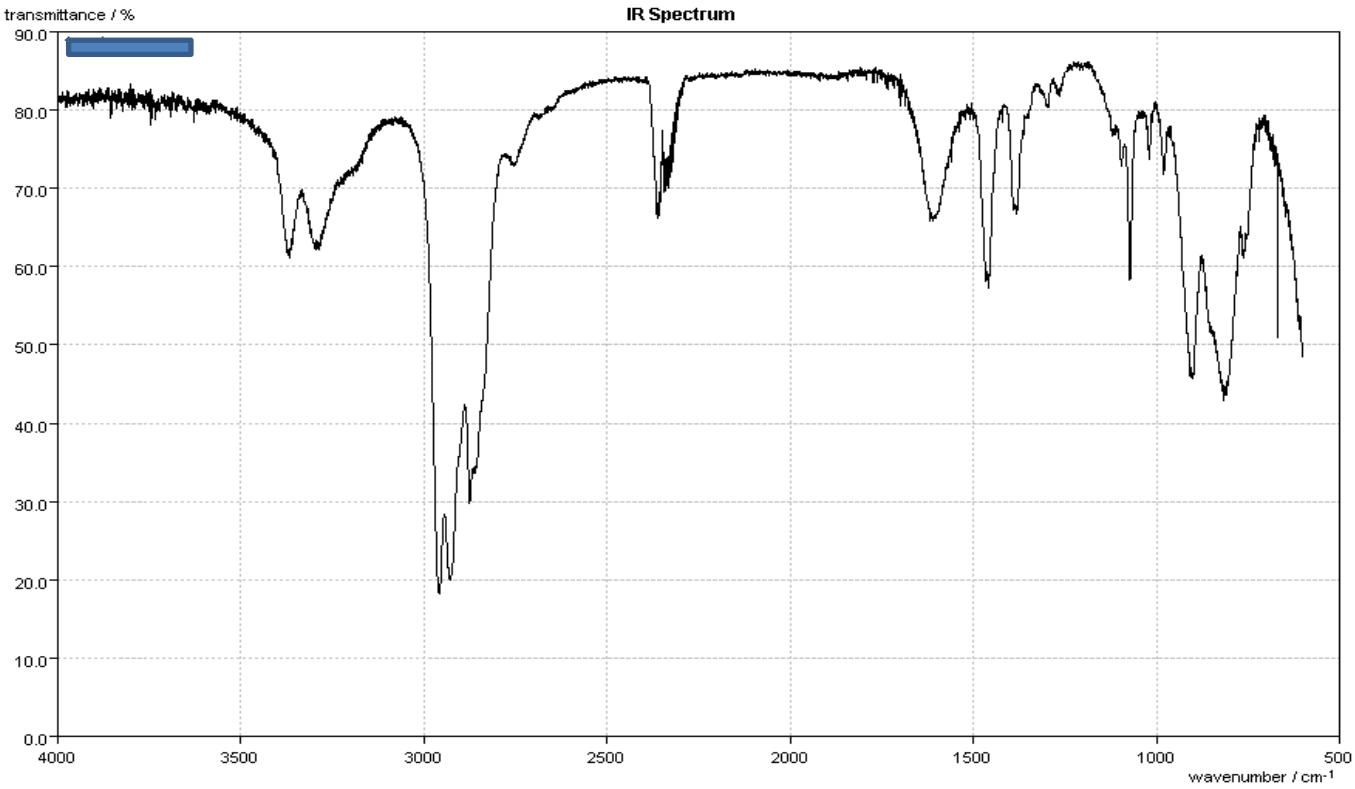
description: Colourless liquid

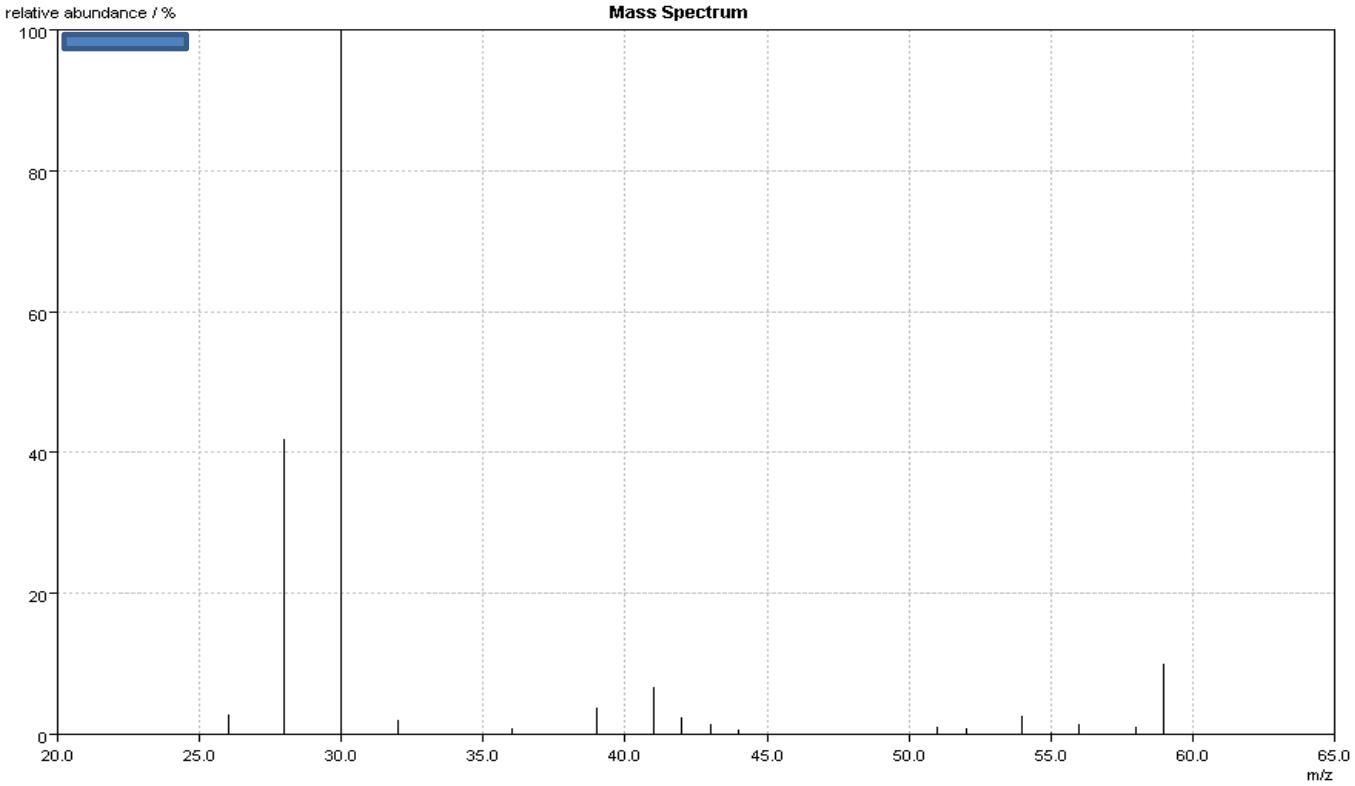
composition: C 61.02%; H 15.25%; N 23.73%

molecular formula: _____









Further Information:

Compound has a fishy smell.

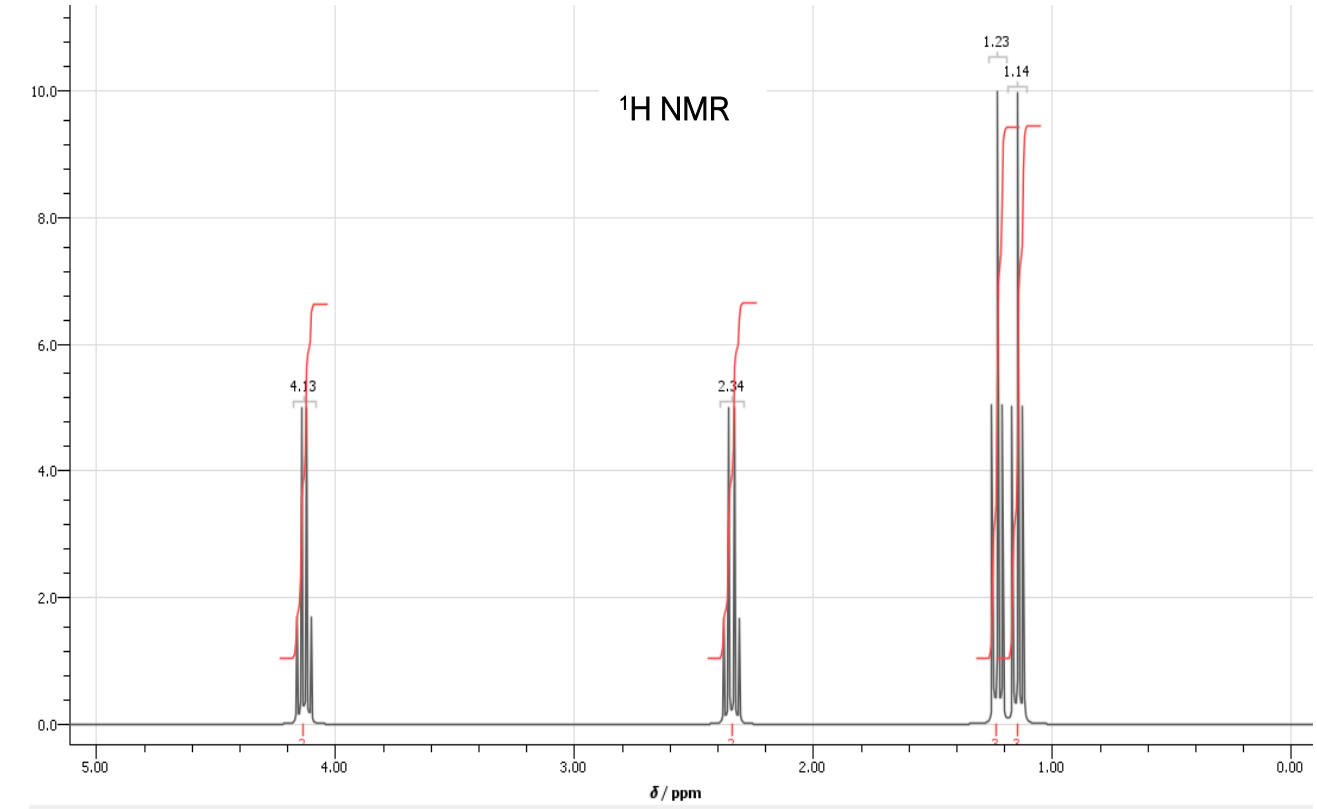
Compound dissolves in water to form an alkaline solution and reacts with hydrochloric acid to form a salt.

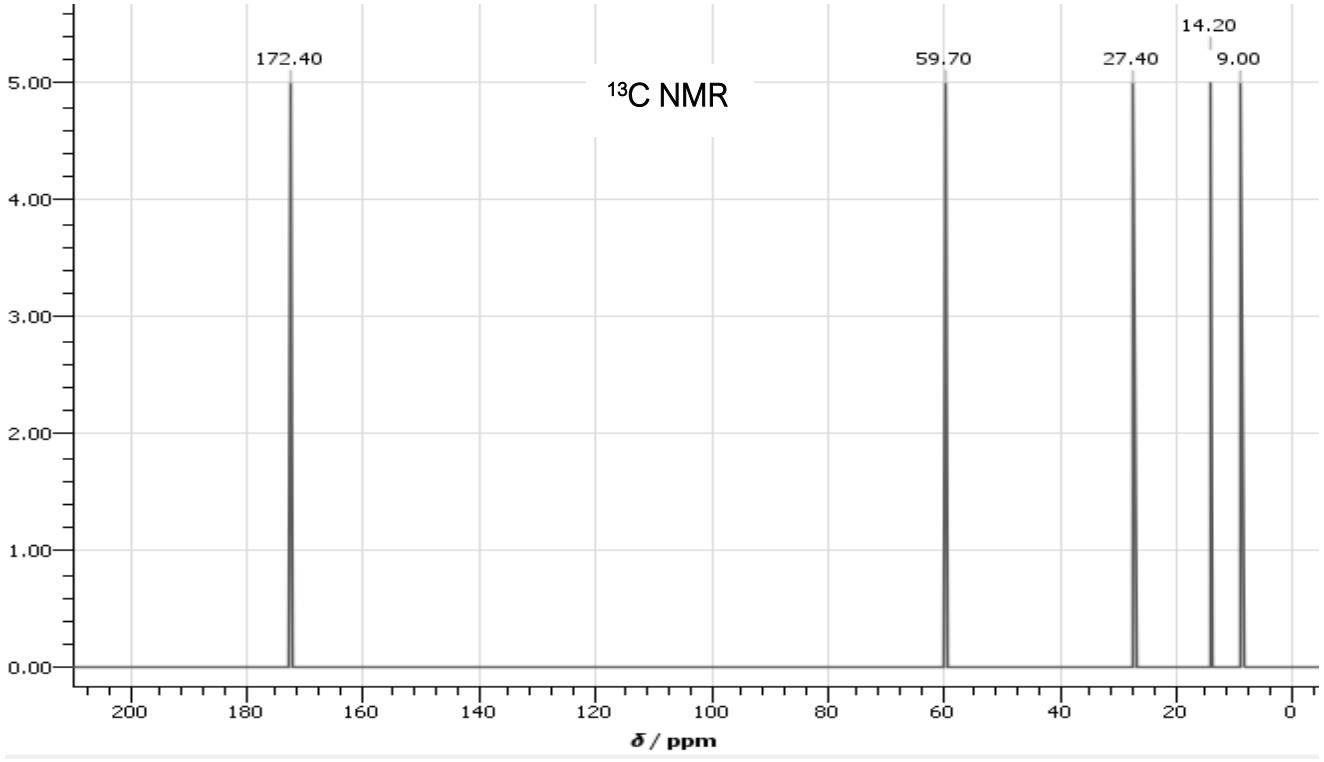
Compound 5

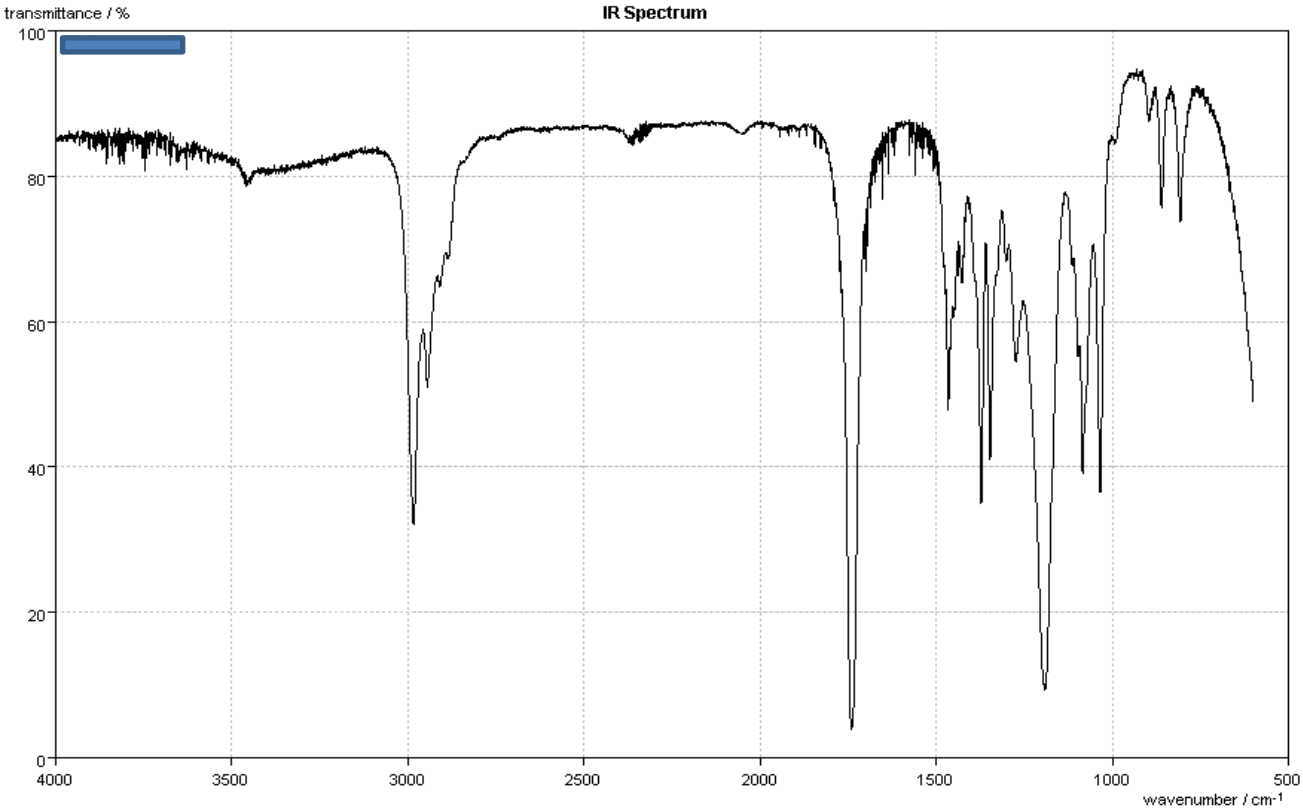
description: Colourless liquid

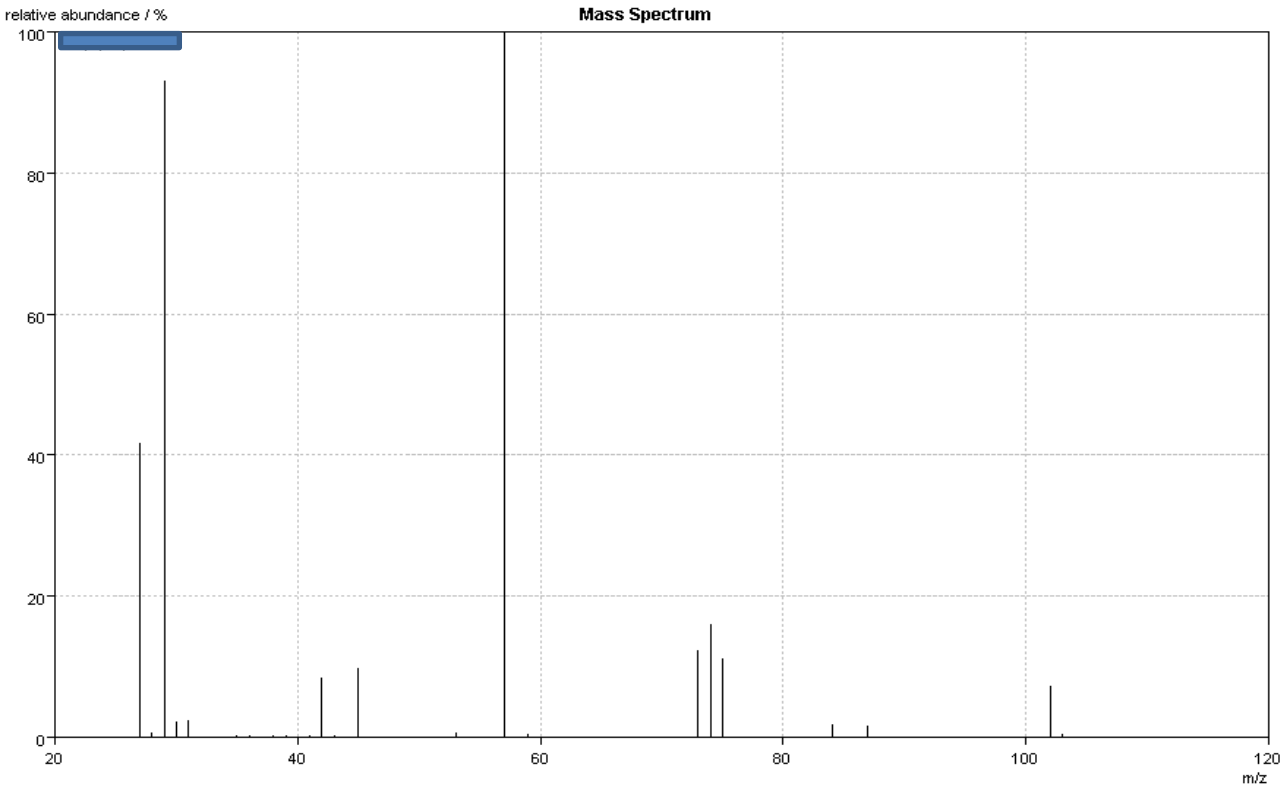
composition: C 58.80% H 9.87% O 31.33%

molecular formula: _____









Further Information:

Compound has a fruity smell.

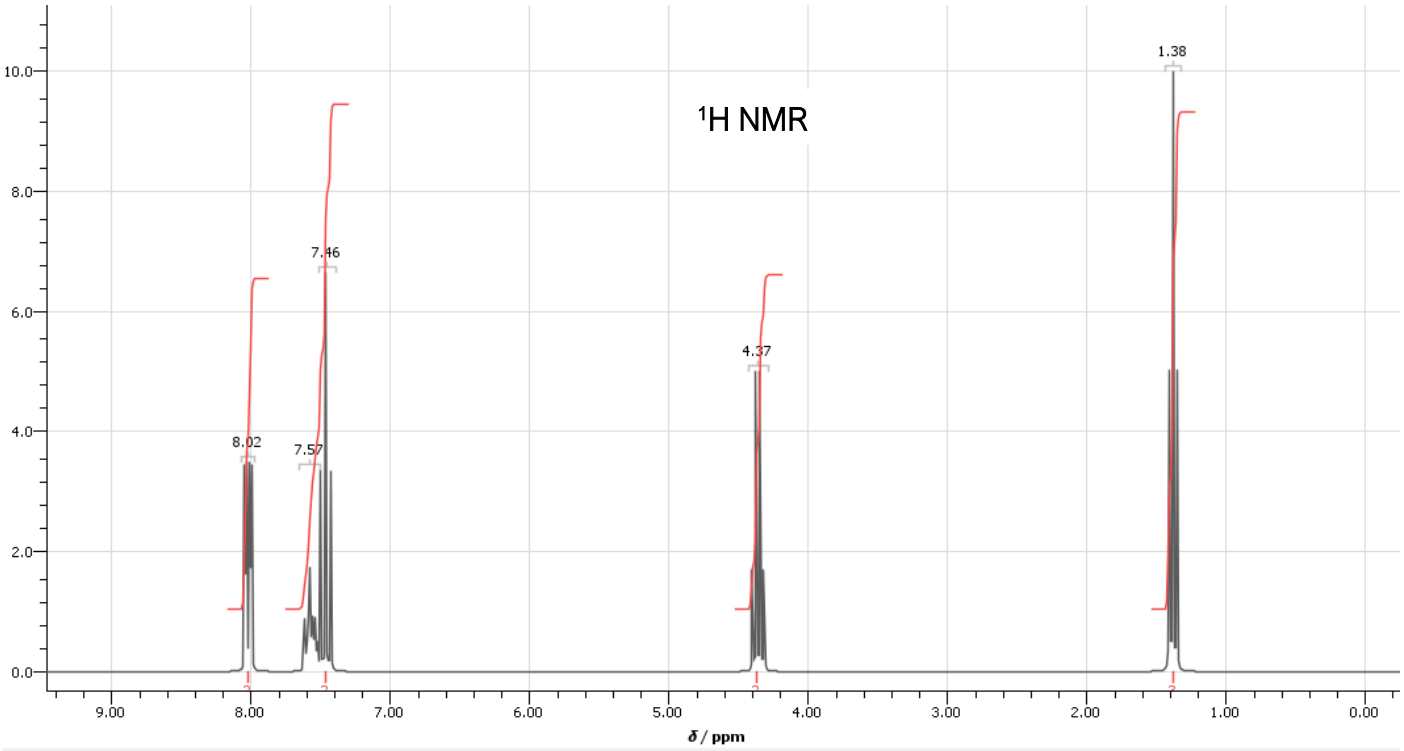
Compound hydrolyses in aqueous hydroxide to give sodium propanoate as one of two products.

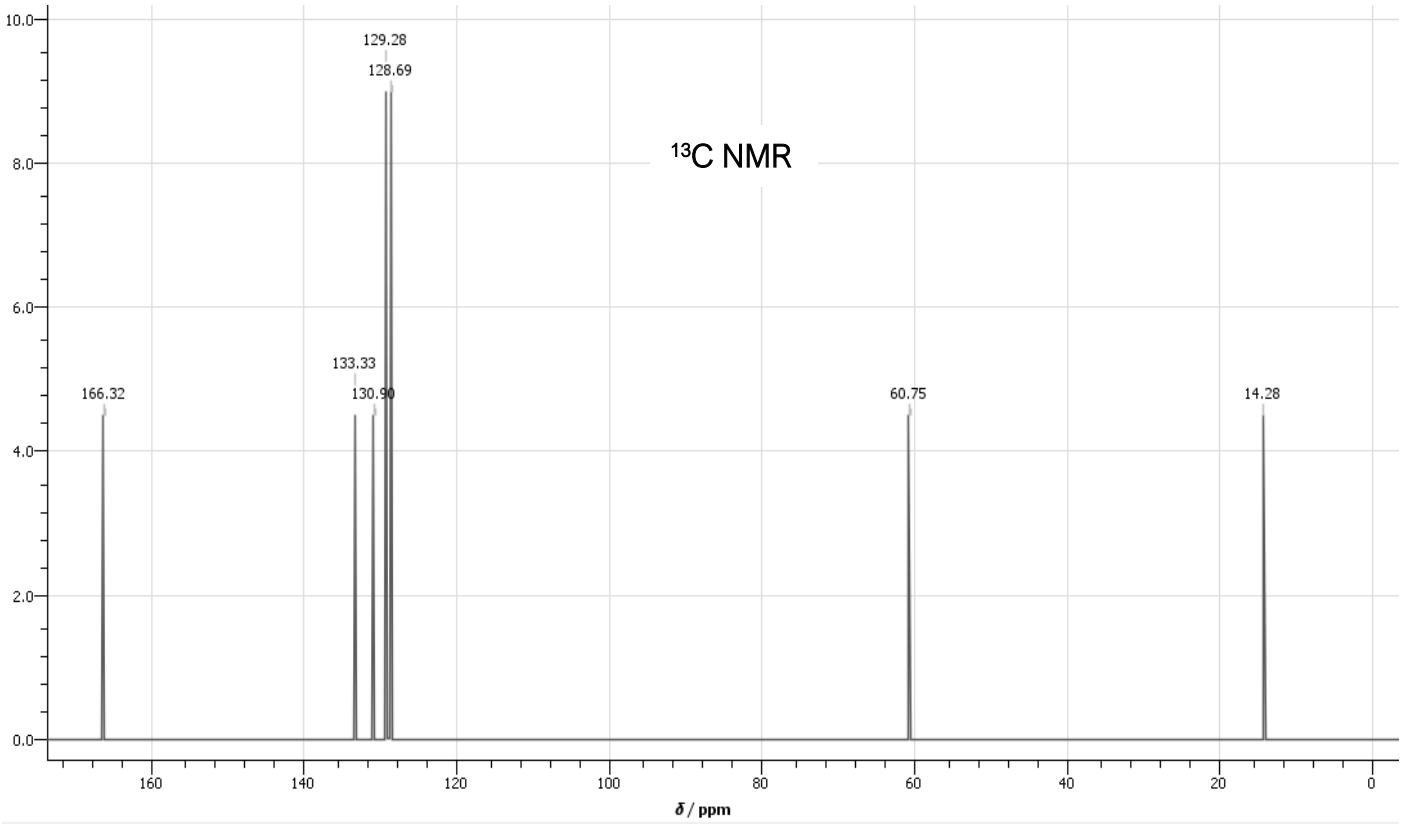
Compound 6

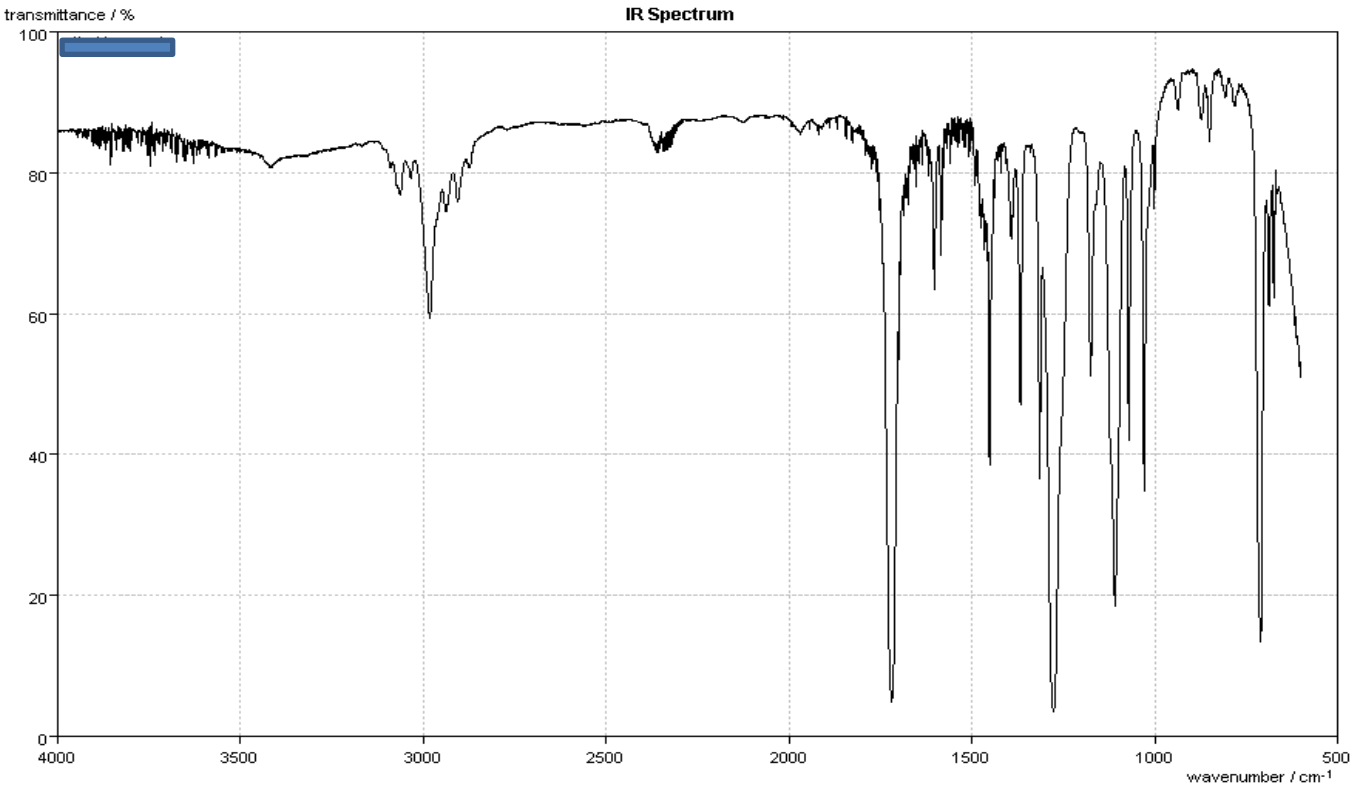
description: Colourless liquid

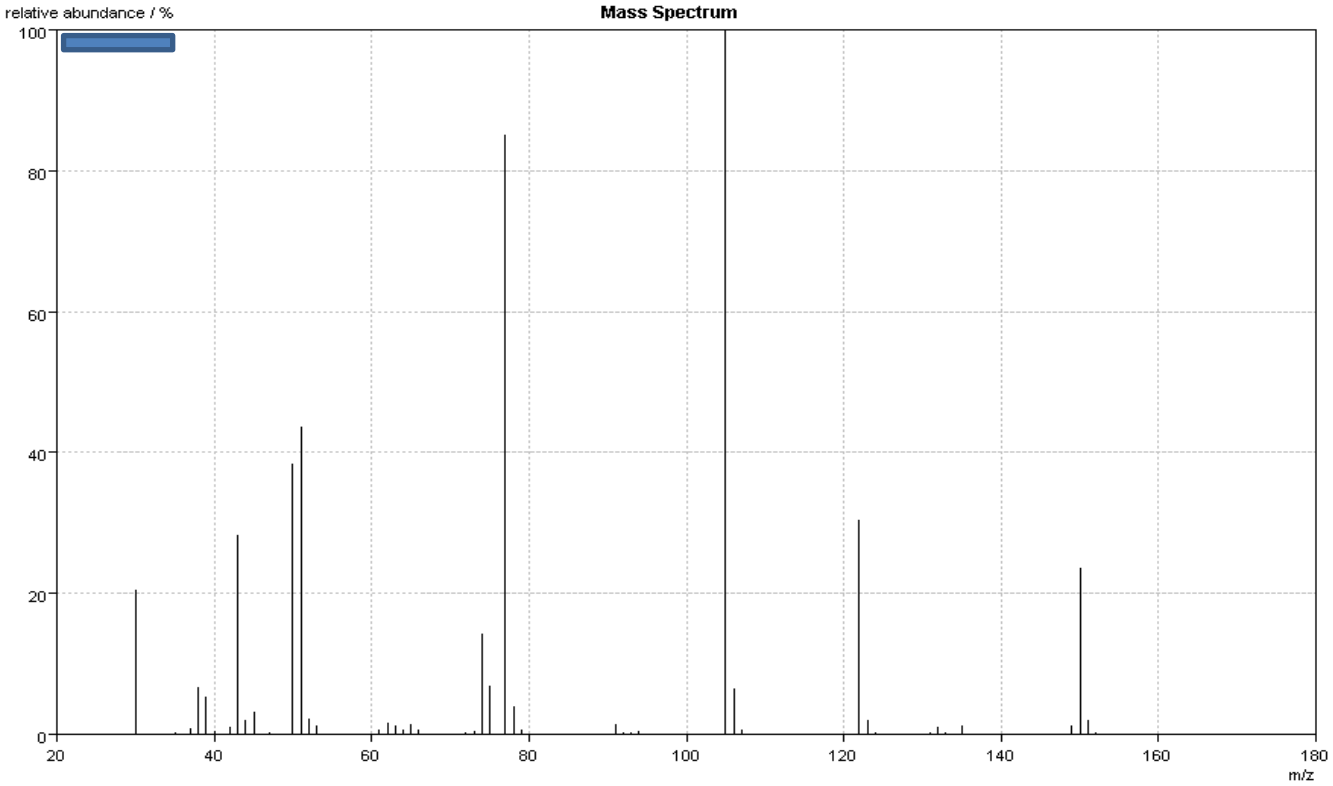
composition: C 71.98% H 6.71% O 21.31%

molecular formula: _____









Further Information:

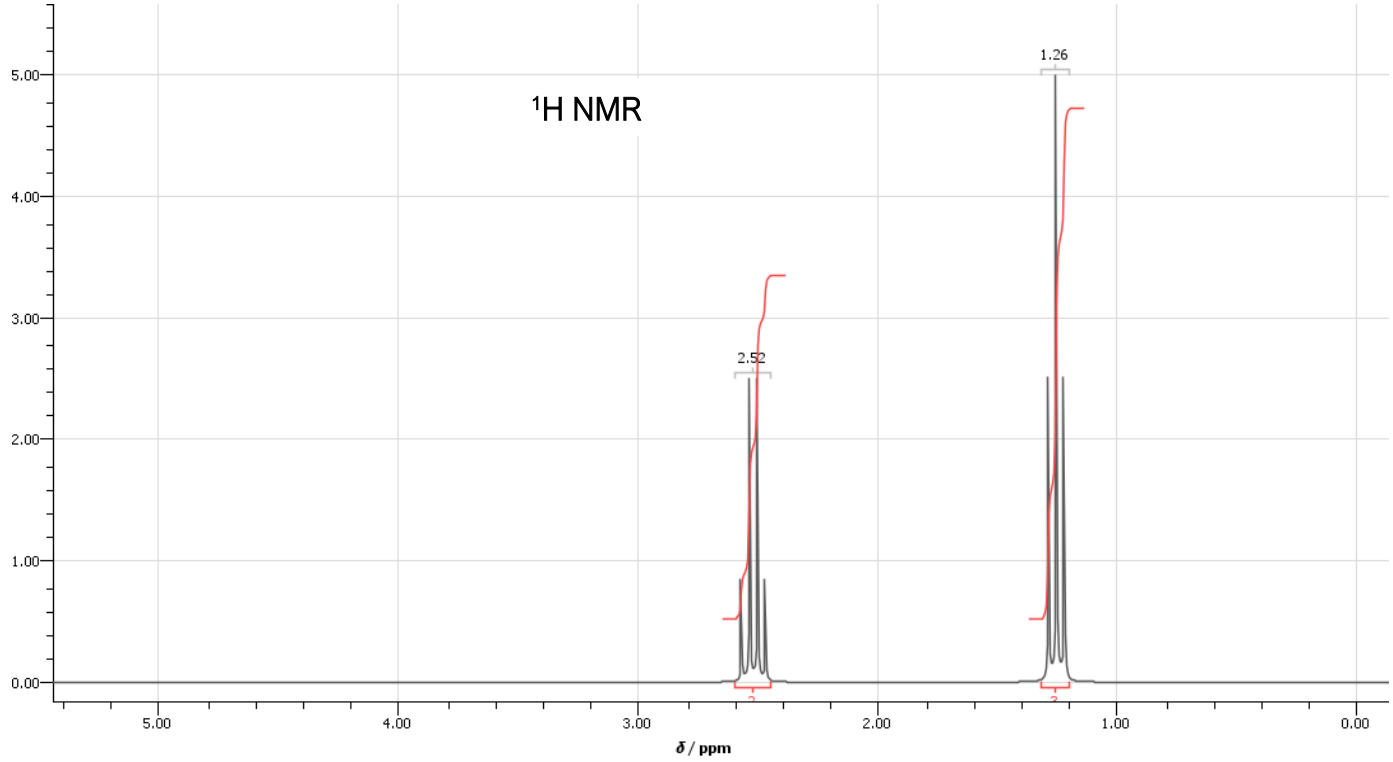
Compound hydrolyses in aqueous hydrochloric acid to give ethanol as one of two products.

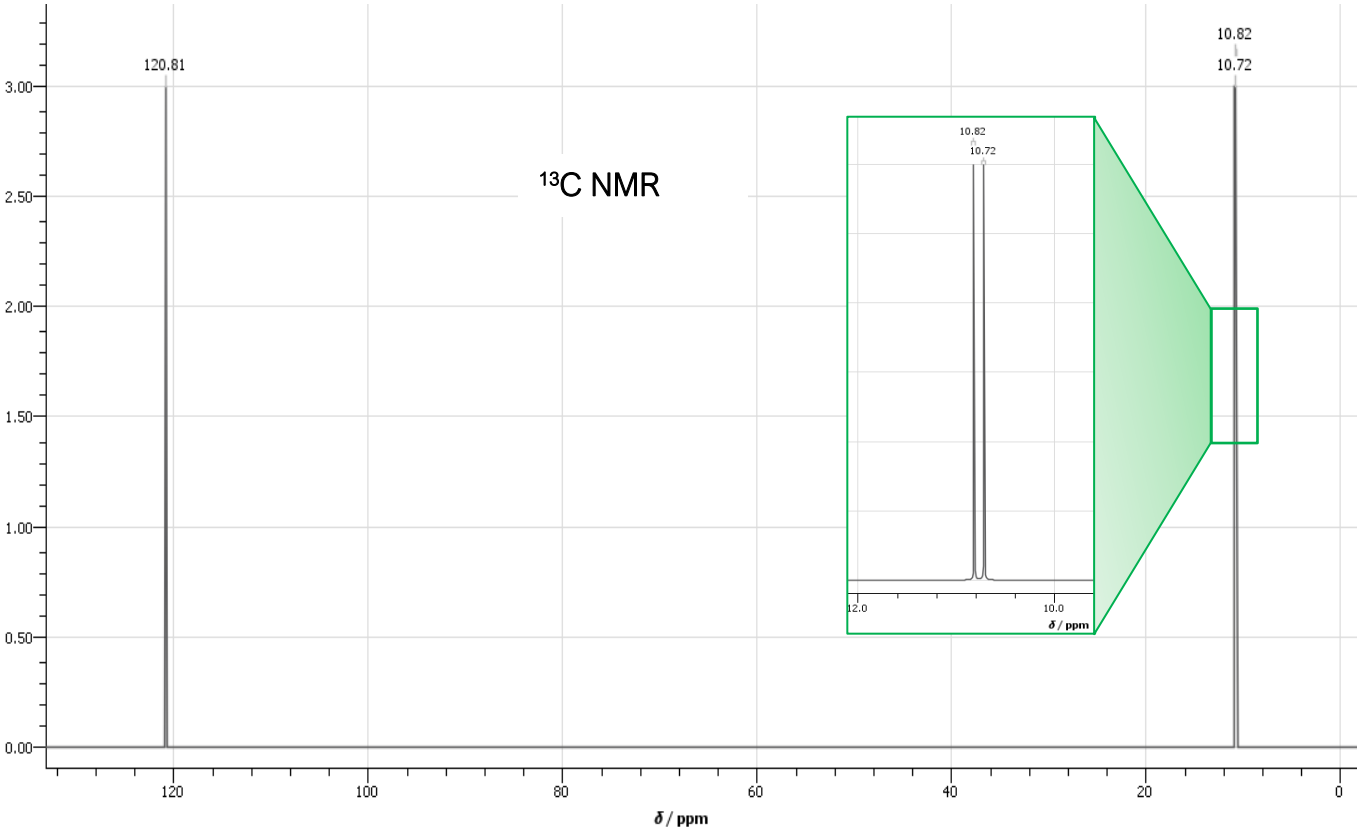
Compound 7

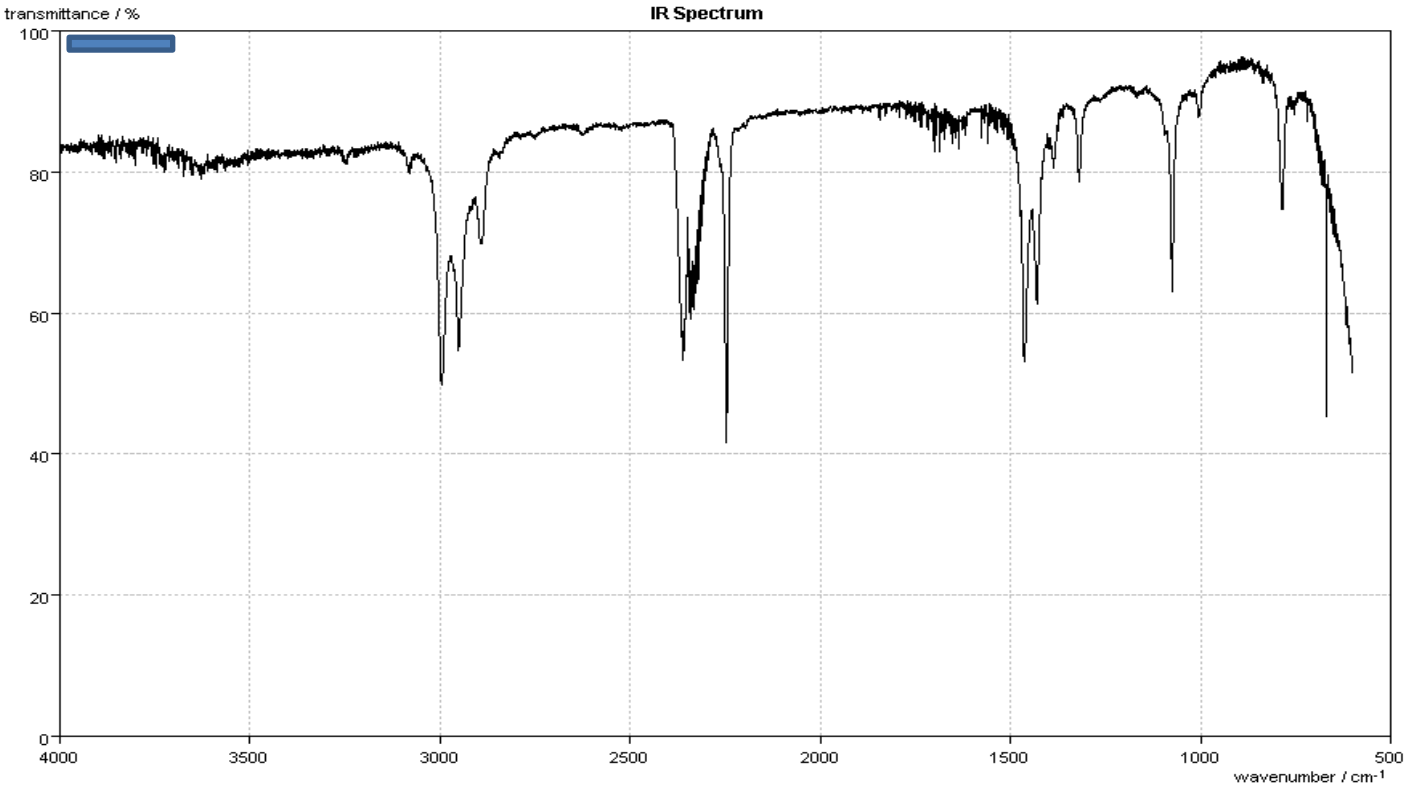
description: Colourless liquid

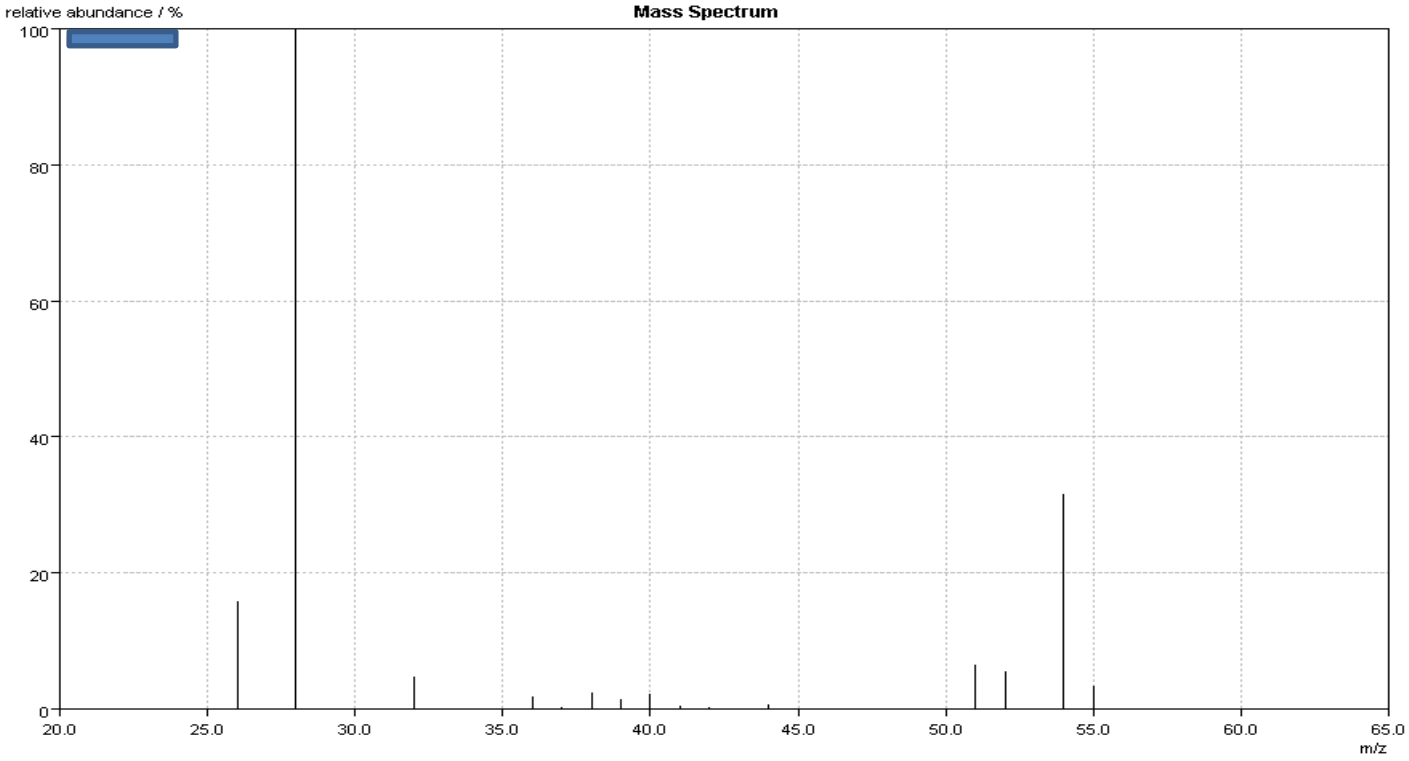
composition: C 65.42%; H 9.15%; N 25.43%

molecular formula: _____









Further Information:

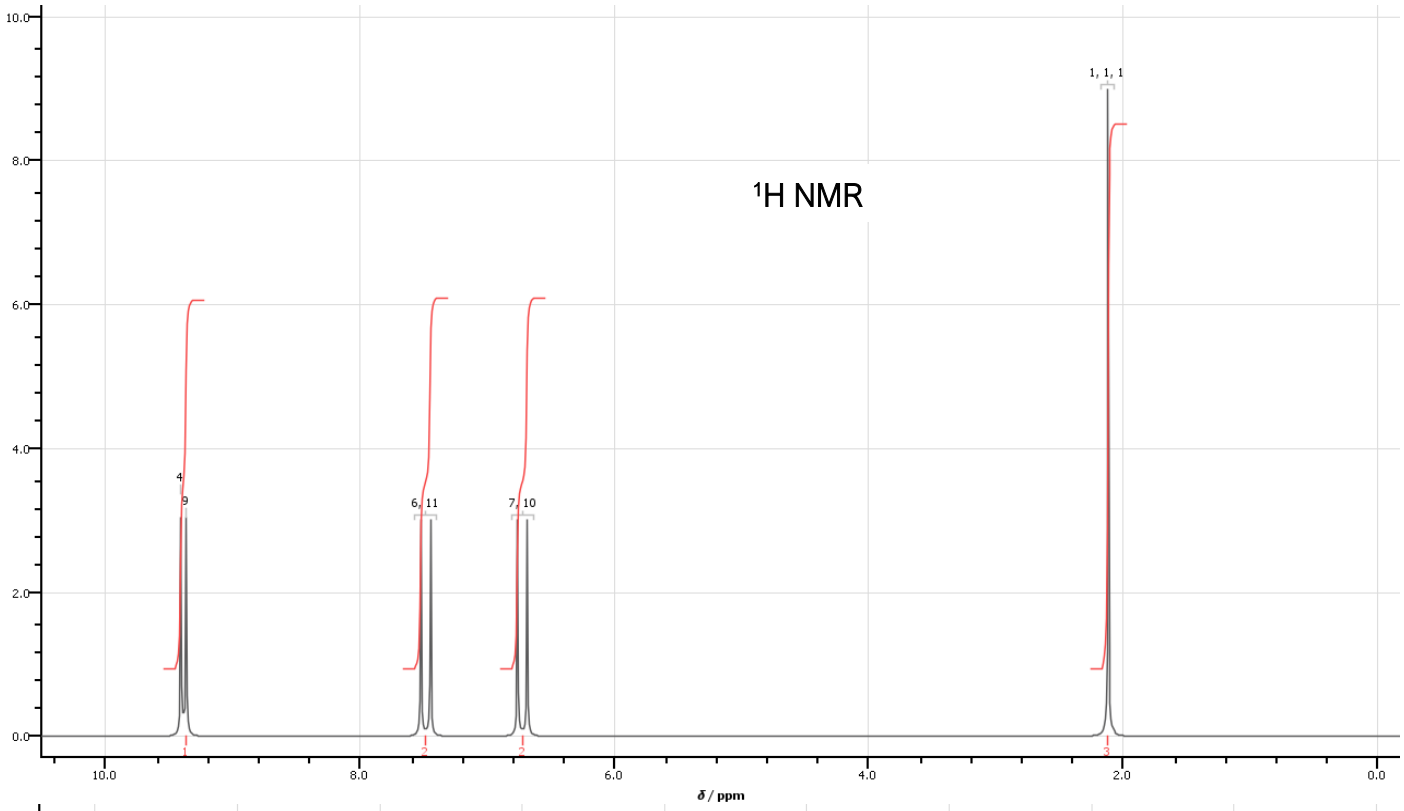
Compound dissolves in water to give a neutral, mildly poisonous solution.

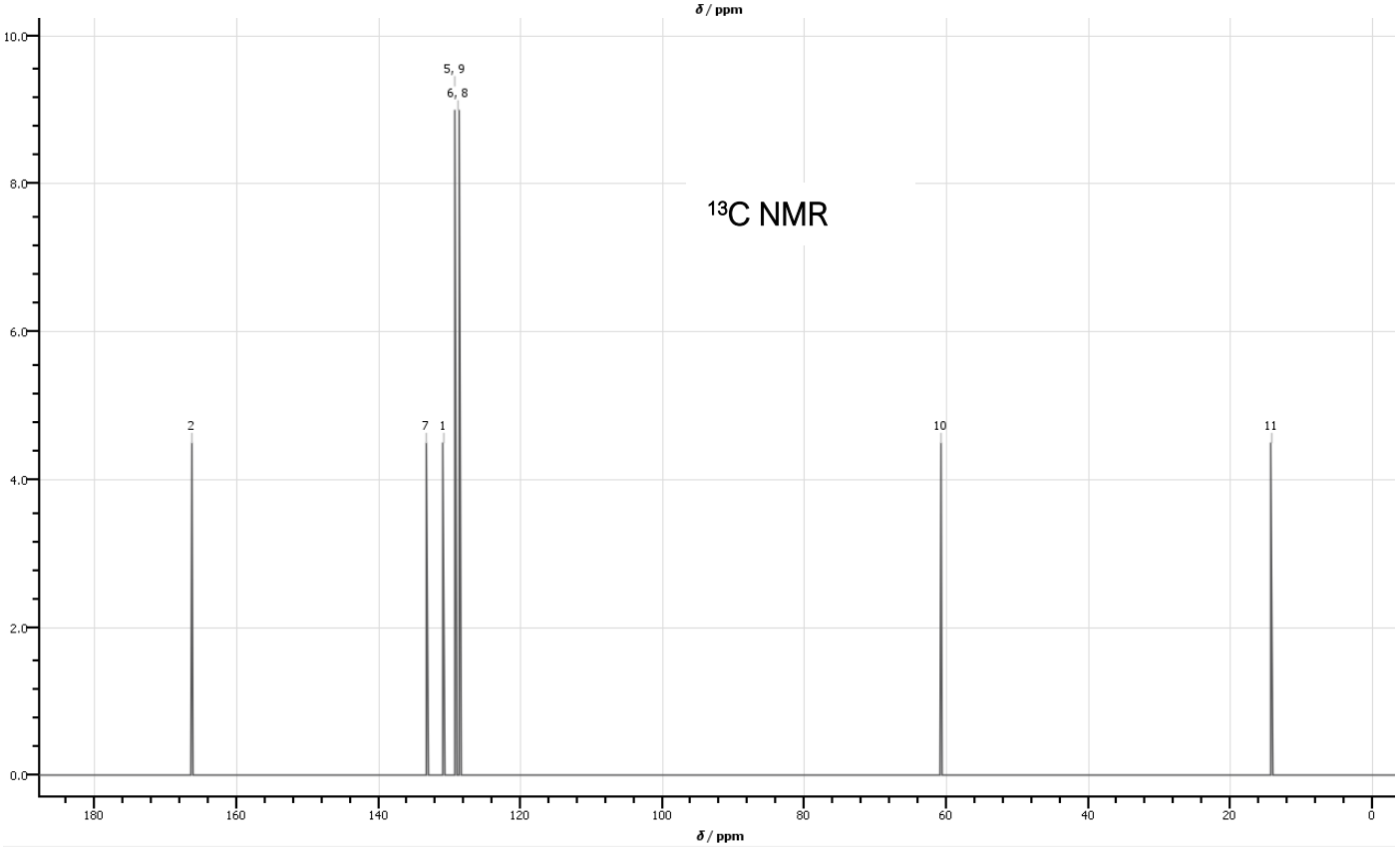
Compound 8

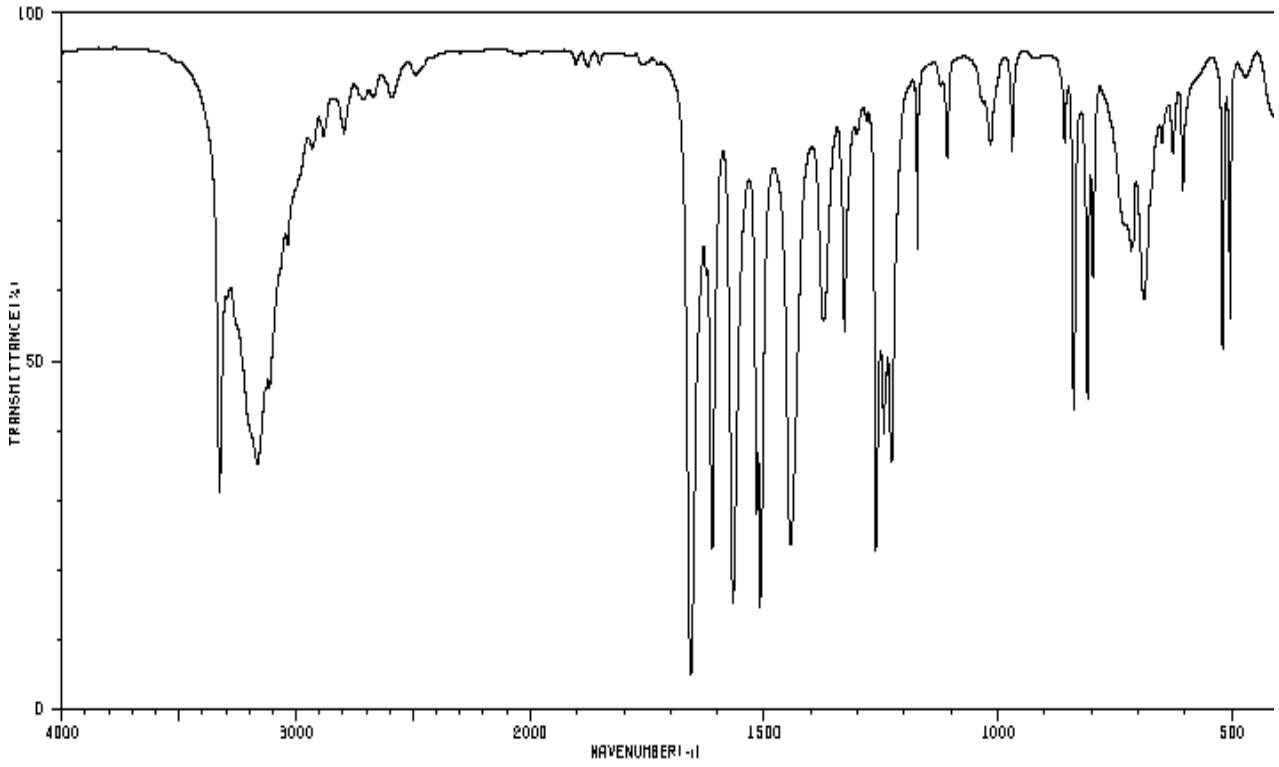
description: White crystalline solid

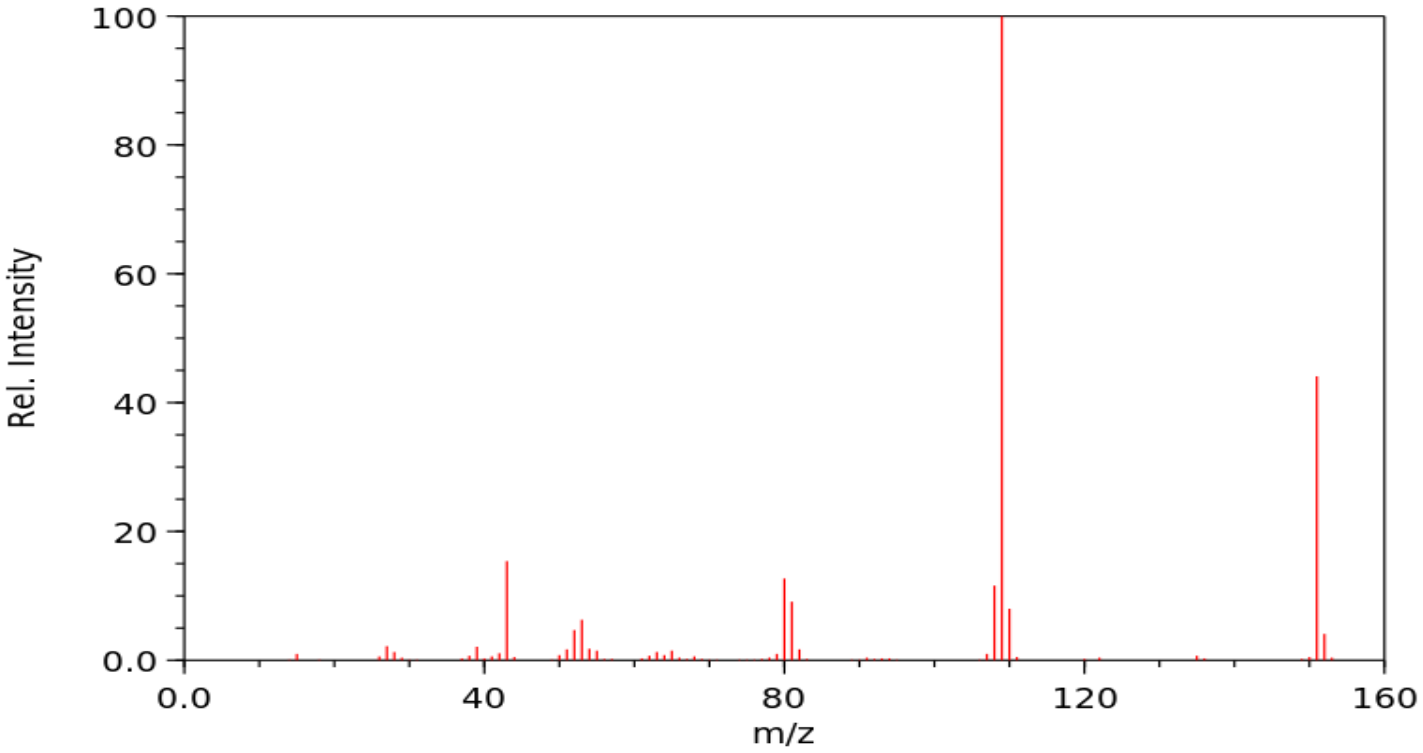
composition: C 63.56%; H 6.00%; N 9.27%; O 21.17%

molecular formula: _____









Further Information:

Compound can be hydrolysed in boiling hydrochloric acid or sodium hydroxide solution to produce, ethanoic acid or sodium ethanoate respectively, as one of the products

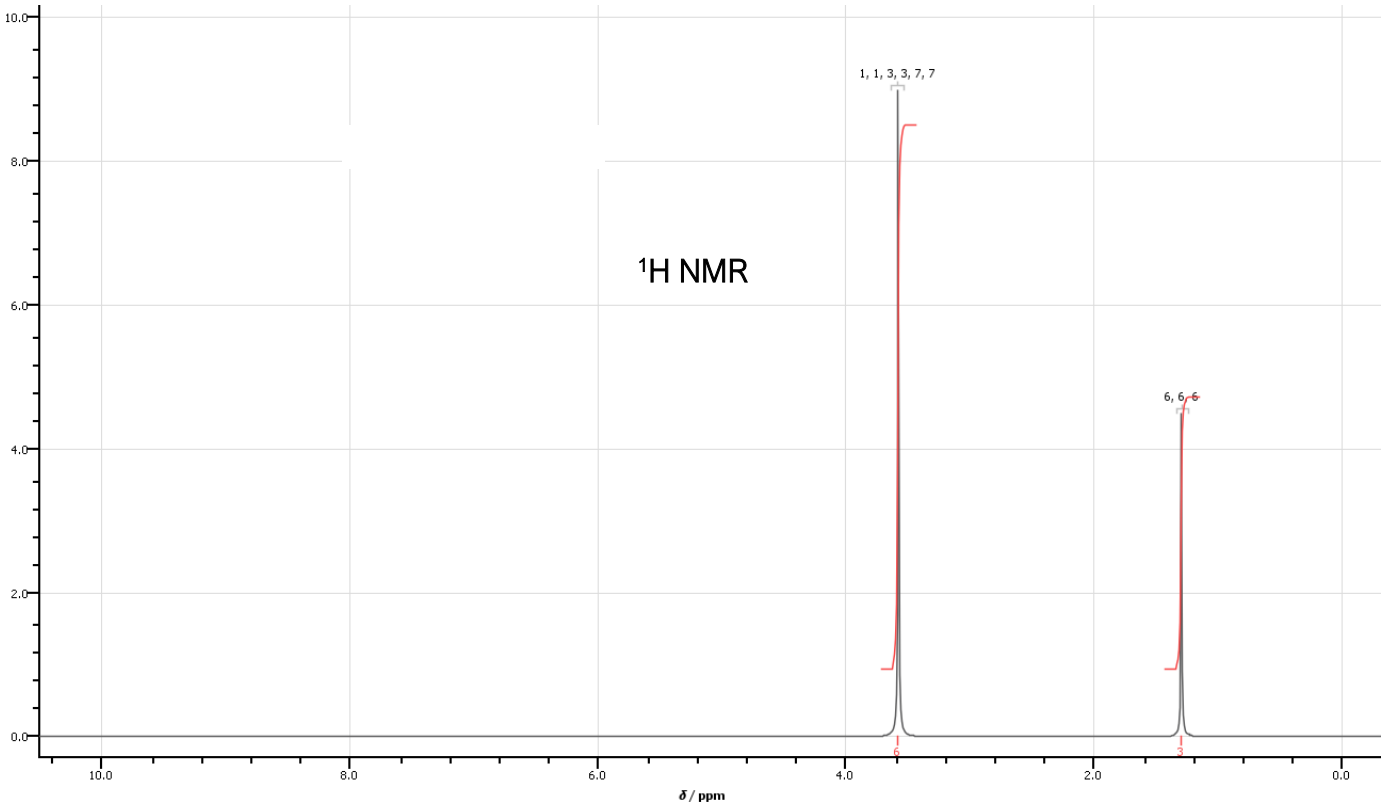
Compound is a pain killer and anti-pyretic. It is also moderately hepatotoxic.

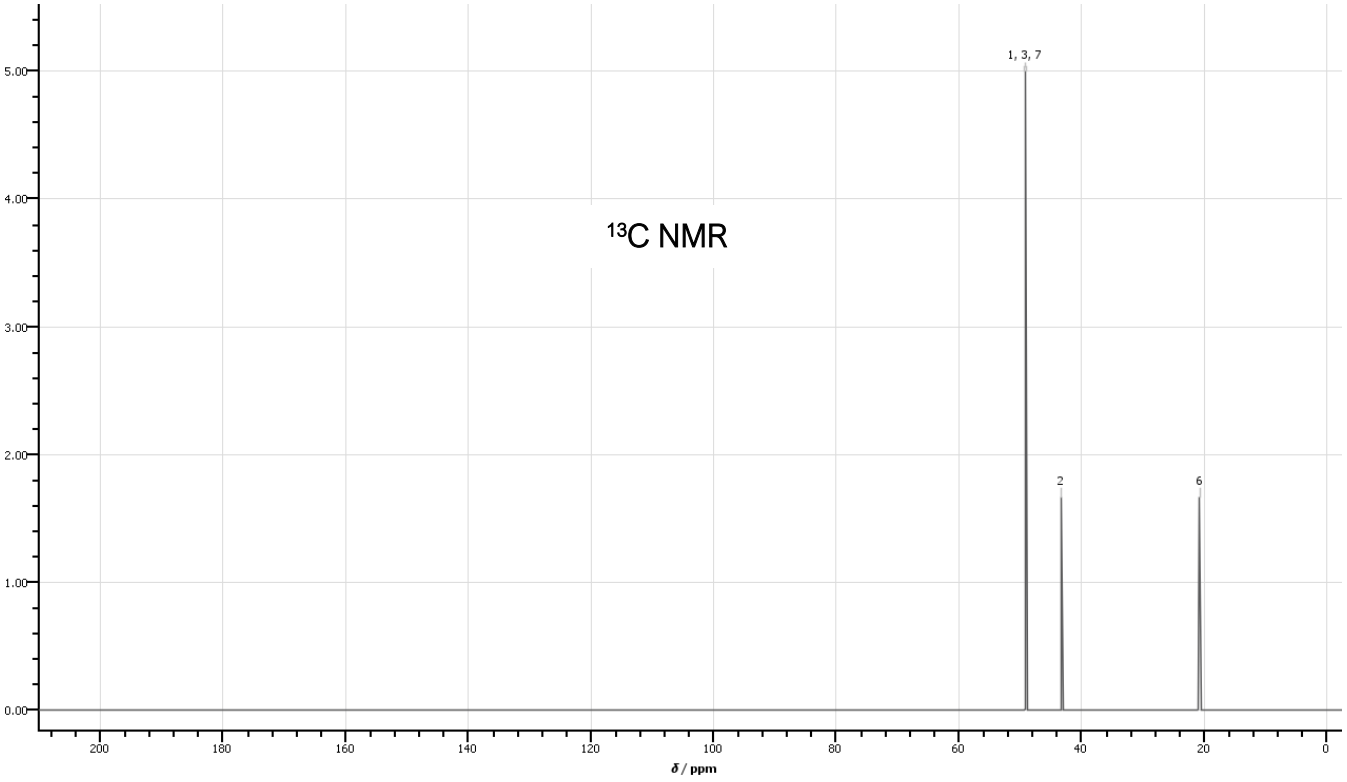
Compound 9

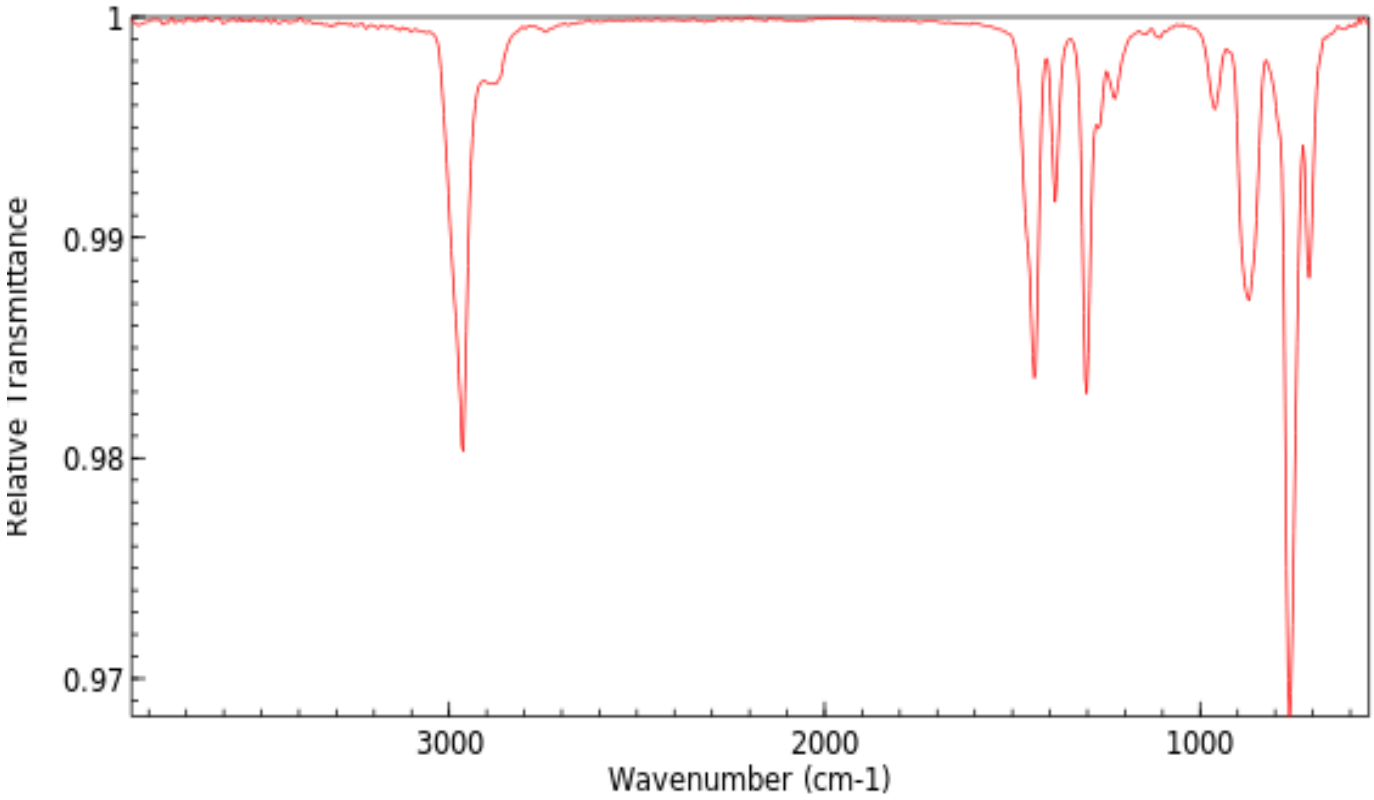
description: colourless volatile liquid

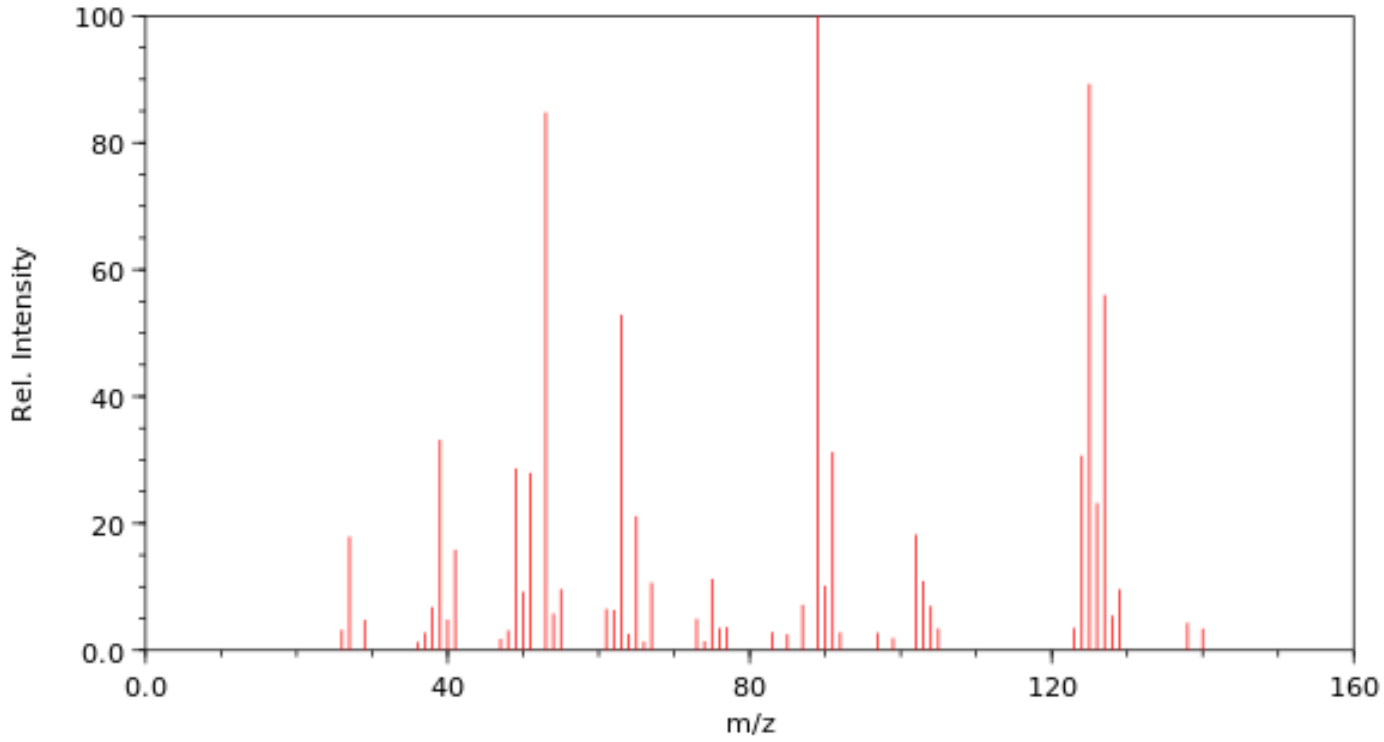
composition: C 34.22%; H 5.17%; Cl 60.61%

molecular formula: _____









Further Information:

Compound is immiscible (insoluble) in water.

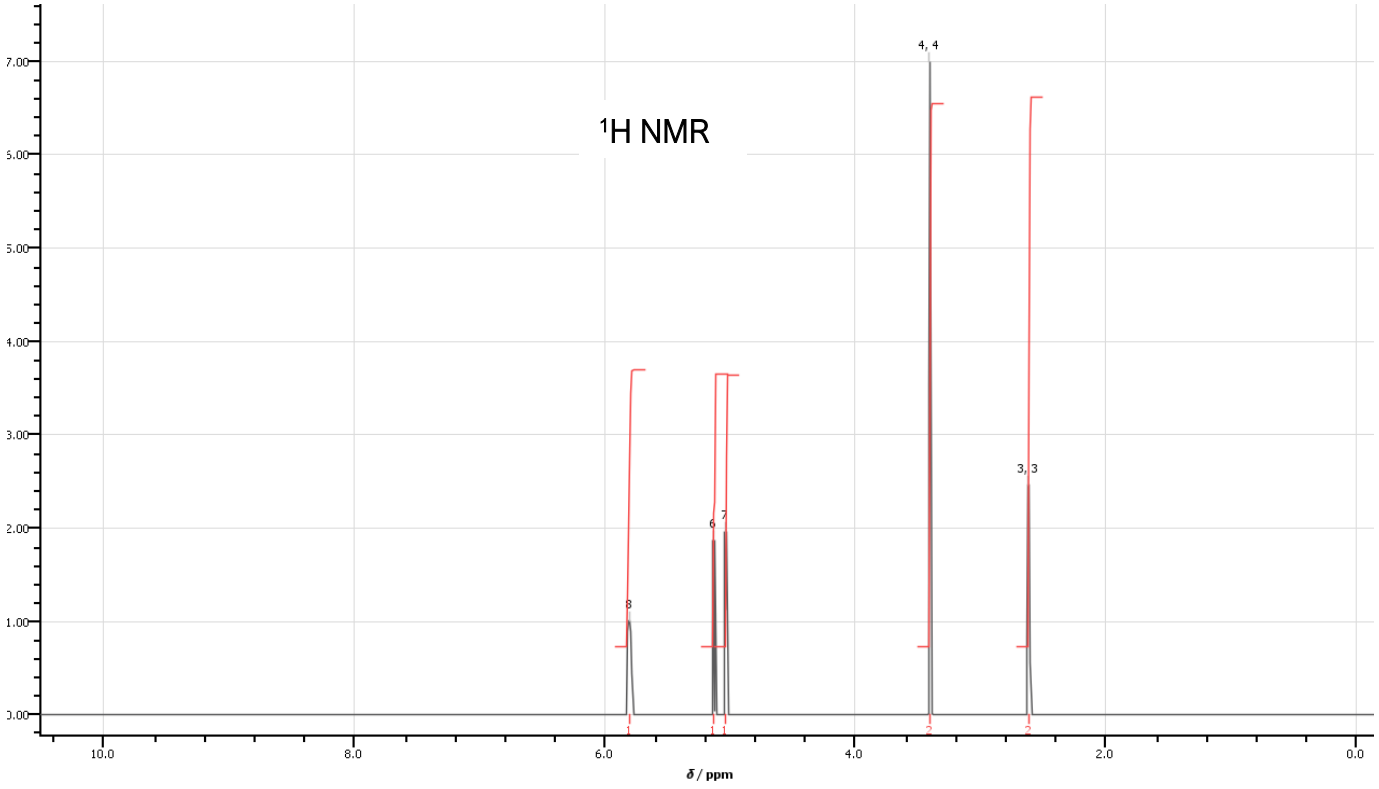
Compound produces a white precipitate when heated in a mixture of sodium hydroxide solution, ethanol and silver nitrate.

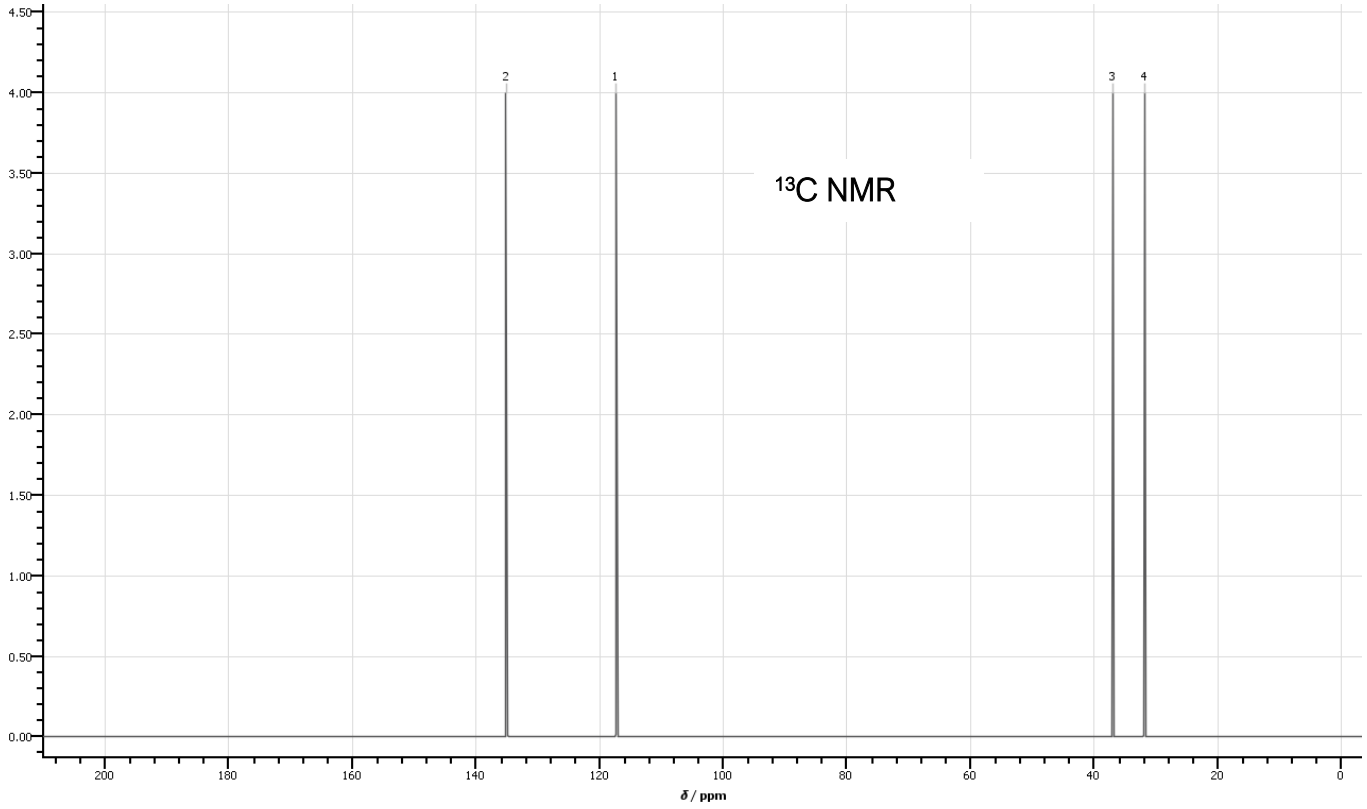
Compound 10

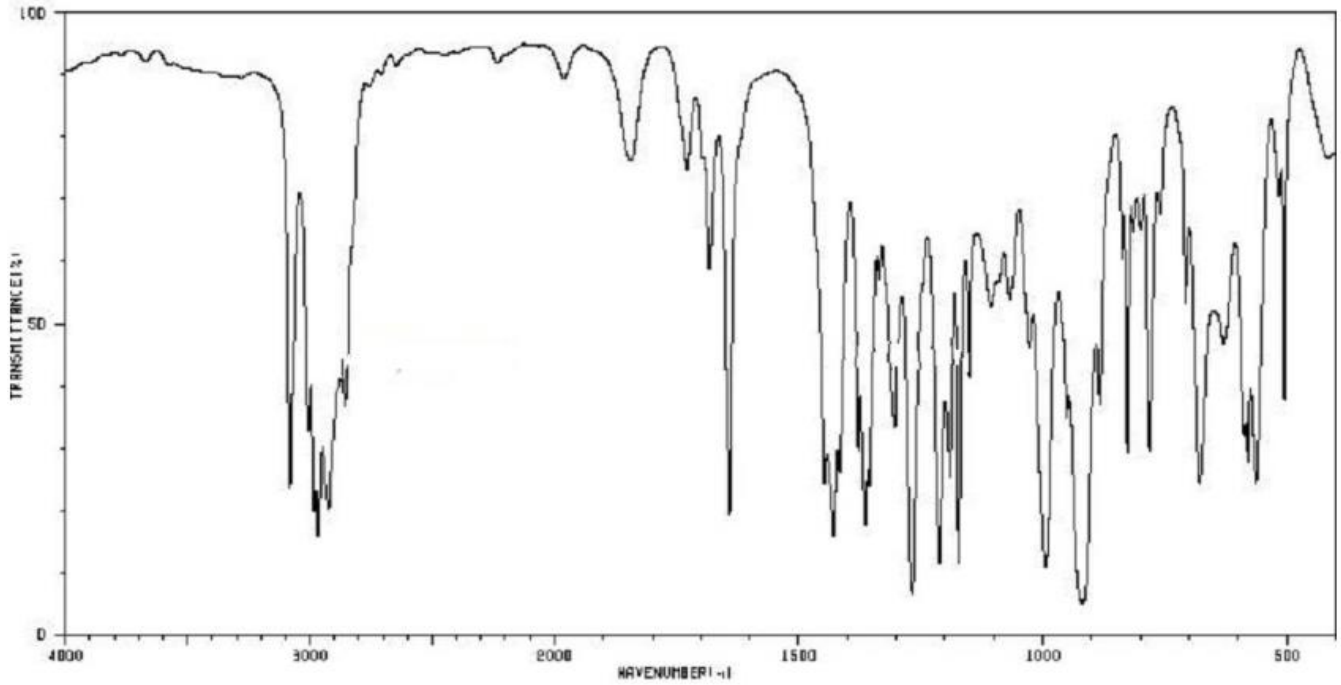
description: Colourless volatile liquid

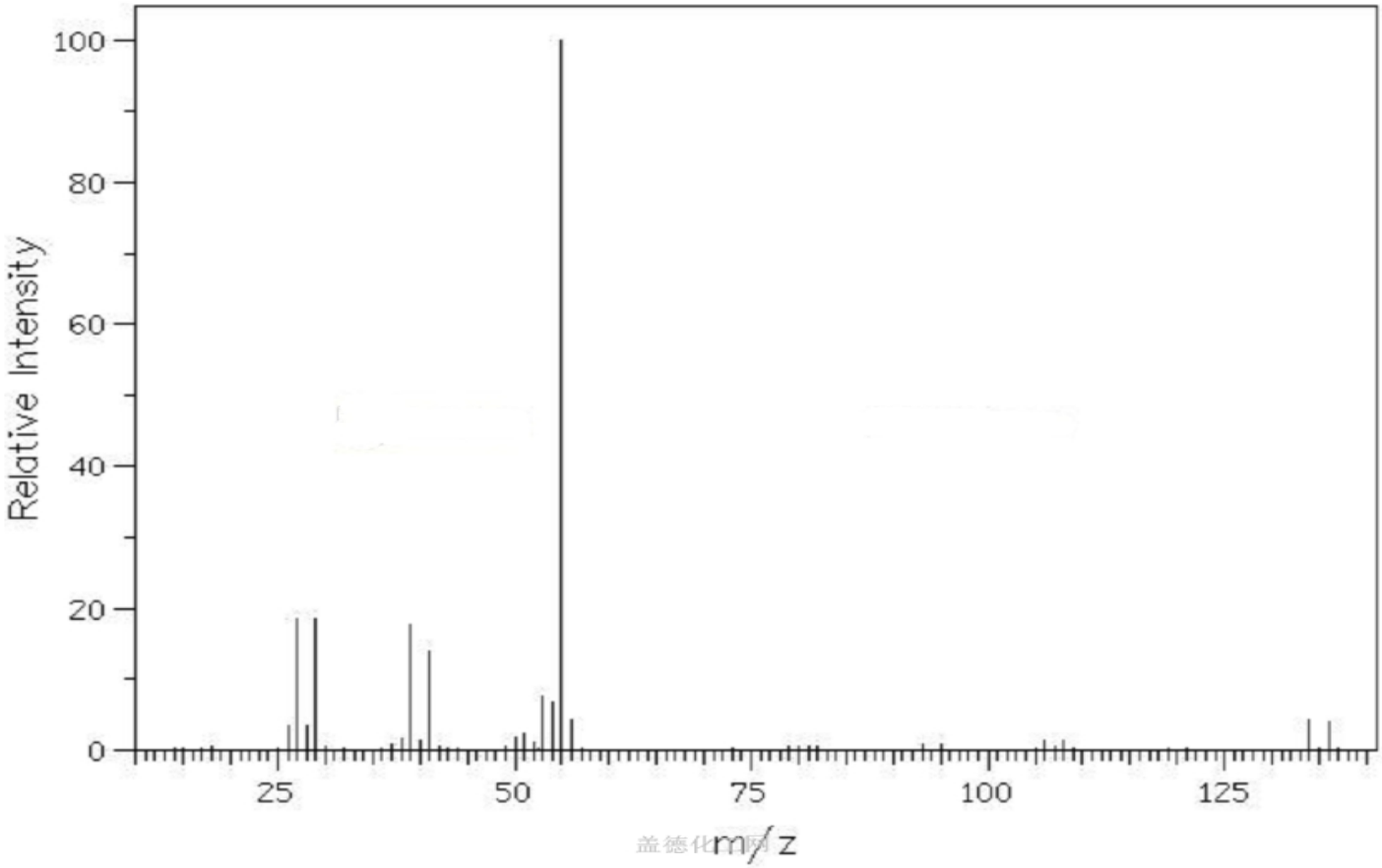
composition: C 35.55%; H 5.19%; Br 59.26%

molecular formula: _____









Further Information:

Compound is immiscible (insoluble) in water.

Compound decolourises bromine water.