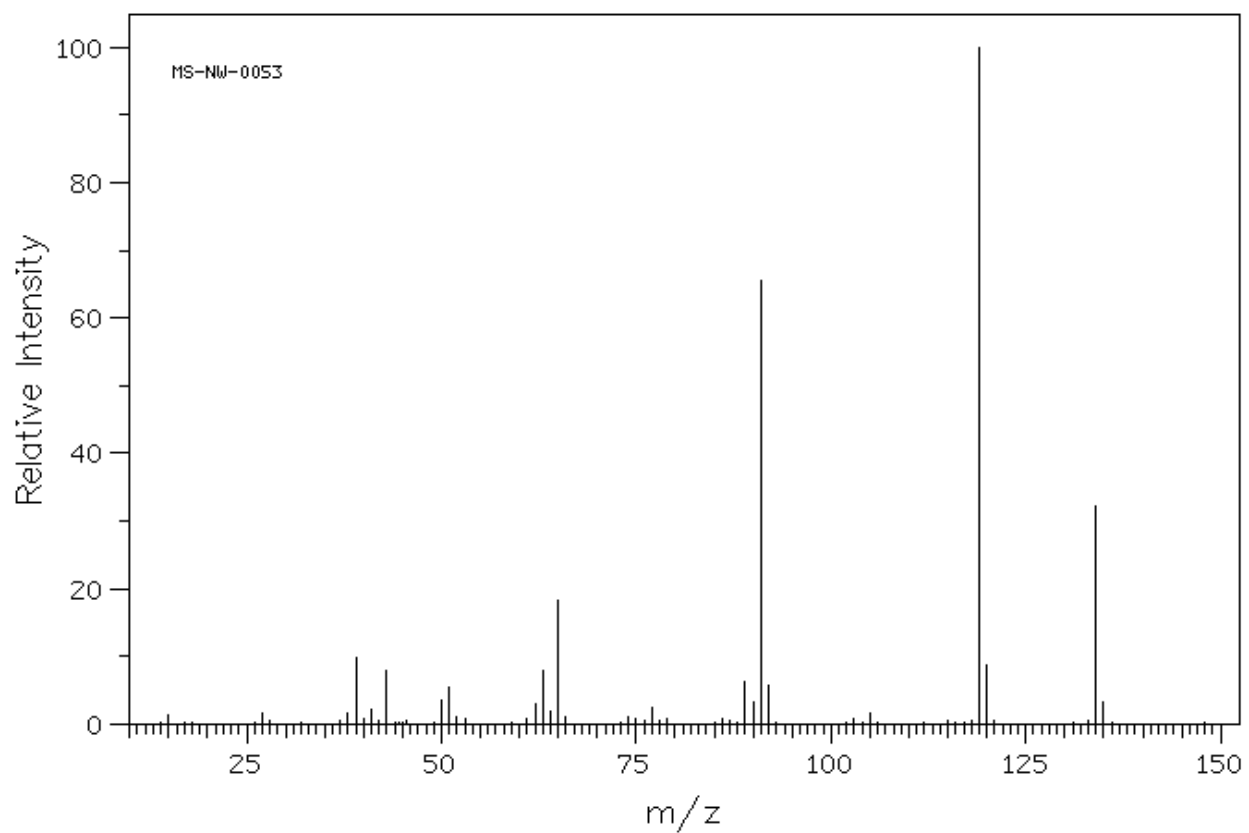


Exercise 3: More characterization of compounds using spectroscopic techniques

Through combustion analysis, 10.00 g of an unknown compound yields 29.52 g CO_2 and 6.72 g H_2O and no other products. In addition, careful experimentation reveals that 26.23 g of oxygen were needed to completely combust the sample.

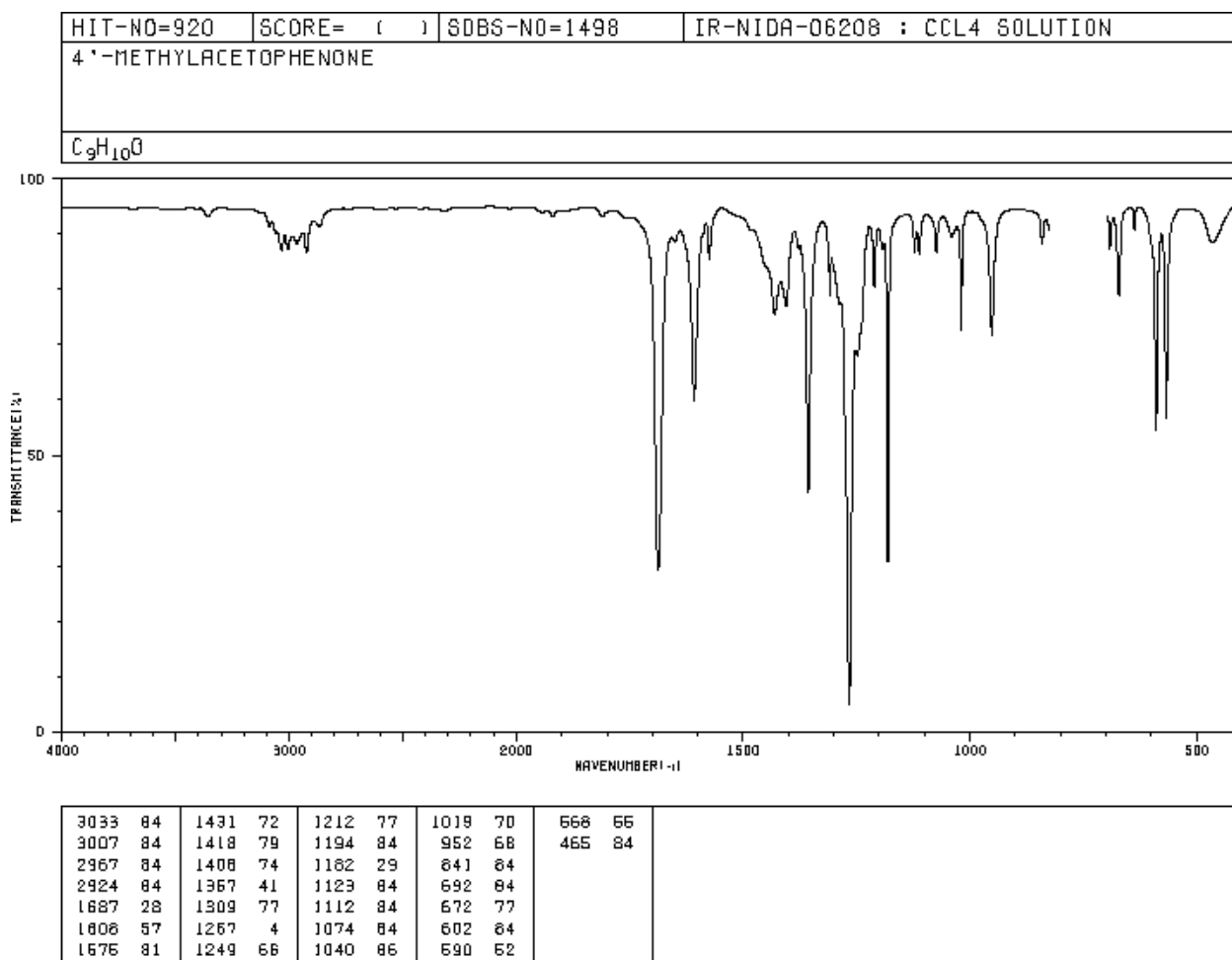
1. Determine the empirical formula of the compound. Show your calculations. Somewhere in your derivation, show whether the compound is a simple hydrocarbon or not.

2. Below is the mass spectrum of the unknown compound; determine the molecular formula of the compound based on your answer to question 1 and the mass spectrum.



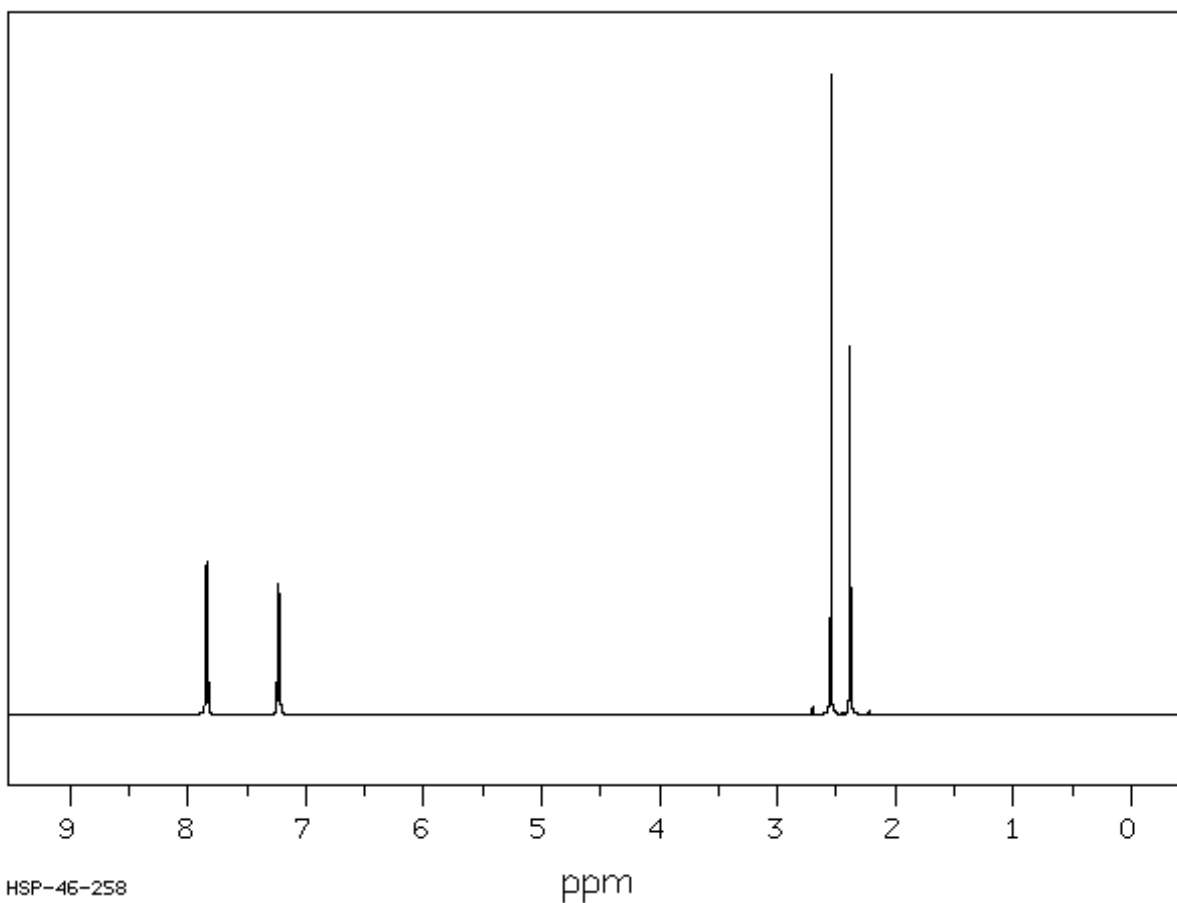
m/z	Intensity
39.0	9.7
43.0	8.0
51.0	5.4
63.0	7.9
65.0	18.2
89.0	6.3
91.0	65.6
92.0	5.7
119.0	100.0
120.0	8.8
134.0	32.2

3. Below is IR spectrum of the unknown.



Pick some key peaks and assign functional groups. Hint: Some of the weaker absorptions in the functional group region of the spectrum will be helpful.

Below is the ^1H -NMR for the unknown compound.



Assign.	Shift (ppm)	Integration	# of protons
A	7.838	654	
B	7.228	689	
C	2.544	1044	
D	2.381	975	

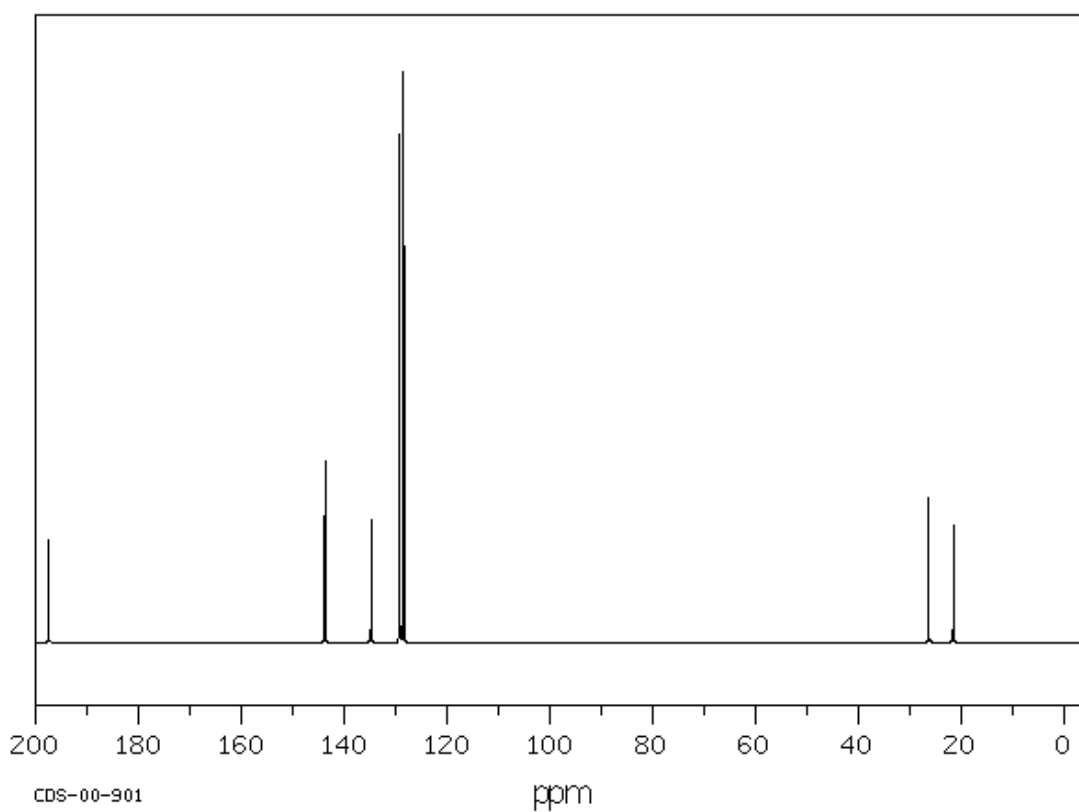
None of the peaks show any splitting.

4. Convert the peak integrations into the correct number of protons represented by each peak.

5. Using information on the IR spectrum as well, what do the peaks at $\delta = 7.8$ and 7.2 tell you about the structure of the molecule?

6. Draw the structure of the molecule and assign peaks A through D (previous page) to the appropriate sets of equivalent hydrogens.

Below is the ^{13}C -NMR for the unknown compound.



ppm	Assign.
197.49	1
143.74	2
134.79	3
129.23	4 *
128.43	5 *
26.37	6
21.52	7

The "assignments" column refers to a particular carbon or carbons (the * indicates the integration of the peak suggests two carbons represented by that peak).

7. On your structure for the unknown molecule, label the carbons with the appropriate assignment number.

8. Justify your assignment of the $\delta = 197$ peak to the particular carbon you chose. In other words, why is it so far downfield?