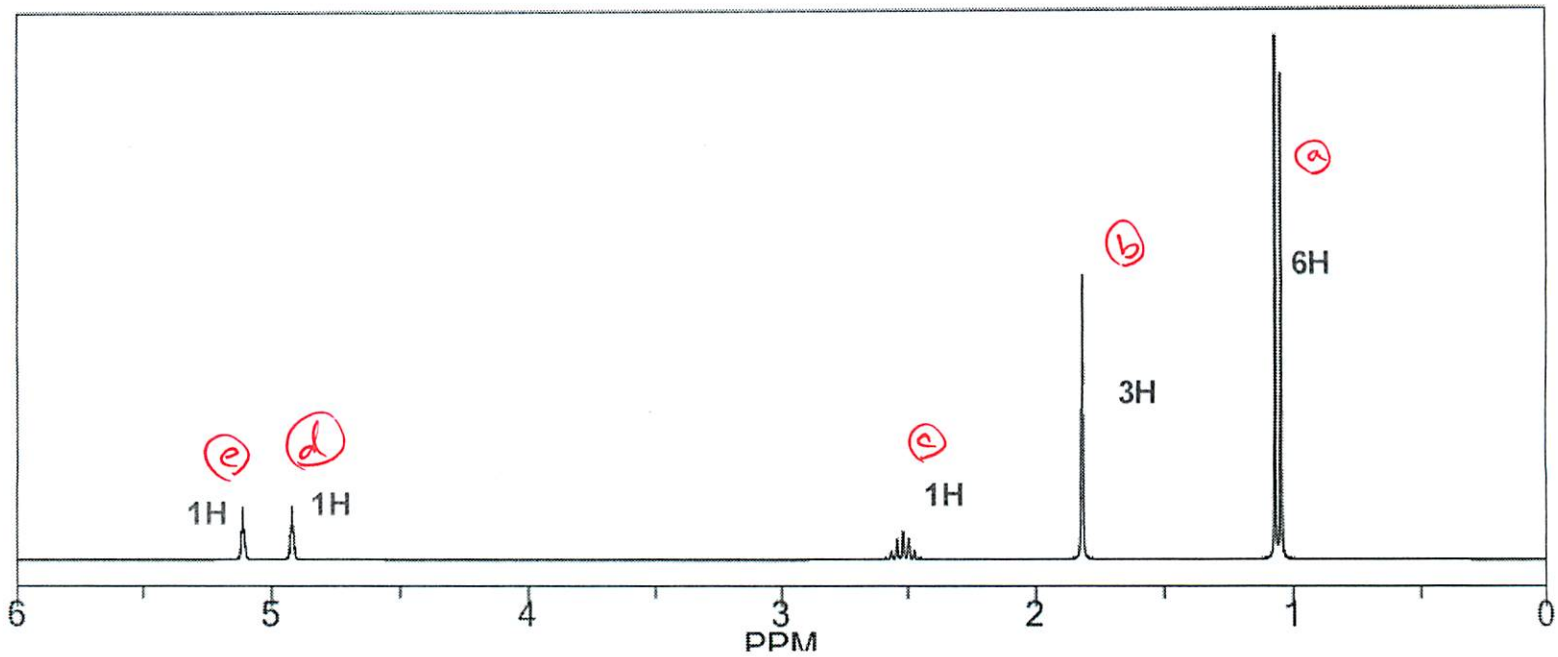
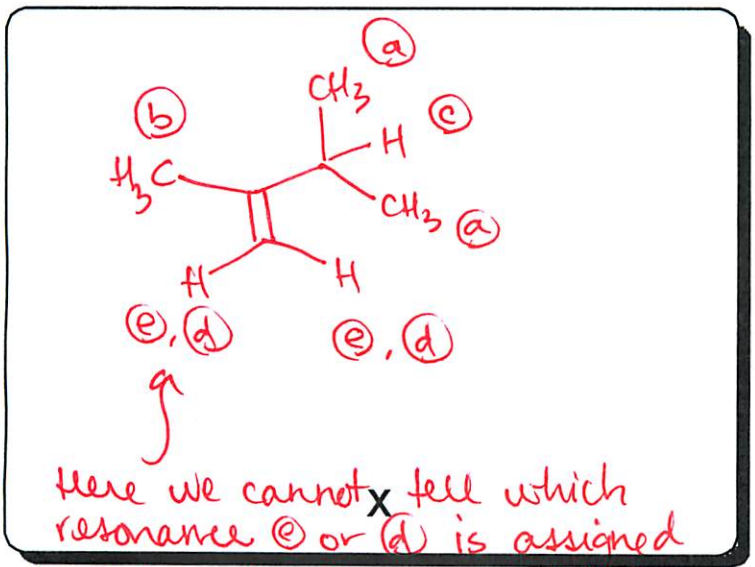
NMR Practice for Your Mastery and Enjoyment!

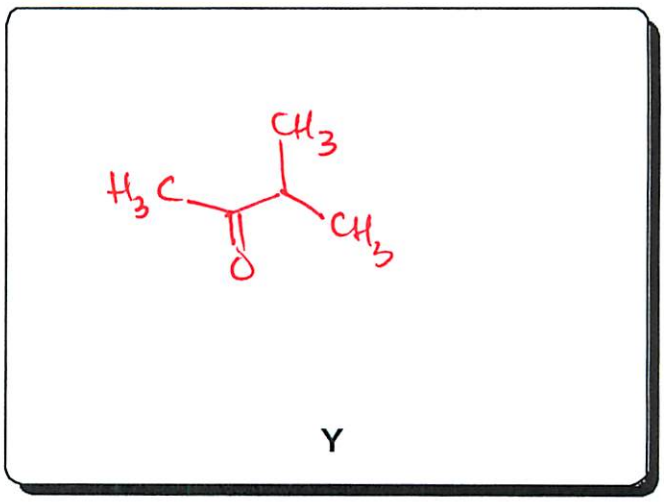
1. An unknown compound **X** (C_6H_{12}) is analyzed by NMR spectroscopy to give the following spectra. Ozonolysis of **X** gives two compounds, one of which is formaldehyde and compound **Y** ($C_5H_{10}O$). Propose structures for **X** and **Y** in the boxes below. Also, label the peaks in the spectra according to the "a,b,c" naming system and assign these resonances to the protons in your proposed structure for **X**.





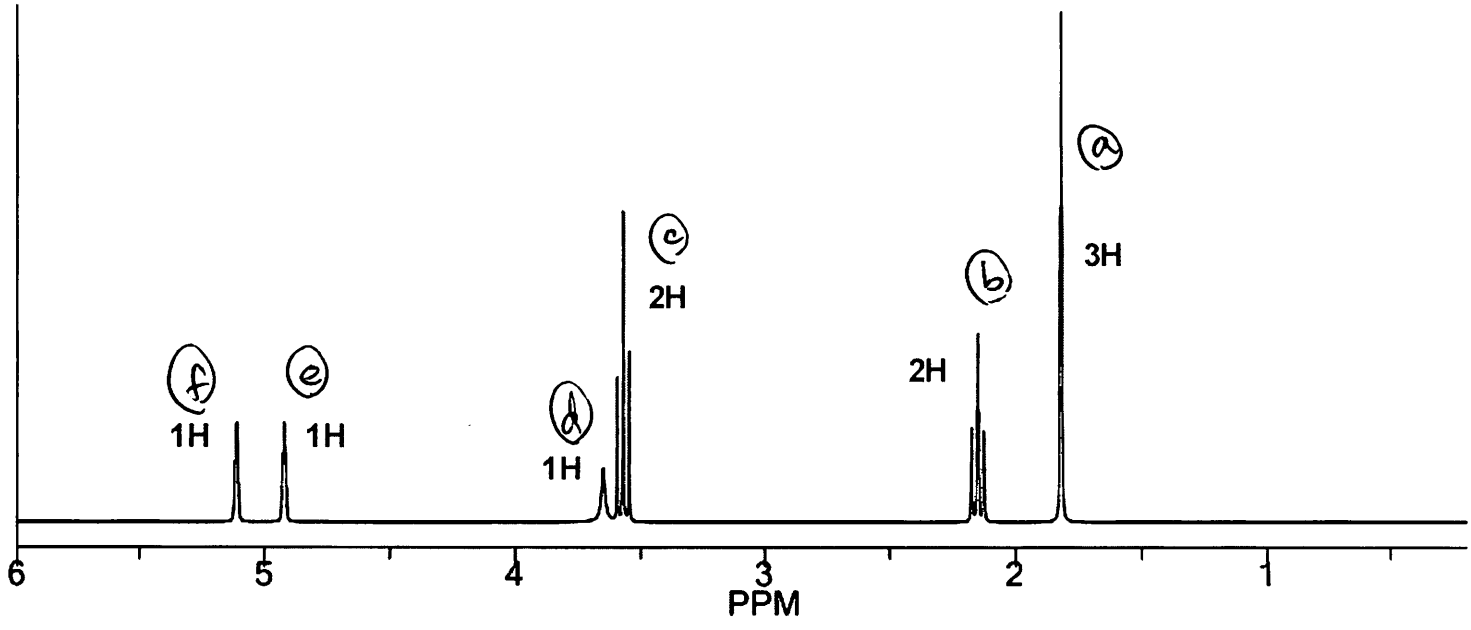
Here we cannot tell which resonance (e) or (d) is assigned to this proton (not enough info.) so we assign both possible resonances.

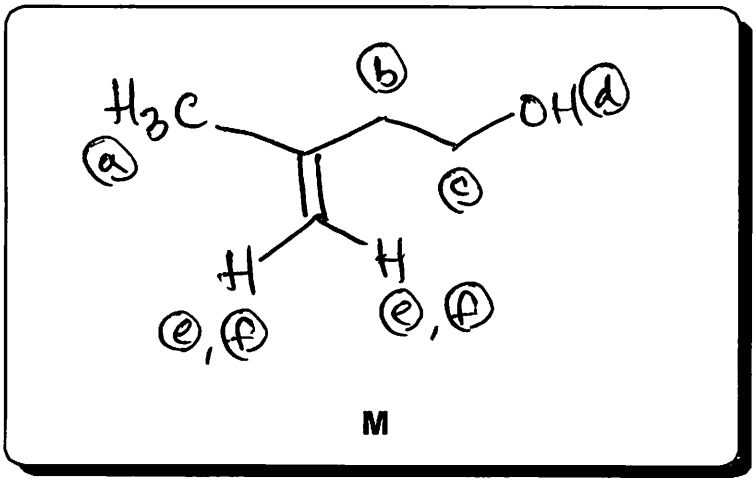
X



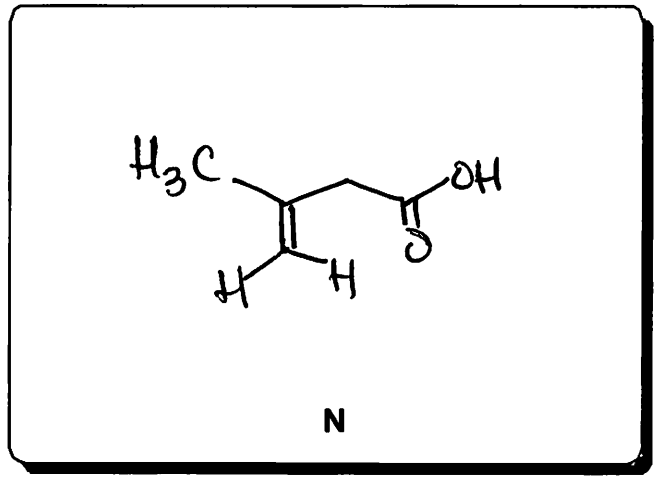
Y

2. A compound **M,** ($C_5H_{10}O$) was shown to give the NMR spectra shown below. Upon exposure to Jones reagent in water, a new compound **N,** ($C_5H_8O_2$) was obtained. Propose structures for **M** and **N**. For **M**, label the peaks in the spectra with the "a,b,c" naming system and assign them to the protons giving rise to the observed resonance.



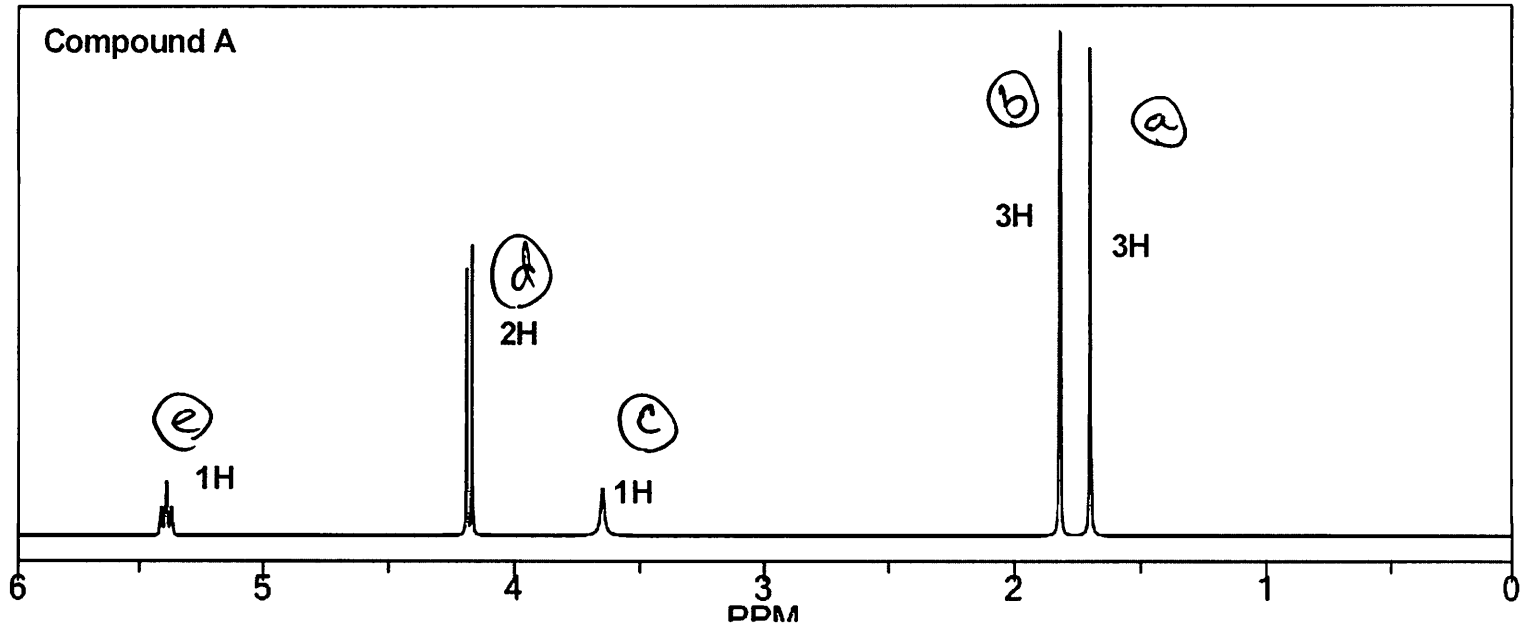


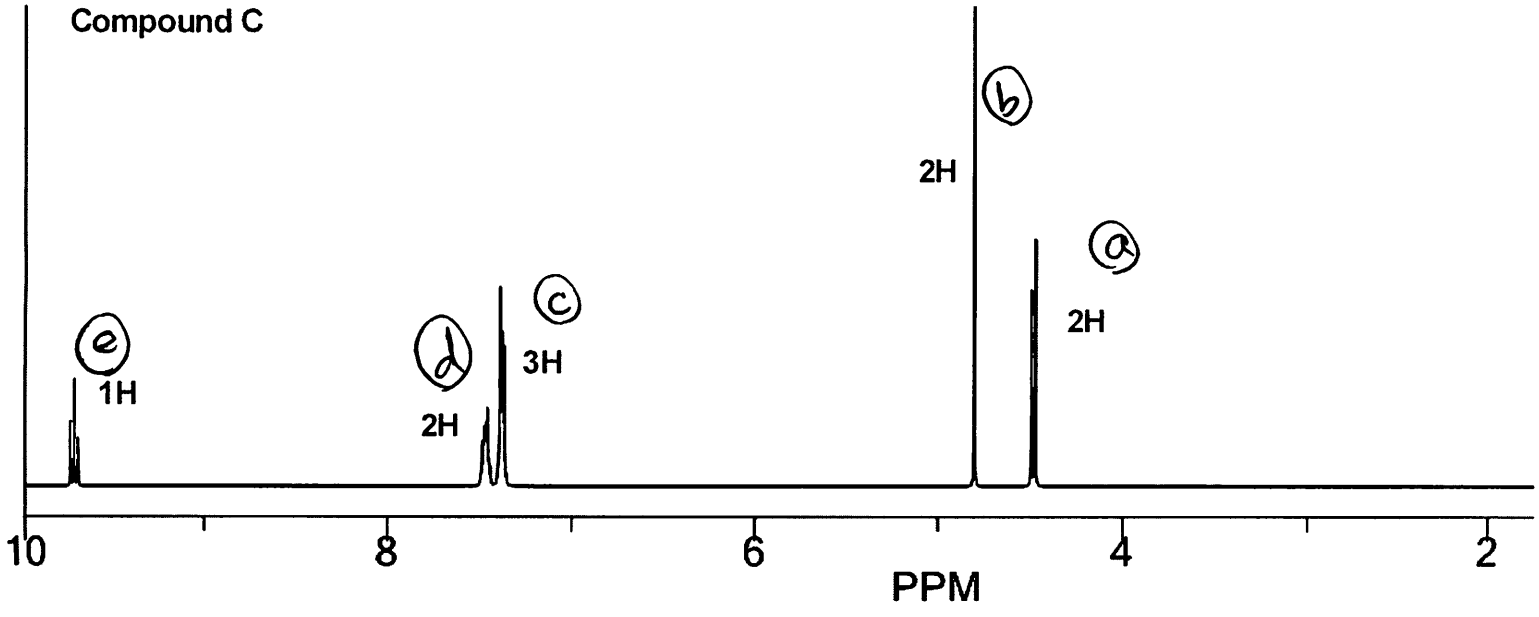
M

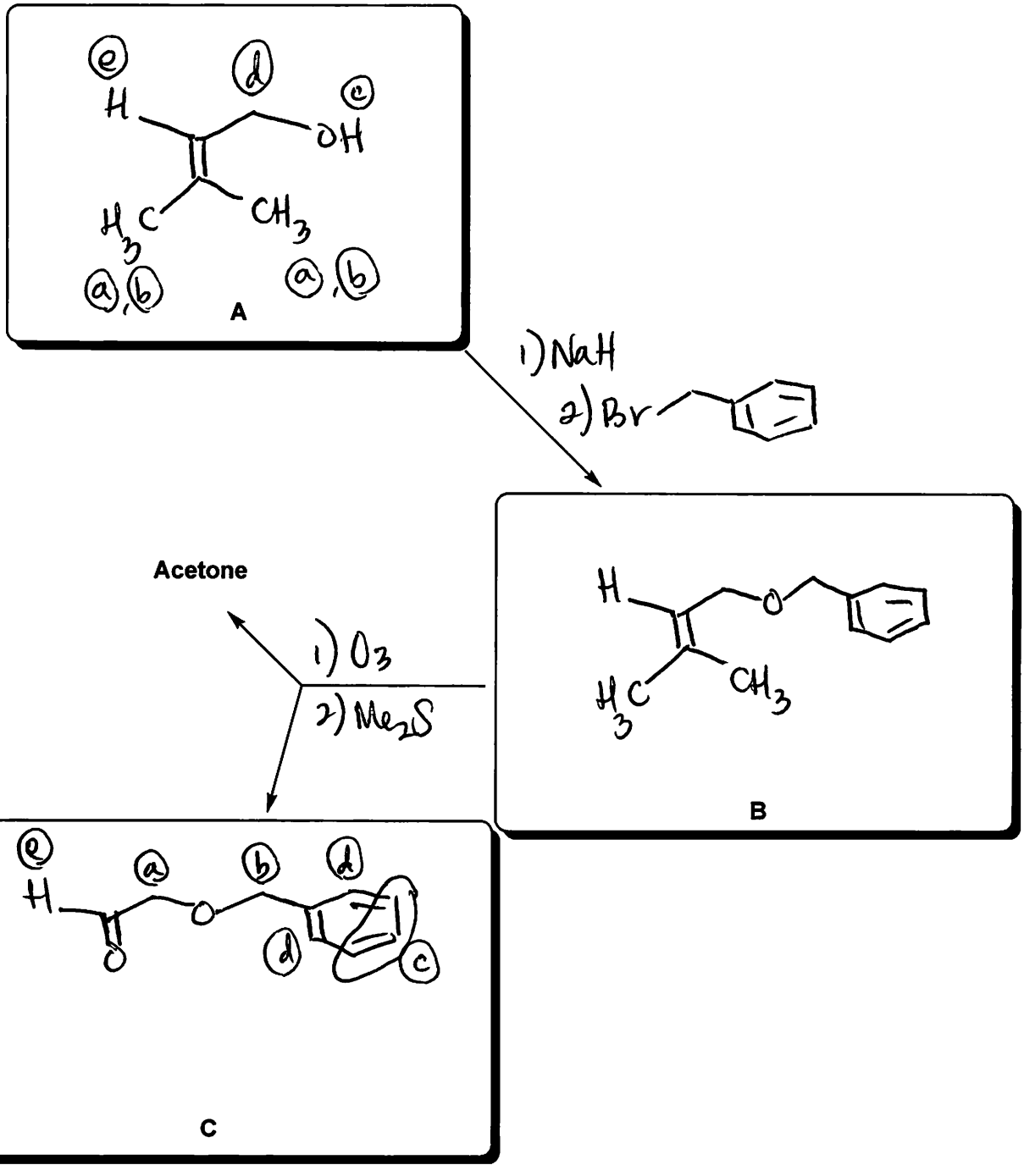


N

3. **A** ($C_5H_{10}O$) is one of nature's building blocks, and regularly employed in the biological synthesis of steroids and other natural compounds. Proton NMR of **A** yields the following spectra. **A** is easily treated with sodium hydride, which when followed with benzyl bromide ($BrCH_2Ph$) gives compound **B.** Ozonolysis of **B** provides acetone and unknown compound **C**($C_9H_{10}O_2$), which also has the following proton NMR spectra. Provide structures for compounds **A** – **C**. Formulate each reaction by writing the reaction conditions over the arrows leading to each product. Finally, label the resonances in each spectra by the "a,b,c" naming system and assign them to the corresponding protons in your proposed structures.

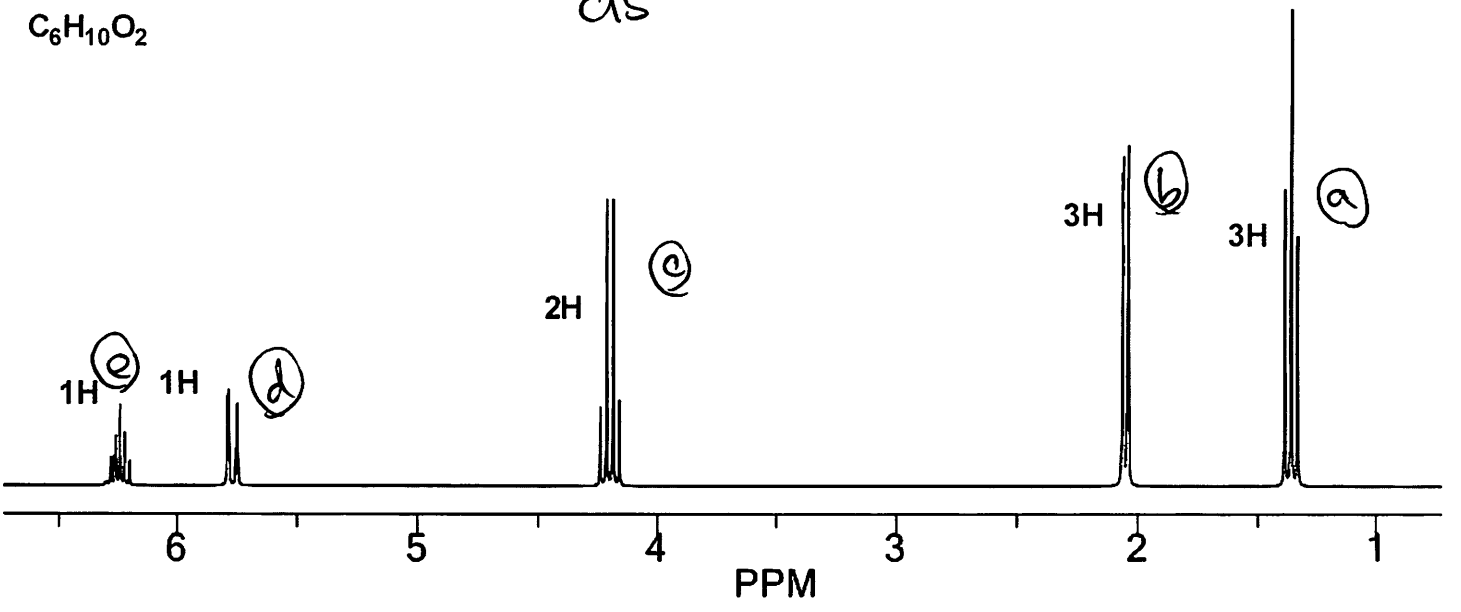


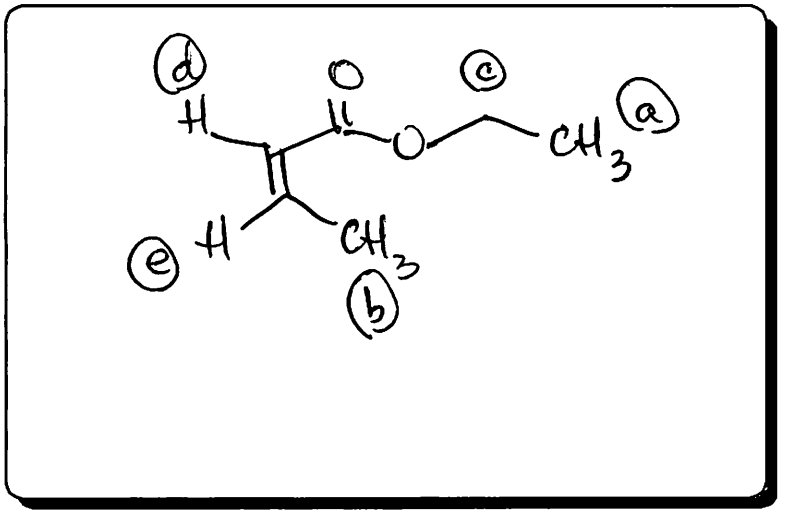



A
1) NaH
2) Br
B
Acetone
1) O3
2) Me2S
C

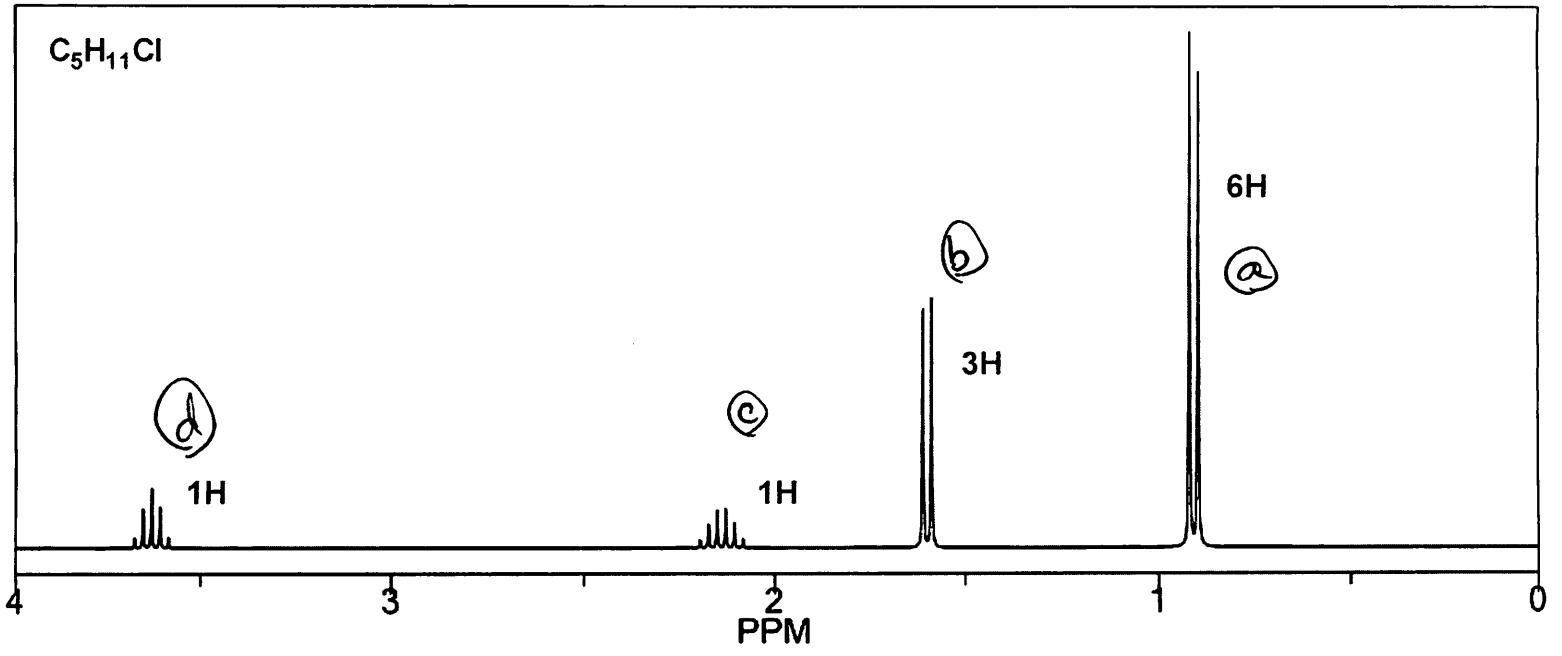
4. Provide a structure based on the NMR data shown below. Also, label the resonances in the spectra by the "a,b,c" naming system and assign them to the corresponding protons in your proposed structure.
^{1}H NMR: δ 6.25 (qn, 1H), 5.77 (d, J = 10.8 Hz, 1H), 4.20 (q, 2H), 2.05 (d, 3H), 1.36 (t, 3H).

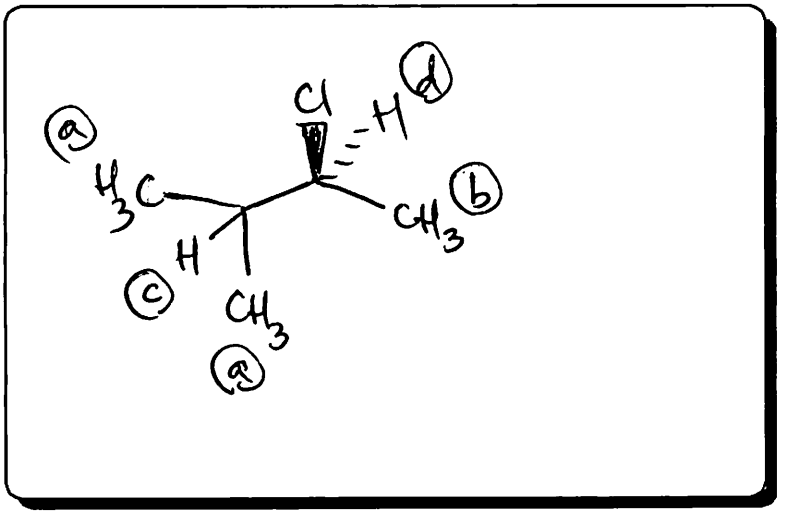
cis





5. Provide a structure based on the NMR data shown below, drawing it as the (S)-enantiomer. Provide the name of this compound. Also, label the resonances in the spectra by the "a,b,c" naming system and assign them to the corresponding protons in your proposed structure.

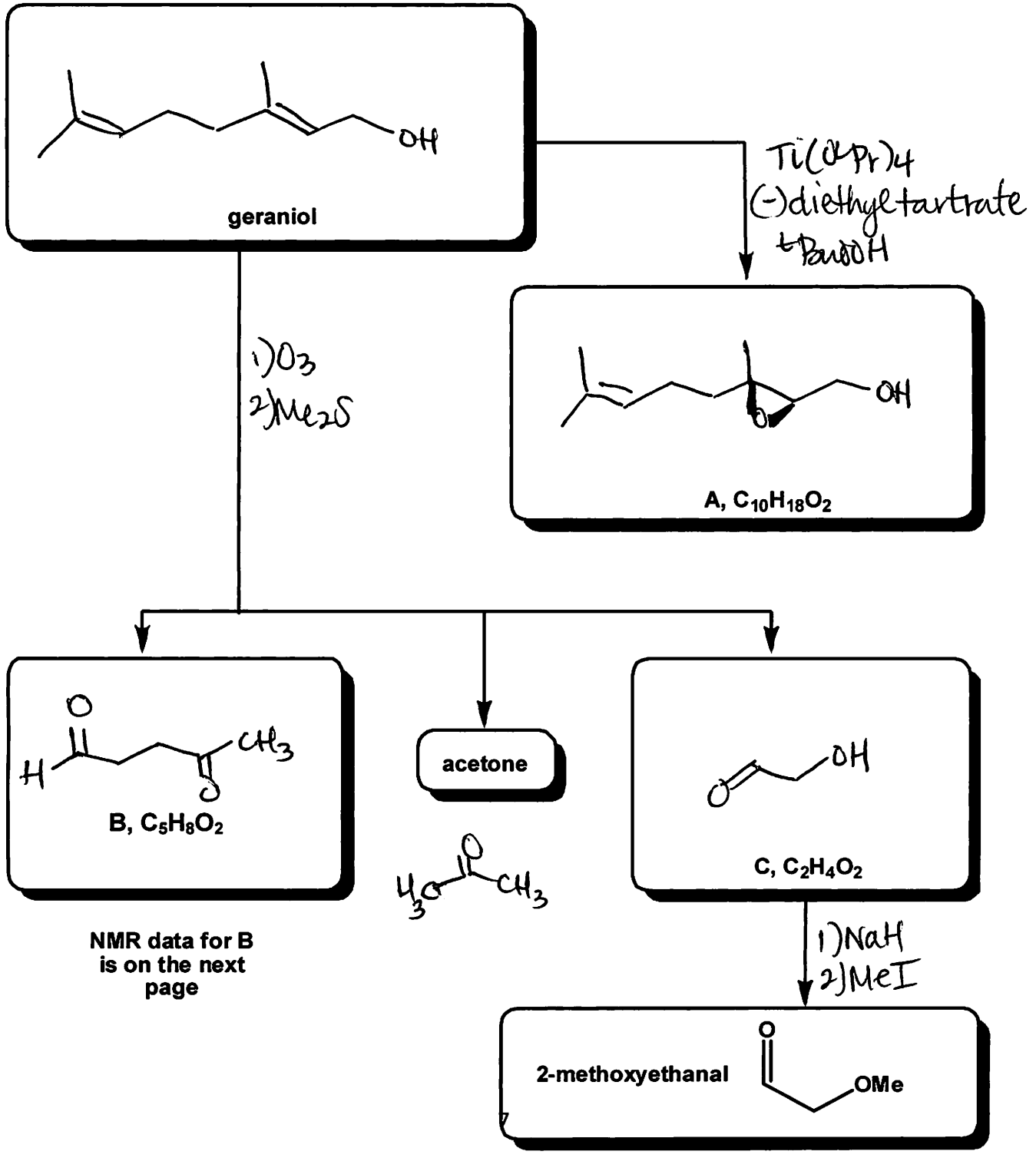




(S)-2-chloro-3-methylbutane

NAME

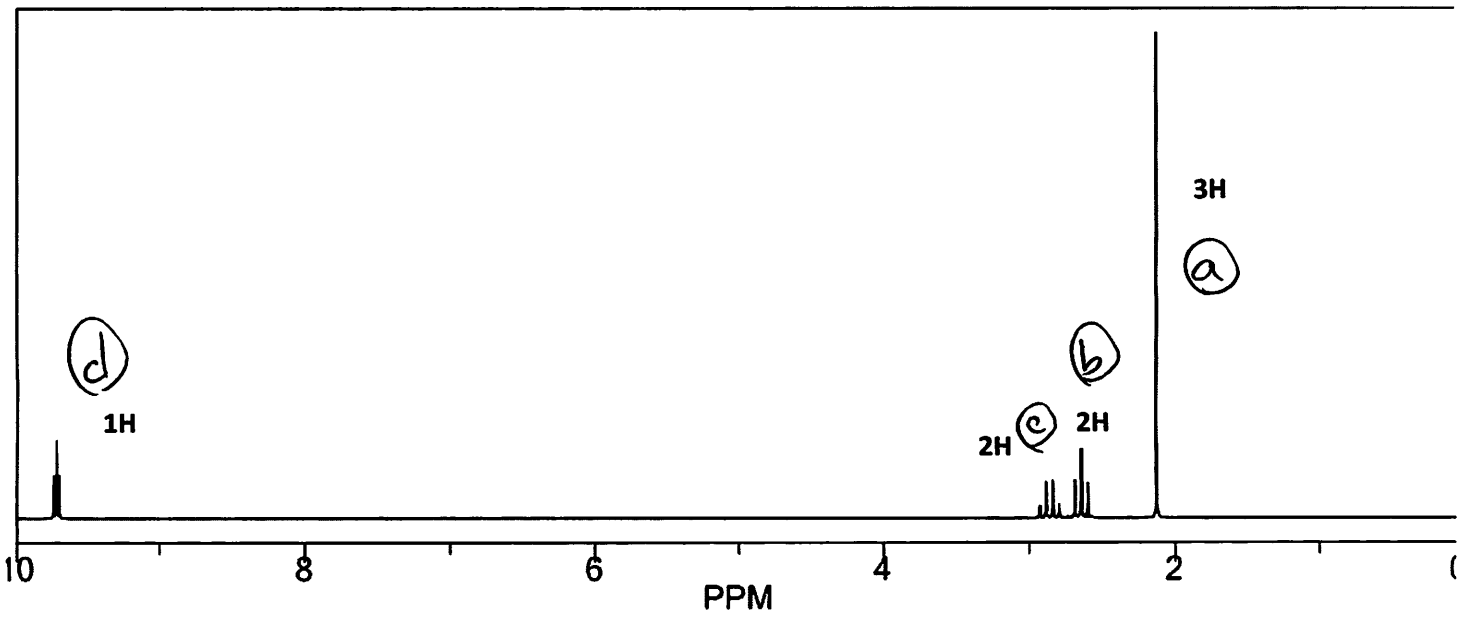
6. Puzzle time. Geraniol ($C_{10}H_{18}O$) is a fragrant *diterpene*, which is a primary component in the "essence of rose". When geraniol is reacted under asymmetric epoxidation conditions with (-) diethyl tartrate, Compound **A** ($C_{10}H_{18}O_2$) is produced. Exposure of geraniol to ozonolysis conditions yields three products: acetone, and Compounds **B**, **C**. When **C** is treated with sodium hydride, then methyl iodide, 2-methoxyethanal is the major product isolated. Propose structures for geraniol, and Compounds **A – C**. Formulate the reactions by writing the reaction conditions over the proper reaction arrows.

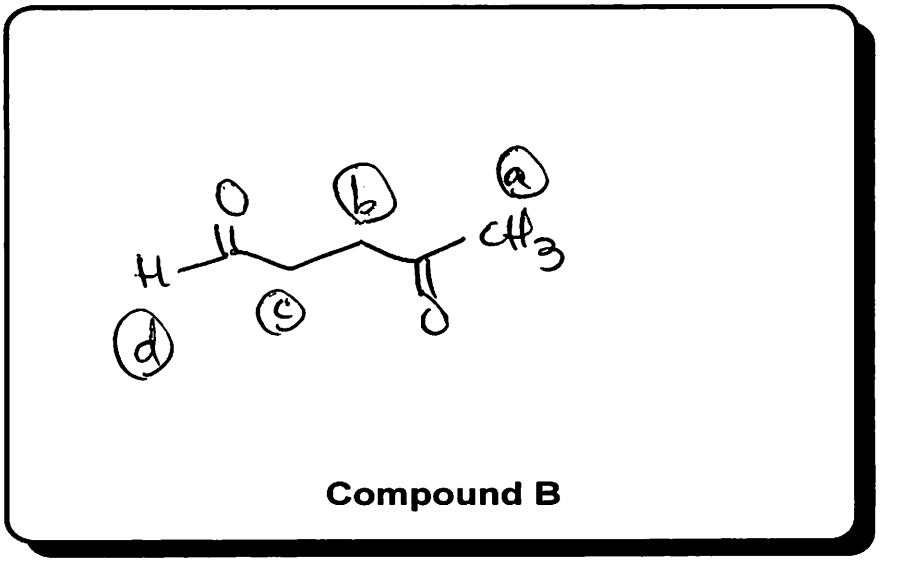


NMR data for B is on the next page

Cont. . . The following ^{1}H NMR spectrum is that of compound B. Redraw compound B in the box below, label the peaks in the spectra using the "a,b,c" naming system and assign them to the protons in Compound B.

^{1}H NMR: δ 9.7 (t, 1H), 2.9 (q, 2H), 2.6 (t, 2H), 2.1 (s, 3H)

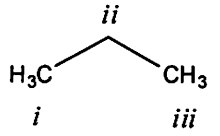




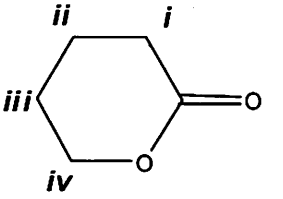
Compound B

7. For the following compounds, use the "a,b,c" naming system to indicate non-equivalent hydrogens by writing the letters on the lines given. Also include what splitting pattern would be predicted for each proton. Propane is provided as an example. If non-equivalent protons will split a set differently, all signals must be listed (i.e. list complex splitting patterns if possible, not the simpler "n + 1")

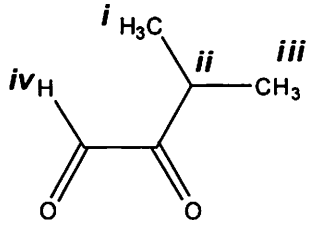
EXAMPLE:



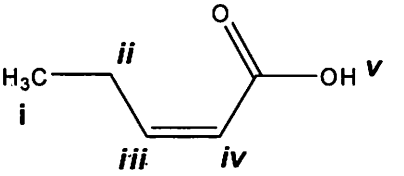
i. _a,_triplet__
ii. _b,_septet_
iii. _a,_triplet__



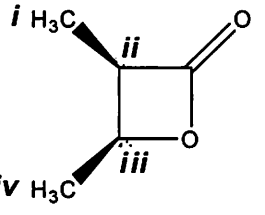
i
ii
iii
iv



i a- doublet
ii b- septet
iii a- doublet
iv c- singlet

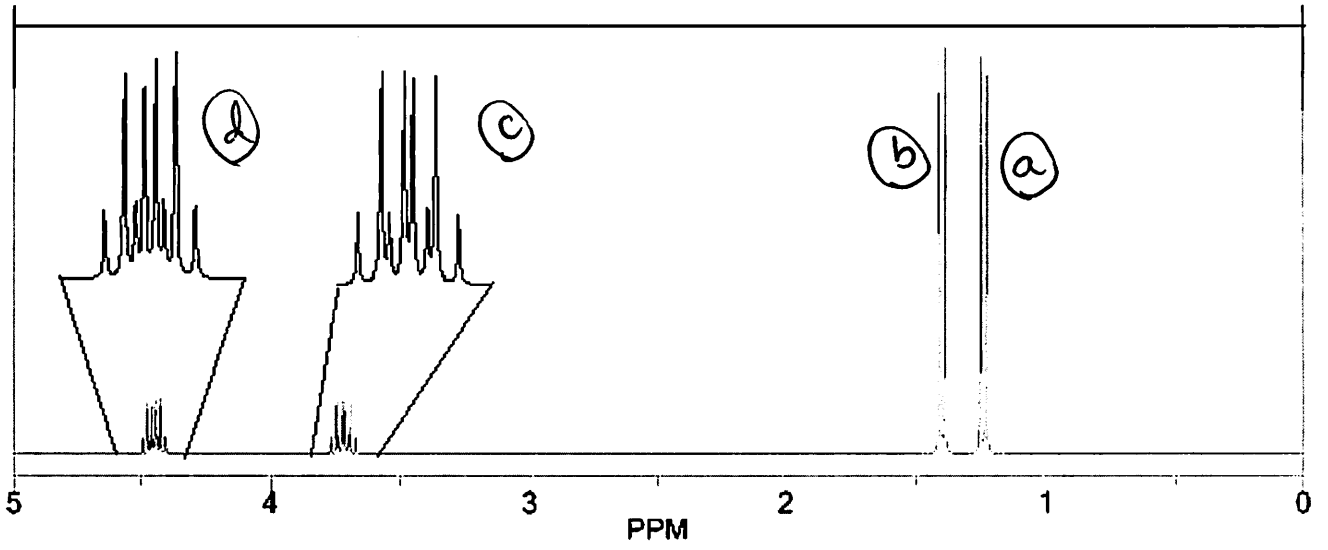


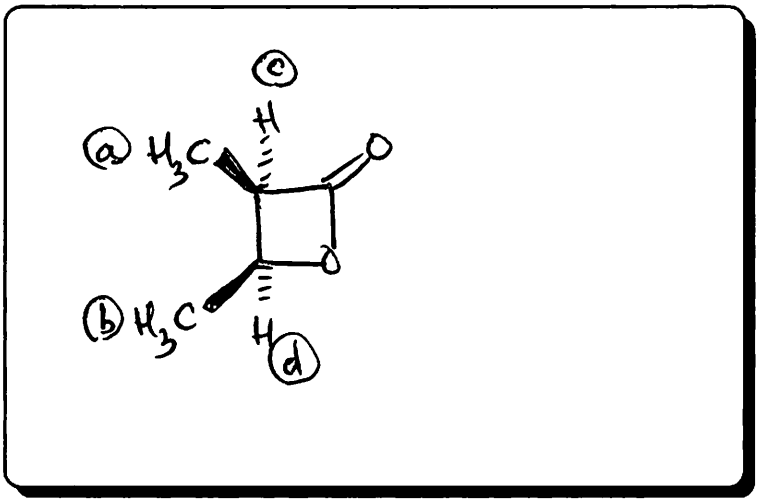
i a- triplet
ii b- doublet of quartets (dq)
iii c- doublet of triplets (dt)
iv d- doublet
v e- singlet



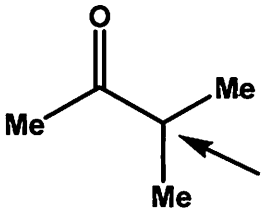
i a- doublet
ii b- dq
iii c- dq
iv d- doublet

8. Determine which of the compounds in #7 produces the NMR spectrum given below. Draw the structure in the box, label the resonances with the "a,b,c" system and assign them to the protons in your proposed structure.



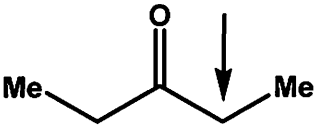


9. For the following constitutional isomers of $C_5H_{10}O$ below, write how many sets of equivalent protons exist on the top line under each structure. Using the bottom line space, predict what splitting pattern (singlet, doublet, etc.) might be observed in the ^1H NMR spectrum for the proton(s) indicated by an arrow in each structure. If a complex splitting pattern is possible, list the predicted for complex splitting.



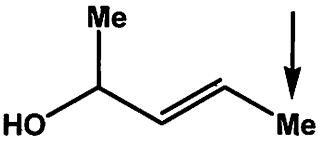
H's: 3

septet



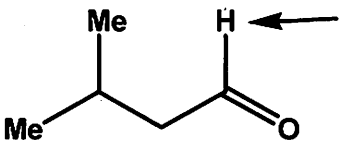
H's: 2

quartet



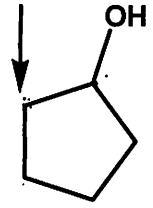
H's: 6

doublet



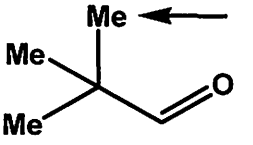
H's: 4

triplet



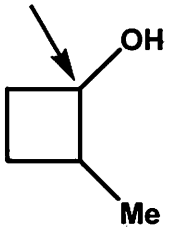
H's: 6

multiplet (dddd)



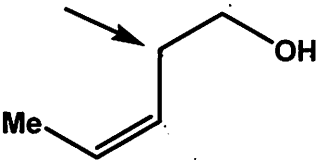
H's: 2

singlet



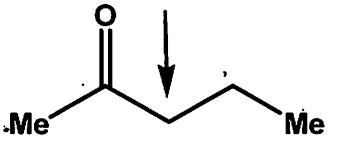
H's: 8

multiplet (dddd)



H's: 6

doublet of triplets (dt)

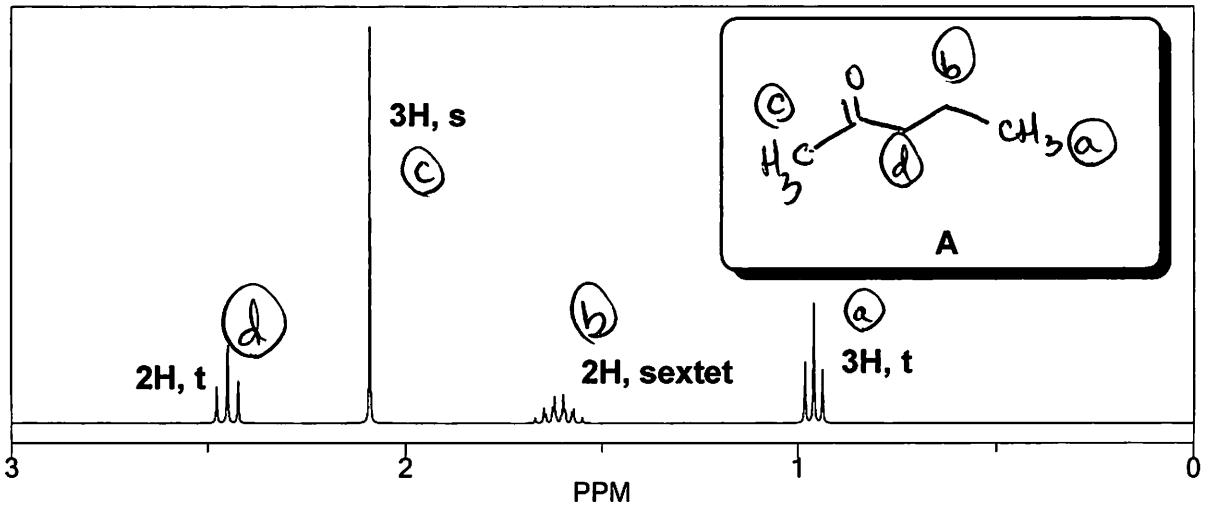


H's: 4

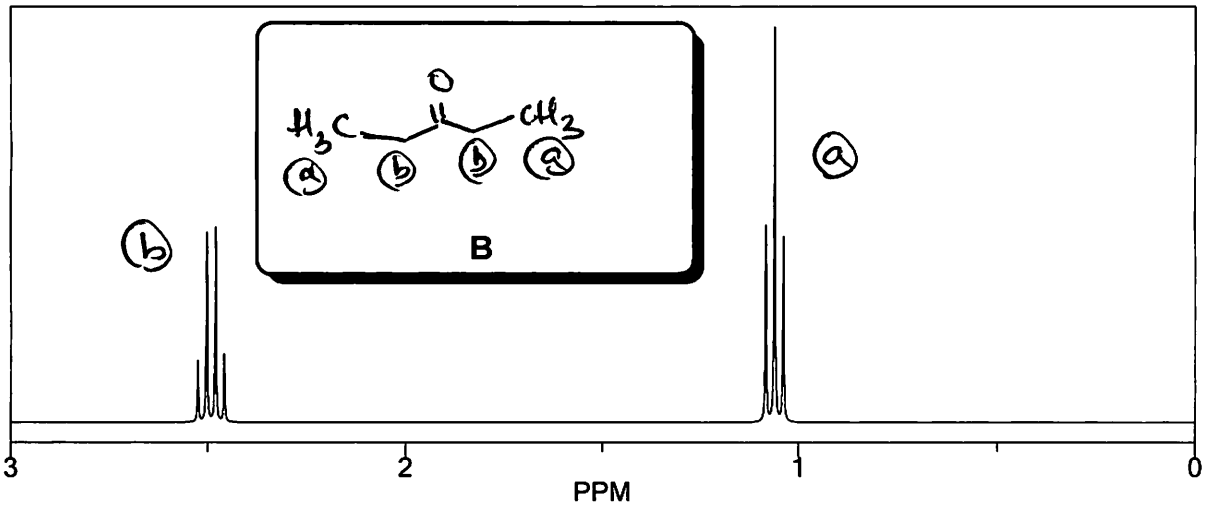
triplet

10. NMR Spectra. The following three spectra are of three of the structures in #5. Assign the correct compound to each spectrum in the boxes provided, label the resonances in each spectra according to the "a,b,c" naming system and assign all signals to the corresponding protons in your proposed structure.

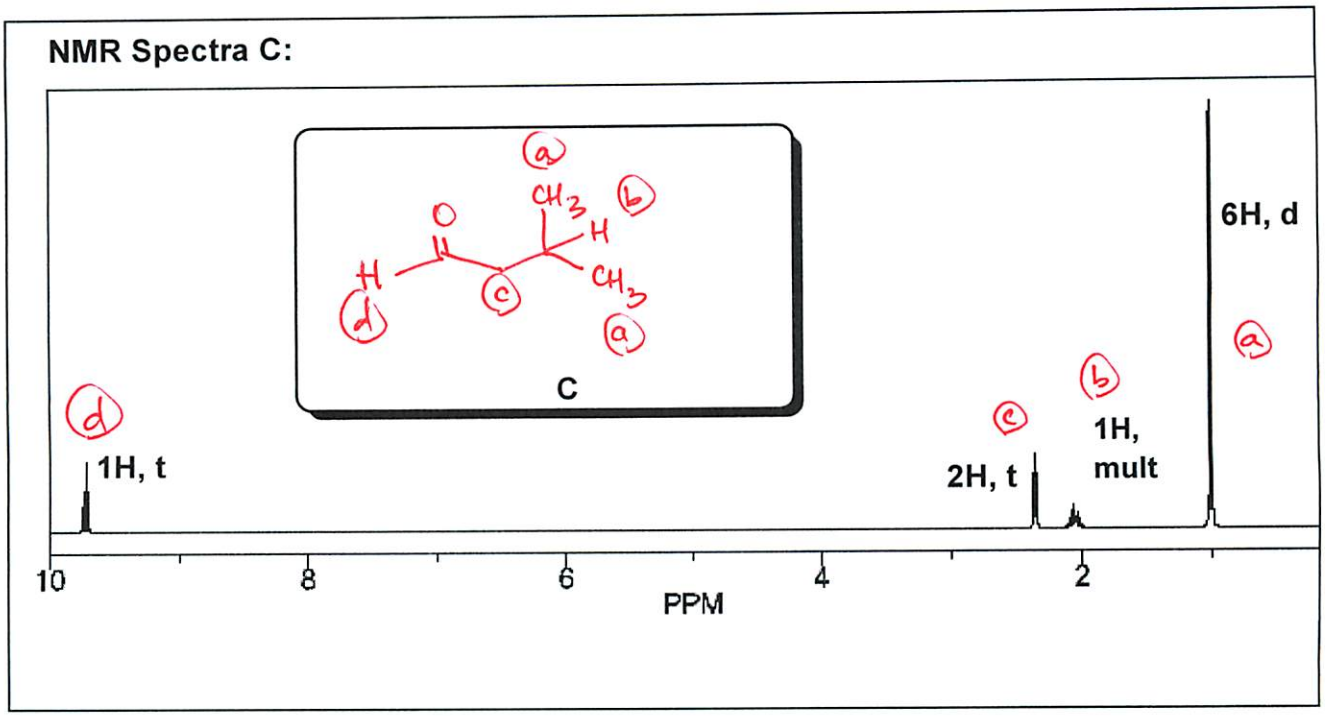
NMR Spectra A:



NMR Spectra B:



NMR Spectra C:



NMR Spectra D