Nuclear Magnetic Resonance
3 - Carbon – 13 NMR

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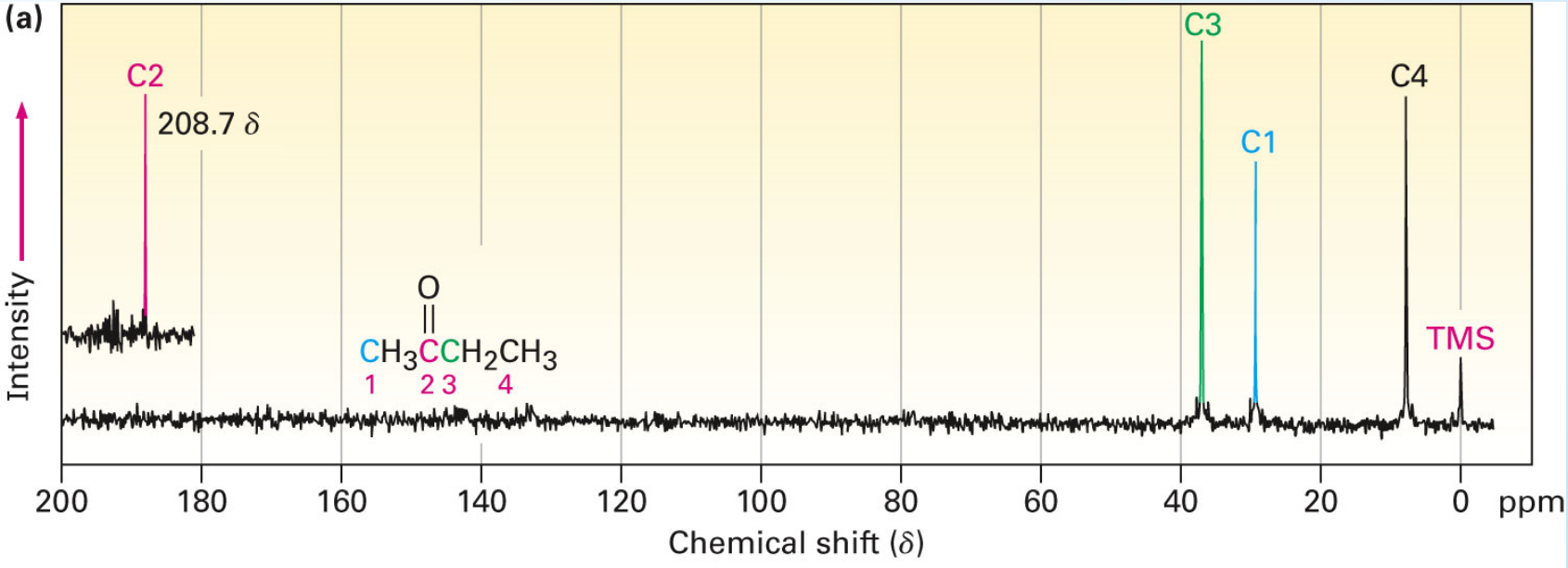
Introduction and Theory of Carbon-13 NMR

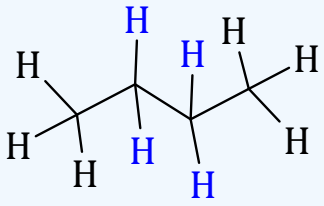
- ^{12}C has no magnetic spin since the nuclear particles are all paired hence it is diamagnetic.
- ^{13}C has a magnetic spin, but there is only 1% of the carbon in a sample so signals are weak, sometimes getting lost in noise. During spectroscopy hundreds of spectra are taken, averaged.
- There is no coupling as the chances of C-13 next to another will be very low. Usually, decoupling is done to avoid this if it happens. All signals are singlets.
- The chemical shift scale is from 0-230 ppm, very high energy.
- The intensity can still give an idea of how many carbons are under the peak.
- The number of peaks indicate the number of carbons. Note that if the carbons are equivalent, they will appear as one signal.
- Techniques such as DEPT (Distortionless Enhancement by Polarization Transfer) allows to see how many carbons are attached on each carbon, classifying the carbons as, 1^o, 2^o, 3^o and 4^o.
- C-13 can be corelated with H-NMR to give HETCOR – hetero correlation to help assign each carbon to proton peaks.

C-13 NMR First Look

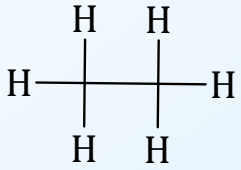
- Below is the spectrum for **2- butanone**



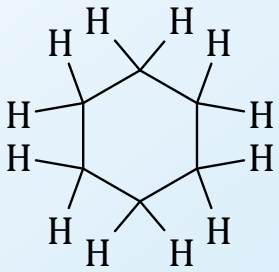
Number of Signals



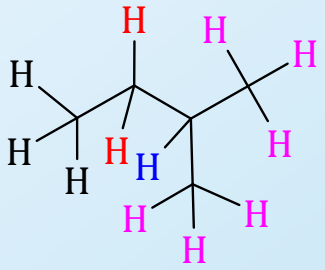
2 peaks



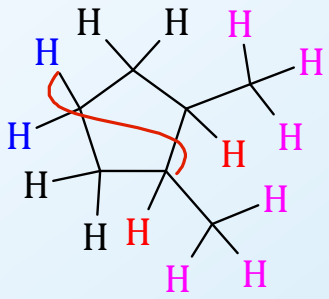
1 peak



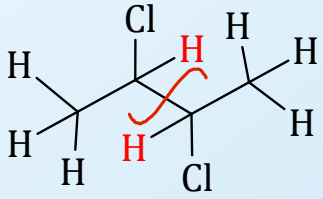
1 peak



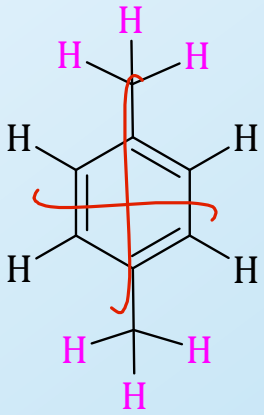
4 peaks



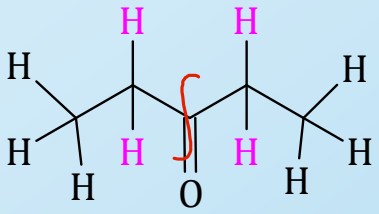
4 peaks



2 peaks

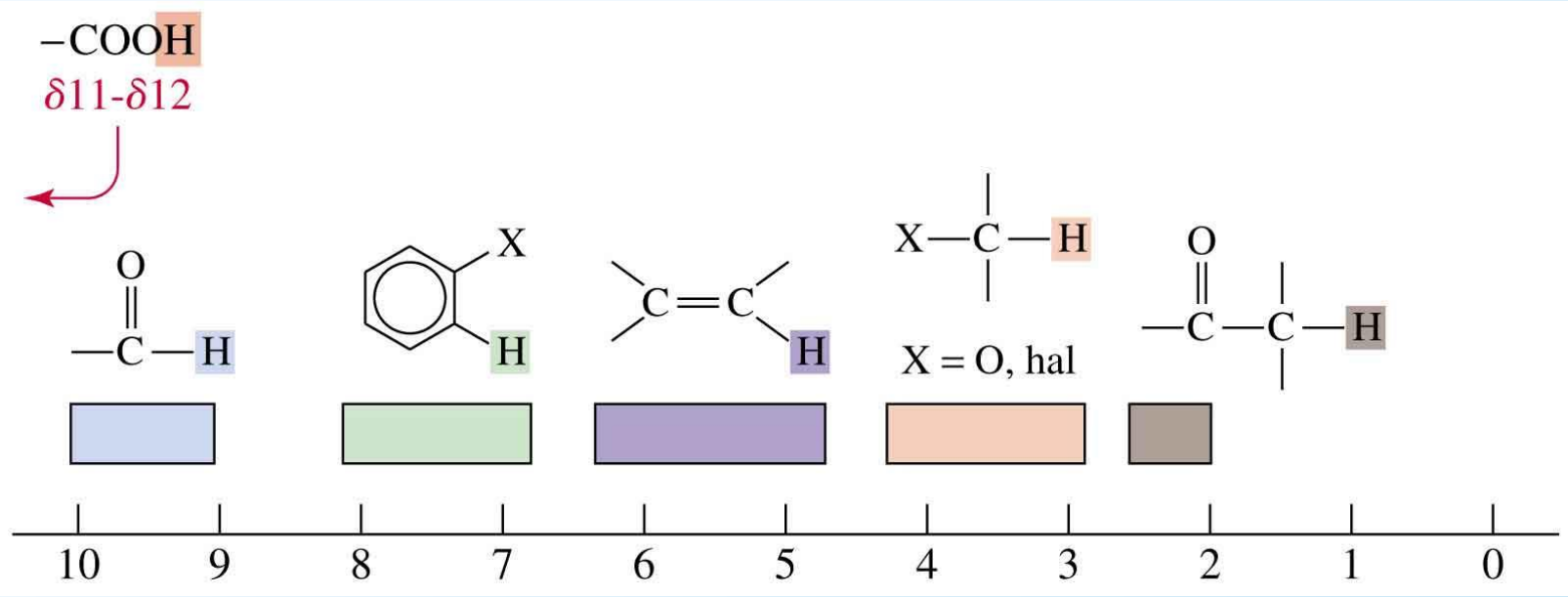


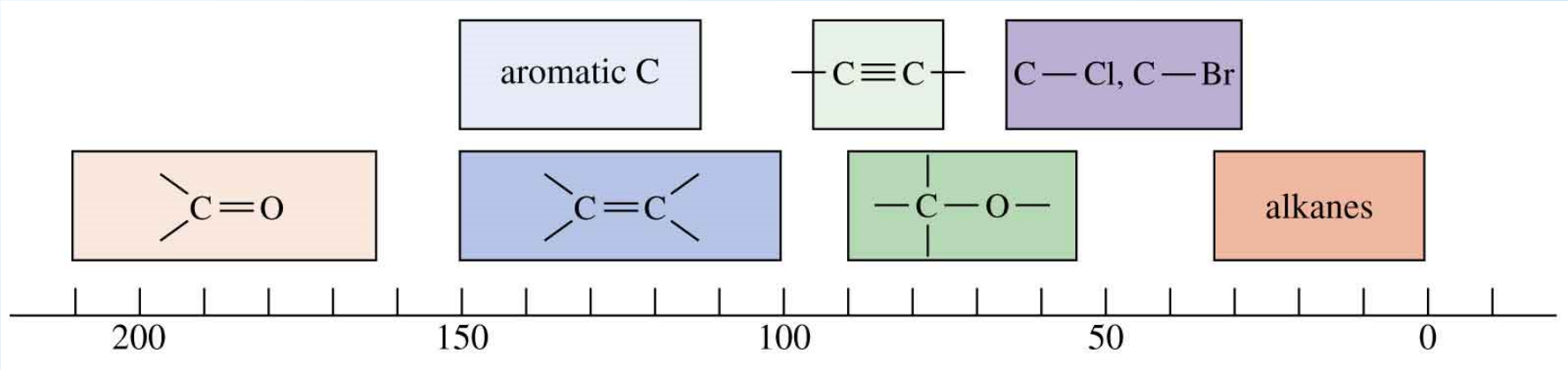
3 peaks



3 peaks

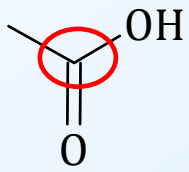
Hydrogen and Carbon Chemical Shifts



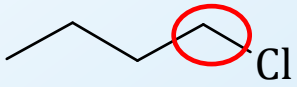


Chemical Shift

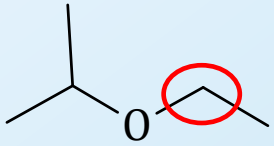
a CH_3COOH



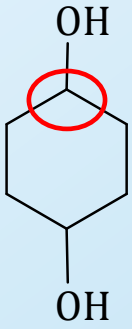
b $CH_3CH_2CH_2CH_2Cl$



c $(CH_3)_2CHOCH_2CH_3$



d Cyclohexane-1,4-diol

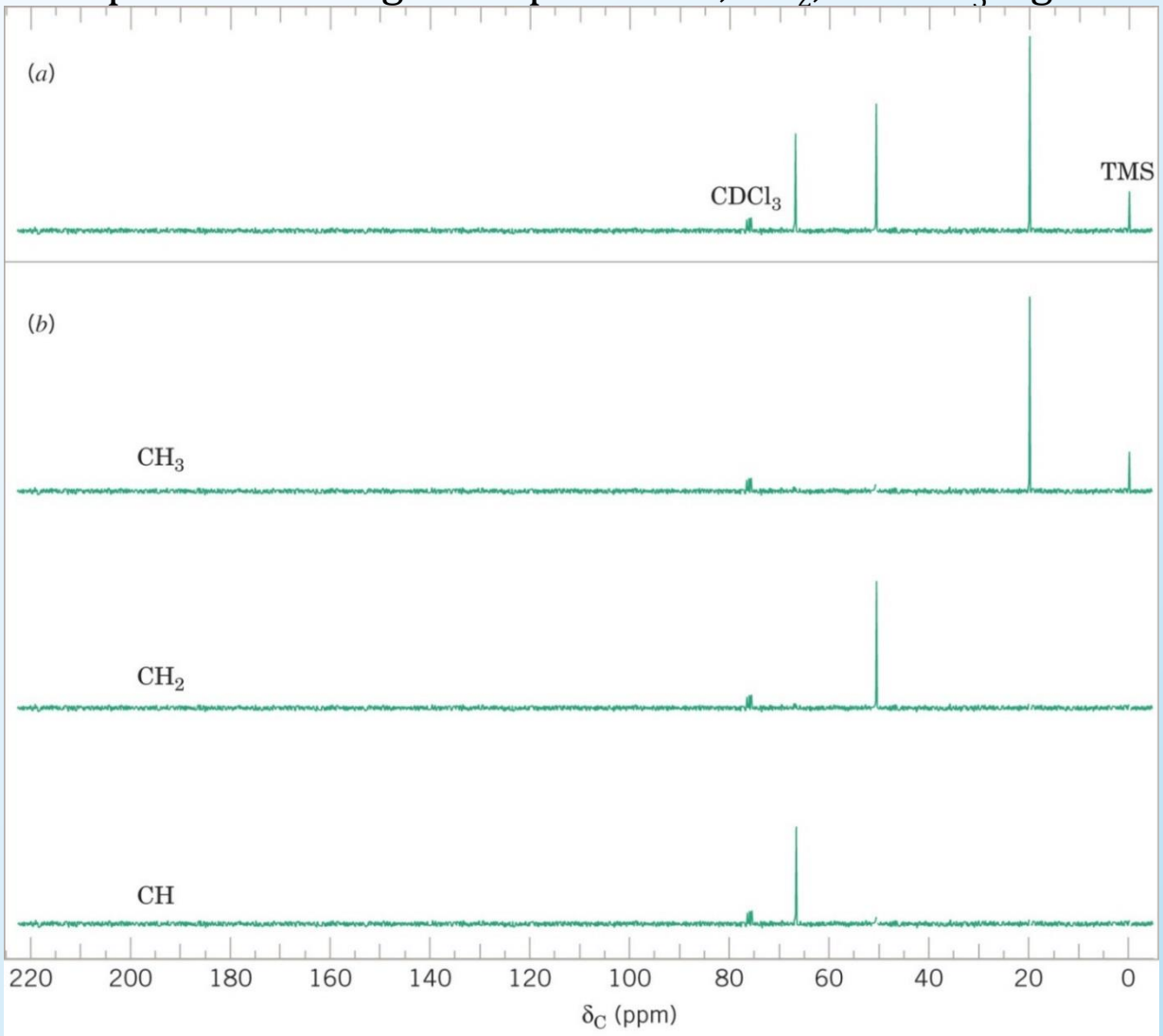


DEPT for C-13 NMR

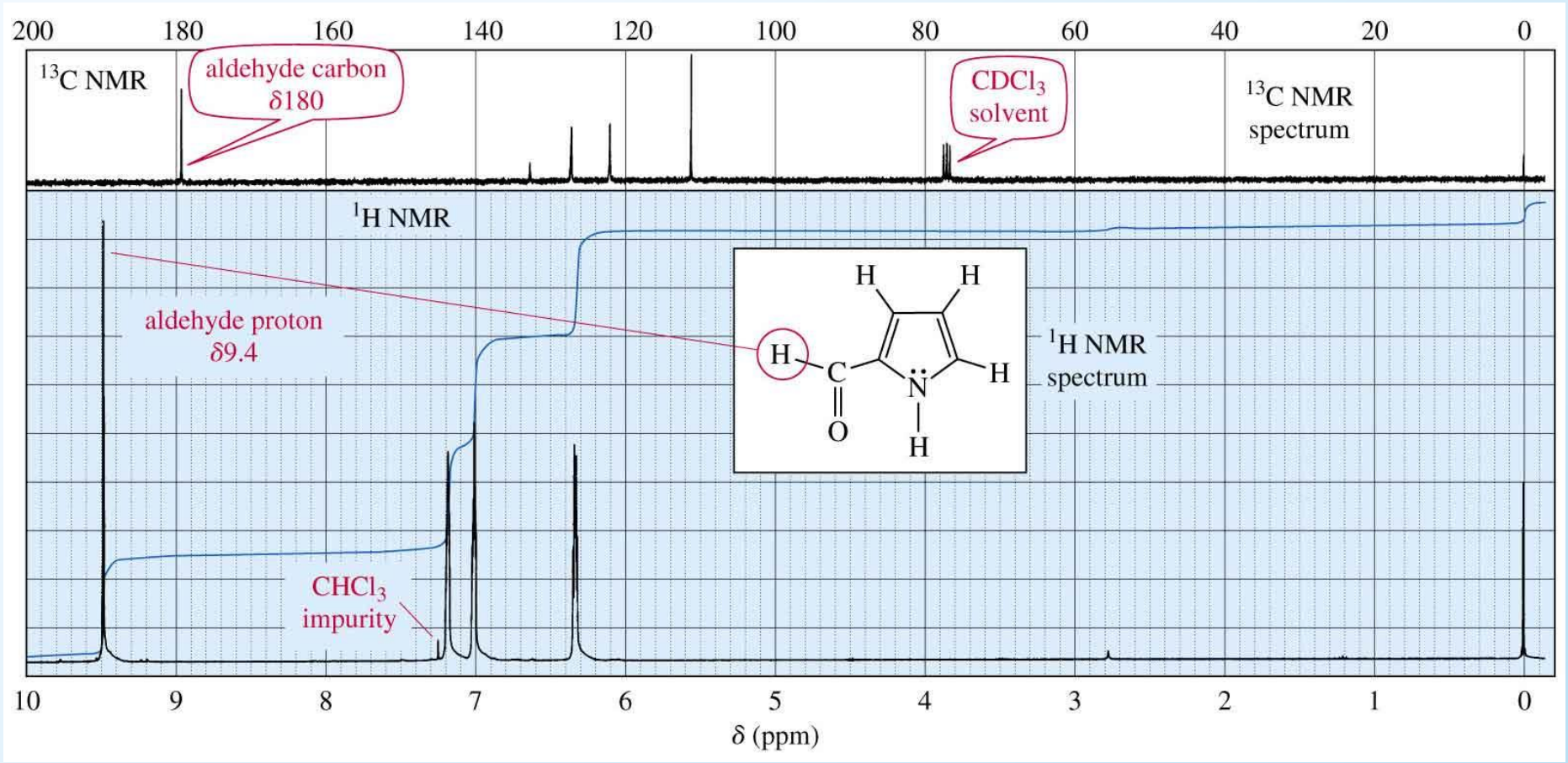
Example: 1-chloro-2-propanol

(a) The broadband decoupled spectrum and

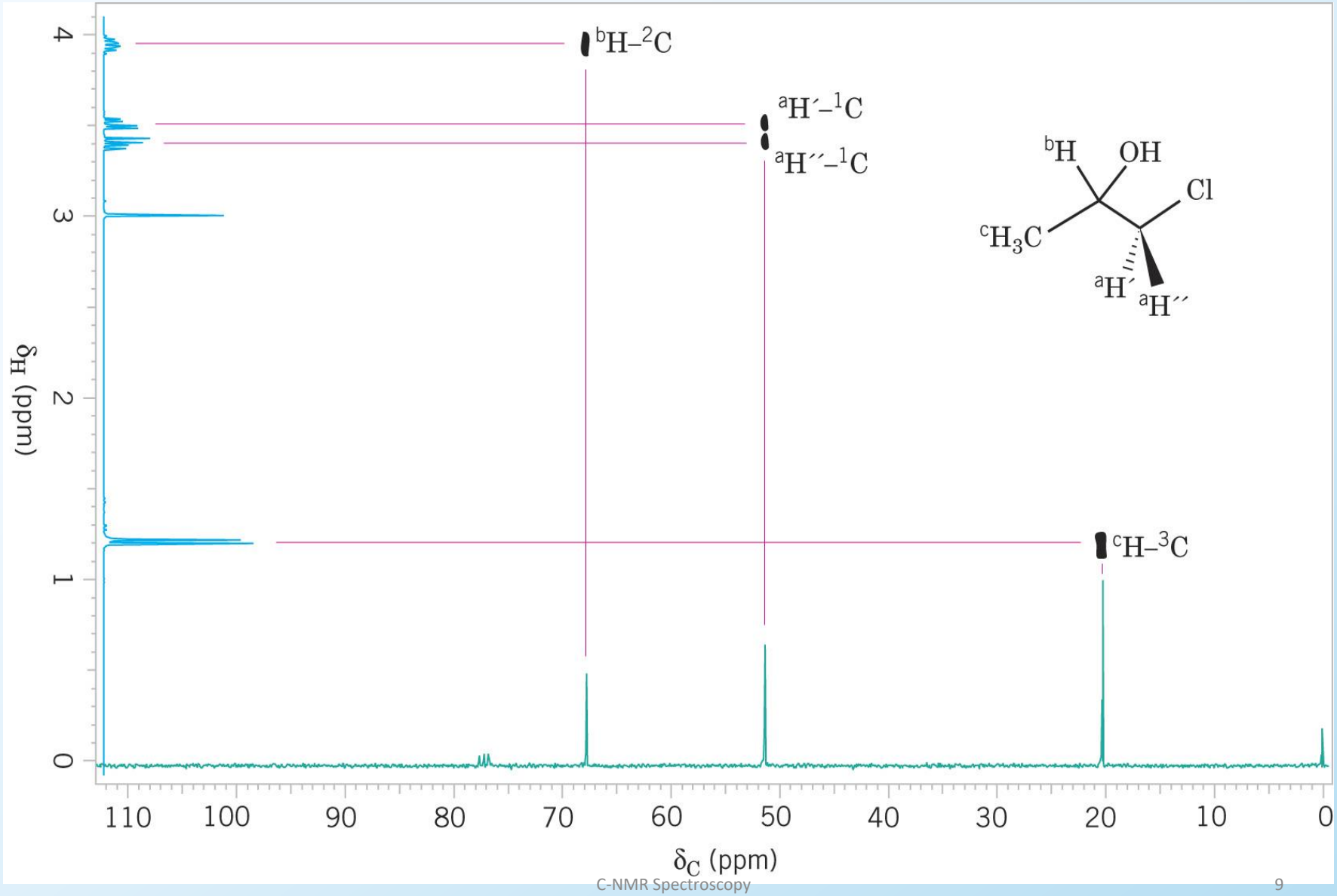
(b) a set of DEPT spectra showing the separate CH, CH_2, and CH_3 signals



Combined ^{13}C and ^{1}H Spectra



H and C coupled NMR (HETCOR)



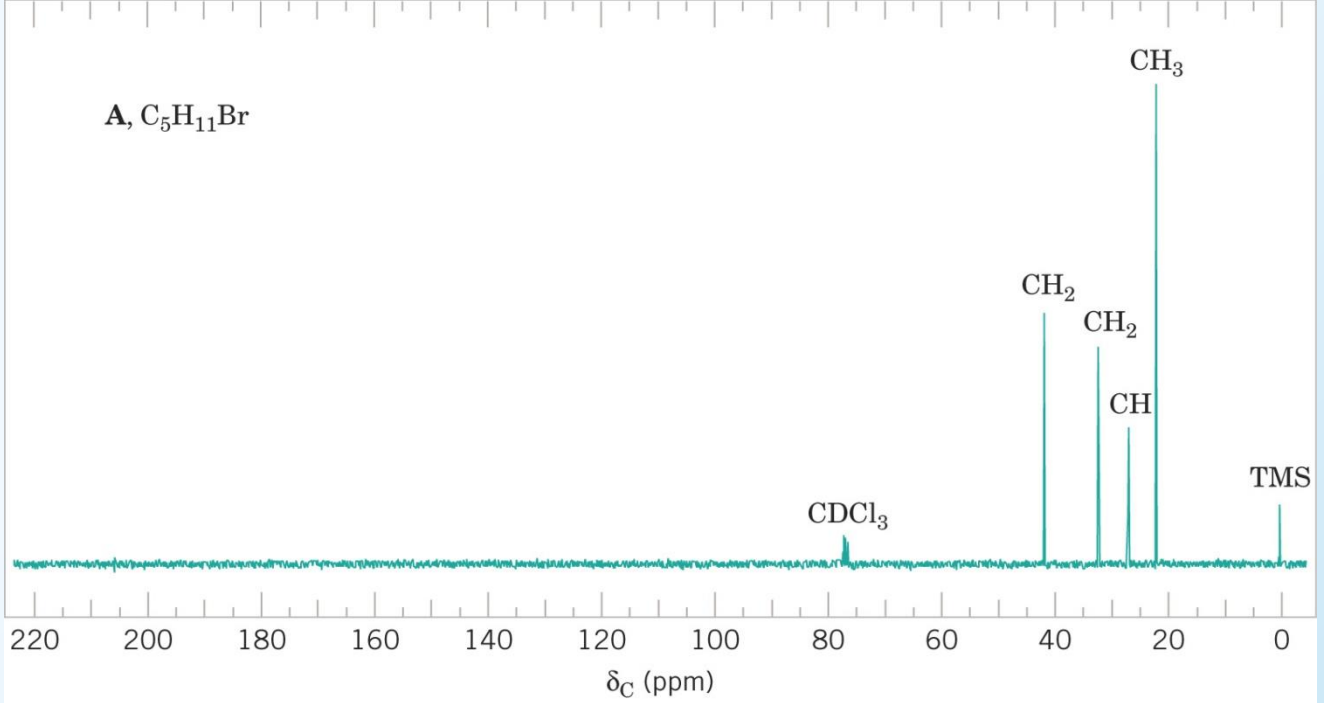
Spin-Spin Splitting

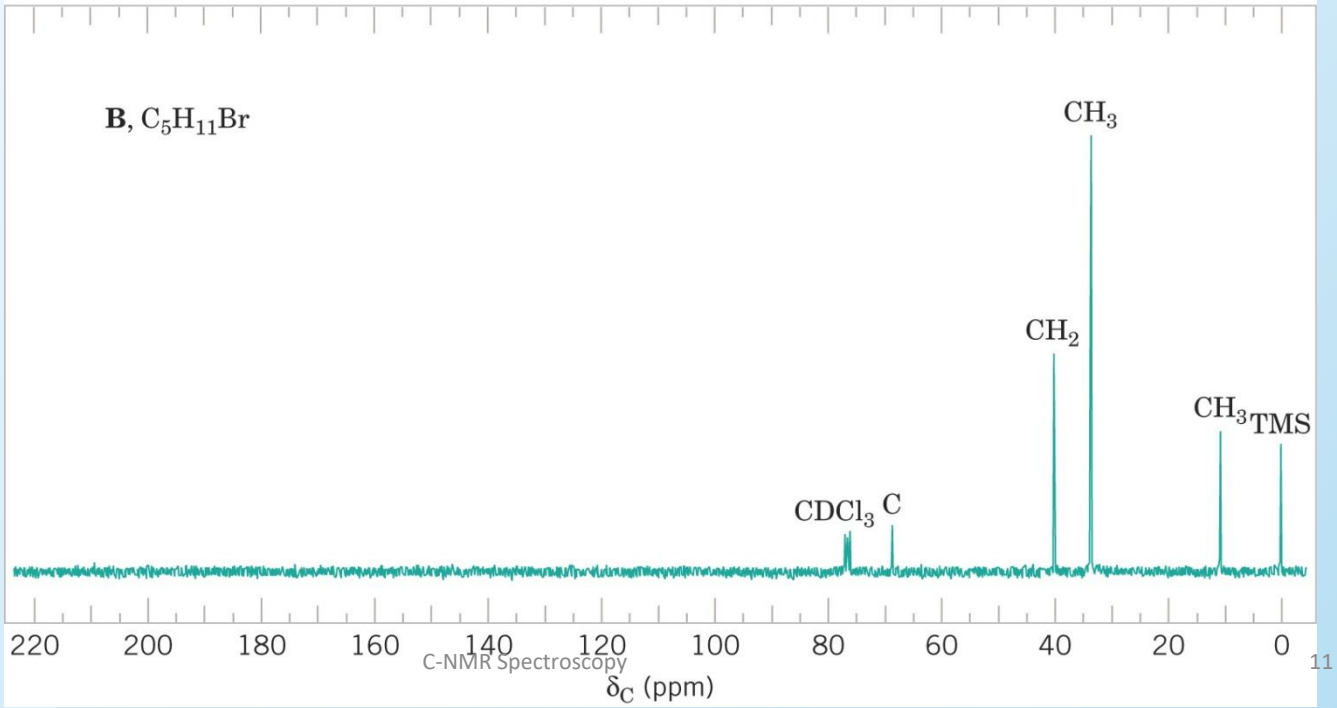
- It is unlikely that a ^{13}C would be adjacent to another ^{13}C, so splitting by carbon is negligible.
- ^{13}C will magnetically couple with attached protons and adjacent protons.
- These complex splitting patterns are difficult to interpret.

Interpreting ^{13}C NMR

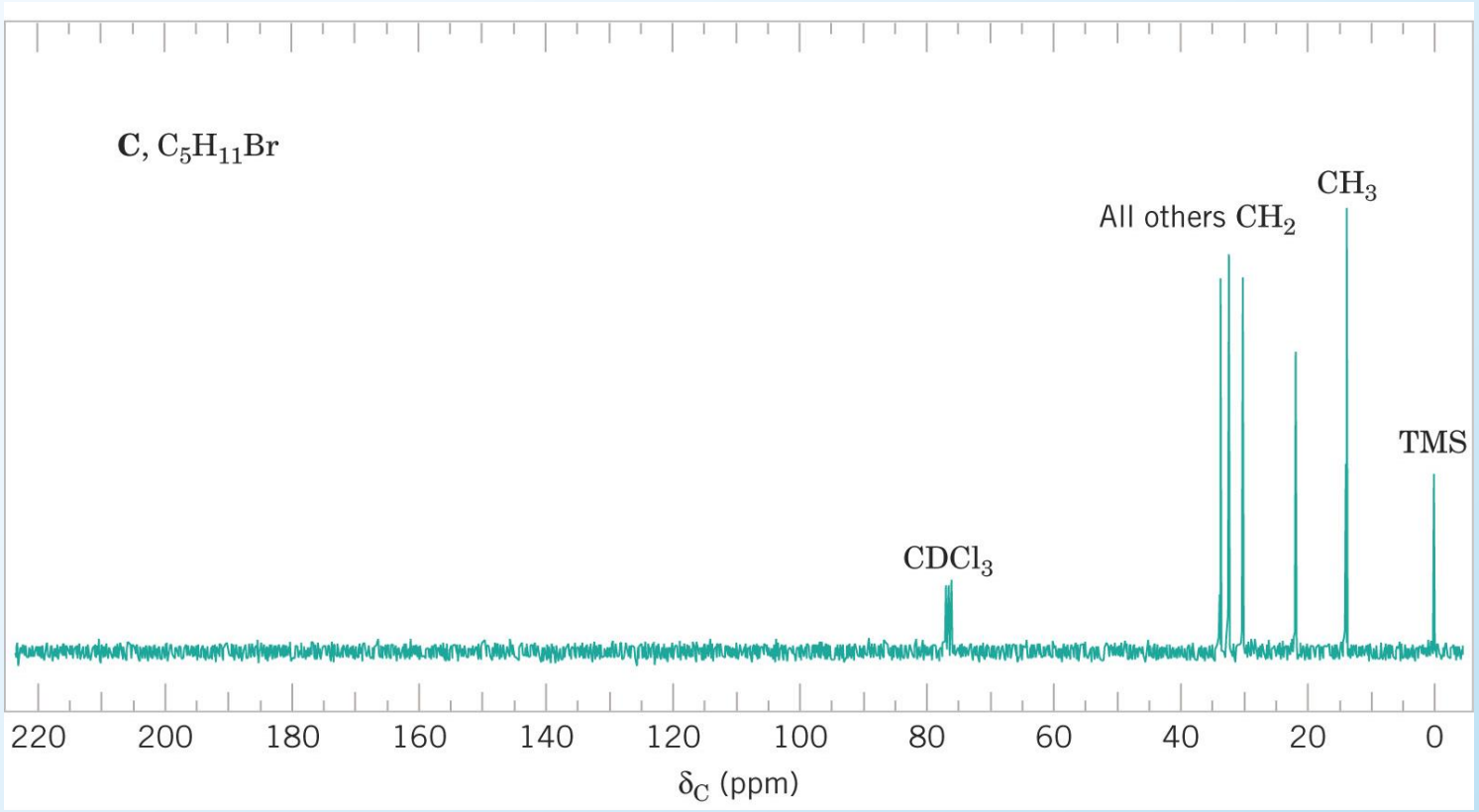
- The number of different signals indicates the number of different kinds of carbon.
- The location (chemical shift) indicates the type of functional group.
- The peak area indicates the numbers of carbons (if integrated).
- The splitting pattern of off-resonance decoupled spectrum indicates the number of protons attached to the carbon.

Problems


A, $C_5H_{11}Br$
CH_3
CH_2
CH_2
CH
TMS
$CDCl_3$
220 200 180 160 140 120 100 80 60 40 20 0
δ_C (ppm)


B, $C_5H_{11}Br$
CH_3
CH_2
CH_3
TMS
$CDCl_3$
C
220 200 180 160 140 120 100 80 60 40 20 0
δ_C (ppm)

Problem


C, $C_5H_{11}Br$
CH_3
All others CH_2
TMS
$CDCl_3$
220
200
180
160
140
120
100
80
60
40
20
0
δ_C (ppm)

Key Concepts

- Chemical shifts
- Interpret a C-13 NMR
- Predict signals for C-13 NMR for a compound