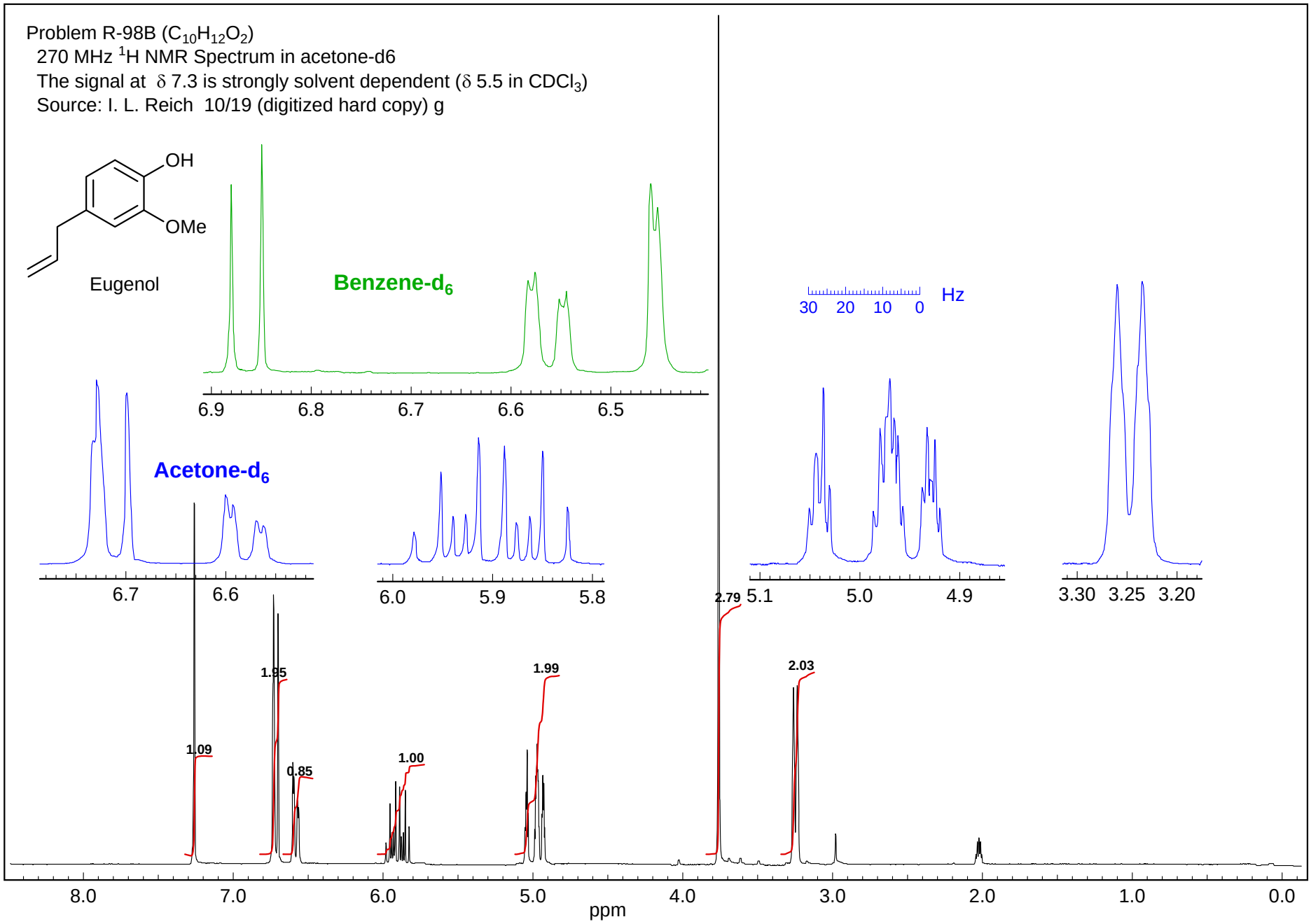
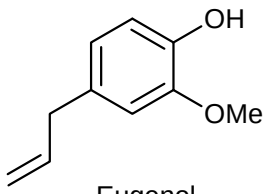


PLT ex-1-1998-g.plt

Problem R-98B

67.5 MHz ^{13}C NMR Spectra in $CDCl_3$

Source: Craig Puckett/Reich (digitized hard copy) 10/19

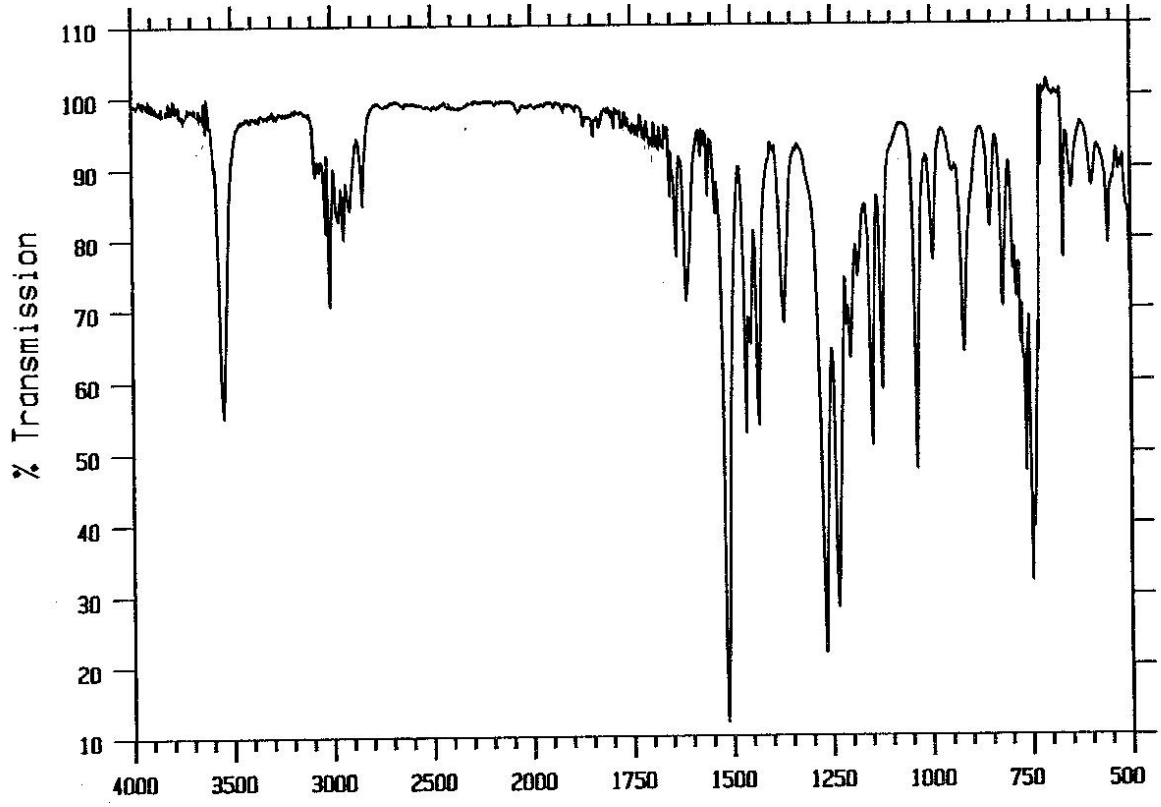


Eugenol

Problem R-98B

FT-IR Spectrum in CCl_4

Source: I. L. Reich 10/19



PLT ex-1-1998-g.plt

Problem R-98B ($C_{10}H_{12}O_2$). Determine the structure (or part structure) of R-98B from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

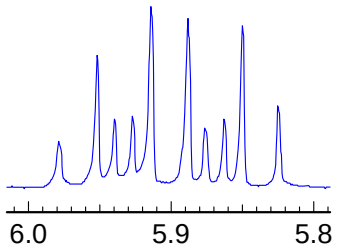
(a) DBE___

(b) What information can you obtain from the IR spectrum?

(c) Interpret the ^{13}C NMR spectrum. The DEPT 135 spectrum shows all CH and CH_3 peaks as positive, and CH_2 peaks negative, the DEPT 90 is CH only. Identify what kind of carbon each signal corresponds to, and write possible part structures.

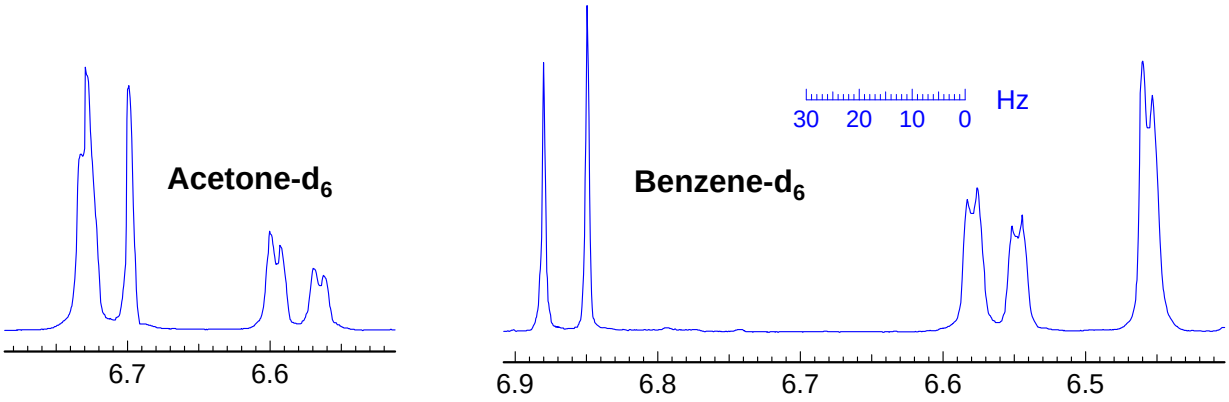
No	ppm	Type of C (e.g. sp^3 CH_2) and/or part structures (e.g. N-CH_2)
1	146.3	_____
2	143.6	_____
3	137.6	_____
4	131.5	_____
5	120.8	_____
6	115.0	_____
7	114.2	_____
8	111.0	_____
9	55.3	_____
10	39.4	_____

(d) Analyze the multiplet at δ 5.9 in the 270 MHz ^{1}H NMR spectrum (shown below). Report multiplicity, coupling constants and part structure you could obtain from the signal. Draw a coupling tree.



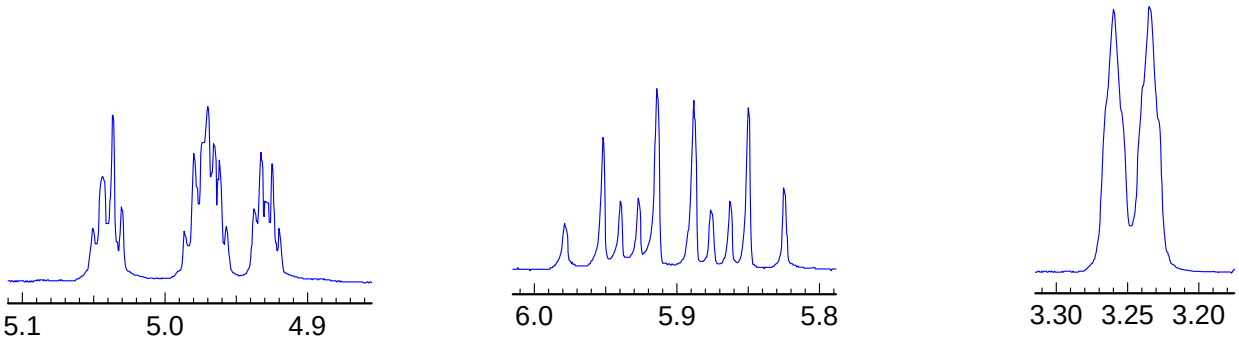
PLT ex-1-1998-g.plt

(e) The peaks between δ 6 and δ 7 have been provided both in acetone-d6 and in benzene-d6 solution. Do an analysis of the pattern in each solvent (give J and δ), and draw the part structure identified by the pattern. Label the part structure to indicate which signals correspond to each proton. (Do not attempt to explain the solvent effect.)



Part structure:

(f) Analyze the remainder of the signals in the ^{1}H spectrum. Label structure fragments with J and δ values.

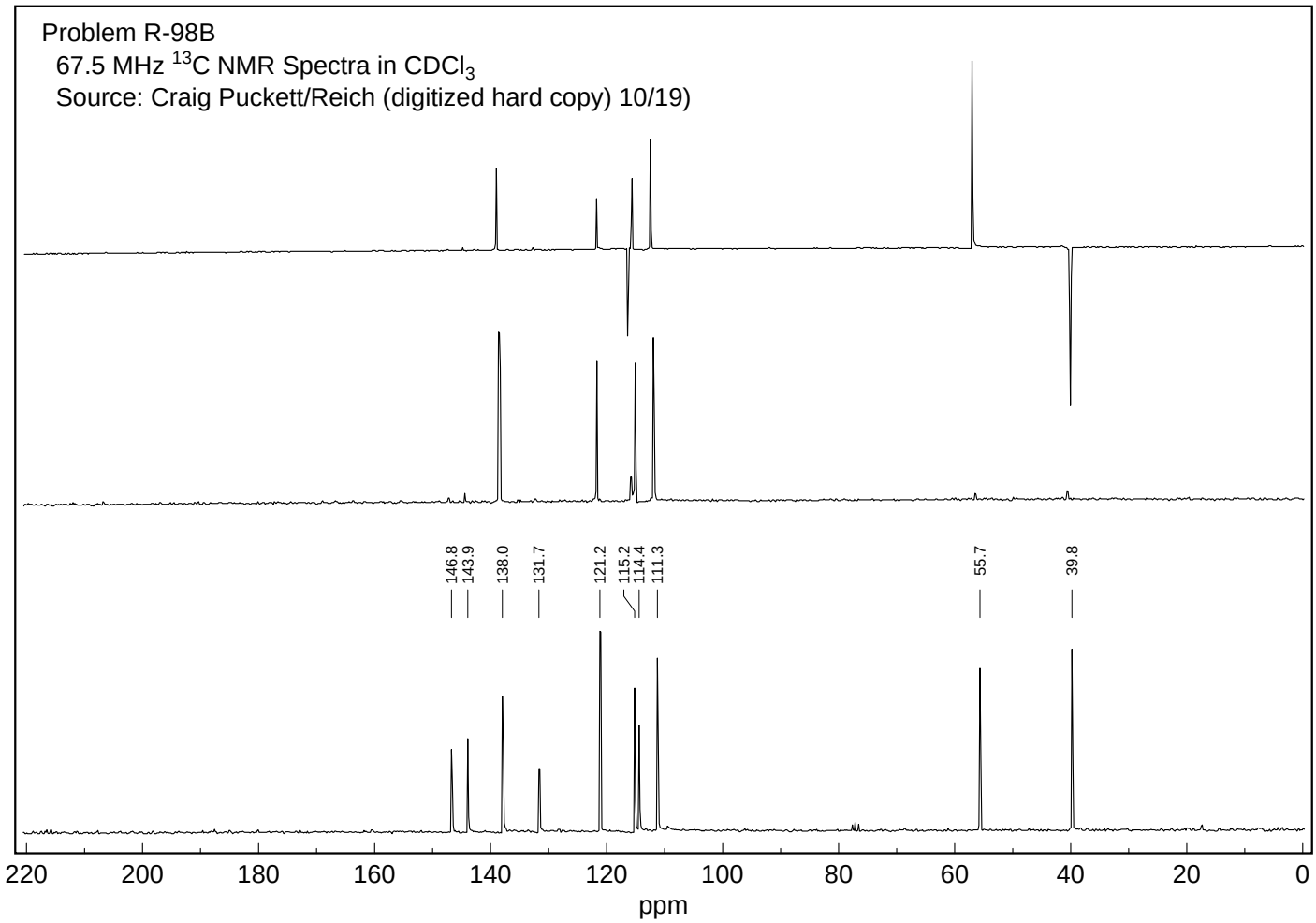


(g) The data provided do not allow a complete structure assignment. Draw all likely structures below. Do a chemical shift calculation for the proton you have assigned to the multiplet at δ 6.6 in the acetone spectrum for each possible structure, and label the structure with the calculated value (do not try to calculate all of the proton shifts). Circle the most likely structure.

Problem R-98B

67.5 MHz ^{13}C NMR Spectra in $CDCl_3$

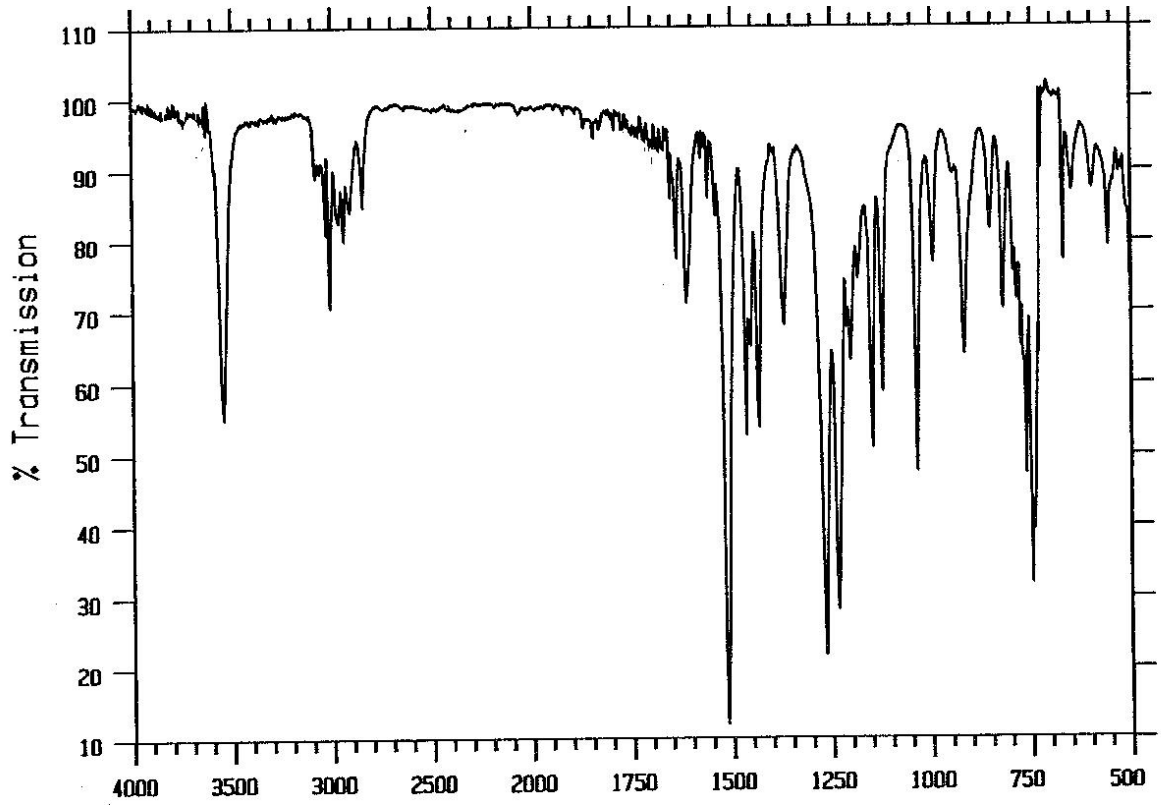
Source: Craig Puckett/Reich (digitized hard copy) 10/19



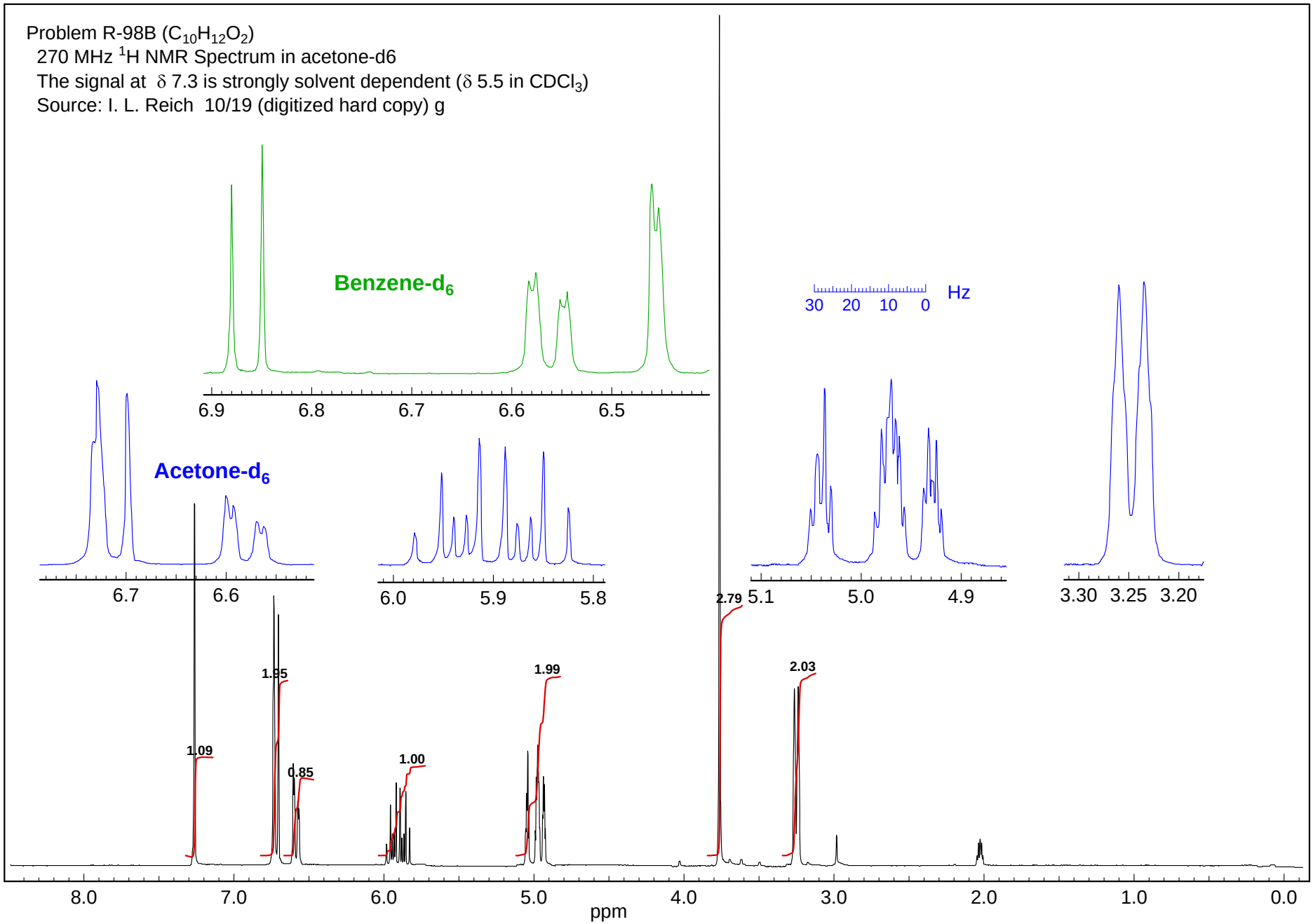
Problem R-98B

FT-IR Spectrum in CCl_4

Source: I. L. Reich 10/19



PLT ex-1-1998-g.plt



PLT ex-1-1998-g.plt

Problem R-98B ($C_{10}H_{12}O_2$). Determine the structure (or part structure) of R-98B from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

2 (a) DBE 5

(b) What information can you obtain from the IR spectrum?

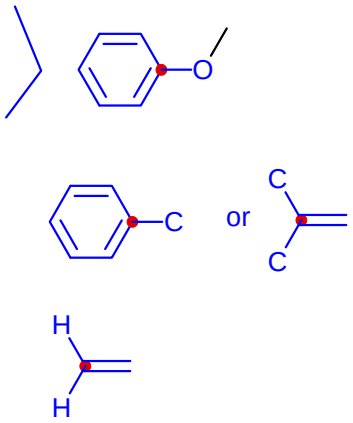
3600 cm^{-1} sharp OH - perhaps intramolecularly H-bonded

No carbonyl 1600-1800 cm^{-1}

3 No acetylene, allene

(c) Interpret the ^{13}C NMR spectrum. The DEPT 135 spectrum shows all CH and CH_3 peaks as positive, and CH_2 peaks negative, the DEPT 90 is CH only. Identify what kind of carbon each signal corresponds to, and write possible part structures.

No	ppm	Type of C (e.g. sp^3 CH_2) and/or part structures (e.g. N-CH_2)
1	146.3	s
2	143.6	s
3	137.6	d Arom, vinyl CH
4	131.5	s
5	120.8	d Arom, vinyl CH
6	115.0	t
7	114.2	d Arom, vinyl CH
8	111.0	d Arom, vinyl CH
9	55.3	q CH_3-O
10	39.4	t C-CH_2-C



There are 11 C-H identified from the multiplicities of the ^{13}C NMR spectrum. There is one H missing - must be an O-H (also seen in IR)

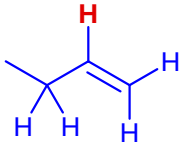
There are 10 different carbons - so no symmetry

4

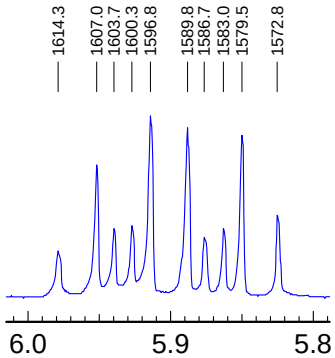
(d) Analyze the multiplet at δ 5.9 in the 270 MHz ^{1}H NMR spectrum (shown below). Report multiplicity, coupling constants and part structure you could obtain from the signal. Draw a coupling tree.

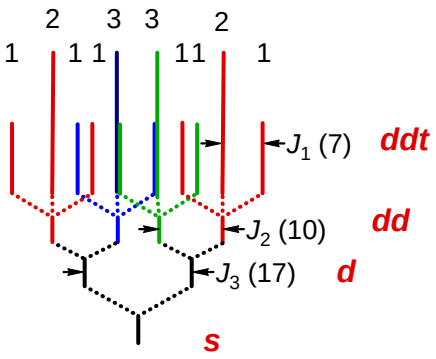
- One vinyl proton (from δ)
- ddt, J = 17, 10, 7 Hz

this defines:

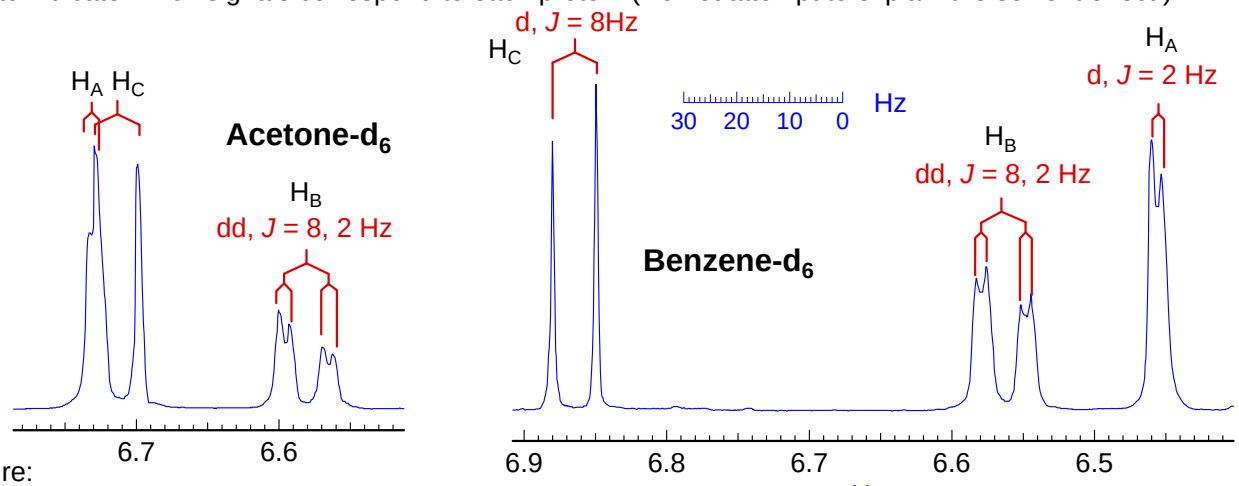


5



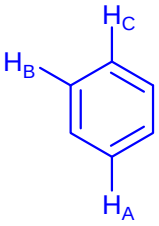


(e) The peaks between δ 6 and δ 7 have been provided both in acetone-d6 and in benzene-d6 solution. Do an analysis of the pattern in each solvent (give J and δ), and draw the part structure identified by the pattern. Label the part structure to indicate which signals correspond to each proton. (Do not attempt to explain the solvent effect.)



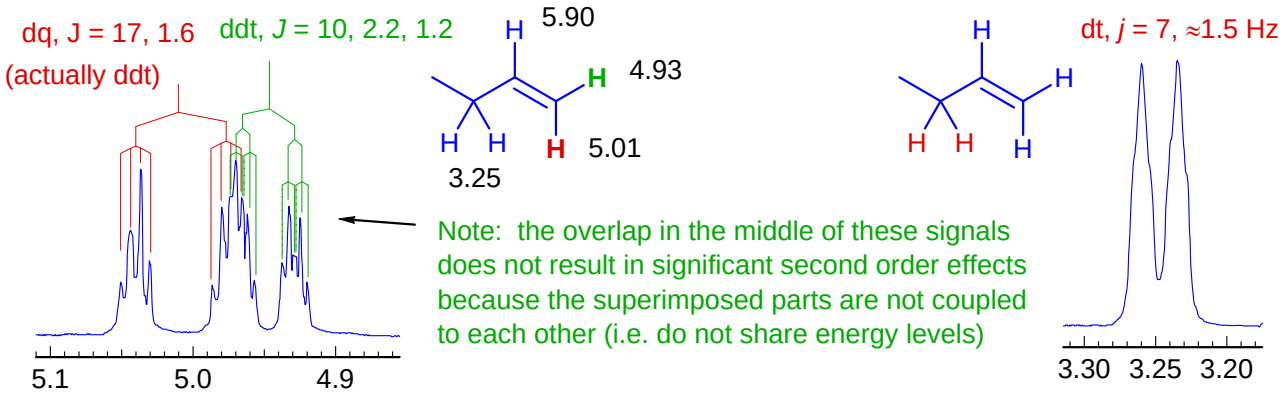
5

Part structure:



- This must be a 1,2,4-trisubsituted benzene (OH, OR)
- The sunstituents must be electrom donating, from δ

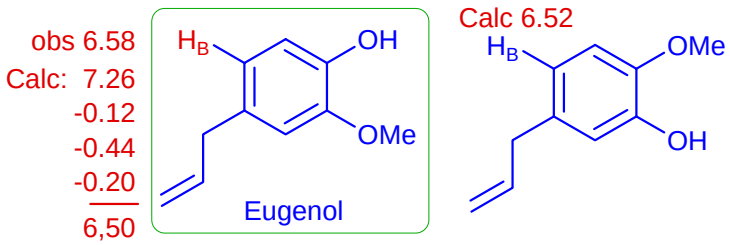
(f) Analyze the remainder of the signals in the ^{1}H spectrum. Label structure fragments with J and δ values.



4

Note: the overlap in the middle of these signals does not result in significant second order effects because the superimposed parts are not coupled to each other (i.e. do not share energy levels)

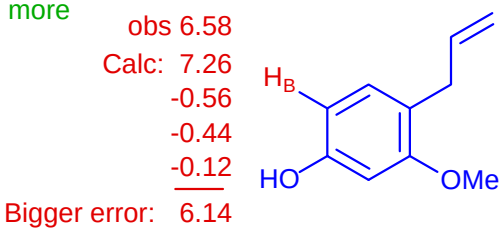
(g) The data provided do not allow a complete structure assignment. Draw all likely structures below. Do a chemical shift calculation for the proton you have assigned to the multiplet at δ 6.6 in the acetone spectrum for each possible structure, and label the structure with the calculated value (do not try to calculate all of the proton shifts). Circle the most likely structure.

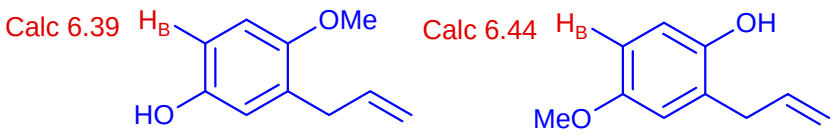


7

These structures fit chemical shift calculations best - hard to distinguish

Other structures fit coupling, but calculated chemical shifts are off a bit more

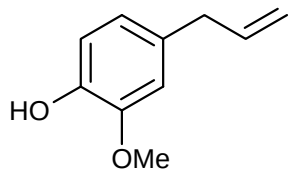




$C_{10}H_{12}O_2$

200 MHz ^{1}H NMR spectrum in **C_6D_6**

Source: Ieva L. Reich (digitized hard copy) g

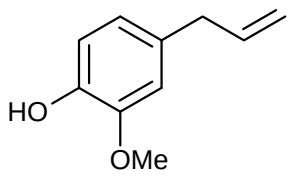


PLT ex-1-1998-g.plt

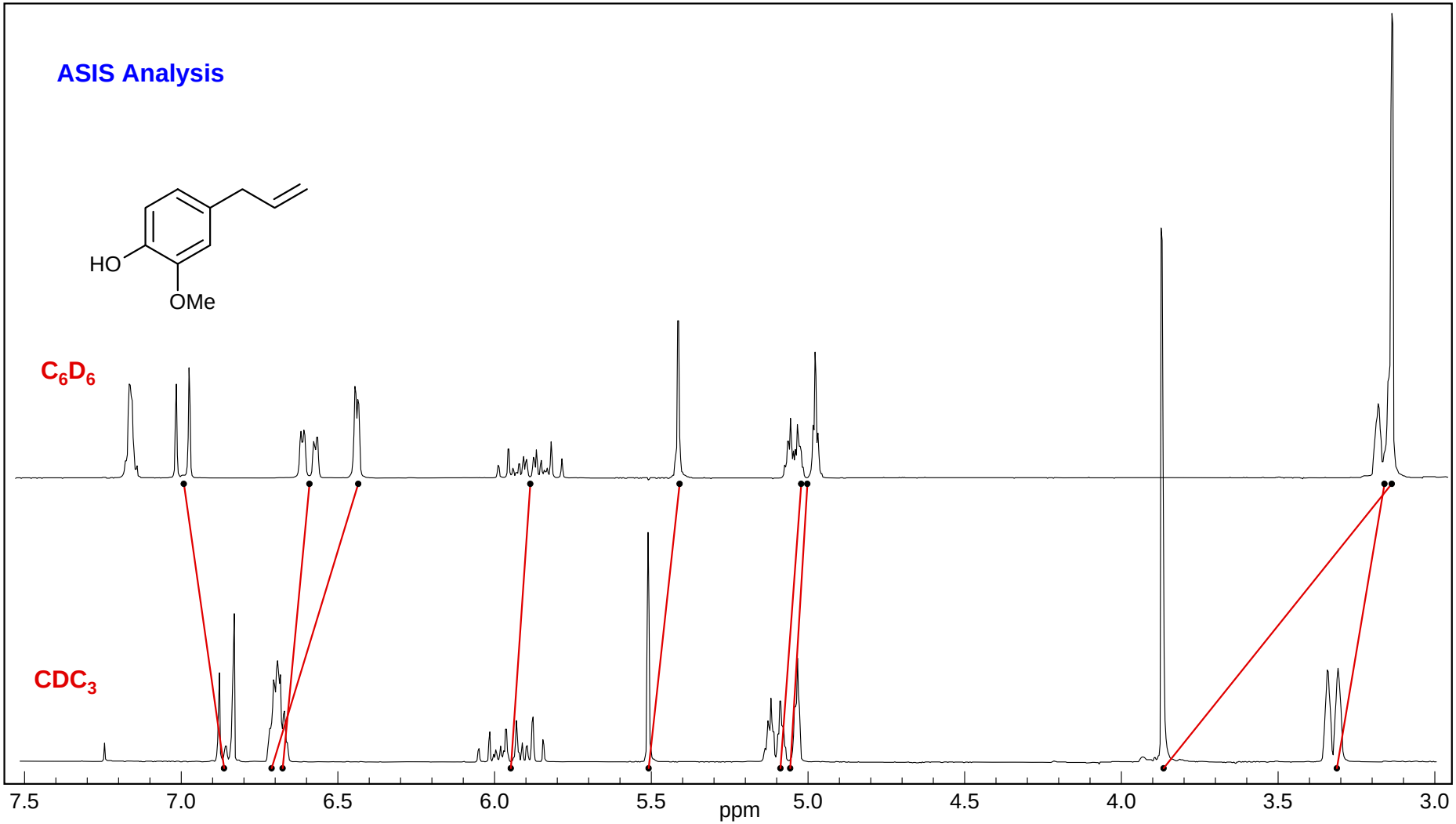
$C_{10}H_{12}O_2$

200 MHz ^{1}H NMR spectrum in **$CDCl_3$**

Source: Ieva L. Reich (digitized hard copy) g



PLT ex-1-1998-g.plt



PLT ex-1-1998-g.plt