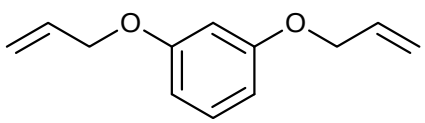
Problem R-90F ($C_{12}H_{14}O_2$)

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

Source: Craig Puckett/Reich 11/20 (digitized hard copy) g

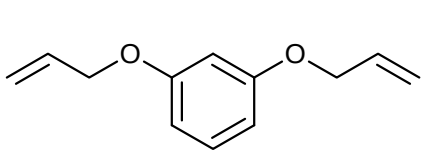


PLT pr-087-to-94.plt

Problem R-90F ($C_{12}H_{14}O_2$)

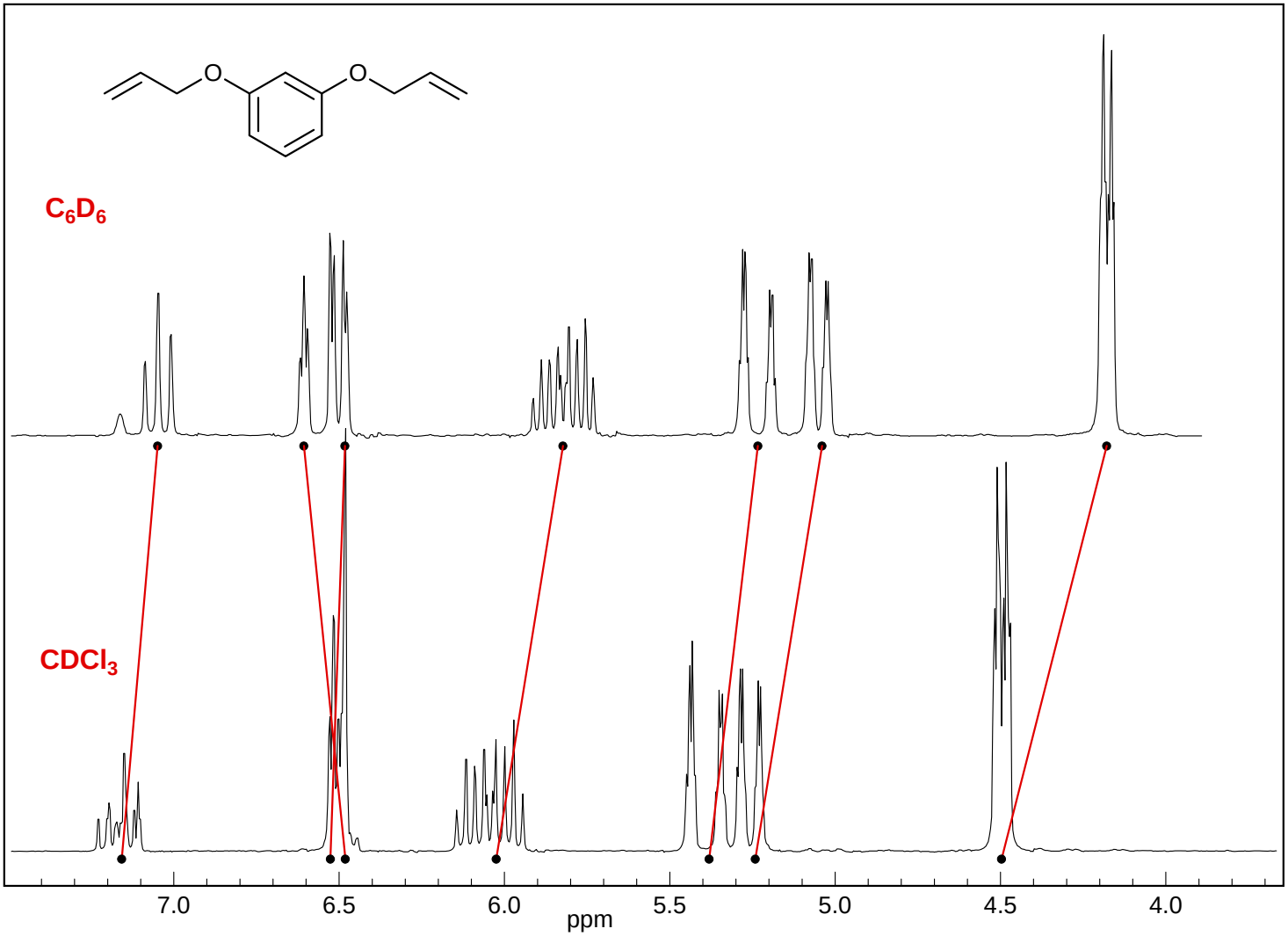
200 MHz ^{1}H NMR spectrum in **$CDCl_3$**

Source: Craig Puckett/Reich 11/20 (diitized hard copy) g



PLT pr-087-to-94.plt

Aromatic Solvent Induced Shifts

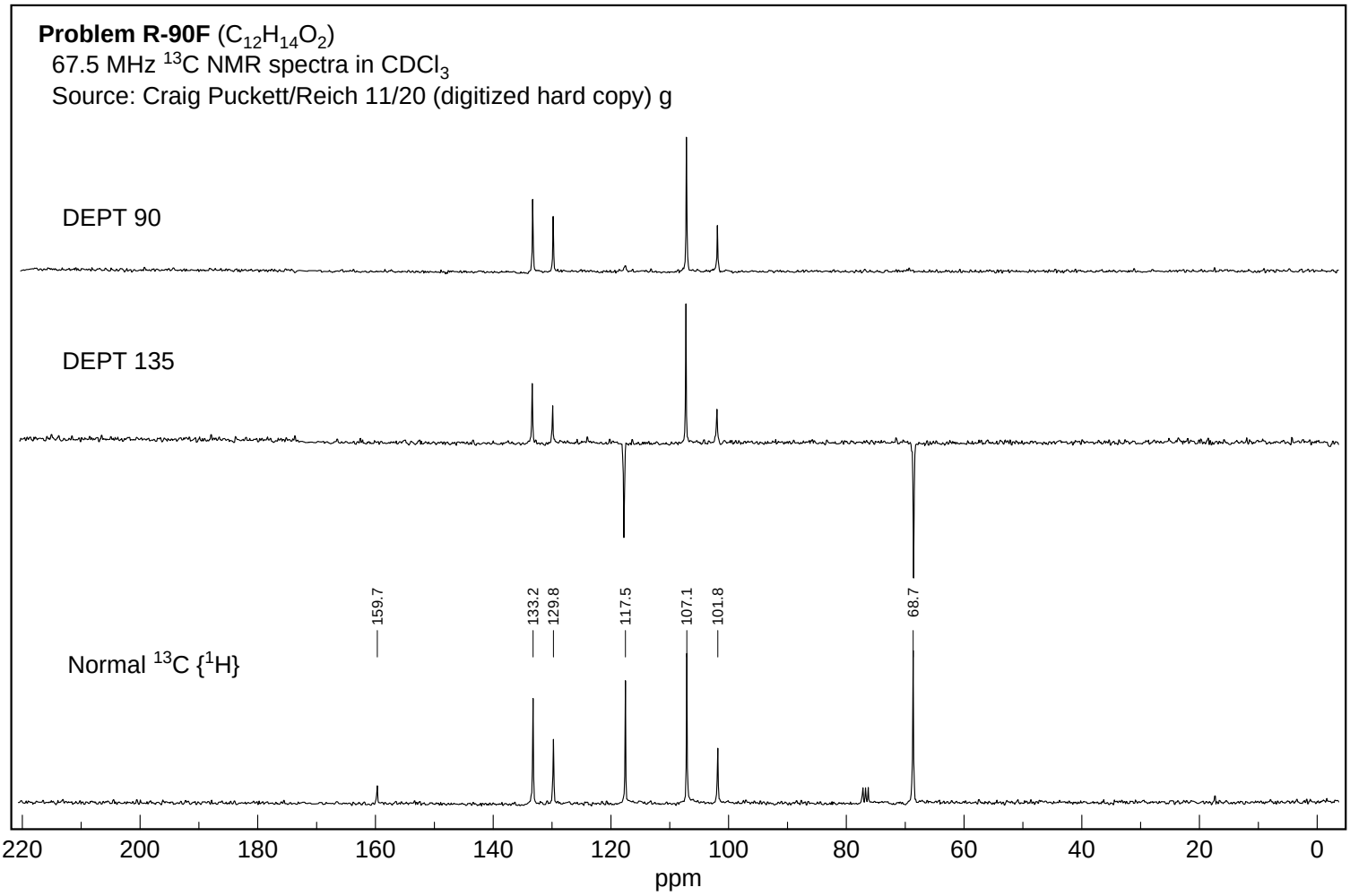


PLT pr-087-to-94.plt

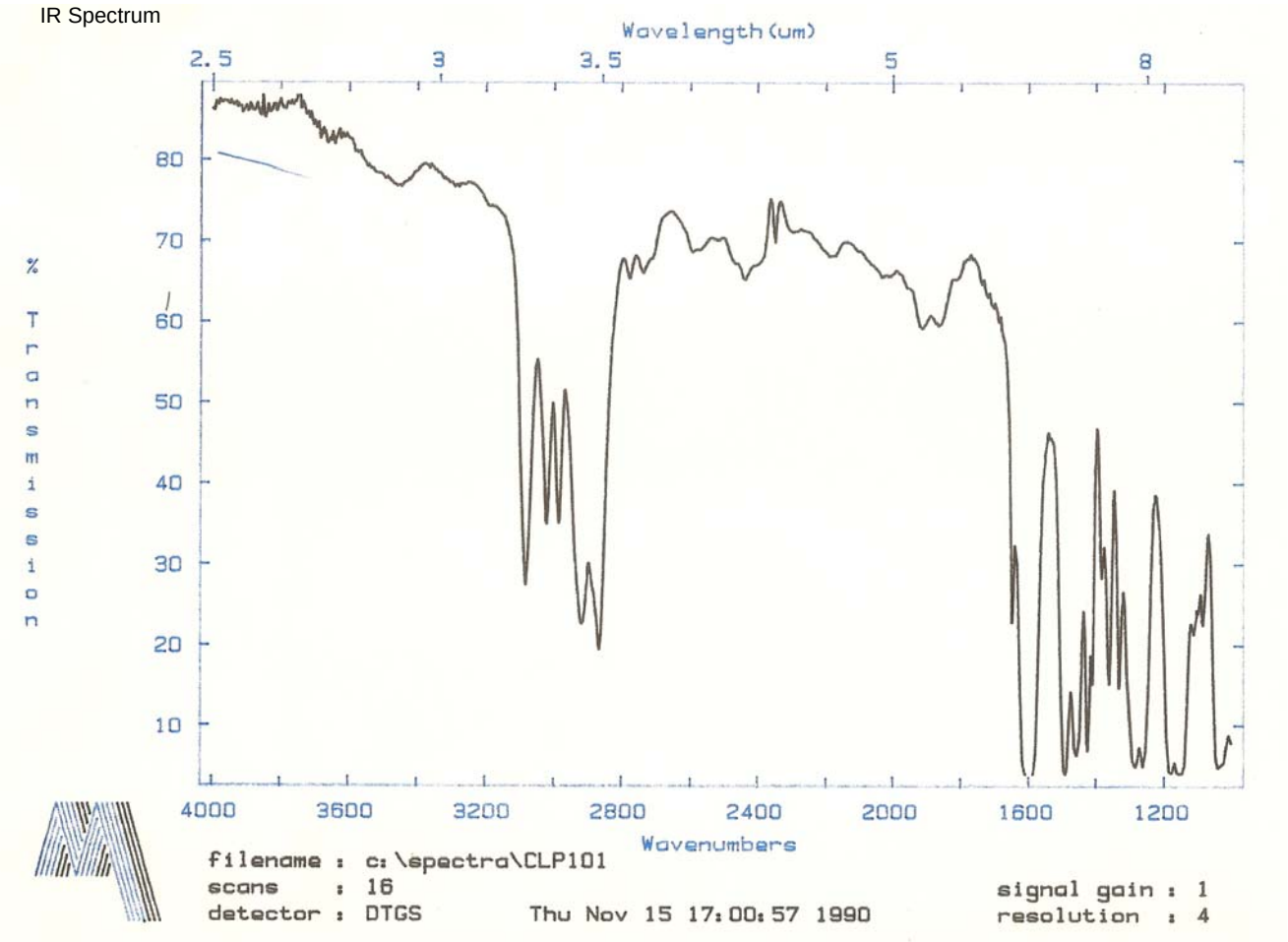
Problem R-90F ($C_{12}H_{14}O_2$)

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

Source: Craig Puckett/Reich 11/20 (digitized hard copy) g



IR Spectrum



PLT pr-087-to-94.plt

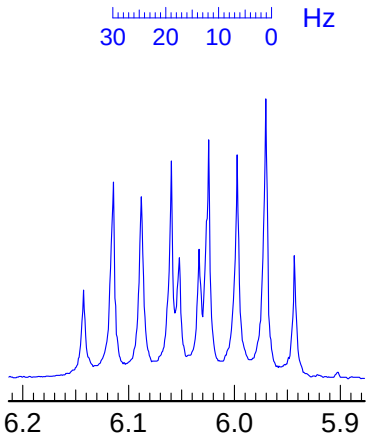
Problem R-90F ($C_{12}H_{14}O_2$). In this problem you are required to determine a structure from the ^{13}C and ^{1}H spectra of a compound. To aid in your analysis, ^{1}H spectra are provided in both $CDCl_3$ and C_6D_6 solution.

(a) DBE____.

(b) Summarize the information you were able to obtain from your analysis of the ^{13}C NMR spectra. Identify part structures that are present in the molecule.

(c) Interpret the signals at δ 6.3 to δ 7.3. What do these signals tell you about the structure? Draw a part structure, and label it with δ and *J* values taken from the C_6D_6 spectrum. Why is the downfield multiplet so much simpler in the C_6D_6 spectrum?

(d) The multiplet at δ 6.0 in the ^{1}H NMR spectrum in $CDCl_3$ is reproduced below. Do a first-order analysis, report multiplicity and *J* values. What does this signal tell you about the structure?



(e) Interpret the signals δ 5.2 to δ 5.5.

(f) Interpret the signals at δ 4.5. Draw below the structure of **R-90F**.

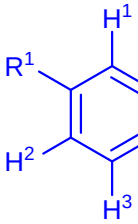
Problem R-90F ($C_{12}H_{14}O_2$). In this problem you are required to determine a structure from the ^{13}C and ^{1}H spectra of a compound. To aid in your analysis, ^{1}H spectra are provided in both $CDCl_3$ and C_6D_6 solution.

(a) DBE 6.

(b) Summarize the information you were able to obtain from your analysis of the ^{13}C NMR spectra. Identify part structures that are present in the molecule.

- the molecule must have some symmetry - only 7 ^{13}C NMR signals for 12 carbons
- the CH_2 at δ 117 must be part of a terminal double bond - $H_2C{=}C$
- probably an aromatic compound with symmetrical substitution
- the peak at 68.7 (t) must be O-CH_2

(c) Interpret the signals at δ 6.3 to δ 7.3. What do these signals tell you about the structure? Draw a part structure, and label it with δ and *J* values taken from the C_6D_6 spectrum. Why is the downfield multiplet so much simpler in the C_6D_6 spectrum?



H^1 t, δ 6.6, *J* = 2 Hz (2 meta *J*)

δ 6.5, dd, *J* = 8, 2 Hz (one ortho, one meta *J*)

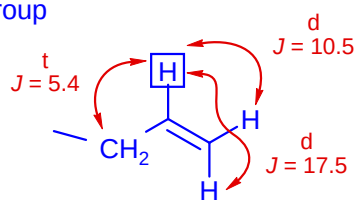
δ 7.1, t, *J* = 8 Hz (two ortho *J*)

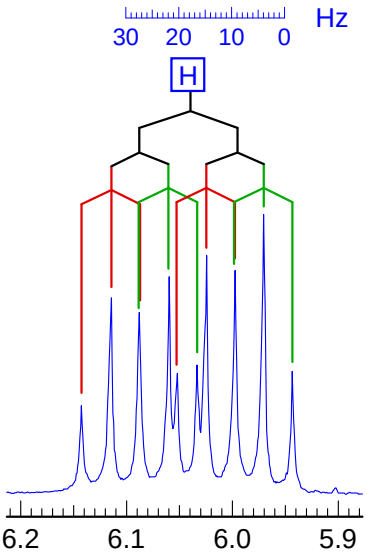
- this must be a meta-disubstituted benzene, with $R^1 = R^2$. The groups must be electron donating, since all protons are upfield of benzene (at δ 7.3)

- in $CDCl_3$ the aromatic signals are more complicated because H^1 and H^2 are strongly coupled (Δδ comparable to or less than *J*). Thus H^3 shows virtual coupling effects.

(d) The multiplet at δ 6.04 in the ^{1}H NMR spectrum in $CDCl_3$ is reproduced below. Do a first-order analysis, report multiplicity and *J* values. What does this signal tell you about the structure?

This is a ddt, *J* = 17.5, 10.5 and 5.4 Hz, which uniquely defines the the middle proton of an allyl group





(e) Interpret the signals δ 5.2 to δ 5.5 in $CDCl_3$.

These are the terminal vinyl protons of the allyl group:

δ 5.25, dq, *J* = 10.9, 1.6 Hz

δ 5.39, dq, *J* = 17.8, 1.6 Hz

- The ^{4}J allylic and ^{2}J gem coupling are almost the same, hence the quartet

- since each of the allyl signals is double intensity, there must be two of these

(f) Interpret the signals at δ 4.5. Draw below the structure of **R-90F**.

δ 4.5, dt, *J* = 5.5, 1.6 Hz