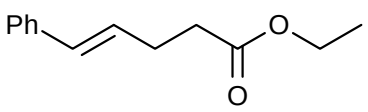
Problem R-08D ($C_{13}H_{16}O_2$)

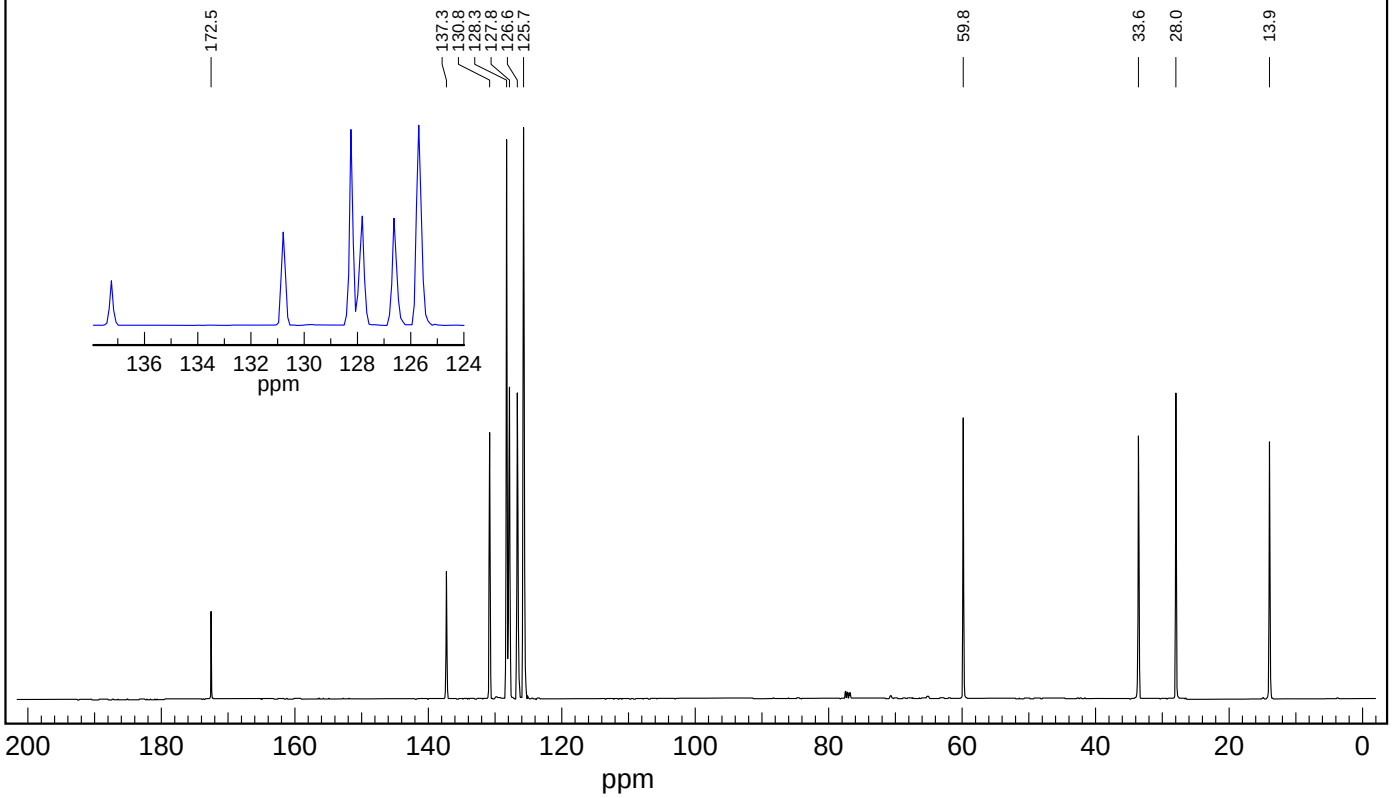
270 MHz ^{1}H NMR Spectrum in $CDCl_3$

Source: J. Ringer/Reich (digitized hard copy) g

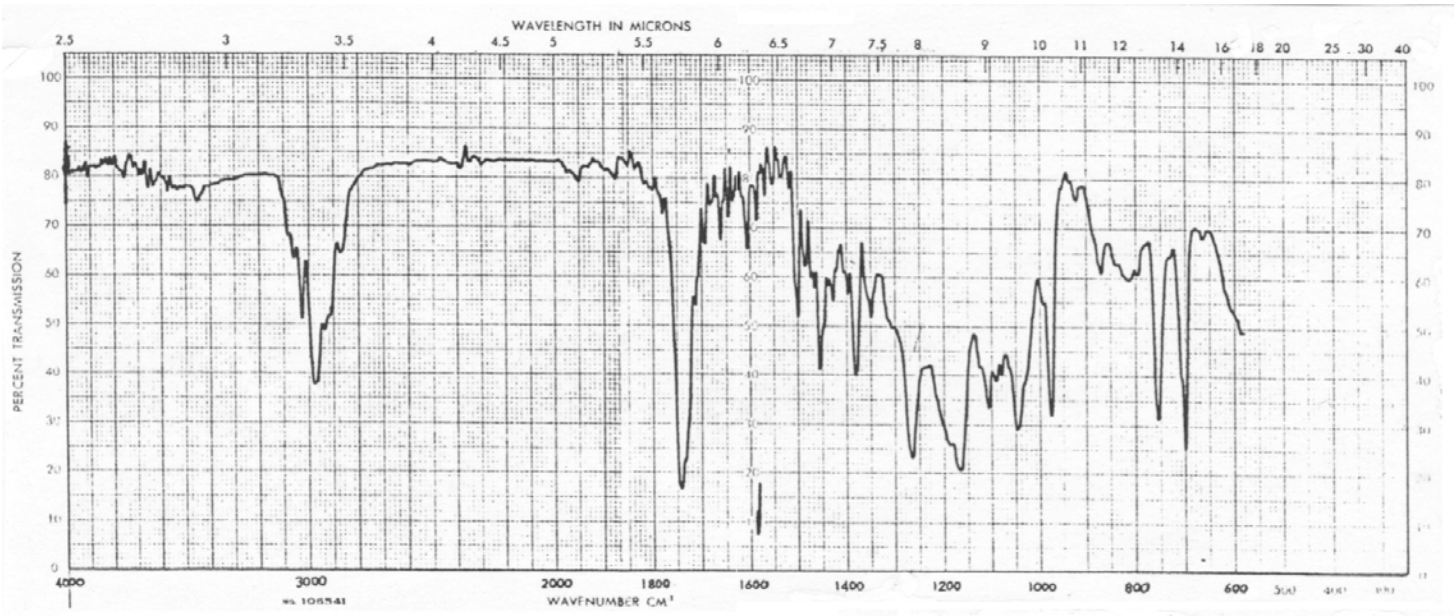


PLT ex-1-2008-09-gq.plt

Problem R-08D ($C_{13}H_{16}O_2$)
125.76 MHz ^{13}C NMR Spectrum in $CDCl_3$
Source: J. Ringer/Reich



Problem R-08D ($C_{13}H_{16}O_2$)
IR spectrum (neat)
Source: J. Ringer/Reich



PLT ex-1-2008-09-gq.plt

Problem R-08D. ($C_{13}H_{16}O_2$). Determine the structure (or part structure) of R-08D from the ^{1}H NMR, ^{13}C NMR and IR spectra provided. (30 points)

(a) DBE____ (b) What information can you obtain from the IR spectrum? List the data, and any conclusions you drew from it.

(b) Analyze the ^{1}H NMR signals. For each group of signals listed below report integration, multiplicity and coupling constants to the extent the signals are amenable to first order analysis, and the part structure each corresponds to. Use the standard reporting method (eg. δ 3.42, qd, J = 7,2, 1H)

1.2

2.4

4.1

6.2

6.4

7.2

(c) Interpret the ^{13}C NMR spectrum. Identify what kind of carbon each signal corresponds to, and write possible part structures.

No	ppm	Type and number of C (e.g. sp^3 CH_2) and/or part structures (e.g. N-CH_2)
1	172.5 (s)	
2	137.3 (s)	
3	130.8 (d)	
4	128.3 (d)	
5	127.8 (d)	
6	126.6 (d)	
7	125.7 (d)	
8	59.8 (t)	
9	33.6 (t)	
10	27.9 (t)	
11	13.9 (q)	

(d) Determine the structure of R-08D. If more than one structure is possible, show them, and circle your best choice.

30

Problem R-08D. ($C_{13}H_{16}O_2$). Determine the structure (or part structure) of R-08D from the ^{1}H NMR, ^{13}C NMR and IR spectra provided. (30 points)

2 (a) DBE 6 (b) What information can you obtain from the IR spectrum? List the data, and any conclusions you drew from it.

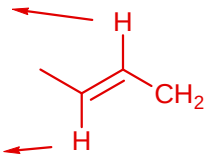
4 1750 cm^{-1} Carbonyl (unconjugated ester) 3020 cm^{-1} Ar-H stretch 1160 cm^{-1} C-O stretch

No triple bonds visible (C≡C) No OH peak

(b) Analyze the ^{1}H NMR signals. For each group of signals listed below report integration, multiplicity and coupling constants to the extent the signals are amenable to first order analysis, and the part structure each corresponds to. Use the standard reporting method (eg. δ 3.42, qd, J = 7,2, 1H)

1.2	t, J = 7Hz, 3H, $\underline{CH_3}$-CH_2	6.2	dt, J = 16, 7Hz, 1H
2.4	m, 4H, CH_2-CH_2 ?	6.4	broad d, J = 16, 1H
4.1	q, J = 7 Hz, 2H, CH_3-$\underline{CH_2}$-O	7.2	m, 5H, Ph?

10



(c) Interpret the ^{13}C NMR spectrum. Identify what kind of carbon each signal corresponds to, and write possible part structures.

No	ppm	Type and number of C (e.g. sp^3 CH_2) and/or part structures (e.g. N-CH_2)
1	172.5 (s)	Ester C=O
2	137.3 (s)	Aromatic C (quat)
3	130.8 (d)	Aromatic (vinyl) C-H
4	128.3 (d)	Aromatic (vinyl) C-H - double intensity (ortho,meta)
5	127.8 (d)	Aromatic (vinyl) C-H
6	126.6 (d)	Aromatic (vinyl) C-H
7	125.7 (d)	Aromatic (vinyl) C-H - double intensity (ortho,meta)
8	59.8 (t)	CH_2-O
9	33.6 (t)	CH_2
10	27.9 (t)	CH_2
11	13.9 (q)	CH_3

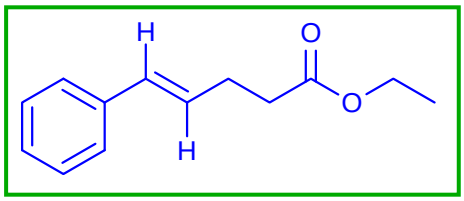
6

There are 2 carbon less than the formula, confirms that two peaks are double, very likely a phenyl group with double intensity o/m carbons

(d) Determine the structure of R-08D. If more than one structure is possible, show them, and circle your best choice.

Calc: 5.25-0.22+1.38 = 6.41 (obs:6.4)

8

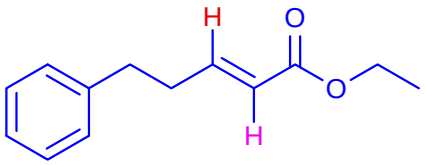


Calc: 5.25+0.36+0.45 = 6.06 (obs:6.2)

ΣΔδ = 0.15

1 bonus point for suggesting both structures

Calc: 5.25-0.45+0.80 = 6.50 (obs:6.2)



Calc: 5.25+0.80-0.22 = 5.83 (obs:6.4)

ΣΔδ = 0.9

- Proton shifts
- IR should be at 1720 cm^{-1} (not at 1750)
- ^{13}C carbonyl shift