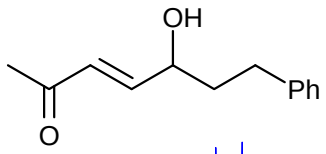
Problem R-09D ($C_{13}H_{16}O_2$)

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

Source: B. Gudmundsson-S. K. Shah/Reich (digitized hard copy) g

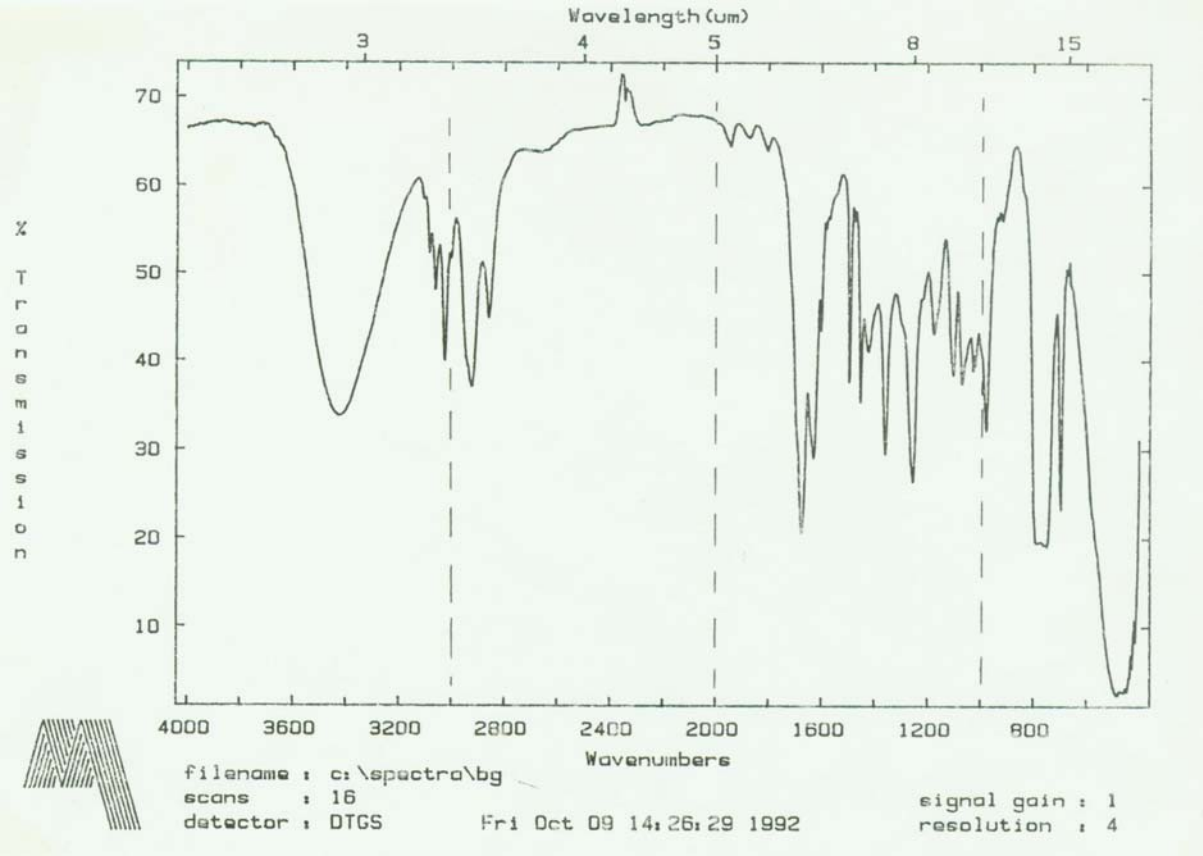


PLT ex-1-2009-10-gq.plt

Problem R-09D ($C_{13}H_{16}O_2$)

IR spectrum (CCl_4)

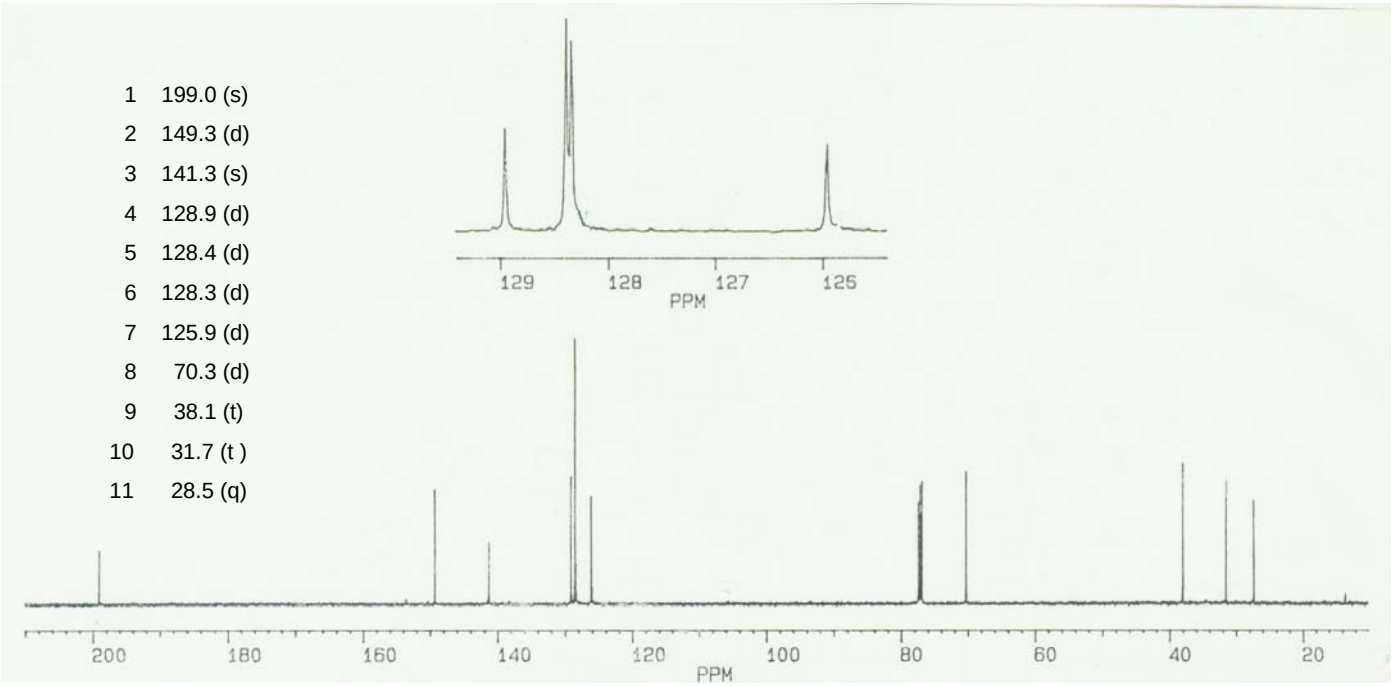
(Source: B. Gudmundsson/Reich 10/22)



Problem R-09D ($C_{13}H_{16}O_2$)

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

(Source: B. Gudmundsson/Reich 10/22)



PLT ex-1-2009-10-gq.plt

Problem R-09D. ($C_{13}H_{16}O_2$). Determine the structure (or part structure) of **R-09D** from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

(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 of the signals listed below report multiplicity and coupling constants to the extent the signals are amenable to first order analysis, and the part structure each corresponds to. (NOTE: the peaks at δ 1.9 and δ 2.8 are not strictly first order)

δ 1.9	δ 4.4
δ 2.1	δ 6.3
δ 2.2	δ 6.8
δ 2.8	δ 7.3

(c) Interpret the ^{13}C NMR spectrum. 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	199.0 (s)	_____
2	149.3 (d)	_____
3	141.3 (s)	_____
4	128.9 (d)	_____
5	128.4 (d)	_____
6	128.3 (d)	_____
7	125.9 (d)	_____
8	70.3 (d)	_____
9	38.1 (t)	_____
10	31.7 (t)	_____
11	28.5 (q)	_____

(d) Determine the structure of **R-09D**. If more than one structure is possible, show them, and circle your best choice. Why are the ^{1}H NMR signals at δ 1.9 and δ 2.8 so complex?

30 **Problem R-09D**. ($C_{13}H_{16}O_2$). Determine the structure (or part structure) of **R-09D** from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

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

3450 cm^{-1} broad OH stretch

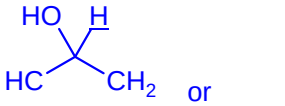
4 1680 cm^{-1} conjugated ketone/aldehyde stretch (CO_2H?)

1660 cm^{-1} C=C stretch

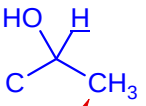
3050 cm^{-1} sp^2 C-H stretch

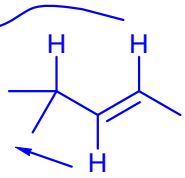
(b) Analyze the ^{1}H NMR signals. For each of the signals listed below report multiplicity and coupling constants to the extent the signals are amenable to first order analysis, and the part structure each corresponds to. (NOTE: the peaks at δ 1.9 and δ 2.8 are not strictly first order)

δ 1.9	2H could be ≈quartet m C(H)-CH_2-CH_2 seems to be coupled to 2.8	δ 4.4	1H, qm (actually tdd, 7,5,2)
δ 2.1	1H broad s, OH	δ 6.3	1H, dd, J = 15.5, 2 Hz
δ 2.2	3H, s, CH_3-C=O, poss CH_3-Ph	δ 6.8	1H, dd, J = 15.5, 5.5 Hz
δ 2.8	2H, m, CH_2-CH_2-EWG ? seems to be coupled to 1.9	δ 7.3	5H, m, monosubstituted Ph



or





Would require a Me doublet

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

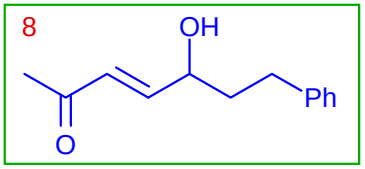
No	ppm	Type of C (e.g. sp^3 CH_2) and/or part structures (e.g. N-CH_2)
1	199.0 (s)	C=O ketone (very likely conjugated)
2	149.3 (d)	sp^2 CH
3	141.3 (s)	sp^2 C (ipso Ph)
4	128.9 (d)	sp^2 CH
5	128.4 (d)	sp^2 CH 2X, o/m phenyl
6	128.3 (d)	sp^2 CH 2X, o/m phenyl
7	125.9 (d)	sp^2 CH
8	70.3 (d)	HC-O- sp^3
9	38.1 (t)	CH_2 sp^3
10	31.7 (t)	CH_2 sp^3
11	28.5 (q)	CH_3 sp^3

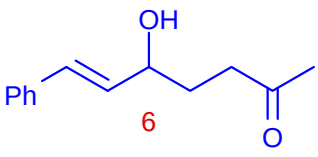
7

There are 2 fewer signals than carbons - 2 must be doubled

(d) Determine the structure of **R-09D**. If more than one structure is possible, show them, and circle your best choice. Why are the ^{1}H NMR signals at δ 1.9 and δ 2.8 so complex?

8



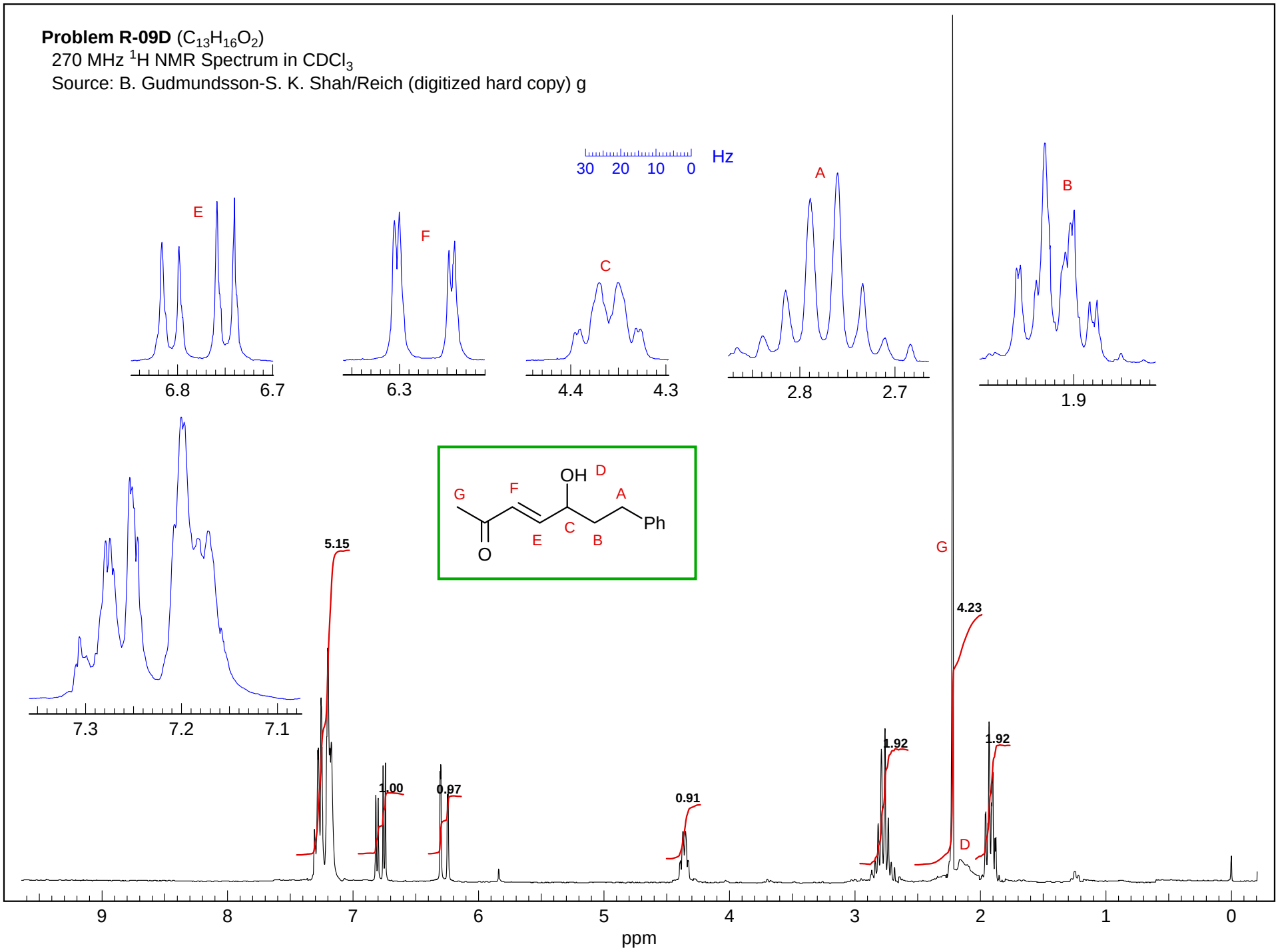


2 These are two adjacent CH_2 groups, which are each diastereotopic, hence an AB MN X system - lots of coupling

Problem R-09D ($C_{13}H_{16}O_2$)

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

Source: B. Gudmundsson-S. K. Shah/Reich (digitized hard copy) g



PLT ex-1-2009-10-gq.plt