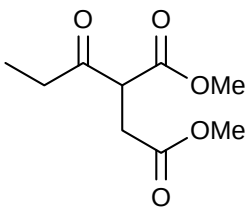
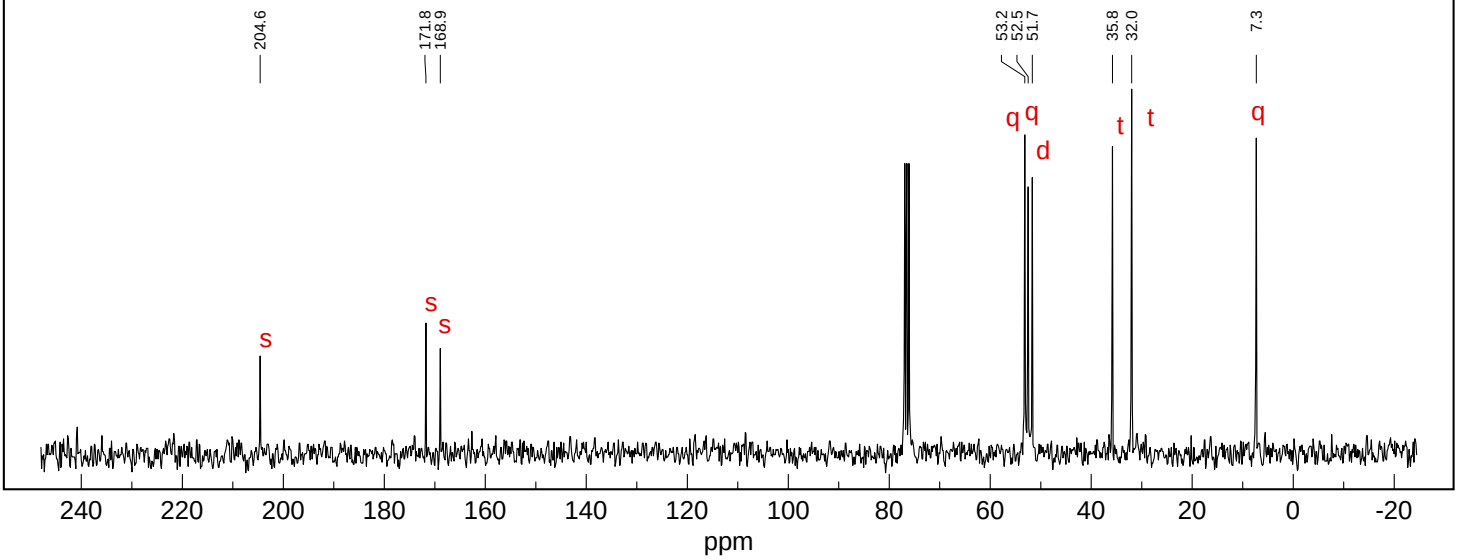
**Problem R-04F** ($C_9H_{14}O_5$)
300 MHz $^1$H NMR Spectrum in $C_6D_6$
Source: Paul Schatz/I. L. Reich (digitized hard copy) 10/27 g







**Problem R-04F** ($C_9H_{14}O_5$)
75.4 MHz $^{13}$C NMR Spectrum in $CDCl_3$
Source: Paul Schatz/I. L. Reich 10/27 g




PLT ex-1-2004-05-gq.plt

**Problem R-04F** ($C_9H_{14}O_5$). Determine the structure (or part structure) of **R-04F** from the $^{1}H$ and $^{13}C$ NMR spectra provided.

(a) DBE ___

(b) Interpret the $^{13}C$ NMR spectrum. The multiplicity of each signal is given on the spectrum. Identify what kind of carbon each signal corresponds to and write possible part structures.

Type of C (e.g. $sp^3$ $CH_2$) and/or part structures (e.g. N-$CH_2$)

δ 7.5 _____

δ 32.2 _____

δ 36.1 _____

δ 52.0 _____

δ 52.7 _____

δ 53.4 _____

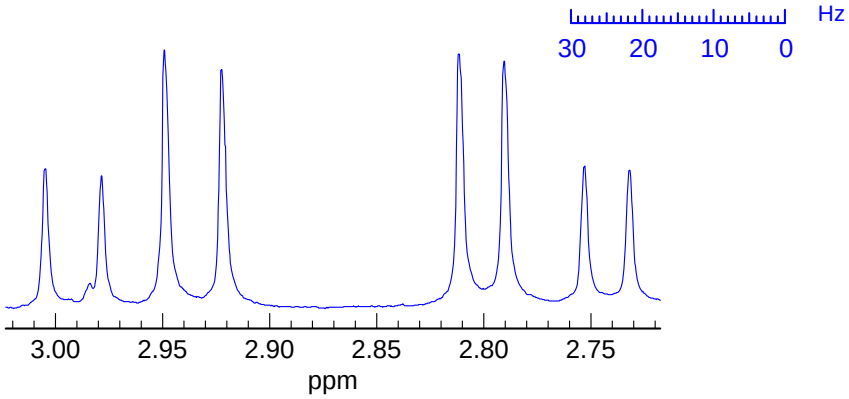
δ 168.9 _____

δ 171.8 _____

δ 204.4 _____

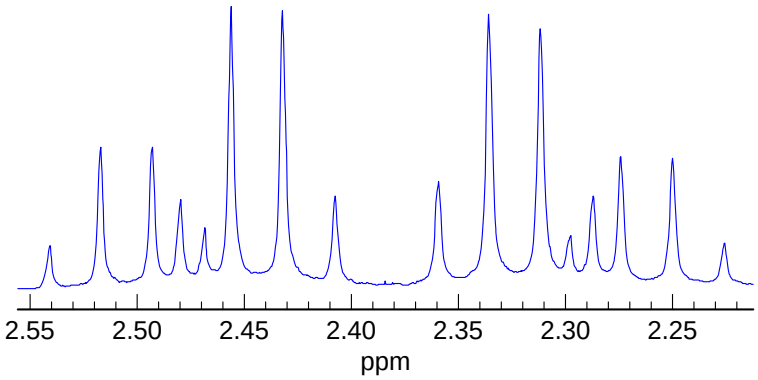
(c) What are the three peaks at δ 77?_____

(d) Analyze the 2-proton multiplet between δ 2.7 and 3.1 (reproduced below). Draw a coupling tree and report coupling constants (in the standard form: e.g., δ 3.9, tq, J = 12, 4 Hz, 1H) and part structure you could obtain from the signal. You may use first-order analysis.




What kind of pattern is this?_____ What other signal is coupled to these protons?

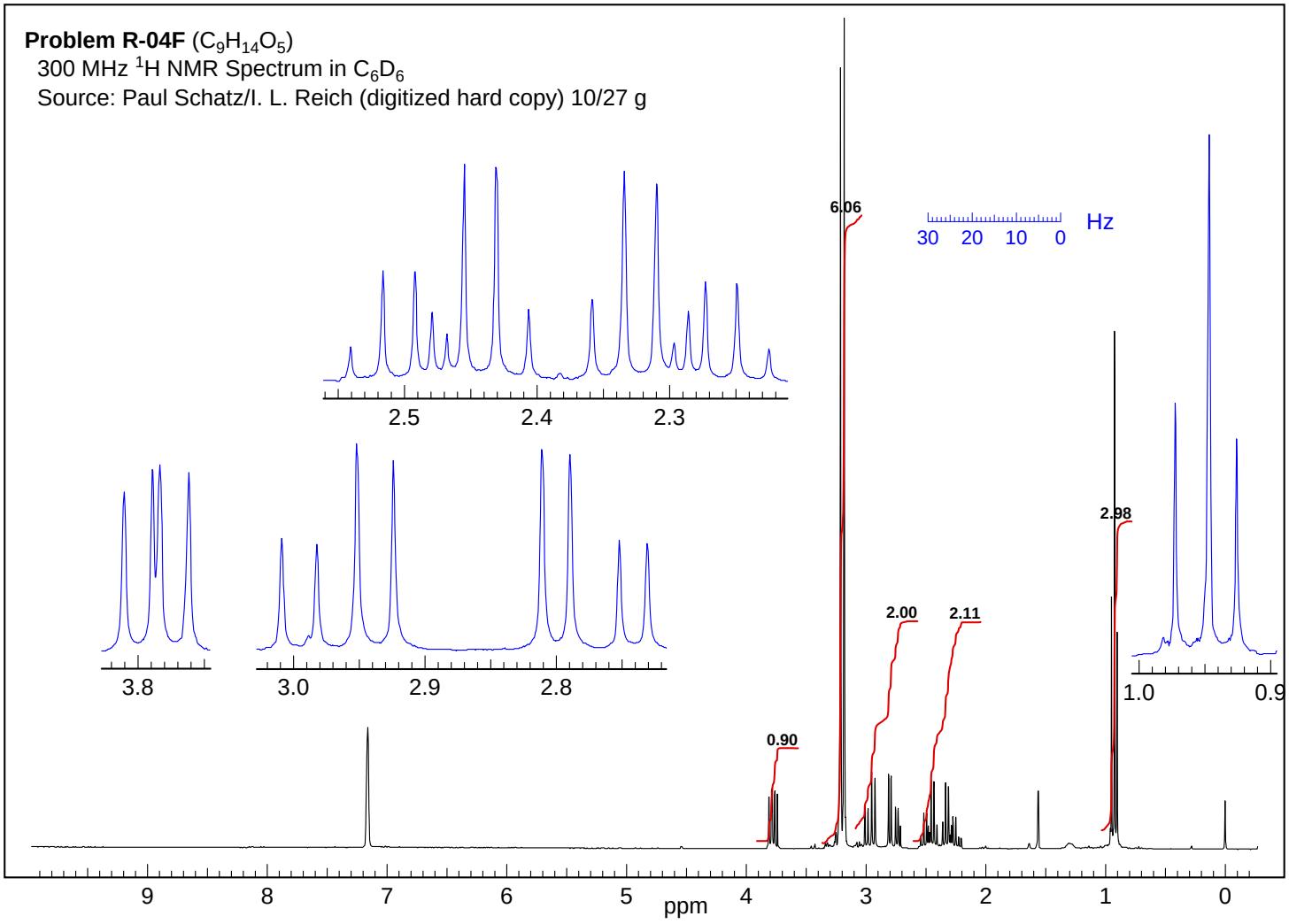
(e) Analyze the two-proton multiplet between δ 2.2 and 2.6 in the 300 MHz $^{1}$H NMR spectrum. The multiplet is reproduced below. Draw a coupling tree and report couplings and a part structure. You may use first order analysis.



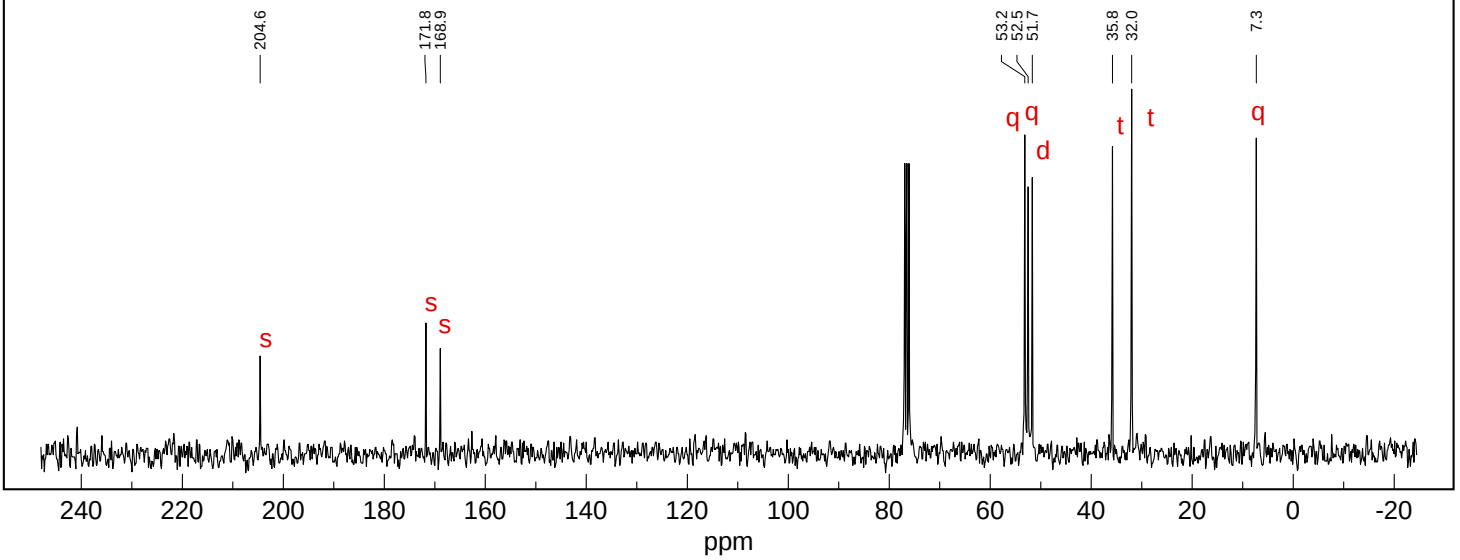

What kind of pattern is this?_____ What other signal is coupled to these protons?

(c) Show a structure for **R-04F**. If there is more than one possibility, circle your best choice.

**Problem R-04F** ($C_9H_{14}O_5$)
300 MHz $^1$H NMR Spectrum in $C_6D_6$
Source: Paul Schatz/I. L. Reich (digitized hard copy) 10/27 g




**Problem R-04F** ($C_9H_{14}O_5$)
75.4 MHz $^{13}$C NMR Spectrum in $CDCl_3$
Source: Paul Schatz/I. L. Reich 10/27 g




PLT ex-1-2004-05-gq.plt

**Problem R-04F** ($C_9H_{14}O_5$). Determine the structure (or part structure) of **R-04F** from the $^1H$ and $^{13}C$ NMR spectra provided.

2 (a) DBE 3

(b) Interpret the $^{13}C$ NMR spectrum. The multiplicity of each signal is given on the spectrum. Identify what kind of carbon each signal corresponds to and write possible part structures.

Type of C (e.g. $sp^3$ $CH_2$) and/or part structures (e.g. N-$CH_2$)

| | |
|---|---|
| δ 7.5 | C-**C**$H_3$ |
| δ 32.2 | C-**C**$H_2$-C $sp^3$ |
| δ 36.1 | C-**C**$H_2$-C $sp^3$ |
| δ 52.0 | **C**-H $sp^3$ |
| δ 52.7 | -O-**C**$H_3$ |
| δ 53.4 | -O-**C**$H_3$ |
| δ 168.9 | Ester |
| δ 171.8 | Ester |
| δ 204.4 | Ketone |

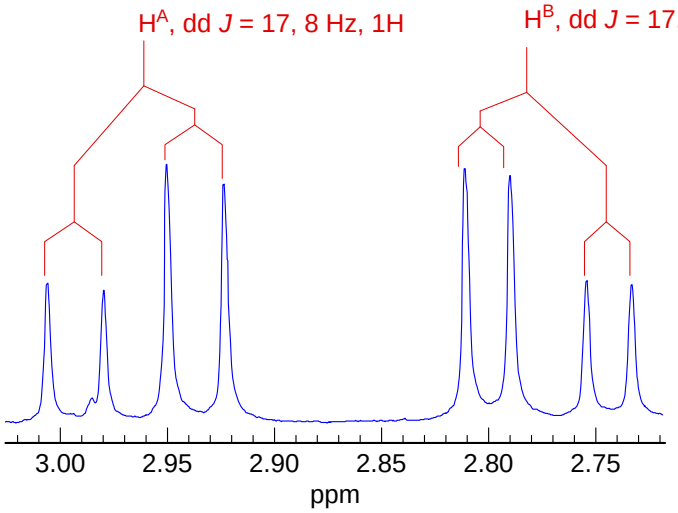
6

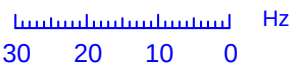
The $^{13}C$ sinals account for all 14 H - so there can be no OH or $CO_2H$

This gives 3 double bond equivalents - so no other rings or double bonds

2 (c) What are the three peaks at δ 77? **C**$DCl_3$: D has spin = 1, giving a 1:1:1 triplet, $J_{CD}$ = 30.7 Hz

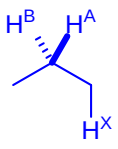
(d) Analyze the 2-proton multiplet between δ 2.7 and 3.1 (reproduced below). Draw a coupling tree and report coupling constants (in the standard form: e.g., δ 3.9, tq, J = 12, 4 Hz, 1H) and part structure you could obtain from the signal. You may use first-order analysis.




6

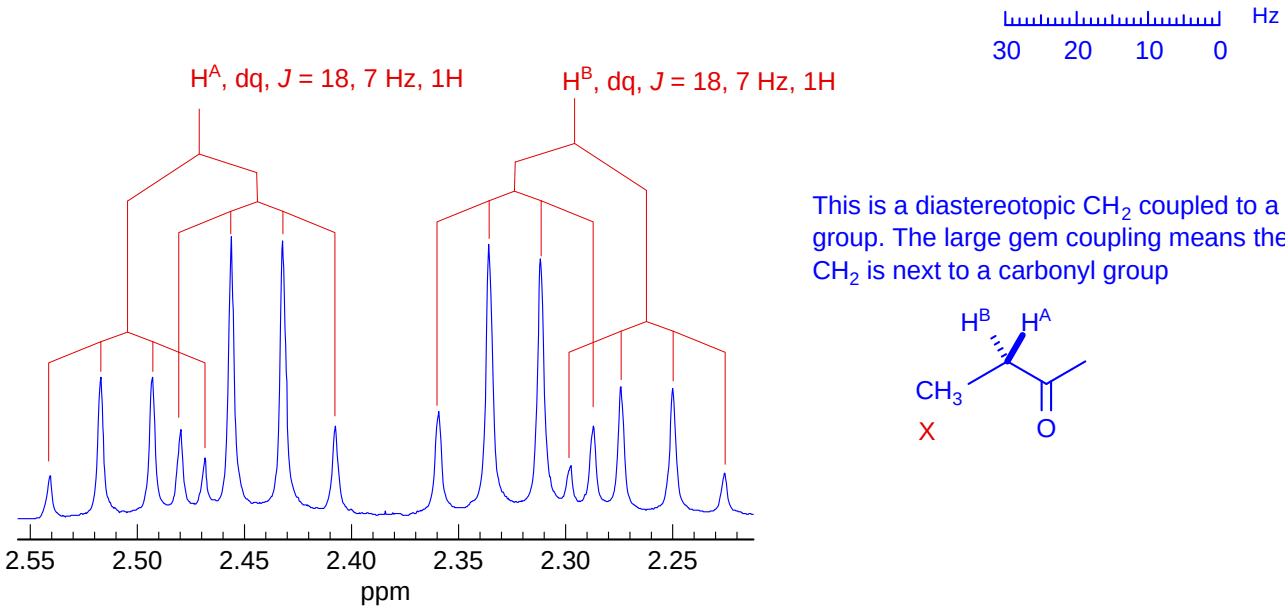
This is probably a diastereotopic $CH_2$ group coupled to one vicinal proton. Large $^2J$ means probably next to carbonyl group




What kind of pattern is this? AB of ABX What other signal is coupled to these protons?

These protons are coupled to dd at δ 3.75 (X)

(e) Analyze the two-proton multiplet between δ 2.2 and 2.6 in the 300 MHz $^1$H NMR spectrum. The multiplet is reproduced below. Draw a coupling tree and report couplings and a part structure. You may use first order analysis.




6

This is a diastereotopic $CH_2$ coupled to a $CH_3$ group. The large gem coupling means the $CH_2$ is next to a carbonyl group




What kind of pattern is this? AB of $ABX_3$ What other signal is coupled to these protons?

These protons are coupled to triplet at δ 0.95 ($X_3$)

(c) Show a structure for **R-04F**. If there is more than one possibility, circle your best choice.

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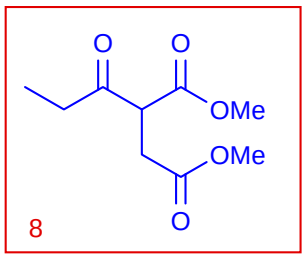
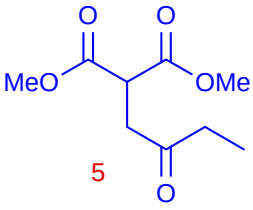
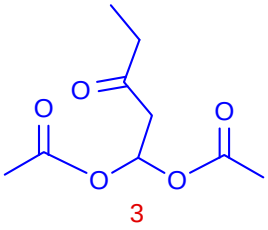
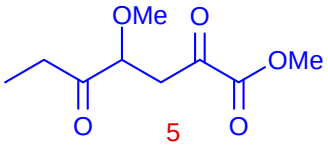
Some alternate solutions:

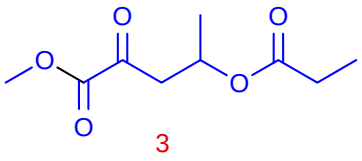


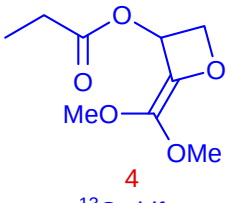



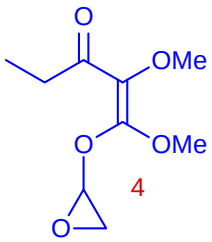



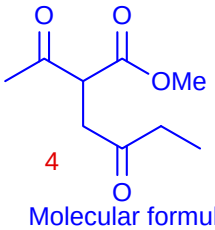

Not chiral - would have no diastereotopic $CH_2$ groups




$^{13}$C shifts




Molecular formula!