Clickers

Joseph Fox
To: "CHEM333-011-2138" undisclosed-recipients:;
[CHEM333-011-2138] videos posted

Class,

A reminder: I have posted links to youtube videos, labeled lectures 2a, 2b, 2c, 2d and 2e. Note, they are required viewing. Please watch them sequentially

https://www.youtube.com/watch?v=SWozhUpRf4o
https://www.youtube.com/watch?v=JCfoP8anSr0
https://www.youtube.com/watch?v=vsC367BxbFM
https://www.youtube.com/watch?v=1H9hatQ5_Mg
http://www.youtube.com/watch?v=HRbv1HL0AuI&feature=youtu.be

JMF

:: UD P.O. Box ::
CHEM333-011-2138 mailing list

Online message archive
and management at https://po-box.nss.udel.edu/
::

a. I watched all 5 videos
b. The dog ate my iphone

lecture 2e, Chem 333, Fall 2013 HD

September 14, 2013 2:50 PM

Edit

59 0 0 0

40%

lecture2d, Chem 333, 2013 HD

September 14, 2013 2:46 PM

Edit

48 0 0 0

33%

lecture2c Chem 333 Fall 2013 HD

September 14, 2013 2:38 PM

Edit

49 0 0 0

33%

lecture2b Chem 333 Fall 2013 HD

September 14, 2013 2:32 PM

Edit

59 0 0 0

40%

lecture2a HD

September 14, 2013 11:10 AM

Edit

87 0 0 0

59%

67% of you: Watch youtube!

PROBLEMS: Complete end of chapter 13 problems 1–10 from Lab Manual
Answers

^{1}H NMR

Protons (nucleus of a hydrogen atom) also have a net spin and can be observed in the NMR.

- Hydrogen atoms are more than 99% ^{1}H.

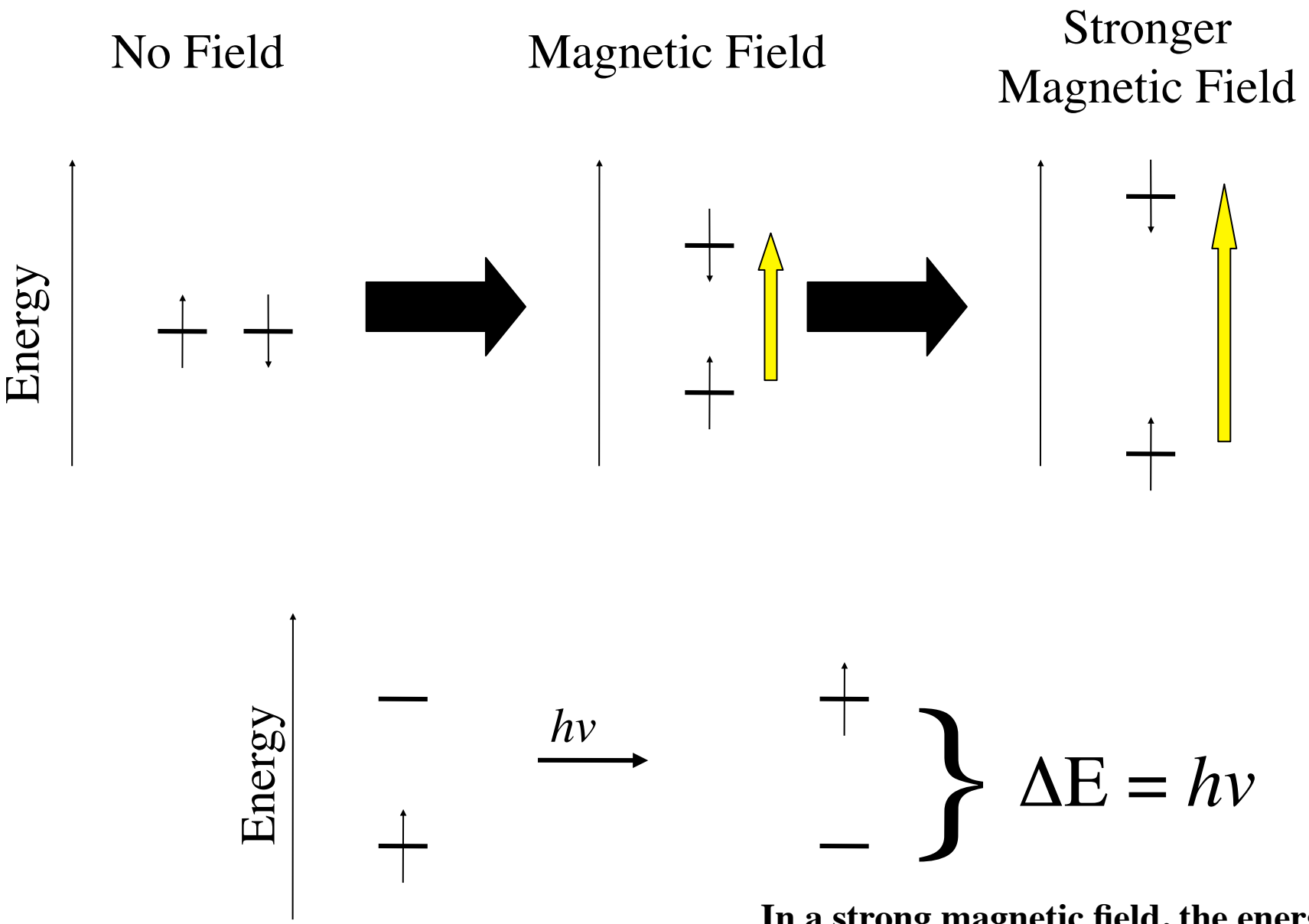
  Other isotopes have special names

  ^{2}H = deuterium

  ^{3}H = Tritium (radioactive)

- Remember ^{13}C is only 1% of carbon; ^{1}H NMR is much more sensitive.

  – Practically speaking for a typical lab molecule, you can obtain a ^{1}H-NMR wit 1-2 mg of compound in a matter of minutes but the same sample may require a few hours to obtain a ^{13}C spectrum.



In a strong magnetic field, the energy level difference corresponds to the energy of radio waves

FYI: Q&A

How strong is a magnetic field in a typical NMR?

300 MHz NMR = 7.0459 tesla

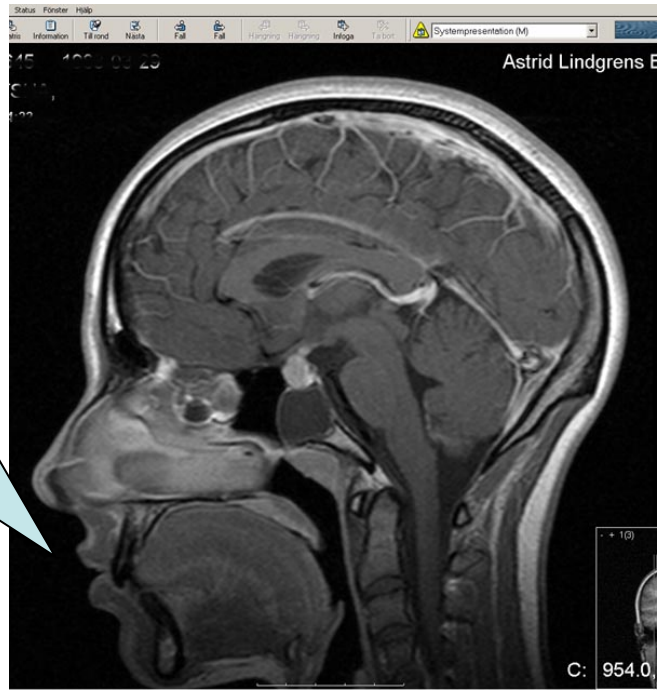
600 MHz NMR = 14.0918 tesla

What other nuclei are commonly observed by NMR?

^{15}N, ^{19}F, ^{31}P

What type of NMR is used in MRI?

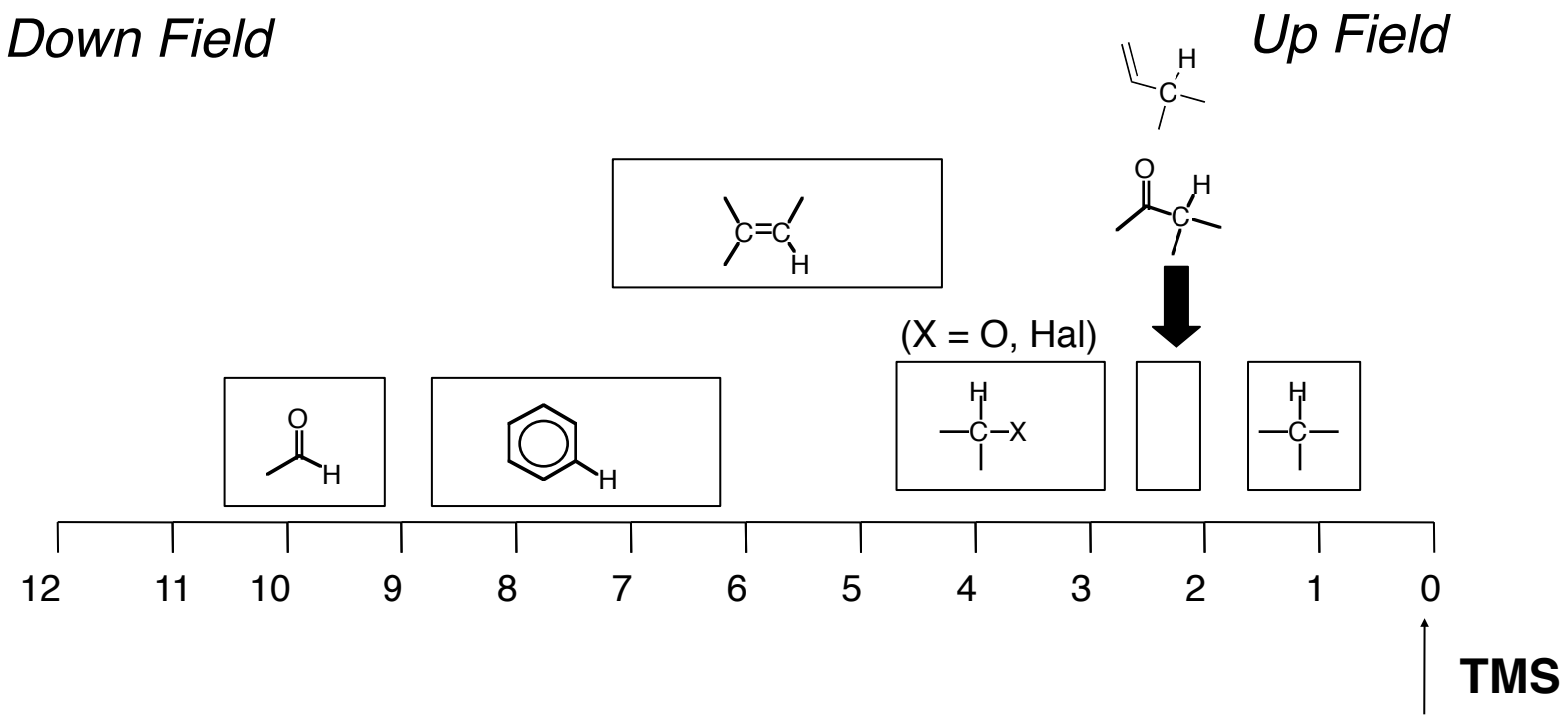
MRI methods typically look at ^{1}H-NMR of water in the different environments of the body.



Protons resonate at a different frequency than ^{13}C's. Typical compounds lie within a smaller range of frequencies than ^{13}C.

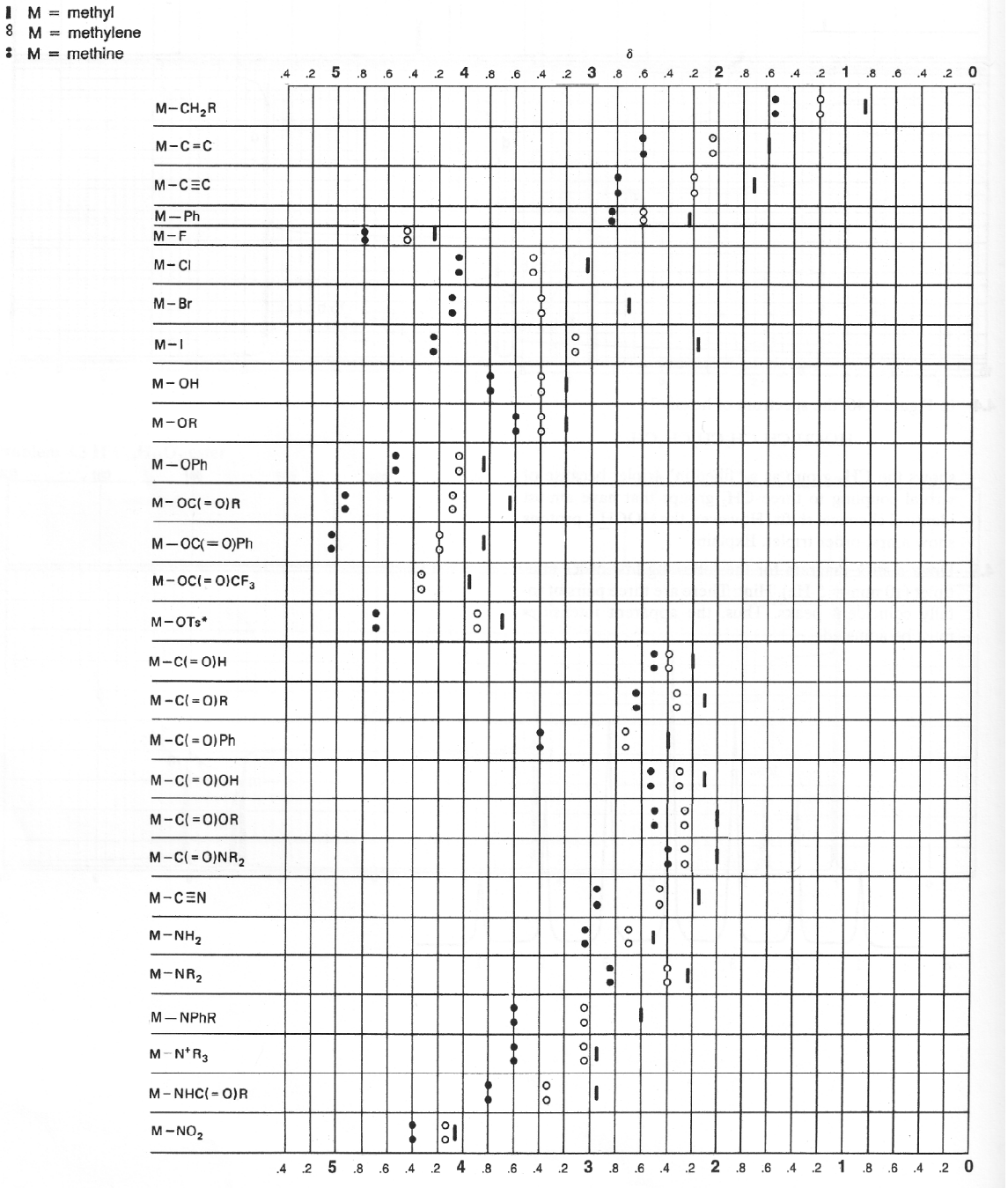
PROTONS ON TYPICAL ORGNIC COMPOUNDS **1-12 ppm**

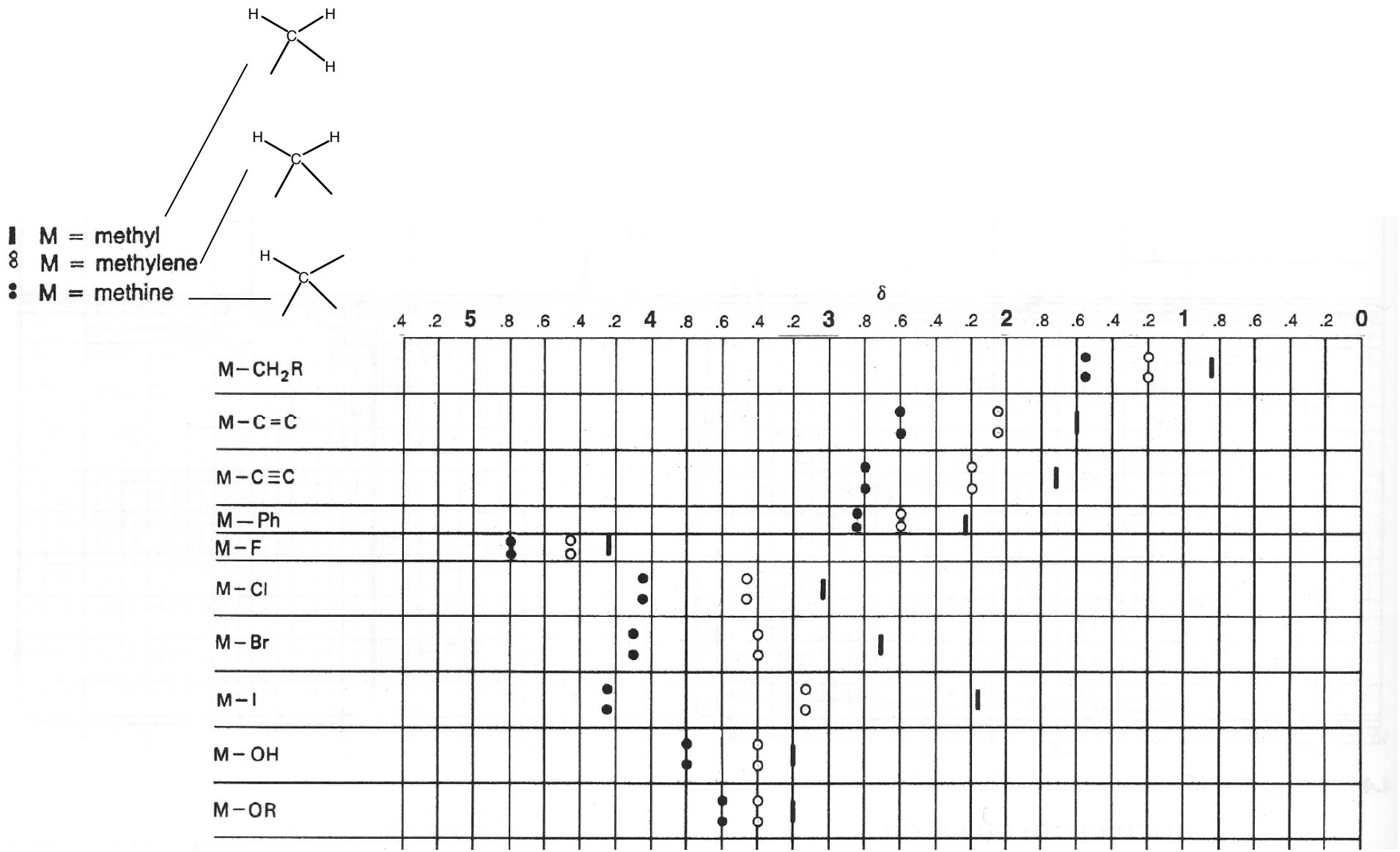
Typical locations of ^{1}H-NMR resonances.

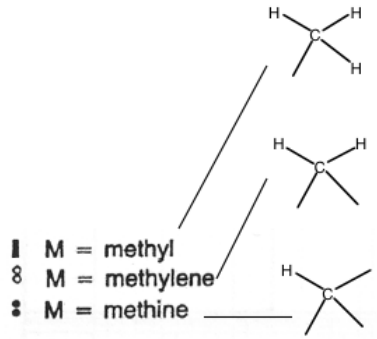


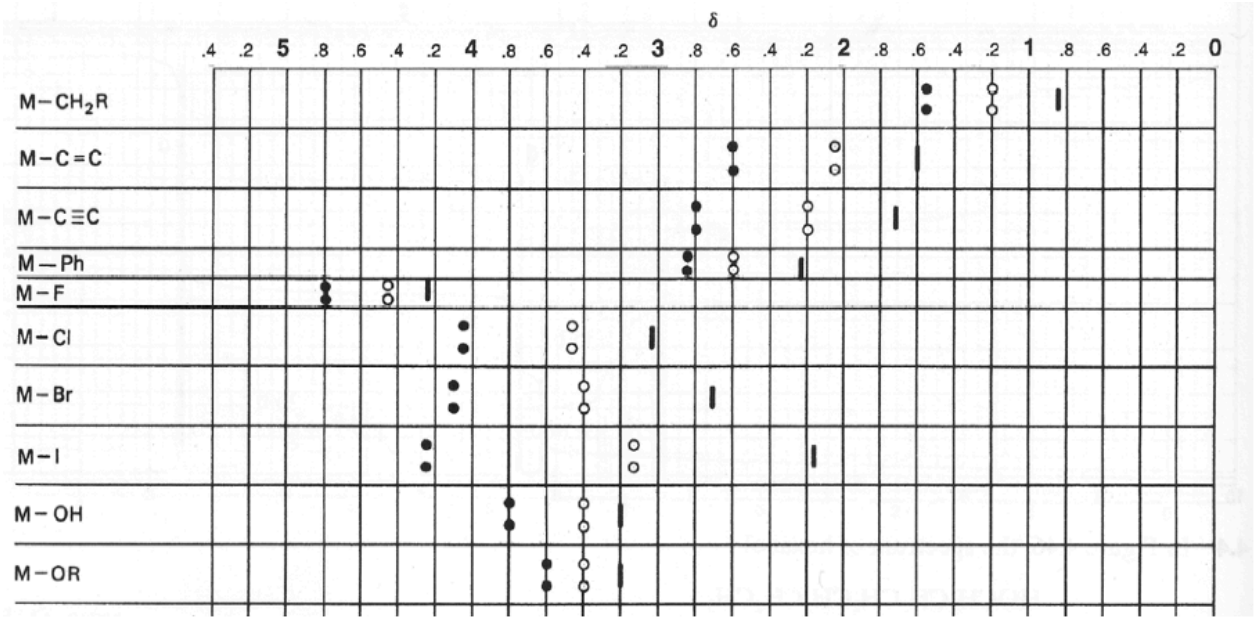
See Table 13.1 in your text (lab manual) or Table H1 (page 258-260 in spec book).

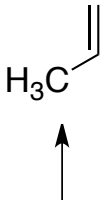
Appendix A CHART A.1 Chemical Shifts of Protons on a Carbon Atom Adjacent (α Position) to a Functional Group in Aliphatic Compounds (M—Y)



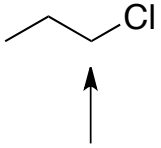

M = methyl
M = methylene
M = methine
δ
.4 .2 5 .8 .6 .4 .2 4 .8 .6 .4 .2 3 .8 .6 .4 .2 2 .8 .6 .4 .2 1 .8 .6 .4 .2 0
M−CH2R
M−C=C
M−C≡C
M−Ph
M−F
M−Cl
M−Br
M−I
M−OH
M−OR



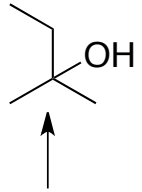




a. 1.2
b. 1.6
c. 2.0
d. 2.6
e. 3.0

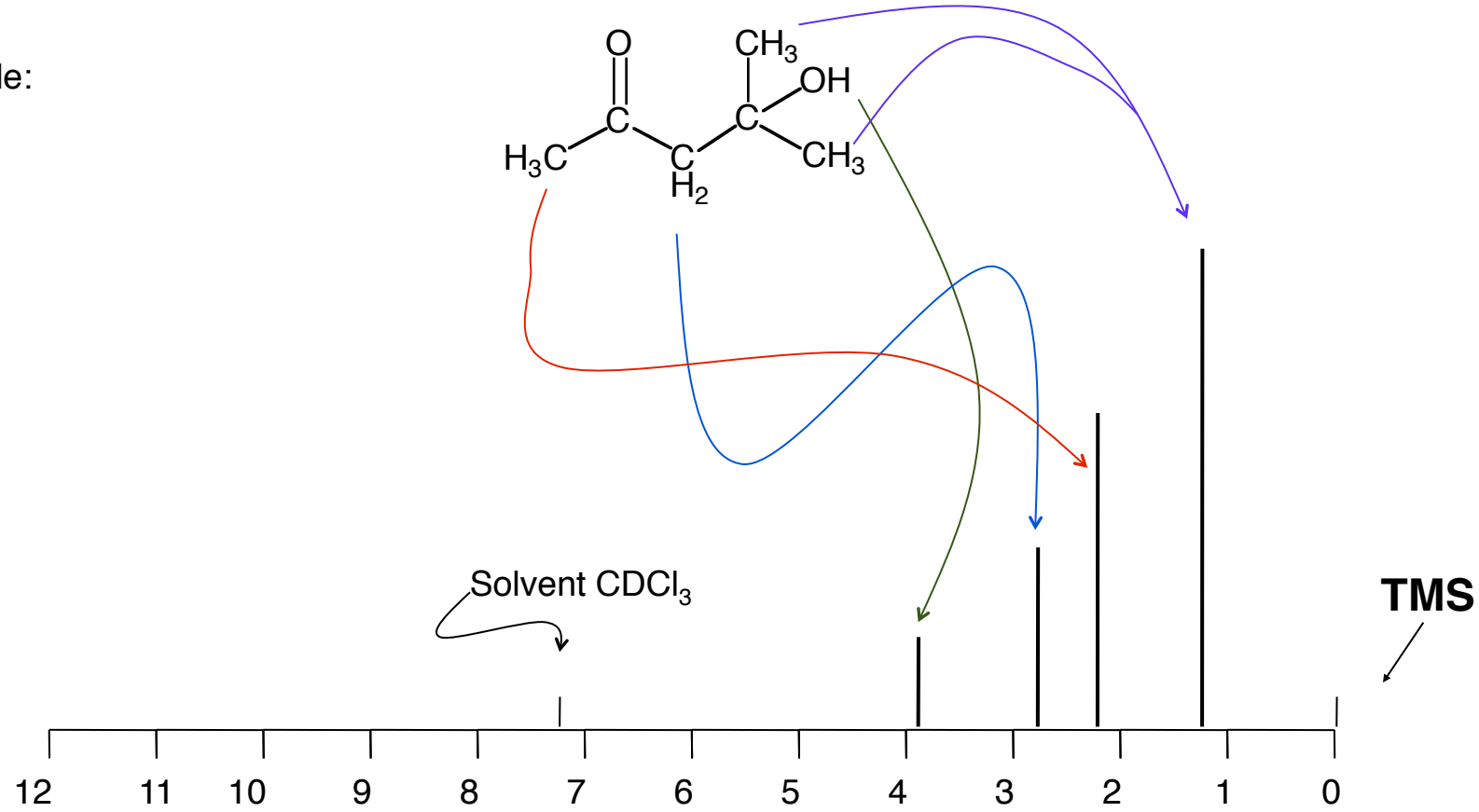


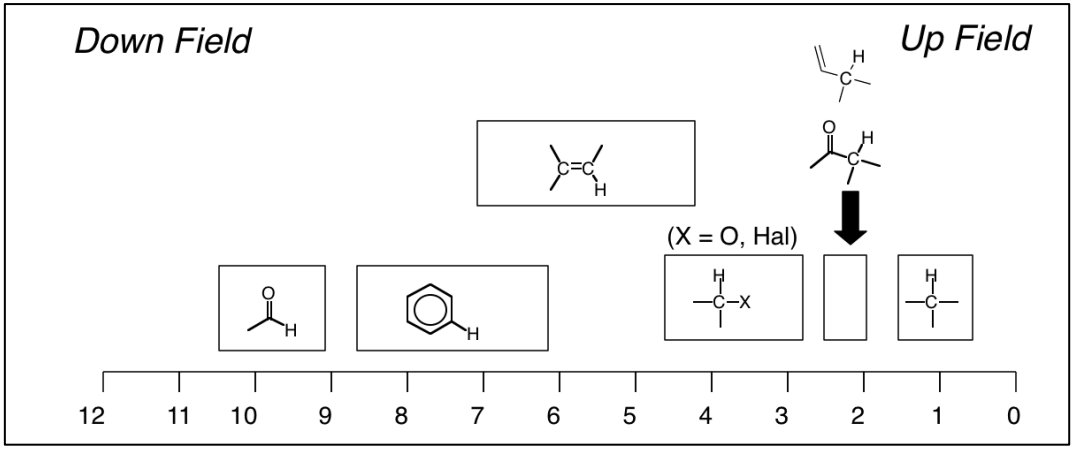
a. 1.2
b. 2.1
c. 3.0
d. 3.4
e. 4.2



a. 1.2
b. 2.1
c. 3.0
d. 3.4
e. 3.8

Example:

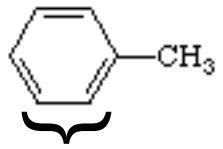

O
CH3
OH
H3C
C
C
CH3
H2
Solvent CDCl3
TMS
12
11
10
9
8
7
6
5
4
3
2
1
0


Down Field
Up Field
(X = O, Hal)
12
11
10
9
8
7
6
5
4
3
2
1
0

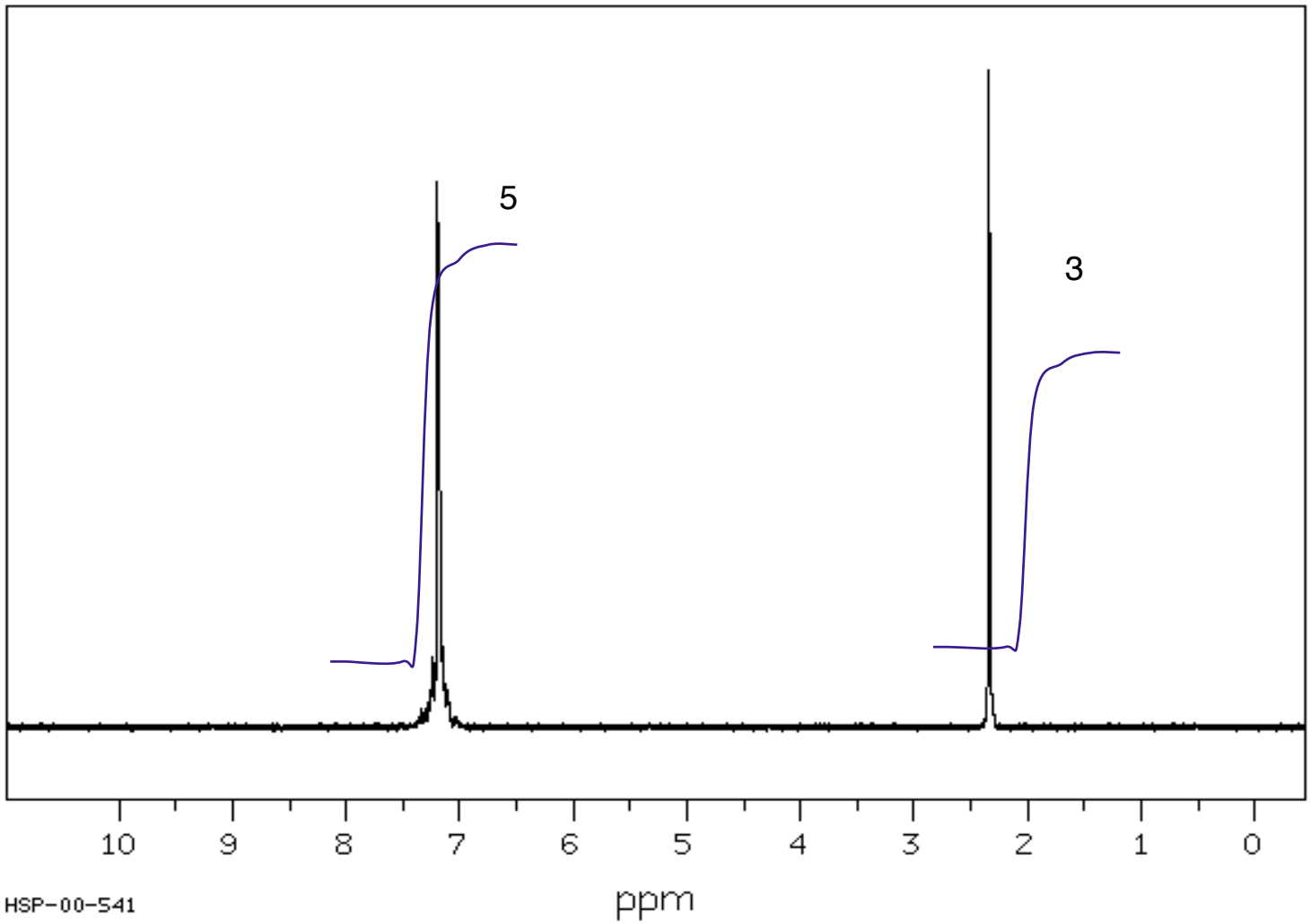
WHAT IS SO SPEC IAL ABOUT PROTON NMR?

- You can accurately integrate your spectra to know how many of each proton type you have.
- Through bond coupling tells us about what is adjacent to the protons of a particular resonance.

^{1}H-NMR peaks can be integrated!!

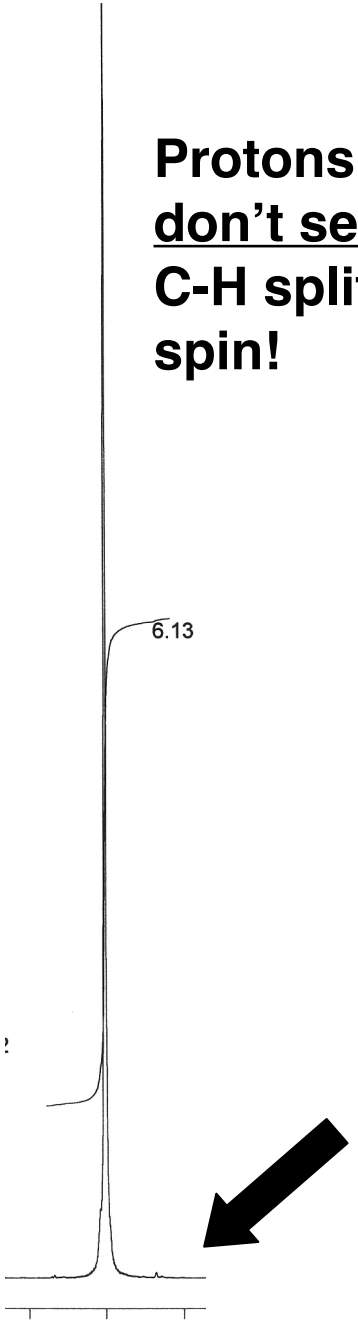


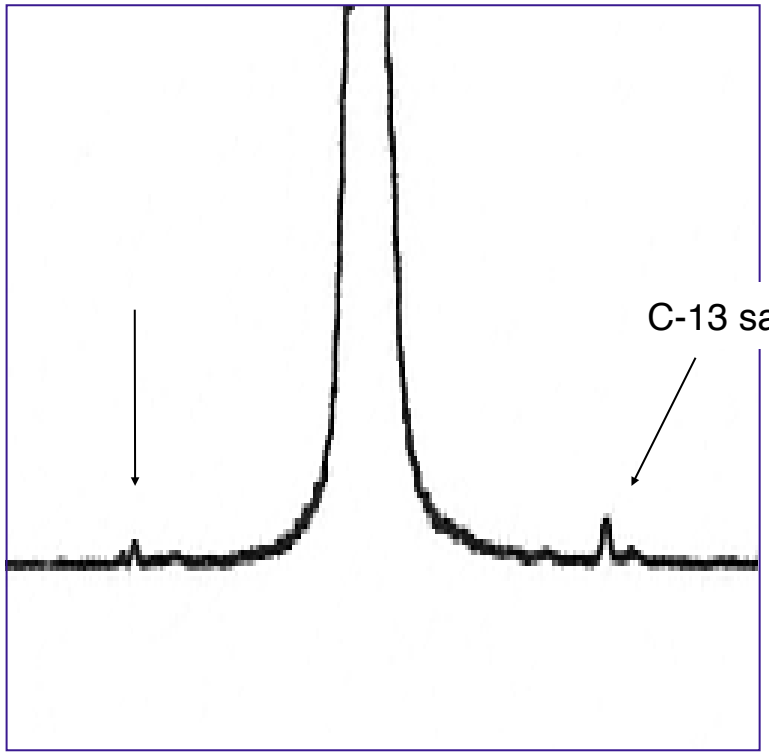
5 : 3



Tells you the ratio of the number of protons that compose each resonance.
This is particularly helpful when you have symmetry.

Protons can couple (cause splitting) of C-13 peaks but <u>we don't see splitting of proton peaks by carbon!!</u>
C-H splitting is very small because only 1% of Carbon has a spin!



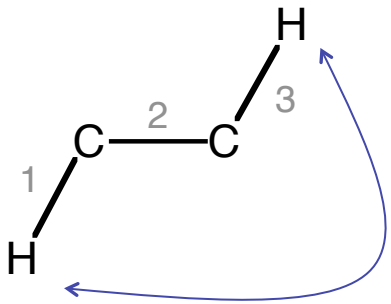


You can see carbon-proton coupling if you look very closely!

Protons can split other protons by through bond J-coupling.

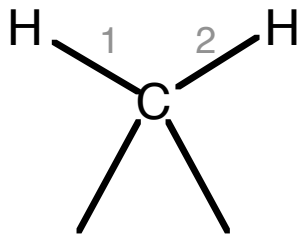
Coupling can be observed between two protons.

The coupling originates because in a magnetic field, the nuclear spin will perturb the local distribution of electrons.



Are coupled over three bonds.

Protons on adjacent carbons are said to be ***Vicinal*** protons



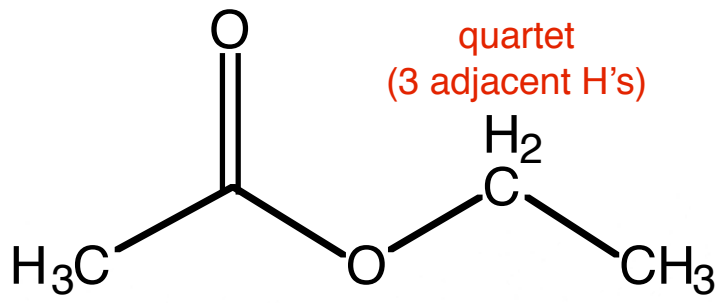
Splitting over two bonds is usually not observed.
Geminal coupling is only observed when the two protons are in different "chemical environments",

Note: there are exceptions when the protons are diastereotopic (more on this later).

Protons on the same carbons are said to be ***geminal*** protons

Proton NMR signals are also split following the N+1 rule

for proton NMR, *N* typically represents the number of adjacent protons.



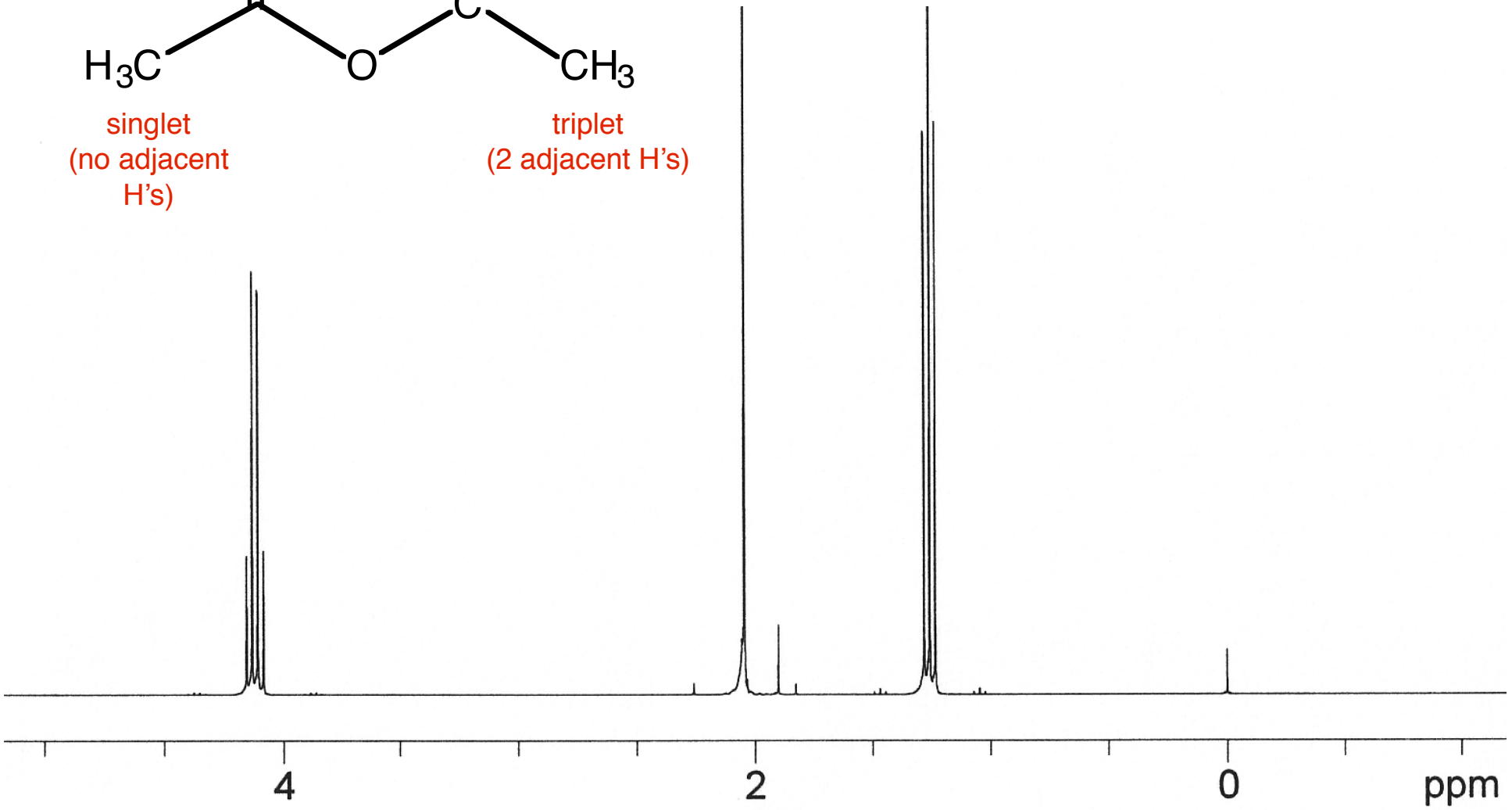
quartet
(3 adjacent H's)

singlet
(no adjacent
H's)

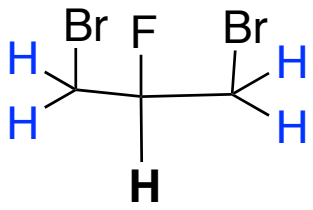
triplet
(2 adjacent H's)

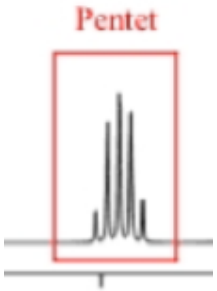
N+1 RULE STILL APPLIES

(note: it can get more complicated)



N+1 rule extends beyond quartets

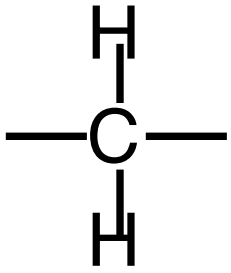




PAY ATTENTION!:

^{13}C-NMR

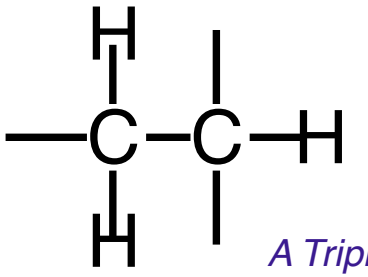
Splitting pattern reflects how many protons are <u>directly attached</u> to the carbon atom



A Triplet

^{1}H-NMR

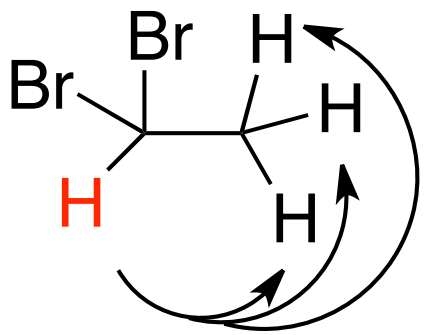
Splitting pattern reflects how many protons are connected to <u>adjacent</u> carbon atom(s)



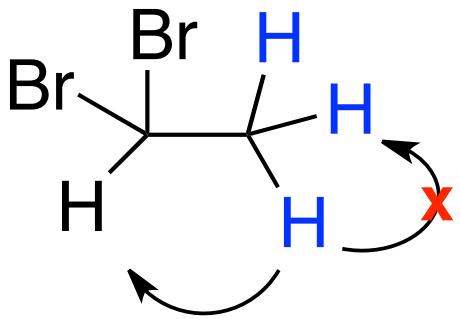
A Triplet

A doublet

clickers



a. singlet
b. doublet
c. triplet
d. quartet ←

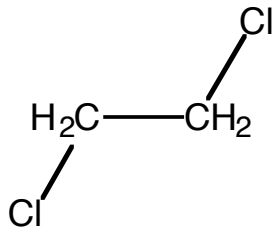


a. singlet
b. doublet ←
c. triplet
d. quartet

Rules for J-coupling

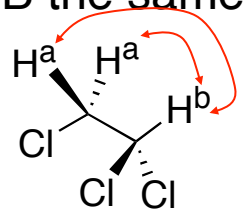
•Nuclei must be chemical shift NON-EQUIVALENT to show (obvious) coupling to each other:

•*1,2-dichloroethane is a singlet*



•The coupling is mutual: A splits B the same amount as B splits A

•$J_{AB} = J_{BA}$



H^a: 3.90 ppm, d (J = 7.1 Hz)

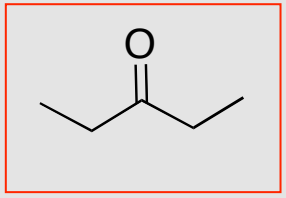
H^b: 5.77 ppm, t (J = 7.1 Hz)

•Coupling constants are reported in Hz and will have the same value on different instruments.

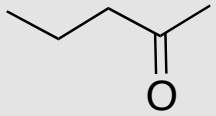
$C_5H_{10}O$

CLICKERS

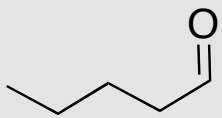
Which is it?



A



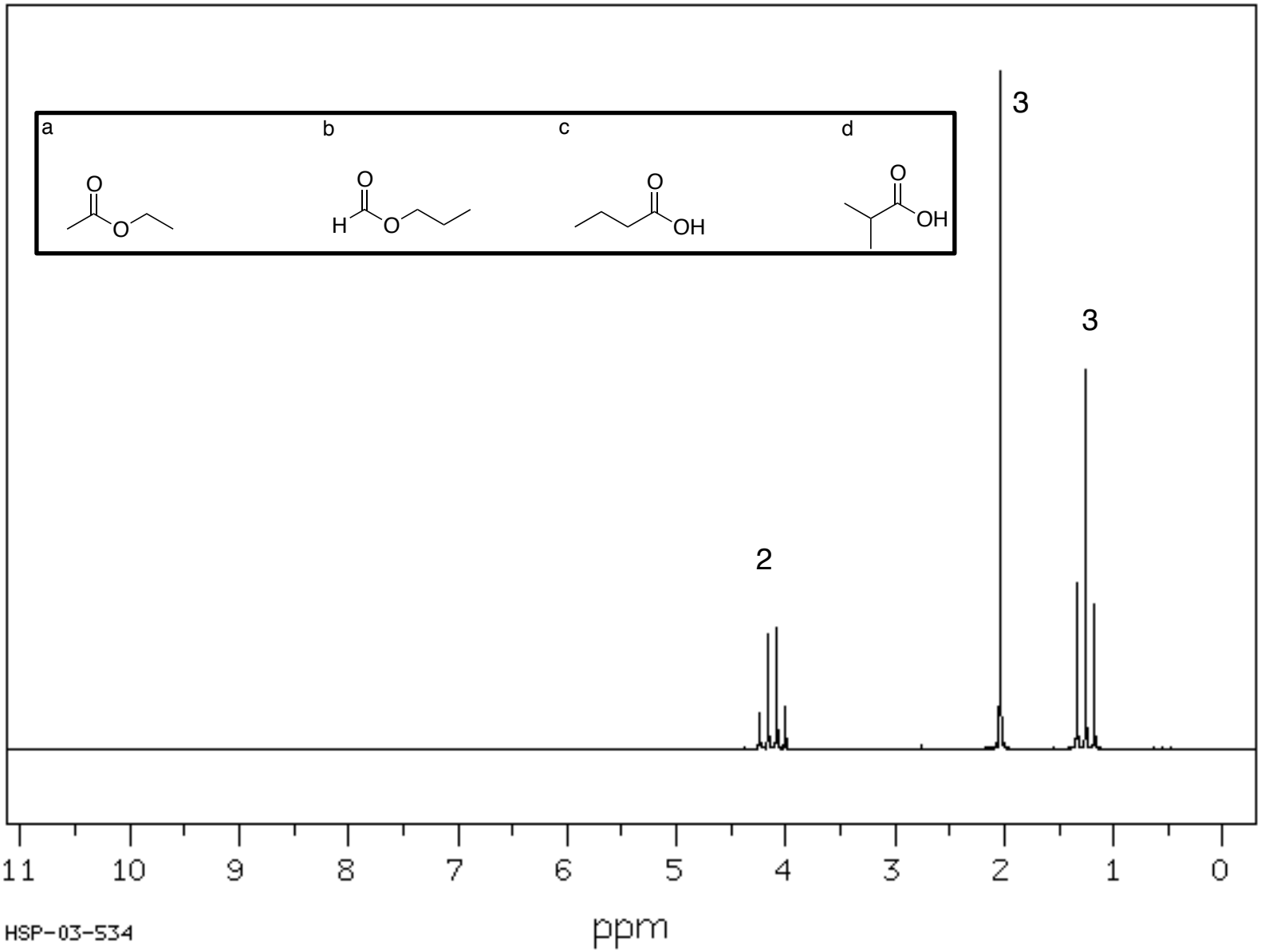
B



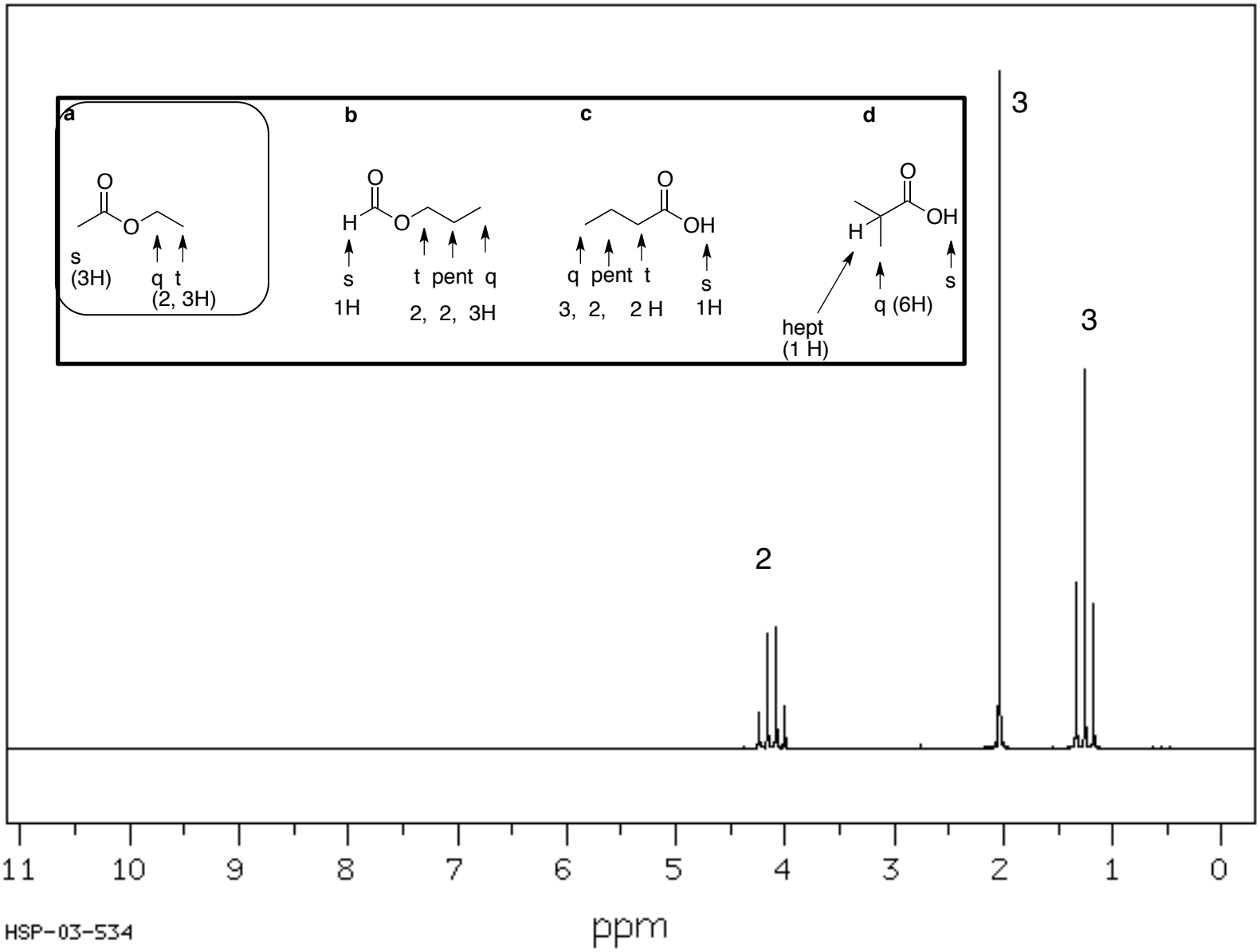
C

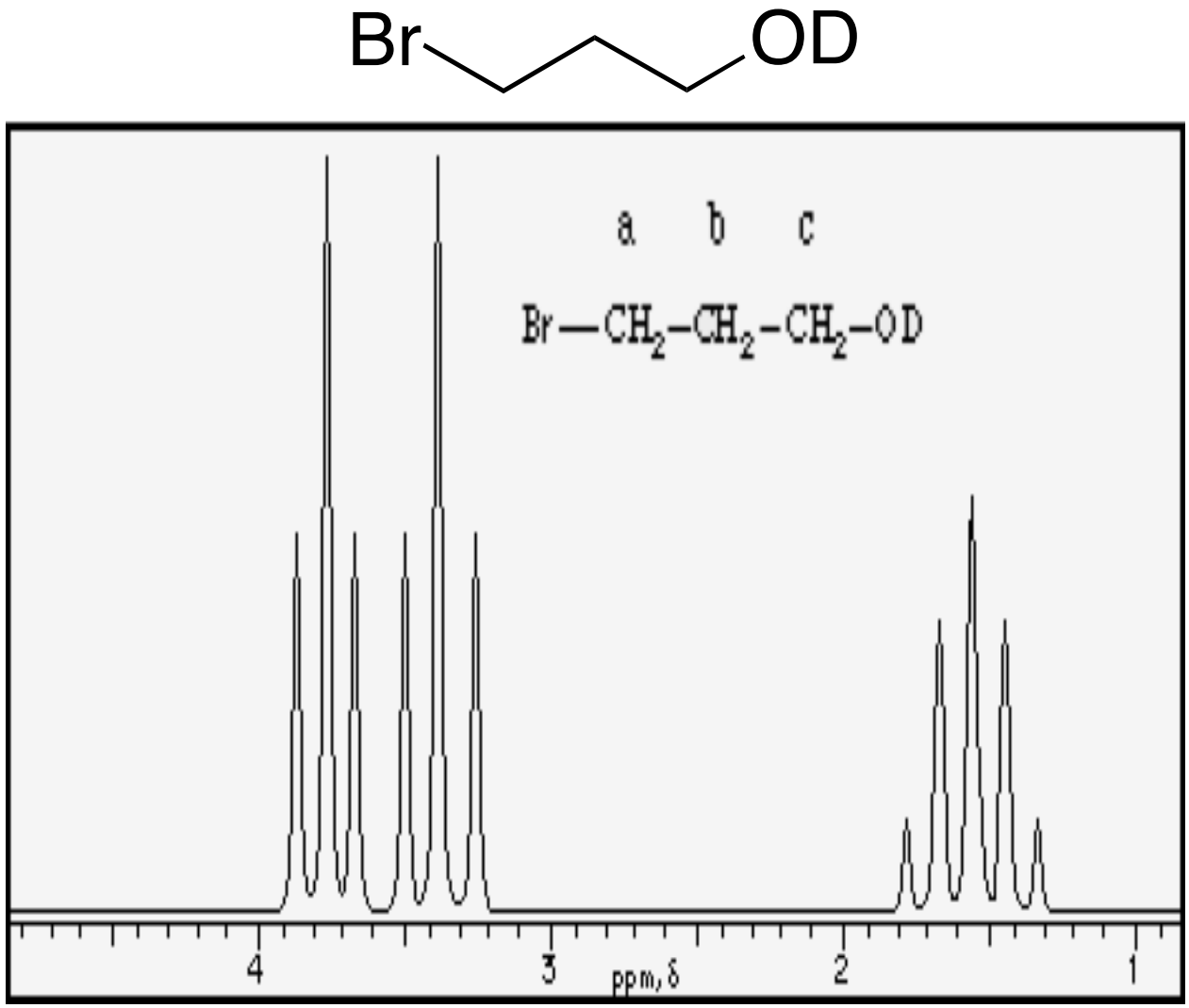
note: 6:4 ratio = 3:2 ratio

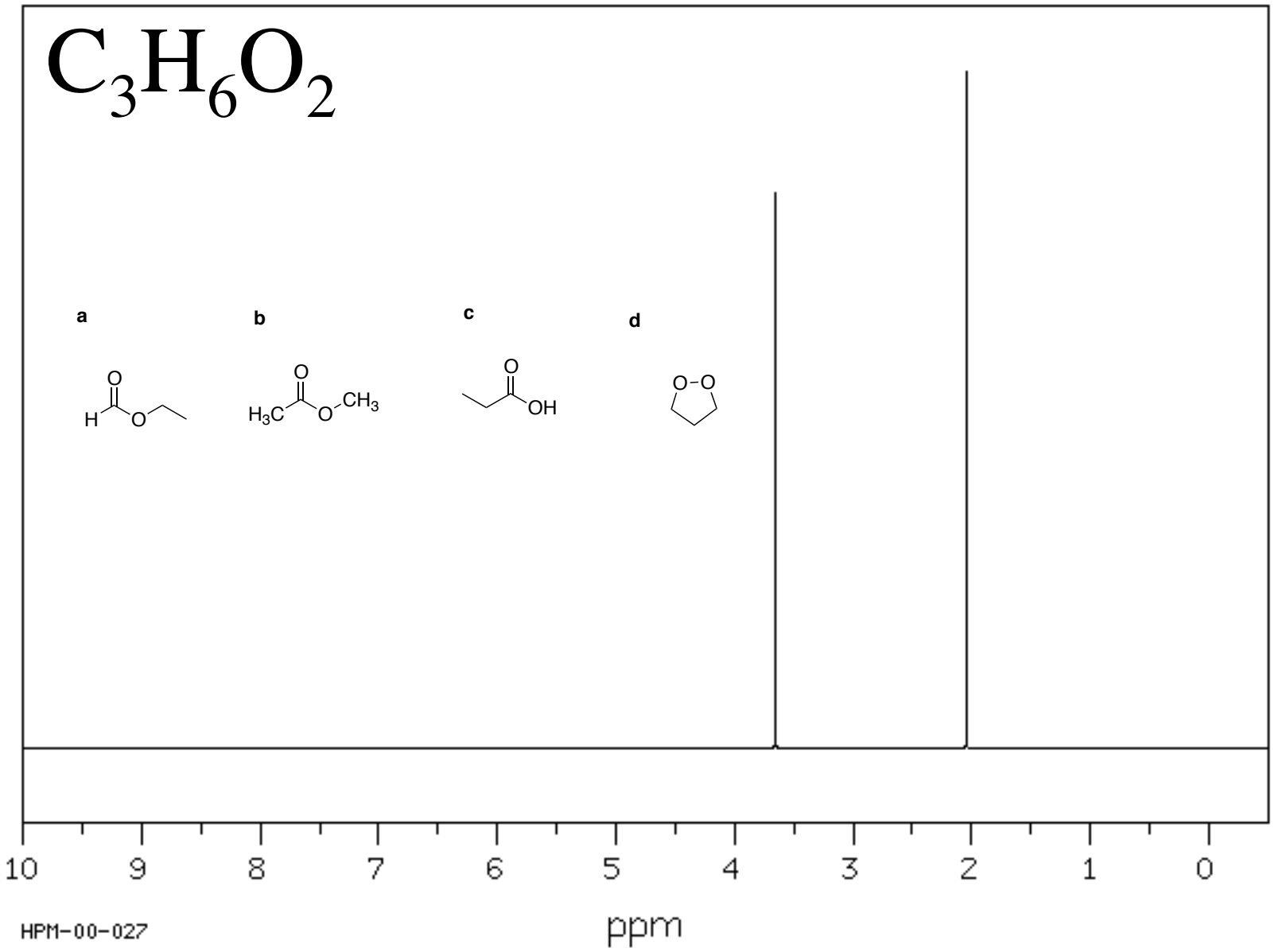
$C_4H_8O_2$

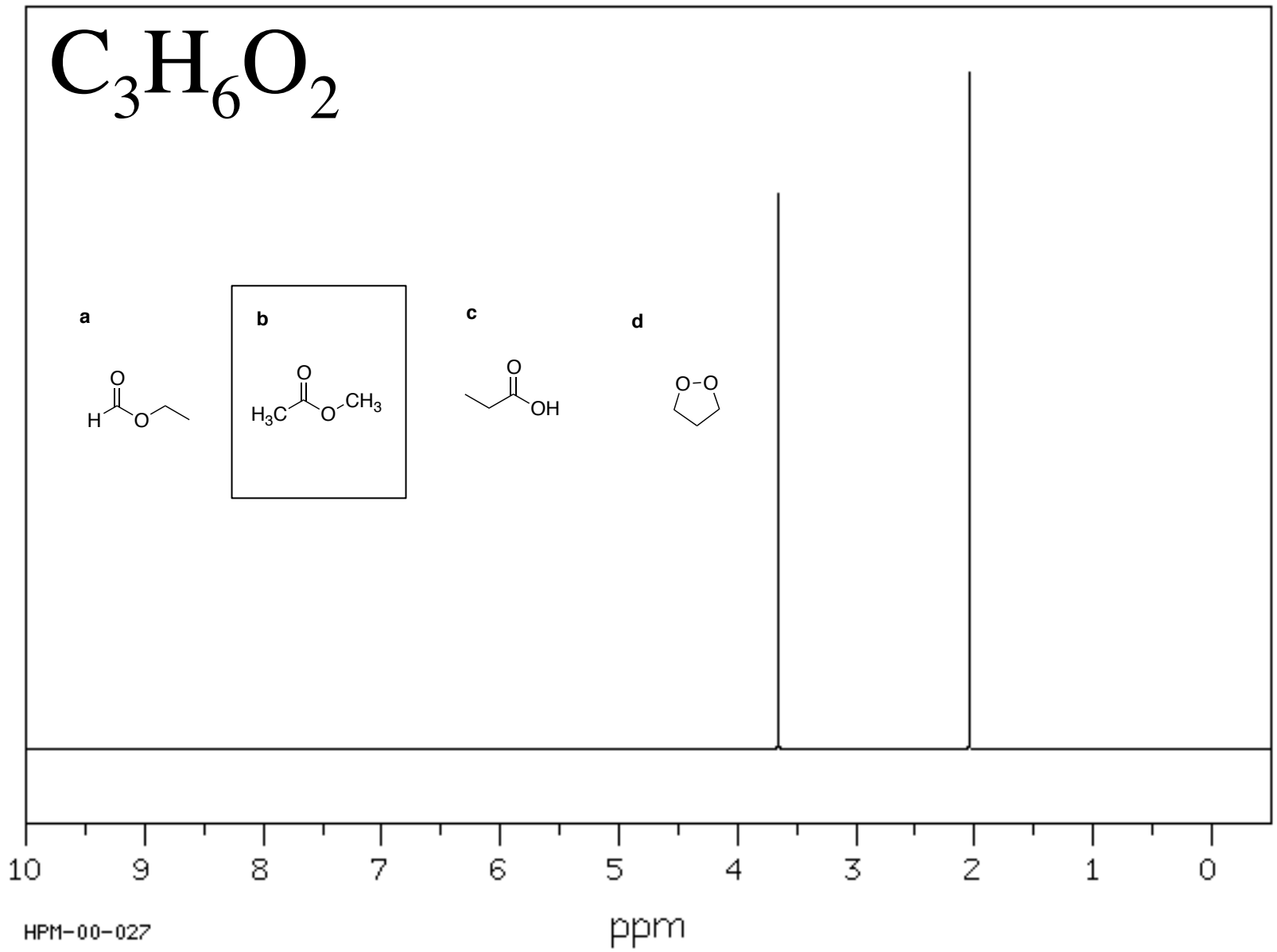


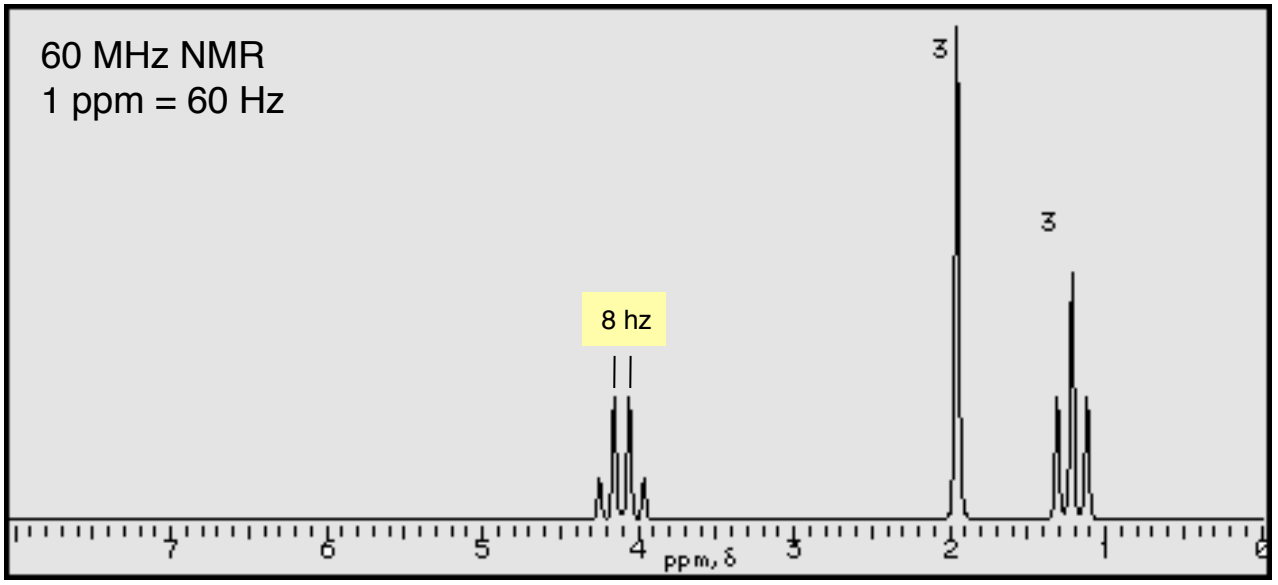
$C_4H_8O_2$


a
O
O
s
(3H)
q t
(2, 3H)
b
O
H
O
s
1H
t pent q
2, 2, 3H
c
O
OH
q pent t
3, 2, 2 H
s
1H
d
O
OH
H
hept
(1 H)
q (6H)
s
3
3
2
11
10
9
8
7
6
5
4
3
2
1
0
ppm
HSP-03-534

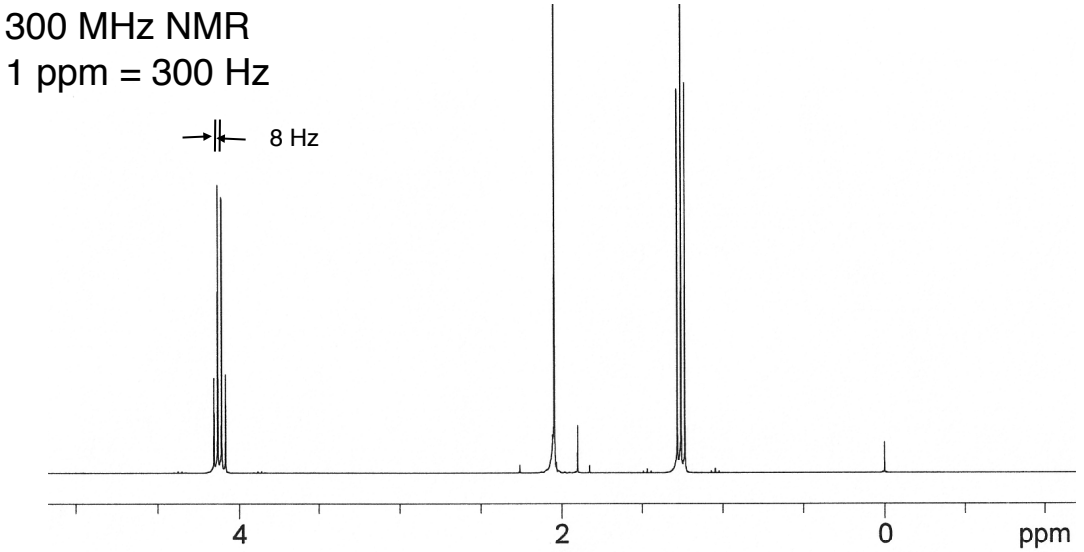

Br
OD
a b c
Br—CH2–CH2–CH2–OD
4
3
ppm, δ
2
1


C3H6O2
a
b
c
d
O
H
O
H3C
O
CH3
OH
O-O
10
9
8
7
6
5
4
3
2
1
0
ppm
HPM-00-027


C3H6O2
a
b
c
d
10
9
8
7
6
5
4
3
2
1
0
ppm
HPM-00-027

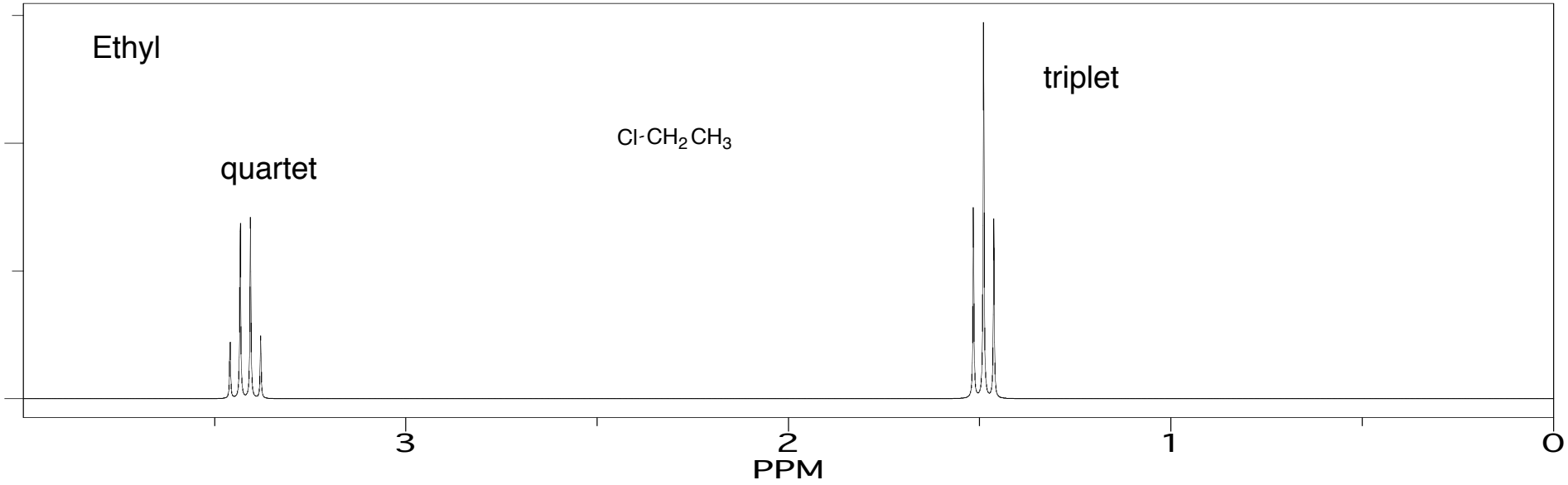


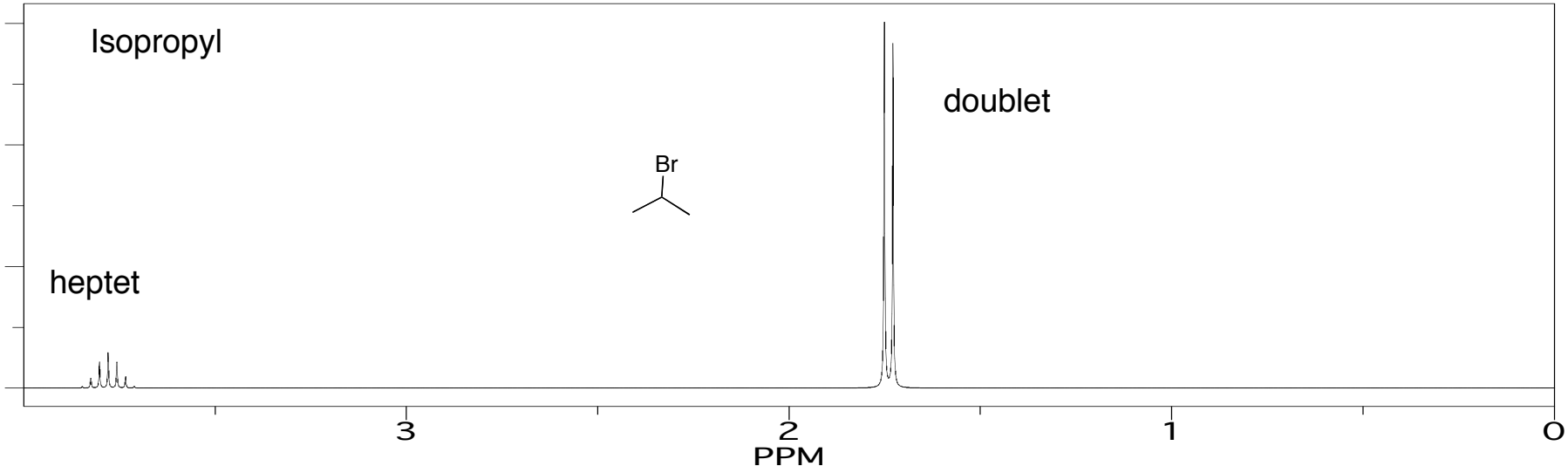
Same compound run on a 60 MHz and a 300 MHz spectrum

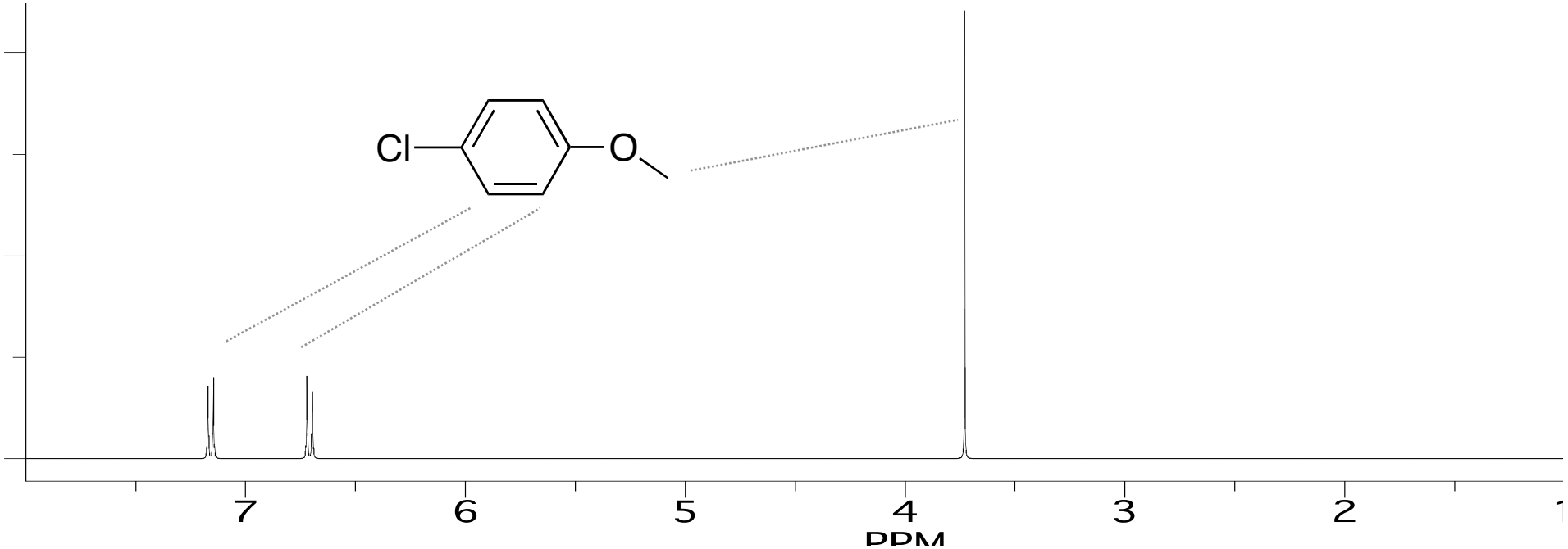


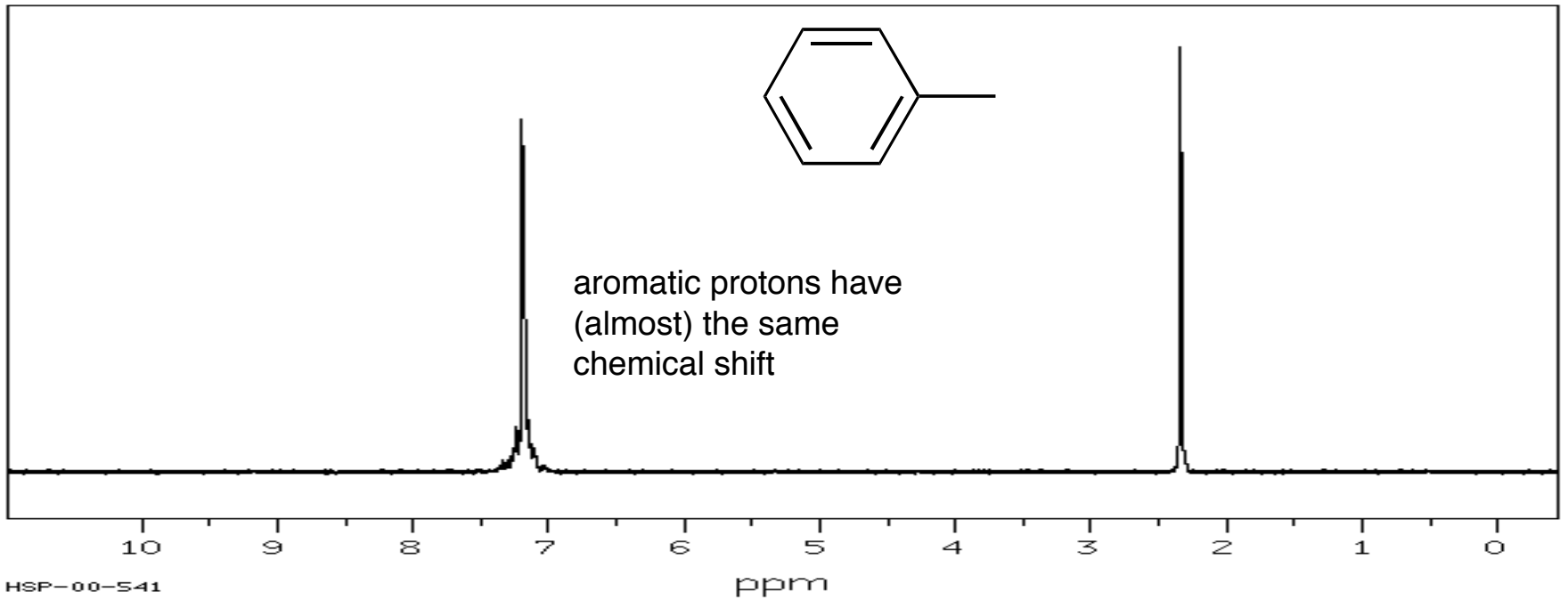
Coupling constant is measured by taking the difference between peaks (in ppm)within a multiplet and multiplying by the field strength in Hz.

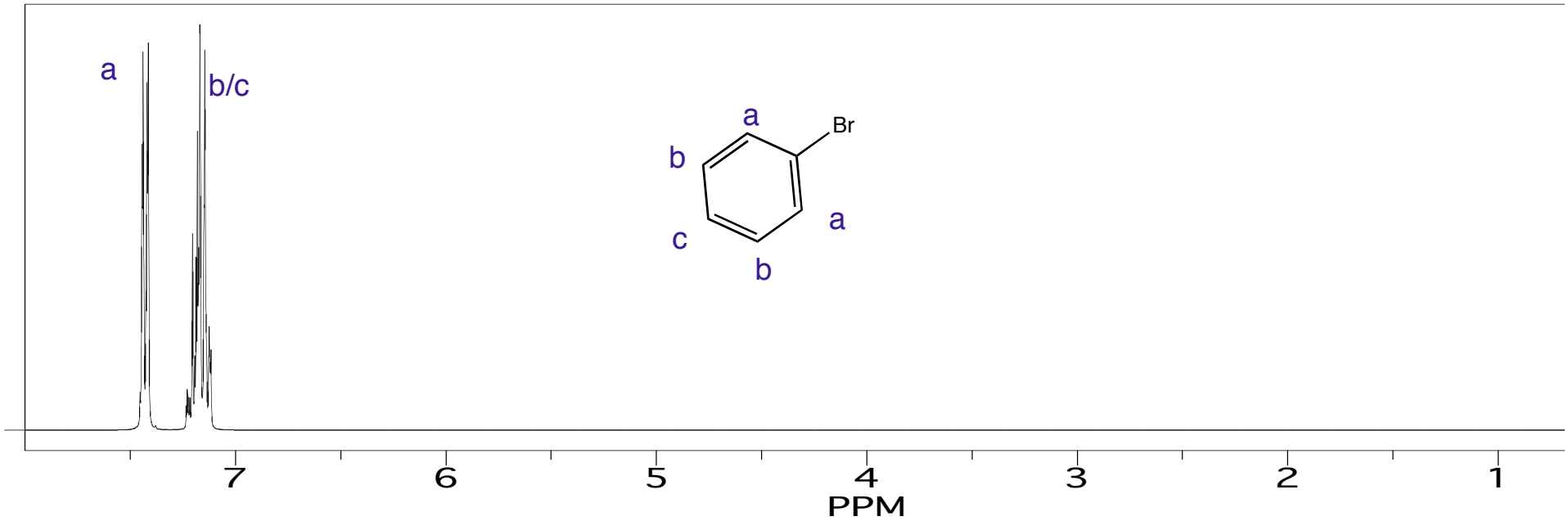
Common Splitting Patterns





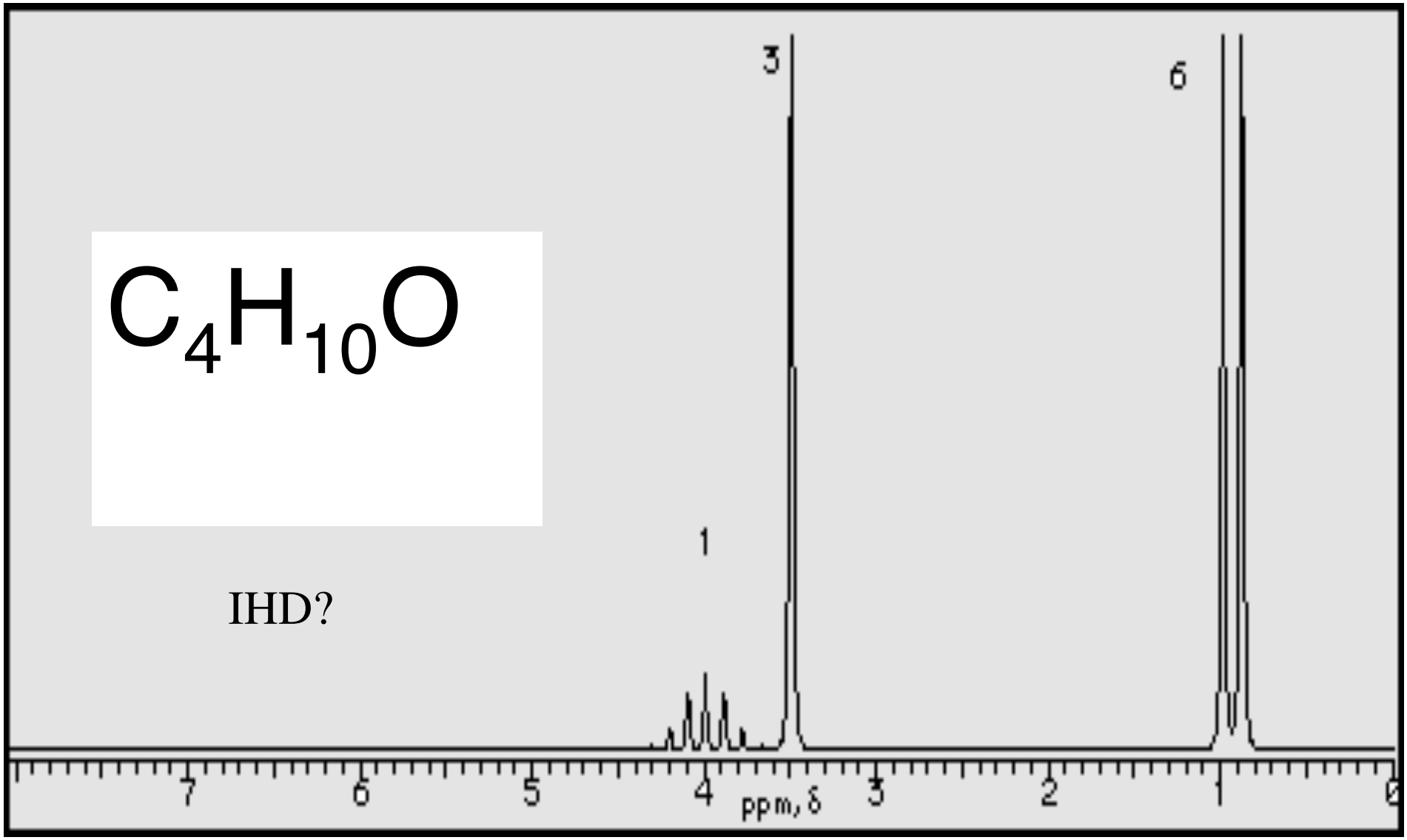

Cl
O
7
6
5
4
3
2
PPM


aromatic protons have
(almost) the same
chemical shift
10
9
8
7
6
5
4
3
2
1
0
HSP-00-541
ppm


a
b/c
a
Br
b
a
c
b
7
6
5
4
3
2
1
PPM

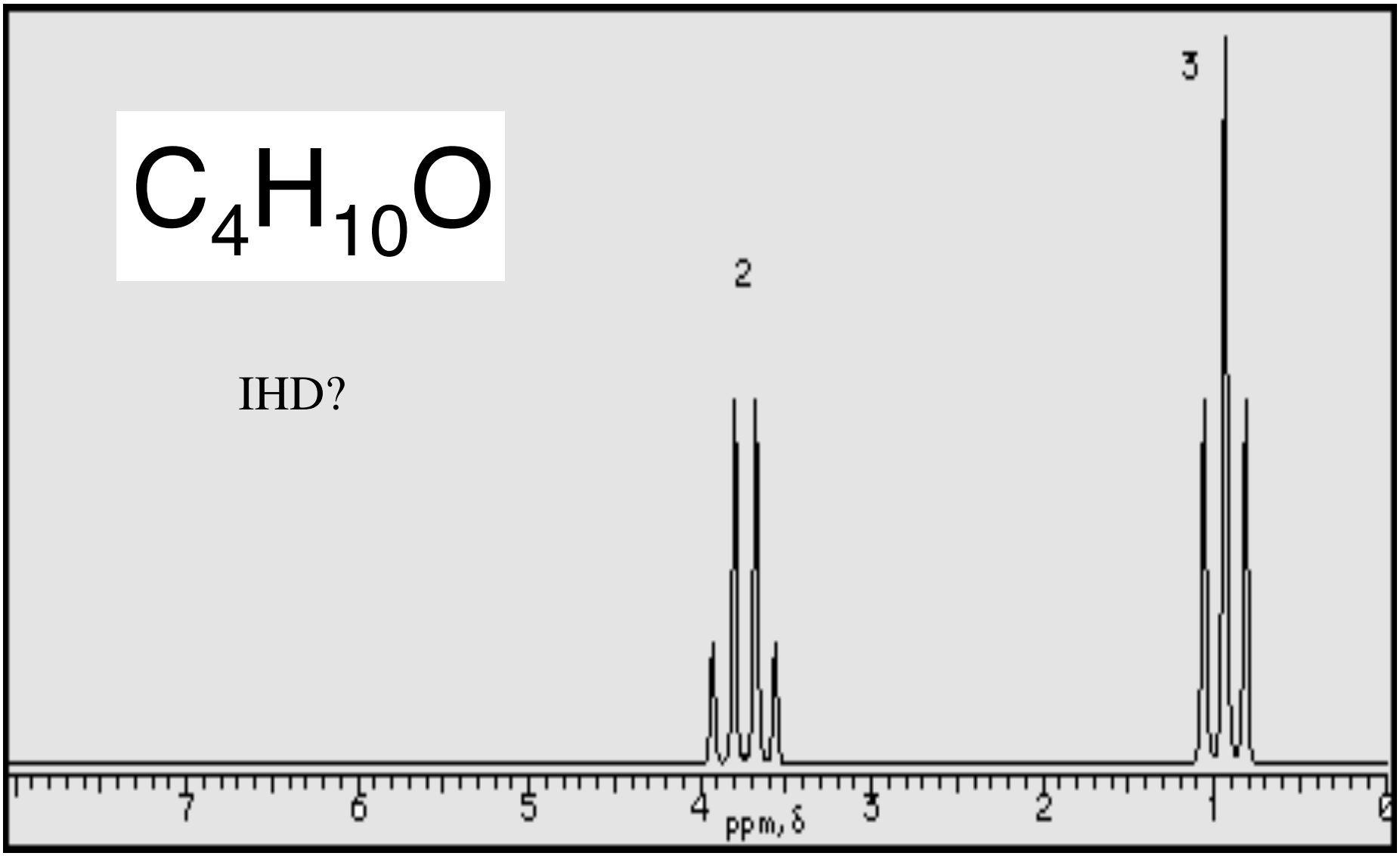
$C_4H_{10}O$

IHD?

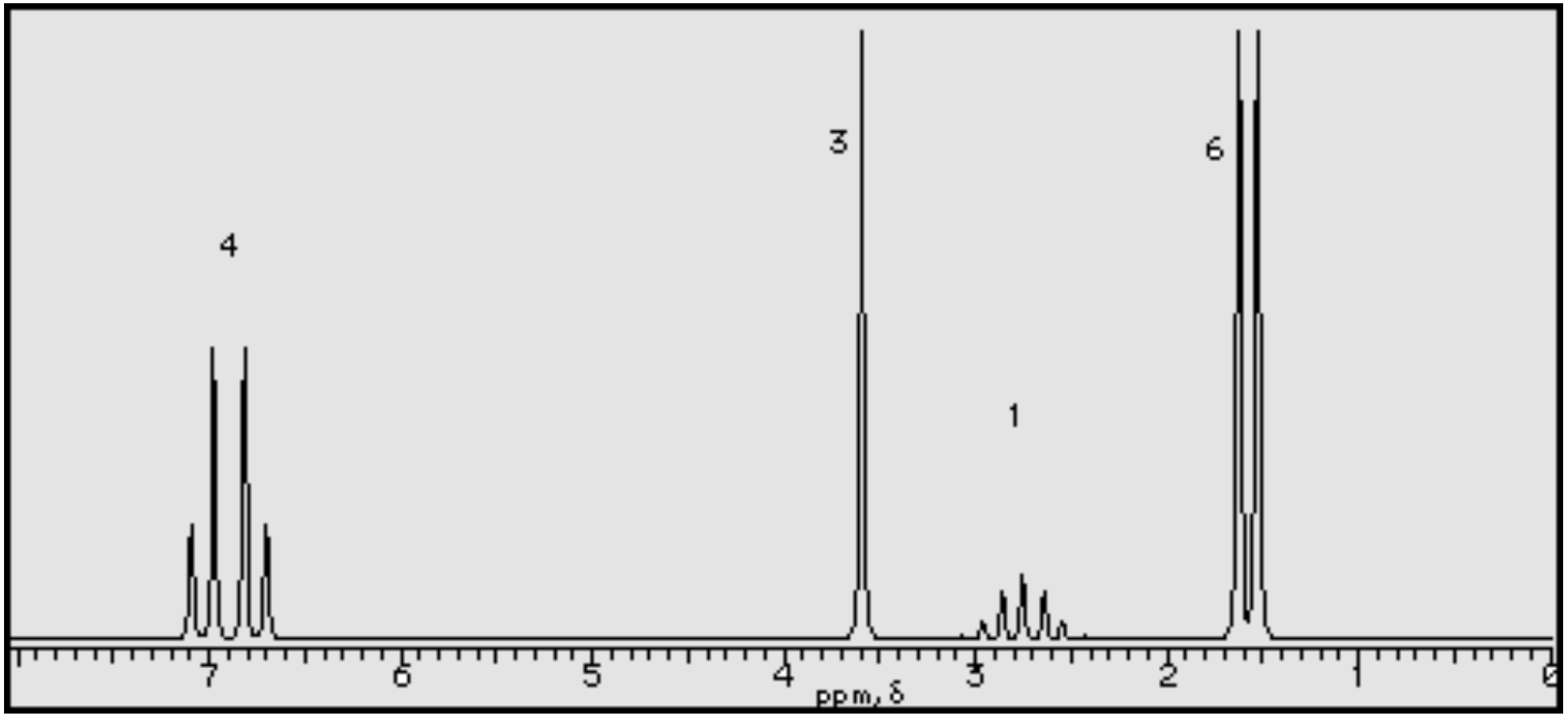


$C_4H_{10}O$

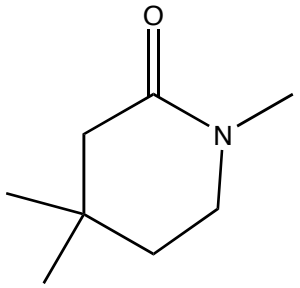
IHD?



$C_{10}H_{14}O$

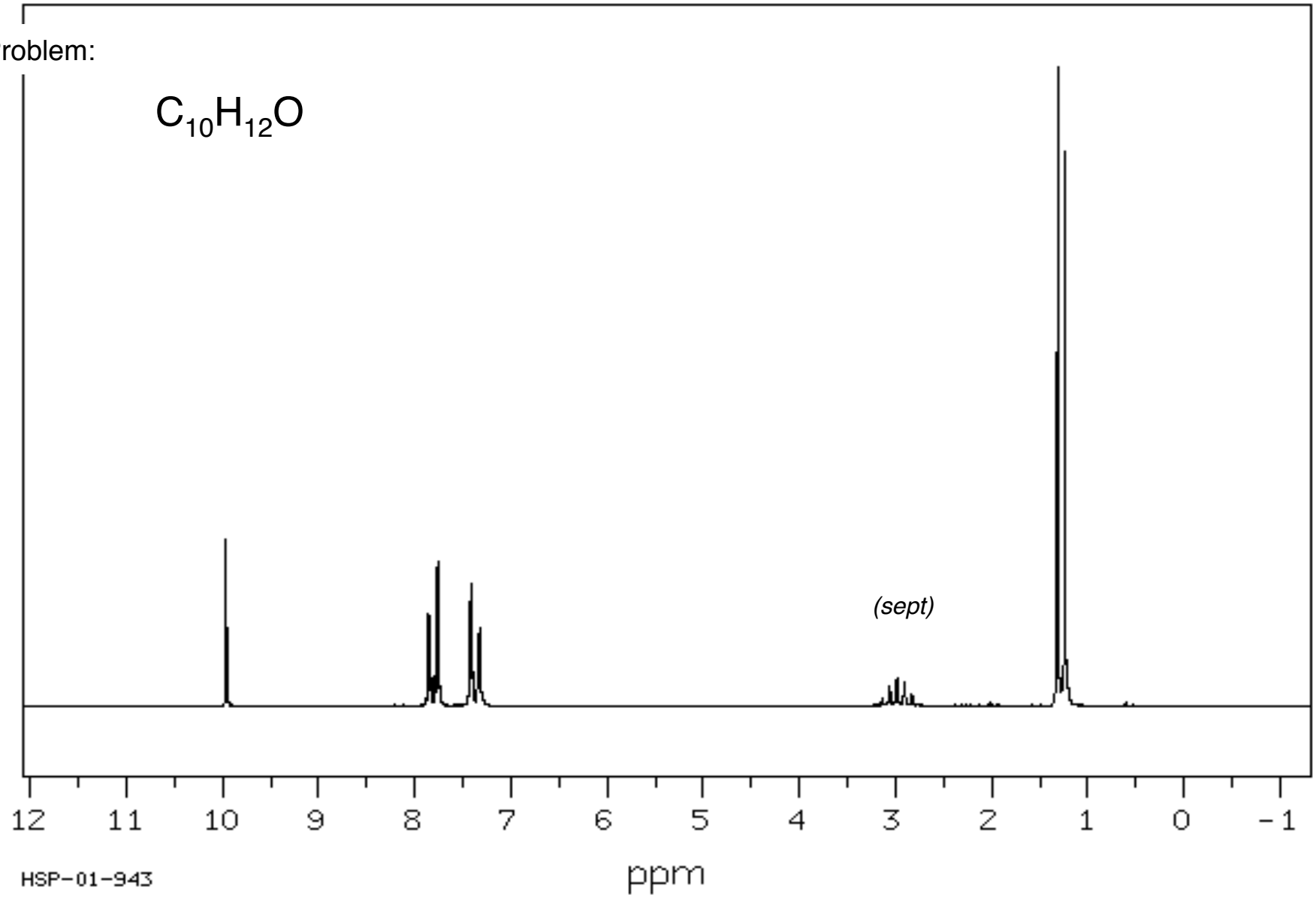


$C_8H_{15}NO$



C-13		**H-1**		
173.2	s	2.90	s	3H
46.9	t	3.2	t	2H
46.5	t	2.10	s	2H
44.2	t	1.47	t	2H
32.6	q	1.11	s	6H
27.4	s			
25.9	q (2)			

Problem:



from: structural database for organic compounds

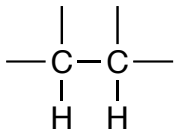
Protons in different chemical structures have different amounts of splitting or "coupling constants".

- Bad News: Life gets more complicated

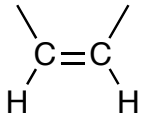
- Good News: Splitting tells us more about the chemical structure.

Coupling Constants Depends on Structure and Geometry

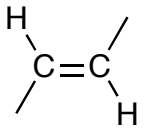
Approximate Coupling Constants.



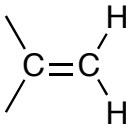
7 Hz
(free rotation)



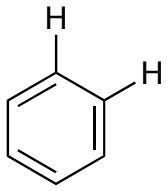
10 Hz



15 Hz

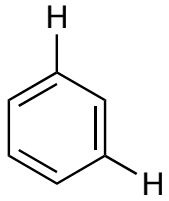


2 Hz

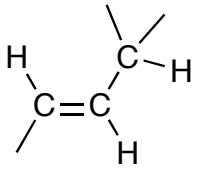


8 Hz

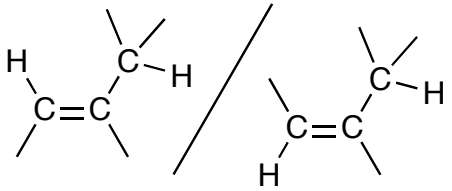
Special Couplings over more than 3 bonds



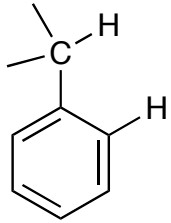
2 Hz



6 Hz

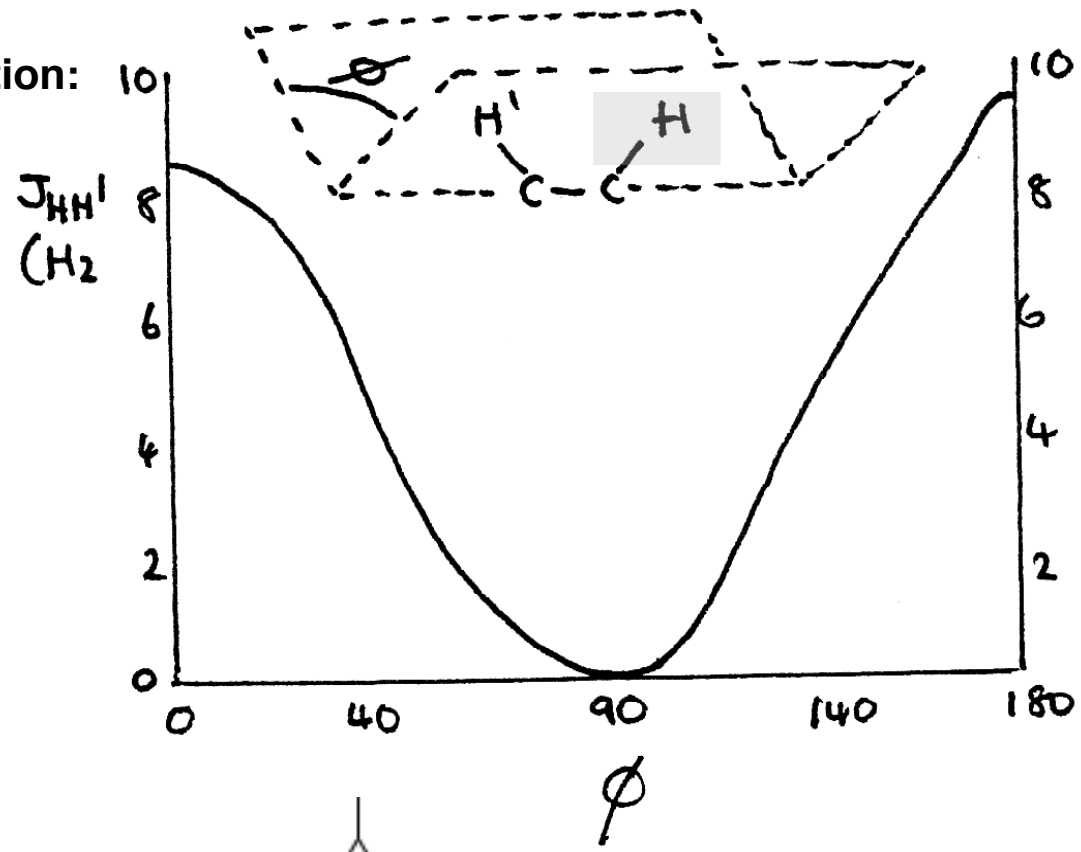


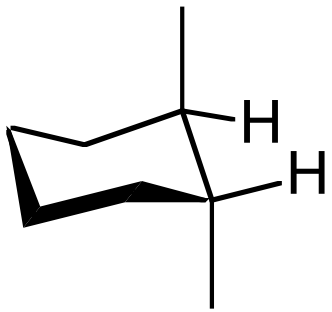
0-3 Hz
depends on geometry



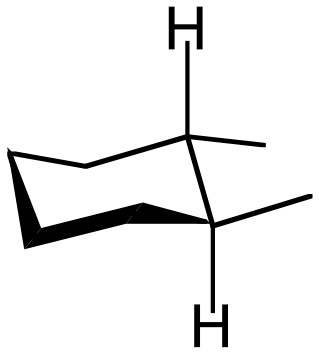
1 Hz

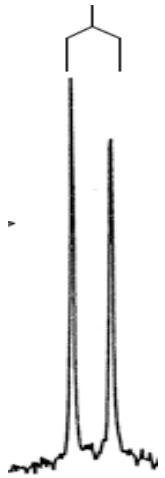
Karplus Equation:





J = 2-3 Hz





J = 8-10 Hz

C

Aldrich M7,970-0 *CAS [108-27-0]*

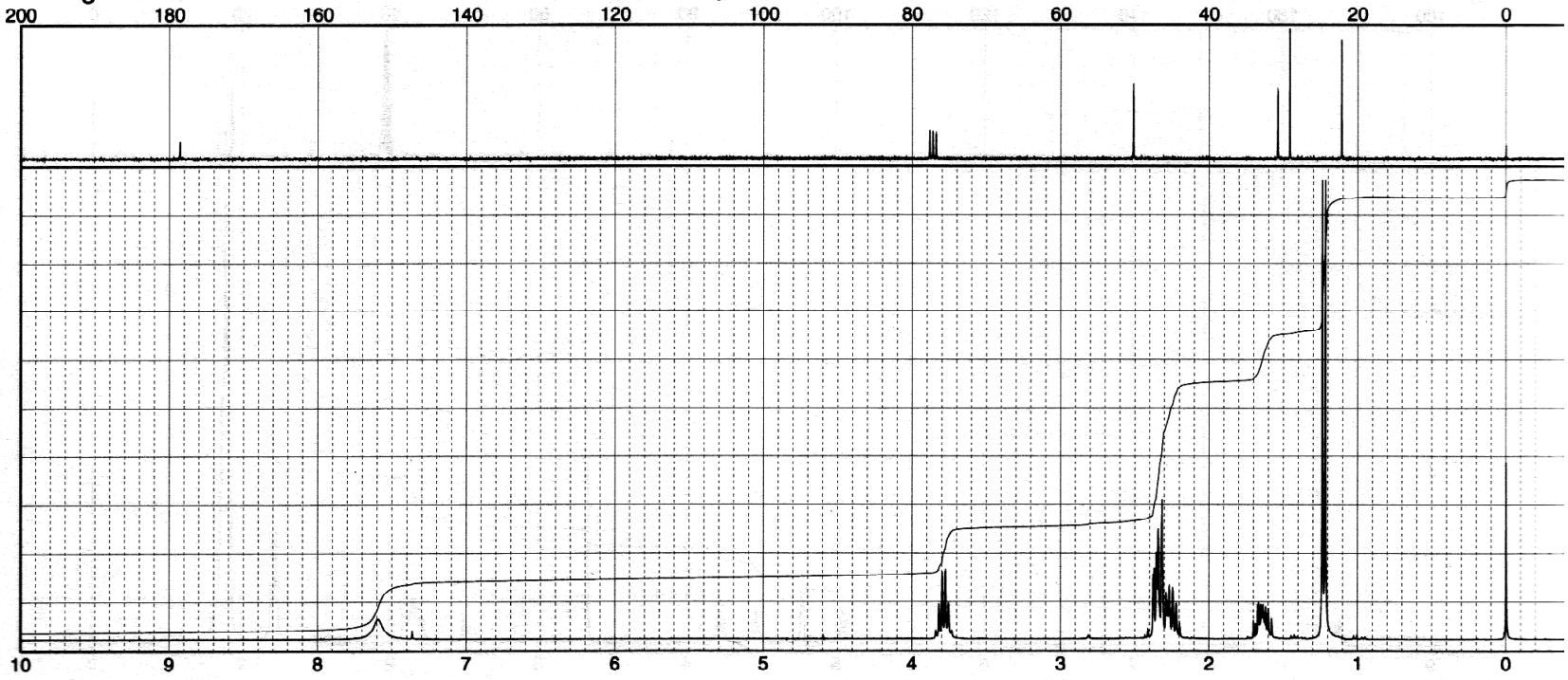
5-Methyl-2-pyrrolidinone, 98%

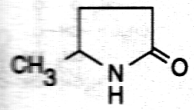
C_5H_9NO
FW 99.13
mp 42°C
bp 248°C
Fp >230°F

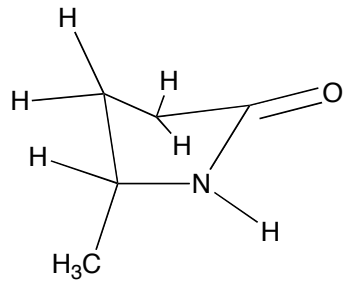
60 MHz: ***1***, 661D
FT-IR: ***1***, 789B
VP-FT-IR: ***3***, 788C

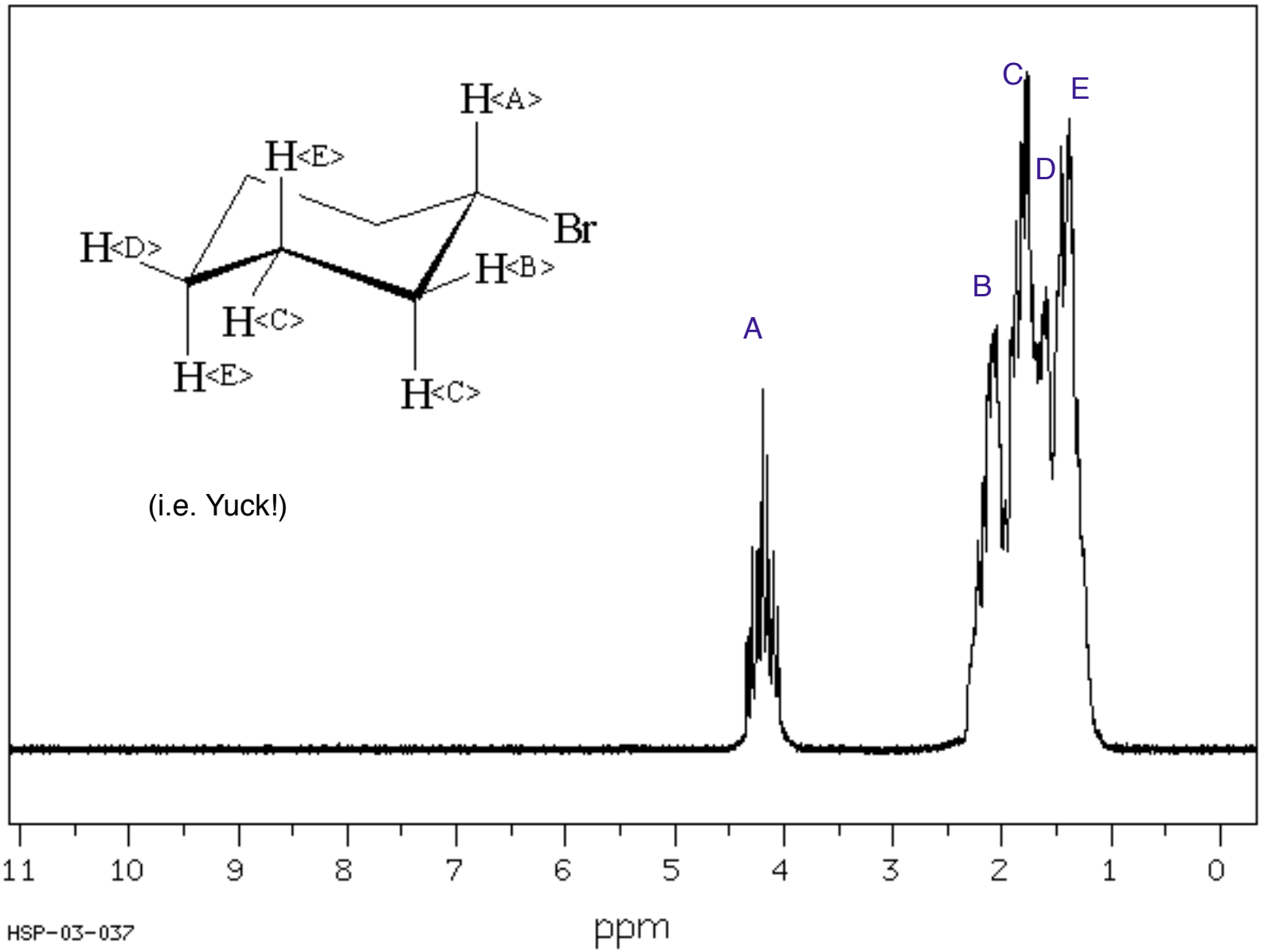
178.56
50.23
30.77
29.17
22.17

$CDCl_3$ QE-300



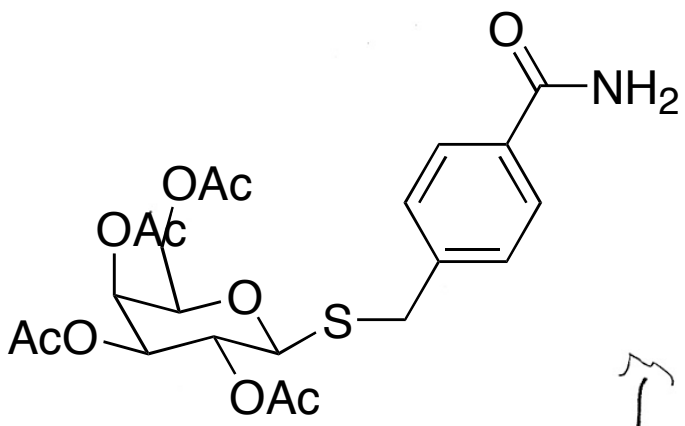




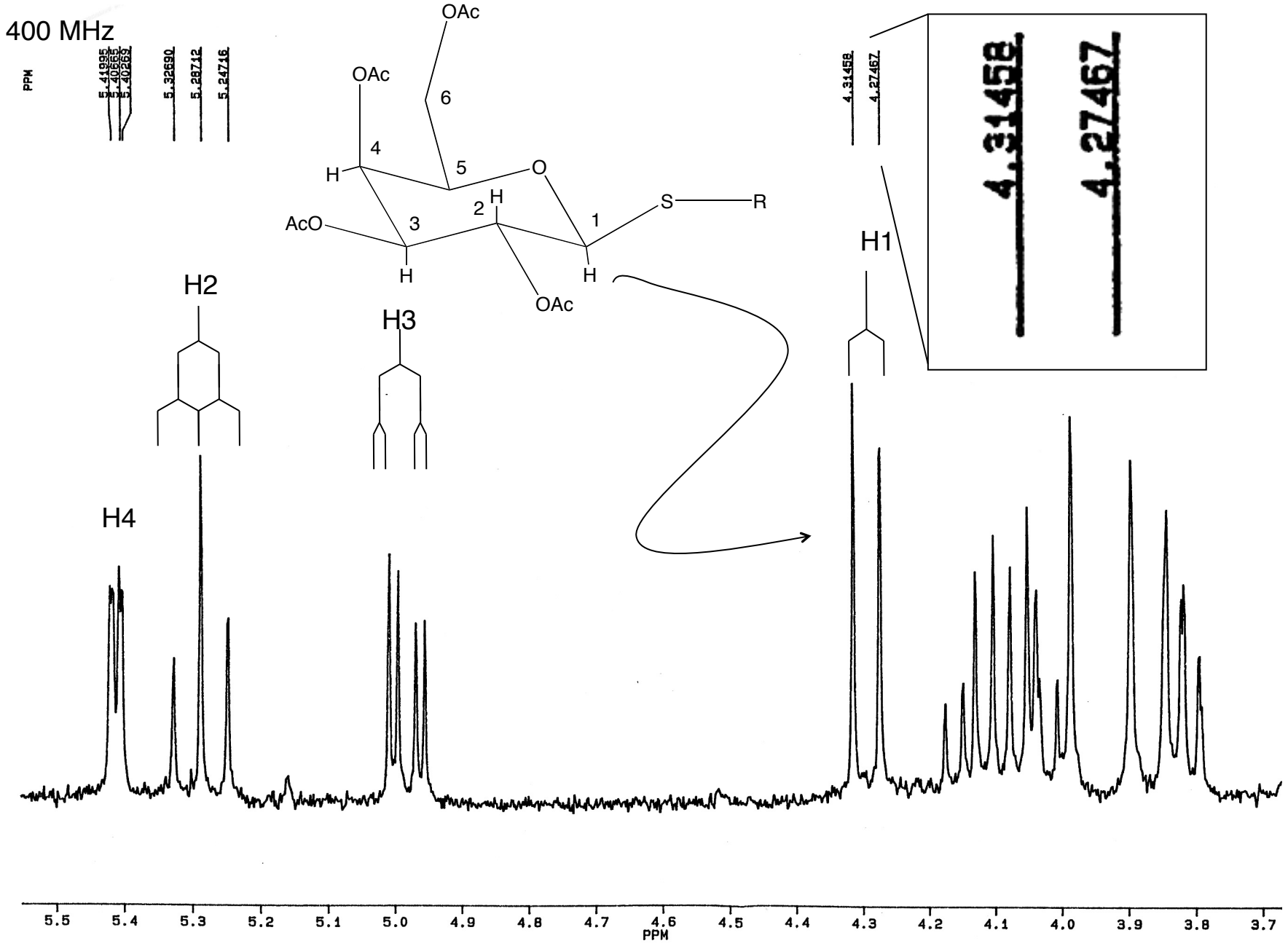

H<A>
H<E>
H<D>
Br
H<B>
H<C>
H<E>
H<C>
(i.e. Yuck!)
A
B
C
D
E
11
10
9
8
7
6
5
4
3
2
1
0
ppm
HSP-03-037

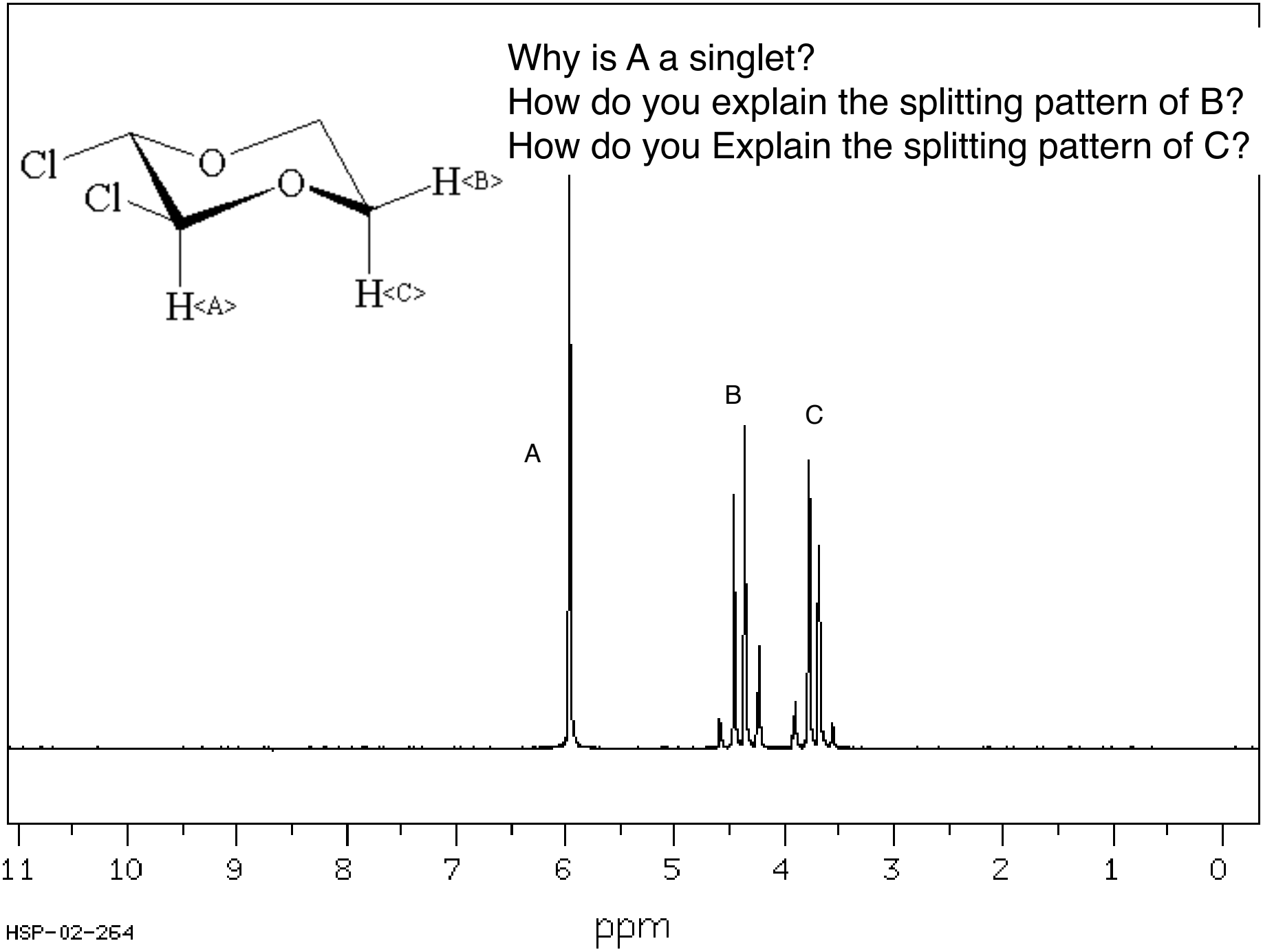
400 MHz


H
H
J = 2-3 Hz
H
H
J = 8-10 Hz

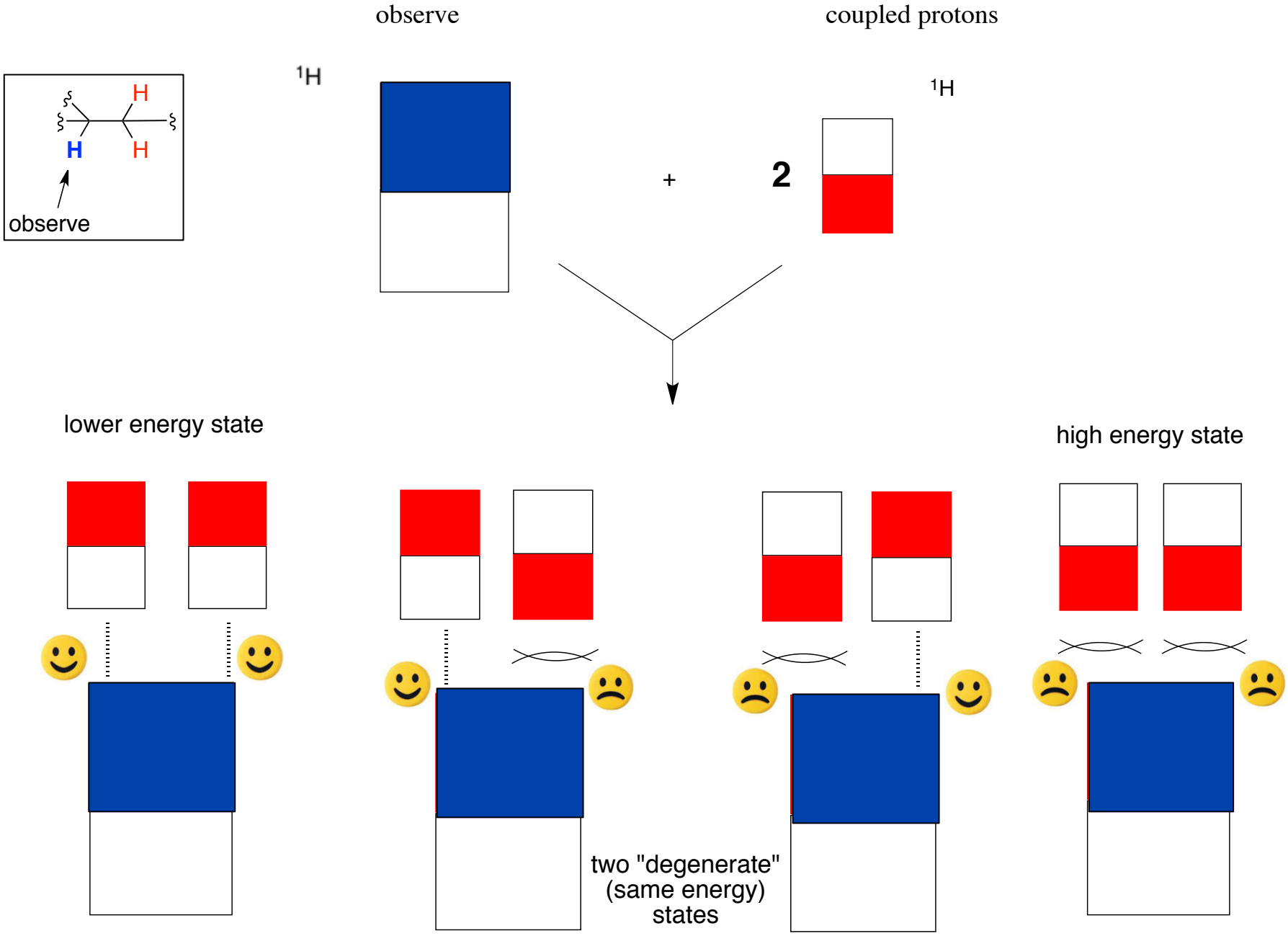

O
NH2
OAc
OAc
O
S
AcO
OAc

1H
1H
1H
1H
197.4
215.1
100.7
107.1
102.7
120.3
353.8
238.6
205.9
305.3
190.6
284.2
318.2
289.9
973.4
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.5
0.0
PPM

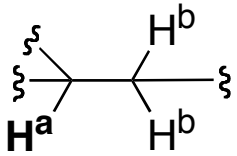

400 MHz
PPM
5.41995
5.40665
5.40269
5.32690
5.28712
5.24716
OAc
OAc
6
4
H
5
O
2
H
S
R
AcO
3
1
H
H
OAc
H1
H2
H3
H4
4.31458
4.27467
4.31458
4.27467
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
PPM

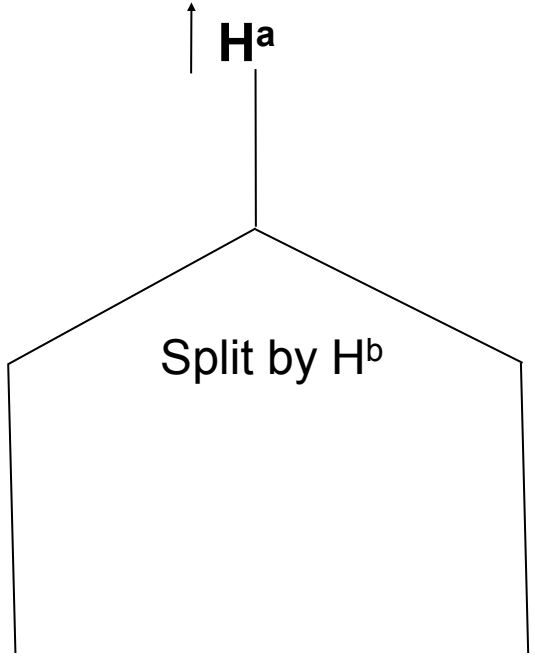

Why is A a singlet?
How do you explain the splitting pattern of B?
How do you Explain the splitting pattern of C?
Cl
Cl
O
O
H<B>
H<A>
H<C>
A
B
C
11
10
9
8
7
6
5
4
3
2
1
0
ppm
HSP-02-264

Magnet analogy

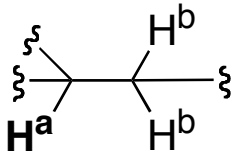


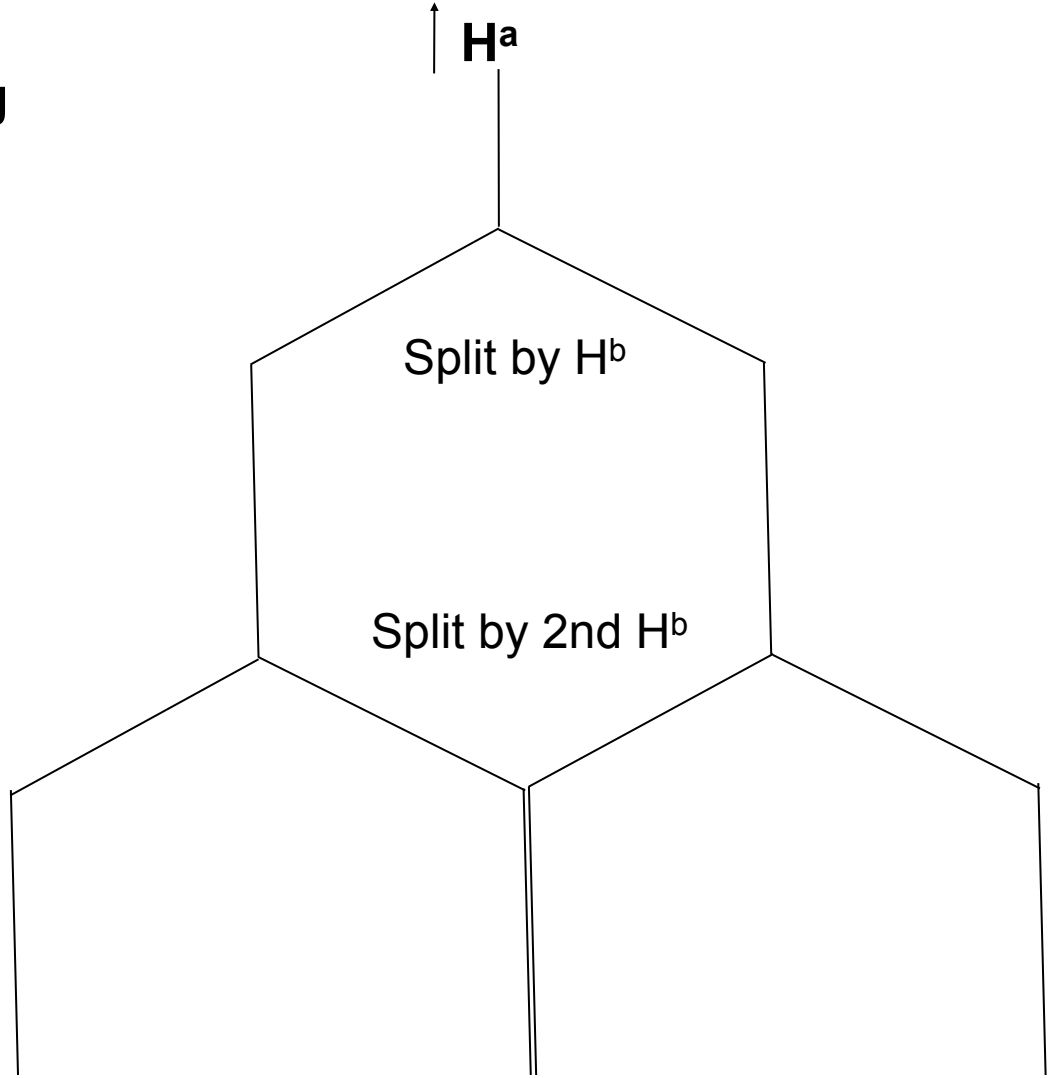
Spin splitting diagrams



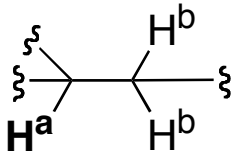


Spin splitting diagrams

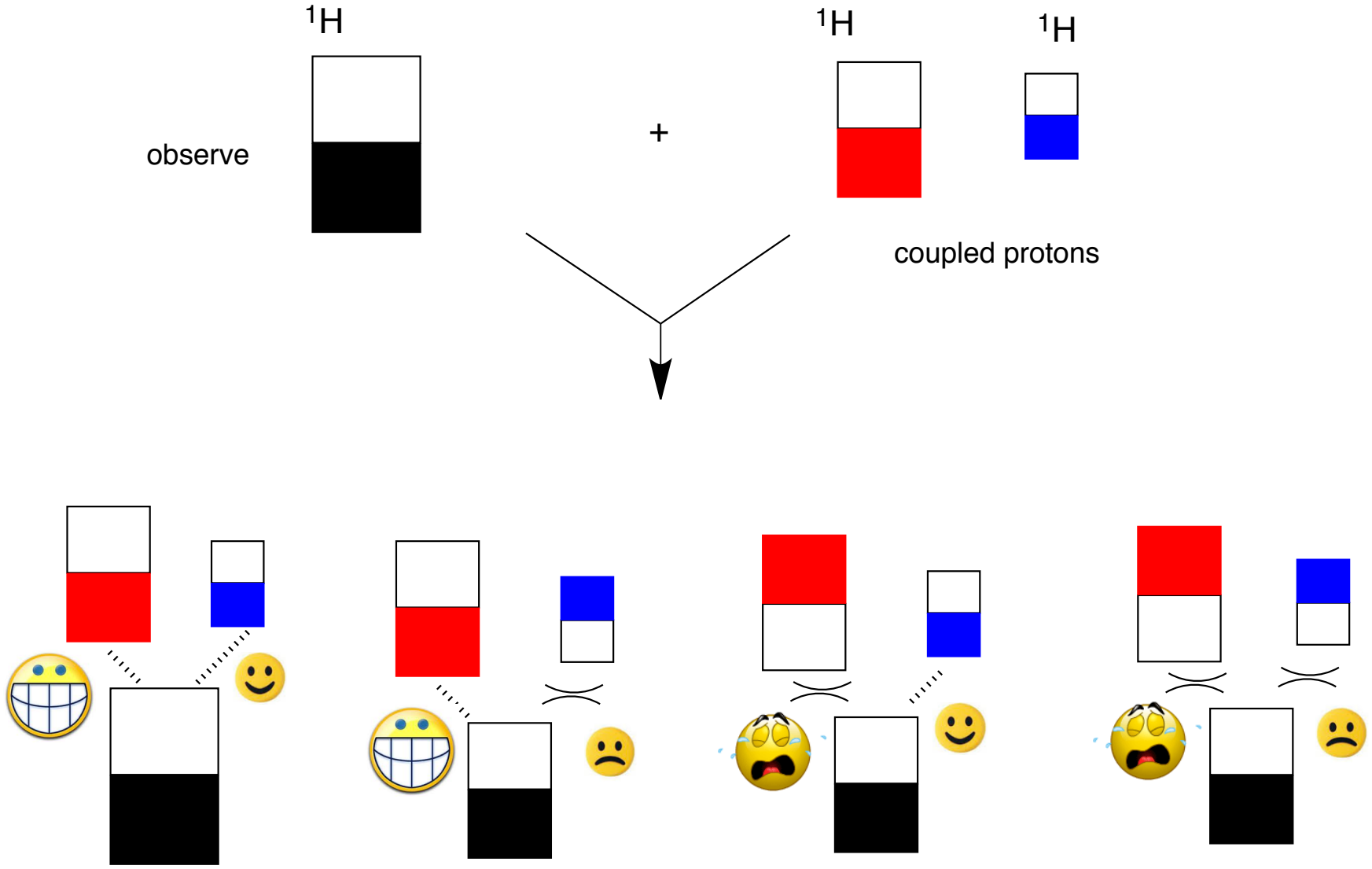




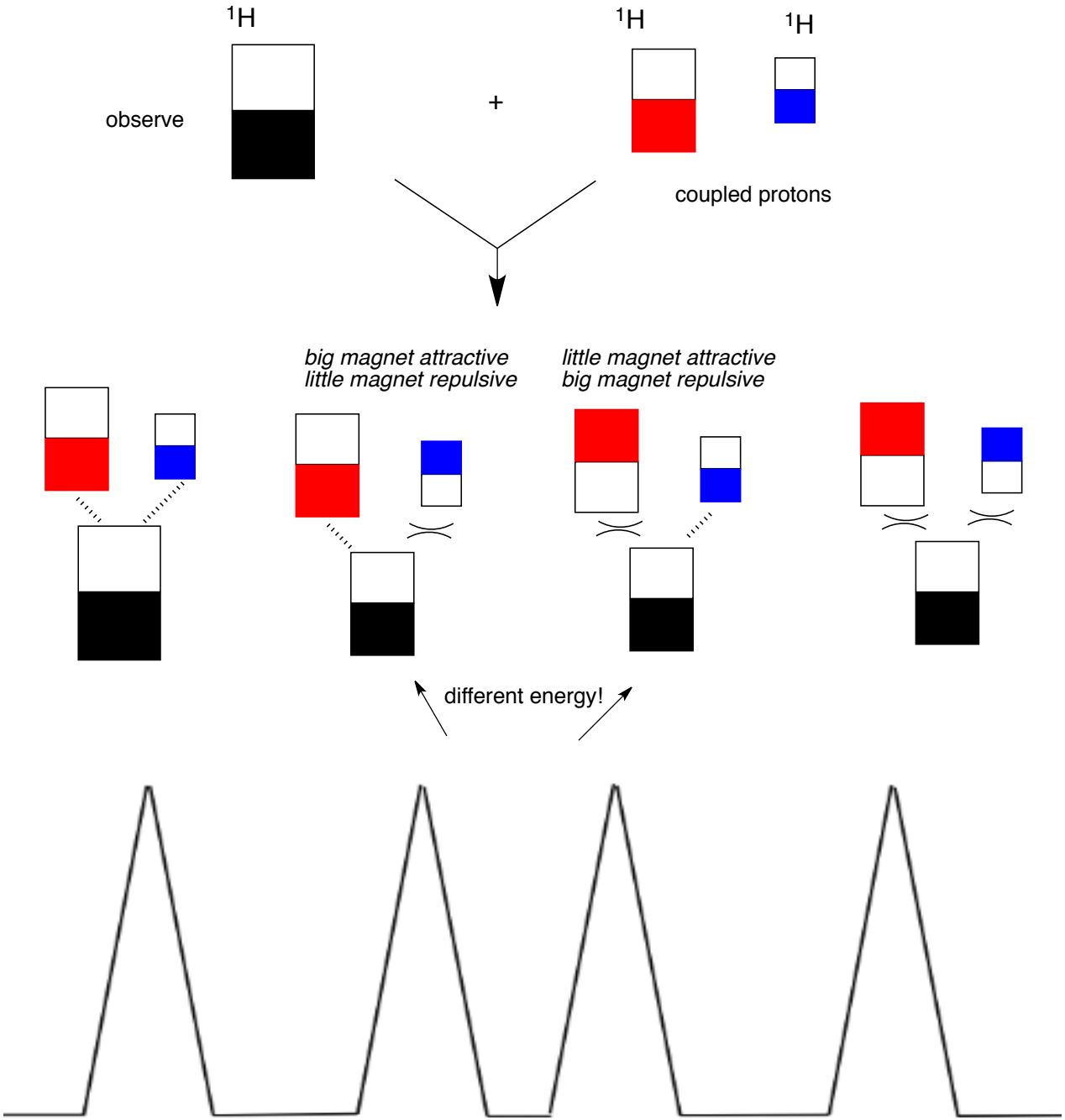
Spin splitting diagrams



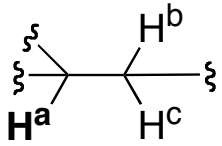
What if we have a strong and weak magnet?



What if we have a strong and weak magnet?

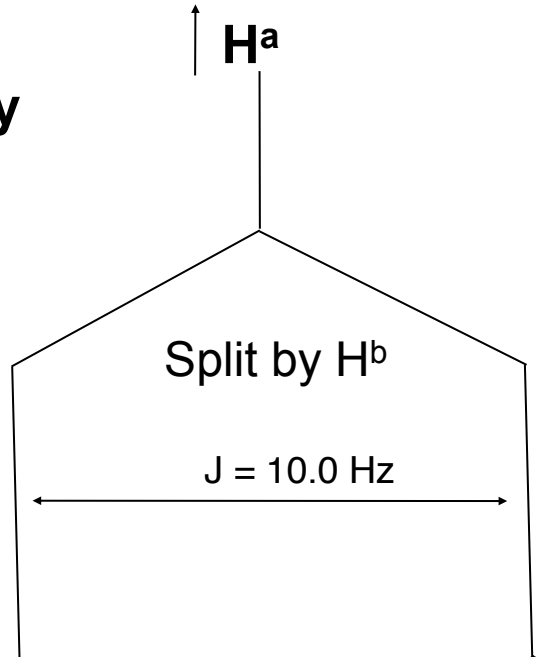


What if the H's are different, and have very different coupling constants??!

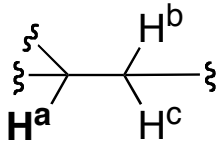


J_{AB} 10 Hz

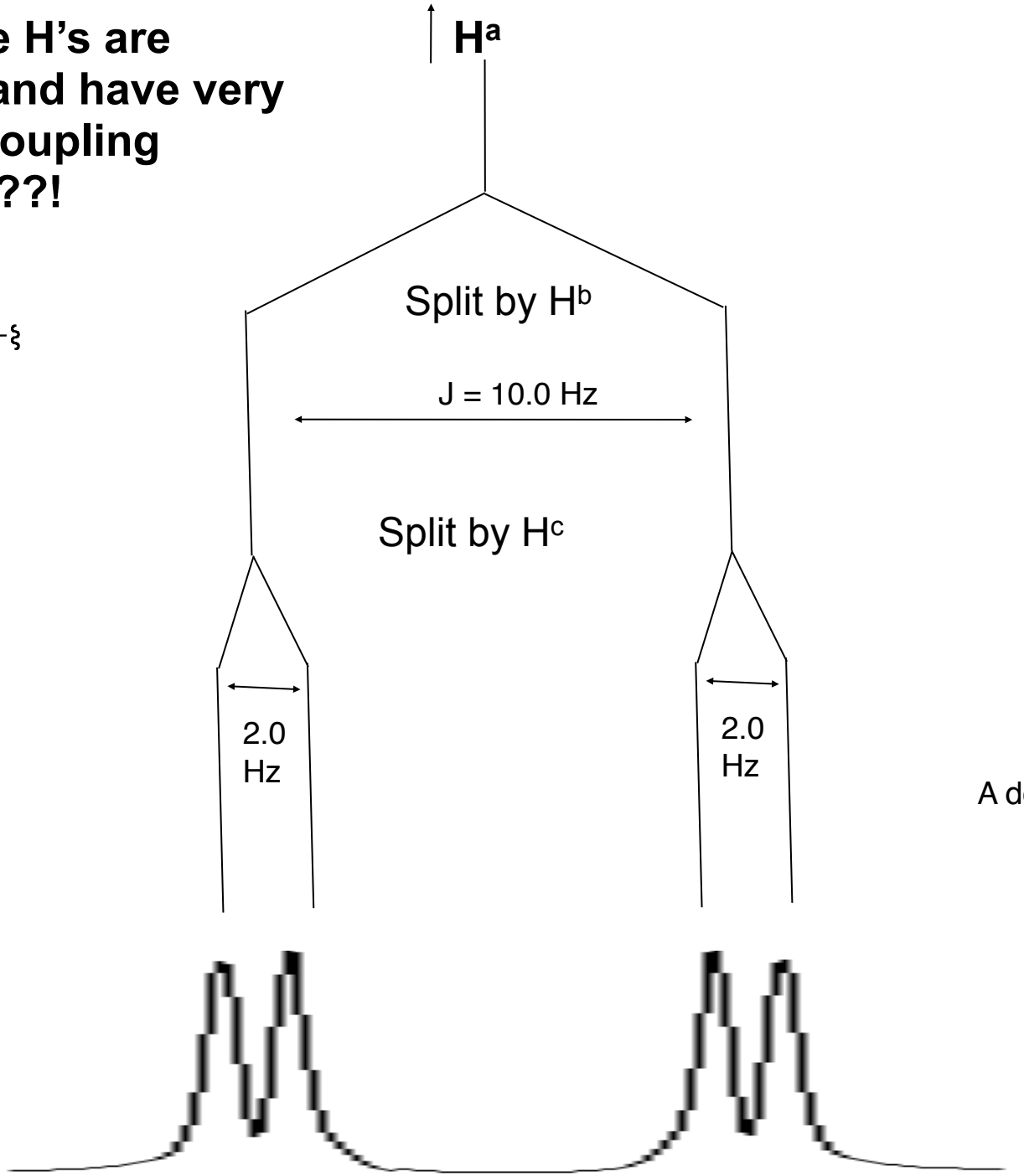
J_{AC} 2 Hz



What if the H's are different, and have very different coupling constants??!



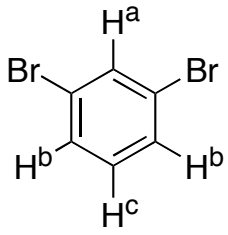
J_{AB} 10 Hz
J_{AC} 2 Hz



A doublet of doublets

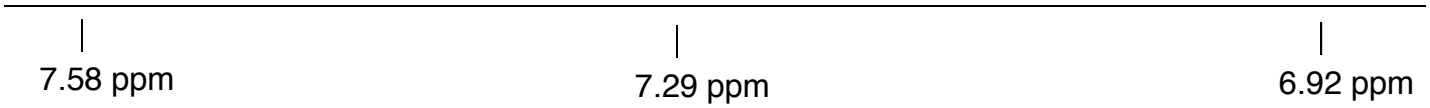
Aromatic (o,m,p) coupling constants

H^a 7.58 ppm
H^b 7.29 ppm
H^c 6.92 ppm



$J(H_{ab})$ 1.9 Hz
$J(H_{bc})$ 8.0 Hz
$J(H_{ac})$ 0.3 Hz

3 doublets of doublets



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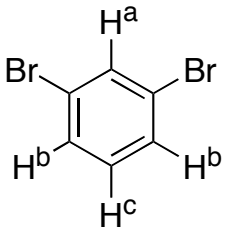

$J(H_{ab})$ 1.9 Hz
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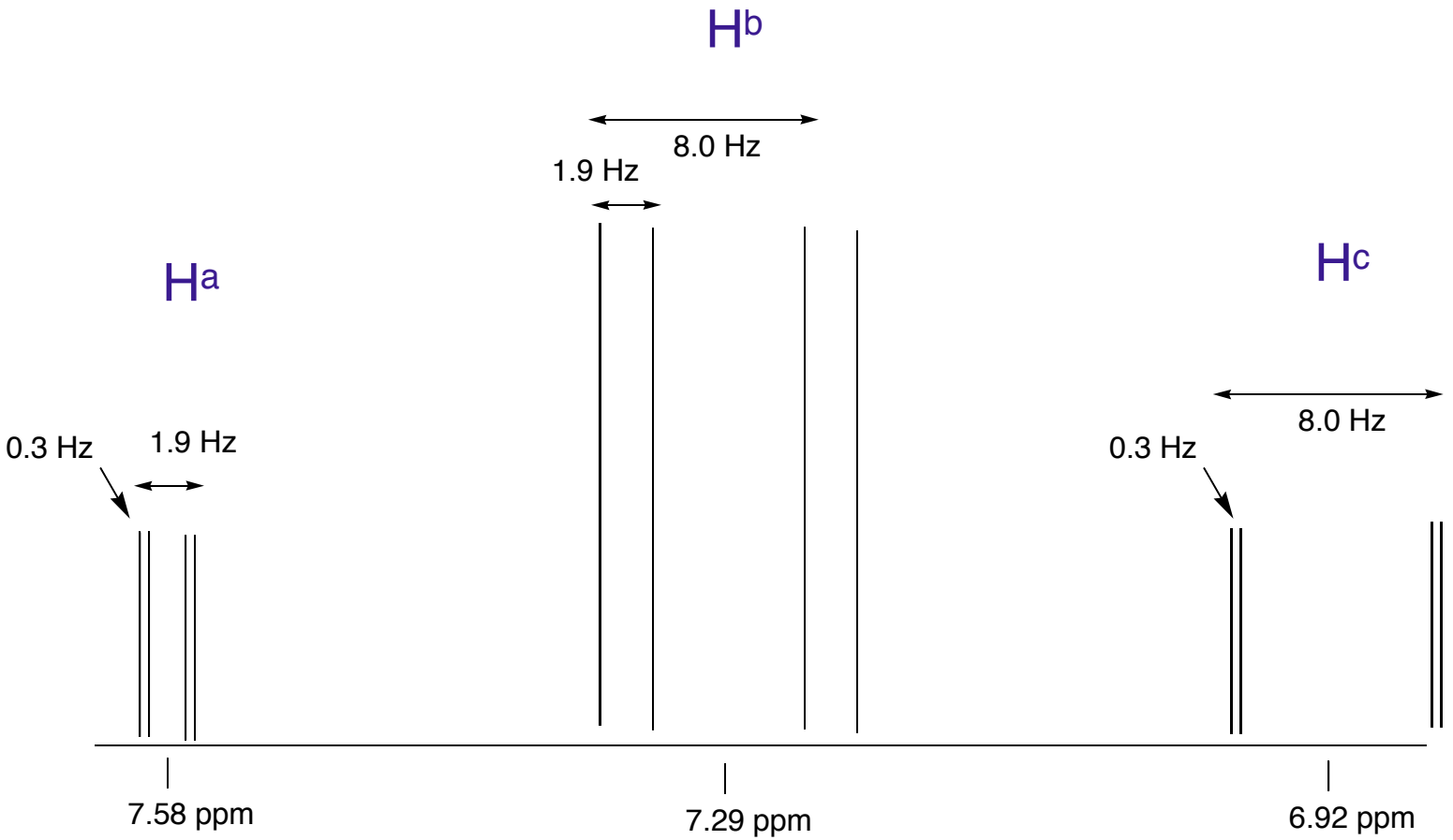
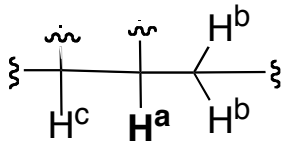
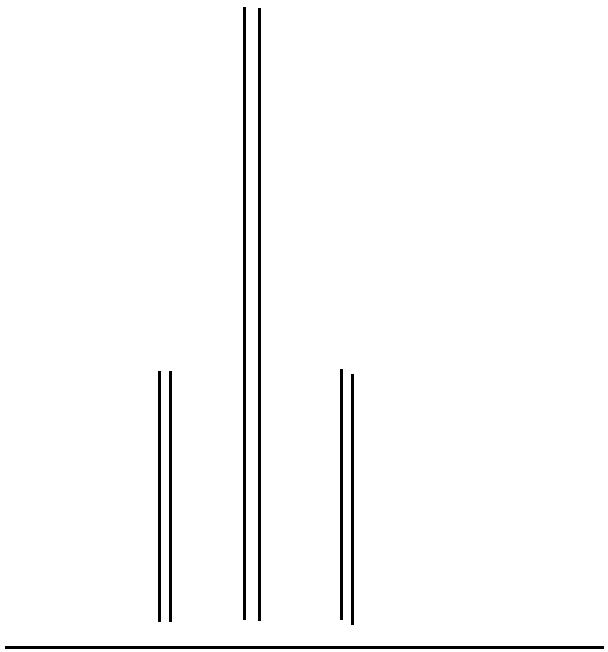
3 doublets of doublets



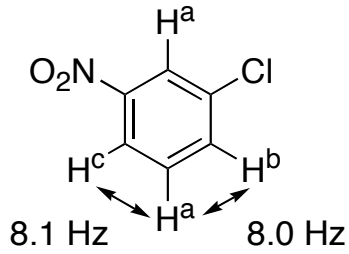
‘Doublets of triplets’ and ‘doublets of quartets’

where $J_{ab} \neq J_{ac}$





What if Jab ~ Jac



Still looks like a triplet, even on a high field spectrometer:

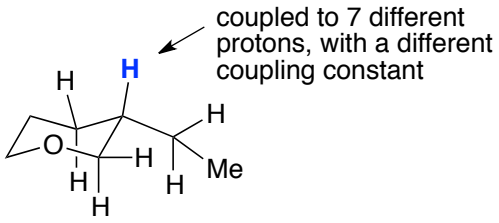
Referred to as an

‘apparent triplet’

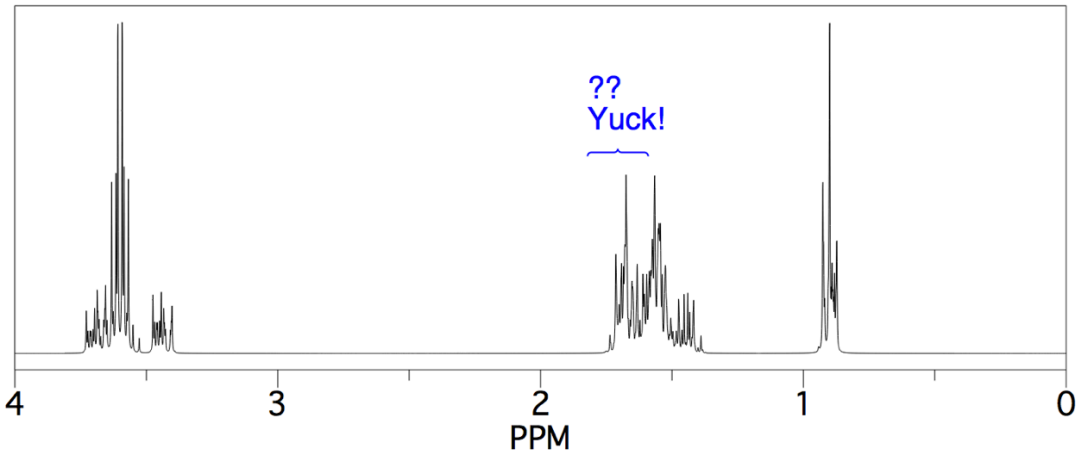
When the going gets really tough...

... we call things a **multiplet (m)**

situation 1: coupling pattern is very complex



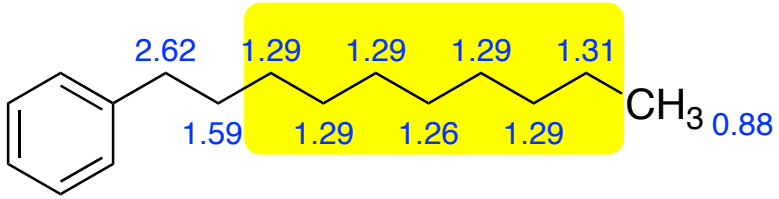
described as: 1.5–1.3 (**m**, 1H)



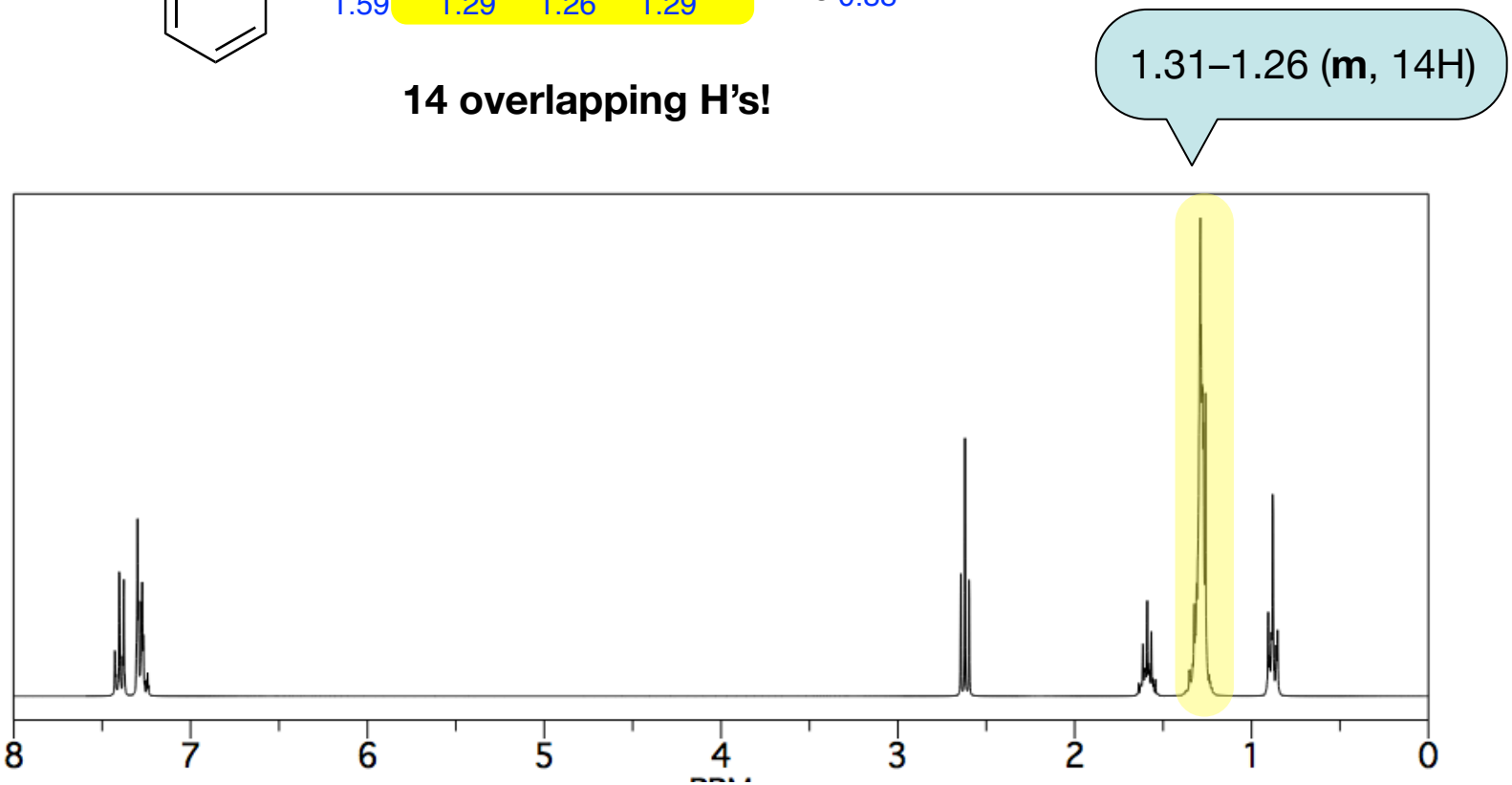
When the going gets really tough...

... we call things a **multiplet (m)**

situation 2: your peak overlaps with other resonances



14 overlapping H's!

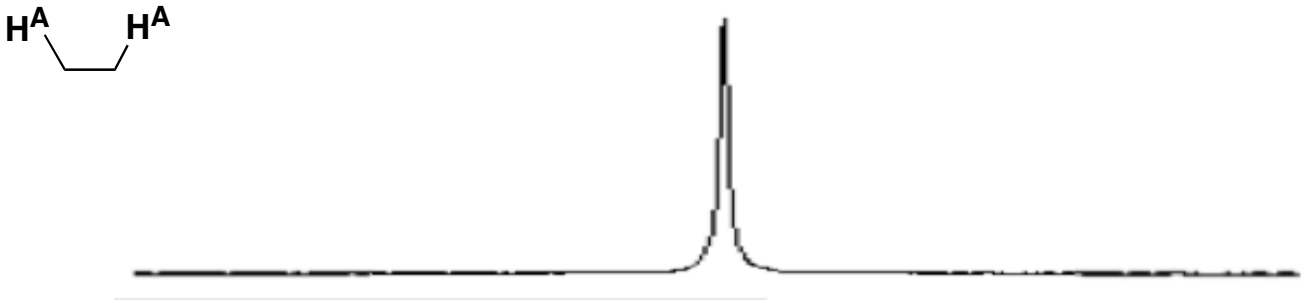


skip to slide 70

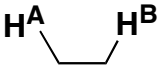
Higher order effects in NMR

When chemical shifts are different: 2 doublets

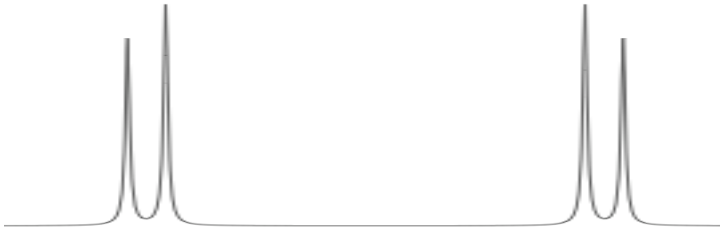
When chemical shifts are the same, coupling is not observed



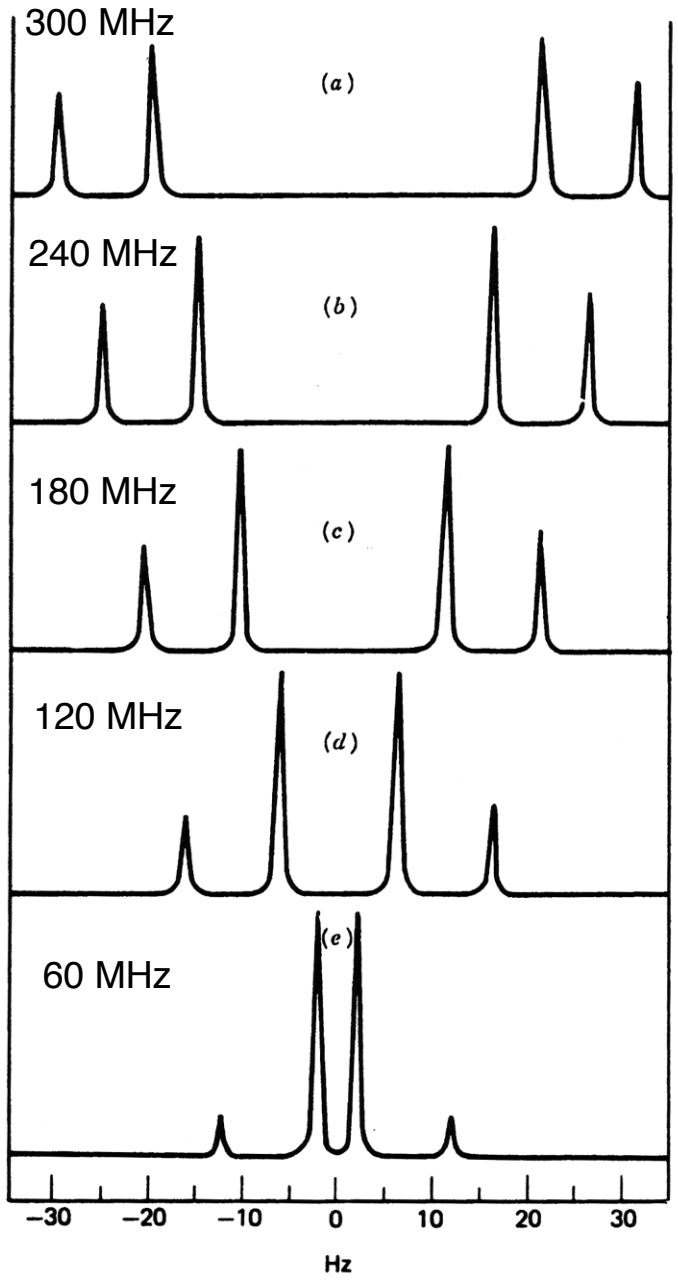
What if chemical shifts are similar but not identical?



We observe ‘collapsed’ doublets



WHEN PROTONS DON'T SPLIT!!



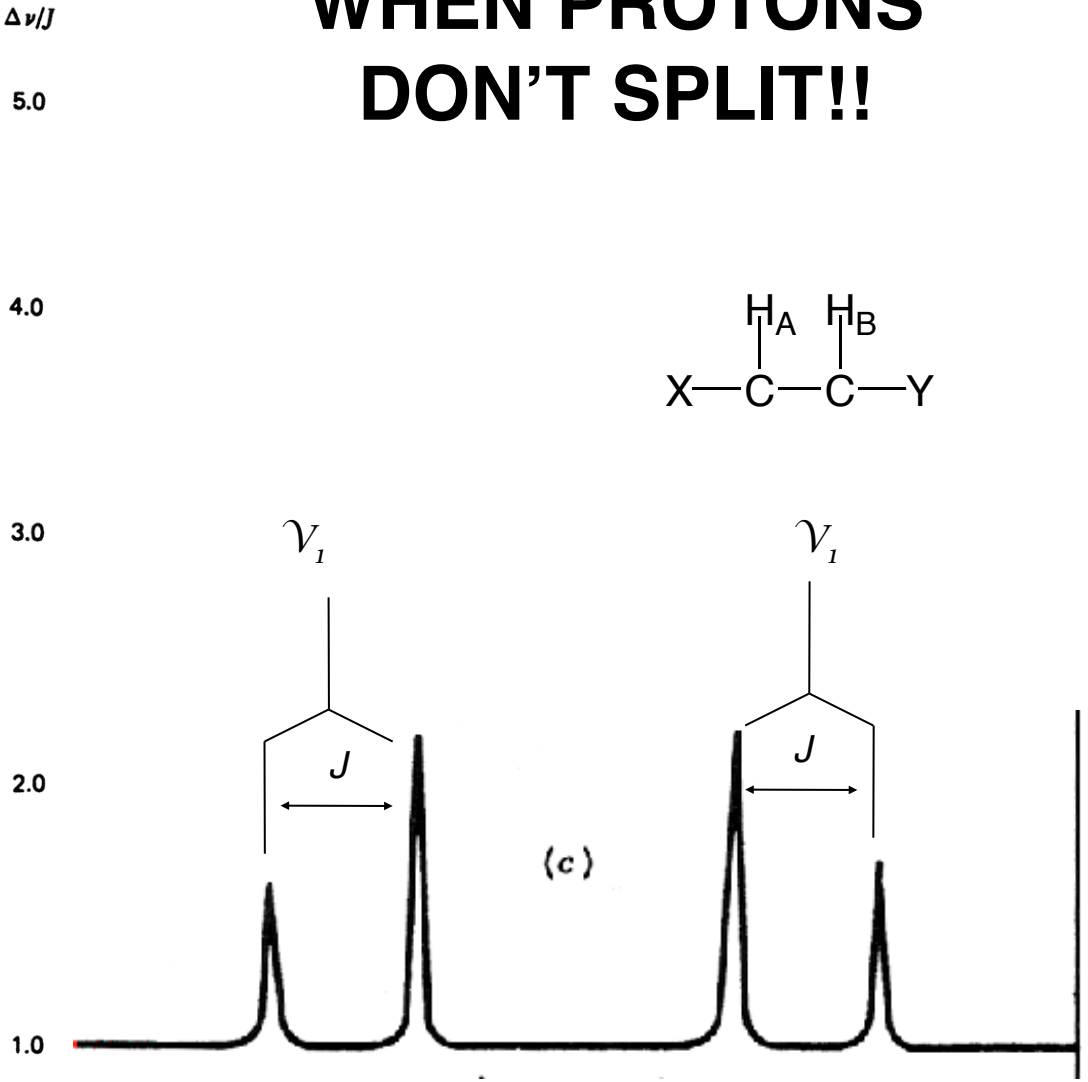


FIGURE 4.23. A two-proton system spin coupling with a decreasing difference in chemical shifts and a large *J* value (10 Hz); the difference between *AB* and *AX* notation is explained in the text (p. 179).

Dynamic NMR

What happens when we take an NMR spectrum of a molecule in conformational equilibrium?

Try taking a picture of a fan spinning ~40 times a second (i.e. 40 Hz). Your picture will depend on your shutter speed

Shutter speed

1/10th Second - total blur
You photograph the average

1/40th Second - similar to the frequency of the fan

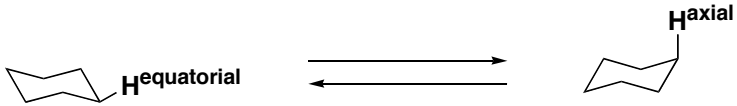
"coalescence" (in NMR speak) is the point where the fan speed and the shutter speed are ~equal

1/200th Second - no movement

In NMR, the movement of the fan is analogous to movement of the molecule, and the frequency of the magnet is equivalent to shutter speed

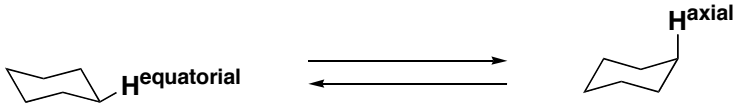
http://cameradojo.com/2008/08/31/capturing-a-sense-of-motion-with-shutter-speed/

Dynamic NMR

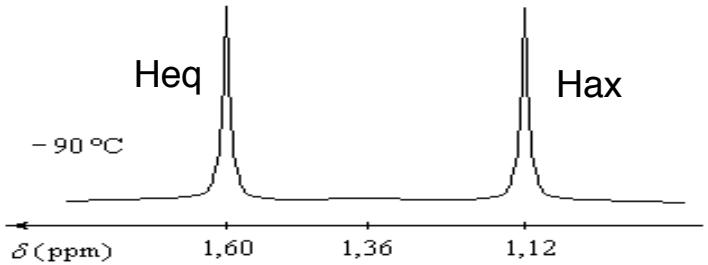


The rate of interconversion (fan speed) is slower at low temperature

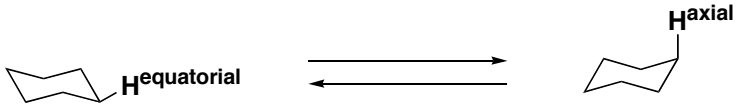
Dynamic NMR



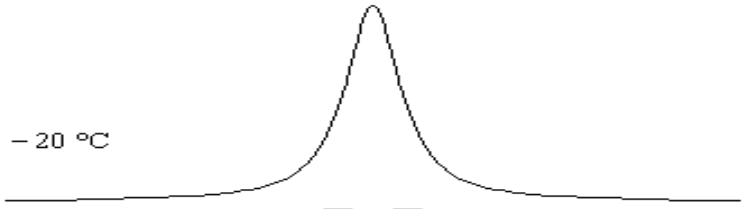
The rate of interconversion (fan speed) is slower at low temperature

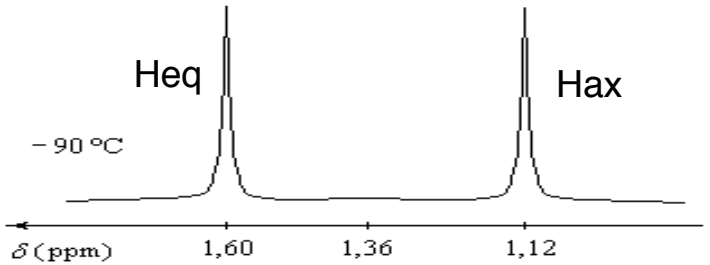


Dynamic NMR

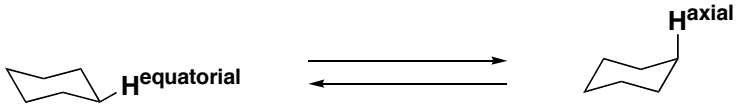


The rate of interconversion (fan speed) is slower at low temperature

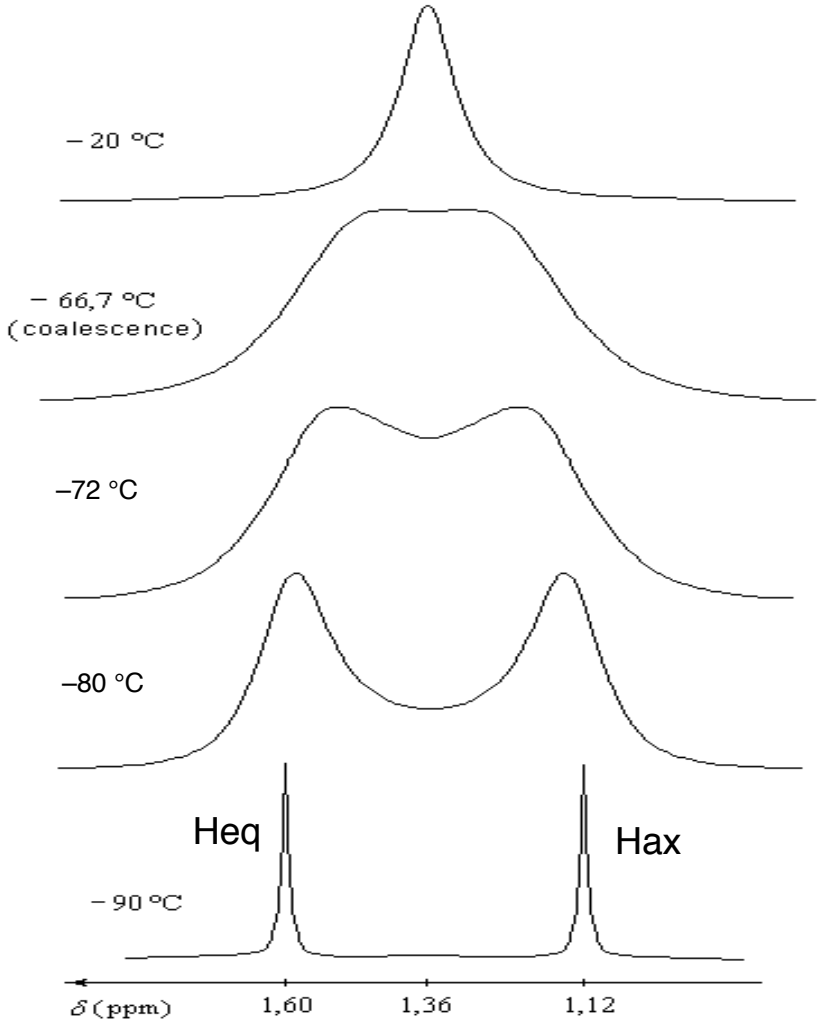




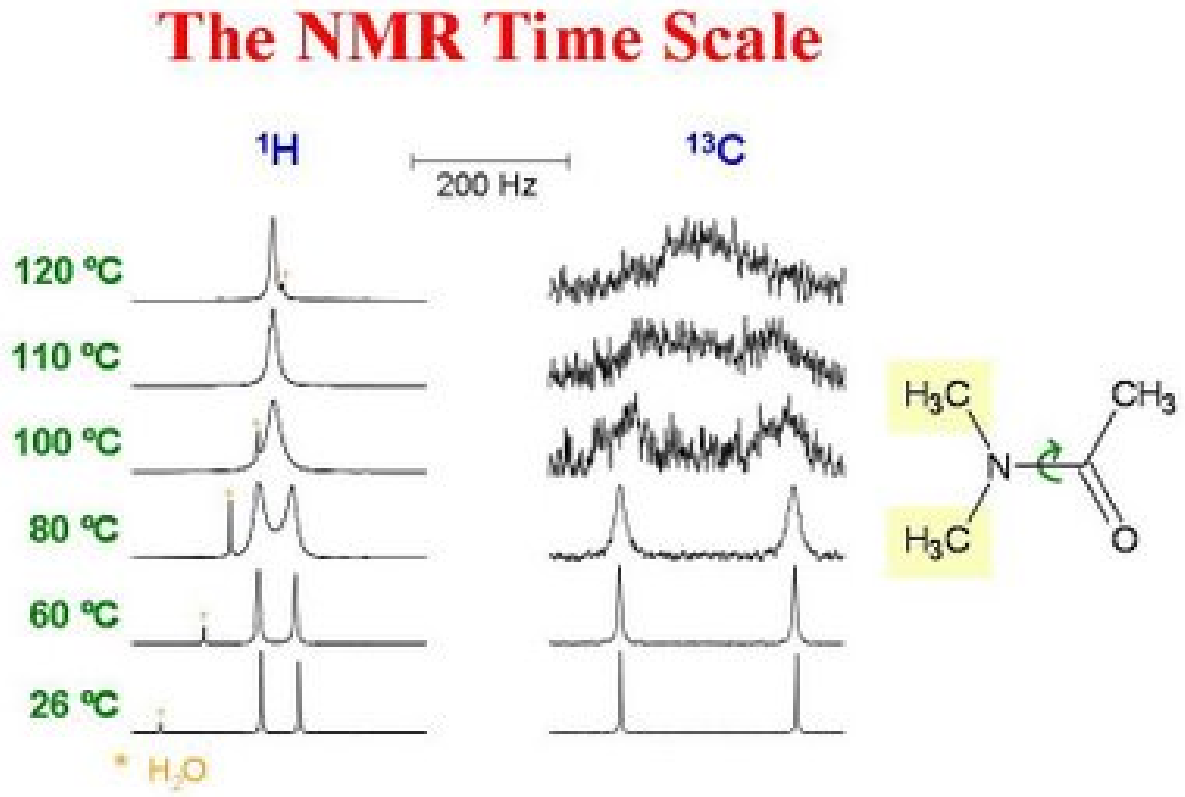
Dynamic NMR



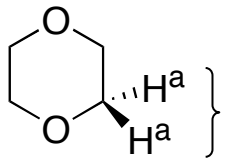
The rate of interconversion (fan speed) is slower at low temperature



Dynamic NMR

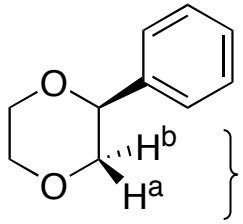

The NMR Time Scale
^{1}H
200 Hz
^{13}C
120 °C
110 °C
100 °C
80 °C
60 °C
26 °C
* H_2O
H_3C
CH_3
N
H_3C
O

Diastereotopic Protons



Same chemical shift

No coupling

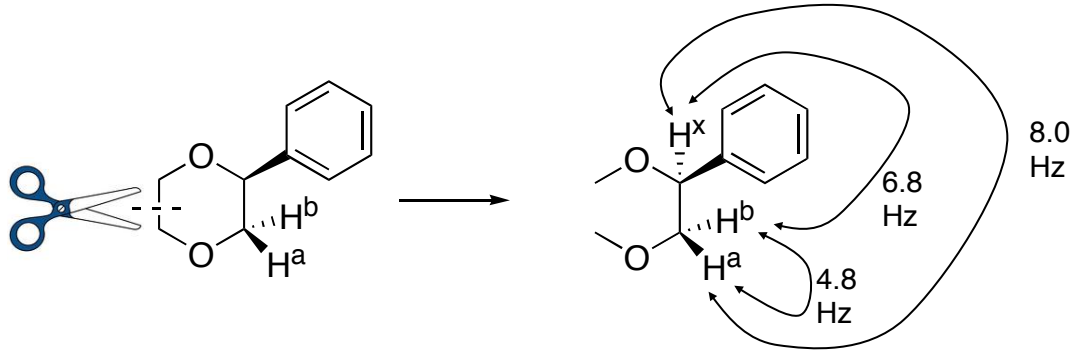


Different! “diastereotopic” protons

Diastereotopic protons:

- CH_2 nearby a chiral center
- Chemical shifts differ (but may overlap in some cases)
- Coupling can often be observed

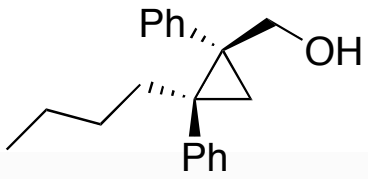
Diastereotopic Protons: not just in cyclic systems



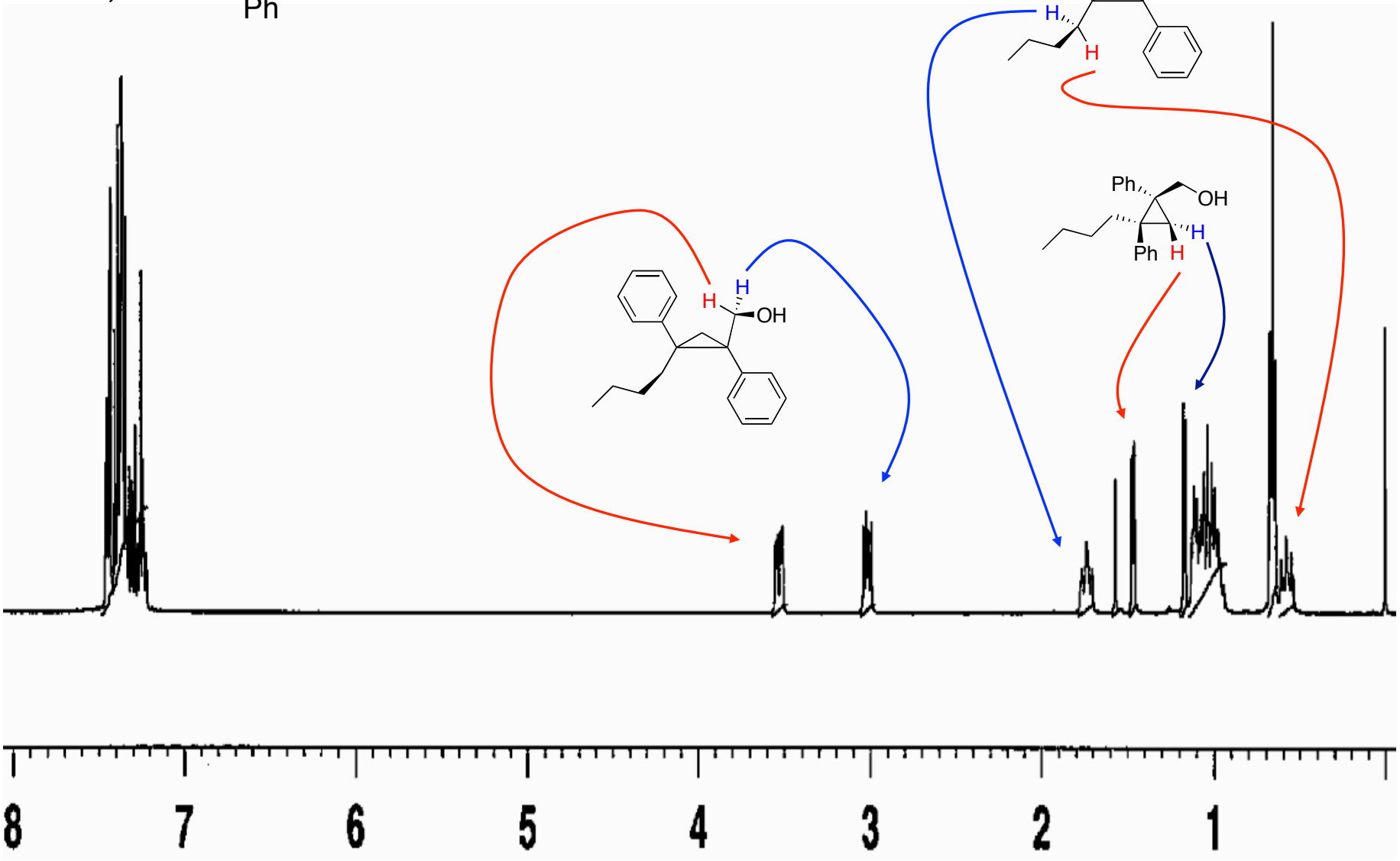
Still diastereotopic

H^a: 3.6 ppm,dd, J = 8.0 Hz, 4.8 Hz
H^b: 3.9 ppm, dd, J = 6.8, 4.8 Hz
H^x: 4.5 ppm, dd, J= 6.8, 8.0 Hz

3 diastereotopic methylenes

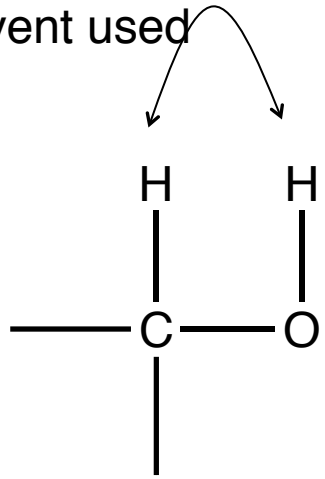


Notice the coupling!

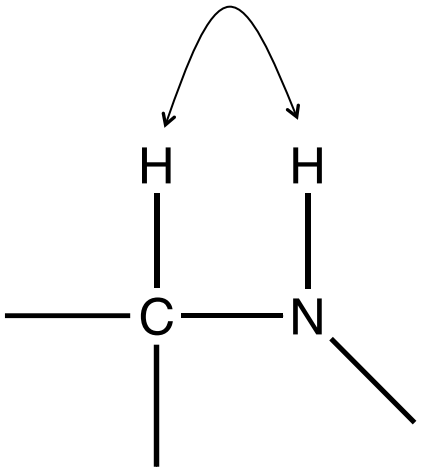


Coupling to Hetero Atoms

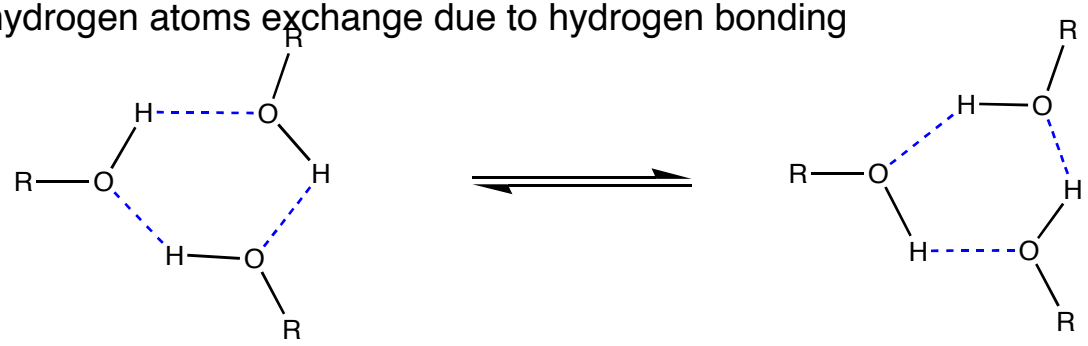
Observed Only Sometimes
Depends on Concentration
Solvent used



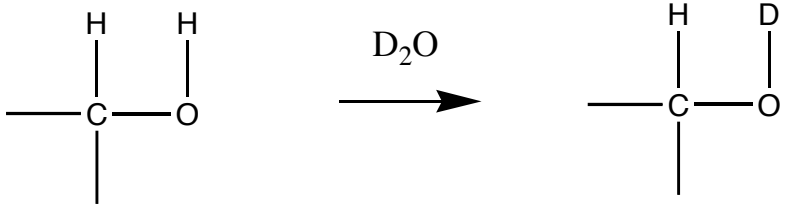
Rarely Observed



WHY: hydrogen atoms exchange due to hydrogen bonding



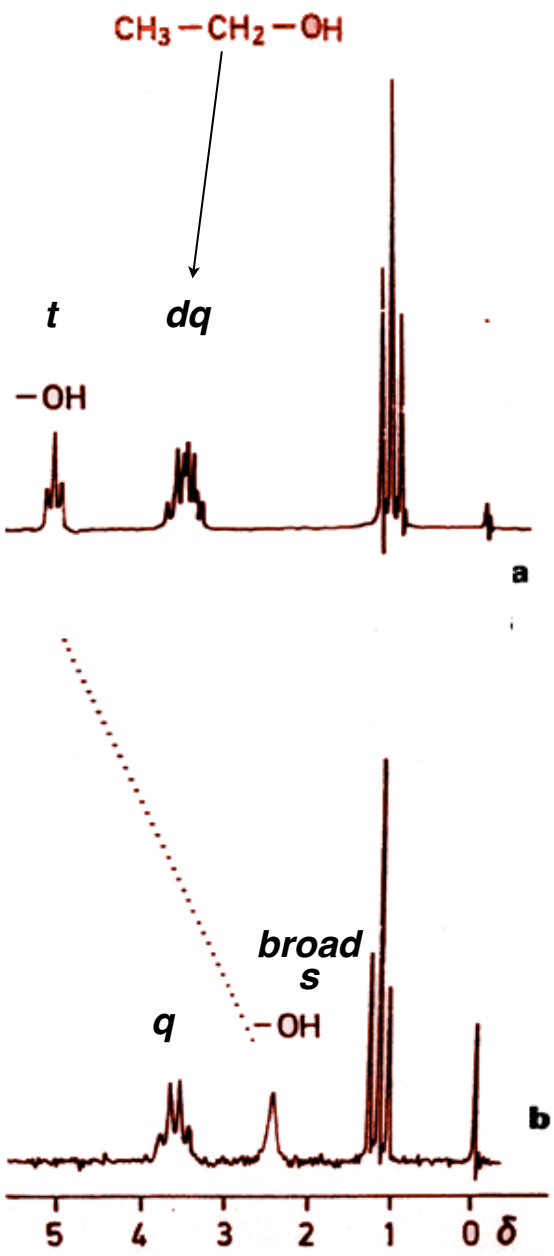
Easy Test



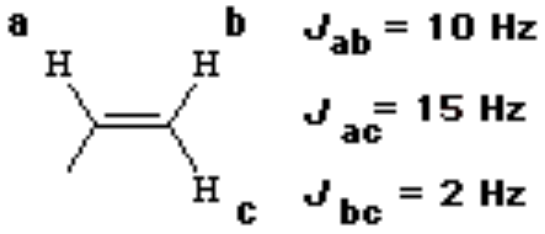
The hydrogen isotope deuterium (^{2}H) has no spin and cannot couple

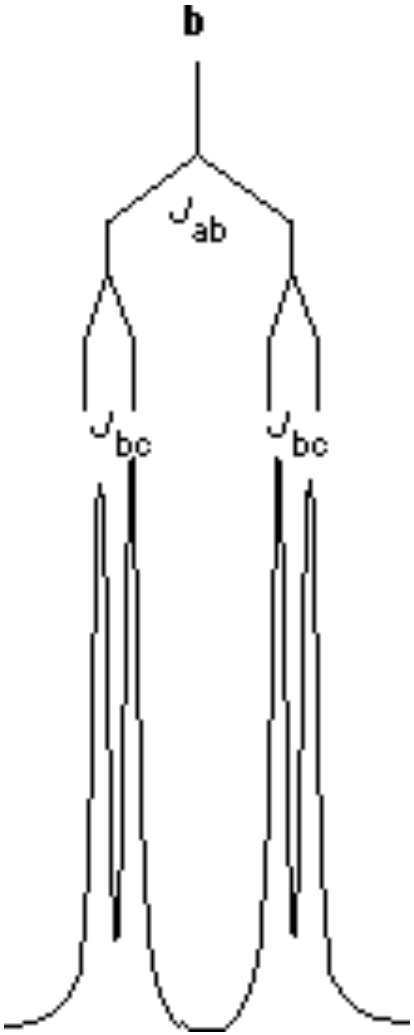
dilute solution of ethanol

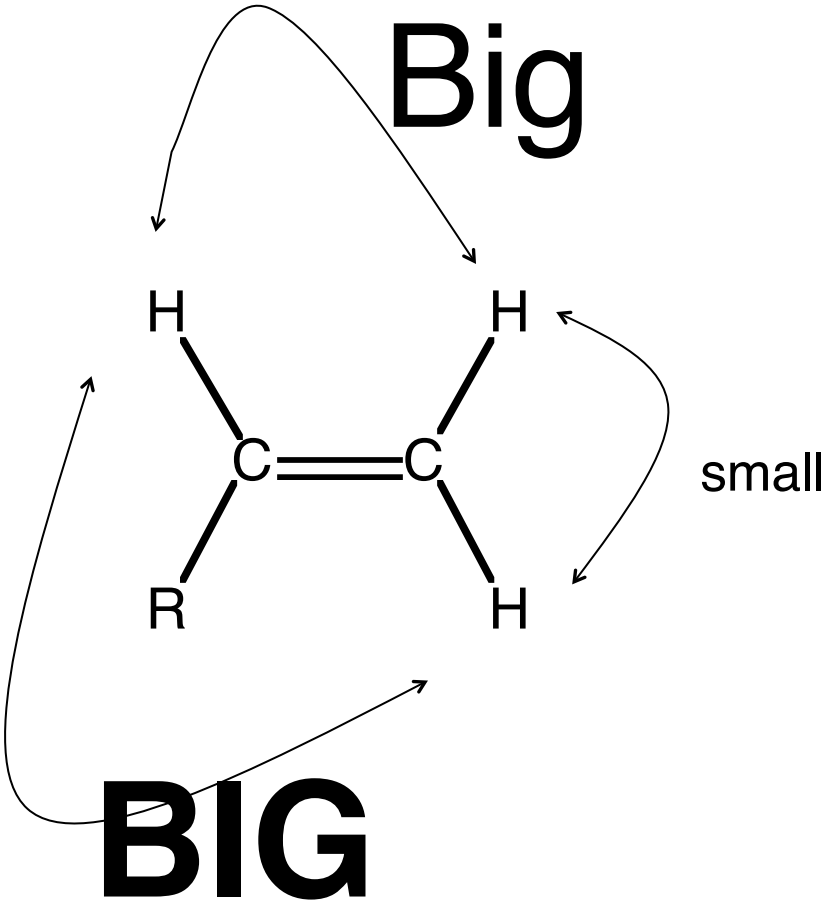
concentrated solution of ethanol

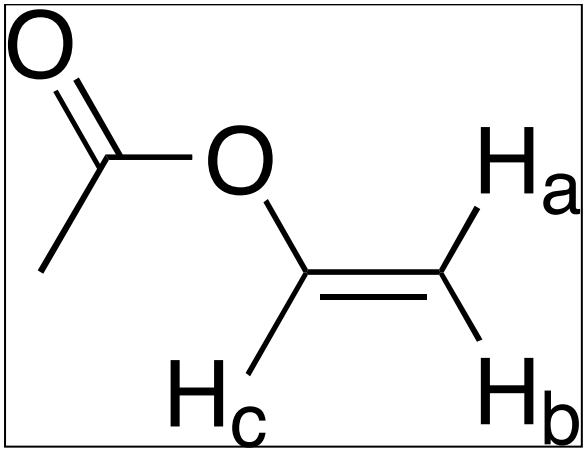


from M. Denk http://131.104.156.23/Lectures/CHEM_207/CHM_207_NMR.htm


a
b
H
H
H
c
J_{ab} = 10 Hz
J_{ac} = 15 Hz
J_{bc} = 2 Hz


b
J_{ab}
J_{bc}
J_{bc}

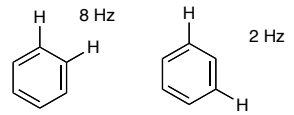

Big
H
H
C
C
R
H
small
BIG

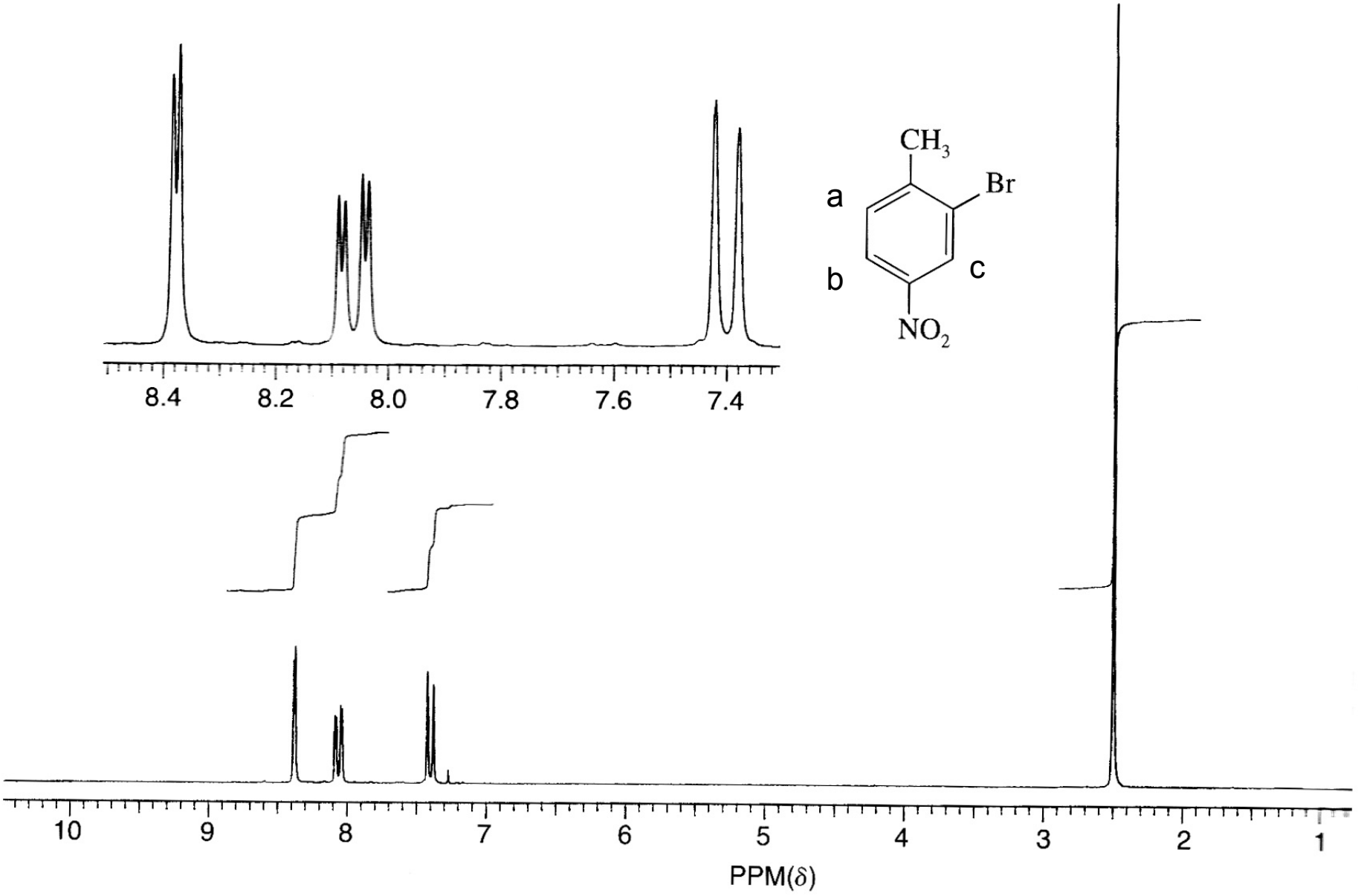


How can coupling constants help you assign the spectrum of vinyl acetate?

Assign the aromatic protons:

Hint:





Assembling Structures Using ^{1}H and ^{13}C NMR.

1. Determine and Assign IHD (Mol formula/C13)
2. Identify symmetry (C13)
3. Identify Functional Groups (C13 or other techniques).
4. Explore around functional groups

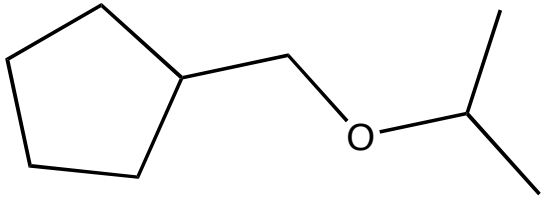
$C_9H_{18}O$

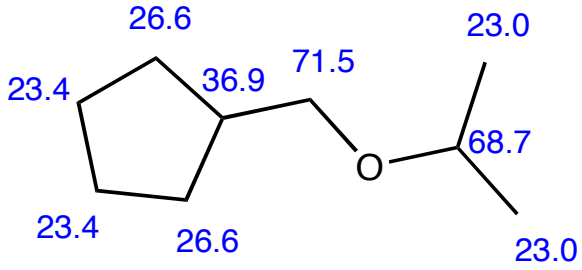
C-13

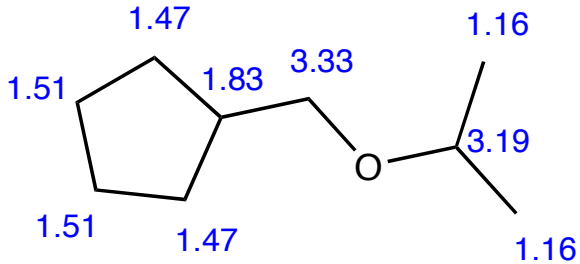
71.5	t
68.7	d
36.9	d
26.6	t (2)
23.4	t (2)
23.0	q (2)

H-1

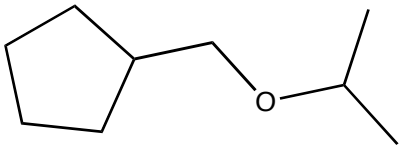
3.33	d	2H
3.19	sept	1H
1.83	m	1H
1.47	m	4H
1.51	m	4H
1.16	d	6H

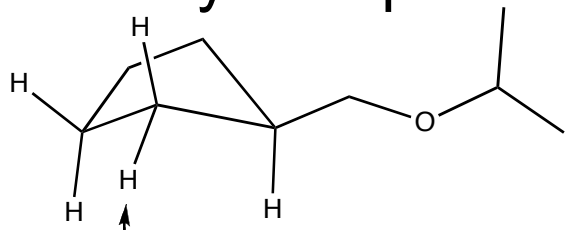






Why Multiplets?





Should be 1.83 t,t,t = multiplet

Should be 1.47 d,d,d,d = multiplet

13.3

$C_9H_{16}O_2$

^{13}C- NMR:

173.6, s

51.3, q

42.0, t

34.9, d

33.1, t (2)

26.2, t (2)

26.1, t

^{1}H- NMR:

3.67, s, 3H

2.19, d, J = 6.4 Hz, 2H

1.70, m, 6H

0.9-1.3, m, 5H

13.4

$C_8H_{11}N$

127.1, s	5.79, t, J = 6.2 Hz, 1H
126.3, d	2.97, s, 2H
117.7, s	2.02, m, 4H
28.0, t	1.70, m, 4H
25.8, t	
25.1, t	
22.5, t	
21.8, t	

$C_{11}H_{18}O_4$

^{1}H NMR

214.9, s

173.5, s

73.6, d

61.7, t

55.1, d

44.1, t

29.9, t

29.2, t

28.1, t

24.0, t

14.1, q

^{13}C-NMR

4.20, q, J = 6.0 Hz, 2H

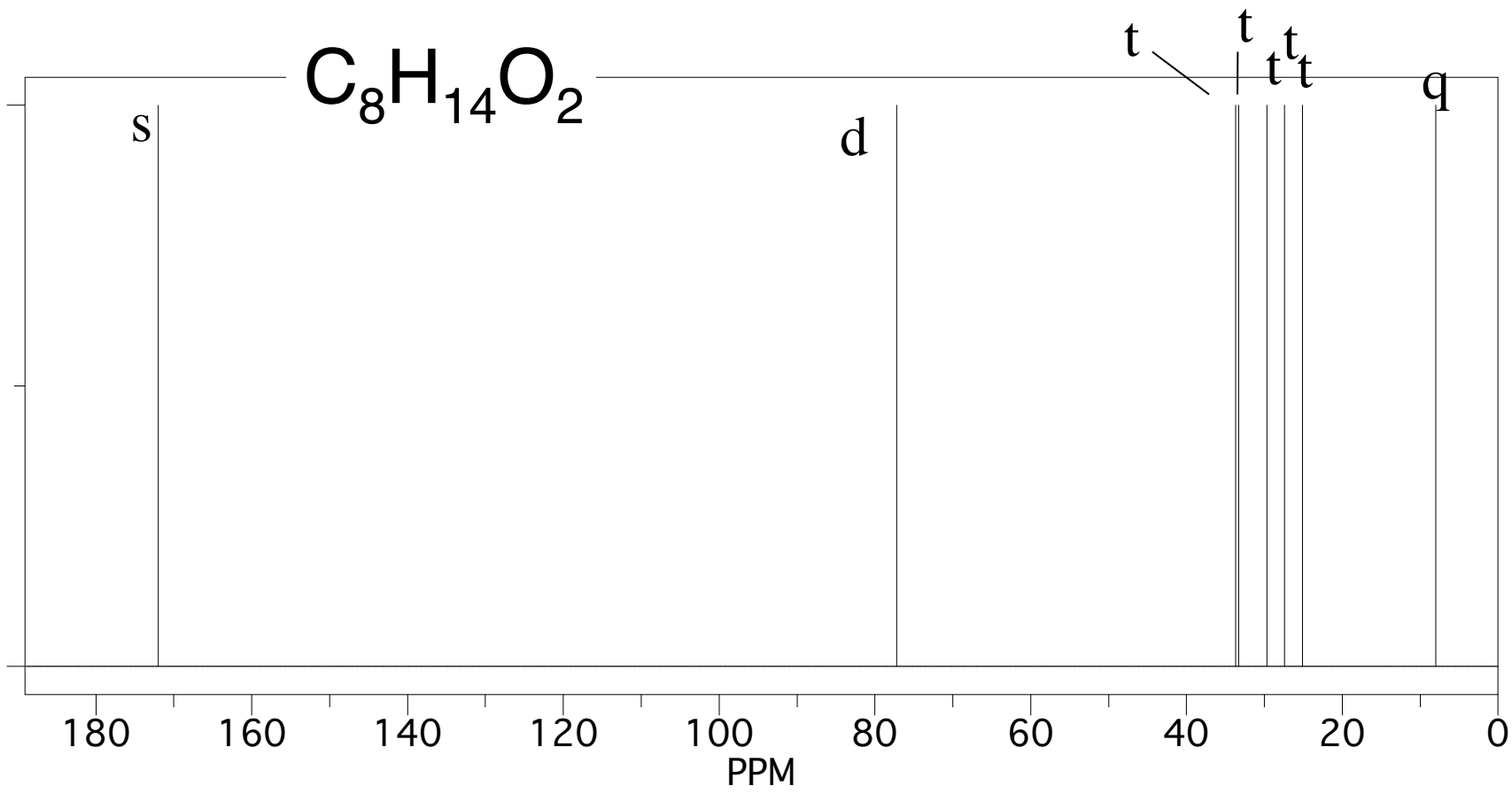
4.00, d, J = 3.5, 1H

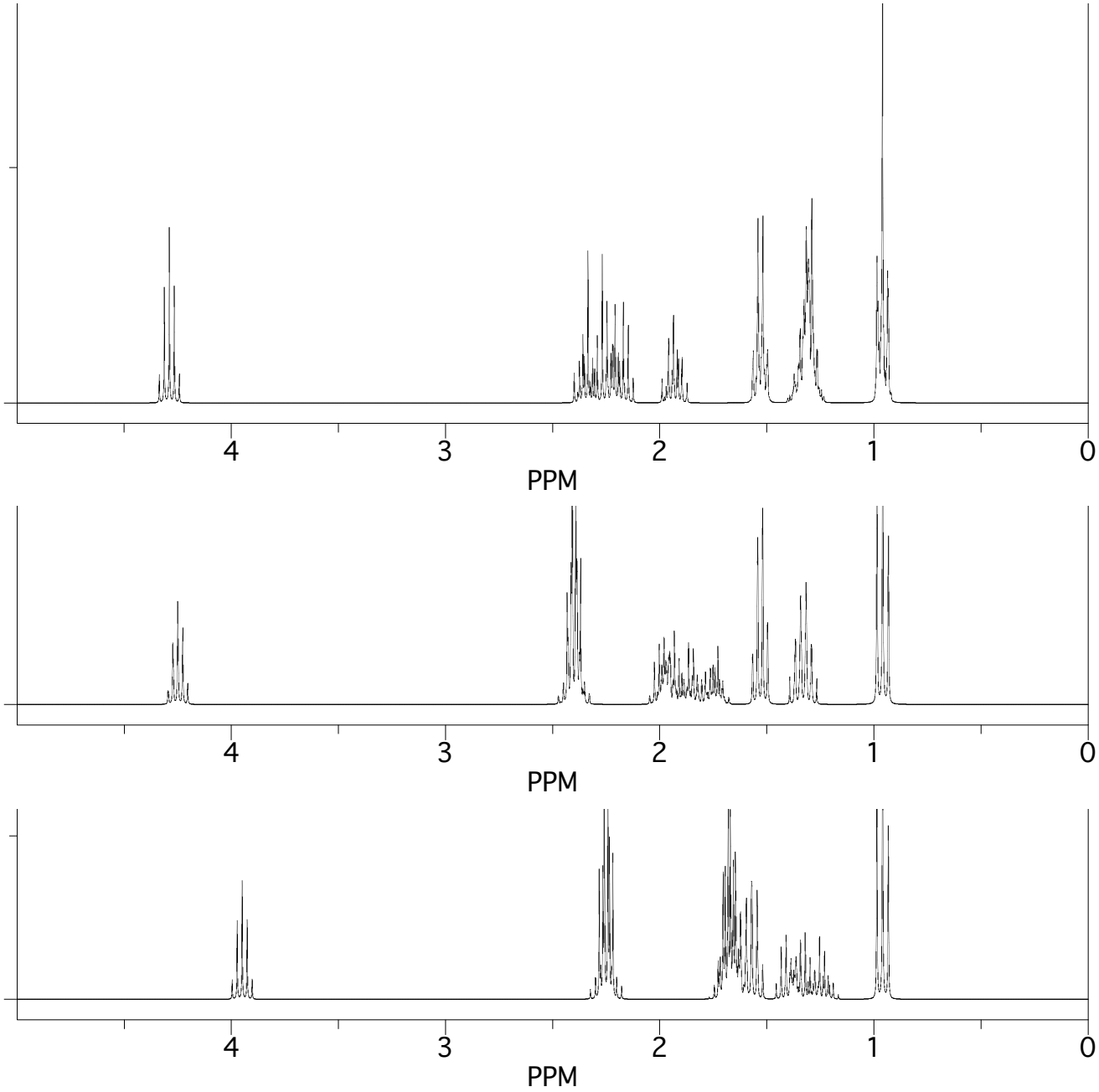
2.9, d, bs, 1H (exchanges)

2.3-2.7, m, 3H

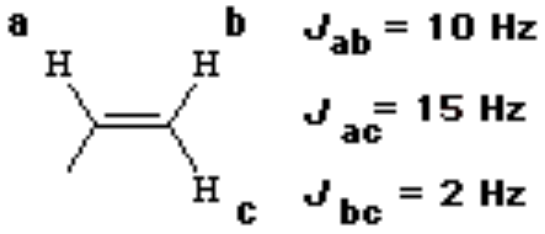
1.2-1.9, m, 8H

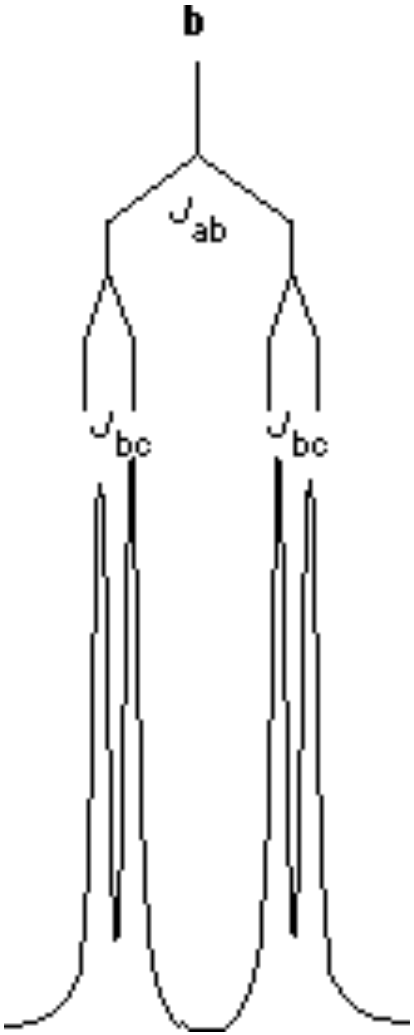
1.30, t, J = 3H

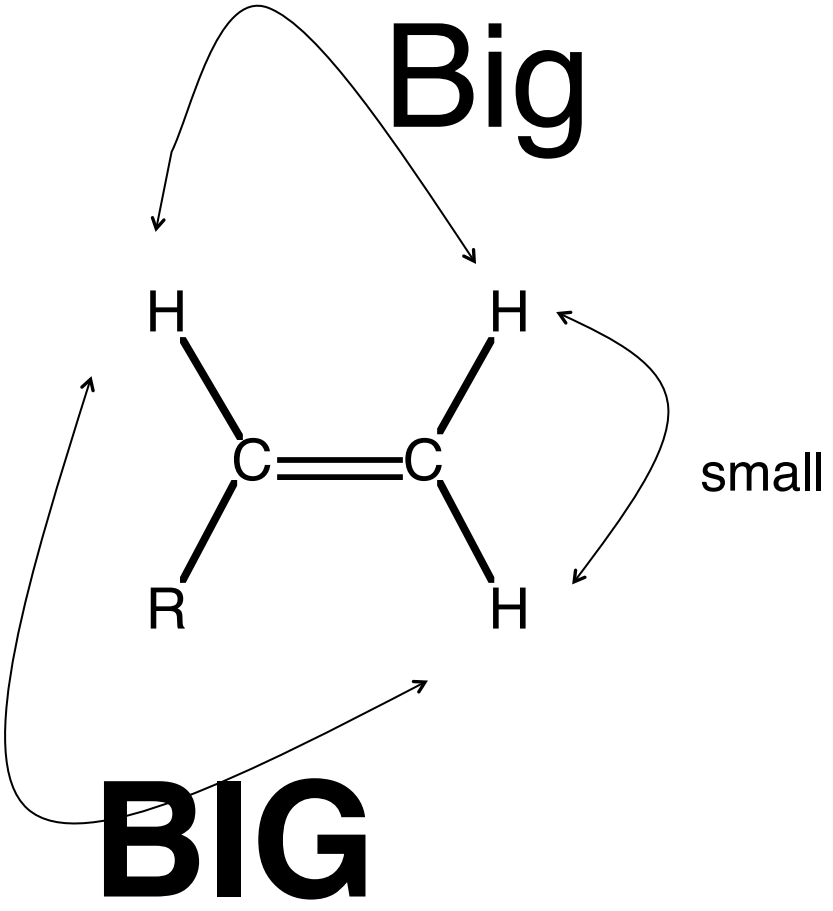

C8H14O2
s
d
t
t
t
t
t
q
180
160
140
120
100
80
60
40
20
0
PPM



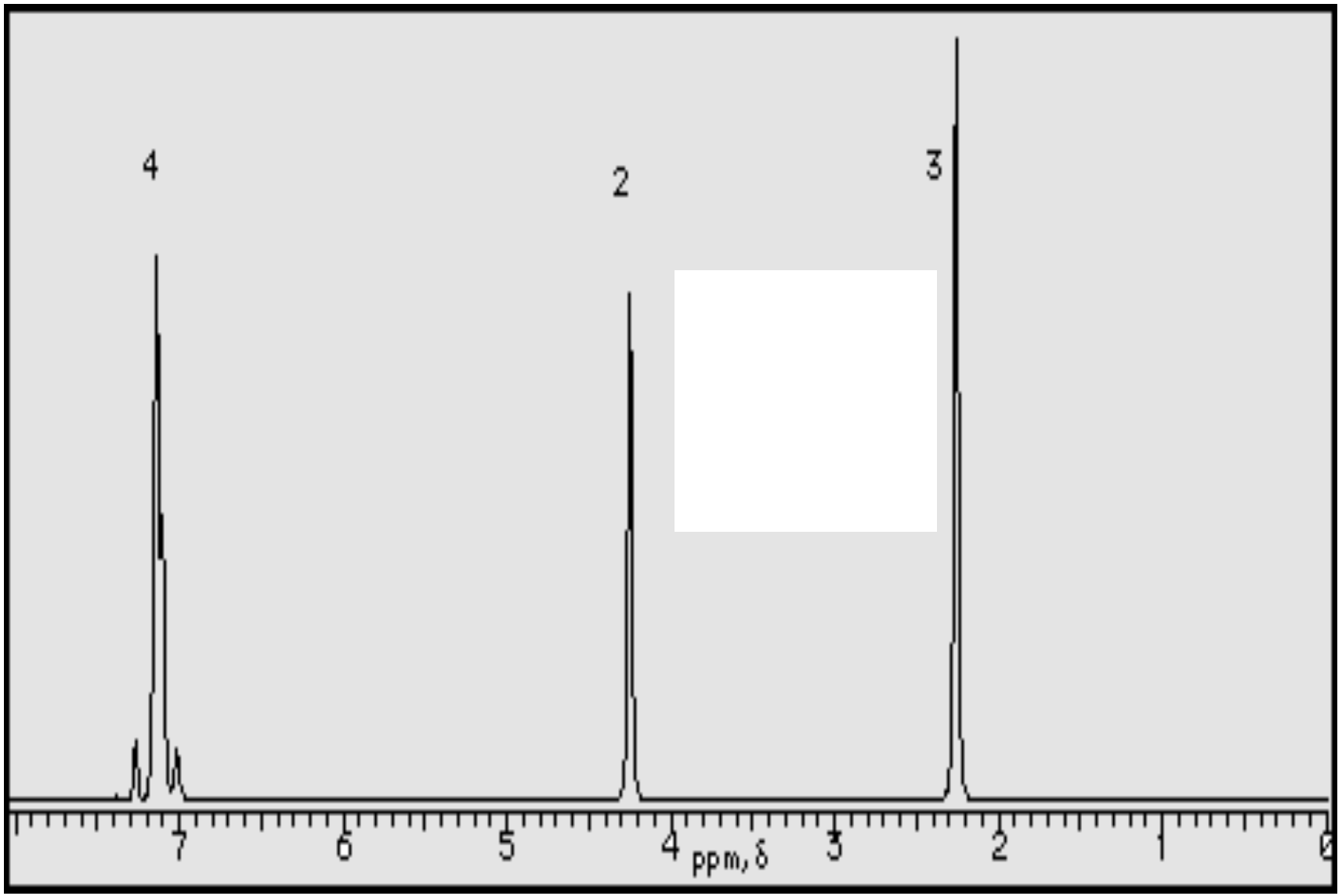
Difficult to determine structure by ^{1}H and ^{13}C NMR only.


a
b
H
H
H
c
J_{ab} = 10 Hz
J_{ac} = 15 Hz
J_{bc} = 2 Hz

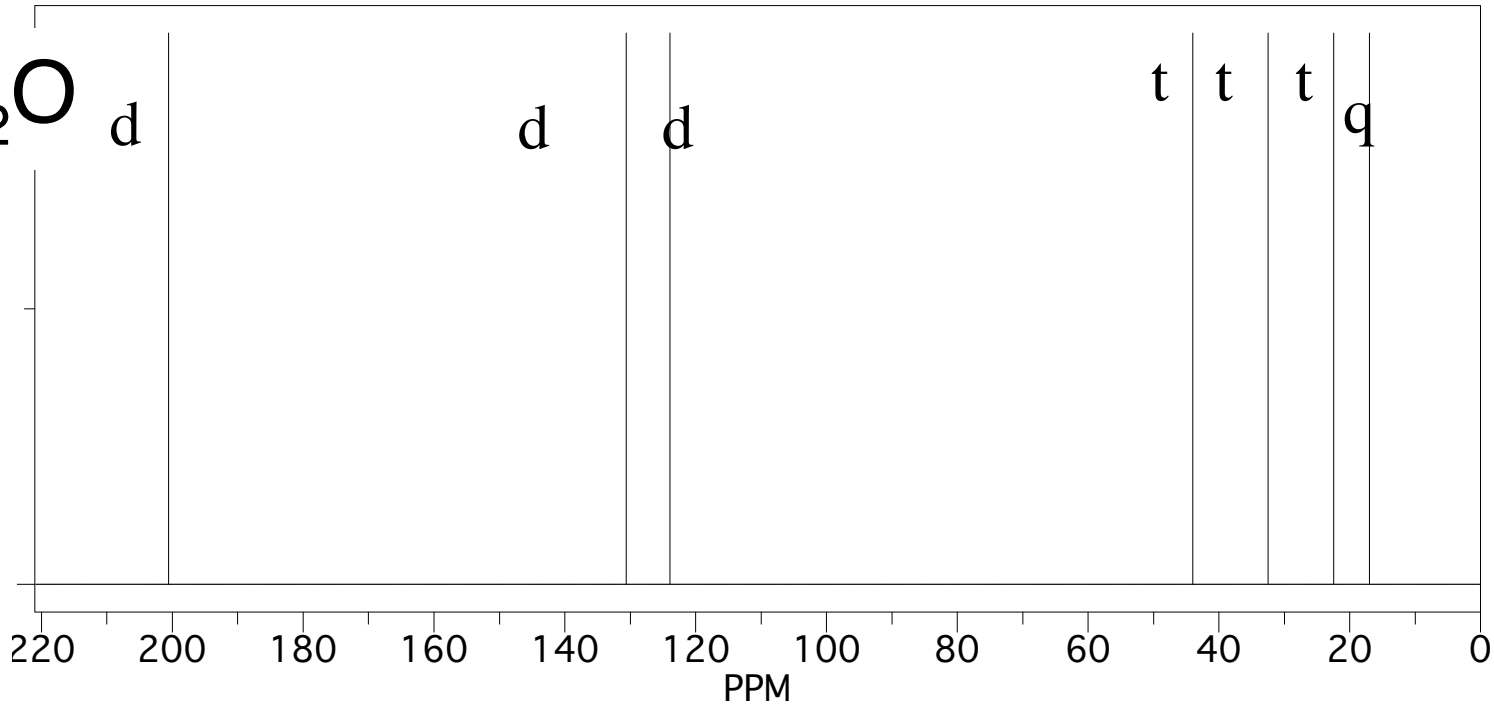

b
J_{ab}
J_{bc}
J_{bc}

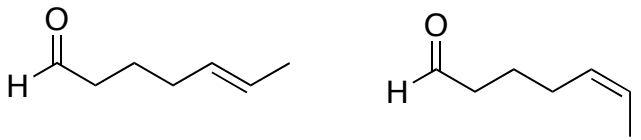

Big
H
H
C
C
R
H
small
BIG

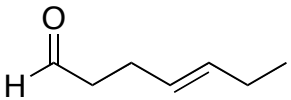
C_8H_9Br

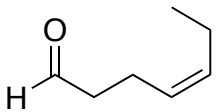


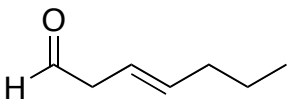
$C_7H_{12}O$

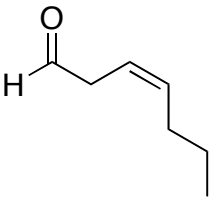








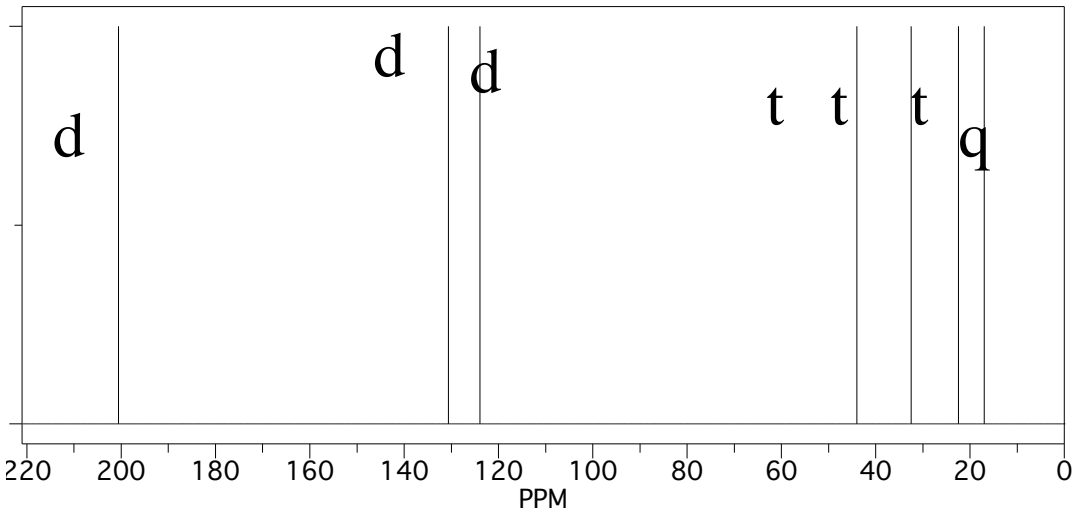


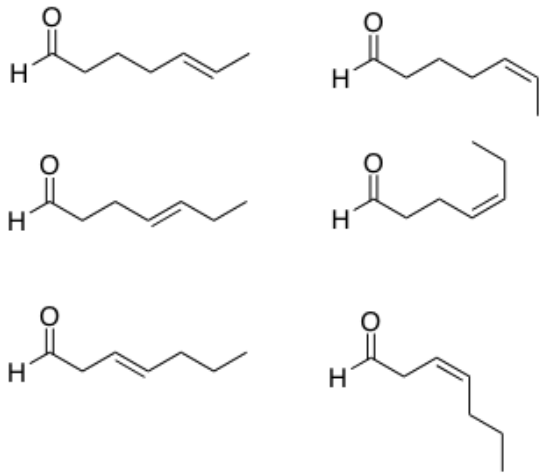


Where is the double bond?

Is the double bond cis or trans?

$C_7H_{12}O$





1H-NMR

9.72 t J = 8.1 Hz, 1H

5.48 dq, J = 15.9, 6.2 Hz, 1H

5.20, dt, J = 15.9, 5.9, 1H

2.40 dt, J = 8.1, 7.4 Hz, 2H

1.96, td, J = 7.9, 5.9Hz, 2H

1.66, quint, J = 8.0 Hz, 2H

1.71, d, J = 6.2 Hz, 3H

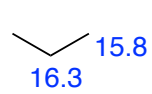
Carbon NMR essentials

Table 5.2 The ^{13}C Shifts for Some Linear and Branched-Chain Alkanes (ppm from TMS)

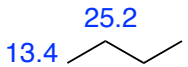
Compound	C-1	C-2	C-3	C-4	C-5
Methane	−2.3				
Ethane	5.7				
Propane	15.8	16.3	15.8		
Butane	13.4	25.2	25.2		
Pentane	13.9	22.8	34.7	22.8	13.9
Hexane	14.1	23.1	32.2	32.2	23.1
Heptane	14.1	23.2	32.6	29.7	32.6
Octane	14.2	23.2	32.6	29.9	29.9
Nonane	14.2	23.3	32.6	30.0	30.3
Decane	14.2	23.2	32.6	31.1	30.5
Isobutane	24.5	25.4			
Isopentane	22.2	31.1	32.0	11.7	
Isohexane	22.7	28.0	42.0	20.9	14.3
Neopentane	31.7	28.1			
2,2-Dimethylbutane	29.1	30.6	36.9	8.9	
3-Methylpentane	11.5	29.5	36.9	(18.8, 3-CH_3)	
2,3-Dimethylbutane	19.5	34.3			
2,2,3-Trimethylbutane	27.4	33.1	38.3	16.1	
2,3-Dimethylpentane	7.0	25.3	36.3	(14.6, 3-CH_3)	

CH_4 -2.3 methane

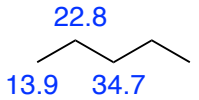
ethane H_3C-CH_3 5.7



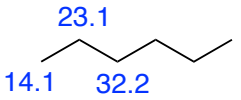
propane



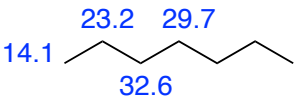
butane



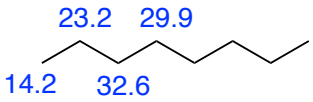
pentane



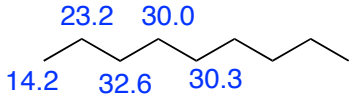
hexane



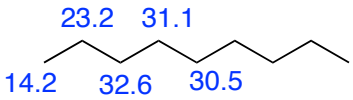
heptane



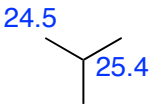
octane



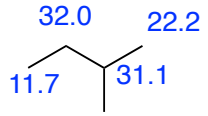
nonane



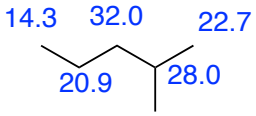
decane



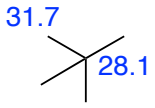
isobutane



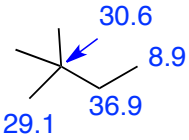
isopentane



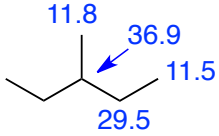
isohexane



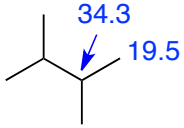
neopentane



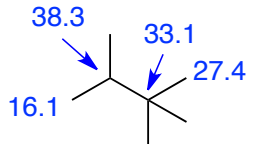
2,2-dimethylbutane



3-methylpentane



2,3-dimethylbutane



2,2,3-trimethylbutane