Practice Exam 1

Chemistry 605 (Reich)

FIRST HOUR EXAM

Thur, March 9, 2012

Question/Points

R-11A_____/7

R-11B_____/10

R-11C_____/5

R-11D_____/25

R-11E_____/20

R-11F_____/20

R-11G_____/13

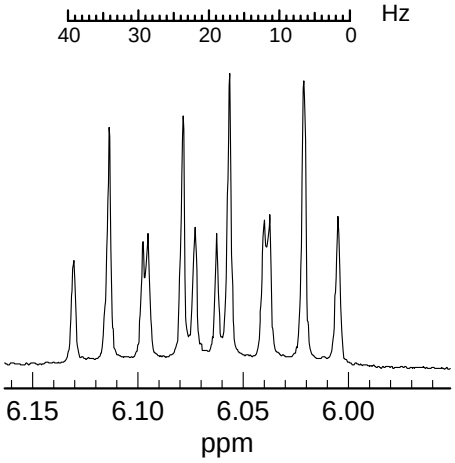
Total _____/100

Name_____

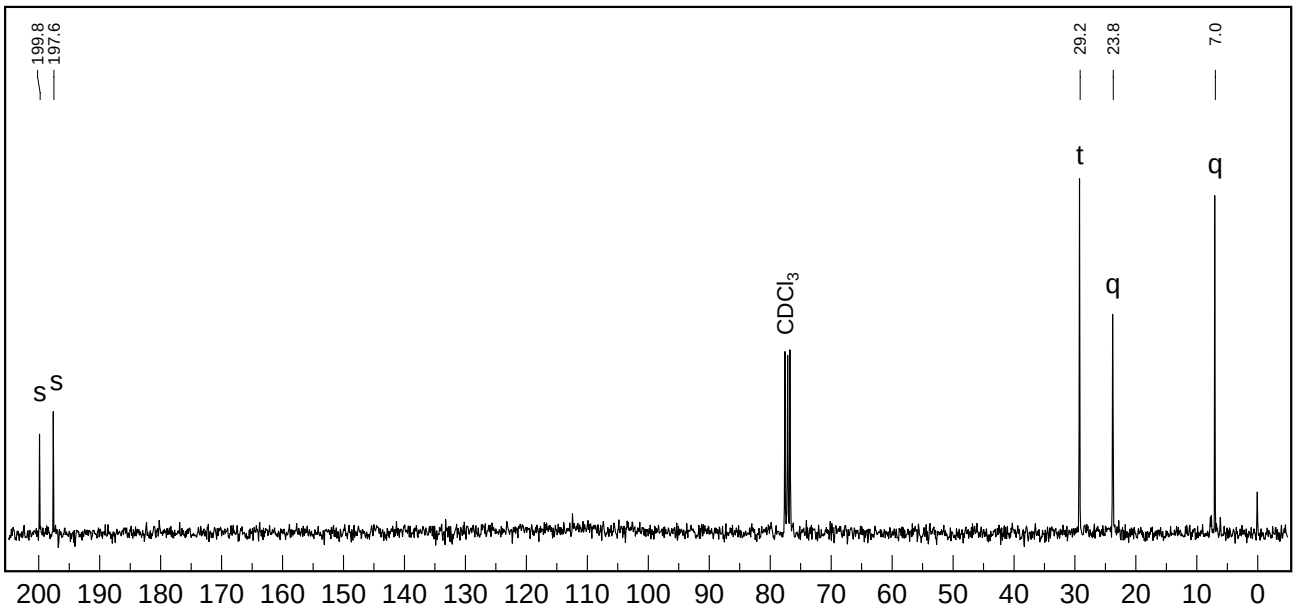
If you place answers anywhere else except in the spaces provided, (e.g. on the spectra or on extra pages) clearly indicate this on the answer sheets.

Problem R-11A. Analyze the muiltiplet below, and report in the standard format

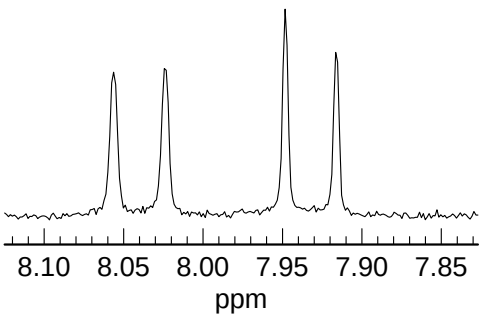
Suggest a part structure that fits the multiplet, indicate which proton is being observed, and label the structure with J values



Problem R-11B Determine the structure of the compound $C_5H_8O_2$ whose 75 MHz ^{13}C NMR spectrum is shown below. Assign the carbons.



Problem R-11C. Are the two one-proton multiplets below coupled to each other? What criteria are you using?



Problem R-11D (C_5H_8O). Determine the structure of **R-10E** from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

(a) DBE_____ (b) What information can you obtain from the IR spectrum (give frequency and peak assignment).

(c) Analyze the ^{1}H NMR spectrum. For each of the groups of signals marked on the spectrum, report the multiplet structure in the standard format (e.g., 0.0 δ, dtd, J = 0.0, 0.0, 0.0 Hz, 2H) and any part structure you could obtain from the signal(s).

A _____

B _____

C _____

D _____

E _____

(d) Give your answer below. If more than one structure fits the data, draw them, but indicate your best choice by circling the structure

(e) The ^{13}C NMR chemical shifts are listed below. Write the δ values on your structure.

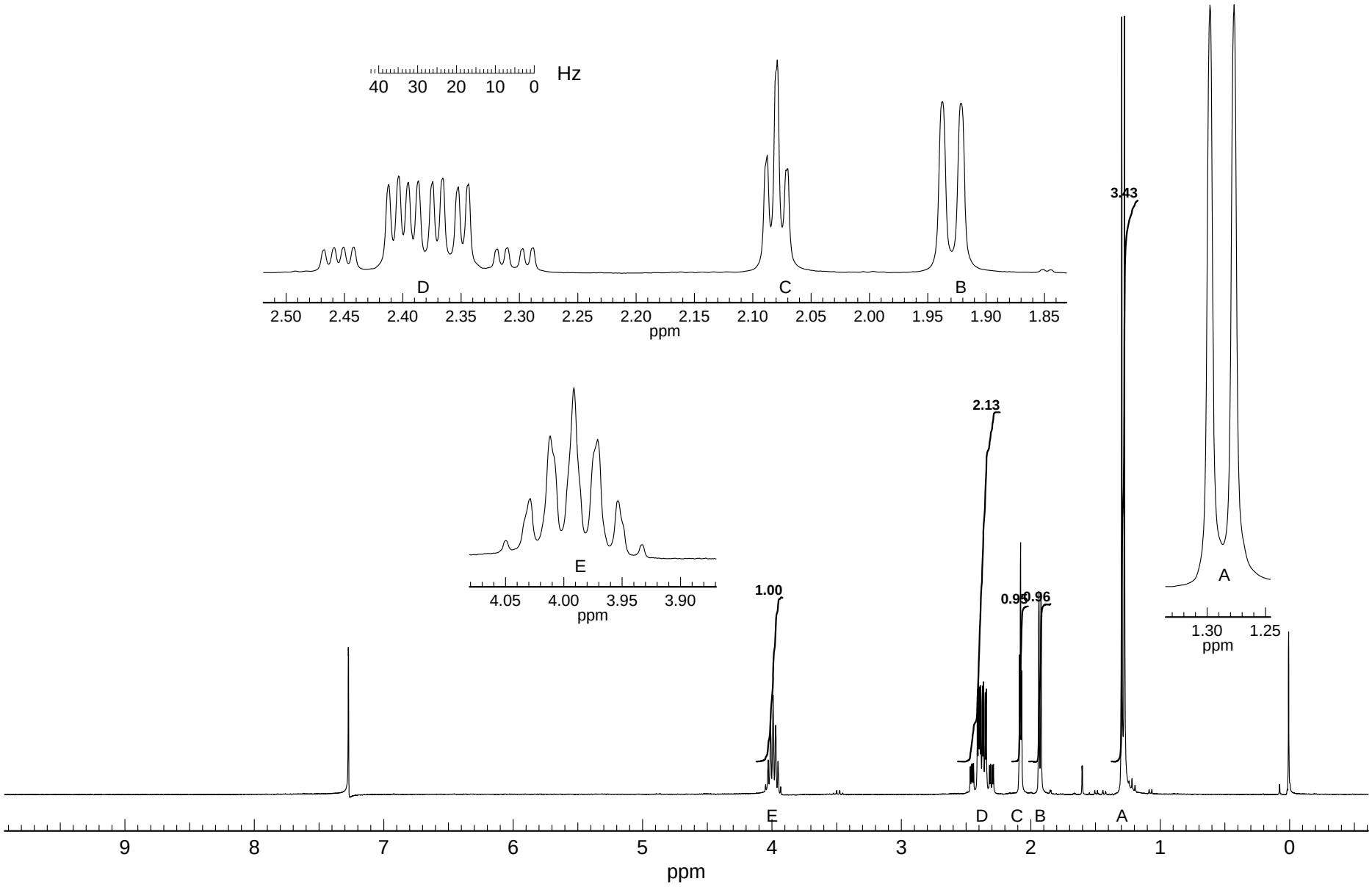
δ
22.5
29.2
66.4
71.0
81.0

(f) To confirm your assignment (and structure) calculate the chemical shifts of the carbons in your structure assigned to the 29.2 and 66.4 signals. Use a suitable model compound, and appropriate chemical shift $\Delta\delta$ values.

Problem R-11D (C_5H_8O)

300 MHz ^1H NMR Spectrum in $CDCl_3$

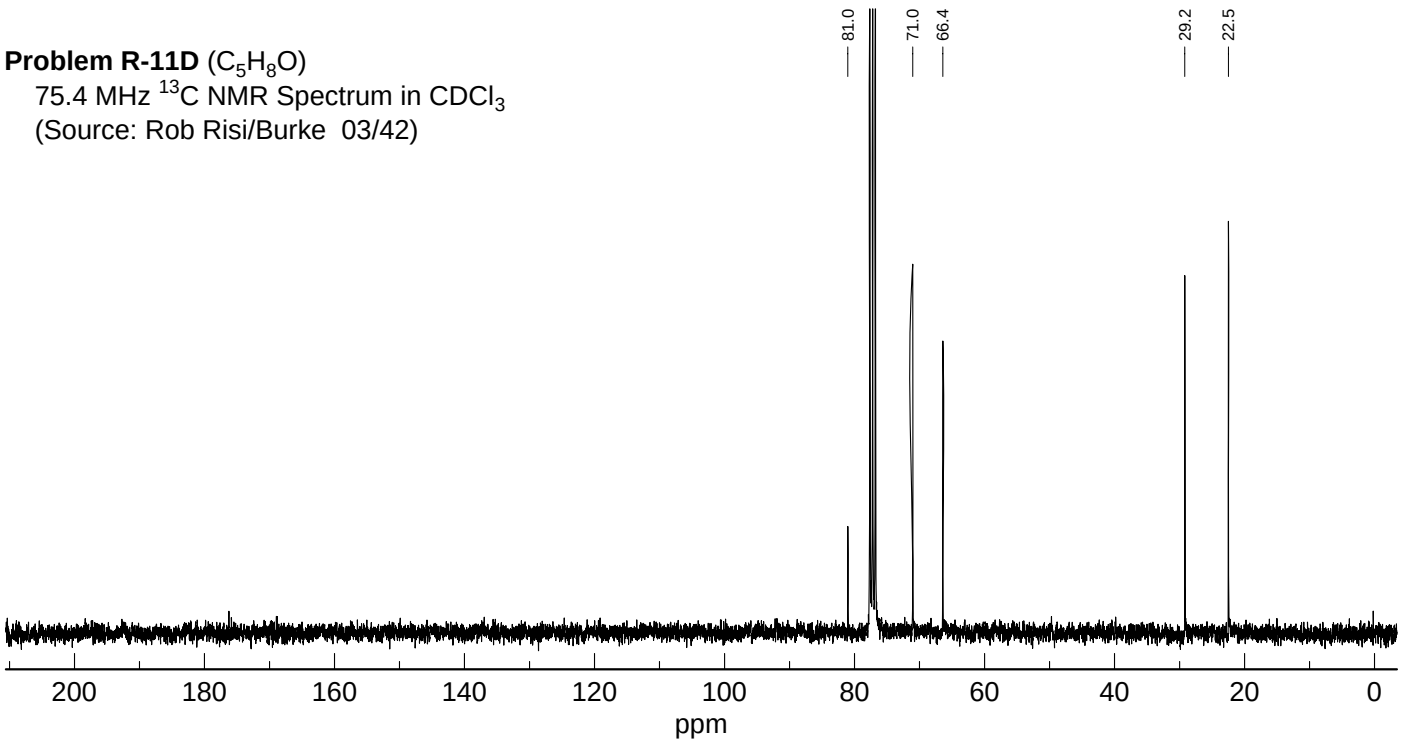
(Source: Rob Risi/Burke 03/42)



Problem R-11D (C_5H_8O)

75.4 MHz ^{13}C NMR Spectrum in $CDCl_3$

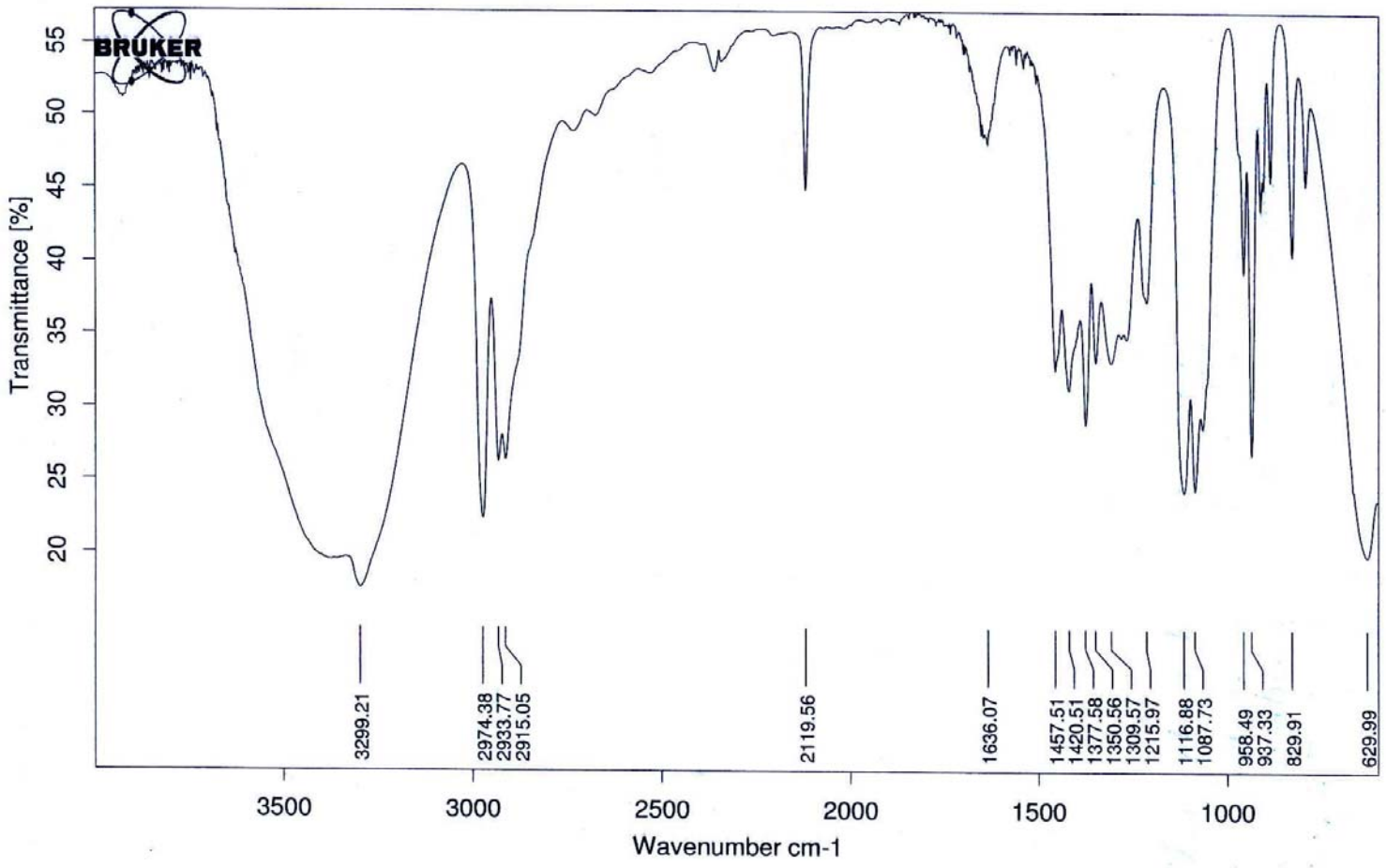
(Source: Rob Risi/Burke 03/42)



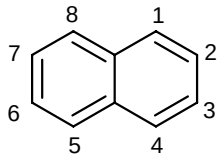
Problem R-11D (C_5H_8O)

IR Spectrum (neat liquid)

(Source: Rob Risi/Burke 03/42)



Problem R-11E ($C_{11}H_9IO$). You are provided the ^1H NMR spectrum of a disubstituted naphthalene (**the substituents are methoxy and iodo**). You are required to analyze the NMR spectrum, and determine the structure or structures.



(a) For each of the 8 positions on the naphthalene as defined above, give either the substituent at that position, or the NMR signal (δ, multiplicity and J values). If there is more than one plausible structure assignment, draw the alternative structure, and indicate your preference. To make grading easier, **please place the methoxy substituent at either 1 or 2**.

1_____

2 _____

3 _____

4 _____

5 _____

6 _____

7 _____

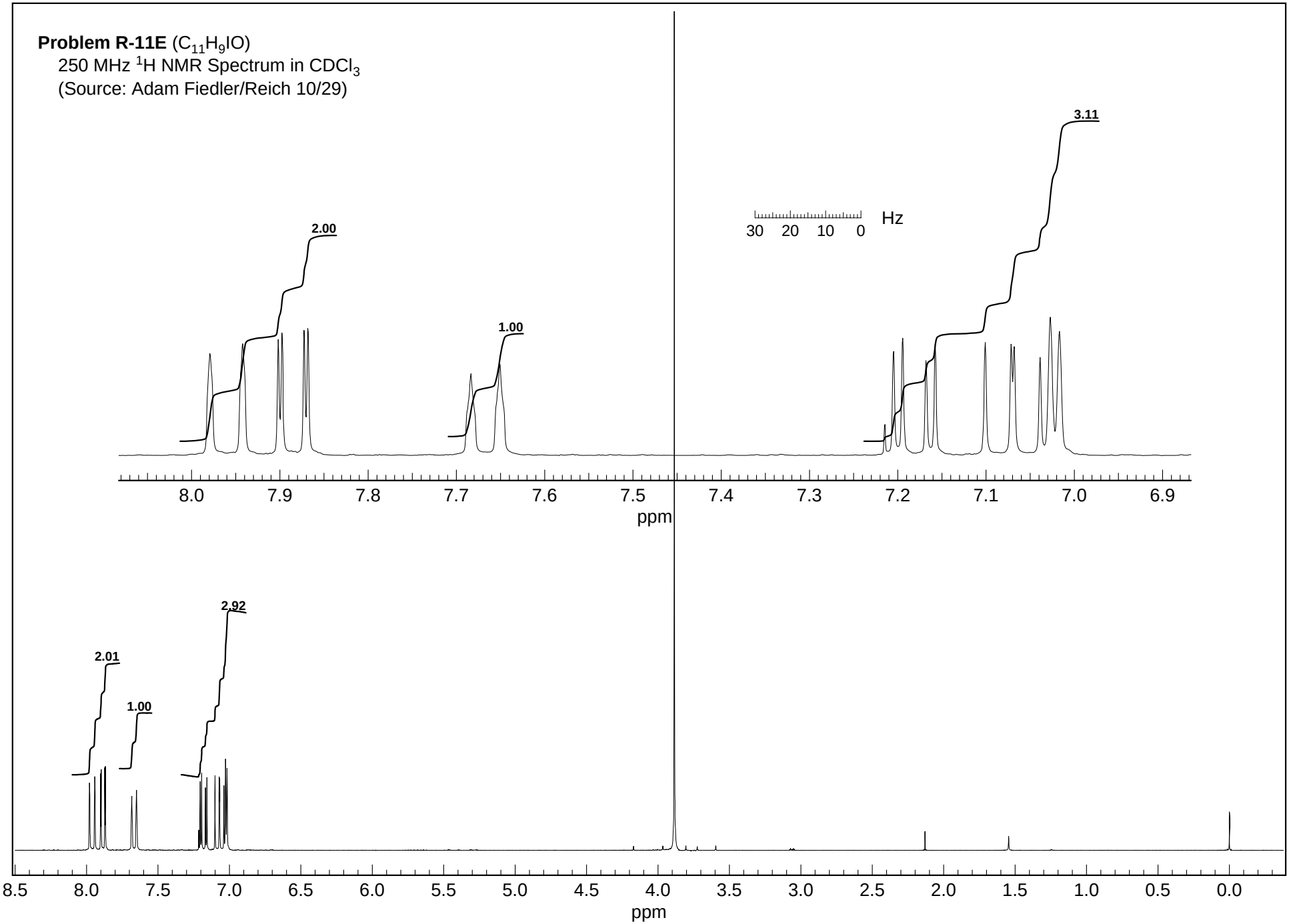
8 _____

(b) Briefly describe the key evidence that led to your structure assignment.

Problem R-11E ($C_{11}H_9IO$)

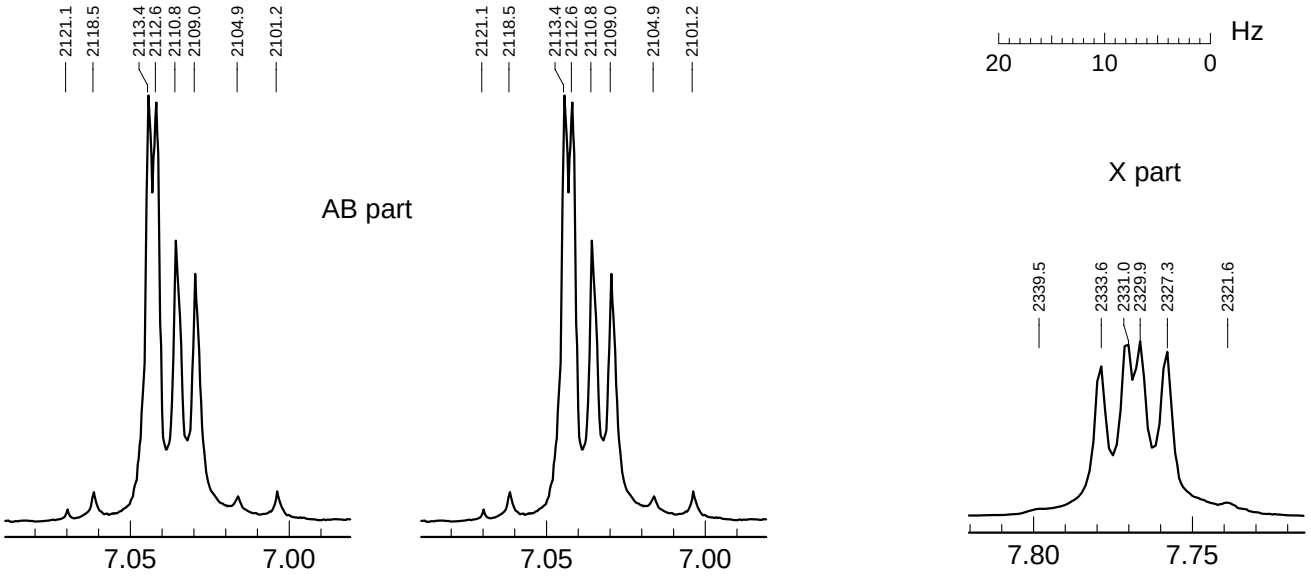
250 MHz ^1H NMR Spectrum in $CDCl_3$

(Source: Adam Fiedler/Reich 10/29)



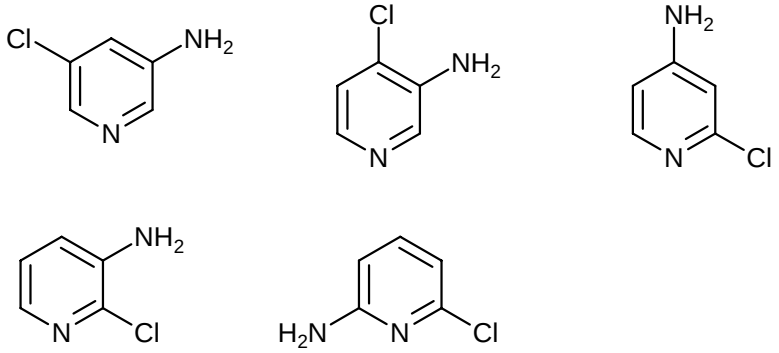
Problem R-11F ($C_5H_5ClN_2$). The 300 MHz NMR spectrum of of a disubstituted pyridine is shown below (the complete spectrum on the next page. This means there are three aromatic protons, which form an ABX pattern.

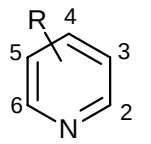
(a) Do an accurate calculation and determine couplings and chemical shifts, and **tabulate your results in an easily readable format**. If there are two solutions, report them both, and draw coupling trees on the spectra. For your convenience two copies of the AB part of the spectrum are shown.



(b) If you are proposing two solutions, suggest at least one criterion which allows you to identify the correct one.

(c) Which of the following structures best fits the NMR *J* and δ values?. Label your preferred structure with H_A, H_B and H_X. For your convenience, the typical coupling constants in pyridines are reproduced below.

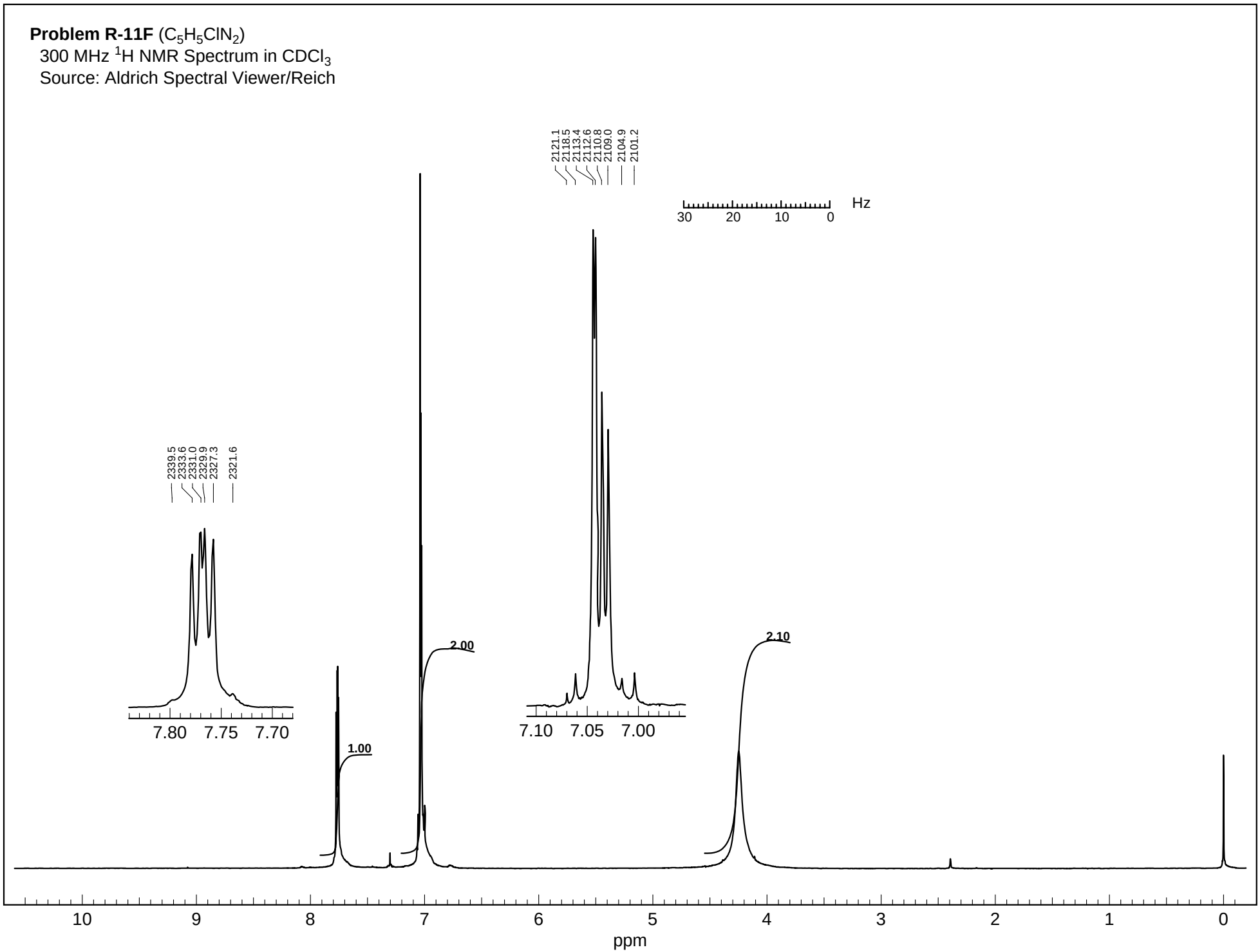




$^3J_{34}$	6.9 to 9.1
$^3J_{23}$	4.0 to 6.0
$^4J_{24}$	0 to 2.7
$^4J_{35}$	0.5 to 1.8
$^4J_{26}$	0 to 0.6
$^5J_{25}$	0 to 2.3

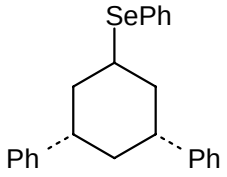
All coupling constants are positive

Problem R-11F ($C_5H_5ClN_2$)
300 MHz ^{1}H NMR Spectrum in $CDCl_3$
Source: Aldrich Spectral Viewer/Reich

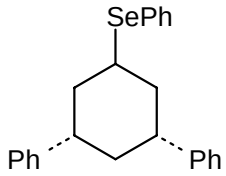


Problem R-11G ($C_{24}H_{24}Se$) This problem requires you to determine which isomer is which from the ^{13}C NMR spectra of cis and trans 3,5-diphenyl-1-phenylselenocyclohexane.

(a) Assign the signals 0-50 ppm for both isomers (place the δ values on the structures below). Briefly explain how you made the assignments.



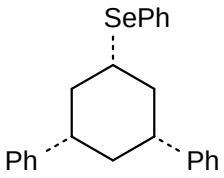
Spectrum A



Spectrum B

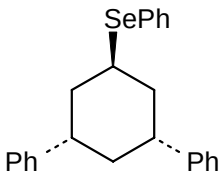
(b) Identify a key feature in the spectra which allows identification of which spectrum corresponds to the cis isomer and which to the trans isomer. Explain your reasoning.

cis isomer



Spectrum ____

trans isomer



Spectrum ____

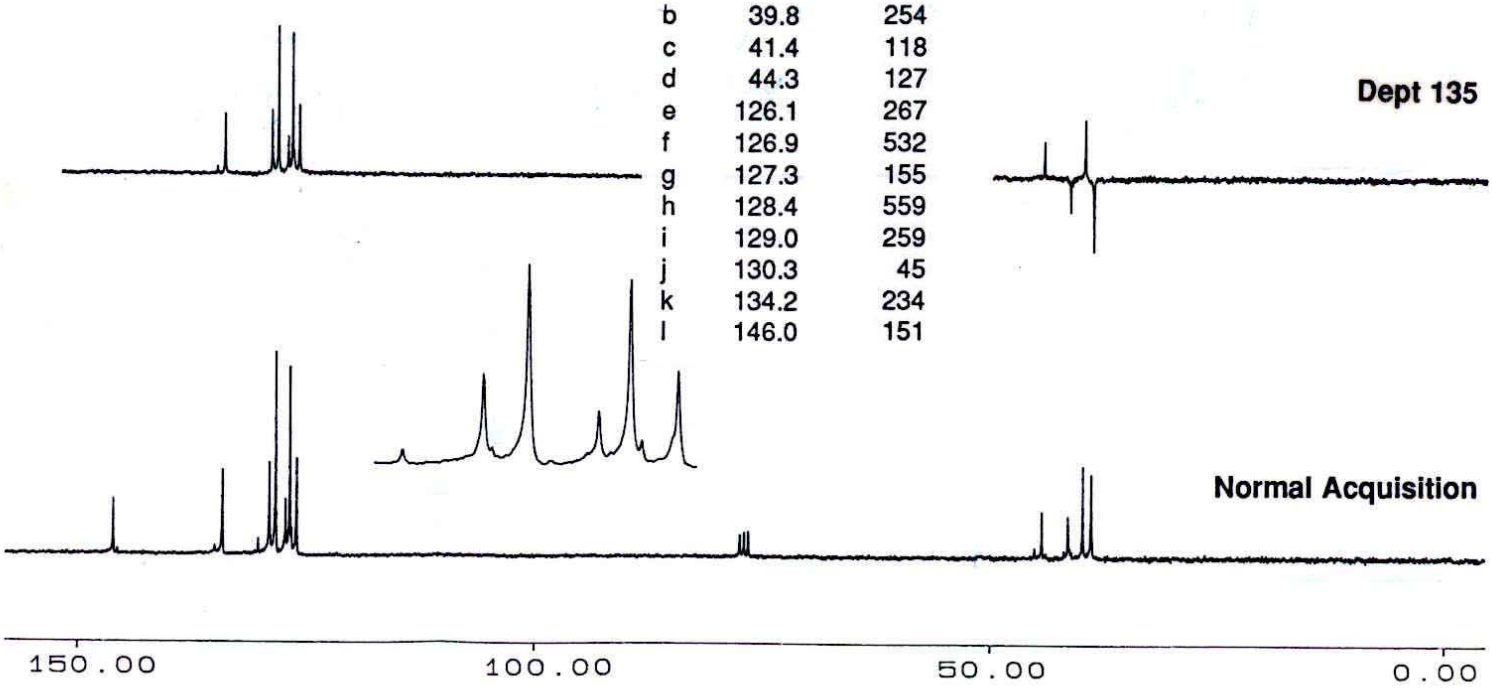
Problem R-11G ($C_{24}H_{24}Se$) **Isomer A**

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

The upper spectrum is a DEPT 135 spectrum (CH_2 negative, CH and CH_3 positive)

(Source: Mike Bowe/Reich 12/20)

	δ	Intensity
a	38.8	232
b	39.8	254
c	41.4	118
d	44.3	127
e	126.1	267
f	126.9	532
g	127.3	155
h	128.4	559
i	129.0	259
j	130.3	45
k	134.2	234
l	146.0	151



Problem R-11G ($C_{24}H_{24}Se$) **Isomer B**

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

The upper spectrum is a DEPT 135 spectrum (CH_2 negative, CH and CH_3 positive)

(Source: Mike Bowe/Reich 12/20)

	δ	Intensity
a	41.0	104
b	41.2	181
c	41.8	94
d	45.1	188
e	126.2	209
f	126.7	406
g	127.5	108
h	128.4	419
i	128.5	82
j	128.9	206
k	135.0	183
l	145.6	115