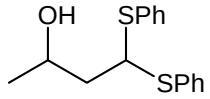
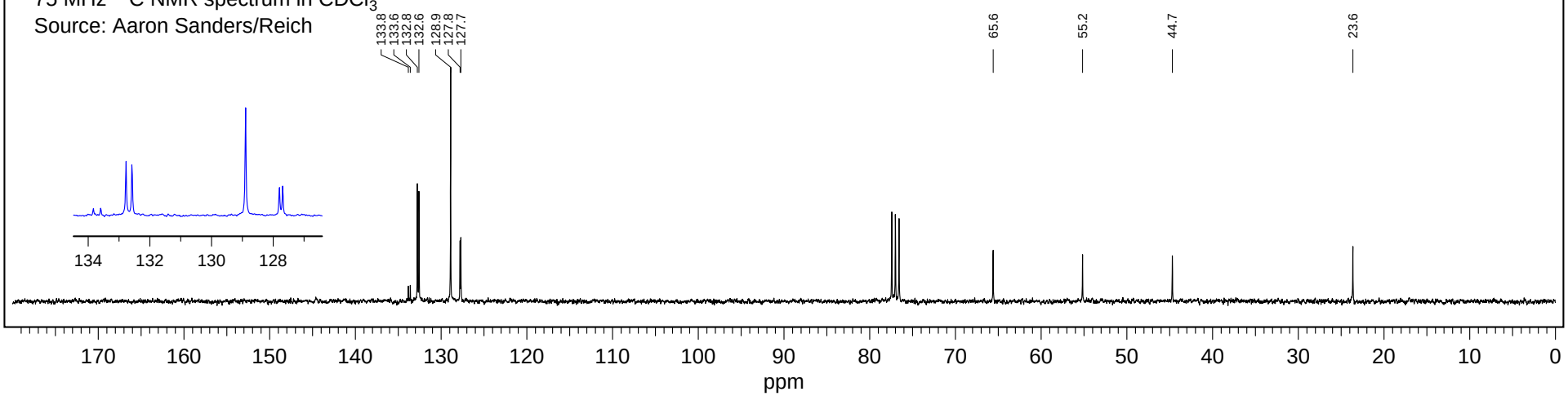
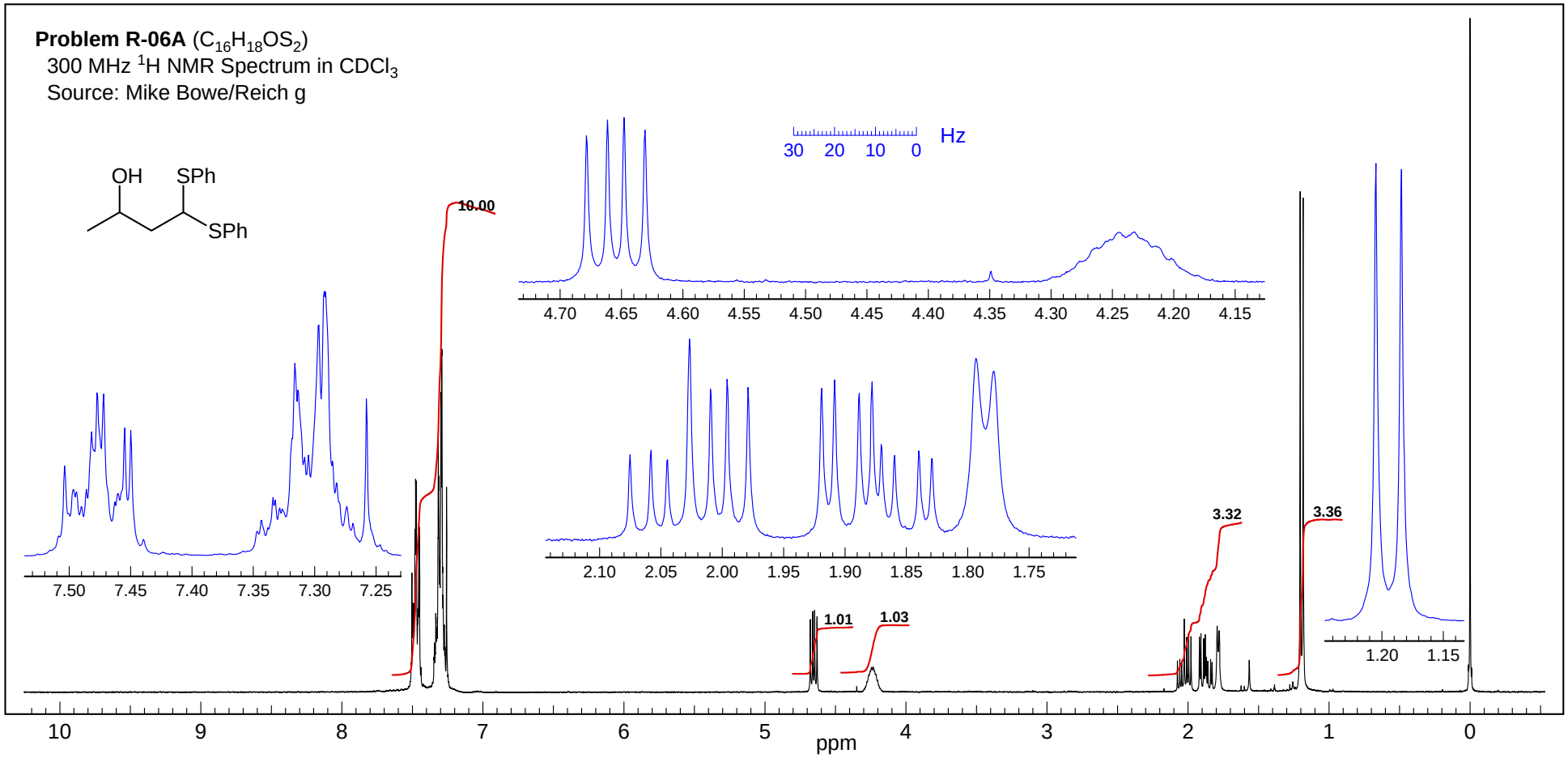
Problem R-06A ($C_{16}H_{18}OS_2$)
300 MHz ^{1}H NMR Spectrum in $CDCl_3$
Source: Aaron Sanders/Reich g



Problem R-06A ($C_{16}H_{18}OS_2$).
75 MHz ^{13}C NMR spectrum in $CDCl_3$
Source: Aaron Sanders/Reich

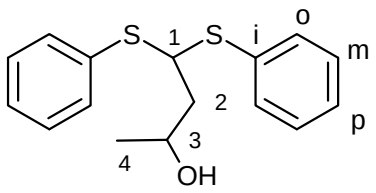


PLT ex-1-2006-07-gq.plt



PLT ex-1-2006-07-gq.plt

Problem R-06A ($C_{16}H_{18}OS_2$). Analyze the spectra shown on the next page. You are given the structure. Please use the numbering system given.



(a) Identify the OH proton.

(b) For the proton or set of protons on each carbon (C-1 to C-4), report the multiplet in the standard format (e.g. δ 3.44, tq, J = 7,2 Hz) .

C-1

C-2

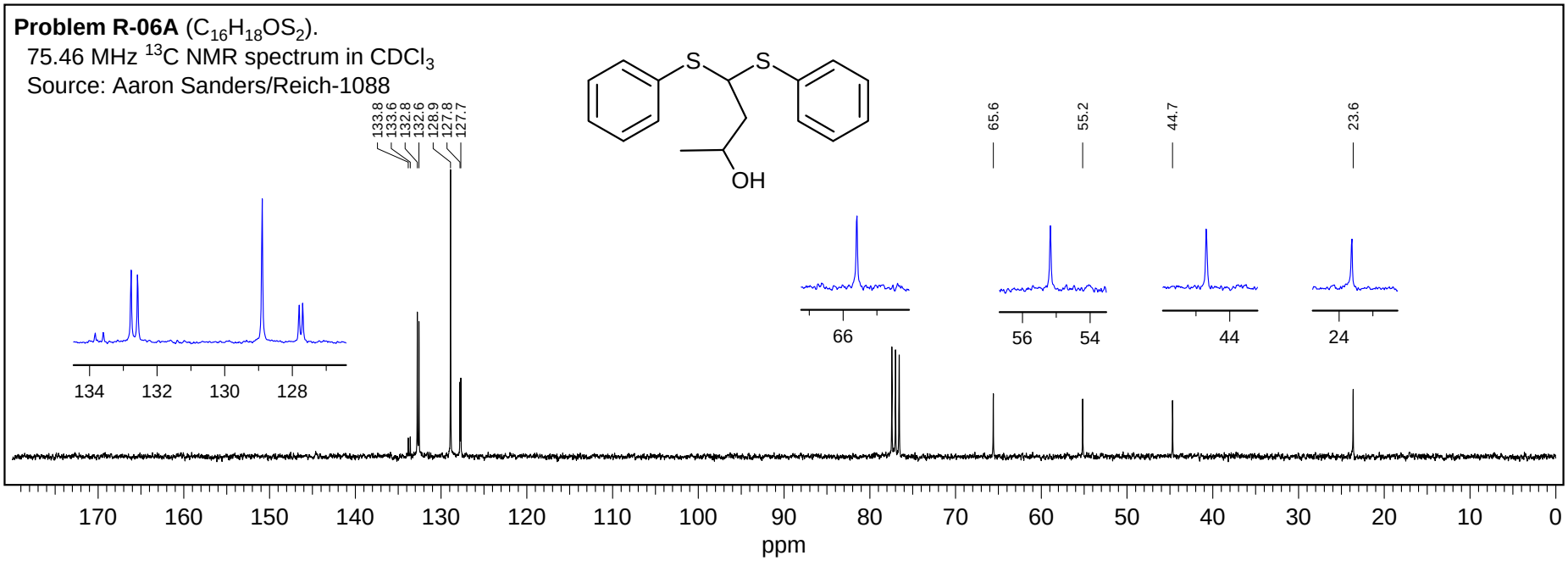
C-3

C-4

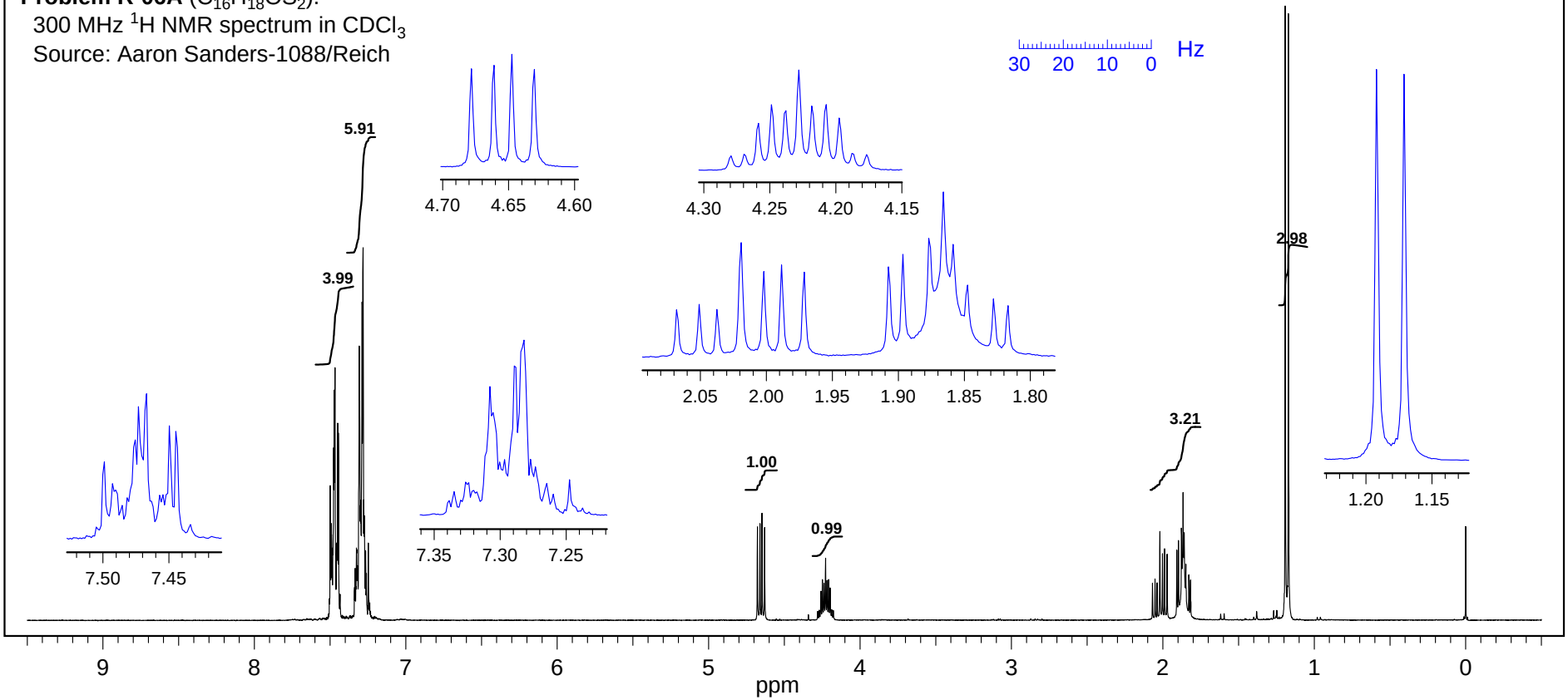
(c) Assign the carbon signals of C-1 to C-4. As part of your answer do a chemical shift calculation for C-3, .

(d) Explain the number of ^{13}C NMR signals in the region from 125 to 135 ppm.

Problem R-06A ($C_{16}H_{18}OS_2$).
75.46 MHz ^{13}C NMR spectrum in $CDCl_3$
Source: Aaron Sanders/Reich-1088

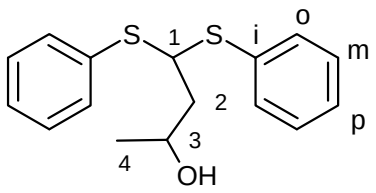


Problem R-06A ($C_{16}H_{18}OS_2$).
300 MHz ^{1}H NMR spectrum in $CDCl_3$
Source: Aaron Sanders-1088/Reich



PLT ex-1-2006-07-gq.plt

Problem R-06A ($C_{16}H_{18}OS_2$). Analyze the spectra shown on the next page. You are given the structure. Please use the numbering system given.



(a) Identify the OH proton.

1 Broad lump underneath the other protons at δ 1.87

(b) For the proton or set of protons on each carbon (C-1 to C-4), report the multiplet in the standard format (e.g. δ 3.44, tq, J = 7,2 Hz) .

1 C-1 δ 4.65, dd J = 10, 5 Hz, 1H

3 C-2 δ 2.02, ddd, J = 15, 9, 5 Hz
δ 1.86, ddd, J = 15, 9, 3 Hz

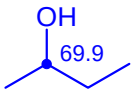
2 C-3 δ 4.23, dqd, J = 9, 6, 3 Hz, 1H

1 C-4 δ 1.19, d, J = 6 Hz, 3H

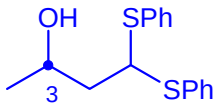
(c) Assign the carbon signals of C-1 to C-4. As part of your answer do a chemical shift calculation for C-3, .

C-3: Probably best to use a model:

4



$\delta_3 = 69.9 + 2(\gamma_{SPh}) = 69.9 + 2(-3) = 63.9$



$\delta_3 = -2.1 + 2\alpha_C + \alpha_{OH\text{-}iso} + \beta + 2(\gamma_{SPh})$
$= -2.1 + 18.2 + 41 + 9.4 - 6 = 60.5$

C-1 54.5
C-2 44.7
C-3 65.6
C-4 23.7

(d) Explain the number of ^{13}C NMR signals in the region from 125 to 135 ppm.

3 Because of the asymmetric center at C-3, the two PhS groups are diastereotopic, so expect 8 signals, actually see only 7 - probably the two meta carbons are at the same shift

only about a third got this correct