Problem R-06A (C_{10}H_{18}OS_{2})
300 MHz $^1$H NMR Spectrum in CDCl$_3$
Source: Aaron Sanders/Reich

Problem R-06A (C_{16}H_{18}OS_{2}).
75 MHz $^{13}$C NMR spectrum in CDCl$_3$
Source: Aaron Sanders/Reich
Problem R-06A (C₁₆H₁₈O₂S₂)
300 MHz ¹H NMR Spectrum in CDCl₃
Source: Mike Bowe/Reich g
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.

\[
\begin{align*}
\text{S} & \quad \text{S} \\
\text{OH} & \quad \text{1} \\
\text{i o} & \quad \text{2} \\
\text{m} & \quad \text{3} \\
\text{p} & \quad \text{4}
\end{align*}
\]

(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. \( \delta 3.44, \text{tq, } J = 7.2 \text{ Hz} \)).

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
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 $\delta$ 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. $\delta$ 3.44, tq, $J = 7.2$ Hz).

1 C-1 $\delta$ 4.65, dd $J = 10, 5$ Hz, 1H

3 C-2 $\delta$ 2.02, ddd, $J = 15, 9, 5$ Hz
$\delta$ 1.86, ddd, $J = 15, 9, 3$ Hz

2 C-3 $\delta$ 4.23, dqd, $J = 9, 6, 3$ Hz, 1H

1 C-4 $\delta$ 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:

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

4

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

(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