Problem R-13A (C_{19}H_{28}O_{3})
270 MHz $^1$H NMR Spectrum in CDCl$_3$
Source: Ieva Reich digitized hard copy (3/27) g
Problem R-13A. Assign several of the protons and analyze multiplets of a steroid.

(a) Assign and analyze the following signals (report multiplicity and $J$ values). Use the steroid numbering on the structure:

\[ \delta 3.55 \]  
\[ \delta 4.55 \]  
\[ \delta 5.40 \]

(b) Assign the 2-proton signal between \( \delta 2.4 \) and \( \delta 2.7 \). Briefly provide a rationale for this assignment. What kind of multiplet is this?

(c) Analyze this multiplet in a mathematically correct fashion (also show a coupling tree), using the frequencies given. Report coupling constants and chemical shifts. If two solutions are possible present them, and provide a rationale for choosing one of them.
Problem R-13A. Assign several of the protons and analyze multiplets of a steroid.

(a) Assign and analyze the following signals (report multiplicity and \( J \) values). Use the steroid numbering on the structure:

\[ \begin{align*}
\delta 3.55 & \quad H^3, \text{tdd, } J = 11, 7, 6 \text{ Hz} \\
\delta 4.55 & \quad H^{15}, \text{ddd, } J = 7, 5, 2 \text{ Hz} \\
\delta 5.40 & \quad H^6, \text{dt, } J = 5, 2 \text{ Hz}
\end{align*} \]

(b) Assign the 2-proton signal between \( \delta 2.4 \) and \( \delta 2.7 \). Briefly provide a rationale for this assignment. What kind of multiplet is this? AB of ABX

Apart from \( H^3 \), \( H^{15} \) and \( H^6 \), the \( H^{16} \) protons will be the most downfield. The large \( ^2J \) (19 Hz) also means the protons must be next to the keto group

(c) Analyze this multiplet in a mathematically correct fashion (also show a coupling tree), using the frequencies given. Report coupling constants and chemical shifts. If two solutions are possible present them, and provide a rationale for choosing one of them.

\[
\begin{align*}
\delta_A & = 2.60 \quad \delta_B & = 2.53 \\
\delta_A & = 2.57 \quad \delta_B & = 2.56
\end{align*}
\]

Solution 1 is correct - can't have a negative \( ^3J \), as in Sol 2. Also the Sol. 2 couplings are unreasonably large
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