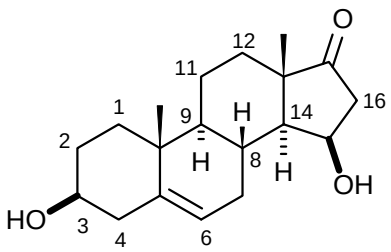
Problem R-13A ($C_{19}H_{28}O_3$)

270 MHz ^{1}H NMR Spectrum in $CDCl_3$

Source: Ieva Reich digitized hard copy (3/27) g



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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:

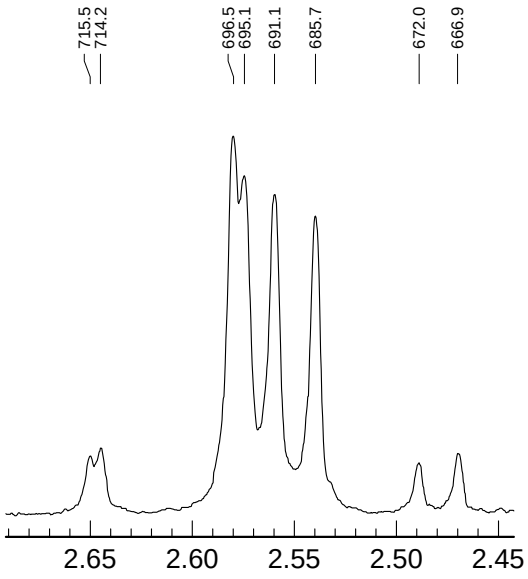
δ 3.55_____

δ 4.55 _____

δ 5.40 _____

(b) Assign the 2-proton signal between δ 2.4 and δ 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:

δ 3.55 H^3, tdd, J = 11, 7, 6 Hz

δ 4.55 H^{15}, ddd, J = 7, 5, 2 Hz

δ 5.40 H^6, dt, J = 5, 2 Hz

(b) Assign the 2-proton signal between δ 2.4 and δ 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 ^{2}J (19 Hz) also means the protons must be next to the keto group

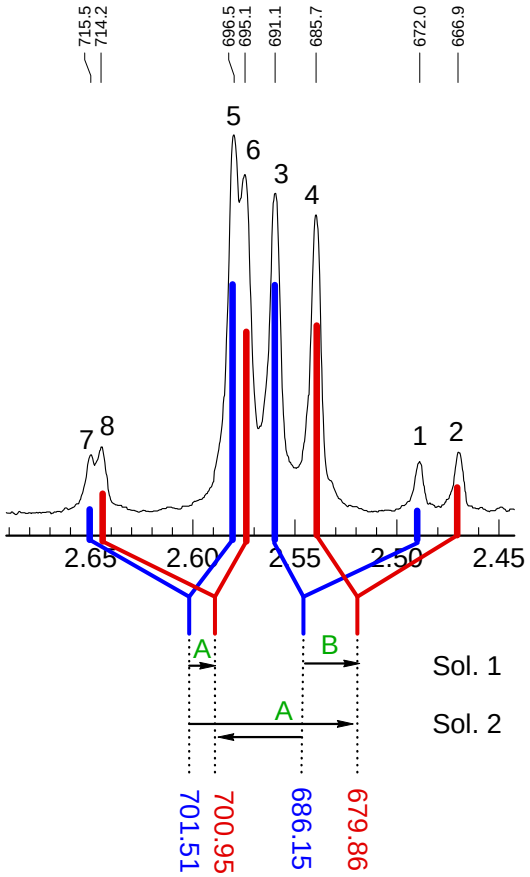
(c) Analyze this multiplet in a mathematically correct fashion (also show a coupling tree), using the frequencies given. Report coupling constants and chemical shifts. If two solutions are possible present them, and provide a rationale for choosing one of them.

$c_- = 693.8$ $\Delta\nu_{ab-} = 15.3$ $c_- \pm \Delta\nu_{ab-}/2 =$ 701.51, 686.15

$c_+ = 690.4$ $\Delta\nu_{ab+} = 21.2$ $c_+ \pm \Delta\nu_{ab+}/2 =$ 700.95, 679.86

	Solution 1	Solution 2
J_{AB}	19.1	19.1
J_{AX}	0.6	-14.8
J_{BX}	6.3	21.6
ν_A	701.2	693.5
ν_B	683.0	690.7
ν_{AB}	18.2	2.9
i14=i15	0.0074	0.47
δ_A	2.60	2.57
δ_B	2.53	2.56

1	671.99
2	666.91
3	691.12
4	685.71
5	696.54
6	695.1
7	715.5
8	714.25



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

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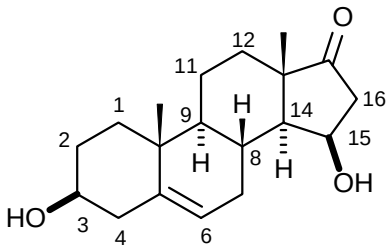

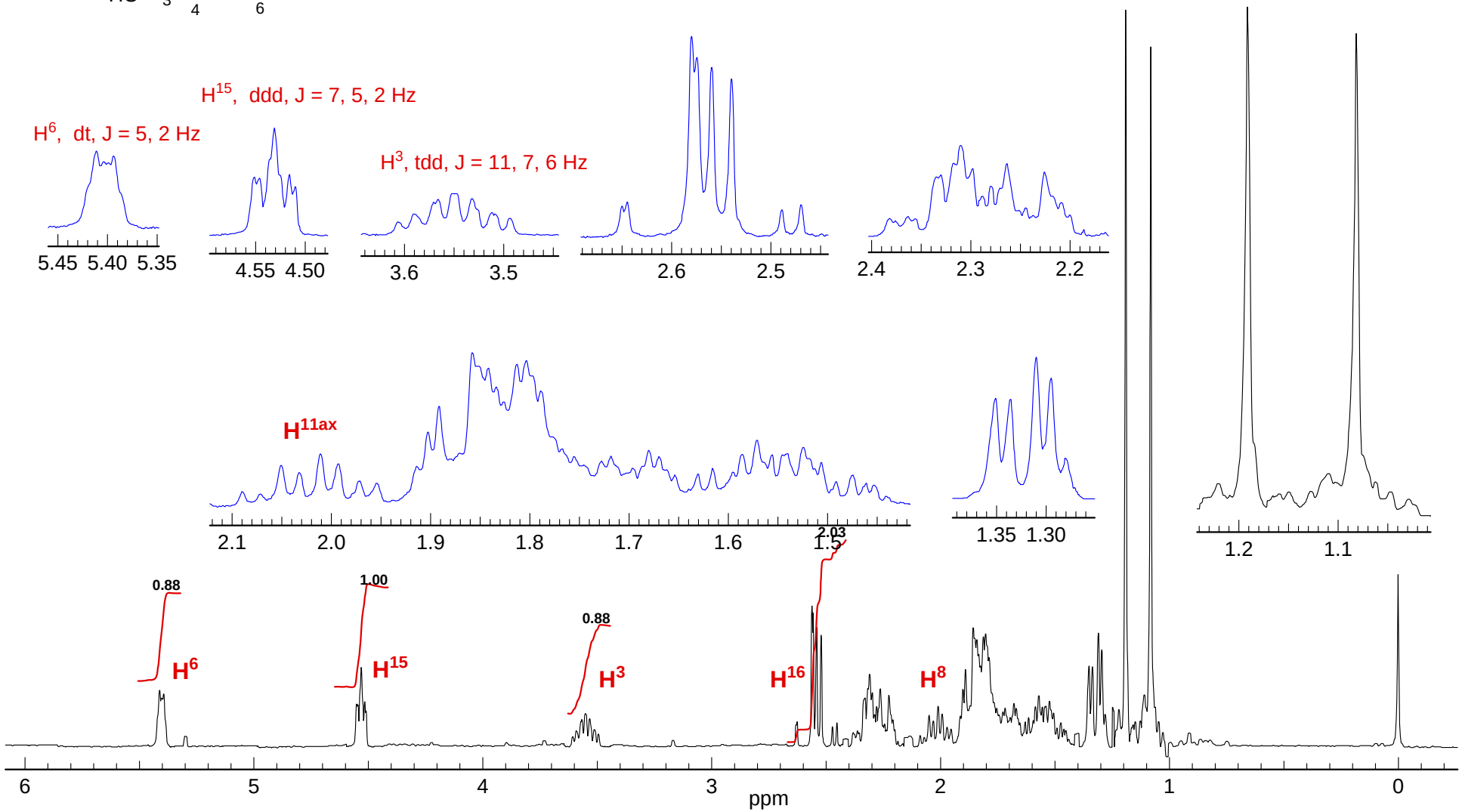

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