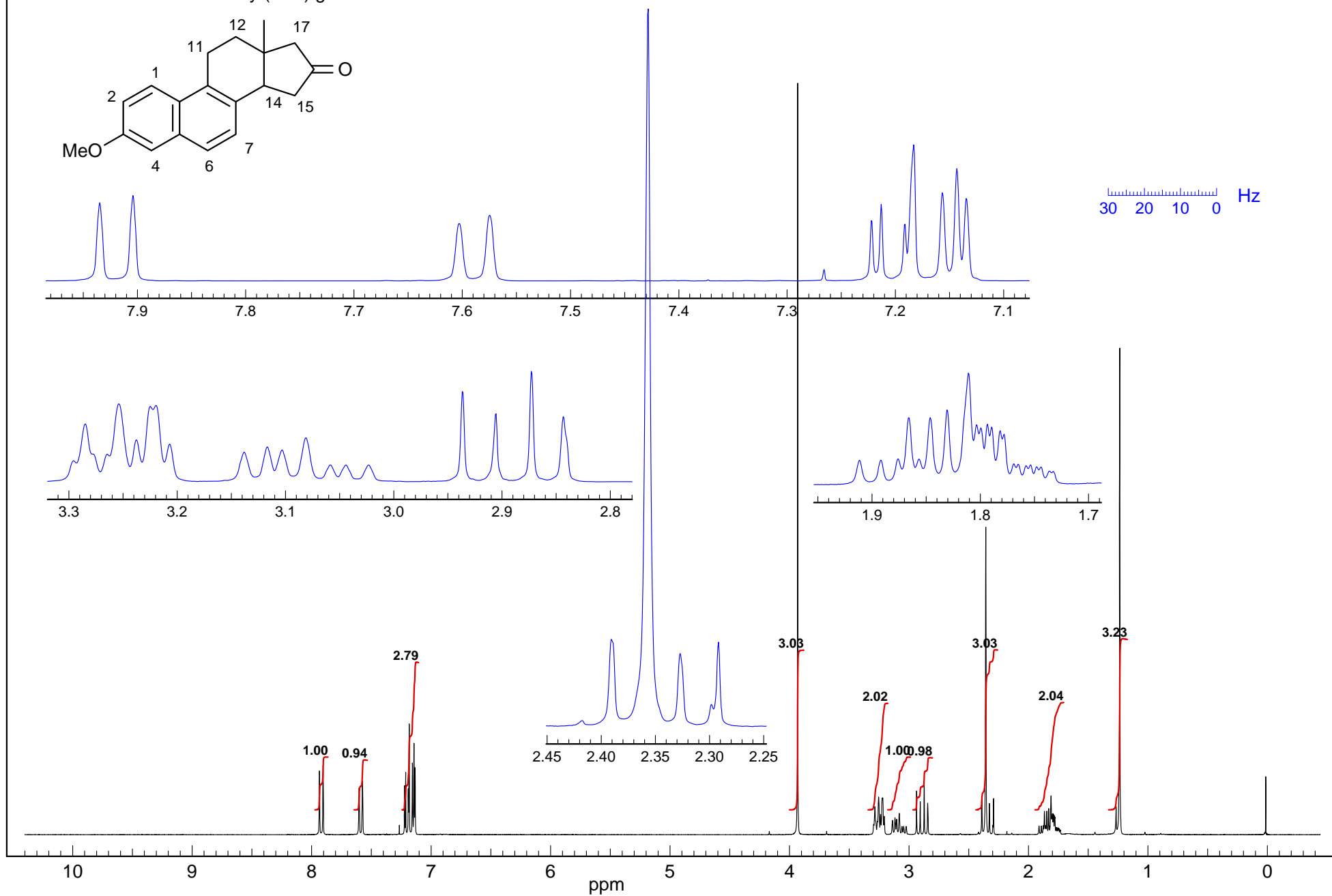
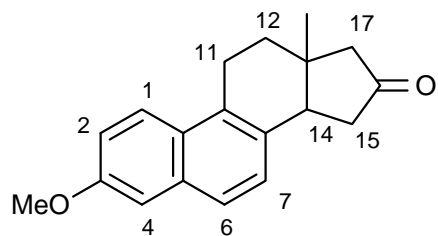


Problem R-08K. C₁₉H₂₀O₂
300 MHz ¹H NMR spectrum in CDCl₃
Source: Wilds/Charlie Fry (C59) g

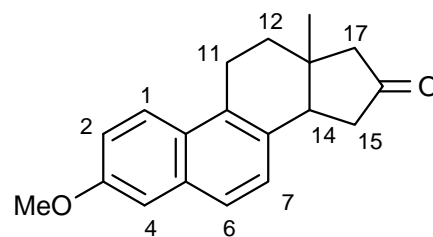


24 **Problem R-08K** ($C_{19}H_{20}O_2$). You are asked to assign the protons of steroid from the 1H NMR spectrum and a H-H COSY. Only answer the questions, it is not necessary to do a complete analysis of all of the signals.

(a) Assign the 2 protons at δ 1.8.

2

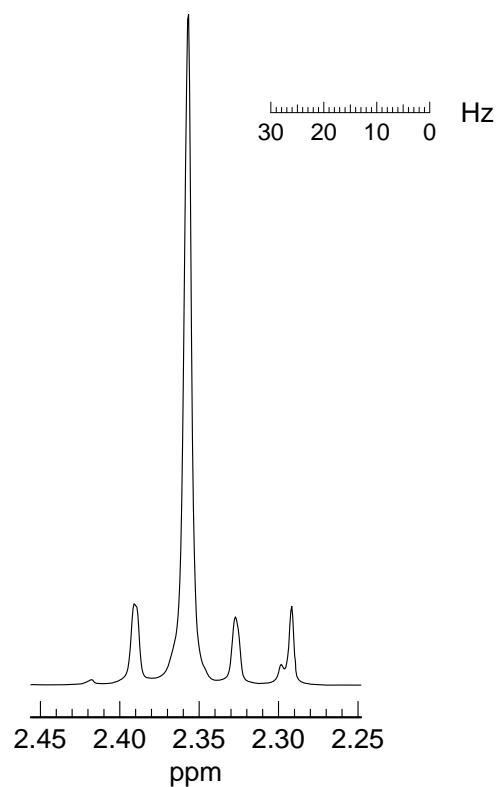
(b) Assign each multiplet from δ 2.8 to 3.3 (4 protons)



8

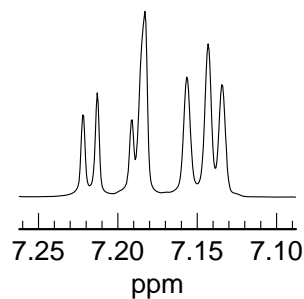
(c) Make a detailed assignment of the 3 protons at δ 2.3. Report δ , multiplicity and couplings.

6

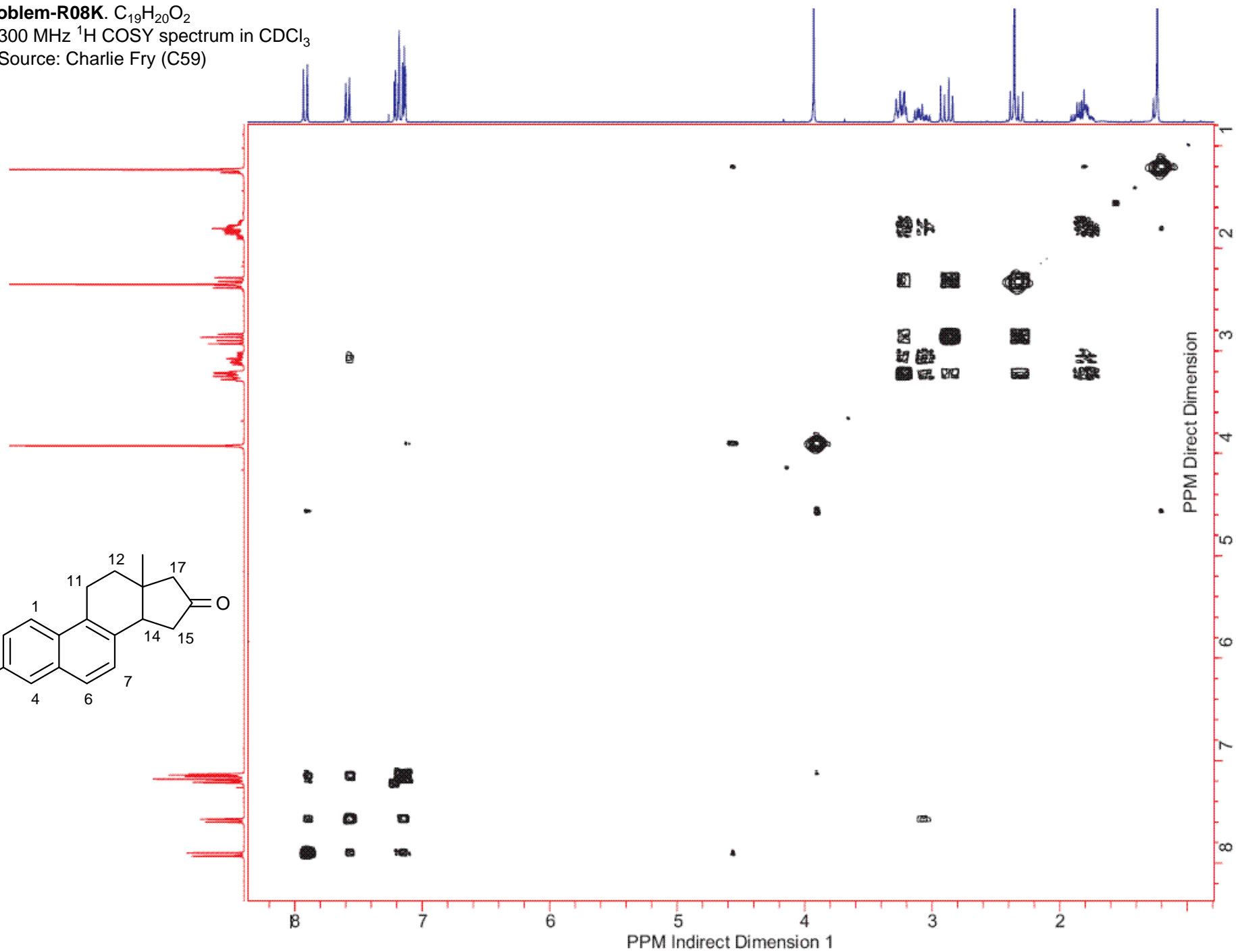
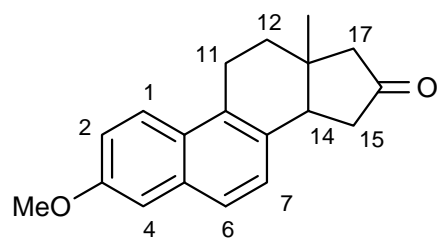


(d) Assign the 3 protons at δ 7.1 to 7.3. Label the expansion below with assignments and coupling trees.

8



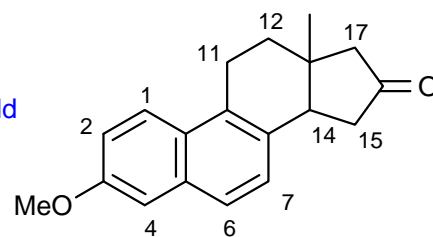
Problem-R08K. C₁₉H₂₀O₂
300 MHz ¹H COSY spectrum in CDCl₃
Source: Charlie Fry (C59)



Problem R-08K ($C_{19}H_{20}O_2$). You are asked to assign the protons of steroid from the 1H NMR spectrum and a H-H COSY. Only answer the questions, it is not necessary to do a complete analysis of all of the signals.

(a) Assign the 2 protons at δ 1.8.

2 From chemical shift consideration, these are the H^{12} protons, one a ddd ($J=14,11,6$) from coupling to the gem and two vicinal protons, the other a dddd ($J=14,7,3,1$) with an additional long-range coupling. In the COSY they are coupled only to the H^{11} protons at 3.08 and 3.25



(b) Assign the 4 protons at δ 2.8 to 3.3.

2.89: H^{15} (dd, $^2J = 19$, $^3J = 9.5$) COSY correlation to other H^{15} and H^{14} . α -keto gives large 2J (19 Hz)

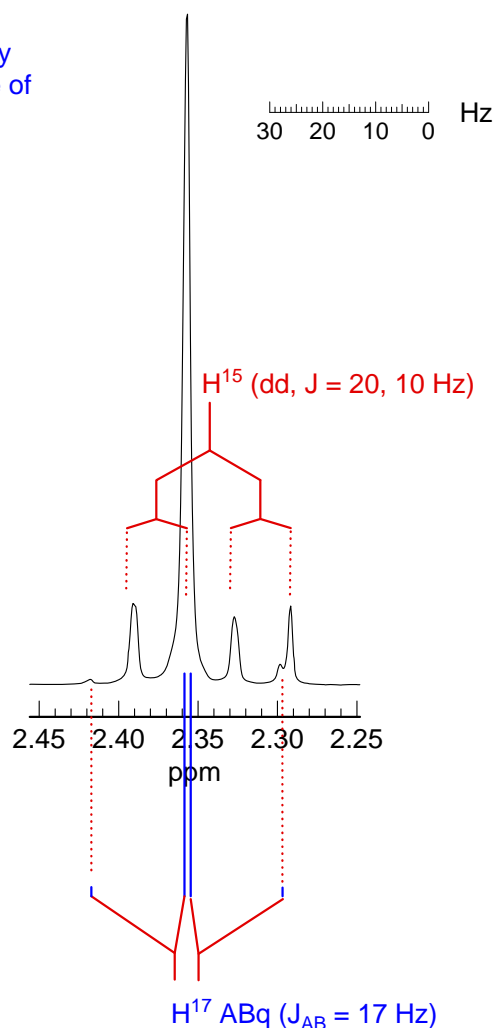
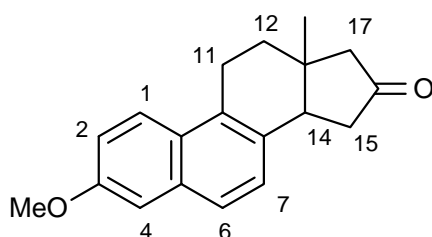
8 3.08: H^{11} - axial (ddd, $^2J = 17$ Hz, $^3J = 11$ Hz, $^3J = 6$ Hz) α -phenyl also raises 2J (17 Hz)

3.25: H^{11} - equatorial COSY correlation to other H^{11} and the H^{12} protons

3.25: H^{14} (t, $J = 10$ Hz) COSY shows correlation to both H^{15} protons

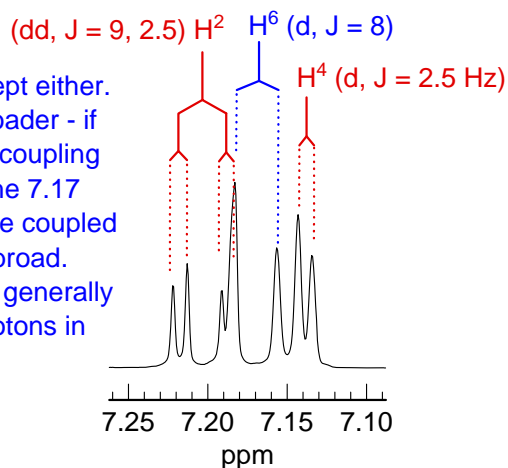
(c) Make a detailed assignment of the 3 protons at δ 2.3. Report δ , multiplicity and couplings.

6 These are the superimposed signals of the two protons at H^{17} (an AB quartet with a very small δ_{AB} and large $^2J_{AB}$ (ca 17 Hz), outer lines barely visible, inner lines not resolved, so it cannot be solved) and a dd for one of the H^{15} protons



(d) Assign the 3 protons at δ 7.1 to 7.3. Label the expansion below with assignments and coupling trees.

8 Hard to assign H^6 and H^7 - accept either. The 7.59 signal is quite a bit broader - if assign to H^7 , could be possible coupling to H^{14} (which is broadened). The 7.17 signal, if assigned to H^6 could be coupled to H^4 (but H^4 is not particularly broad. There is also the argument that generally α protons are downfield of β -protons in naphthalenes.

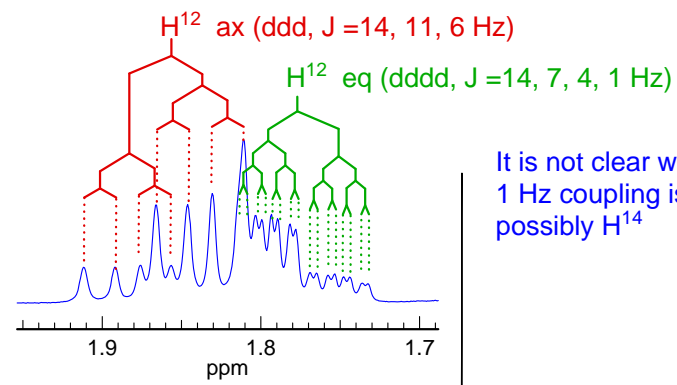
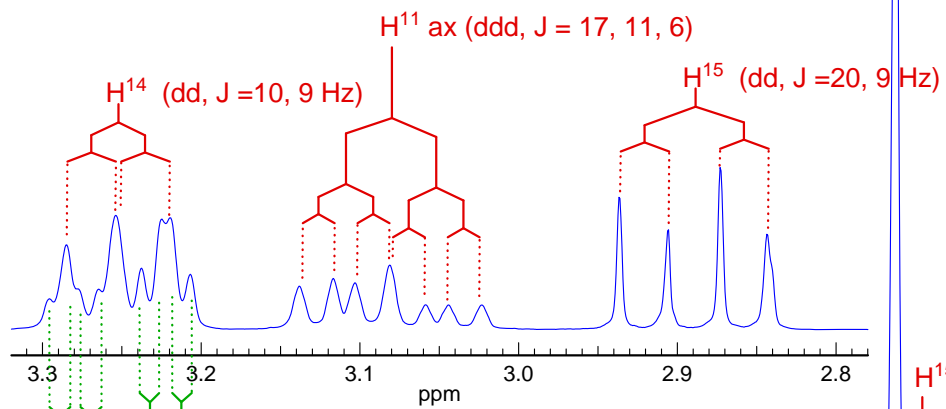
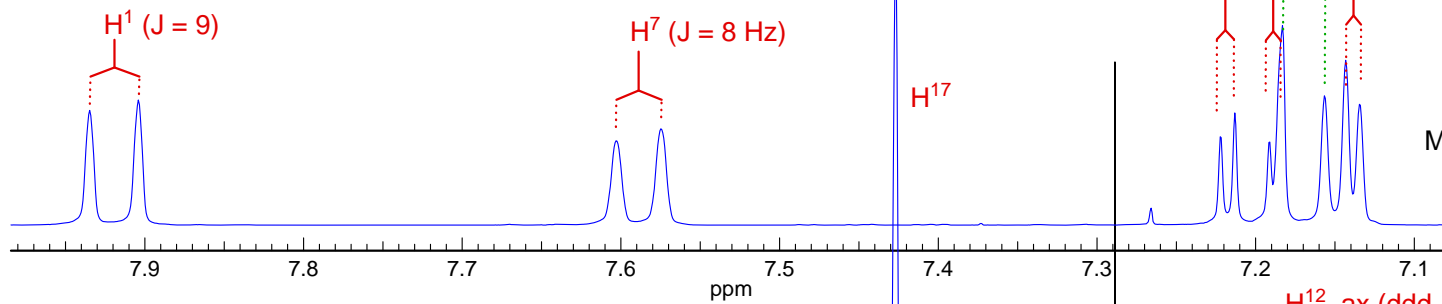
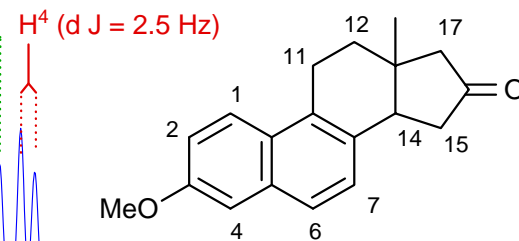


The AB quartet cannot be solved because the middle two lines are not separated.

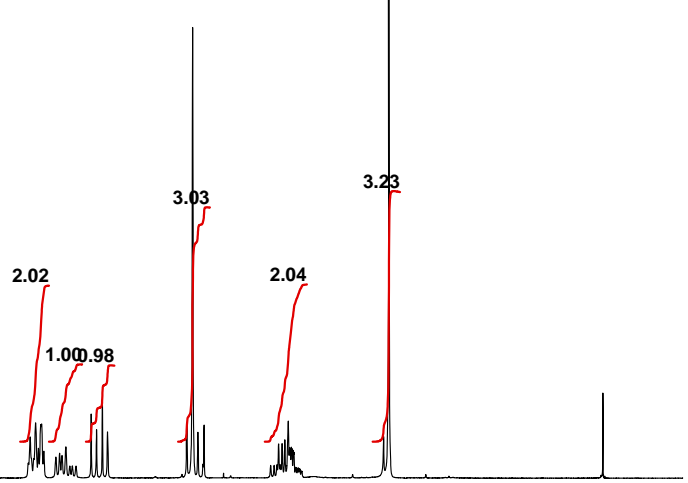
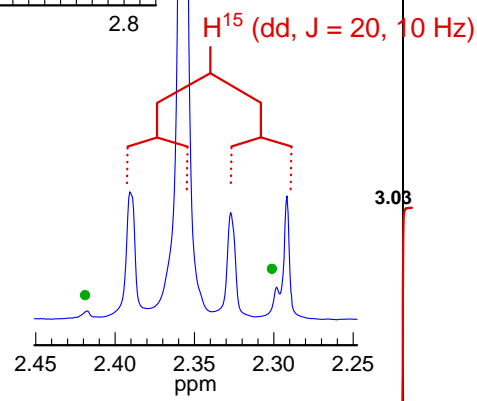
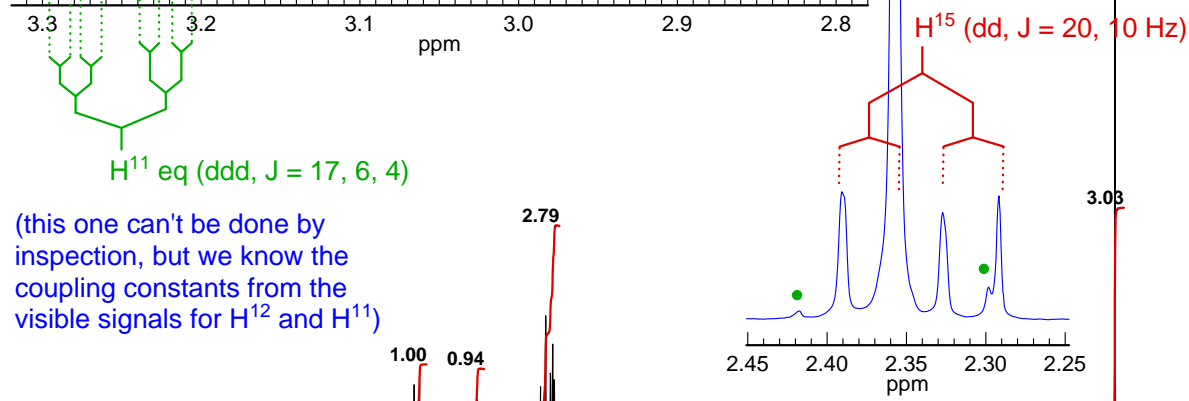
Problem R-08K. C₁₉H₂₀O₂
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 Source: Wilds/Charlie Fry (C59) g

H¹⁷ ABq (J_{AB} = 17 Hz)

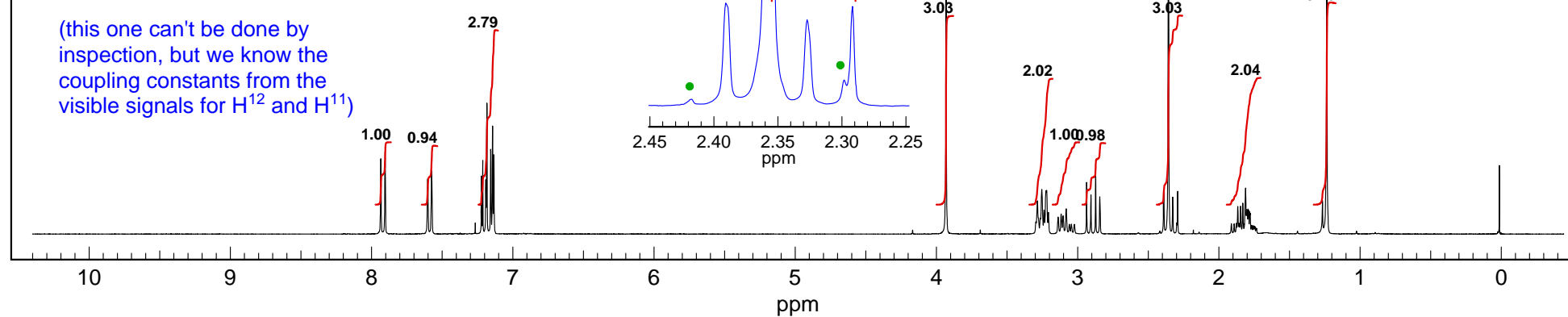
(dd, J = 9, 2.5) H² H⁶ (d, J = 8)



It is not clear what the 1 Hz coupling is - possibly H¹⁴



(this one can't be done by inspection, but we know the coupling constants from the visible signals for H¹² and H¹¹)



Problem-R08K. C₁₉H₂₀O₂
300 MHz ¹H COSY spectrum in CDCl₃
Source: Charlie Fry (C59) 05/39

