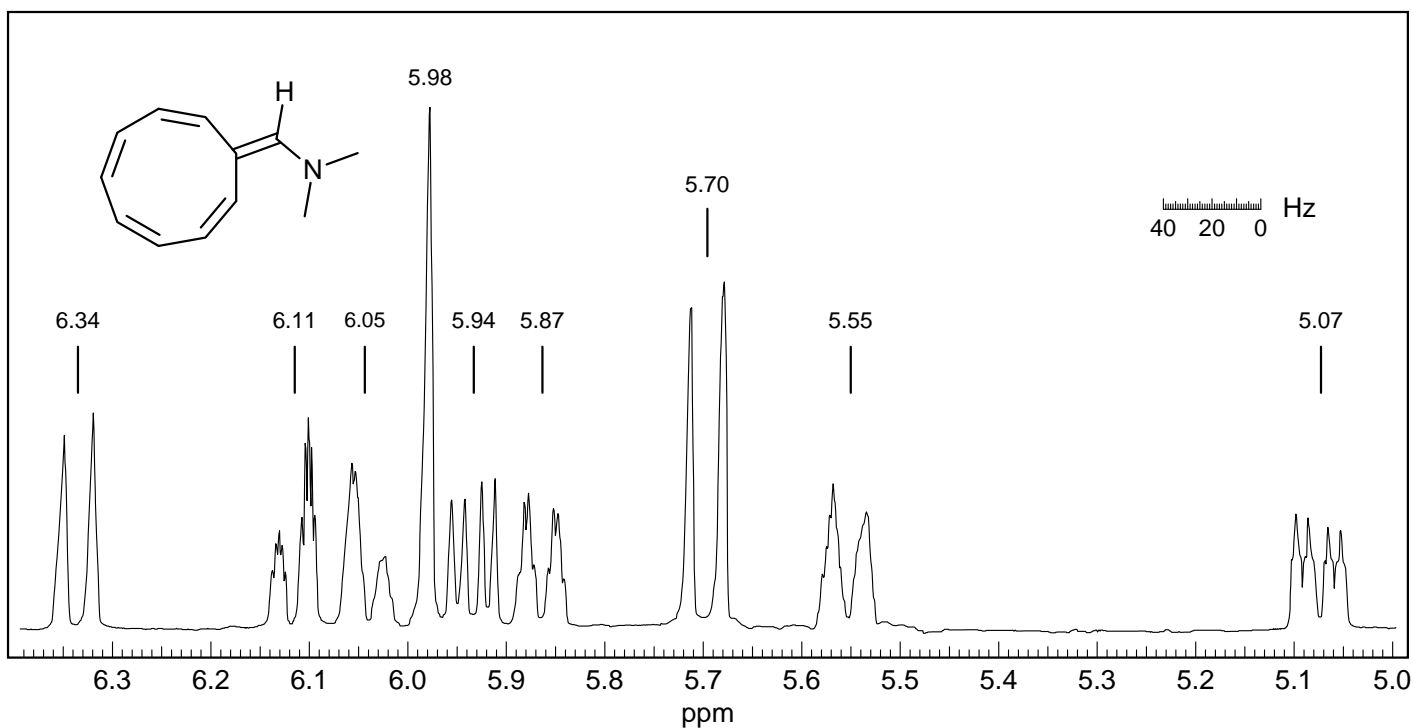


Problem R-920 (C₁₂H₁₅N). Assign the individual signals of the compound whose 400 MHz ¹H NMR spectrum (CDCl₃, -10 °C) is given below. Use couplings, chemical shifts and intensities in your analysis. From their analysis, the authors deduced the conformation shown (Otter, A.; Neuenschwander, M.; Kellerhals, H. P. *Magn. Reson. Chem.* **1986**, 24, 353).



δ

6.34 ____

6.11 ____

6.05 ____

5.98 ____

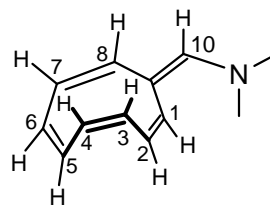
5.94 ____

5.87 ____

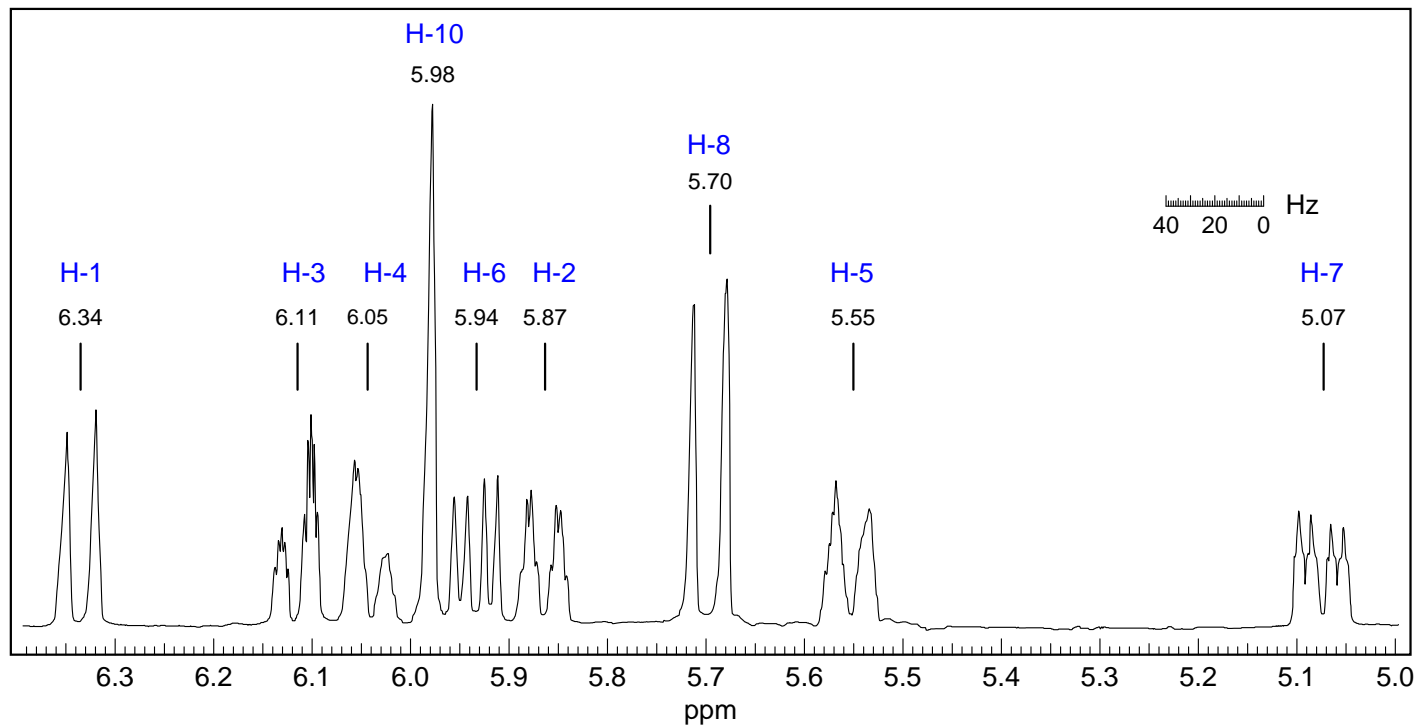
5.70 ____

5.55 ____

5.07 ____



Problem R-920 (C₁₂H₁₅N). Assign the individual signals of the compound whose 400 MHz ¹H NMR spectrum (CDCl₃, -10 °C) is given below. Use couplings, chemical shifts and intensities in your analysis. From their analysis, the authors deduced the conformation shown (Otter, A.; Neuenschwander, M.; Kellerhals, H. P. *Magn. Reson. Chem.* **1986**, 24, 353).



δ

6.34 1
 6.11 3
 6.05 4
 5.98 10
 5.94 6
 5.87 2
 5.70 8
 5.55 5
 5.07 7

1. The two doublets without additional coupling have to be H-1 and H-8. The enamine nitrogen can only conjugate with the 7-8 double bond trans to the NMe₂ group, not with the 1-2 double bond (steric effects) so H7 and H5 are moved upfield (this is how you decide between H-1 and H-8). Once H-7 is assigned, then all the others fall into place by consideration of leanings and the small coupling

2. Start with H-8 (5.70) - leaning shows it must be coupled to 5.07 (H-7) not to 5.55, which would have to lean a lot more (observed leaning is 1.10, coupling to 5.55 would have to lean 1.6).

3. The unique small coupling to H-7 must be to H-6, 5.94

4. Leaning of H-6 (5.94) means coupling to 5.55, which must be H-5 (not to 5.87)

5. Now start with H-1 (6.34). Only remaining proton it can be coupled to is 5.87, which is H-2

6. This leaves H-3 and H-4, 6.11, 6.05. Hard to decide which is which.

