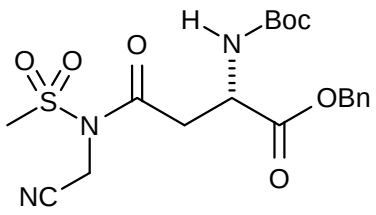
Problem R-09C ($C_{19}H_{25}N_3O_6S$)

300 MHz ^{1}H NMR spectrum in $CDCl_3$

Source: Ron Hinklin/Gellman g



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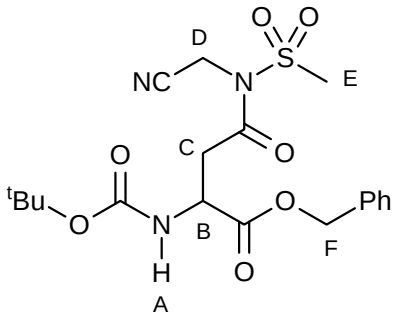
Problem R-09C ($C_{19}H_{25}N_3O_6S$). You are given the 300 MHz ^1H NMR spectrum of a protected amino acid.



(a) Do a chemical shift calculation for protons D and F to help in the assignments below. Show your work.

D:

F:

(b) Identify the proton(s) at δ 3.3 - 3.6 (A-F):_____ What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given. Report the results in the standard format (e.g. G: δ 2.88, 1H, dd, J= 12, 6 Hz)

(c) Identify the proton(s) at δ 4.5 - 4.8 (A-F):_____ What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given.

(d) Identify the proton(s) at δ 5.1 - 5.3:_____ What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given.

(e) Identify the proton at δ 5.5 - 5.7:_____

25

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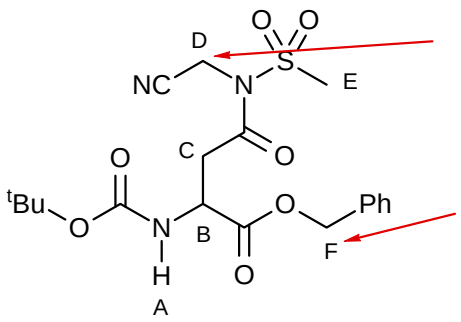


SO_2 is β, not α

Base:	1.2	
α-N-C=O (CH_2)	2.25	(Used Phthalimido)
α-CN (CH_2)	1.10	
	4.55	obs: 4.6

Base:	1.2	
α-Ph (CH_2)	1.45	
α-C(=O)O (CH_2)	2.95	
	5.6	obs: 5.2

4 (a) Do a chemical shift calculation for protons D and F to help in the assignments below. Show your work.

D: See above

F:

(b) Identify the proton(s) at δ 3.3 - 3.6 (A-F): C, E What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given. Report the results in the standard format (e.g. G: δ 2.88, 1H, dd, J= 12, 6 Hz)

5 C: AB of ABX.First order analysis gives dd, J_{AB} = 17.9 Hz, J_{AX} = 4.5 Hz; and dd, J_{AB}=17.9, J_{BX} = 4.3 Hz
To get accurate chemical shifts in "AMX" type of analysis have to remove J_{AX} and J_{BX} and calculate the resulting AB quartet properly (i.e., average 1&2=997.7, 3&4=1015.5, 5&6=1033.0 and 7&8=1050.9, and solve AB quartet: ν_{AB} = 30.5, ν_{center} = 1024.3, ν_A = 1039.5 (δ 3.47), ν_B = 1009.0 (δ 3.36)

3 E δ 3.2, 3H, s

(c) Identify the proton(s) at δ 4.5 - 4.8 (A-F): B & D What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given.

3 B: δ 4.7, dt, 1H, J = 8, 3 Hz,

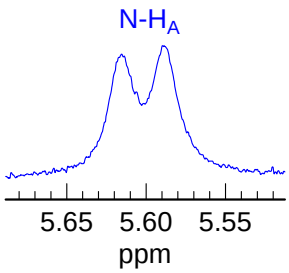
4 D: AB quartet, ν_{AB} = 7.15, J_{AB} = 17.8 (17.4, 18.3) , ν_{center} = 1378.0, ν_A = 1374.4, ν_B = 1381.6
δ_A = 4.58, δ_B = 4.61

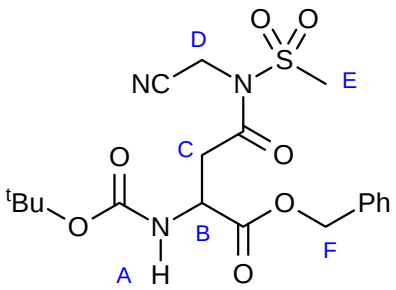
(d) Identify the proton(s) at δ 5.1 - 5.3: F What kind of pattern(s) is (are) these? Calculate accurate shifts and couplings from the peak-pick Hz values given.

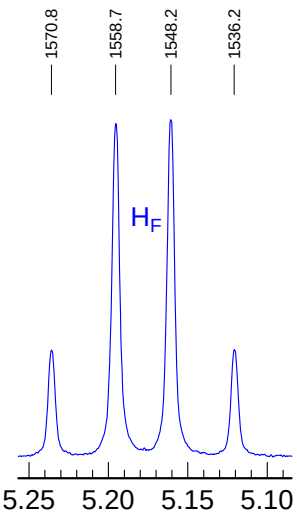
4 AB quartet

F: AB quartet, ν_{AB} = 19.0, J_{AB} = 12 (12.0, 12.1) , ν_{center} = 1553.4, ν_A = 1543.9 (δ 5.15), ν_B = 1562.9 (δ 5.21)

2 (e) Identify the proton at δ 5.5 - 5.7: A







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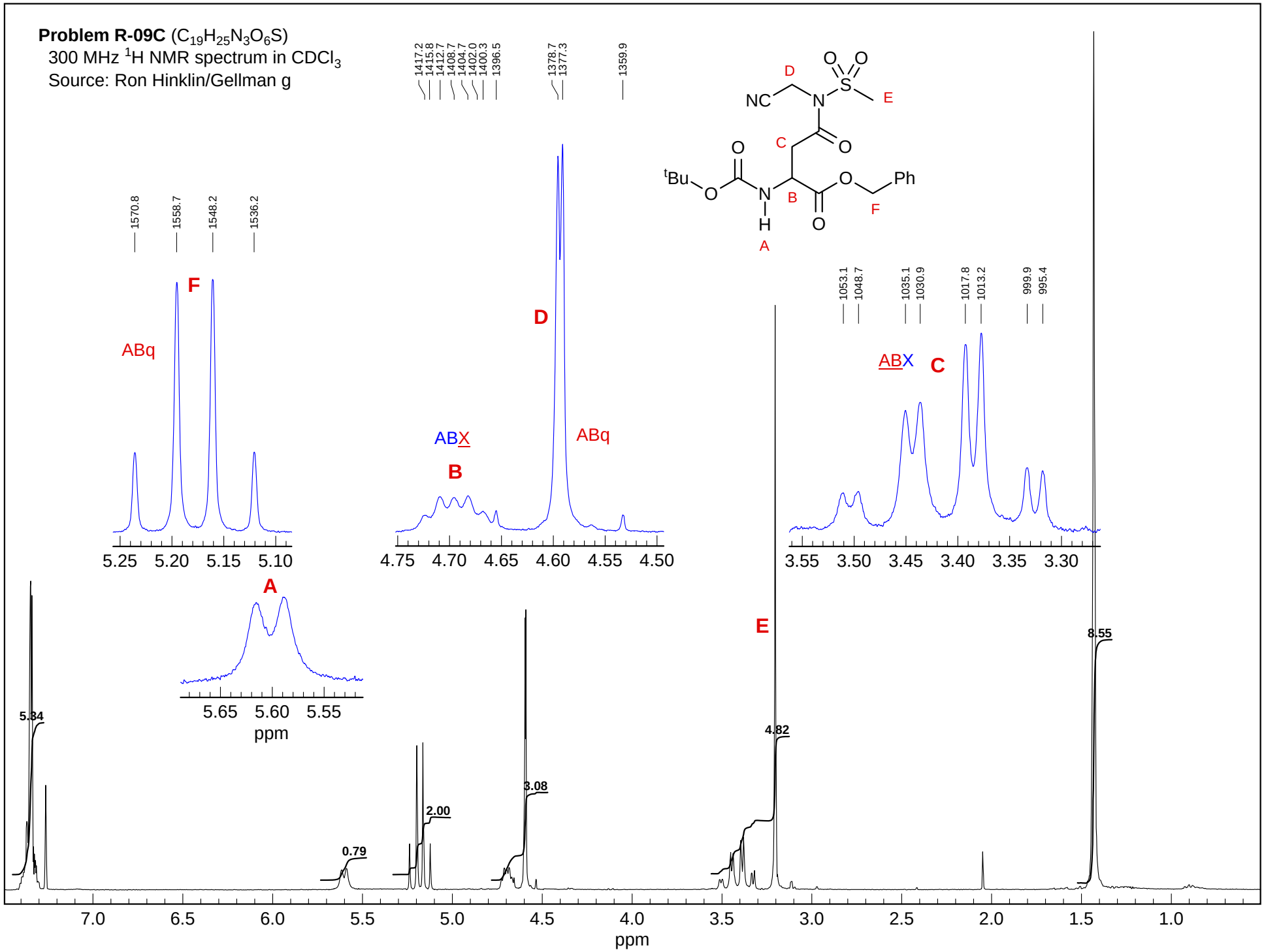

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