Problem of the Month:

February 2015

Coupled and decoupled $^{13}$C NMR spectra, recorded at 100 MHz

List of $^{13}$C chemical shifts: 161.6, 143.5, 127.2, 115.0, 50.5, 27.8
Strategy

(1) First, determine the number of DBE for the molecule. Its molecular formula is $C_8H_{10}FN$.

(2) Various types of $^{13}$C NMR spectra are presented on the first page: Without $^1$H or $^{19}$F coupling (top trace), with only $^{19}$F coupling visible (middle) and with both, $^1$H and $^{19}$F coupling present (bottom). With this information, you can assign the number of attached hydrogen or fluorine atoms.

(3) Now, assign protons to corresponding carbons from the $^1$H NMR spectrum on the next page. Try to assign the multiplets and derive the observable spin systems. Note, the upper spectrum is $^{19}$F decoupled.
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\(^1\text{H}\{^{19}\text{F}\}\) NMR spectrum

\(^1\text{H}\) NMR spectrum, 400 MHz

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Hints

(1) The $^1$H signal at $\delta_1$ 1.38 will cease, once a drop of deuterated water is added to the sample!

(2) This information, together with the number of DBE, should be helpful.

(3) This month’s problem does not contain any through-bond correlation experiments. The only 2D experiment available as additional information is a heteronuclear Overhauser effect (next page). This type of spectrum gives information about nuclei which are (farely) close in space (maximum 4 Å as a rule of thumb).

(4) Also, you might verify the other part of the aromatic spin system with the $^{19}$F NMR spectrum given on the next page.
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H,F HOESY, 400/376 MHz

$^{19}$F NMR spectra, 376 MHz

$^{19}$F NMR spectrum

(ppm)

8.6 & 5.5 Hz

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Solution

(1) The target molecule should be, according to DBE and $^1$H/$^{13}$C chemical shifts in the sp$^2$ region of the spectra, an aromatic compound. From the sum formula and the hint with the perishing signal at $\delta_H$ 1.38 (intensity = 2H), an amine is feasible.

(2) There is an AA‘XX‘ system visible in the $^1$H NMR spectrum, if $^{19}$F is decoupled. In combination with the $^{19}$F coupling observed in the $^{13}$C{$^1$H} spectrum, the substitution pattern can be identified. This is affirmed by the NOE spectrum.

(3) To derive the aliphatic amine part, the $^1$H NMR spectrum is sufficient: Here, a AX$_3$ spin system is observed.

(4) No solution? Enter the chemical shifts of the $^{13}$C signals in nmrsshiftdb2 as a „spectrum search“ (option „complete“) or click here.