NMR Characterization of Microstates of SM07

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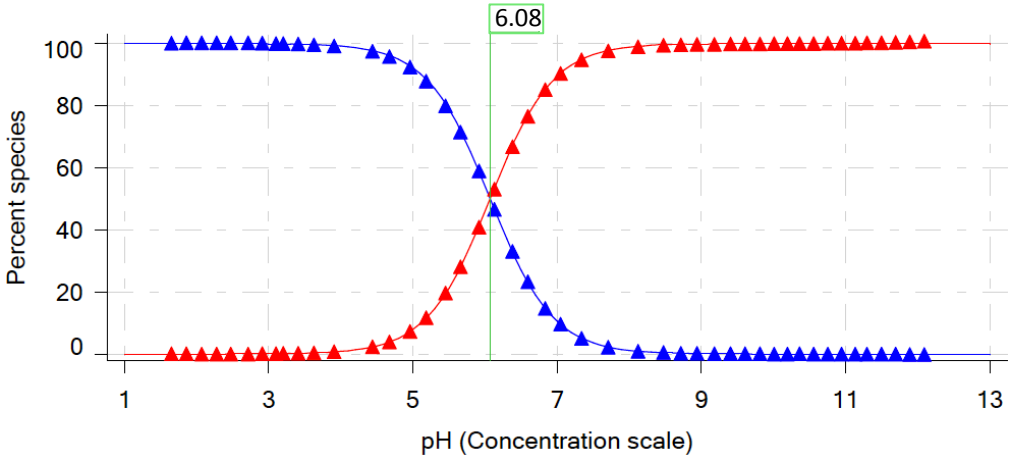
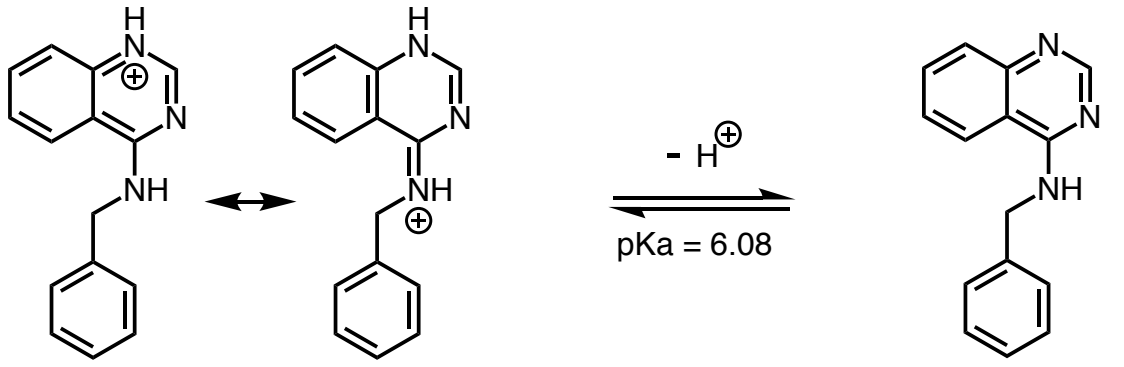
Distribution of species and pKa value of SM07 was determined with UV-metric pKa measurement with Sirius T3.



NMR characterization of SM07 showed that pKa 6.07 was related to a microscopic protonation state transition between SM07_micro006 and SM07_microo004 microstates.



SM07_micro006

SM07_micro004

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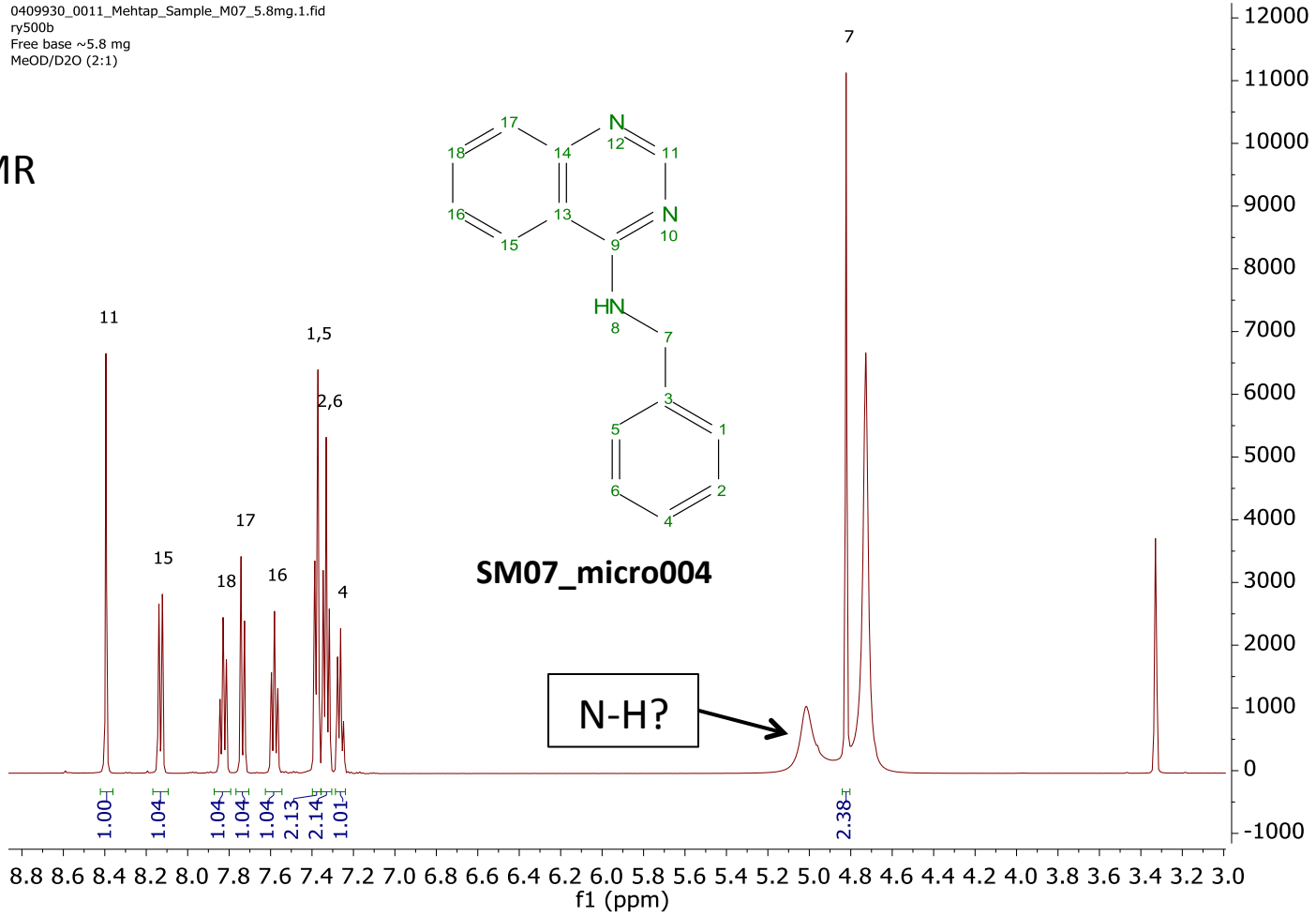
SM07 structure was assigned with ^{1}H NMR, ^{13}C NMR, COSY, HSQC, HMBC and ^{15}N HMBC with 5 – 6 mg of sample in methanol-d4/D_2O in 2:1 ratio (free-base, pH 5-6*) – obtaining reference chemical shifts essential for this study.

^{1}H NMR



* Measured with pH paper.

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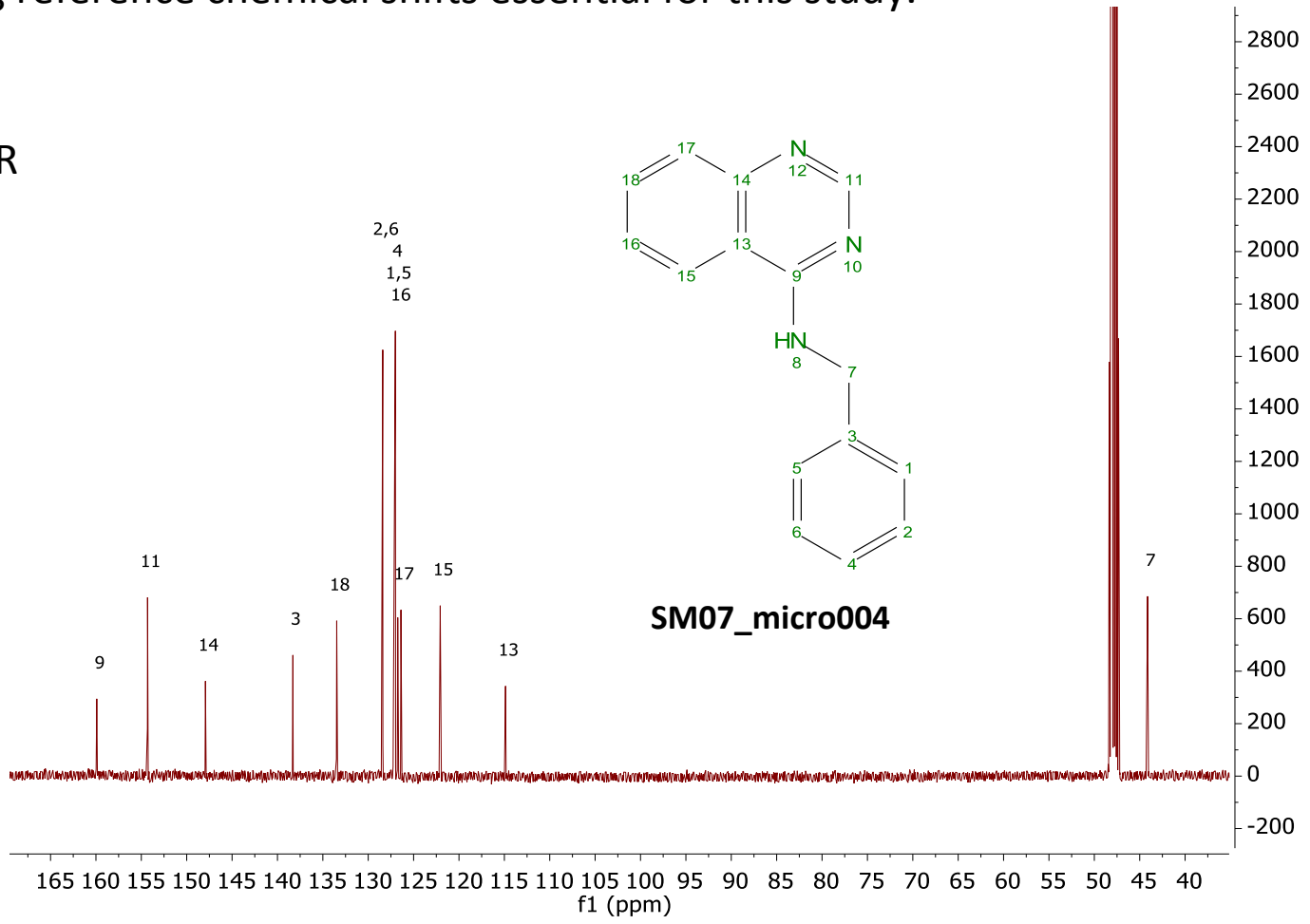
^{13}C NMR



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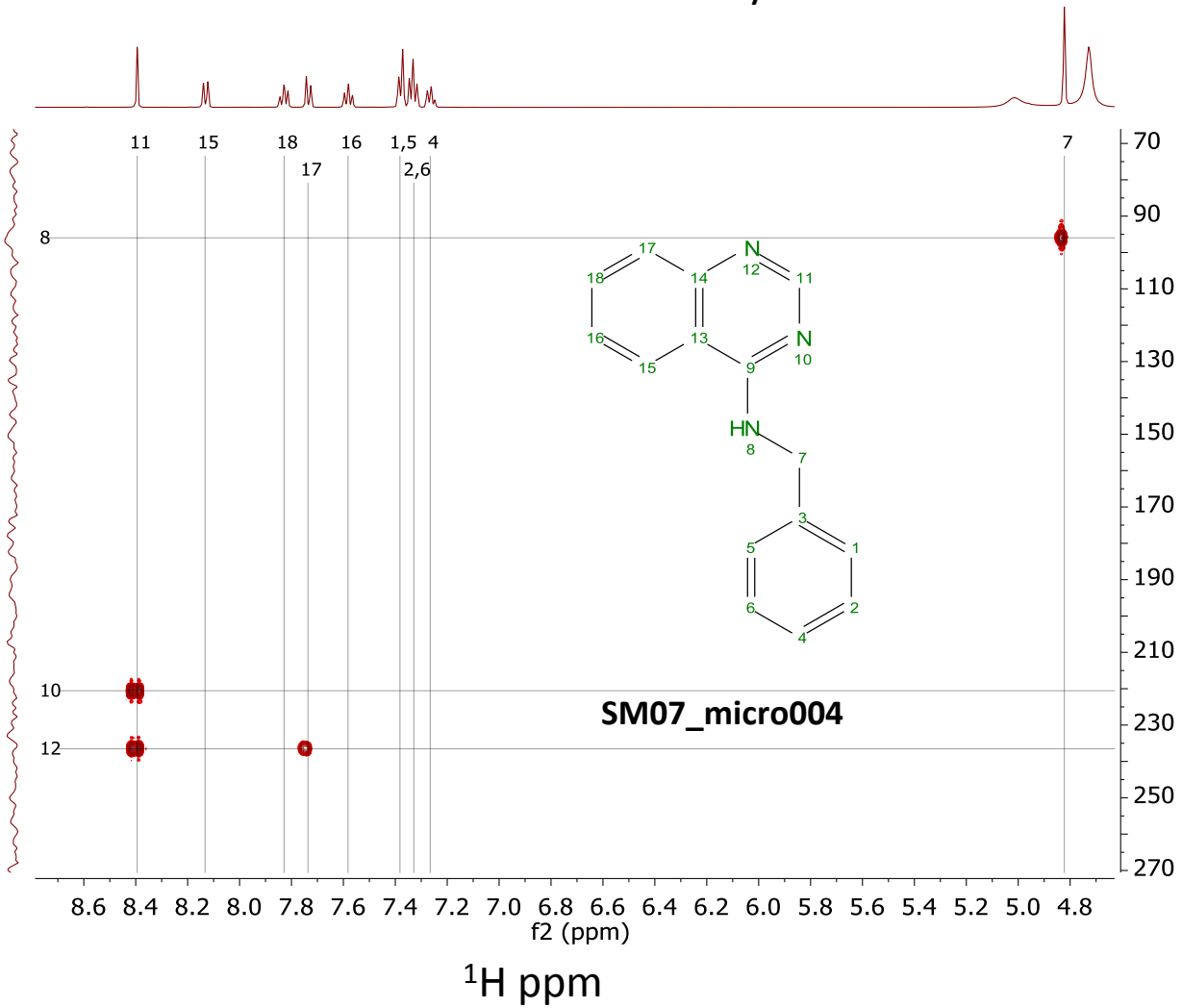
^{15}N HMBC

N-8 = 96 ppm
N-10 = 220.5 ppm
N-12 = 236.5 ppm



^{15}N ppm

^{1}H ppm

* Measured with pH paper.

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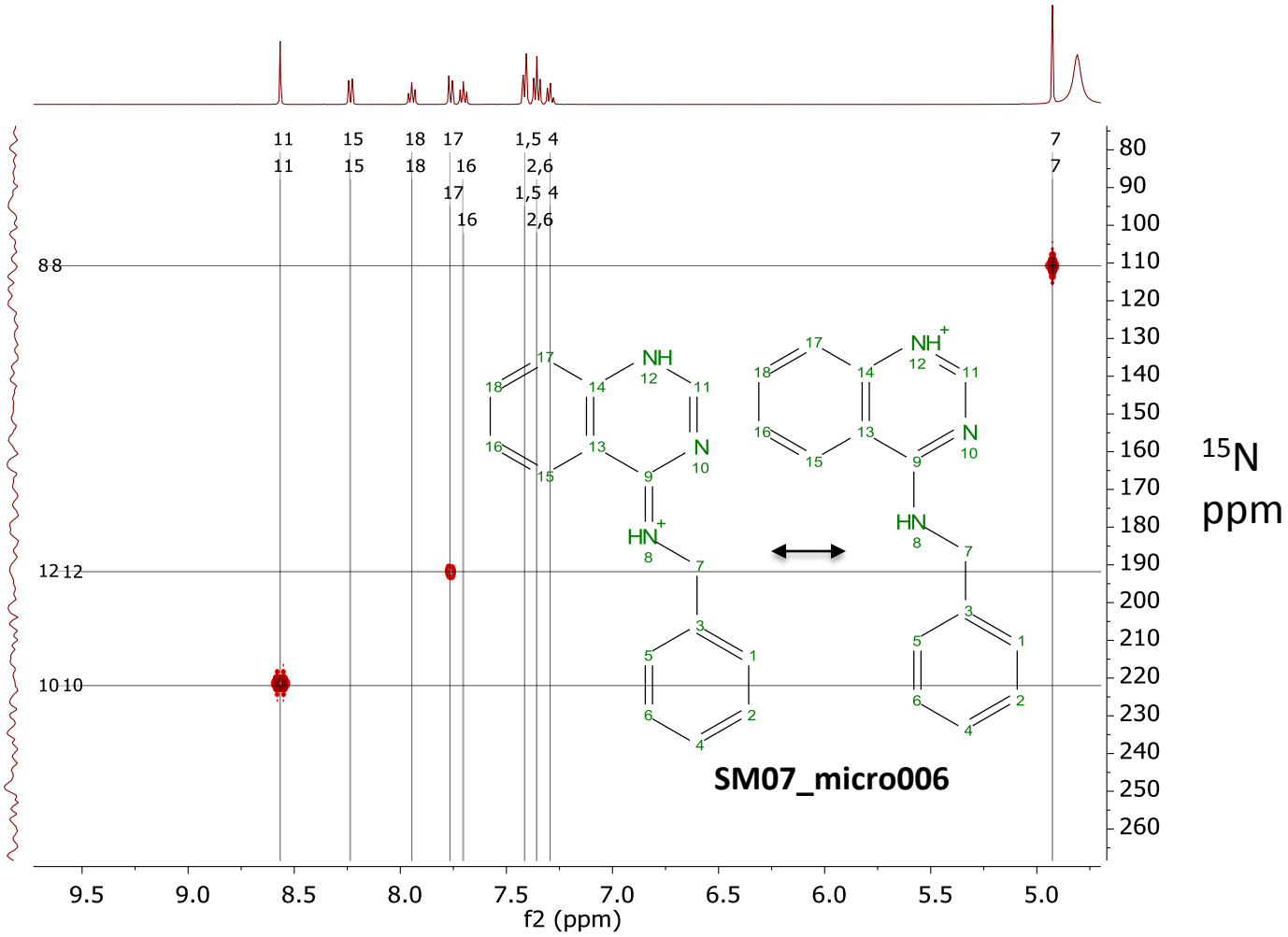
^{15}N HMBC after addition of about 0.5 equivalent of triflouroacetic acid (TFA) to the solution (pH 4.5 – 5.5*).

N-8 = 110.7 ppm
N-10 = 222.0 ppm
N-12 = 191.7 ppm



^{15}N ppm

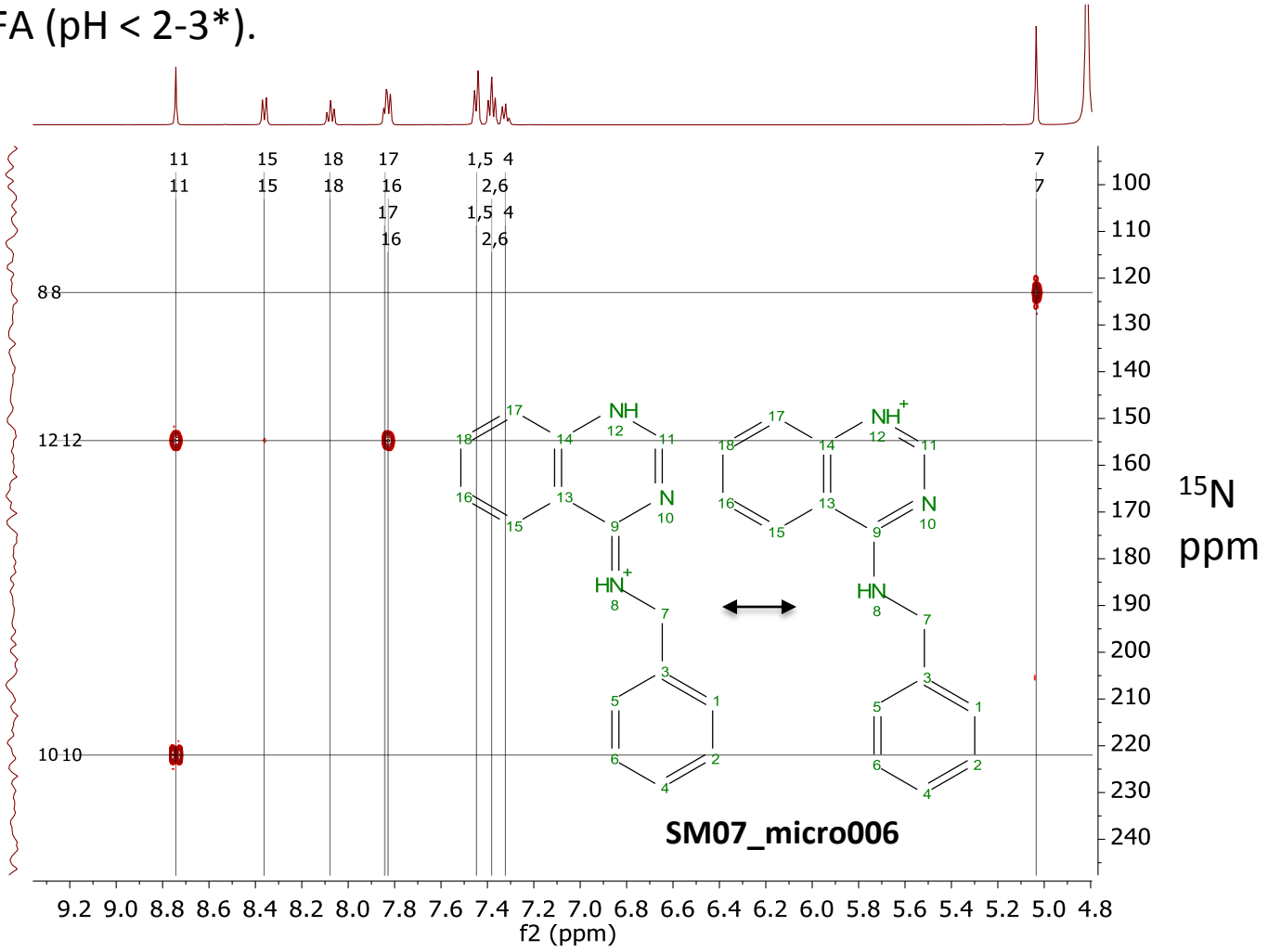
^{1}H ppm

* Measured with pH paper.

NMR Characterization of Microstates of SM07

^{15}N HMBC after addition of about 1 equivalent (less ~5%) of triflouroacetic acid (TFA) to the solution – note that no further change was observed even after addition of ~ 5 equivalents of TFA (pH < 2-3*).

N-8 = 123.1 ppm
N-10 = 222.0 ppm
N-12 = 154.7 ppm



* Measured with pH paper.

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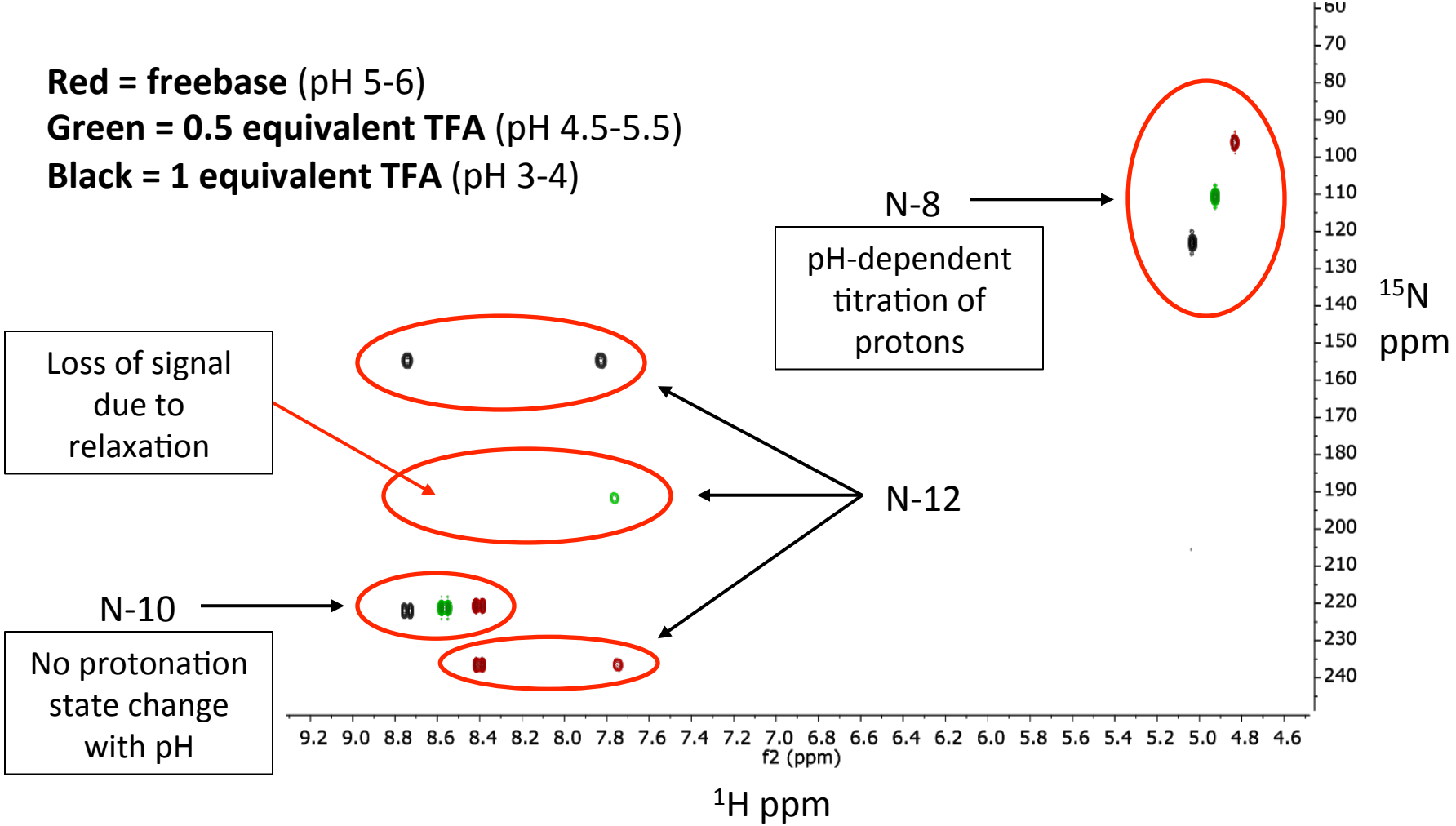
Overlay of ^{15}N HMBC at free-base, 0.5 equivalent and 1 equivalent of TFA. Notice that the chemical shifts of N-8 and N-12 in the 0.5 TFA equivalent solution are at the midpoint between the freebase and the 1 equivalent solution.

Red = freebase (pH 5-6)
Green = 0.5 equivalent TFA (pH 4.5-5.5)
Black = 1 equivalent TFA (pH 3-4)



* Solution pH values were measured with pH paper.