

C8953

NMR structural analysis - seminar

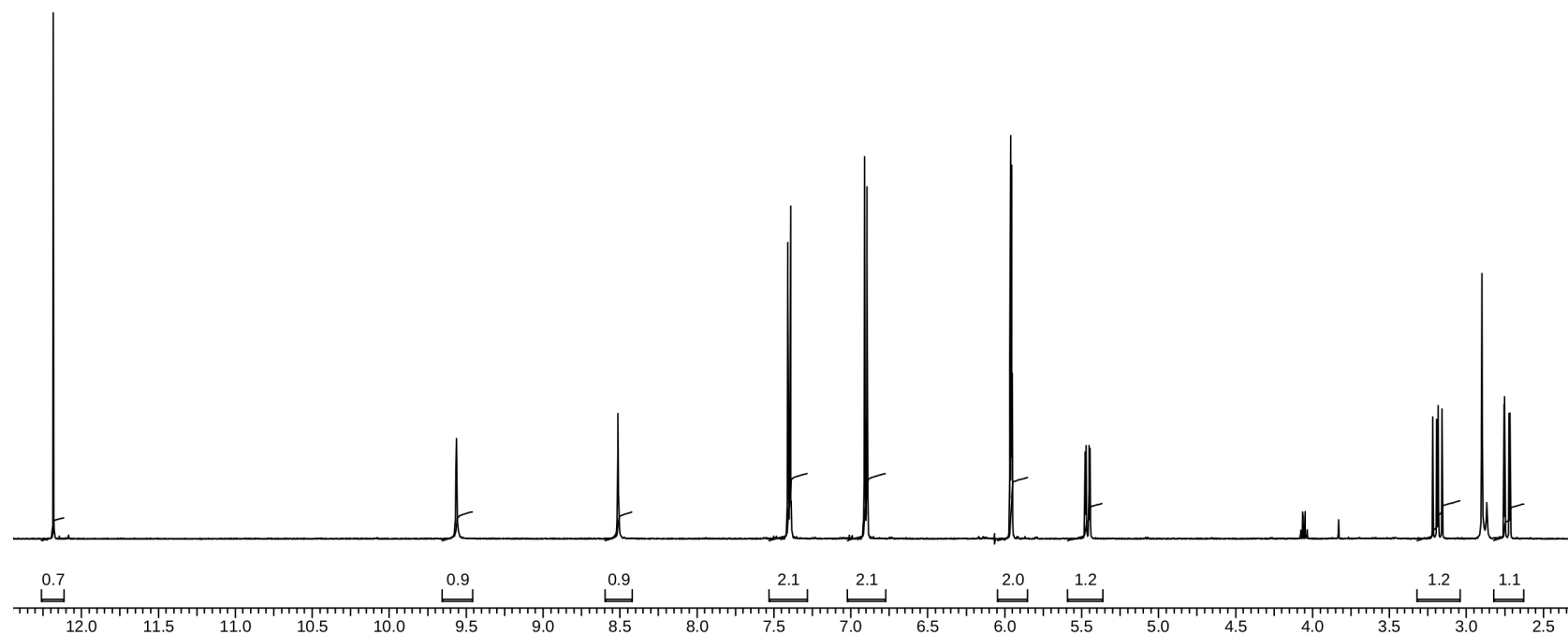
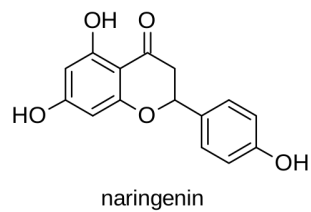
^1H - ^{13}C APT + 2D NMR spectra, COSY

Jan Novotný

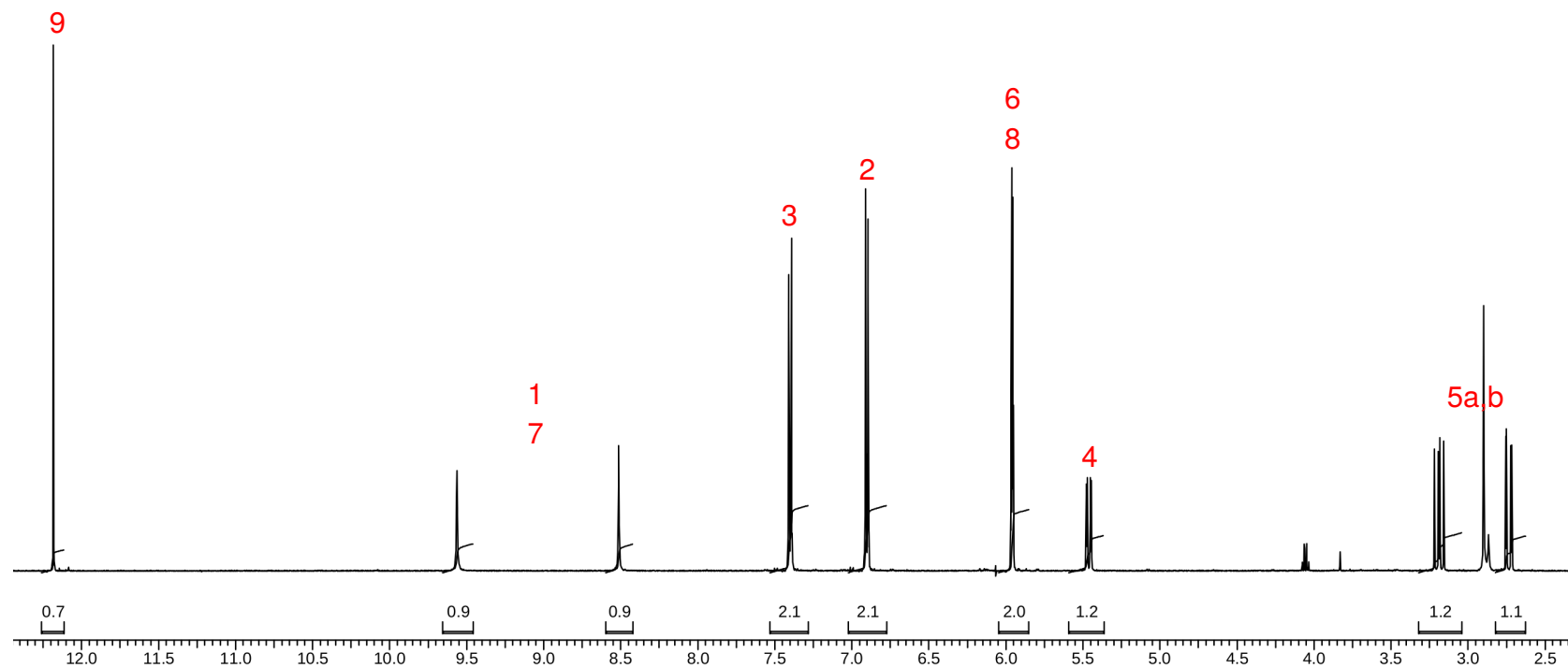
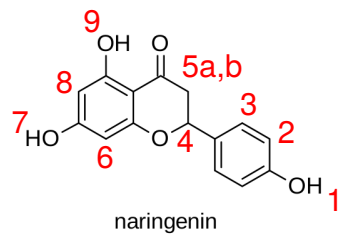
novotnyjan@mail.muni.cz

March 11, 2020

^1H NMR spectrum of naringenin in d_6 -acetone

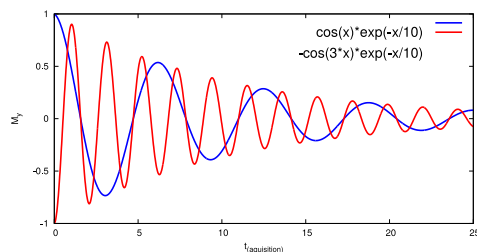
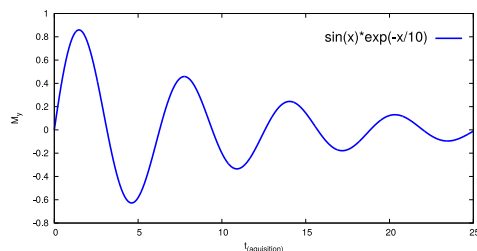
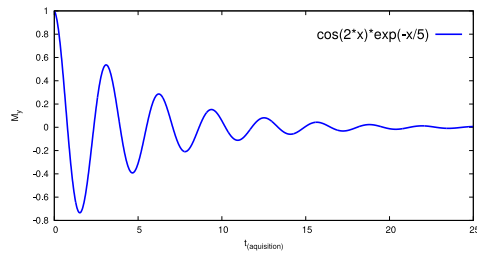
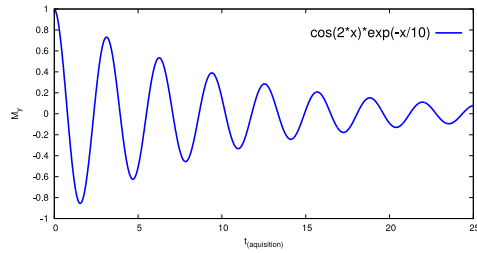
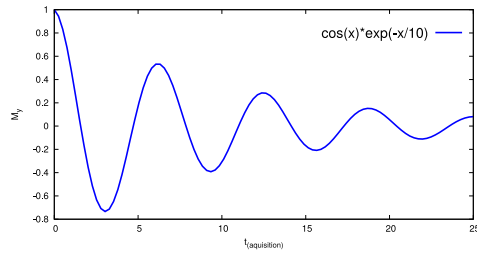


^1H NMR spectrum of naringenin in d_6 -acetone

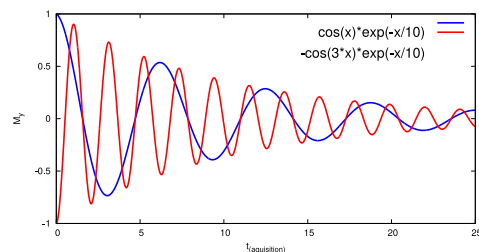
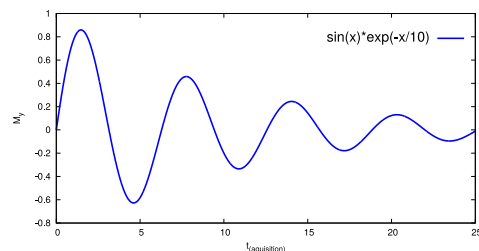
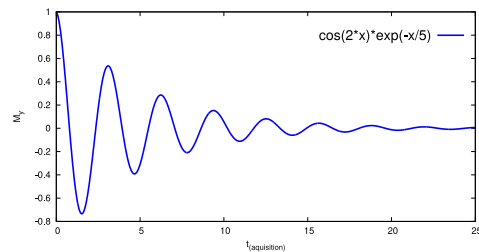
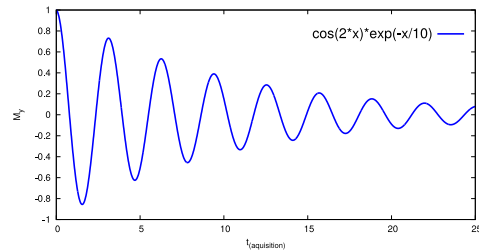
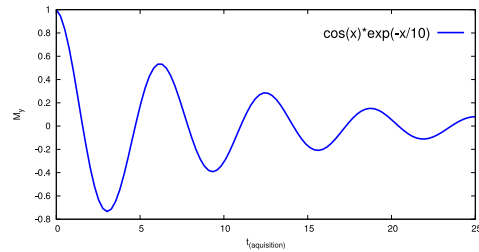


Basics of 1D FT spectroscopy

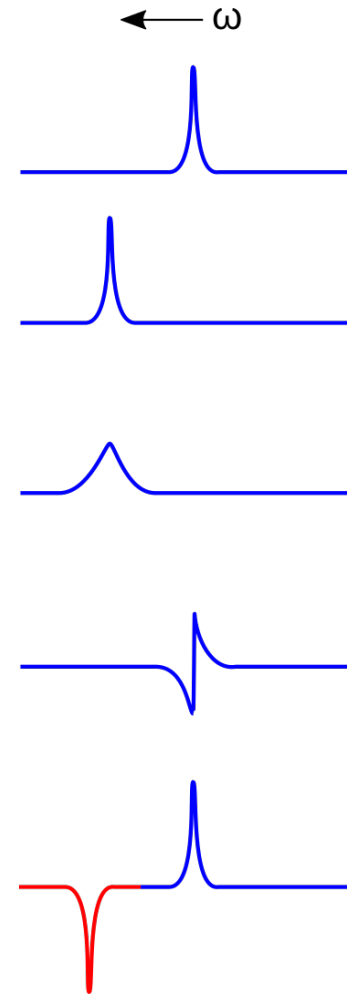
Draw FT representation of attached FID records (reciever is located in the $+y$ direction):



Basics of 1D FT spectroscopy



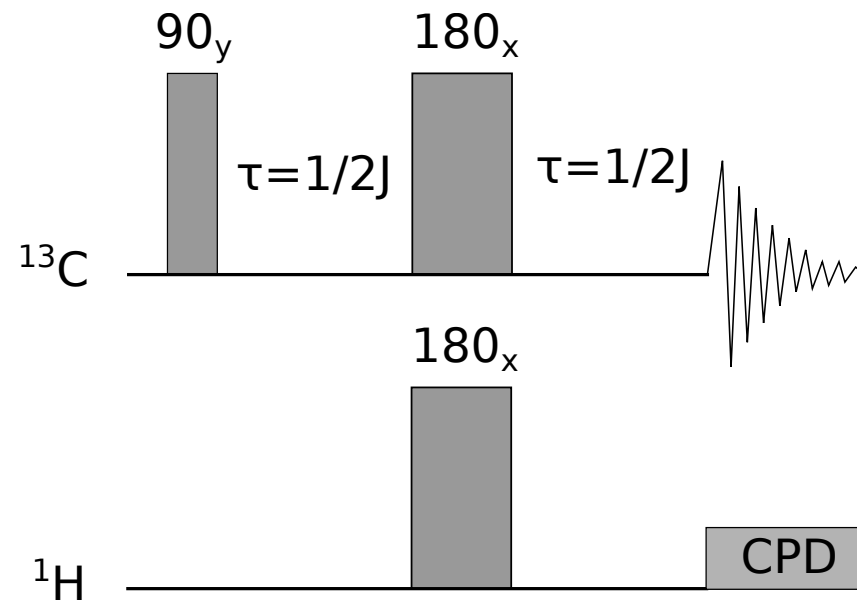
Draw FT representation of attached FID records (receiver is located in the $+y$ direction):



Heteronuclear spin echo

By using vector diagrams determine the result of attached pulse sequence.

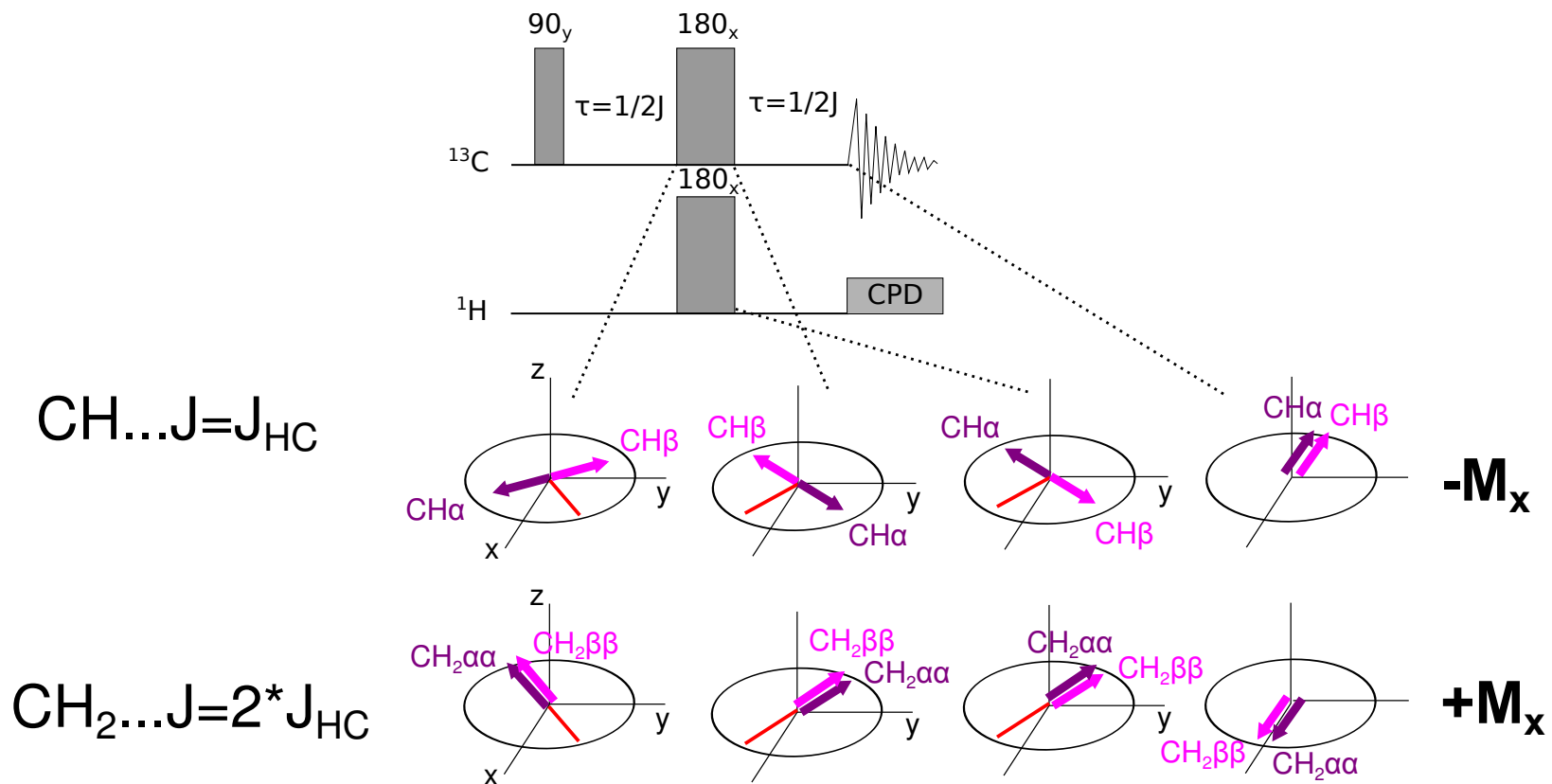
Lets consider isolated spin systems **a)** $^{13}\text{C}-^1\text{H}$ and **b)** $^{13}\text{C}-^1\text{H}_2$.



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APT - Attached Proton Test

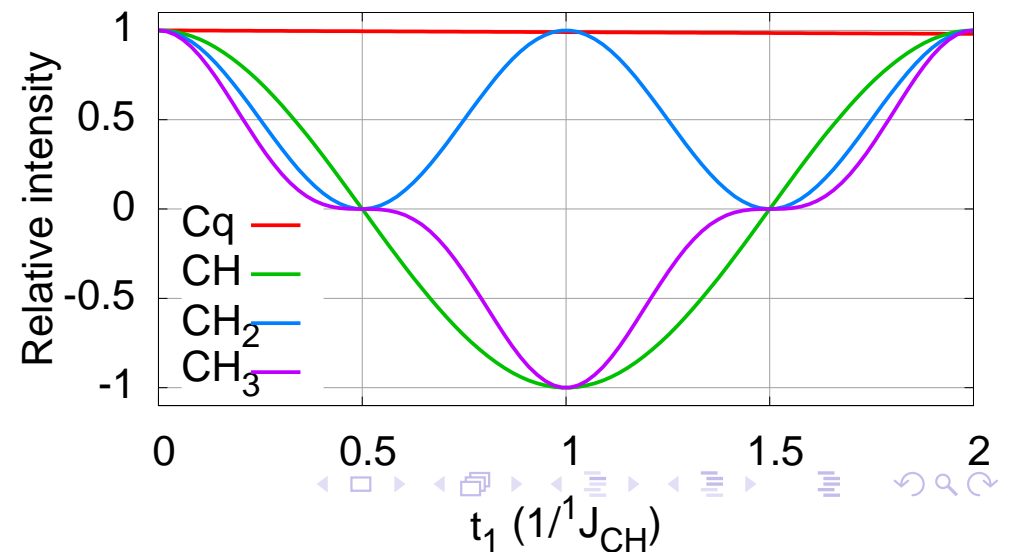
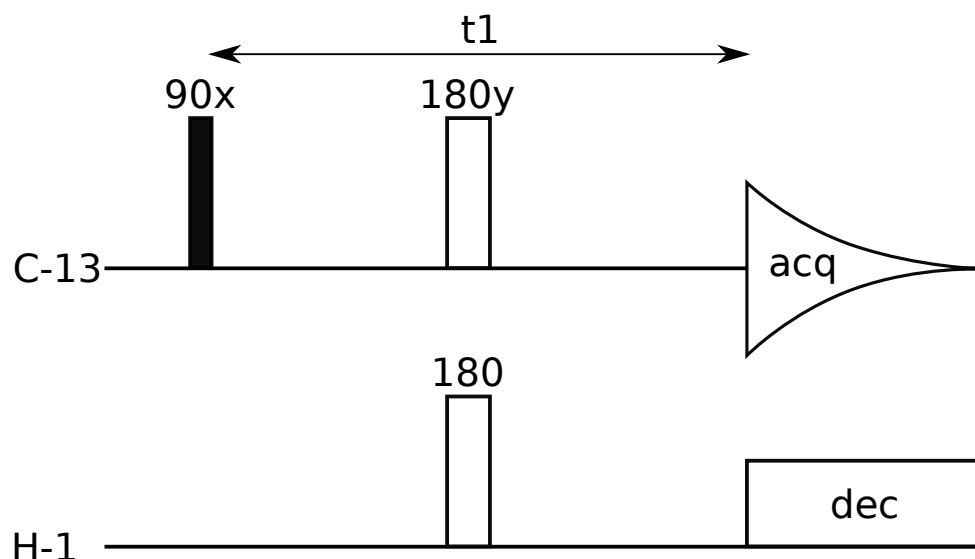
based on heteronuclear spin echo

▶ $t_1 = 1/{}^1J_{CH}$

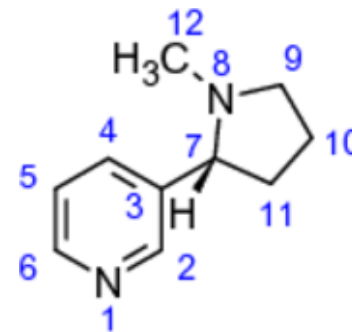
phase of signals distinguished according to numbers of directly attached protons

- ▶ Cq, CH₂ positive
- ▶ CH, CH₃ negative

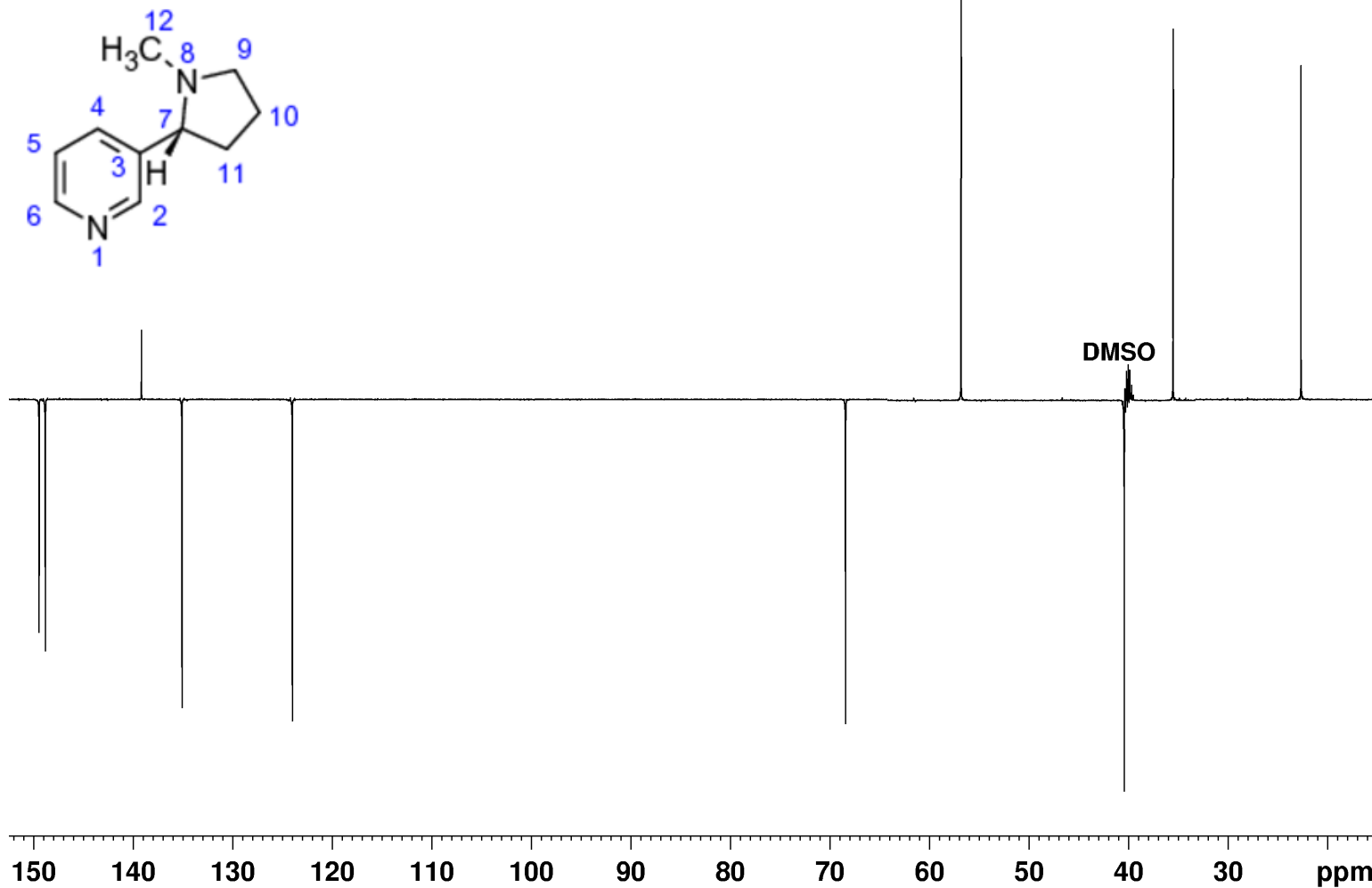
Different ${}^1J_{CH} \implies$ different intensities



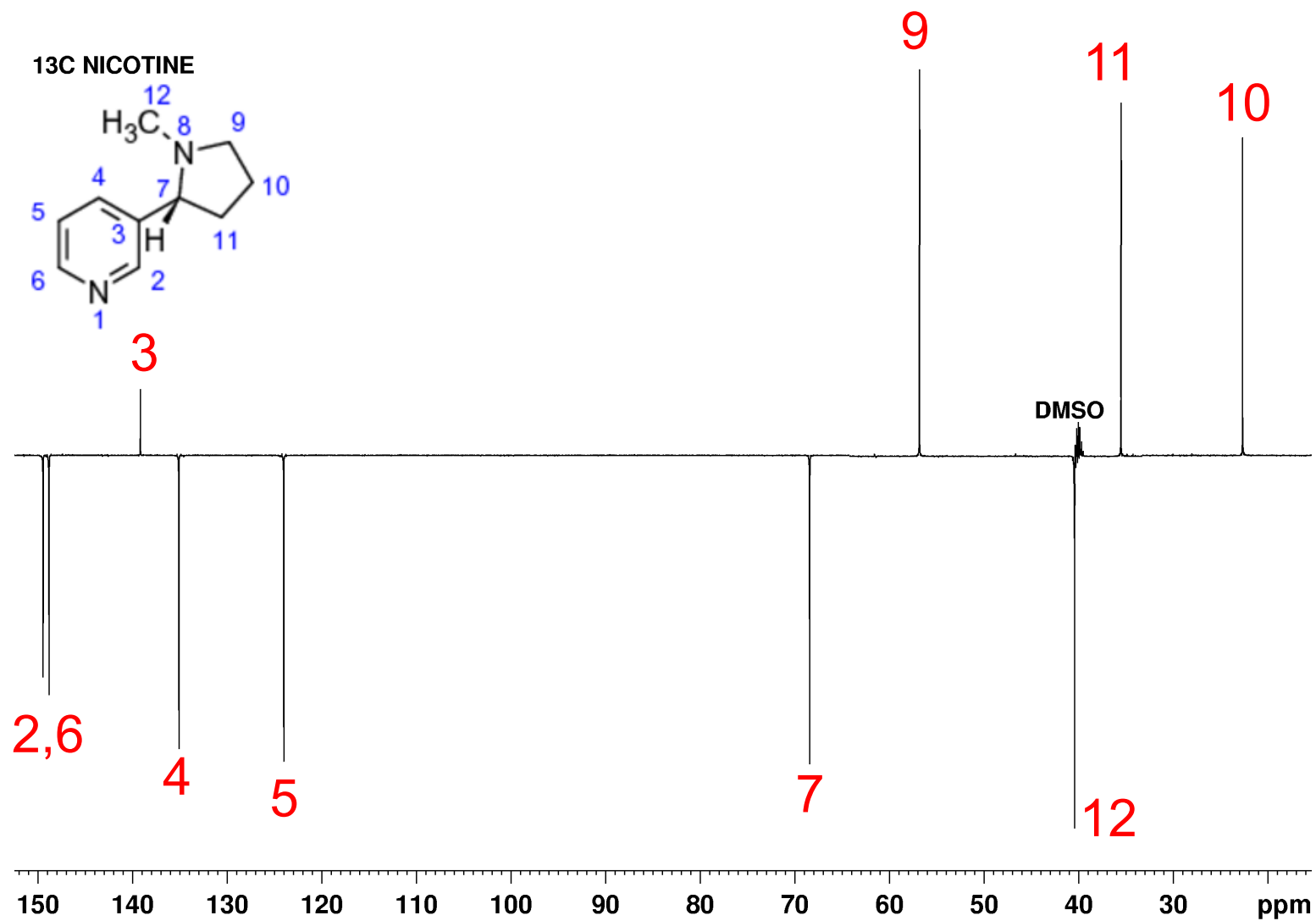
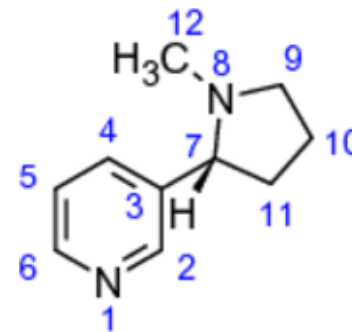
^{13}C APT Nicotine



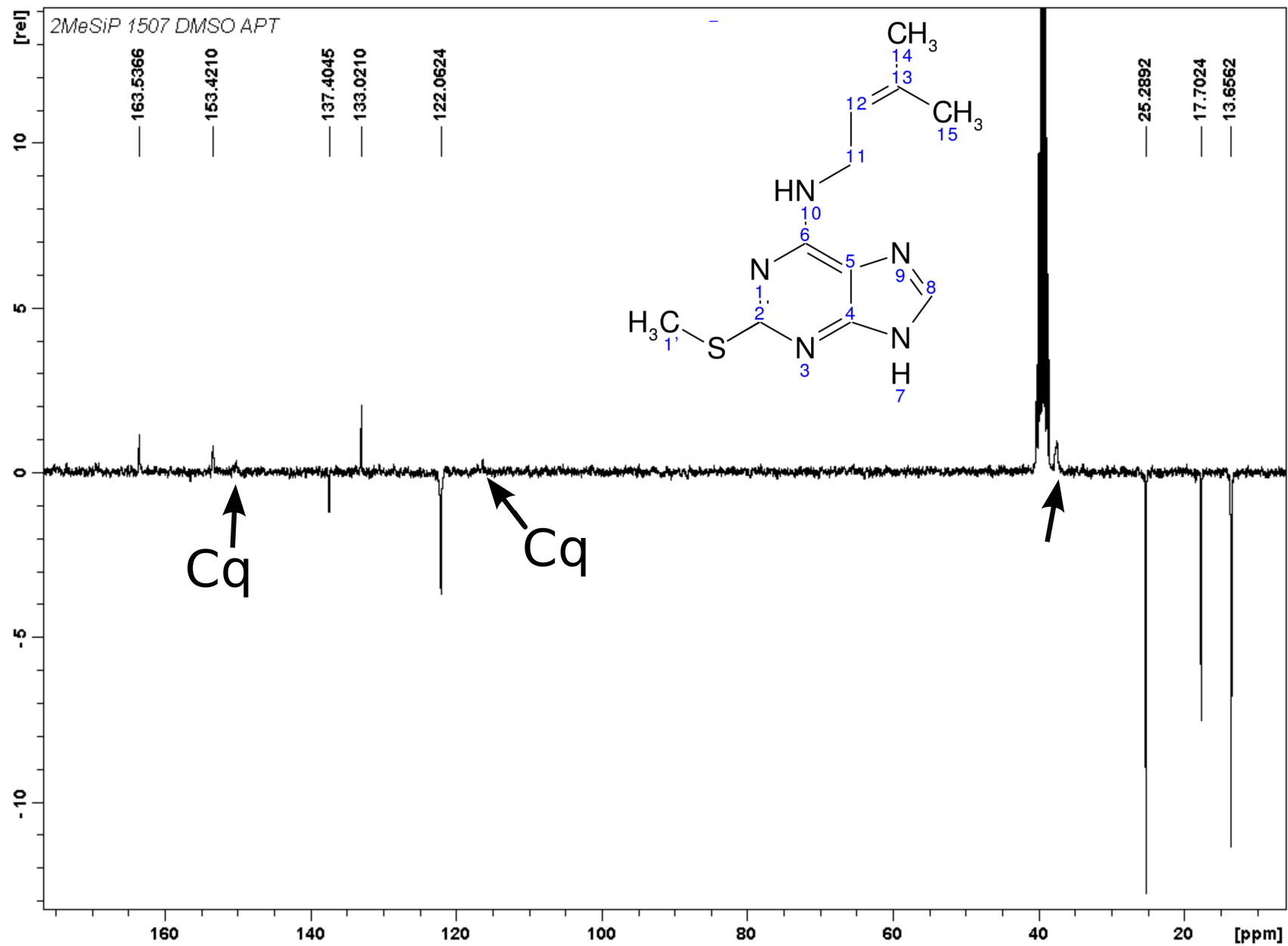
^{13}C NICOTINE



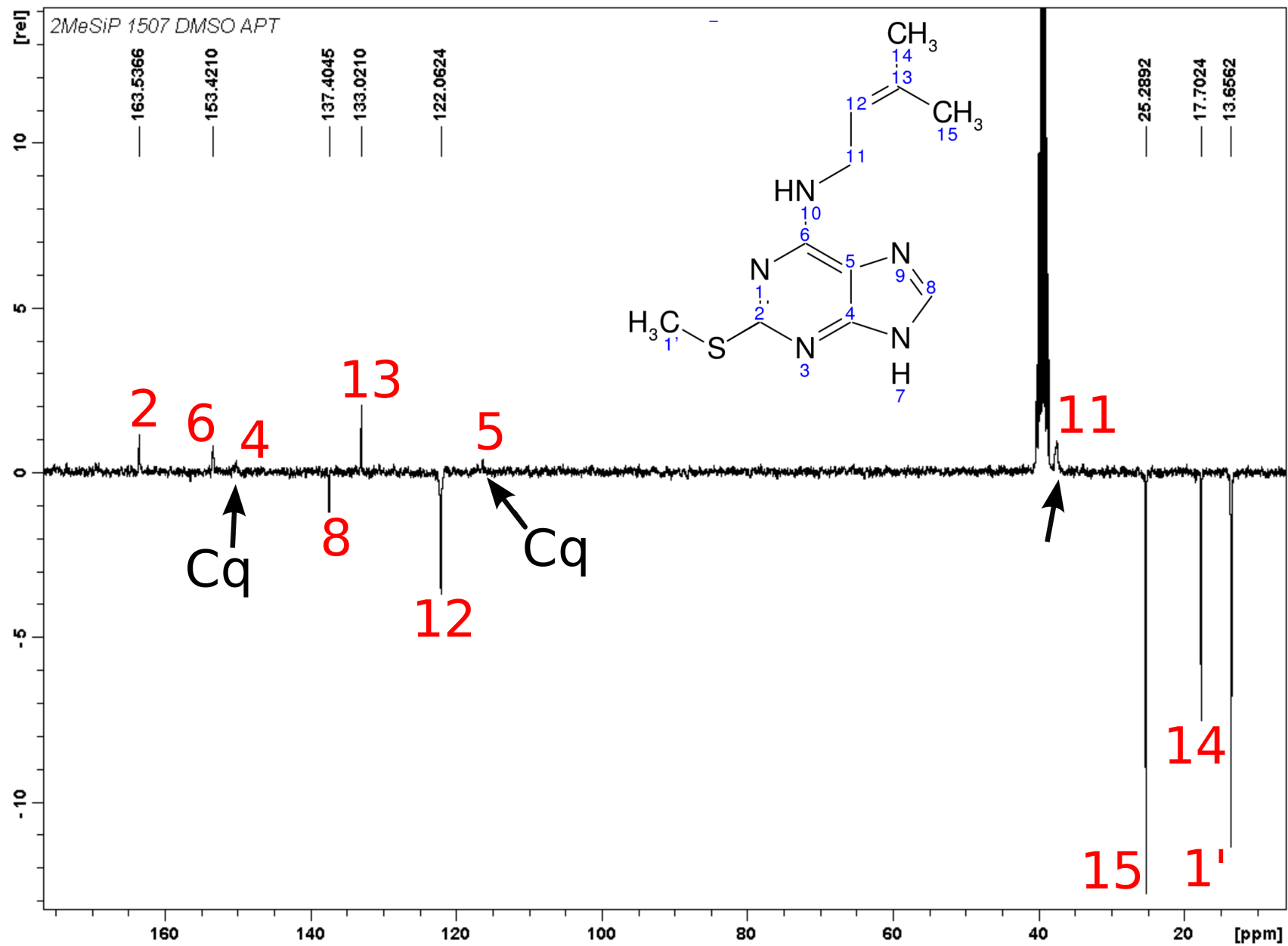
^{13}C APT Nicotine



^{13}C APT 4



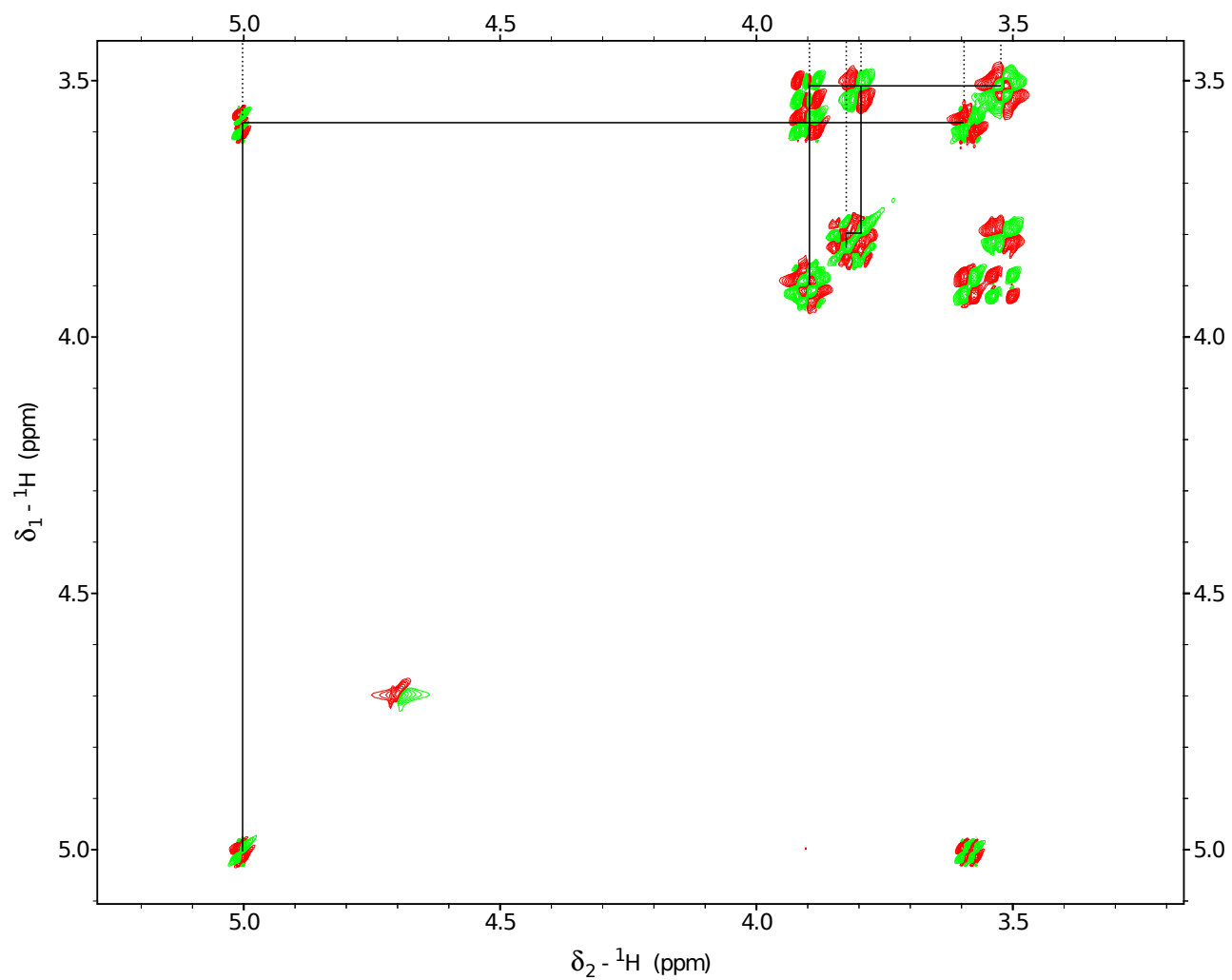
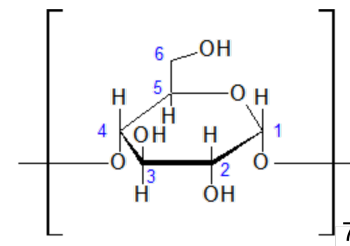
^{13}C APT 4



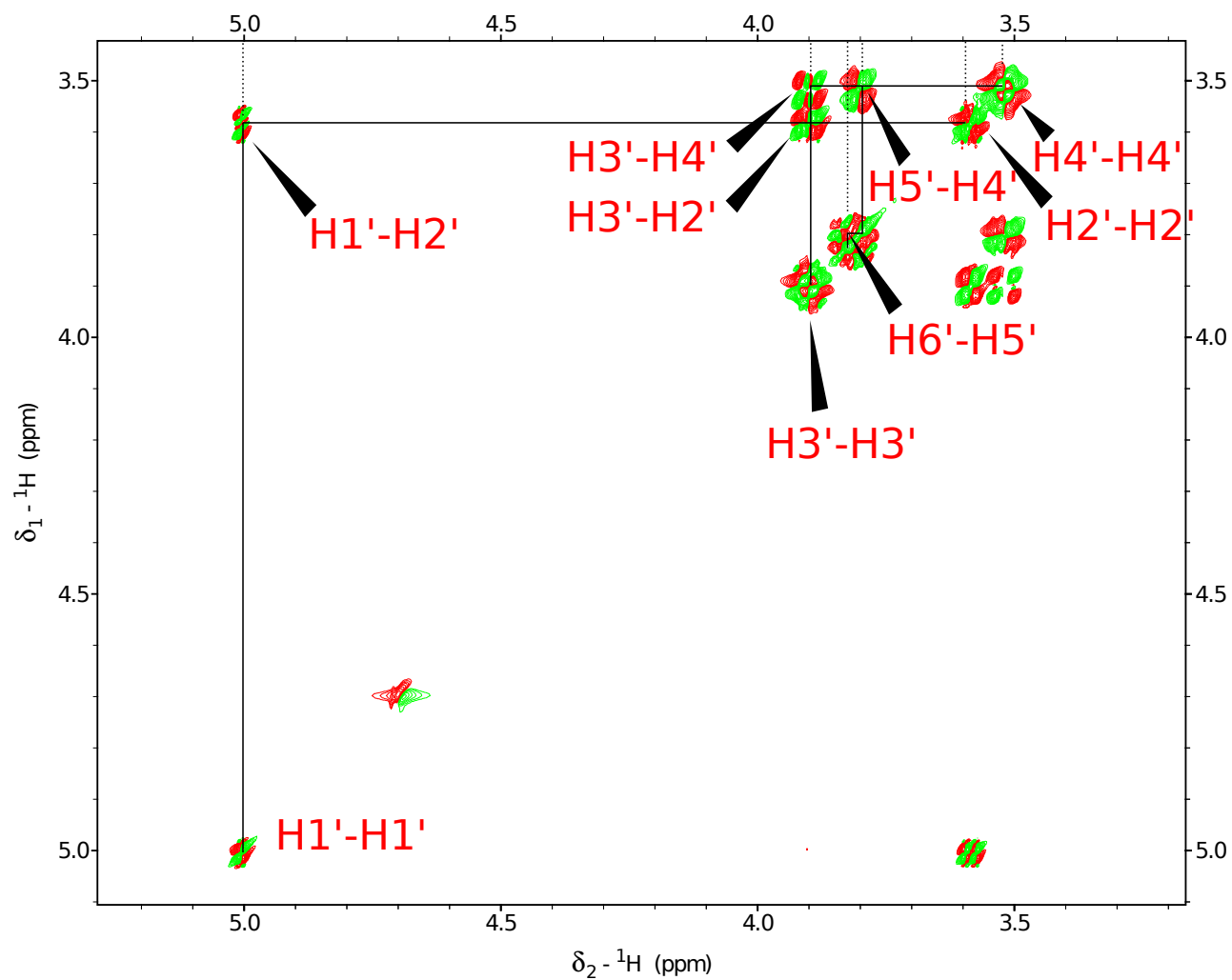
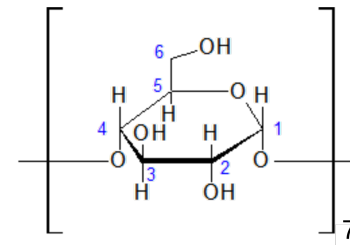
Hints for beginners

- ▶ Determination of **individual spin systems** - sharing **off-diagonal crosspeaks**
- ▶ Isolated protons - only diagonal crosspeak
- ▶ Already known rules: symmetry, diastereotopicity, most shielded/deshielded atoms etc.

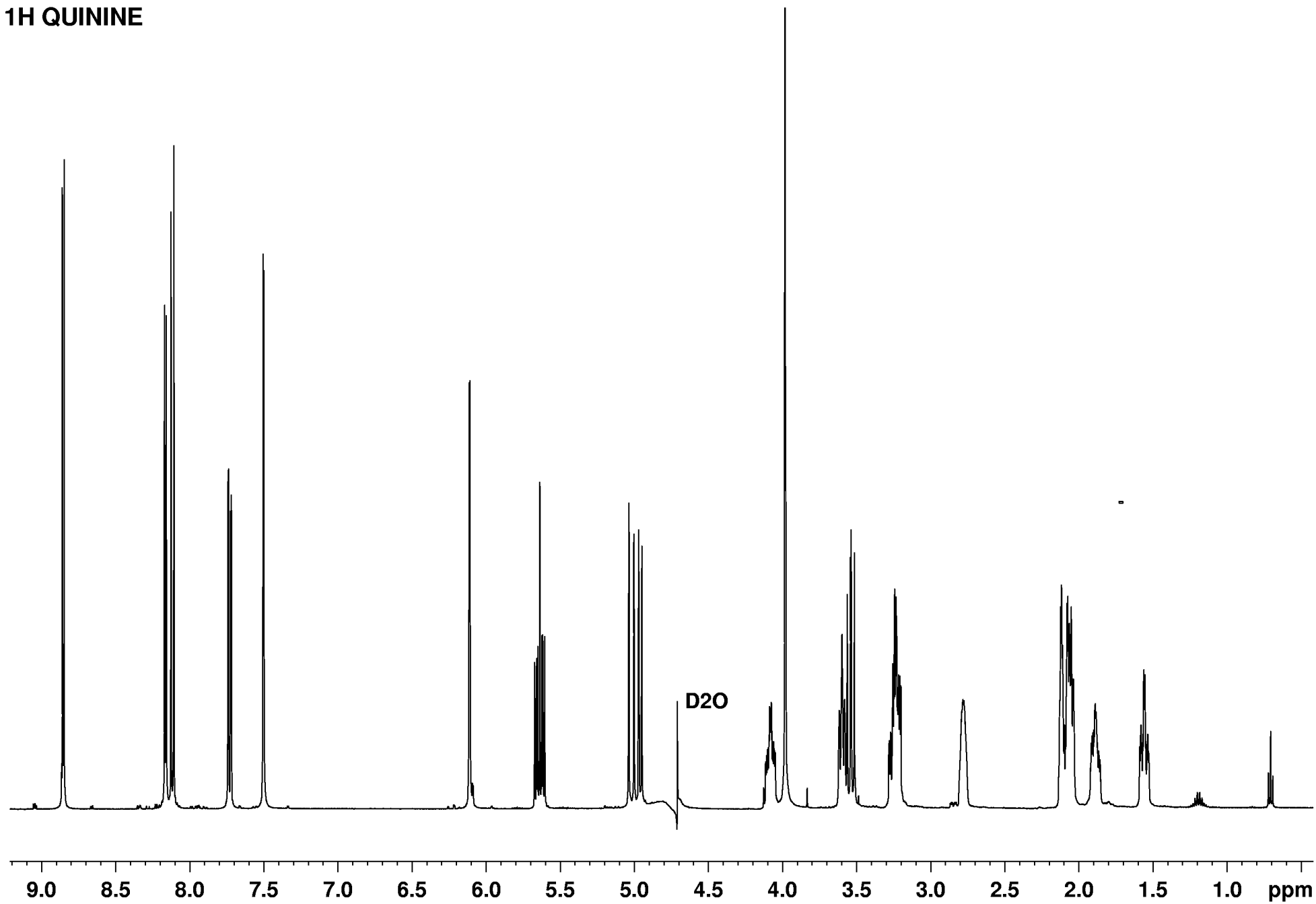
COSY : β -cyclodextrine



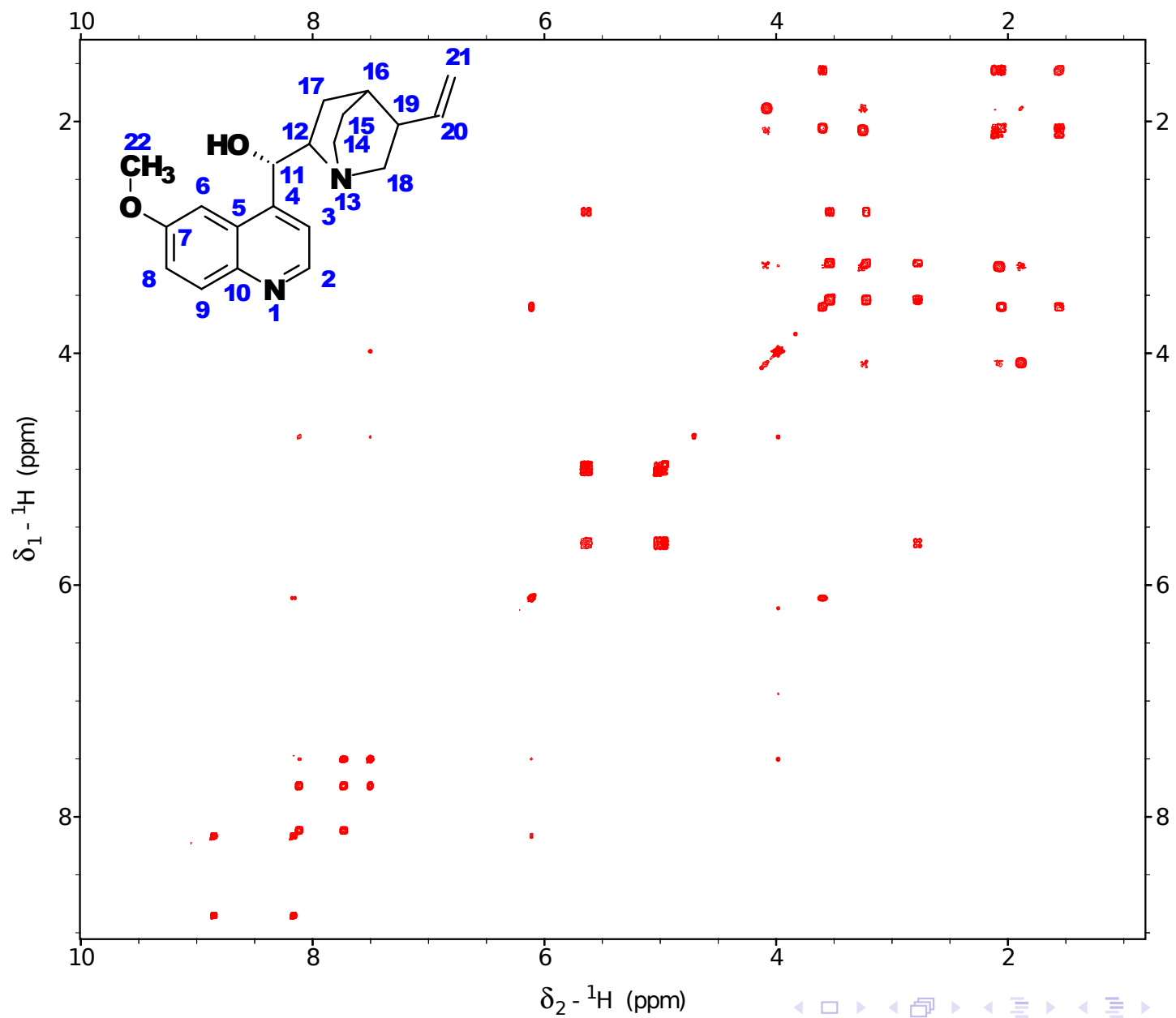
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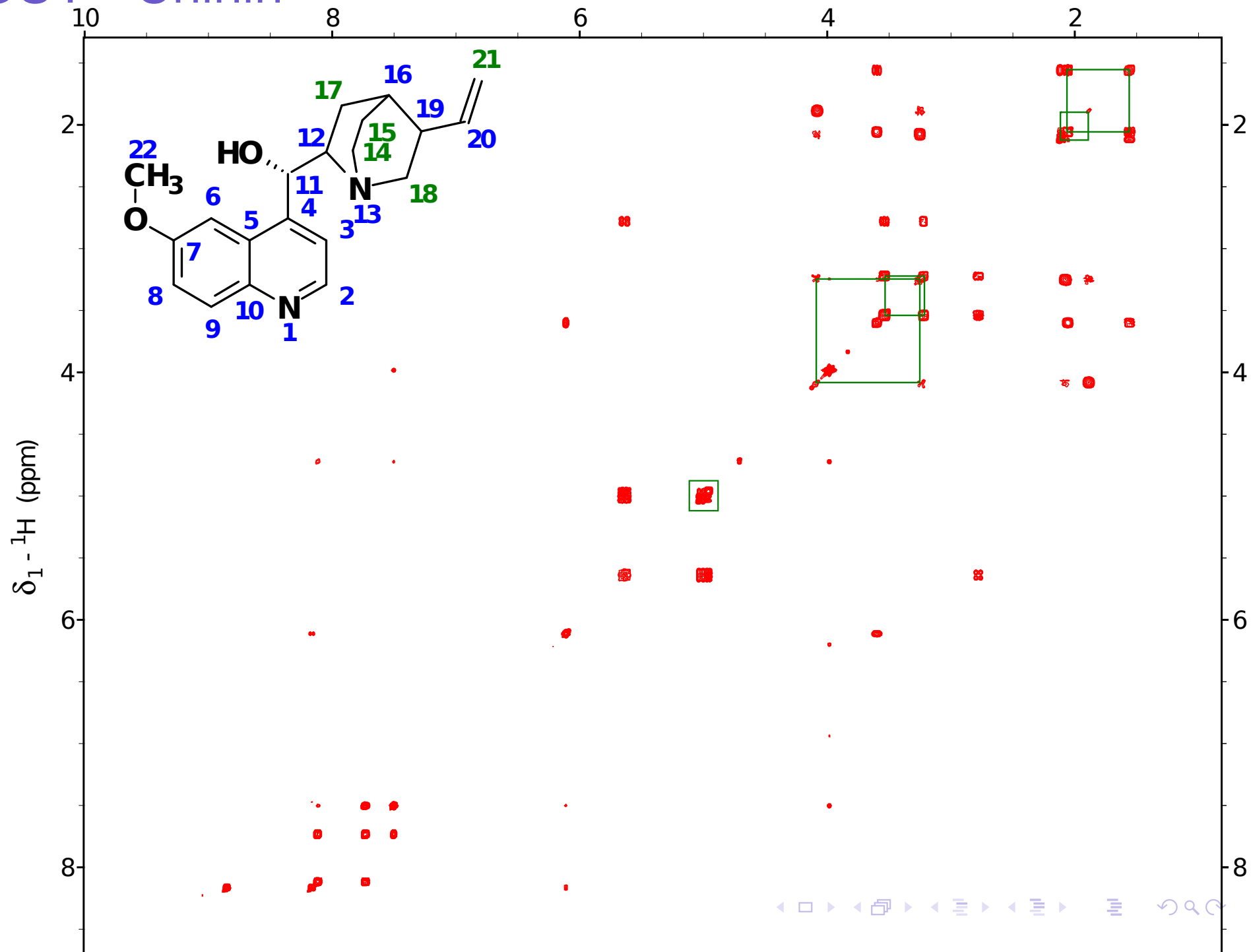
1H QUININE



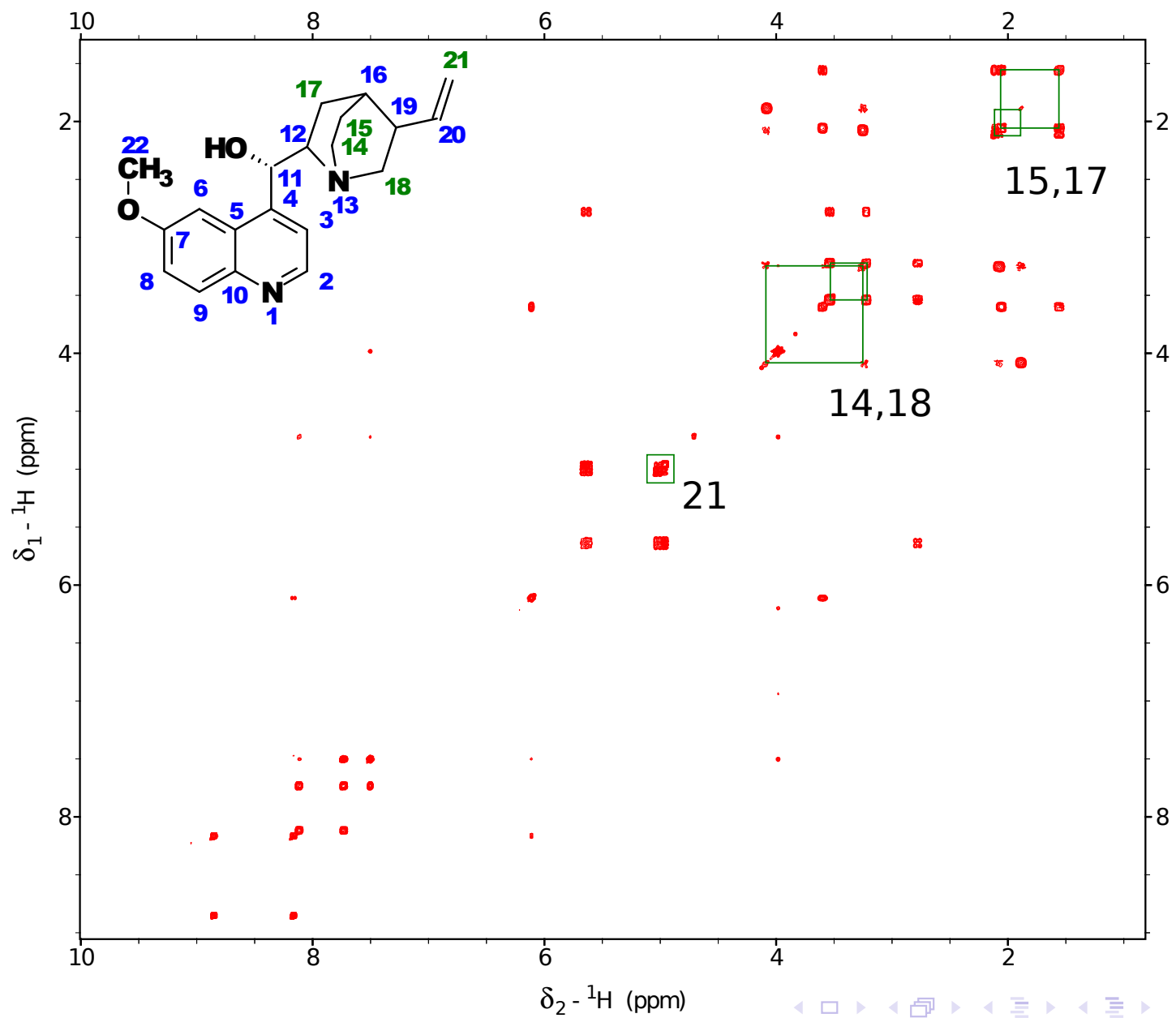
COSY - Chinin



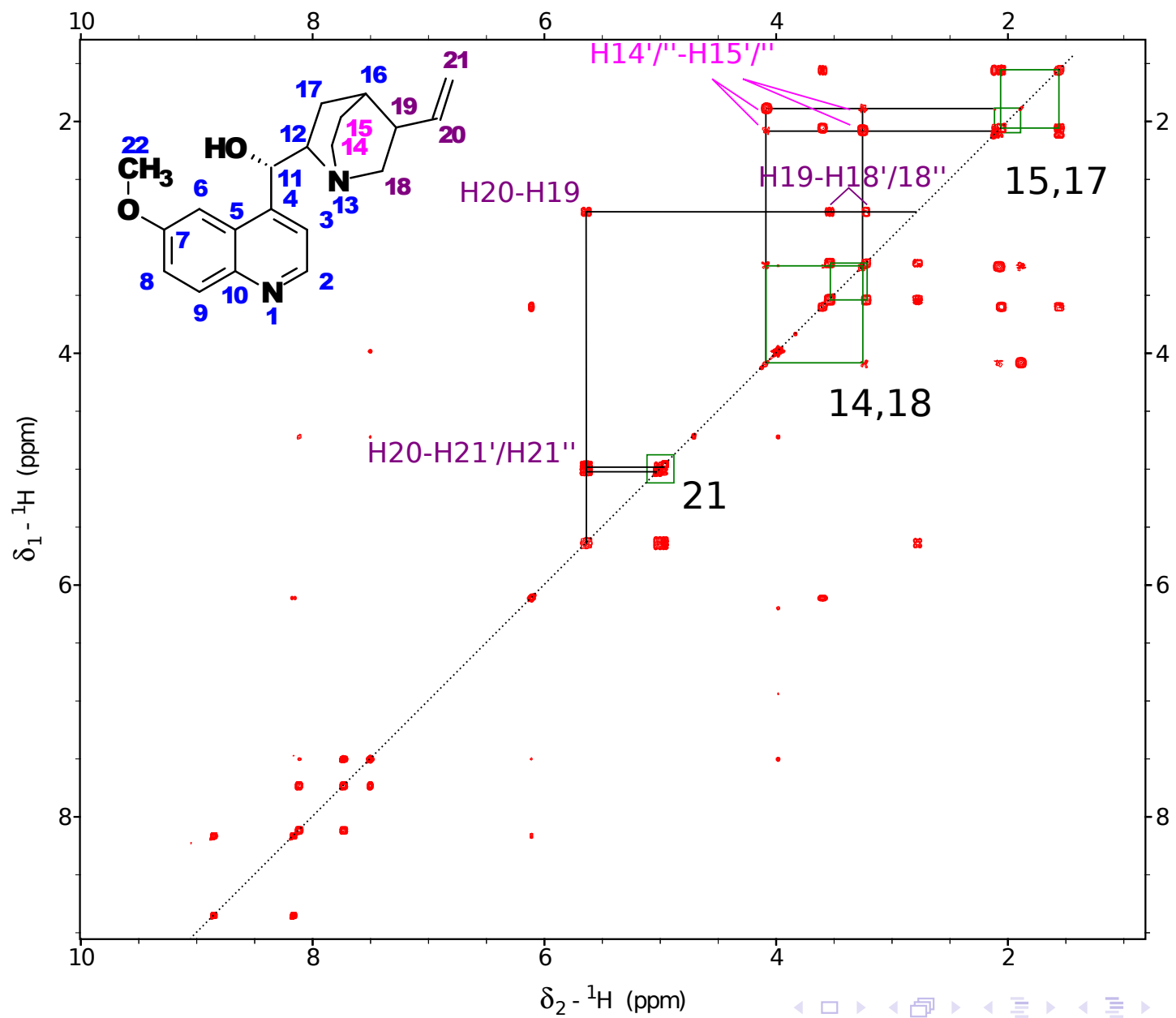
COSY - Chinin



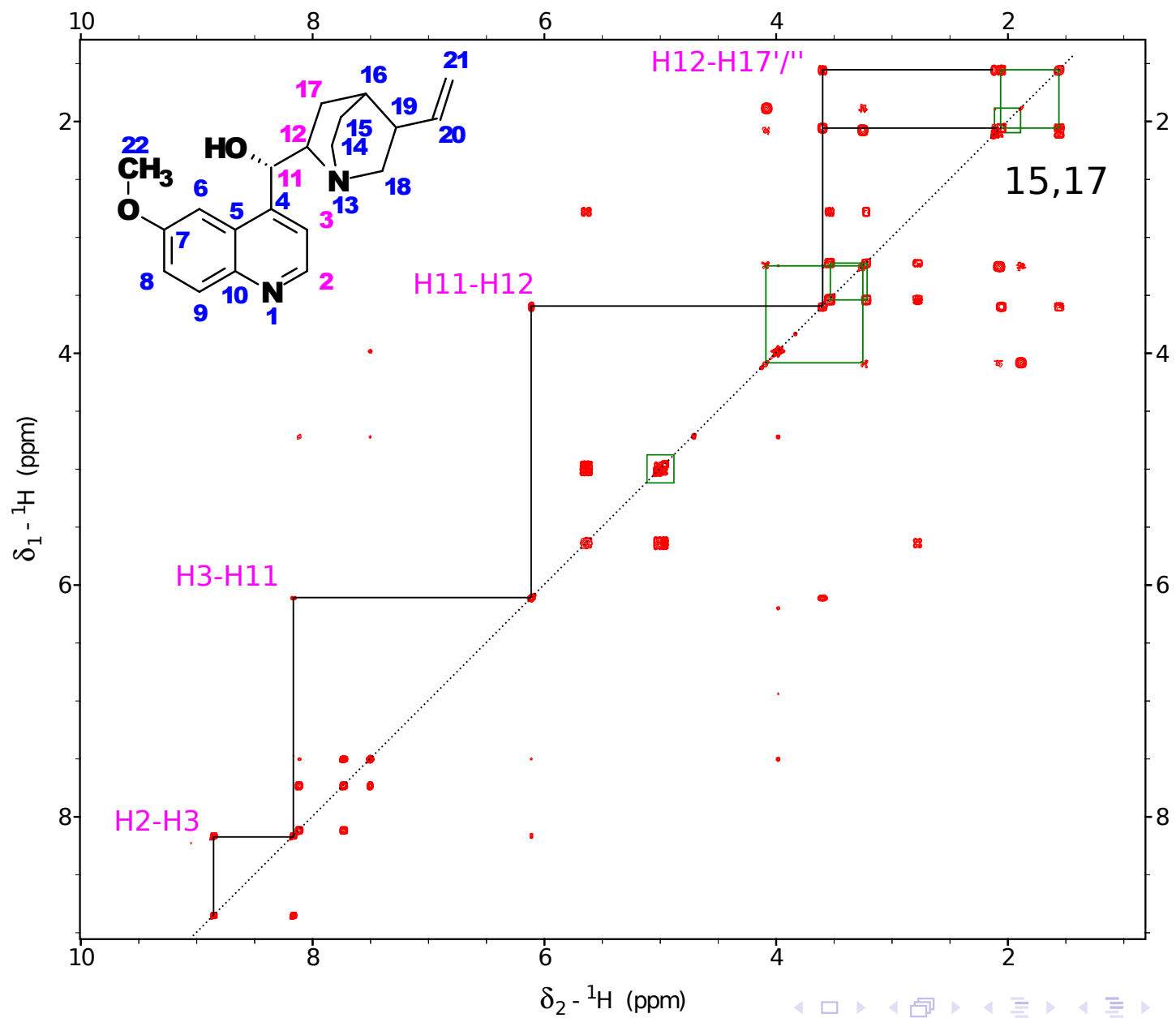
COSY - Chinin



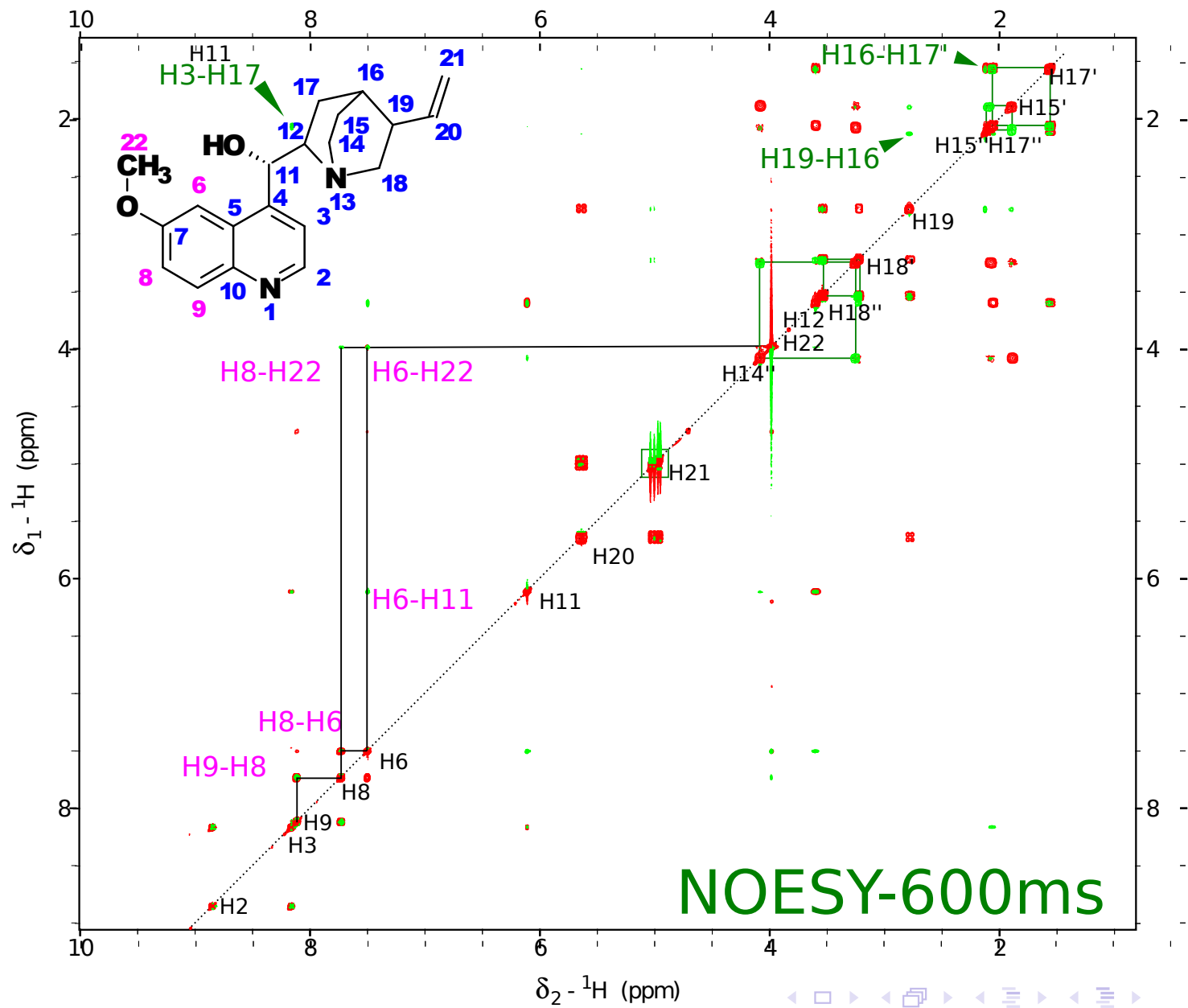
COSY - Chinin



COSY - Chinin



COSY - Chinin



Next topic

^1H - ^1H correlations (NOESY, ROESY, TOCSY)