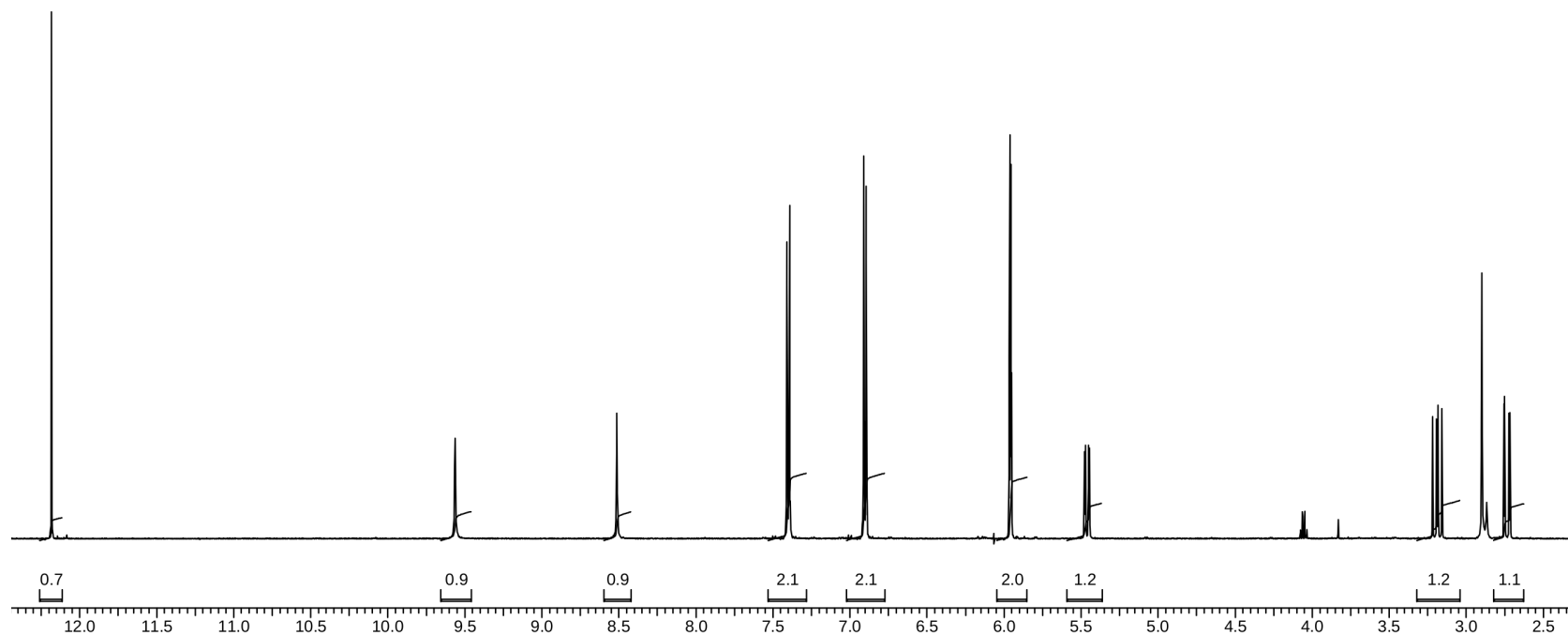
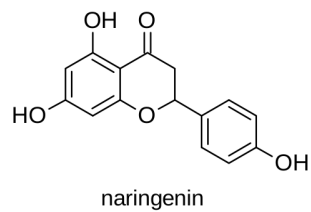


C8953
NMR structural analysis - seminar
2D NMR spectra, COSY

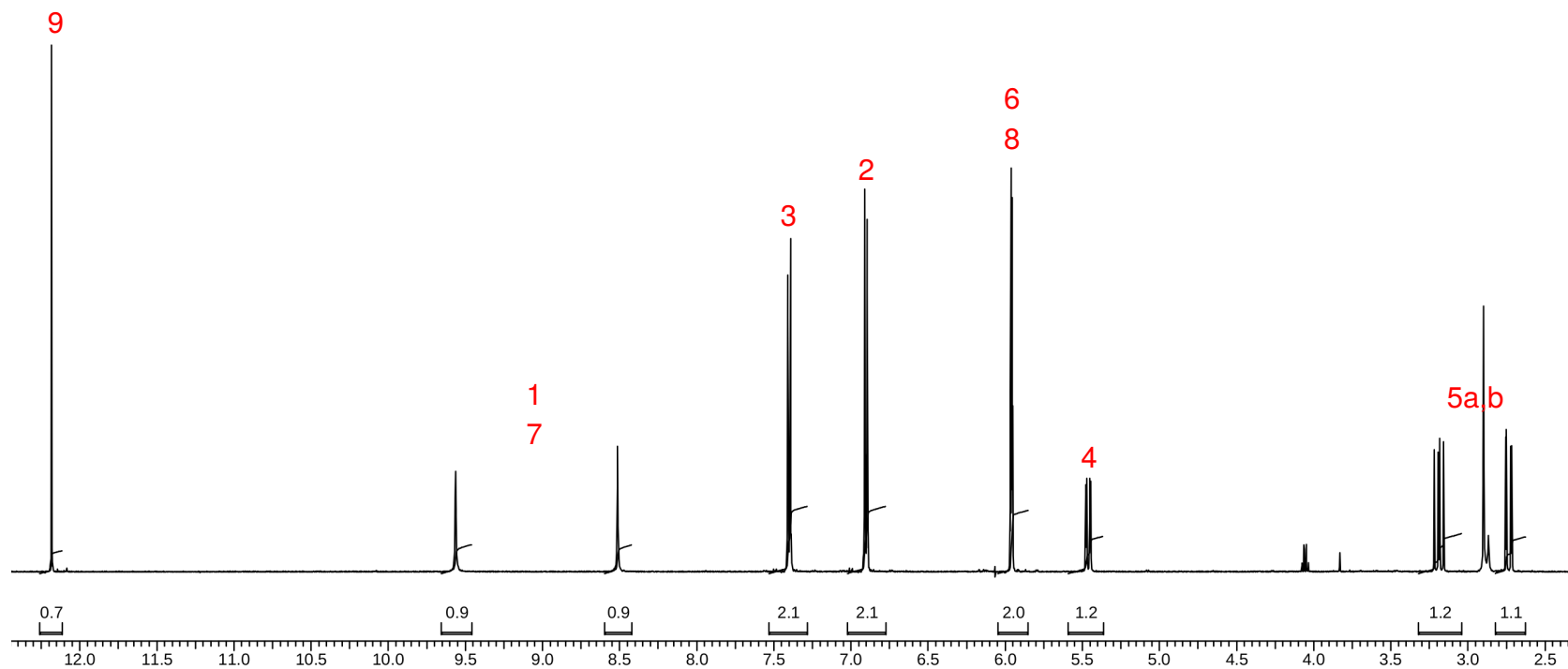
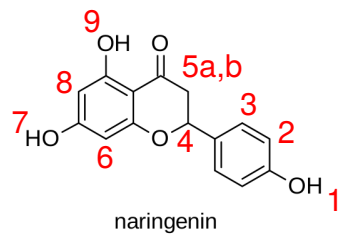
Jan Novotny
176003@mail.muni.cz

March 16, 2022

^1H NMR spectrum of naringenin in d_6 -acetone



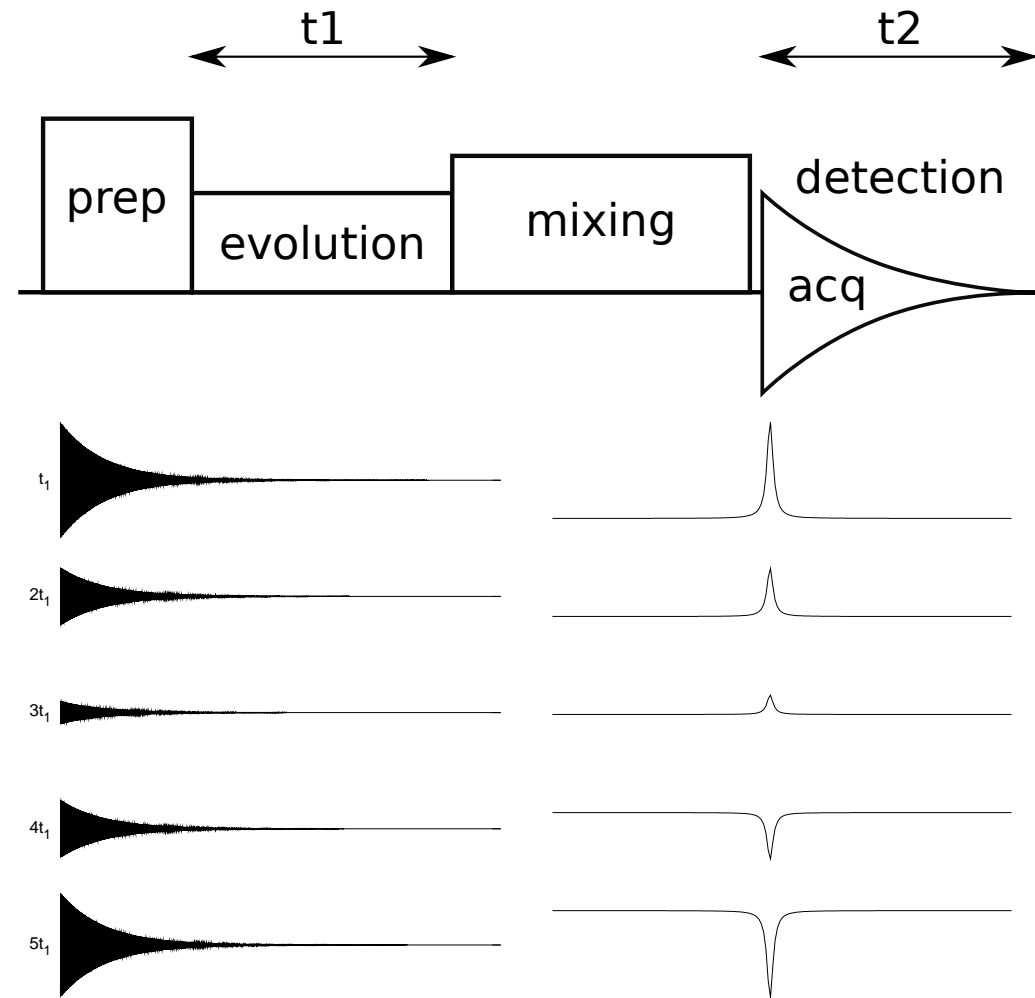
^1H NMR spectrum of naringenin in d_6 -acetone



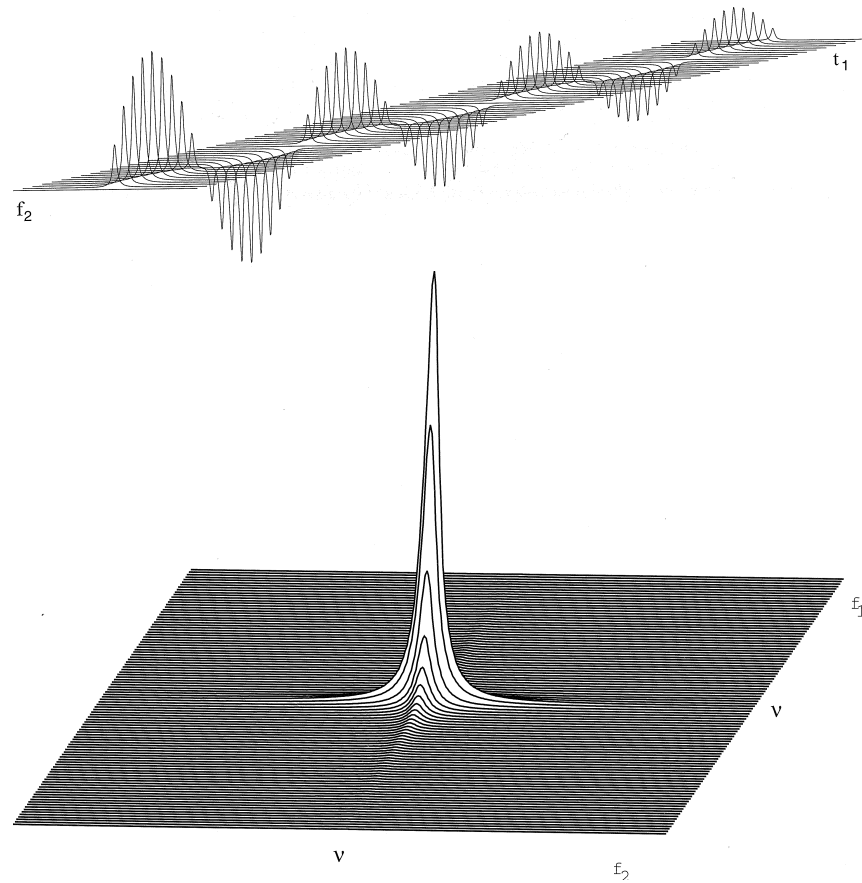
2D NMR

Second dimension f_1

- ▶ preparation period
⇒ coherence
- ▶ evolution period
 $t_1 \xrightarrow{\text{FT}} f_1$
 - ▶ increments
 - ▶ evolution of coherence
- ▶ mixing period
 - ▶ transfer of encoded magnetisation
 - ▶ measurable signal
- ▶ detection of signal
 $t_2 \xrightarrow{\text{FT}} f_2$



2D NMR

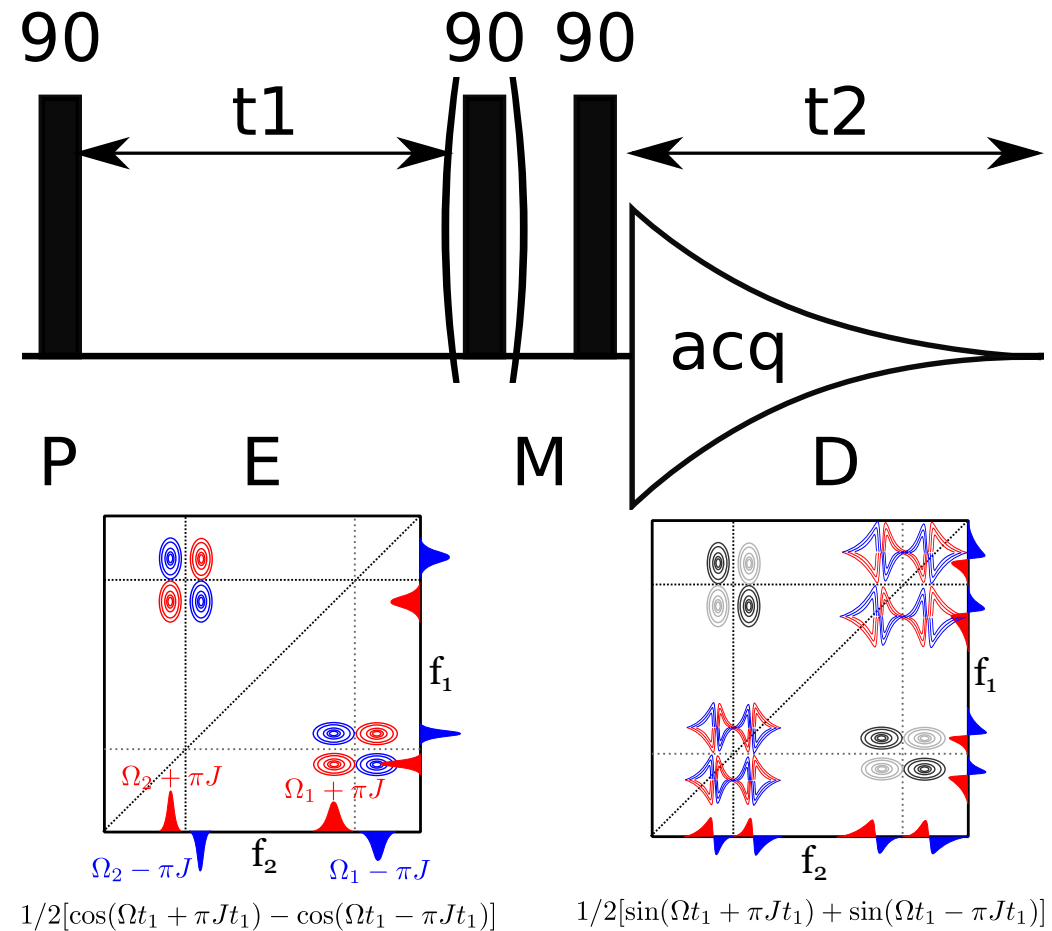


2D spektrum

- ▶ FT in t_1 - modulated 1D spectra
- ▶ FT in t_2 - 2D spectrum

COSY

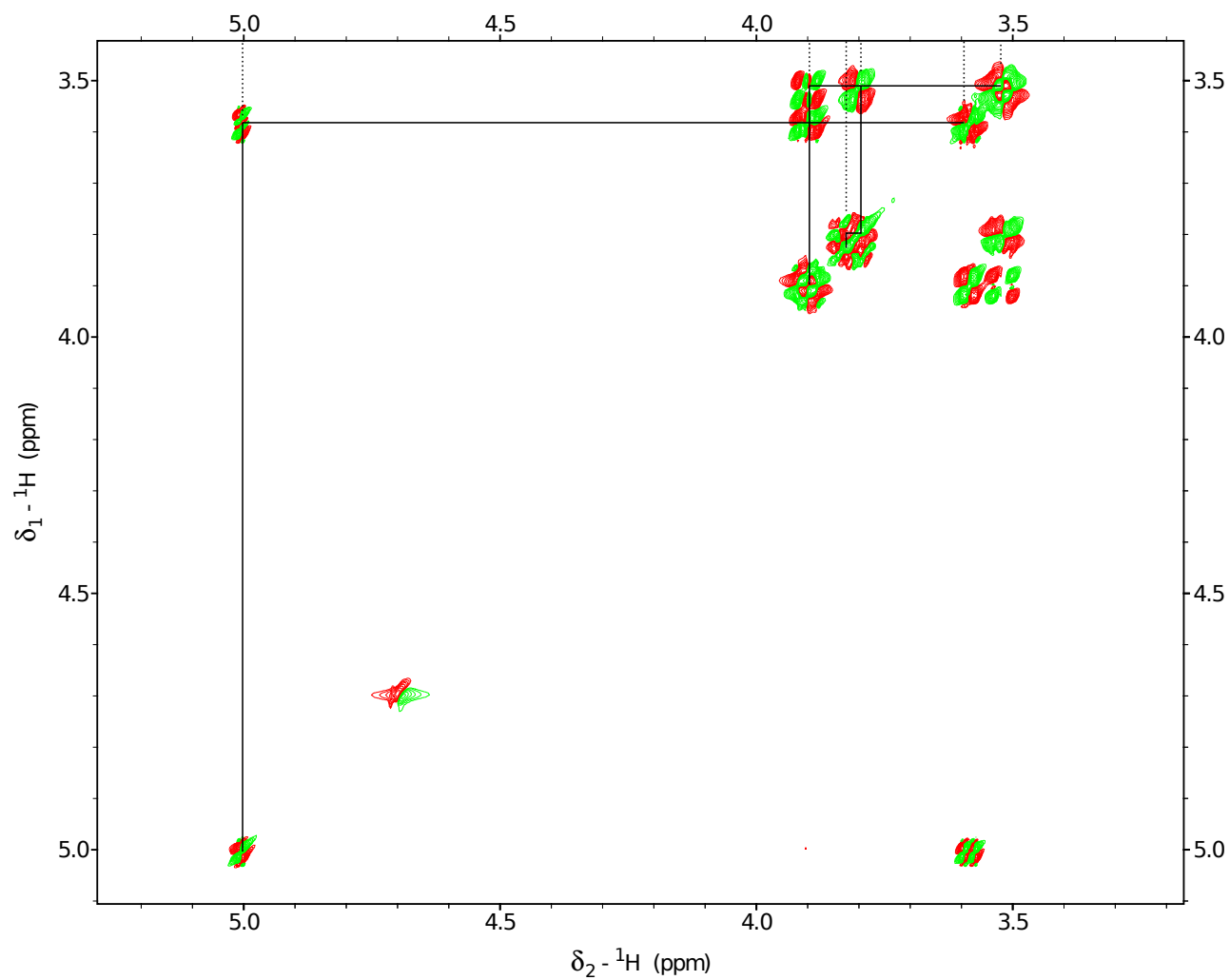
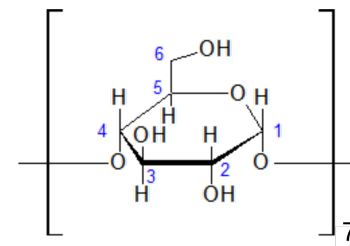
- ▶ easiest 2D experiment
- ▶ **correlates H nuclei based on $^{2/3}J$ coupling**
- ▶ through 2, 3, (4) bonds
- ▶ antiphase off-diagonal crosspeak between coupled atoms
- ▶ DQF-COSY - modification of basic sequence, diagonal crosspeaks in absorption phase



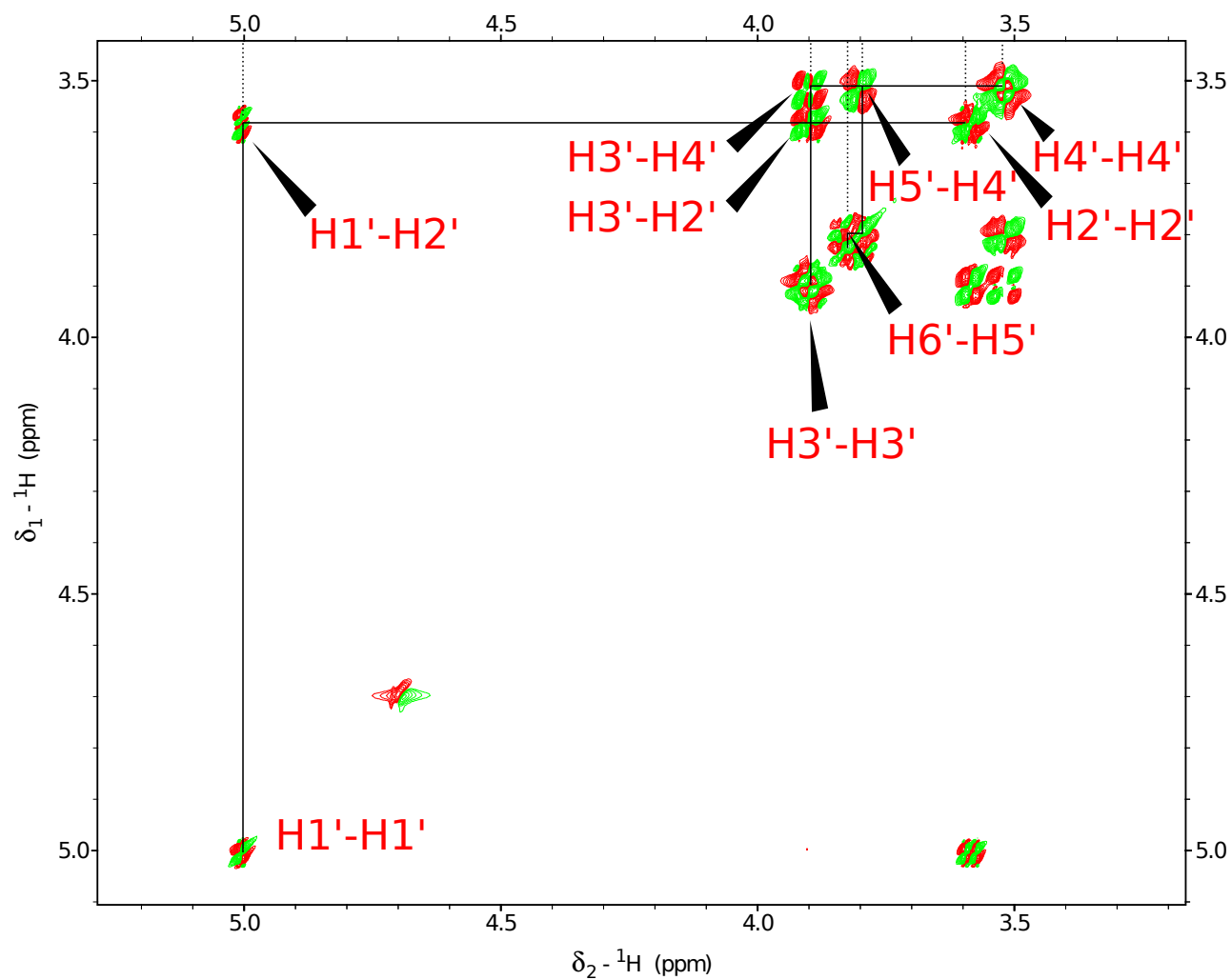
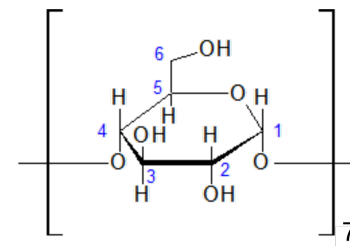
Hints for beginners

- ▶ 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

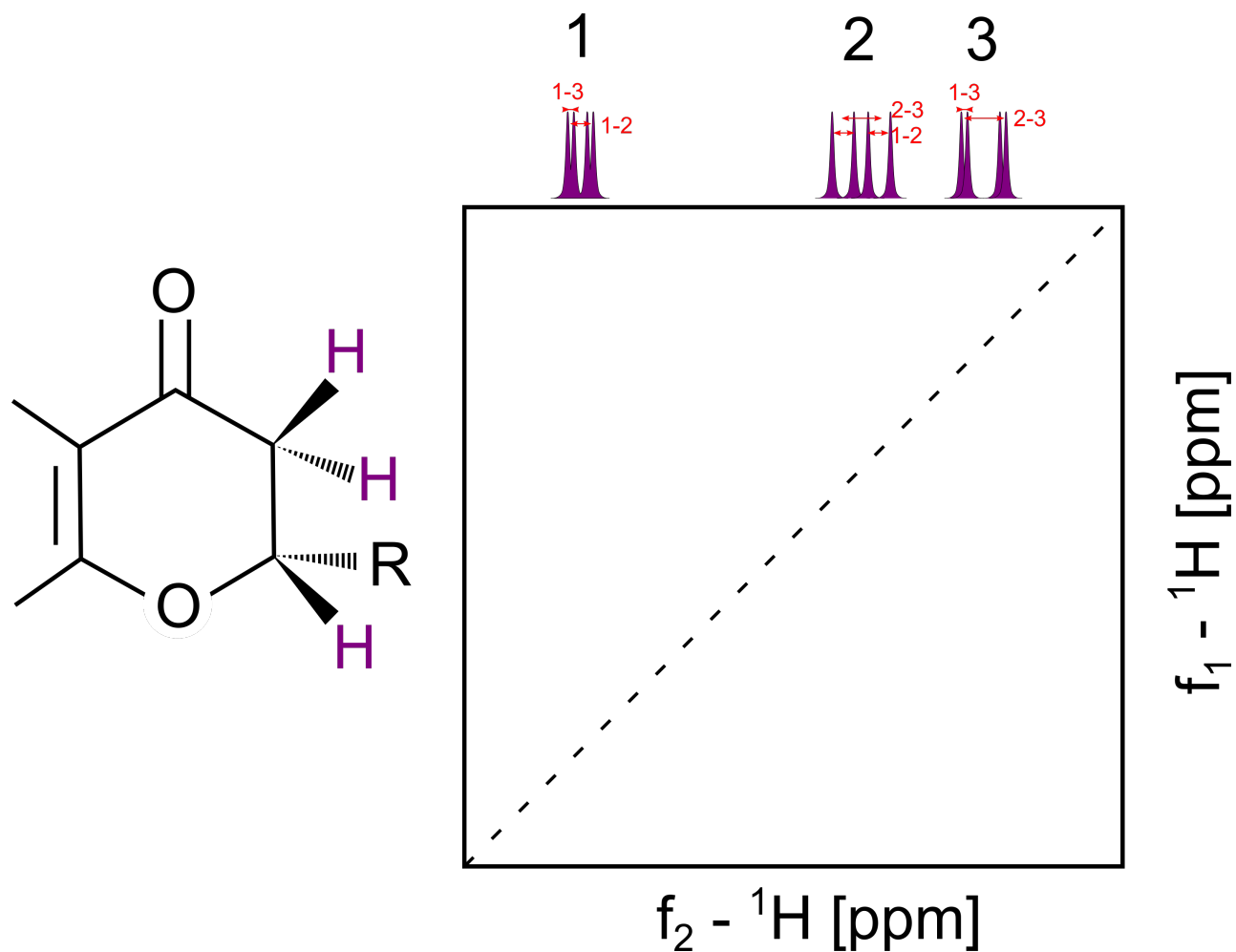


COSY : β -cyclodextrine



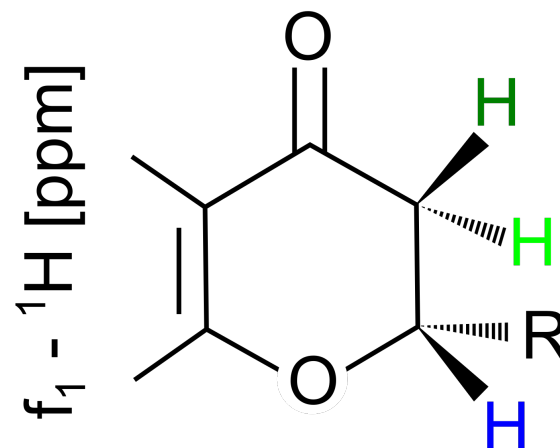
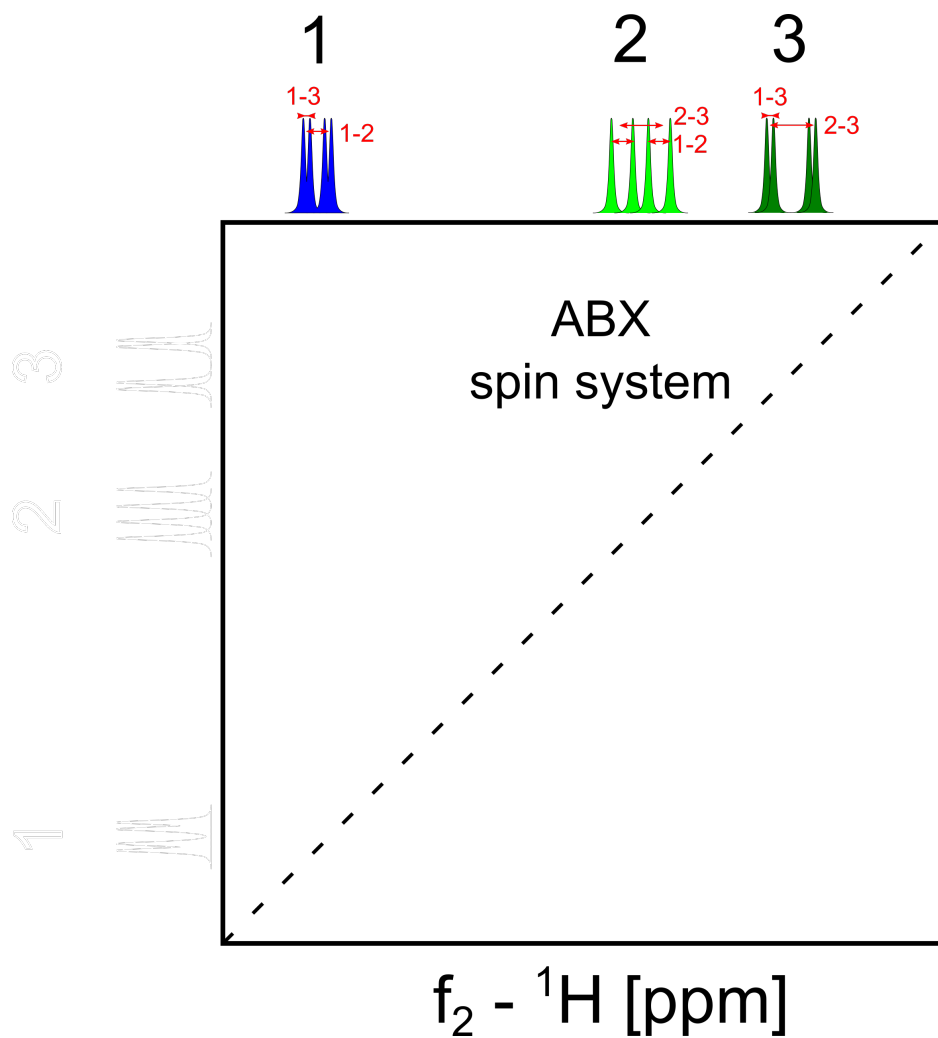
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



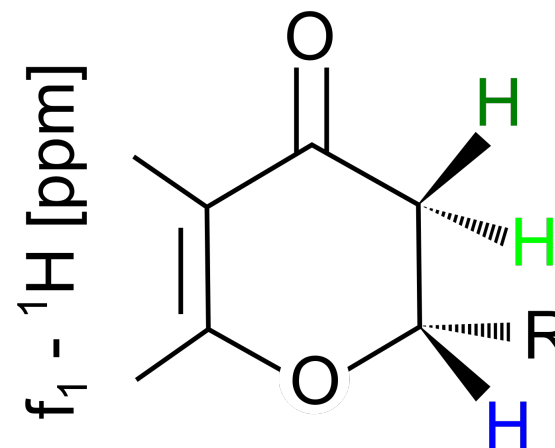
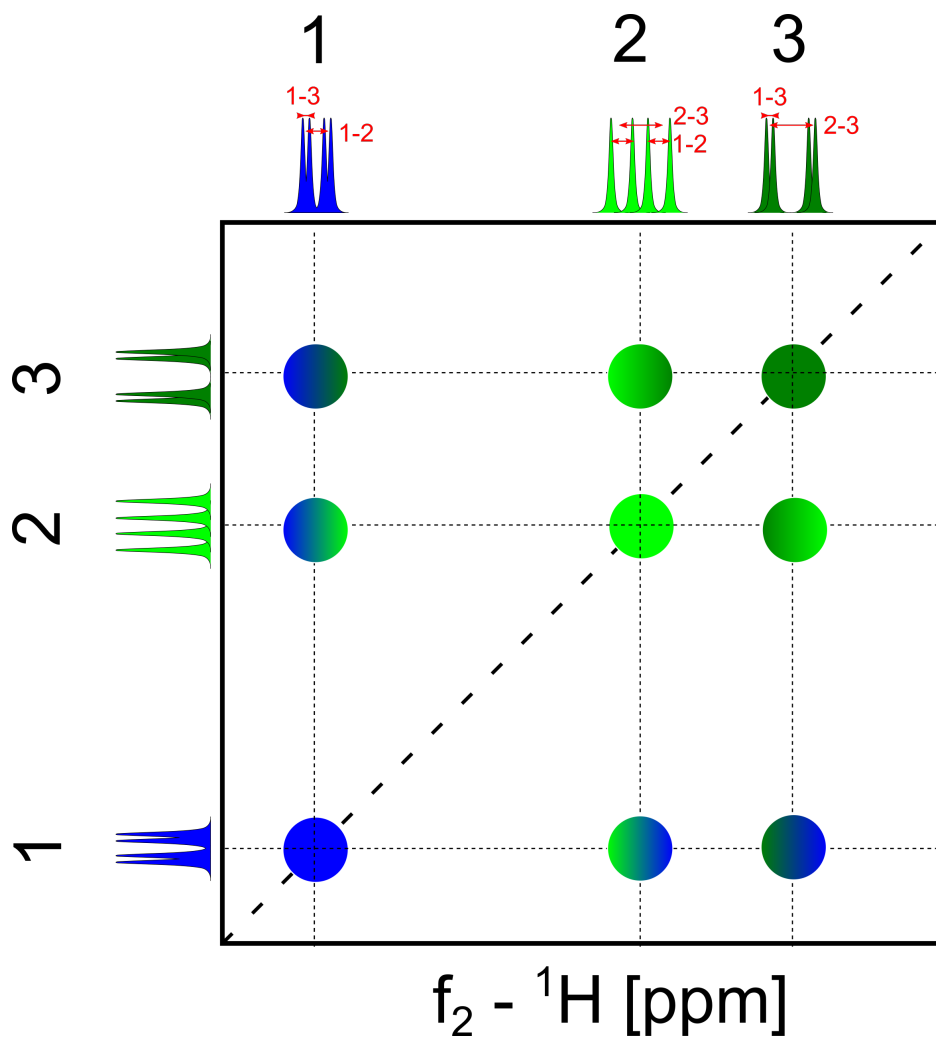
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



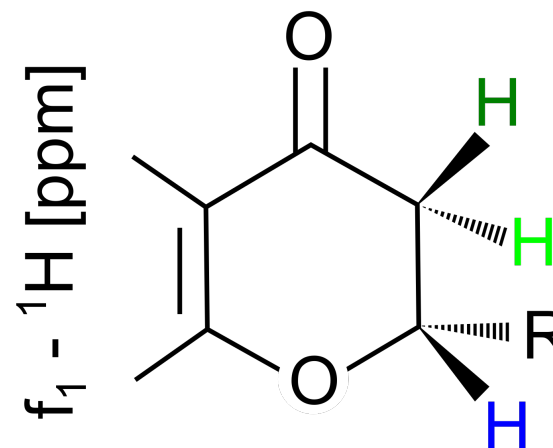
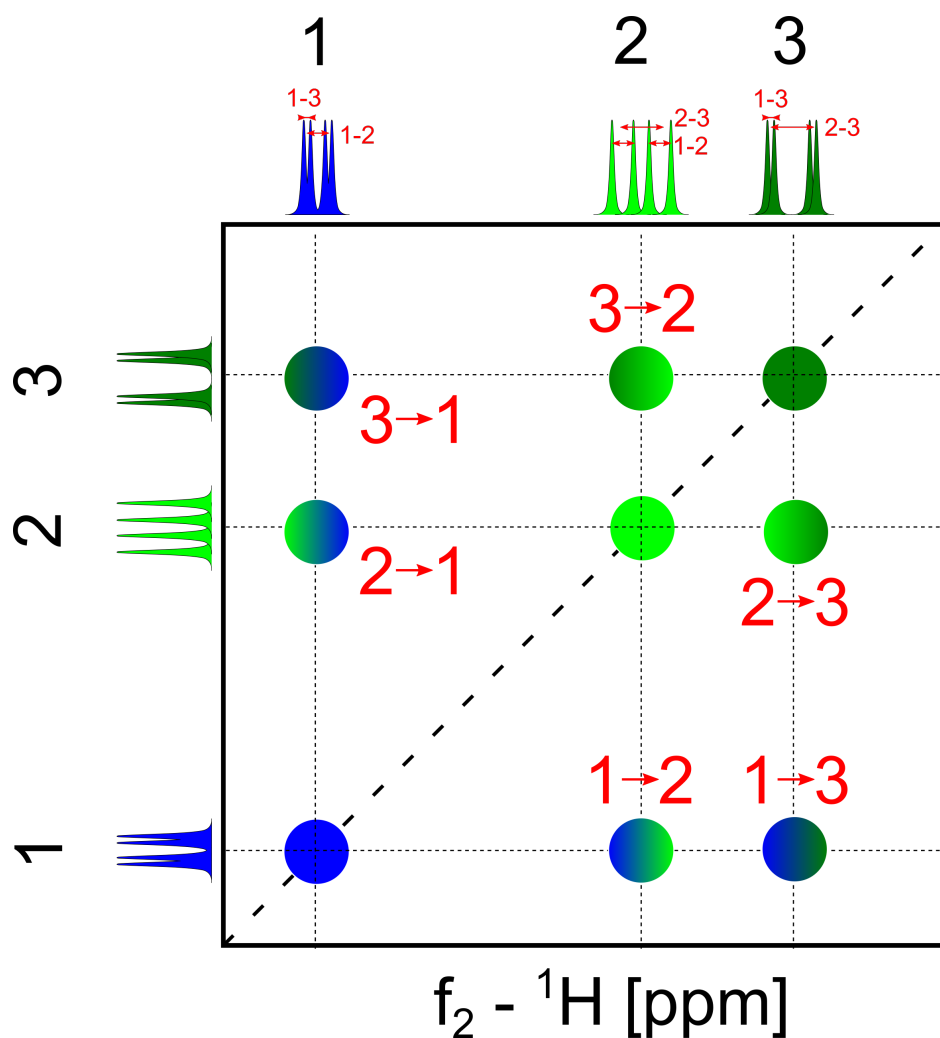
Phase sensitive COSY

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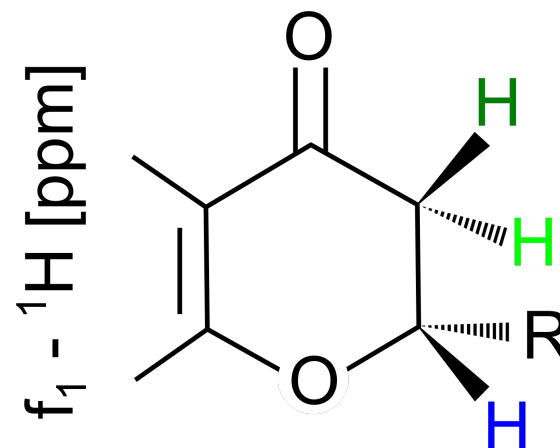
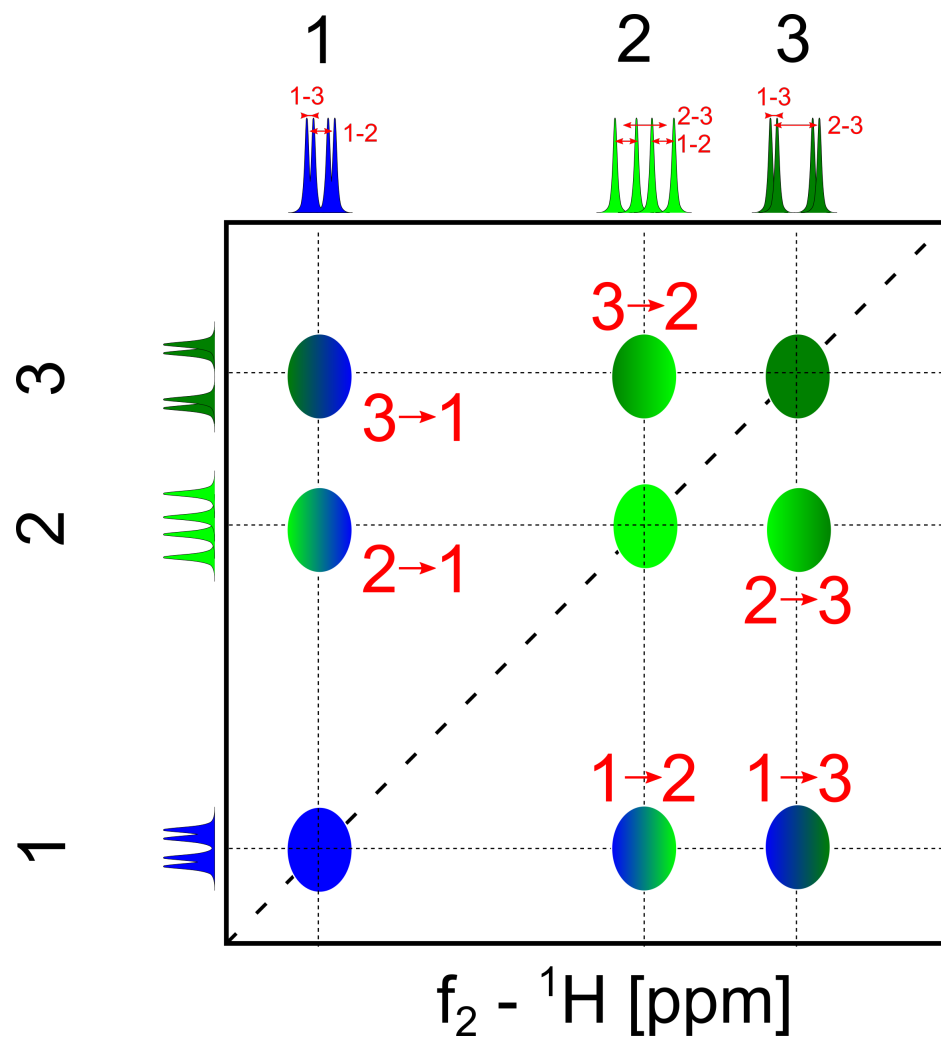
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



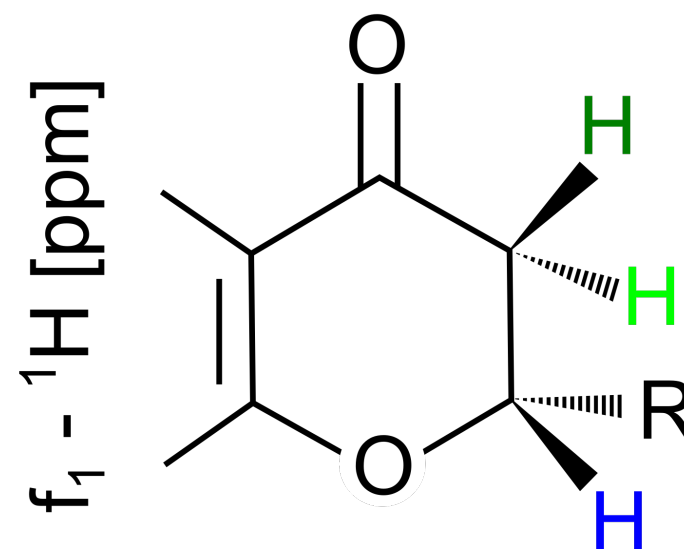
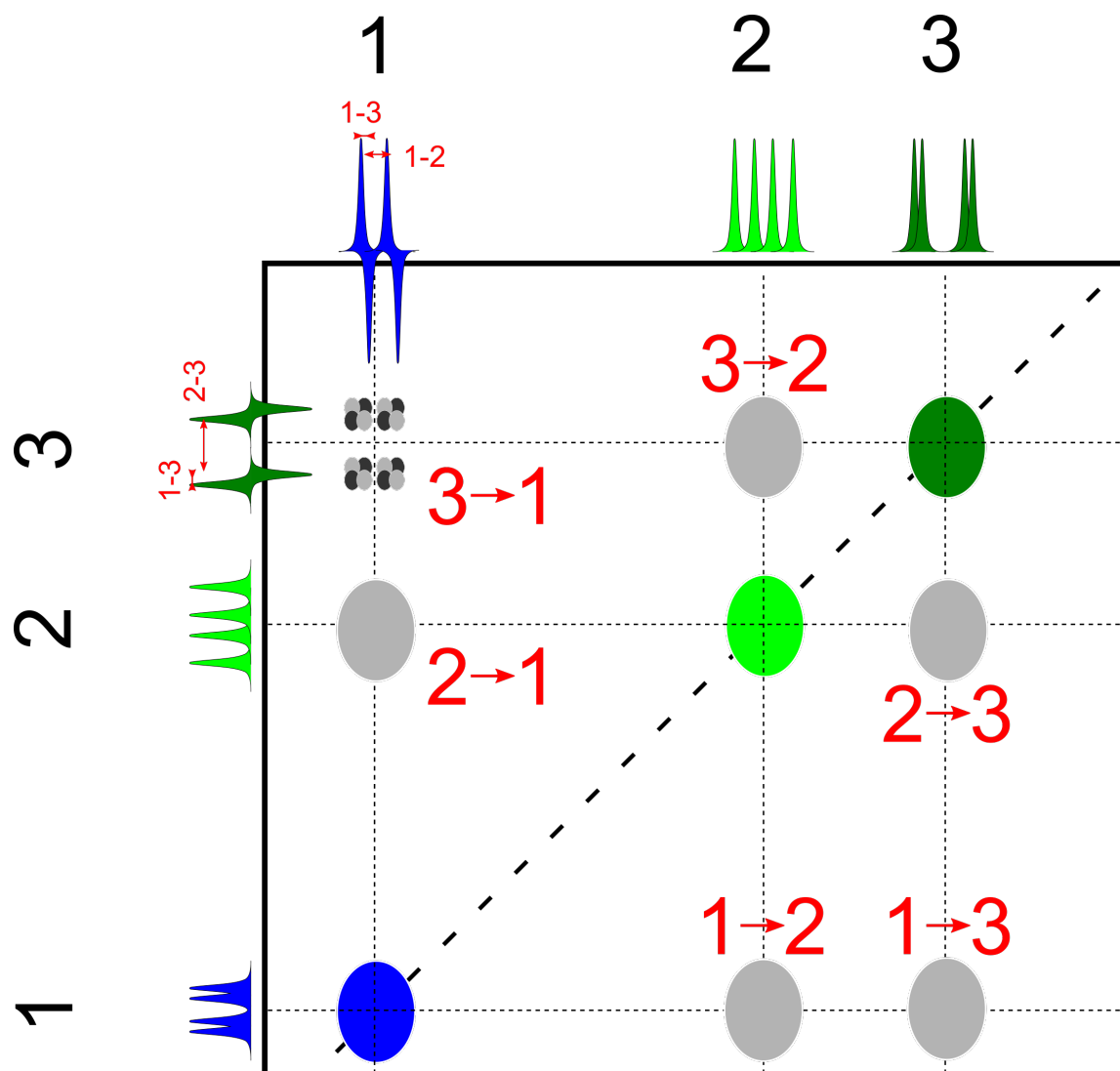
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



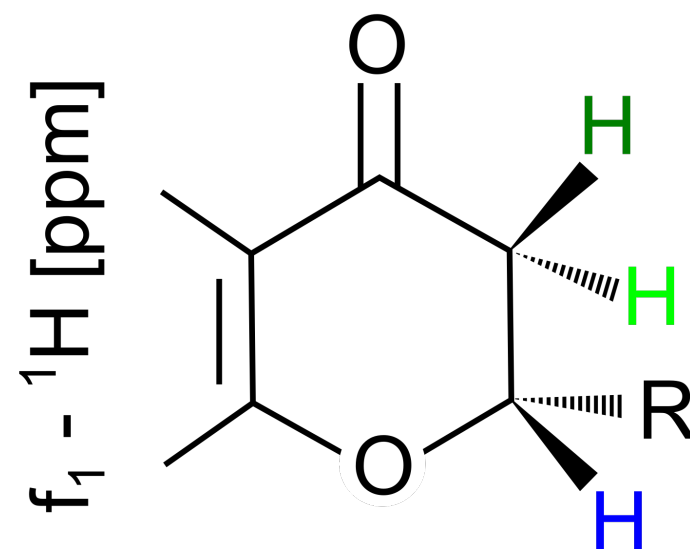
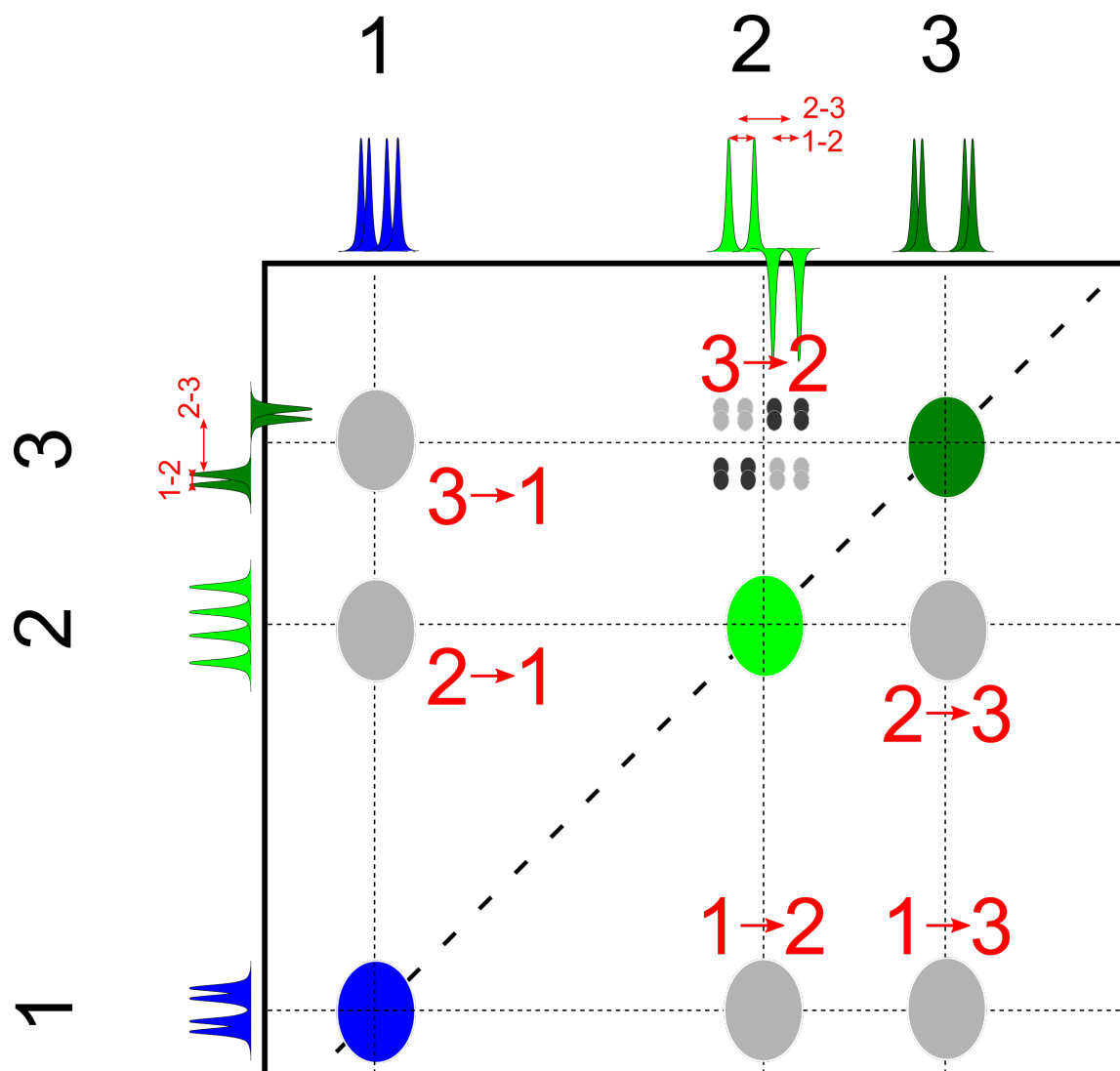
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



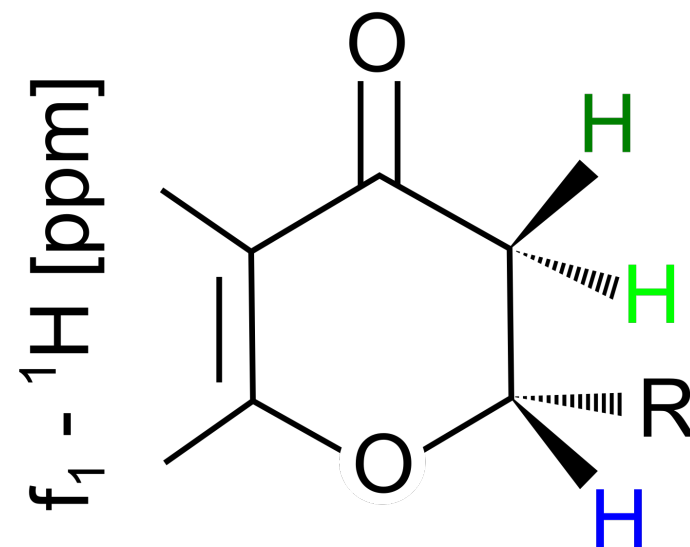
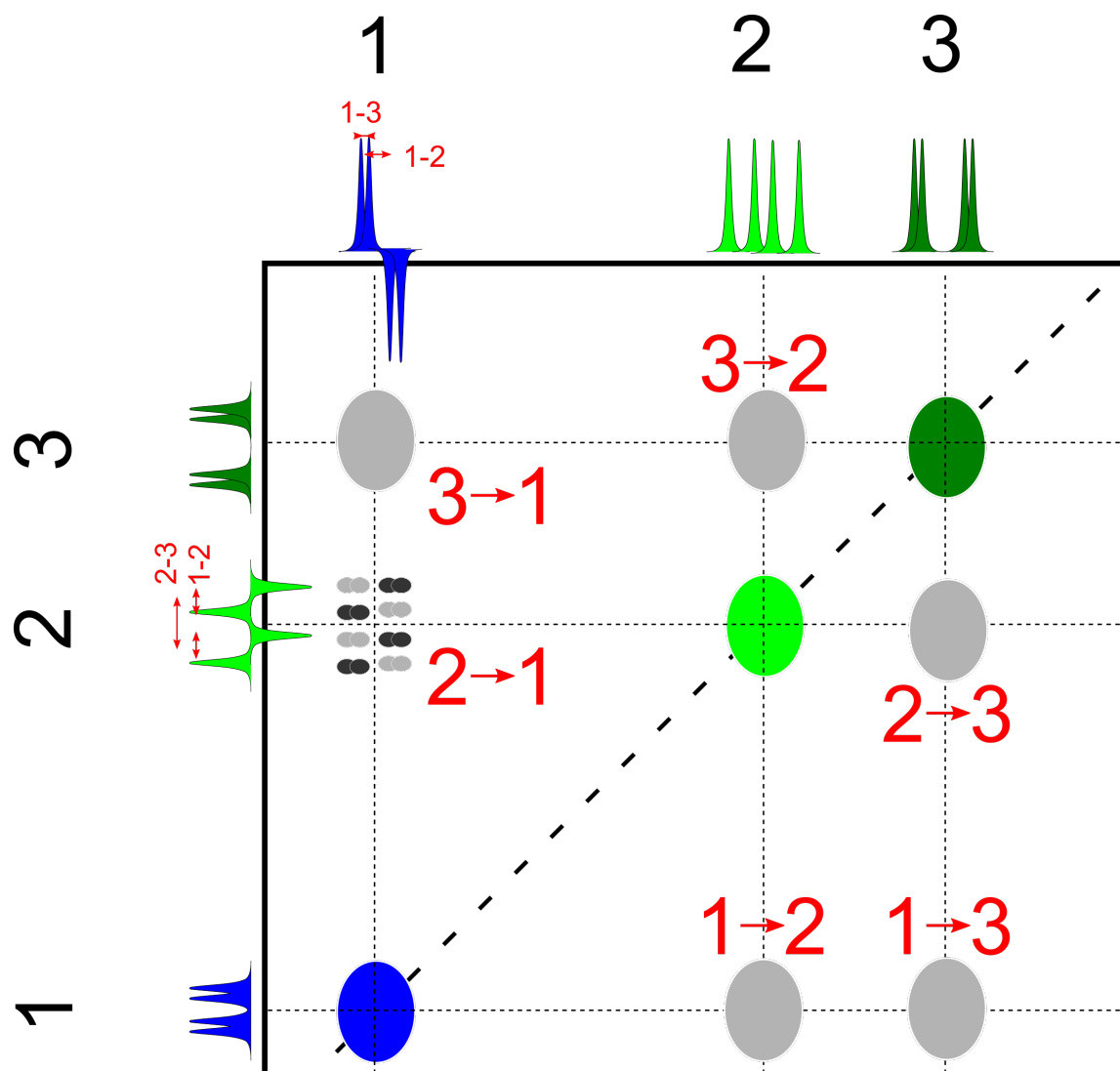
Phase sensitive COSY

- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase

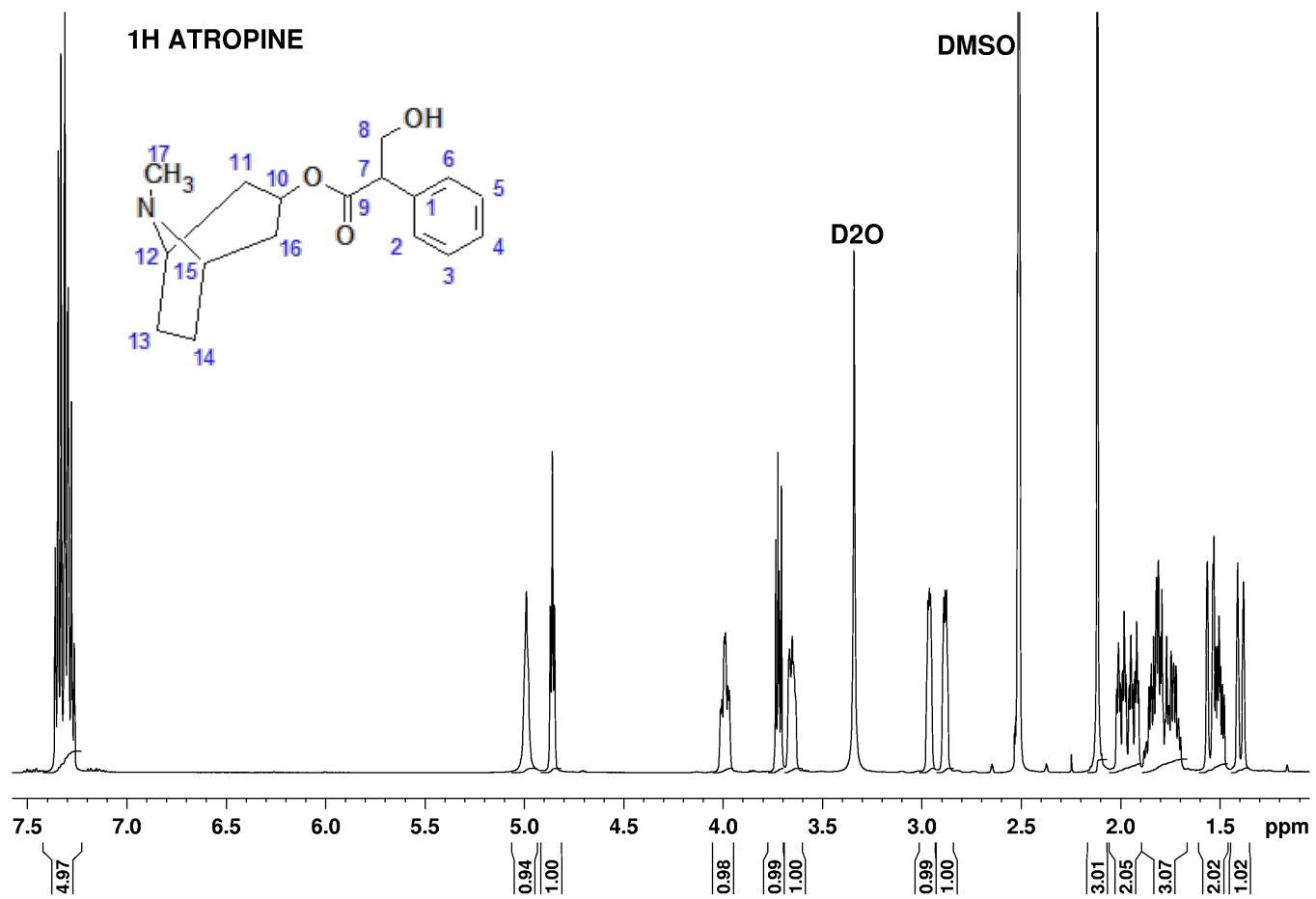


Phase sensitive COSY

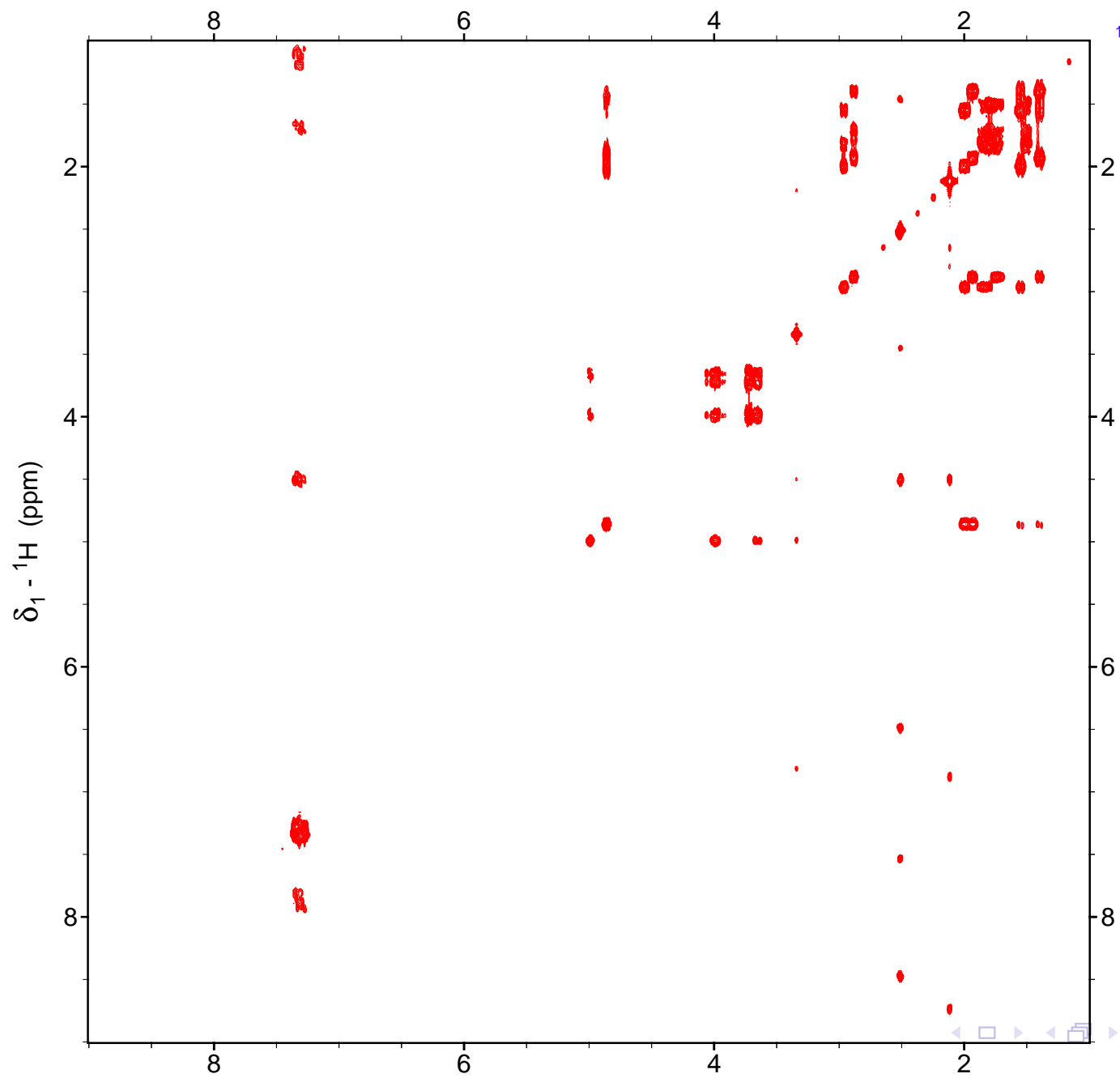
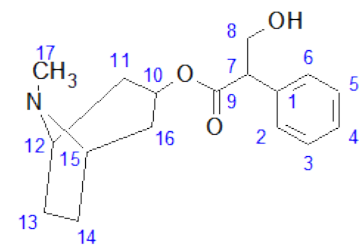
- ▶ direct vs. indirect dimension
- ▶ active coupling - antiphase crosspeak, passive coupling - in-phase



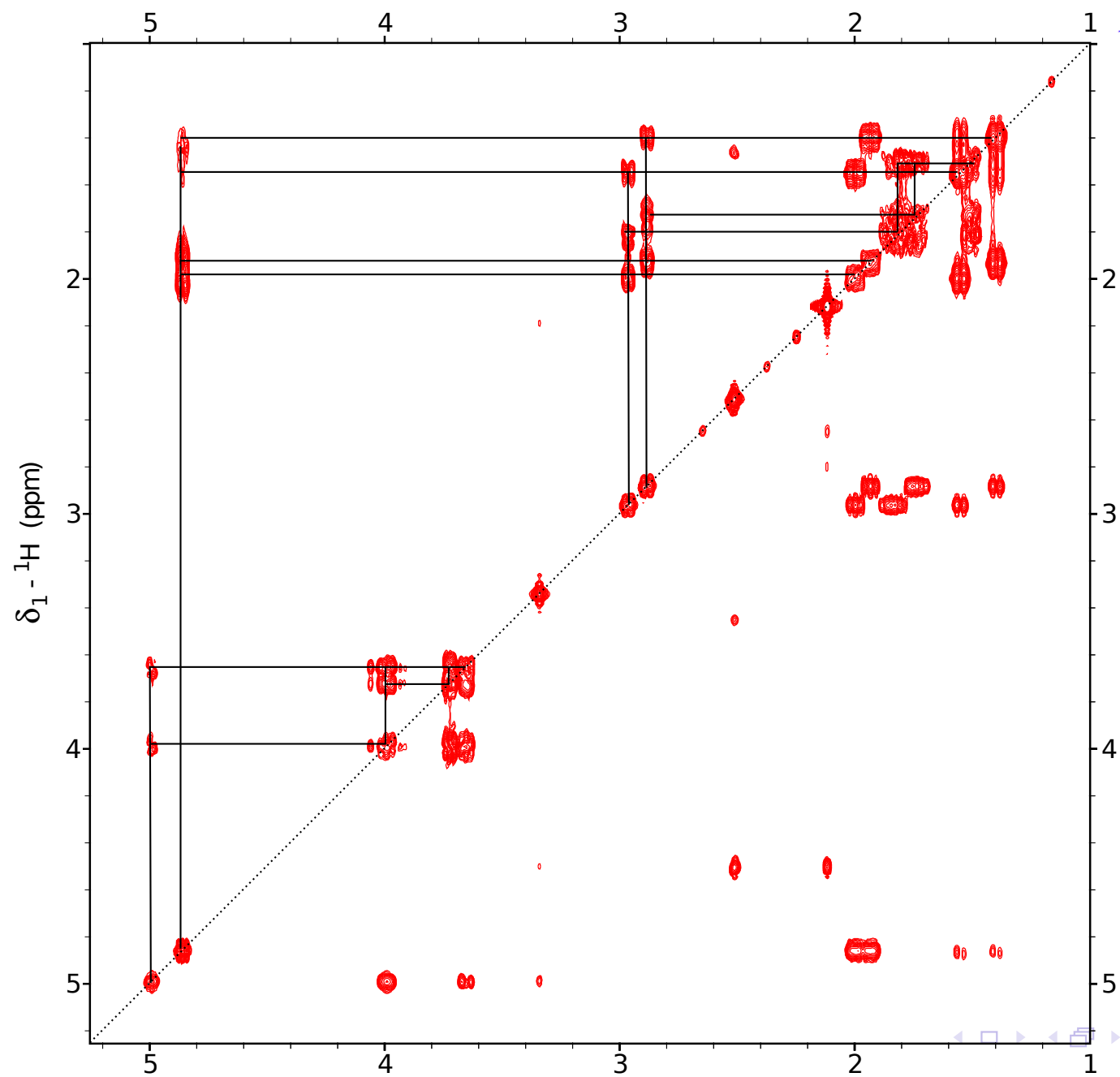
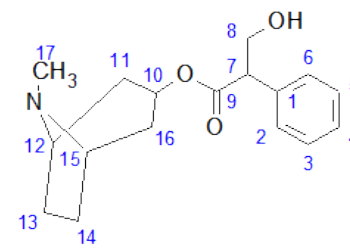
1D ^1H of Atropine in DMSO



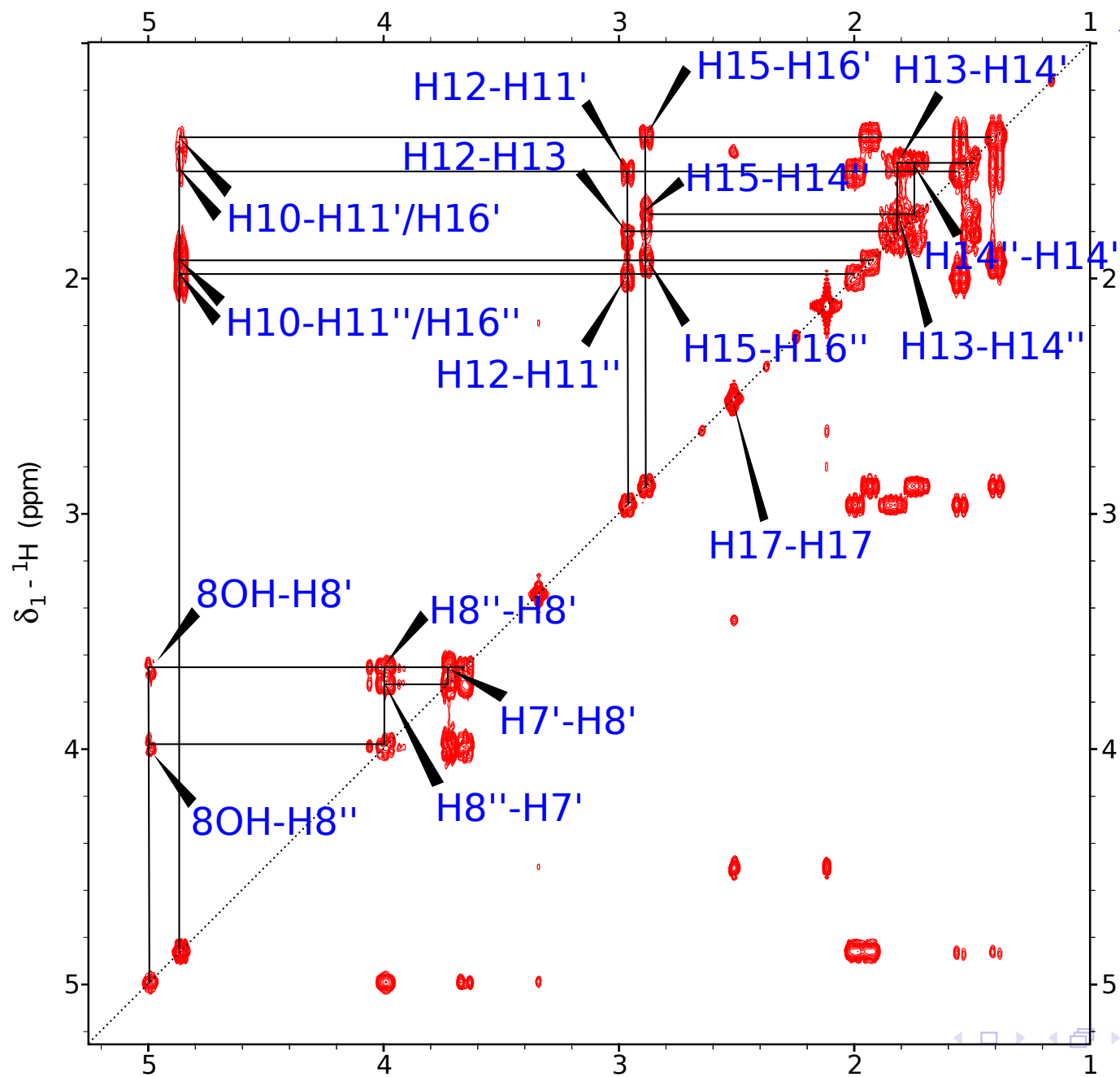
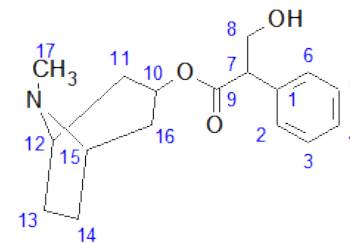
COSY : Atropine



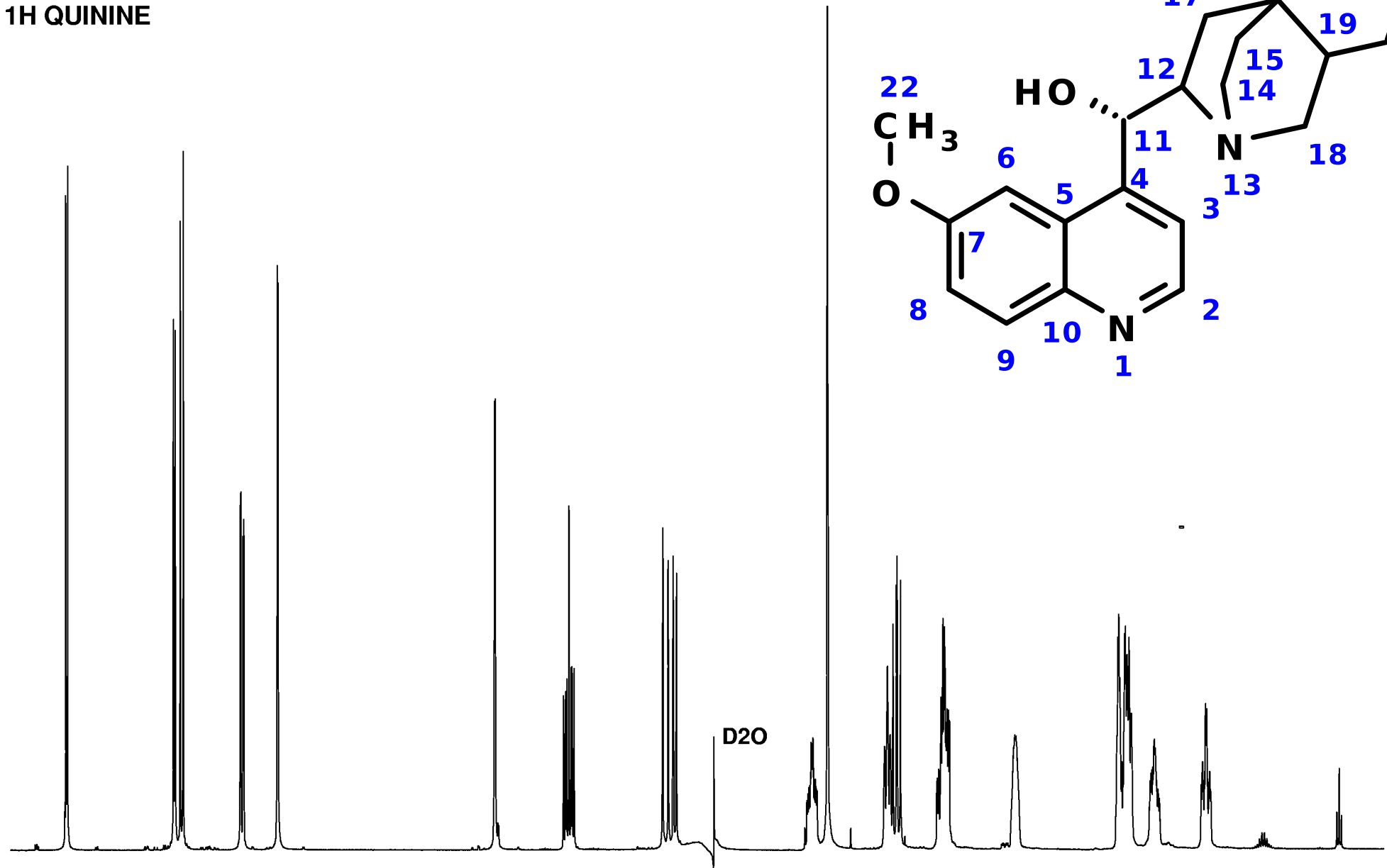
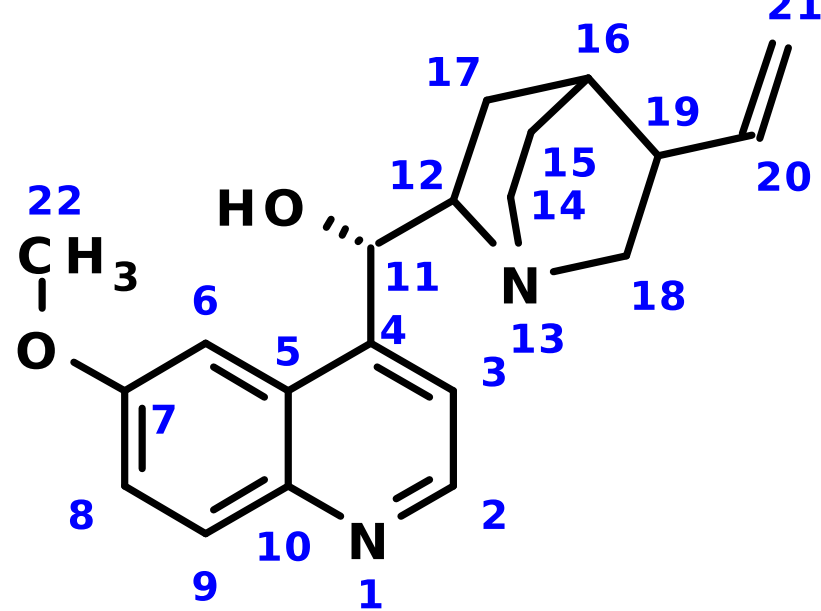
COSY : Atropine



COSY : Atropine

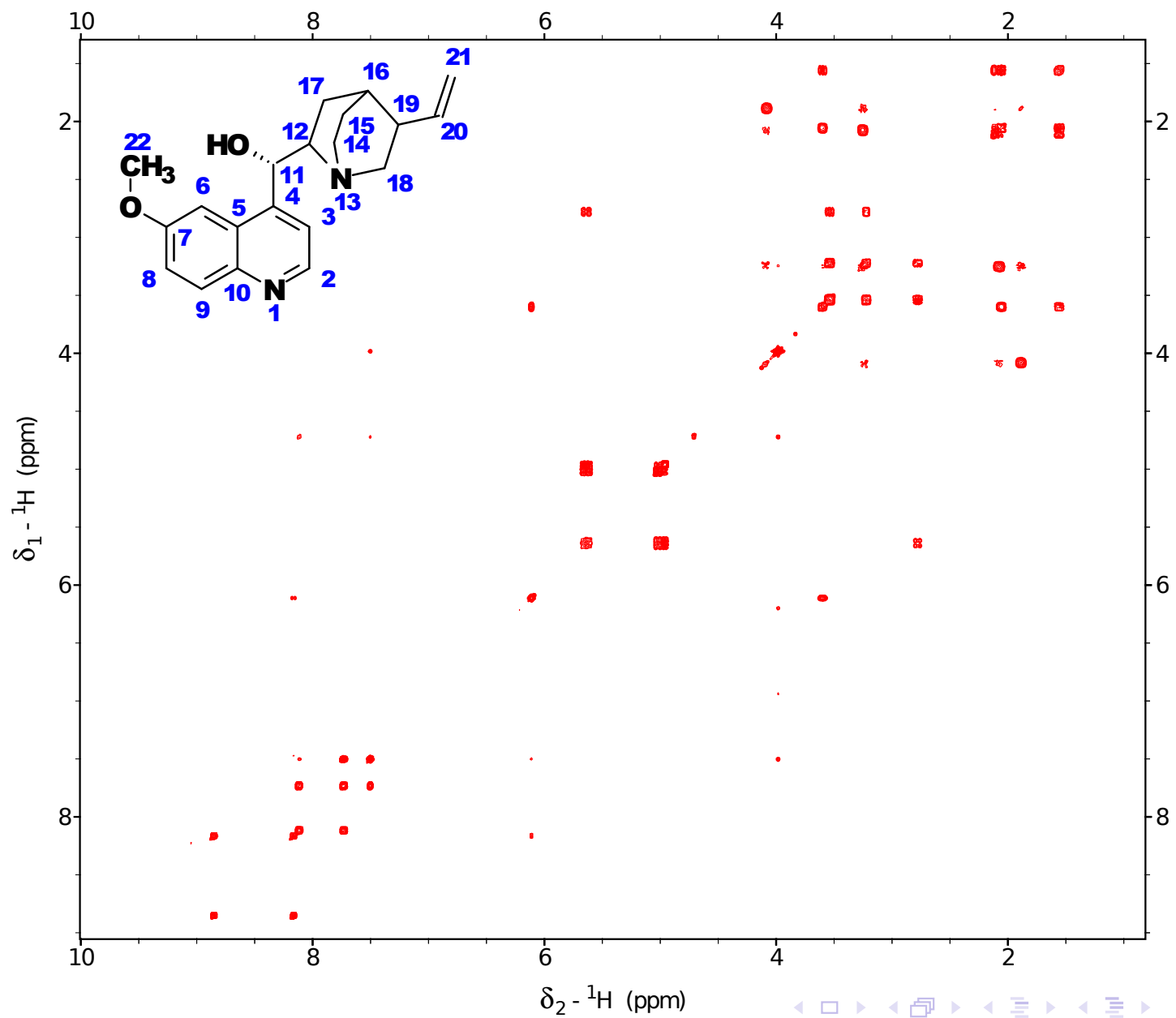


1H QUININE

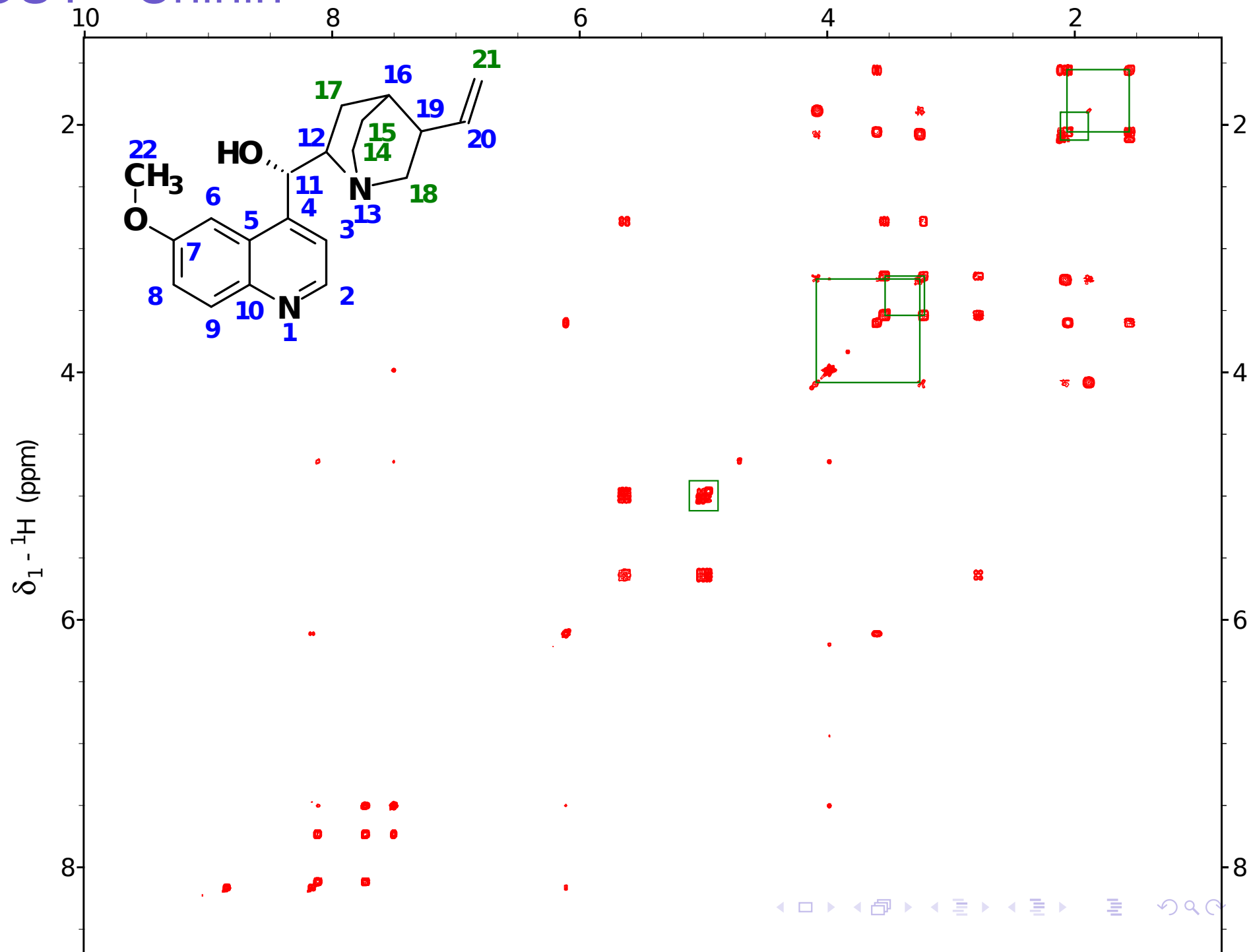


9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

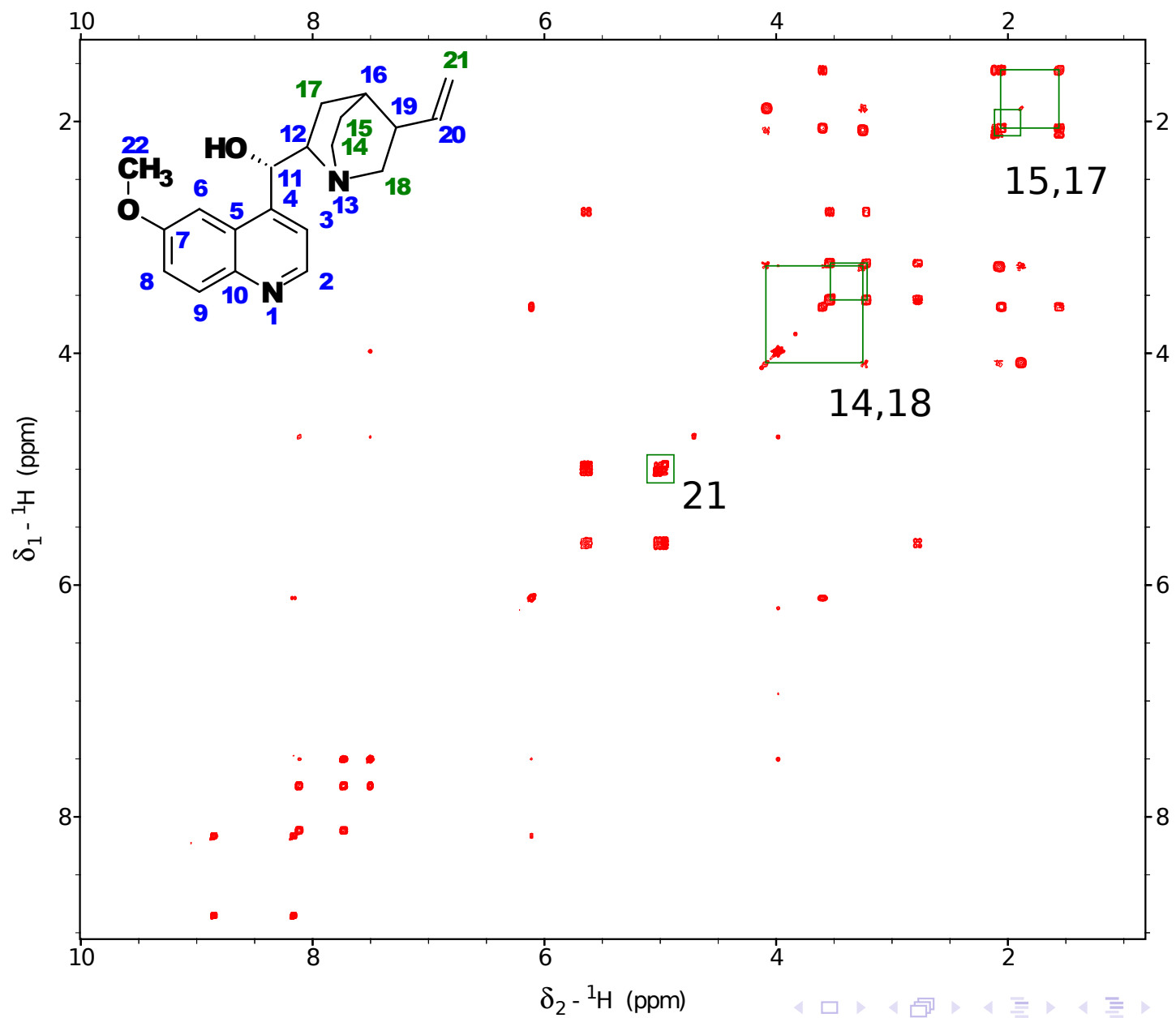
COSY - Chinin



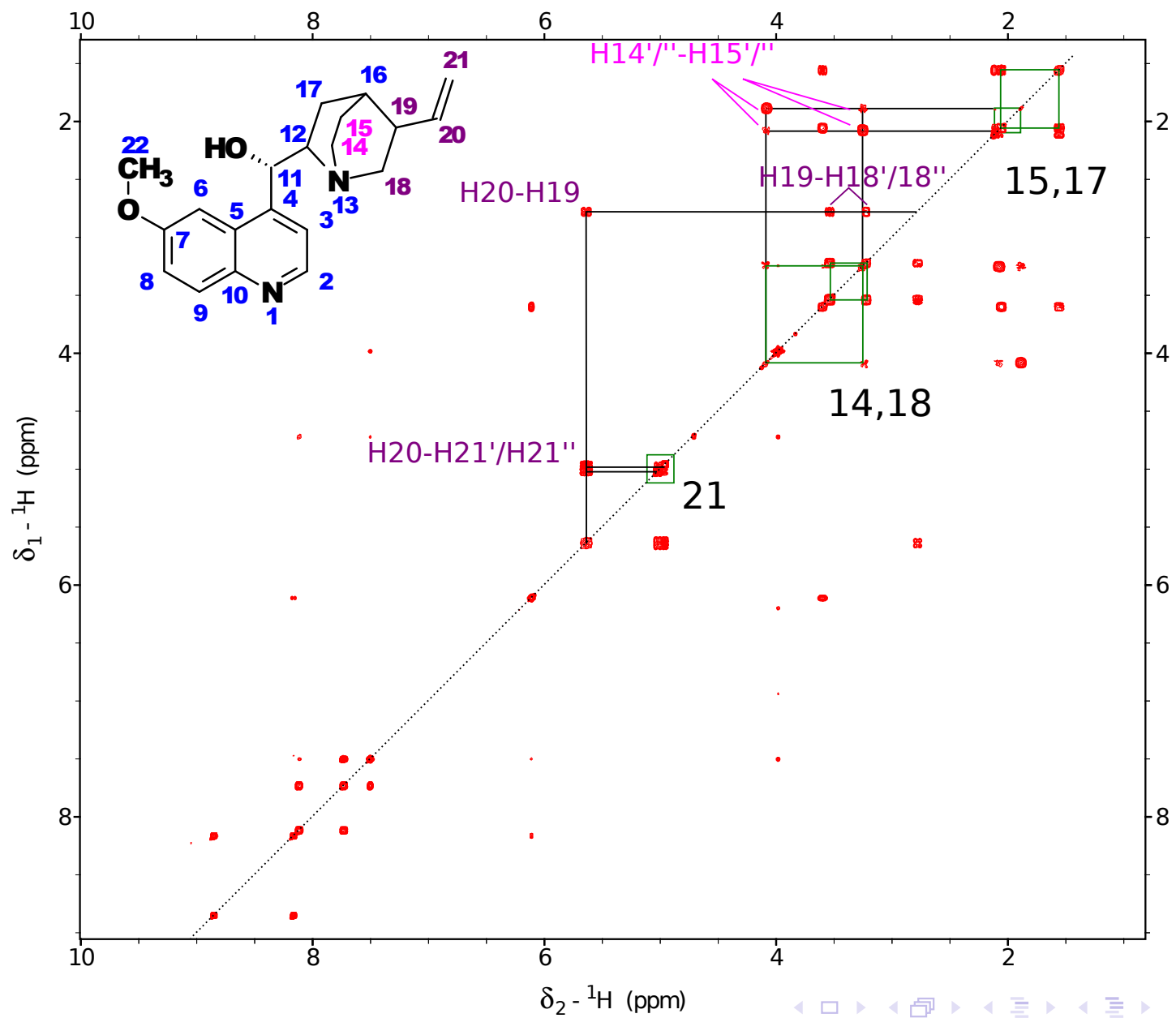
COSY - Chinin



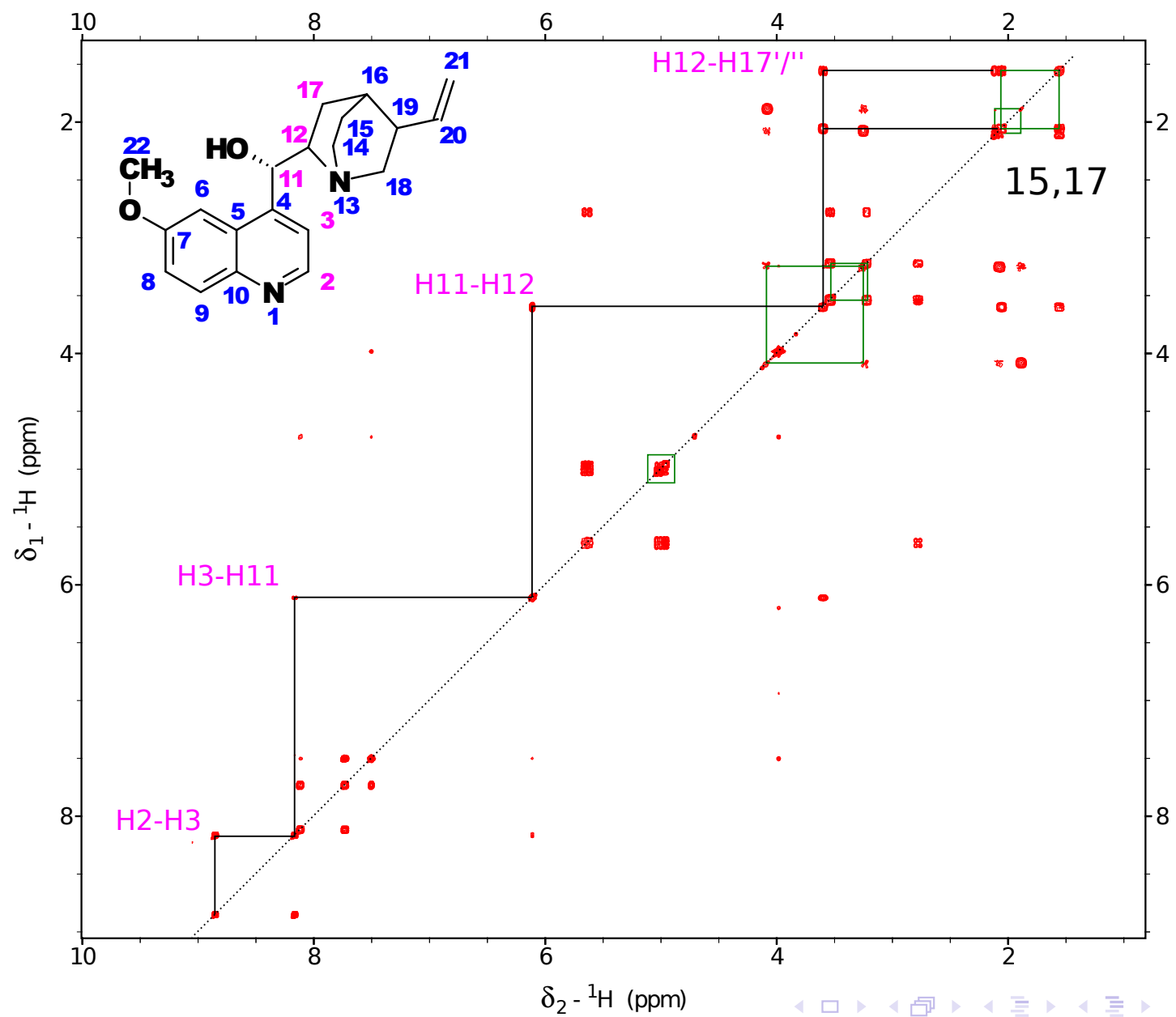
COSY - Chinin



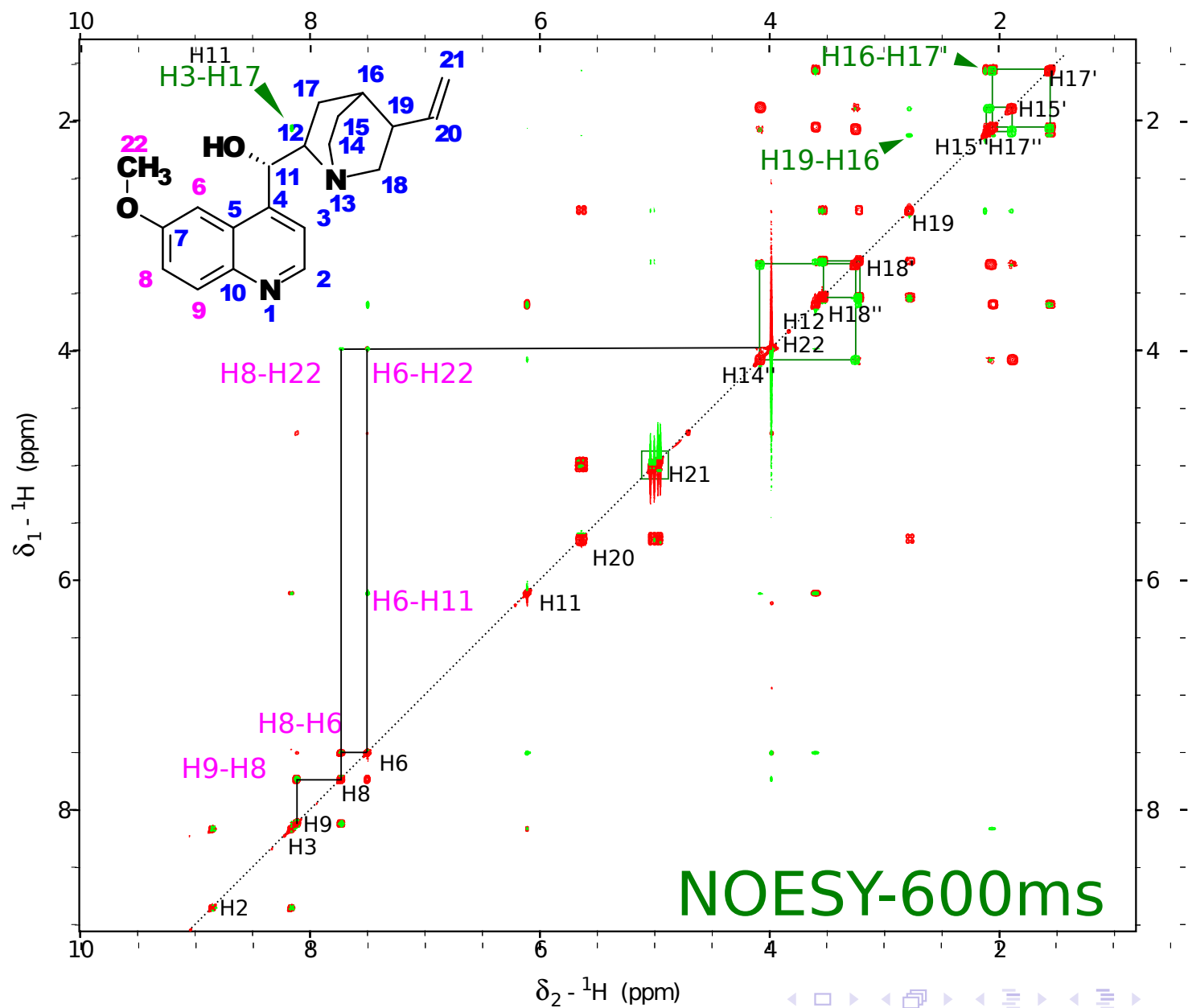
COSY - Chinin



COSY - Chinin



COSY - Chinin



Next topic

^1H - ^1H through space correlations (NOESY, ROESY)