

C8953
NMR strukturní analýza
seminář

Introduction to 2D NMR & Homonuclear correlation - COSY

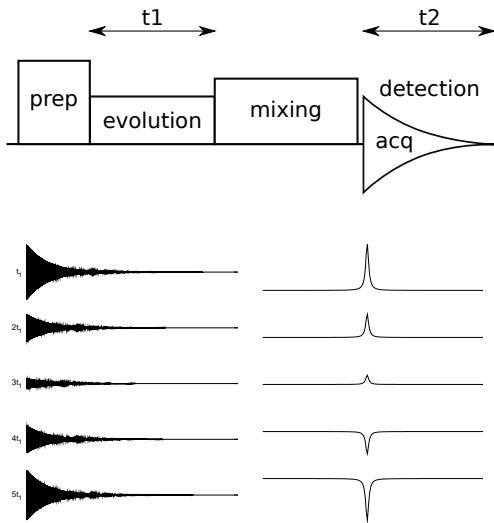
Ondřej Jurček
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March 27, 2017

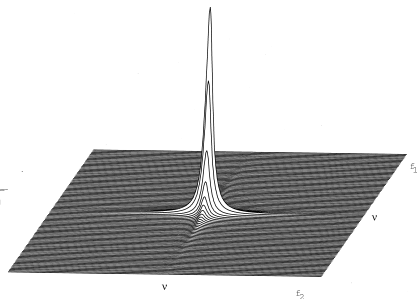
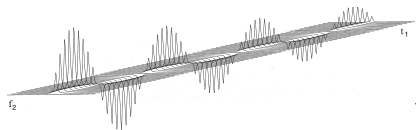
2D NMR

Second dimension f_1

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



2D NMR

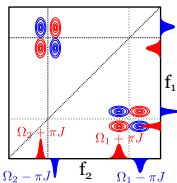
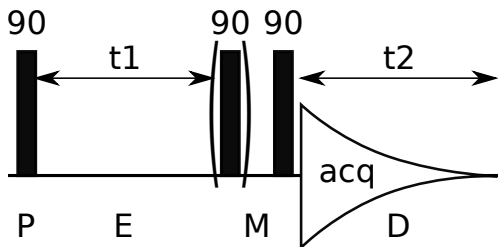


2D spektrum

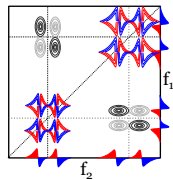
- ▶ FT in t_1 - modulated 1D spectra
- ▶ FT in t_2 - 2D spectrum

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



$$1/2[\cos(\Omega t_1 + \pi J t_1) - \cos(\Omega t_1 - \pi J t_1)]$$

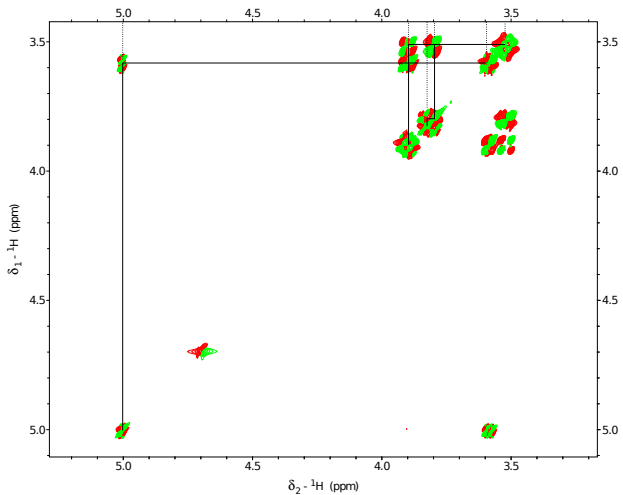
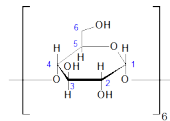


$$1/2[\sin(\Omega t_1 + \pi J t_1) + \sin(\Omega t_1 - \pi J t_1)]$$

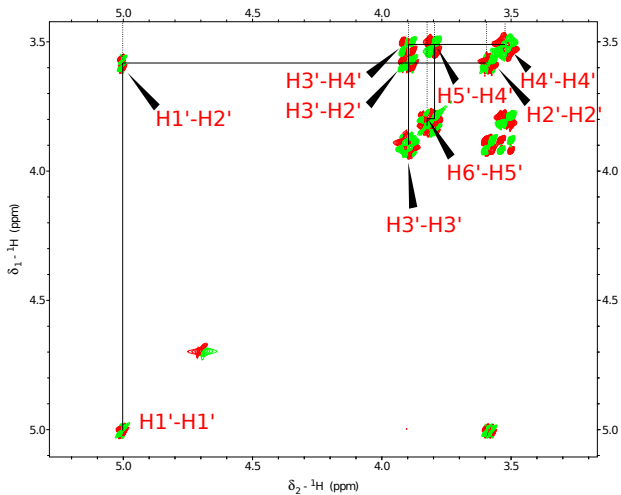
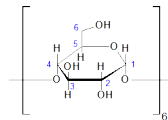
Hints for beginners

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

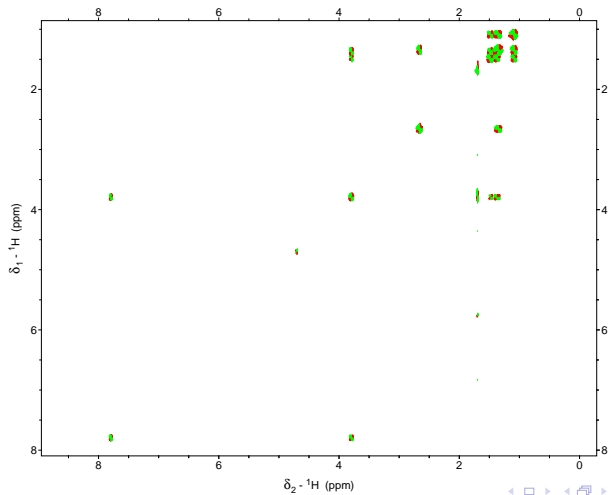
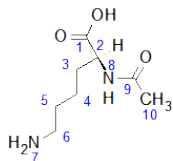
COSY : Betacyklodextrine



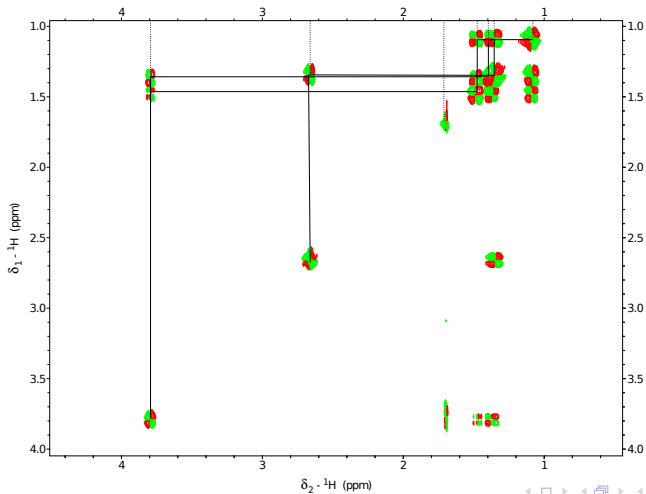
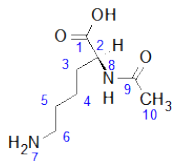
COSY : Betacyklodextrine



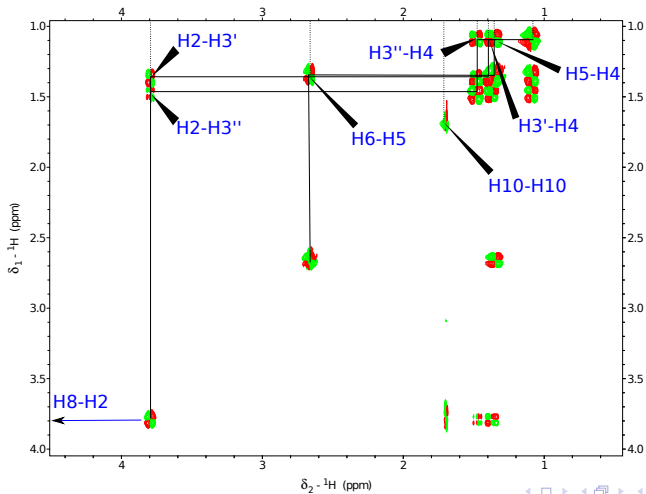
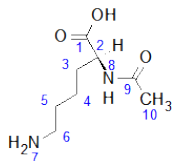
COSY : N-acetyllysine



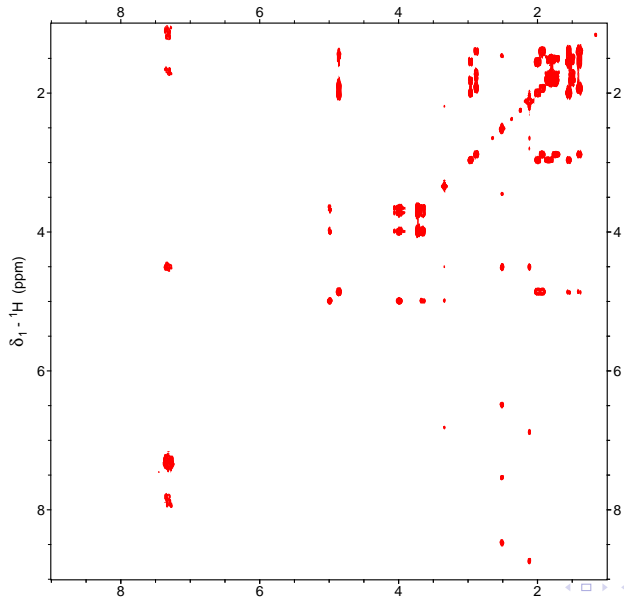
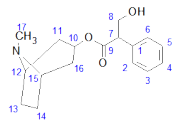
COSY : N-acetyllysine



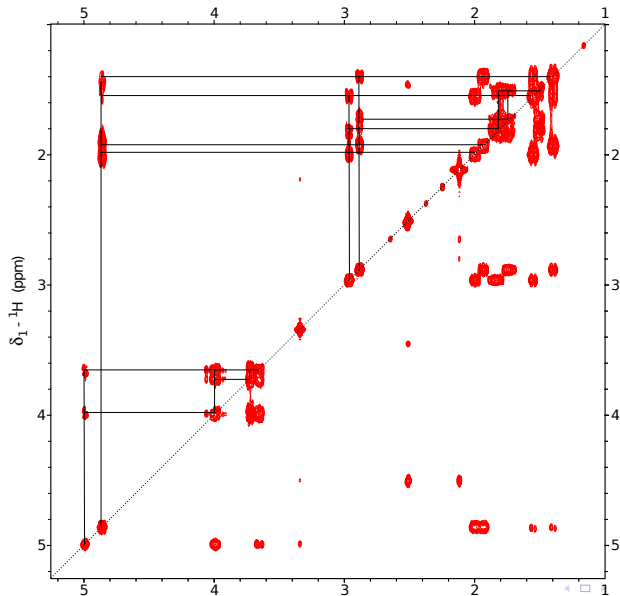
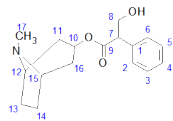
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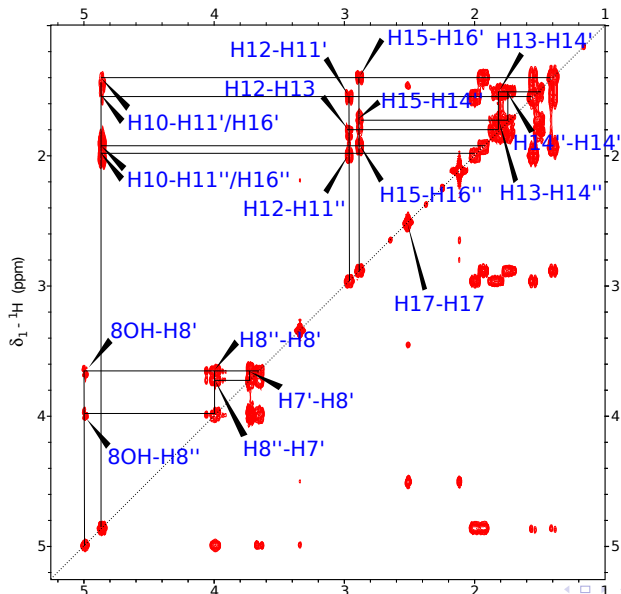
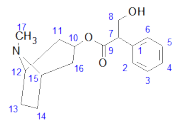
COSY : Atropine



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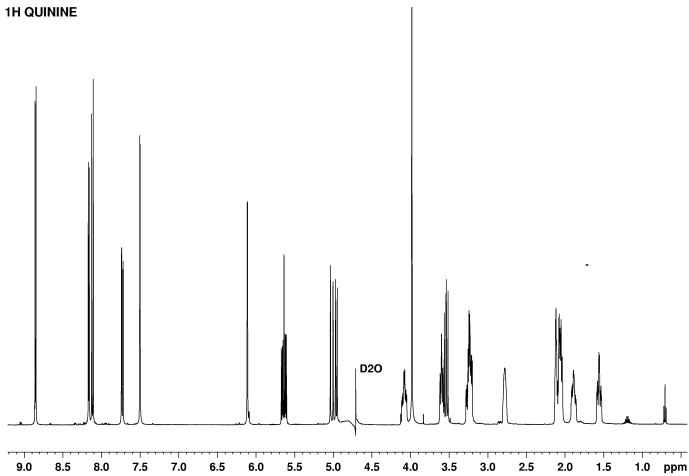


COSY : Atropine

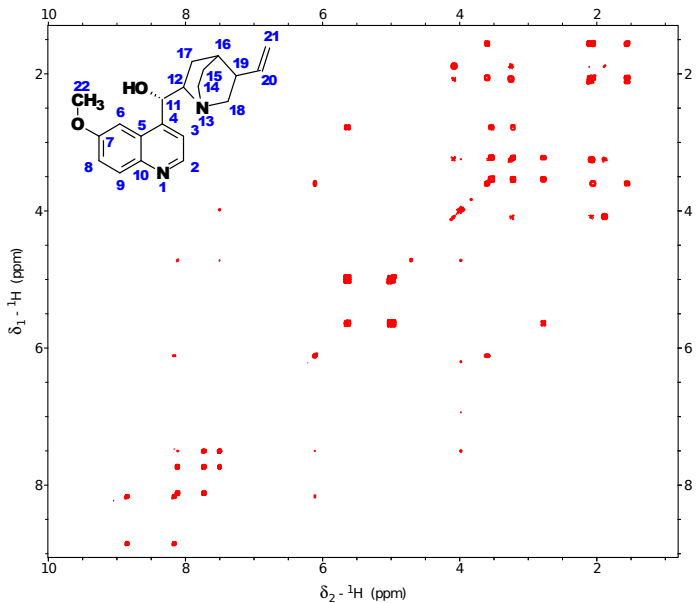


Chinin - 1D ^1H

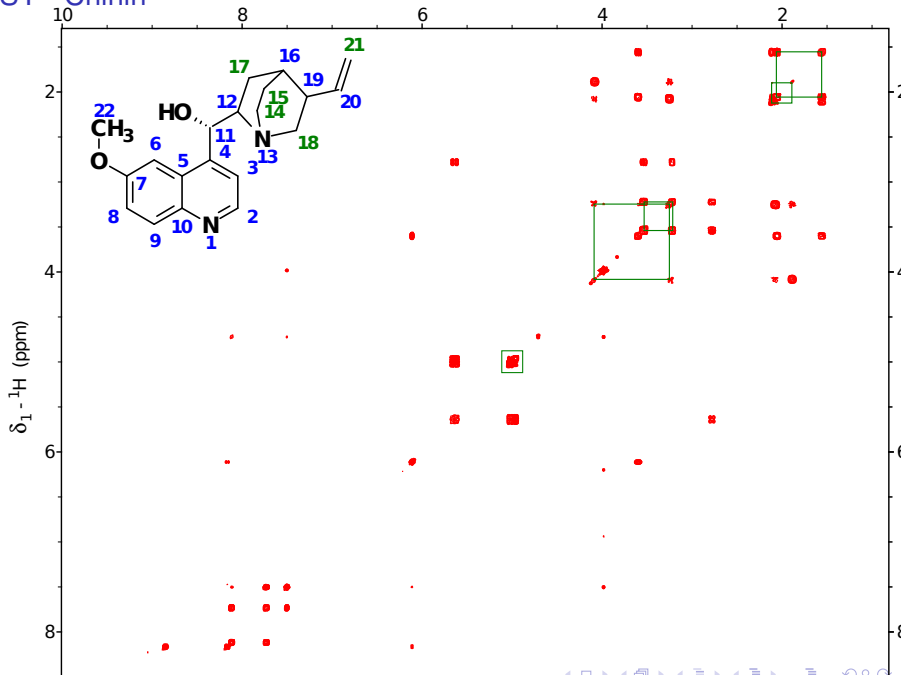
1H QUININE



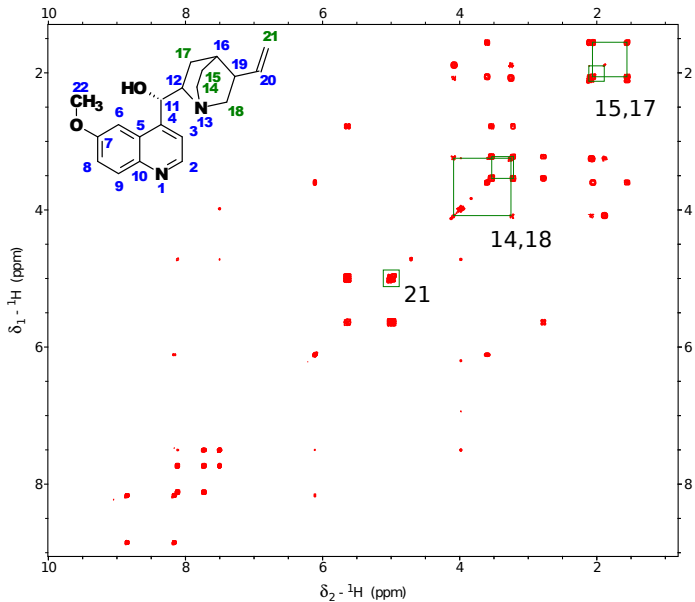
COSY - Chinin



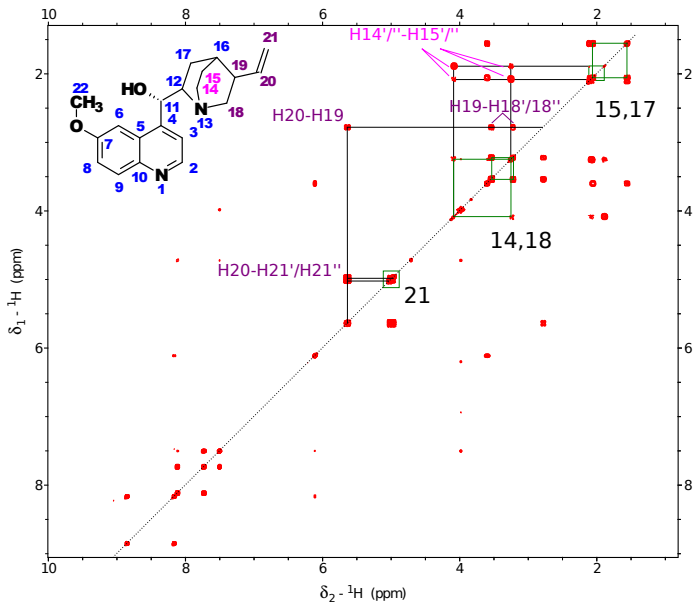
COSY - Chinin



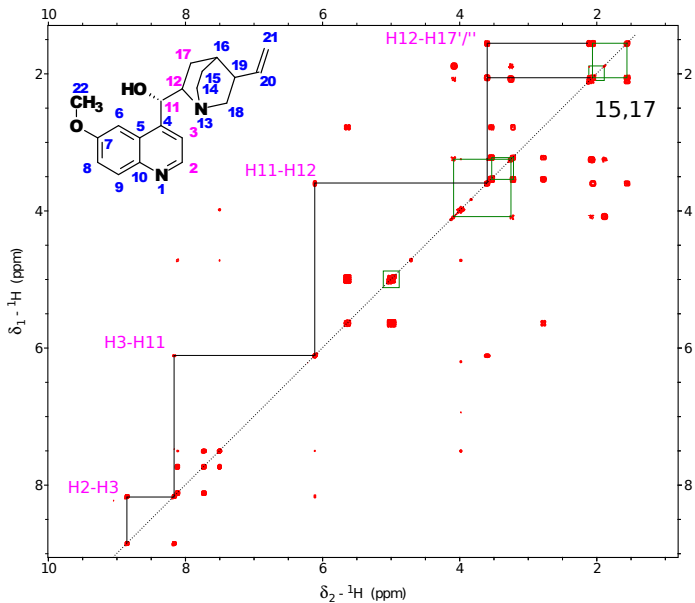
COSY - Chinin



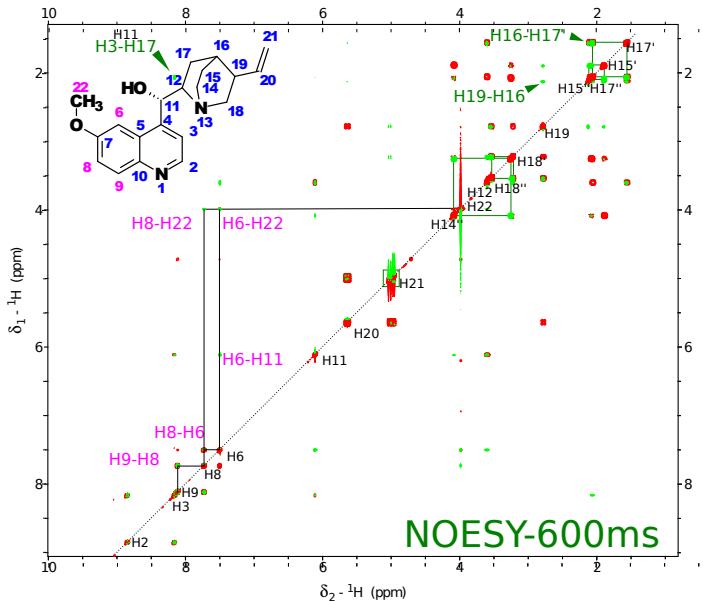
COSY - Chinin



COSY - Chinin



COSY - Chinin



Next seminar: ^1H - ^1H NOESY