

C8953

NMR structural analysis - seminar

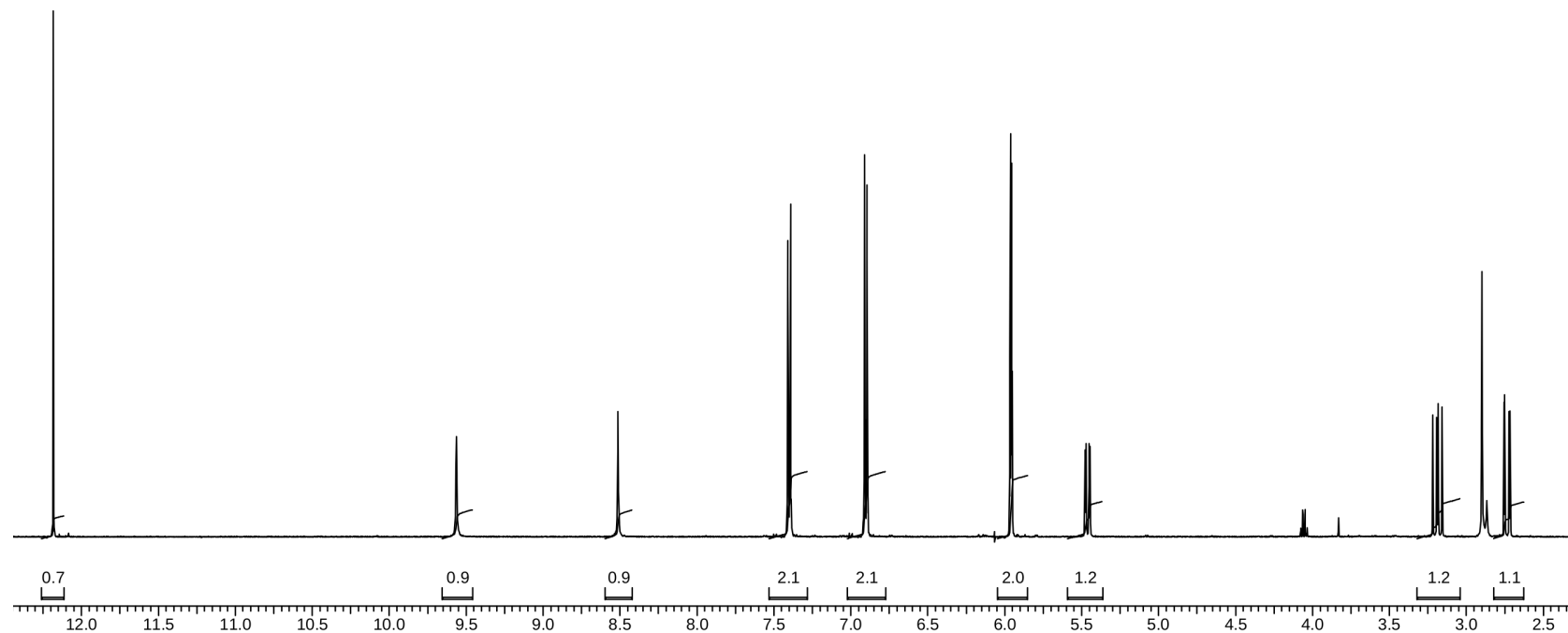
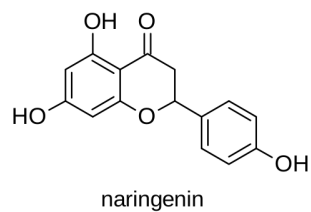
Vector model of NMR experiments + 2D spectra, COSY

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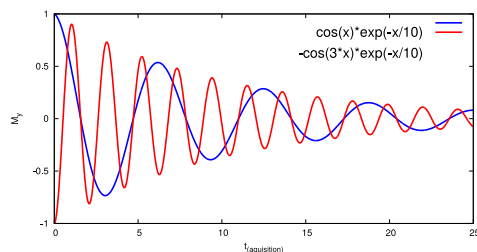
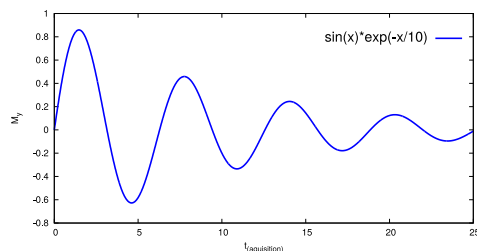
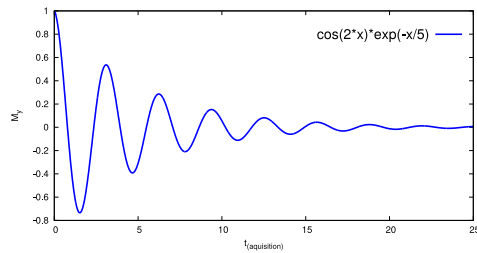
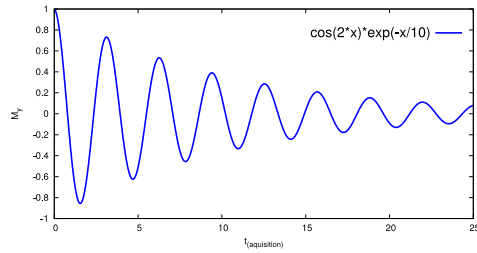
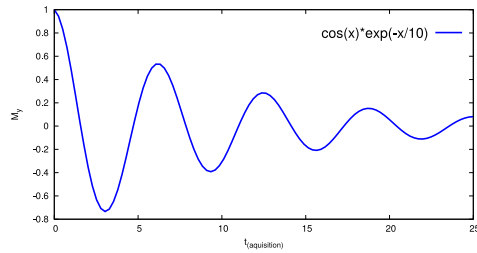
March 21, 2018

^1H NMR spectrum of naringenin in d_6 -acetone



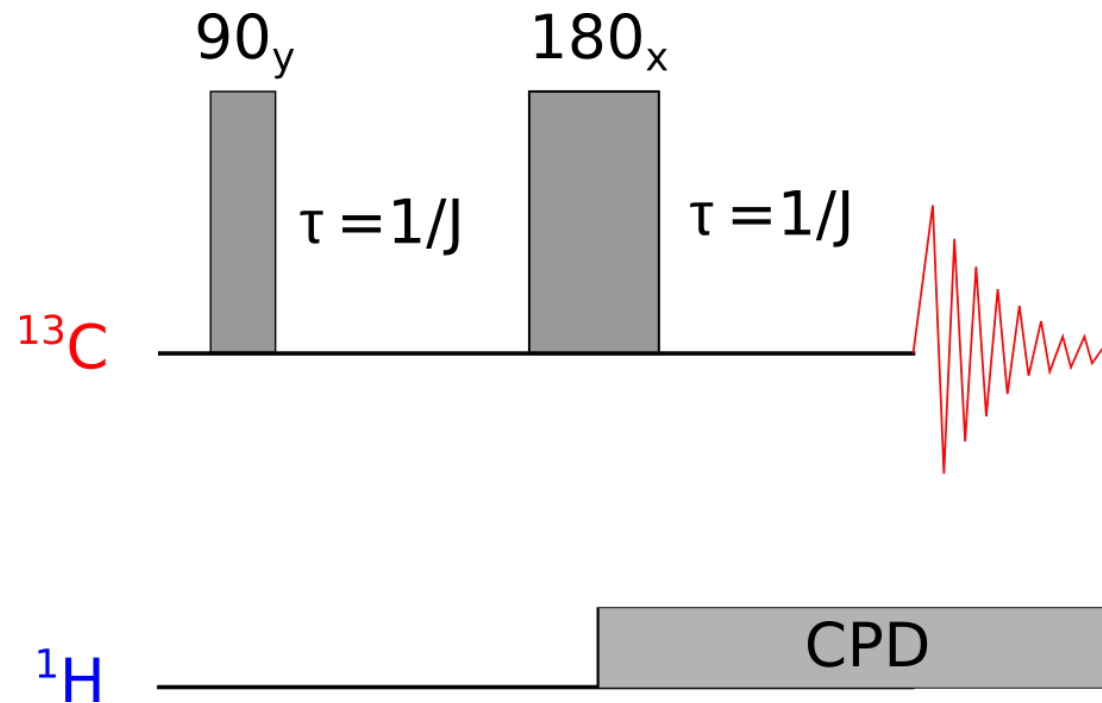
Basics of 1D FT spectroscopy

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



Heteronuclear spin echo of ^{13}C - $^1\text{H}_3$ group

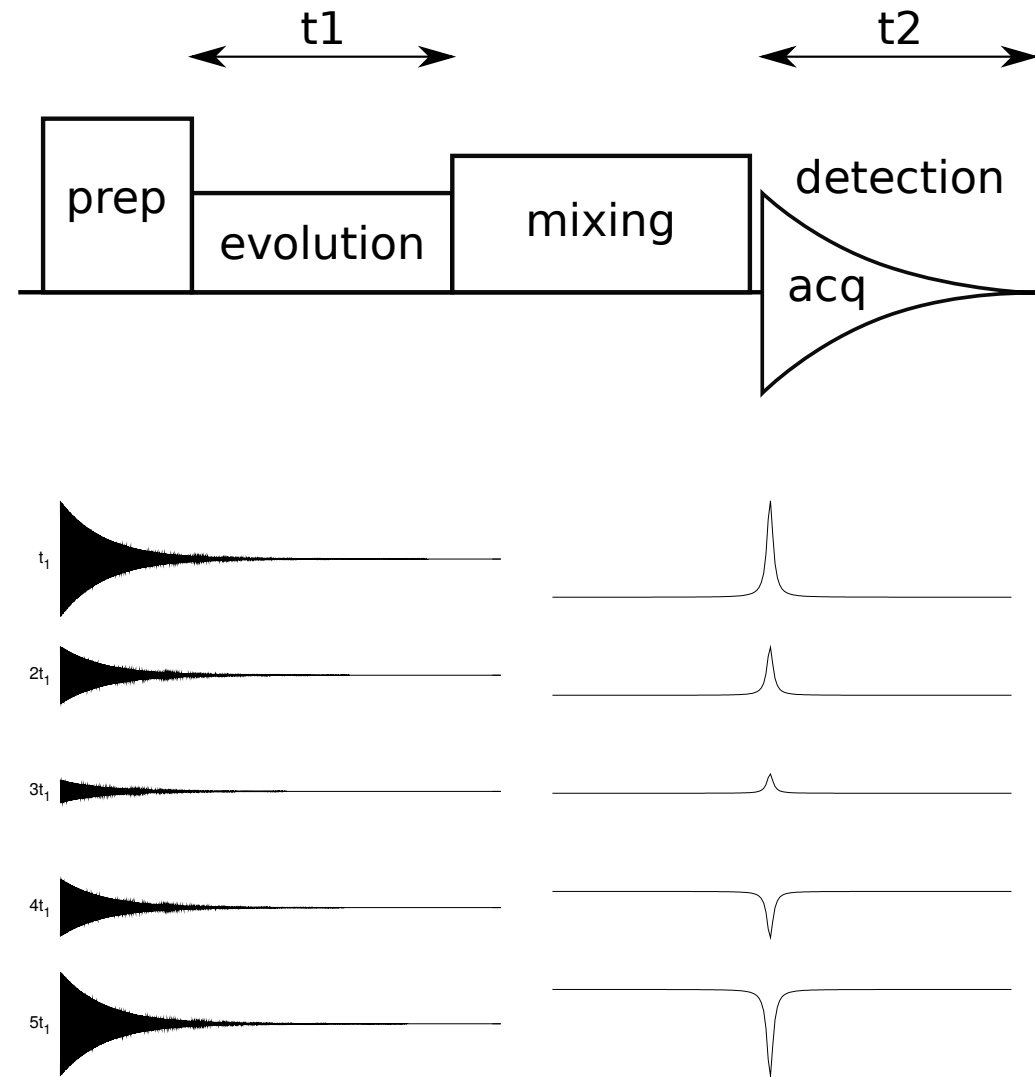
By using vector diagrams determine the result of attached pulse sequence. First realize what is the evolution of ^{13}C signal resulting from offset? CPD=composite pulse decoupling



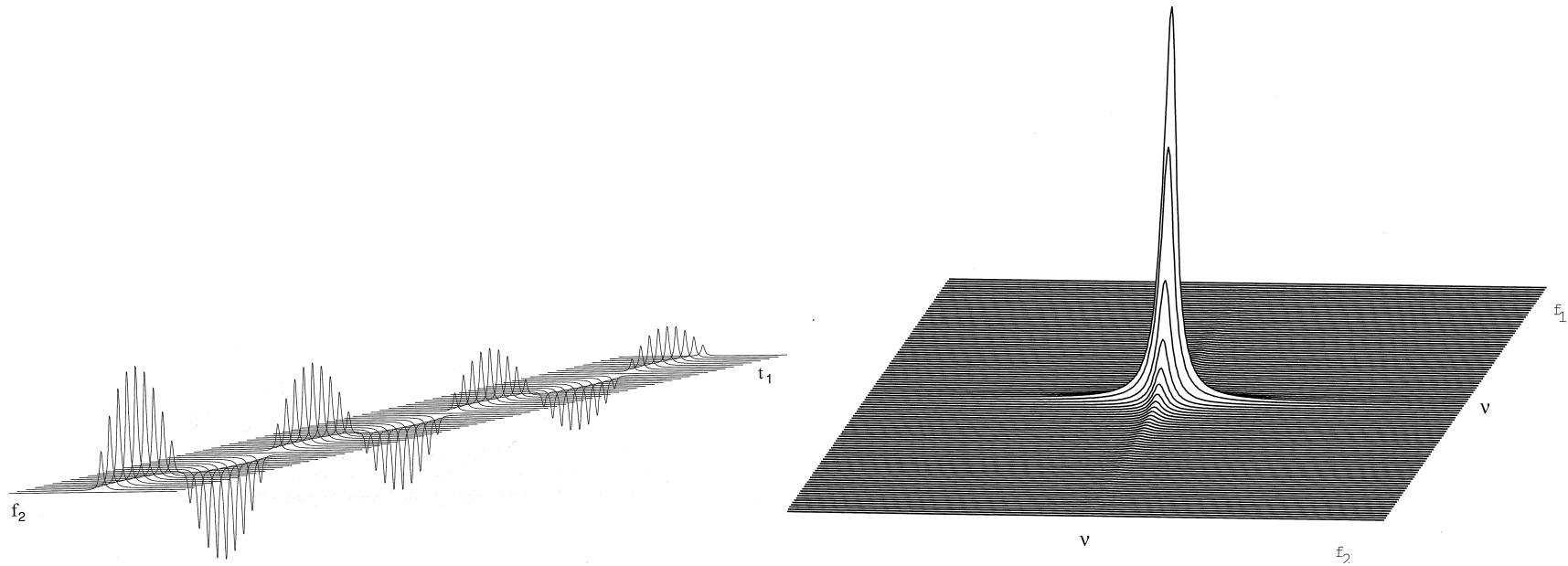
2D NMR

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

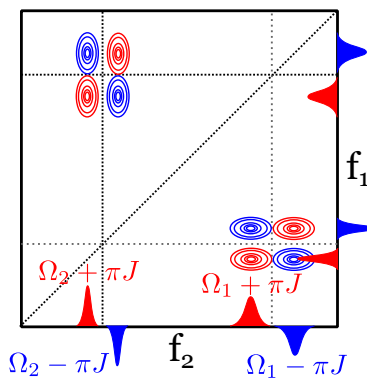
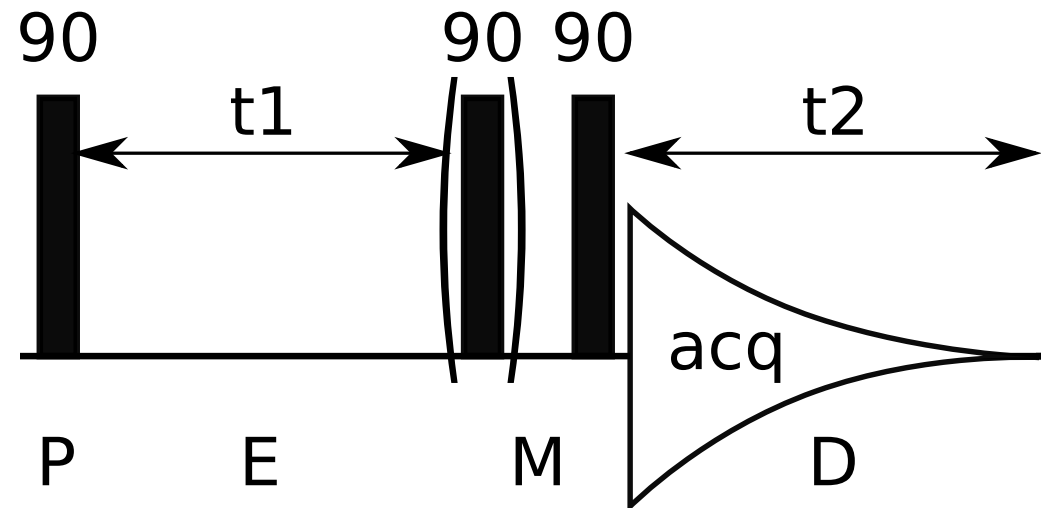


2D spektrum

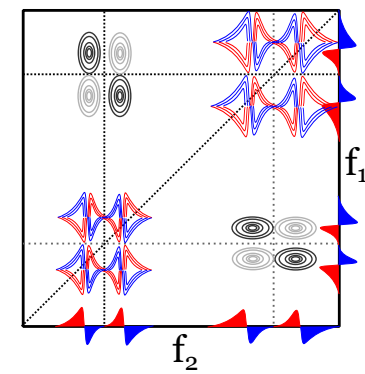
- ▶ FT in t_1 - modulated 1D spectra
- ▶ FT in t_2 - 2D spectrum

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



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

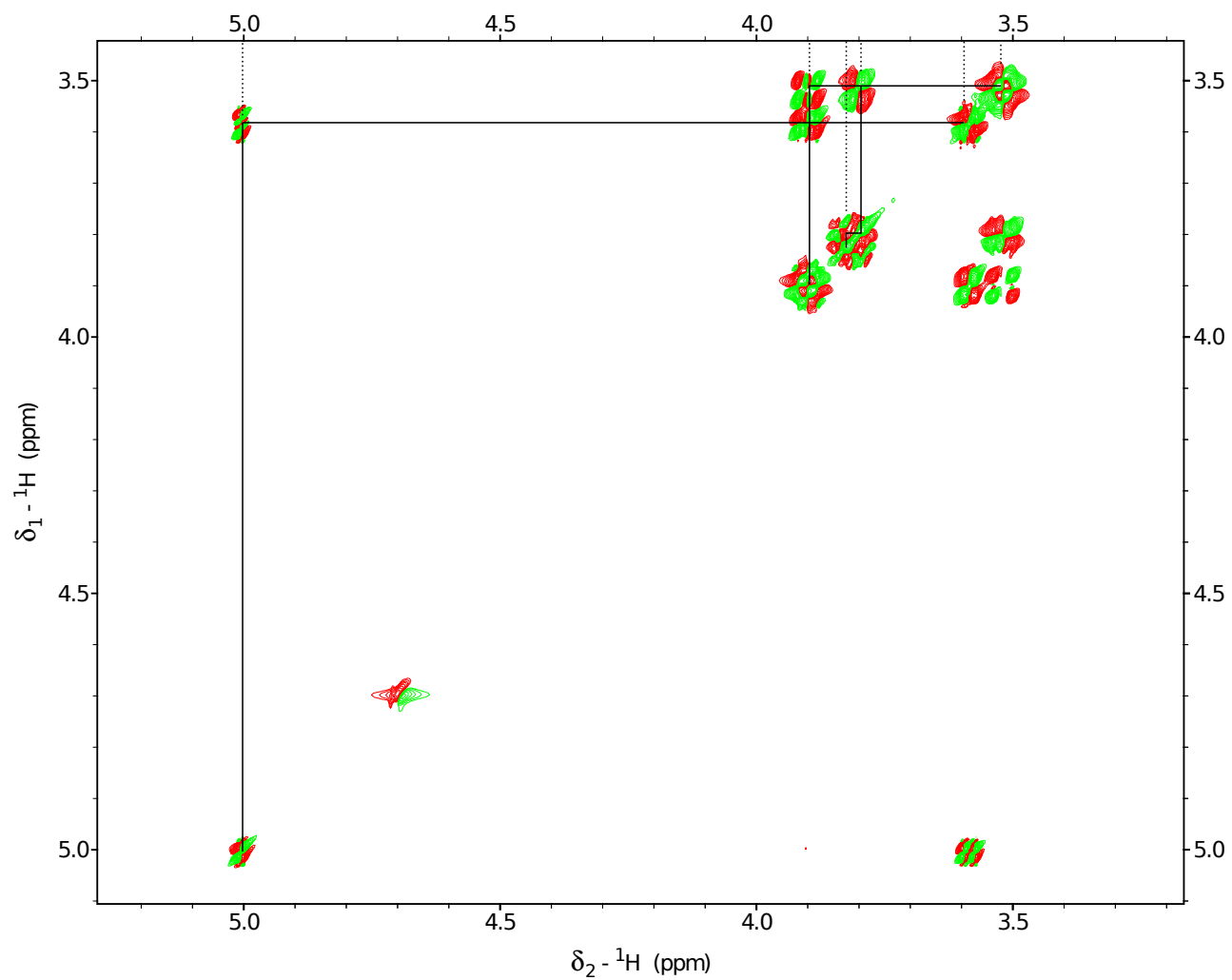
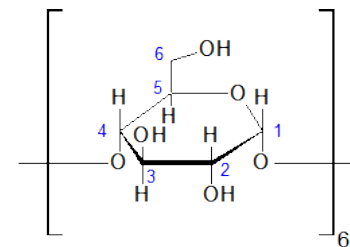


$$1/2[\sin(\Omega t_1 + \pi J t_1) + \sin(\Omega t_1 - \pi J t_1)]$$

Hints for beginners

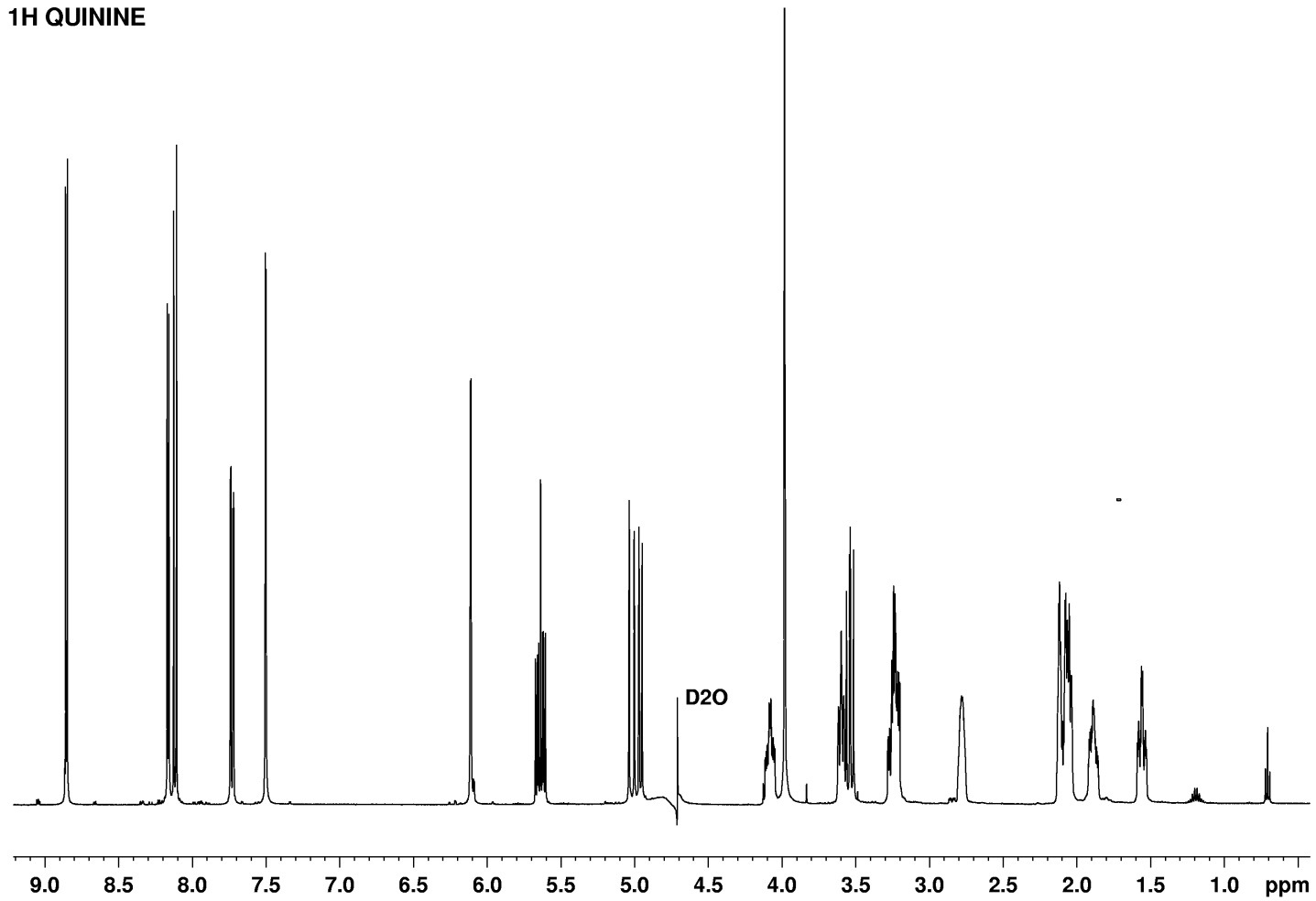
- ▶ Determination of **individual spin systems** - sharing **off-diagonal crosspeaks**
- ▶ Isolated protons - only diagonal crosspeak
- ▶ Already known rules: symmetry, diastereotopicity, most shielded/deshielded atoms etc.

COSY : β -cyclodextrine

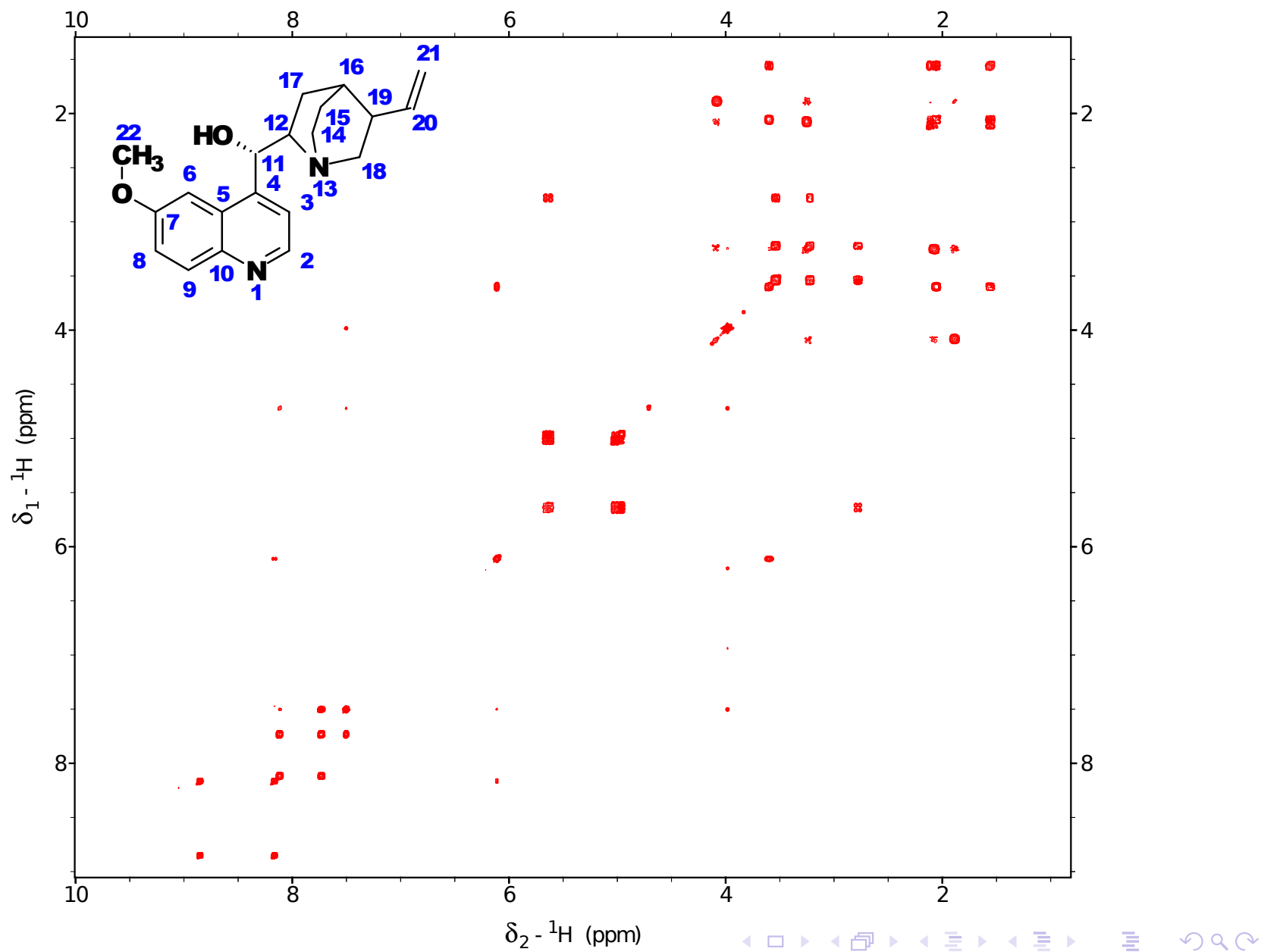


Chinin - 1D ^1H

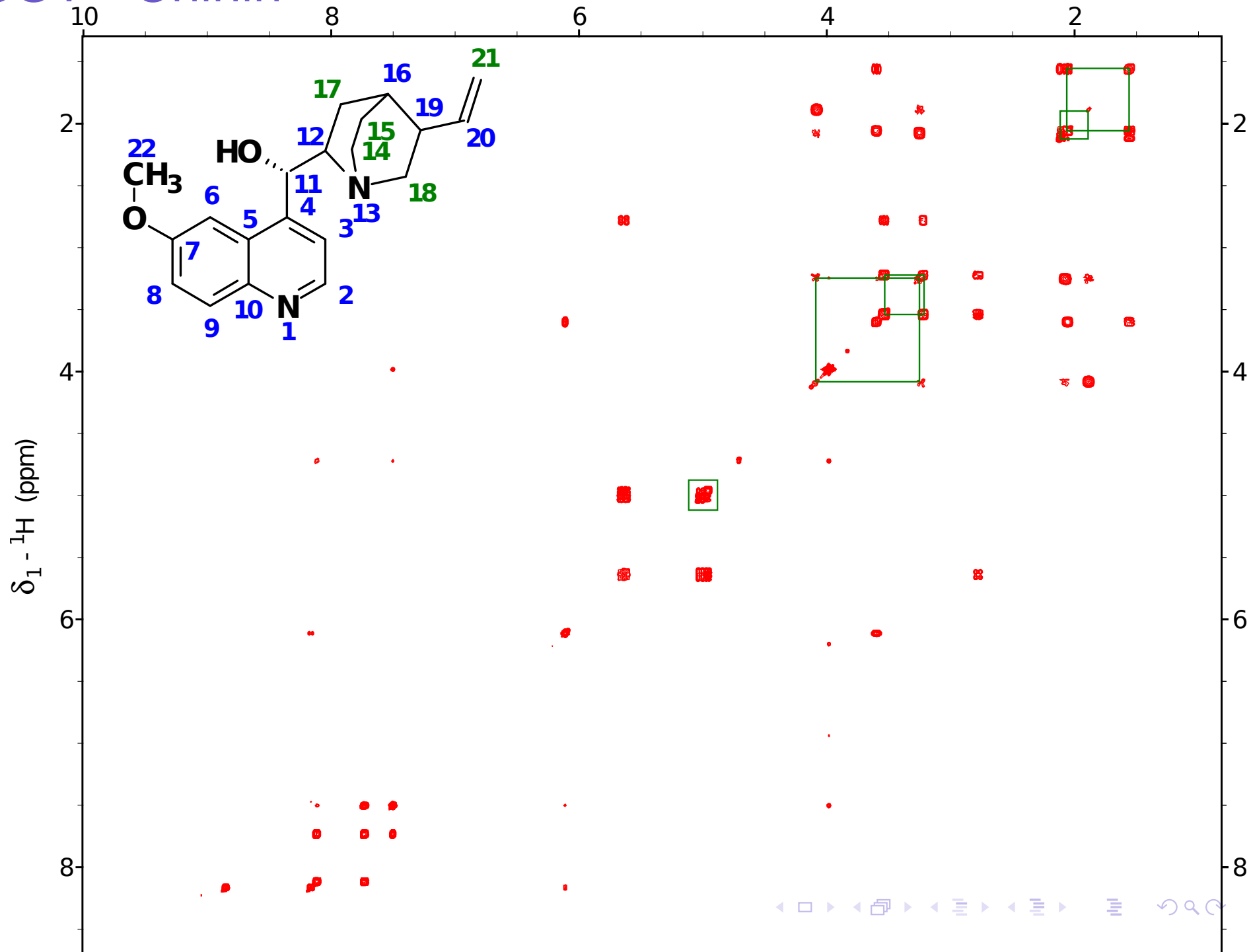
1H QUININE



COSY - Chinin



COSY - Chinin



Next topic

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