

C8953

NMR structural analysis - seminar

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

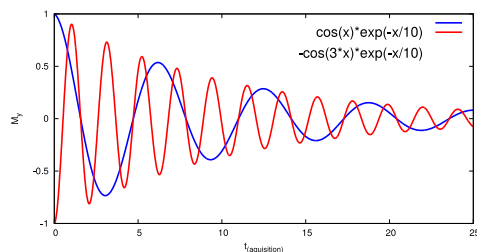
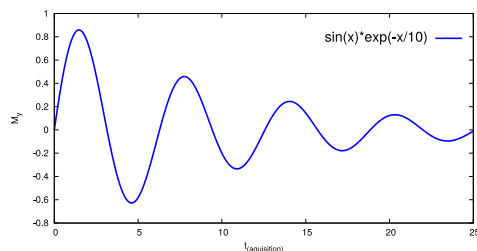
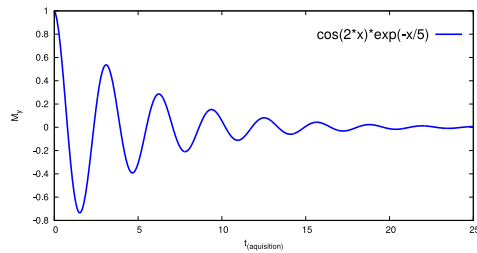
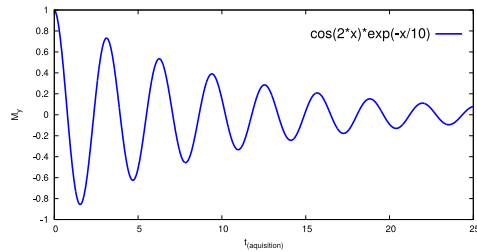
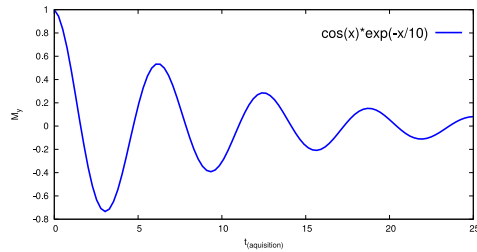
Jan Novotný

novotnyjan@mail.muni.cz

March 20, 2019

Basics of 1D FT spectroscopy

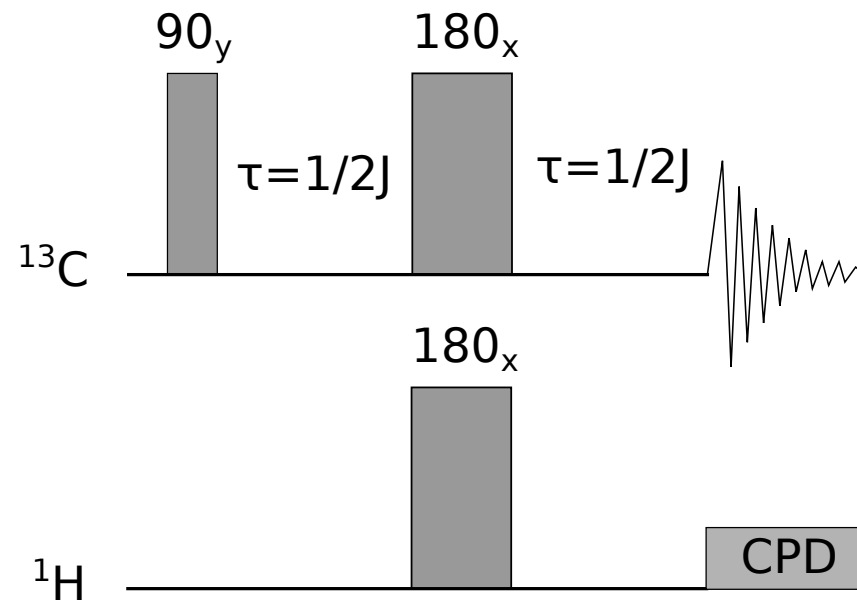
Draw FT representation of attached FID records (reciever is located in the $+y$ direction):



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)** $^{13}\text{C}-^1\text{H}_2$.



APT - Attached Proton Test

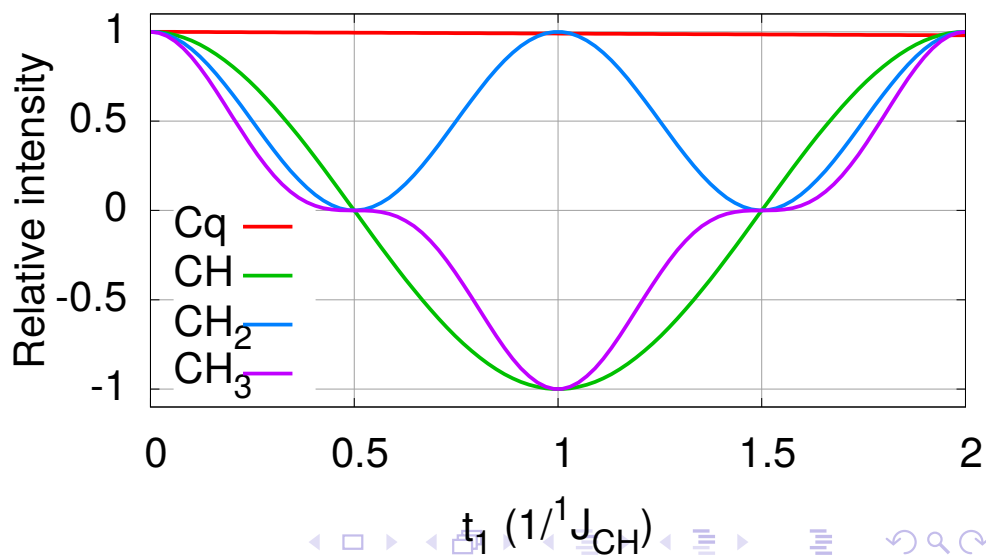
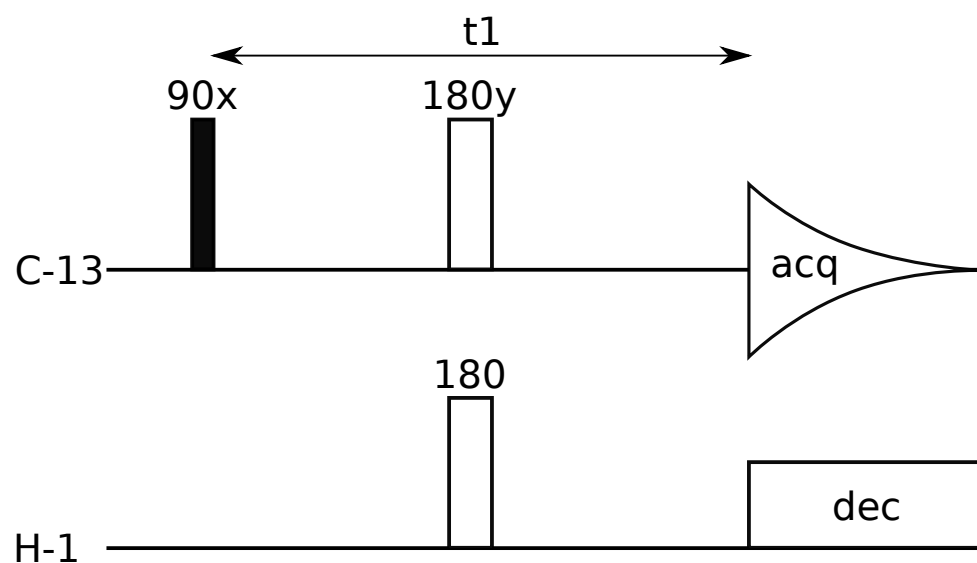
vychází z heteronukleárního spinového echa

▶ $t_1 = 1/{}^1J_{CH}$

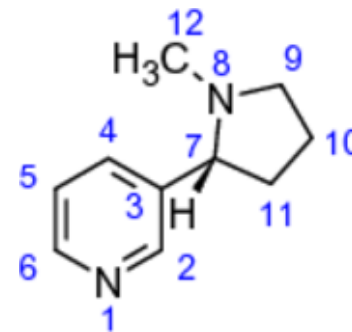
rozlišení signálů C podle počtu navázaných H

- ▶ Cq, CH₂ kladné
- ▶ CH, CH₃ záporné

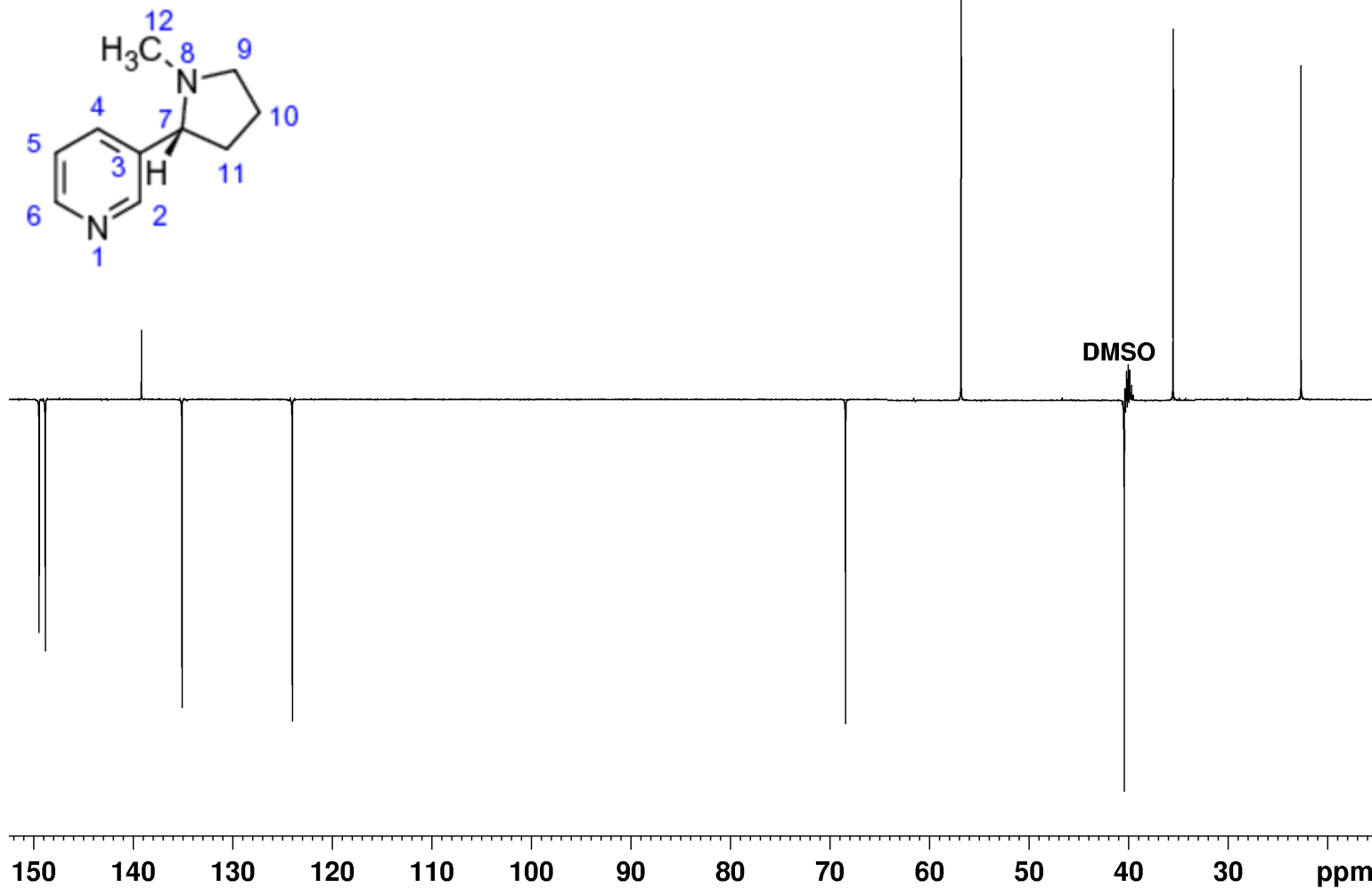
Rozdílná ${}^1J_{CH} \implies$ různé intenzity



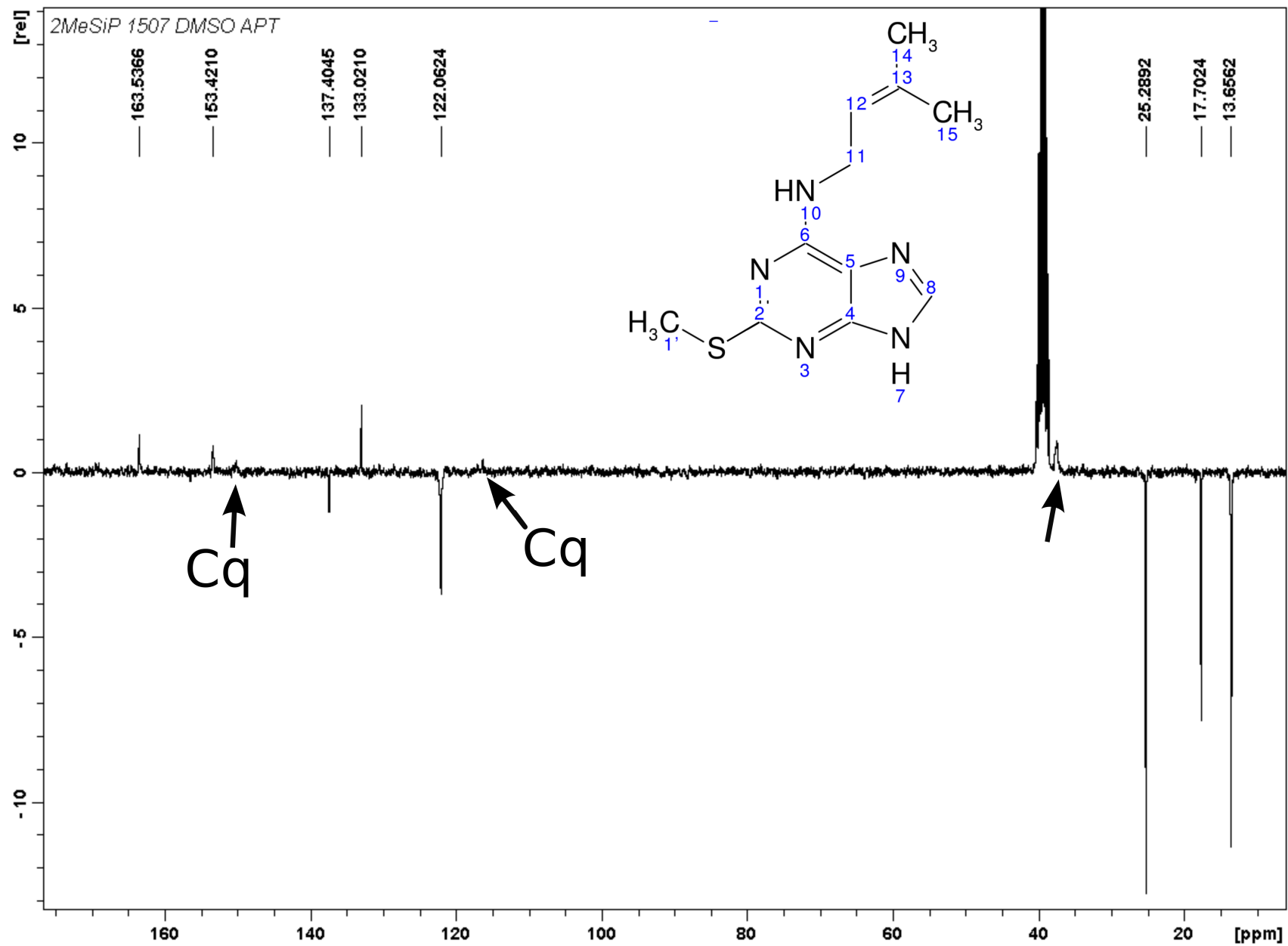
^{13}C APT Nicotine



^{13}C NICOTINE



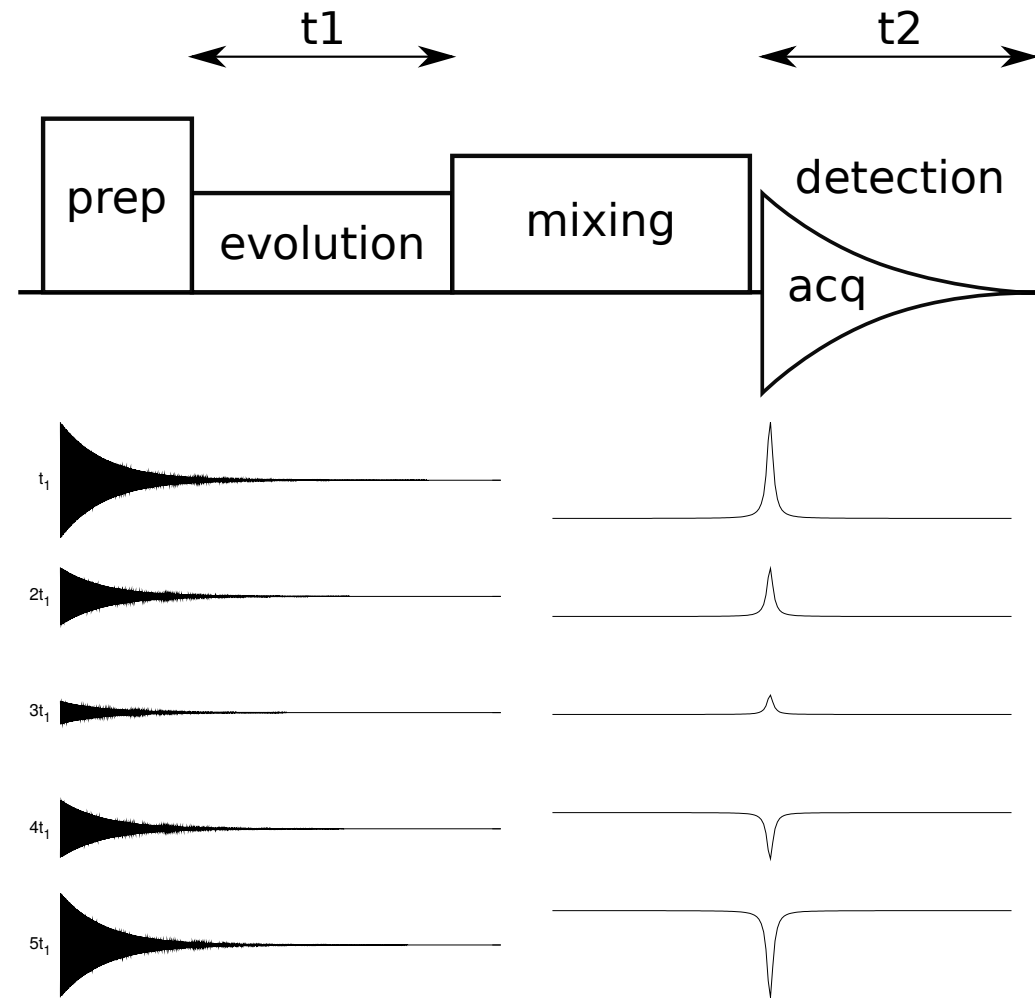
^{13}C APT 4



2D NMR

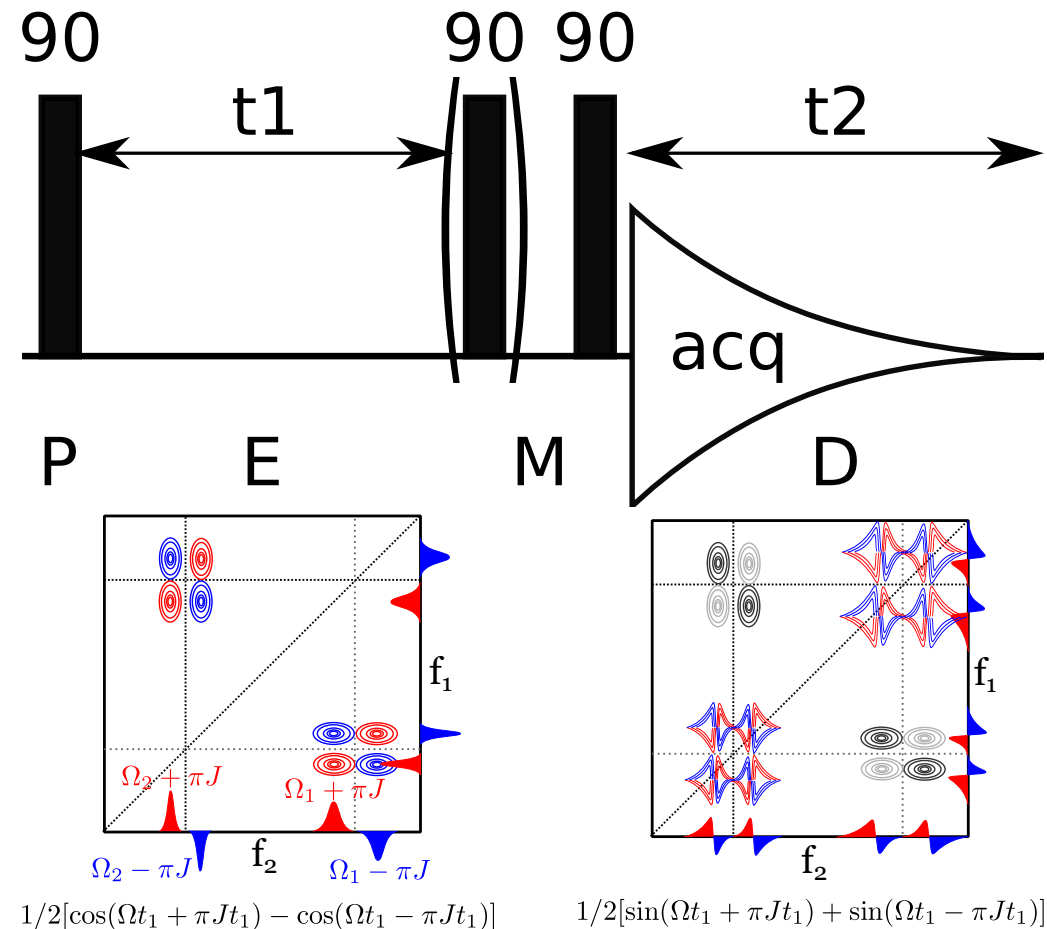
Second dimension f_1

- ▶ 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
 $t_2 \xrightarrow{\text{FT}} f_2$



COSY

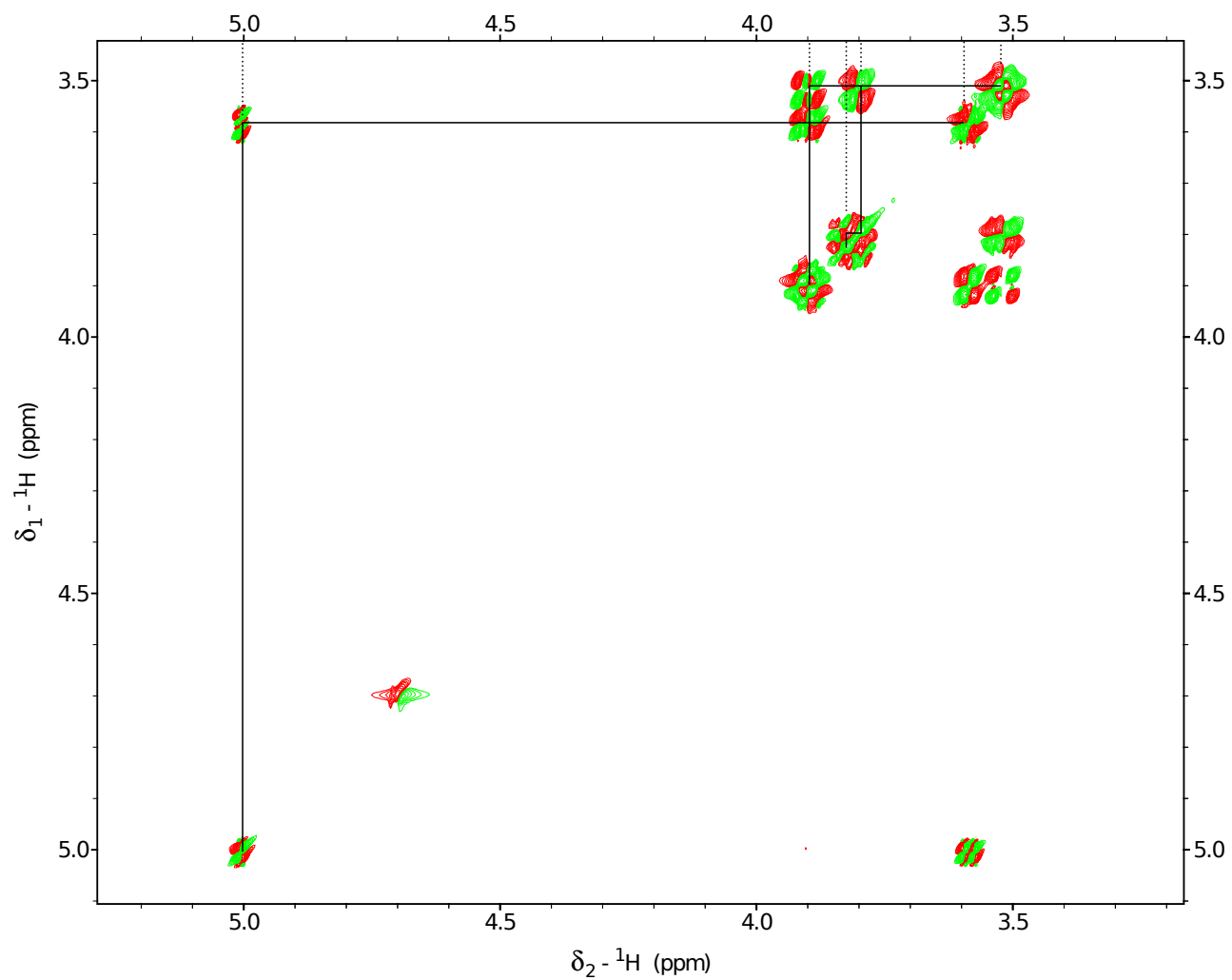
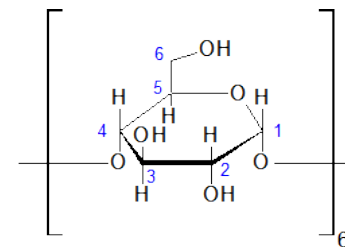
- ▶ easiest 2D experiment
- ▶ **correlates H nuclei based on $^2/3 J$ coupling**
- ▶ through 2, 3, (4) bonds
- ▶ antiphase off-diagonal crosspeak between coupled atoms
- ▶ DQF-COSY - modification of basic sequence, diagonal crosspeaks in absorption phase



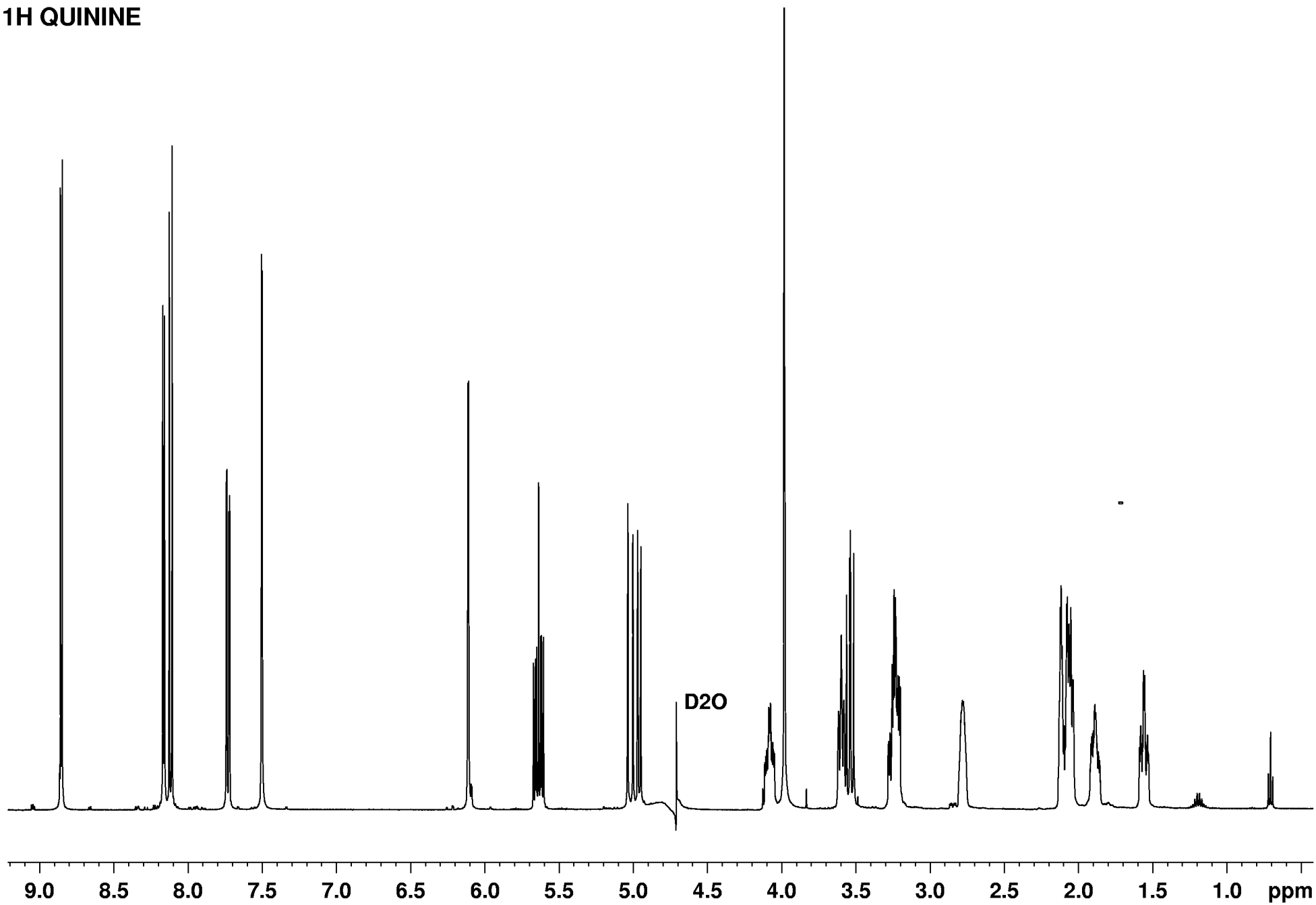
Hints for beginners

- ▶ 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



1H QUININE



COSY - Chinin

