C8953
NMR strukturní analýza
seminář

Introduction to heteronuclear correlations

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Polarization transfer

- bigger population diffrenece of ^{1}H nucleus is transfered via J-coupling to less sensitive nucleus X (^{13}C, ^{15}N)
- fundamental building block of heteronucler correlation experiments: in 2D-HX experiment each crosspeak manifests interaction of H and X nucleus coupled trough bonds
- **Task: Draw the evolution of magnetization during basic INEPT pulse sequence. Consider C-H interacting pair.**

HMQC (Heteronuclear Multiple Quantum Correlation)
HSQC (Heteronuclear Single Quantum Correlation)

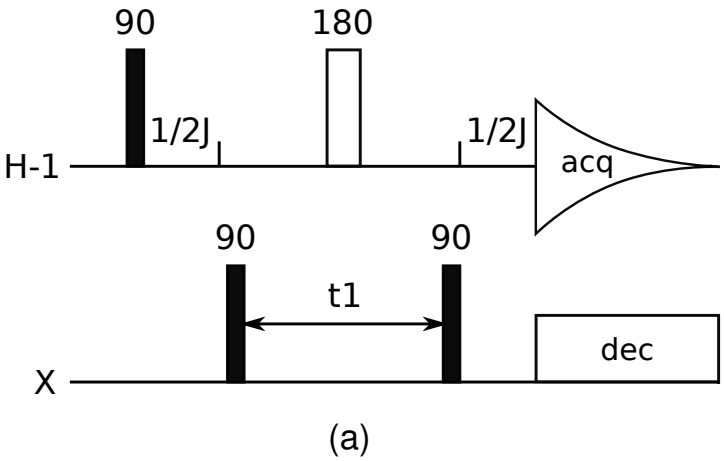
correlate ^{1}H-X (X=^{13}C, ^{15}N,...) based on $^{1}J_{HX}$

HMQC (a)

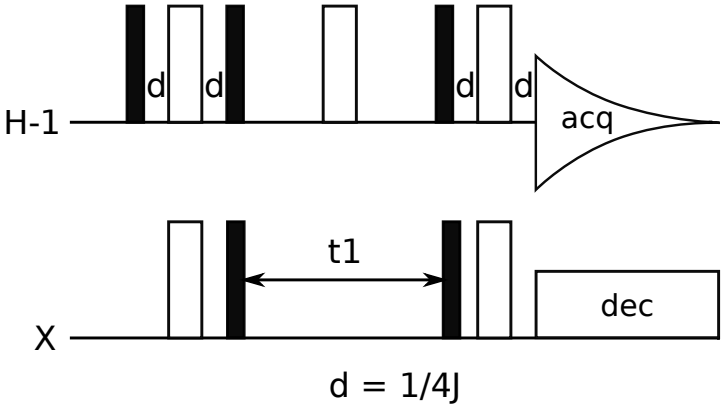
- \+ more robust experiment
- \+ change of parameters - HMBC
- \- lower sensitivity and worse resolution

HSQC (b)

- \+ better resolution, sensitivity
- \+ part of more complex mutlidimensional experiments
- \- less robust



(a)



d = 1/4J

(b)

Practical notes ^{1}H-X HSQC

- resolution of overlaps
- routine experiments to control biomolecular sample
- easy identification of geminal protons
- indirect determination of protons bonded to NMR inactive heteroatom
- heteronuclear correlation $\Rightarrow$ no diagonal crosspeak, no symmetry
- X decoupled during acquisition $\Rightarrow$ singlet crosspeak

HMBC(Heteronuclear Multiple-Bond Correlation)

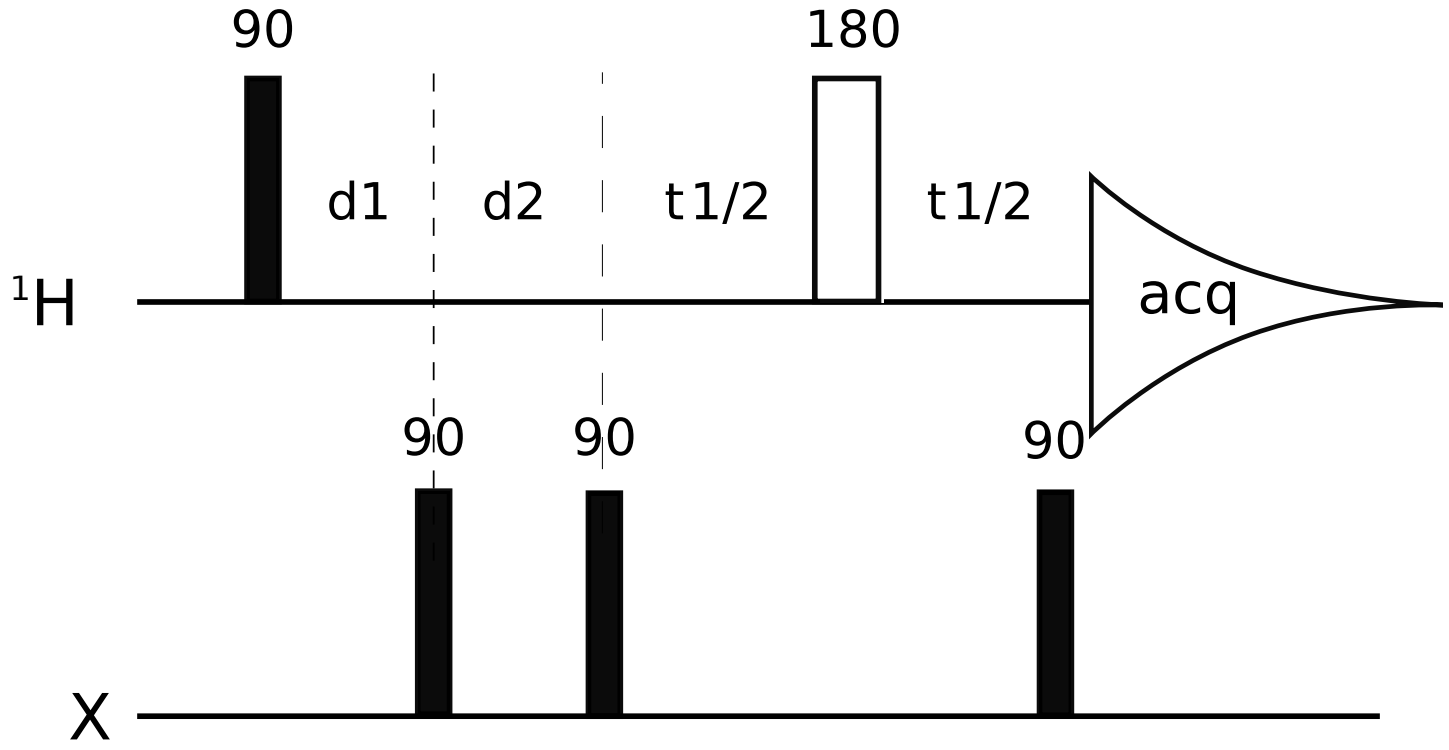
heteronuclear correlation based on long-range H-X spin-spin interaction(${}^{n}J_{HX}$, n>1)

- utilizes polarization transfer from H through 2-5 bonds on heteroatom (^{13}C, ^{15}N)
- allows to detect quaternary heteroatoms (Cq) or connect signals among isolated spin systems

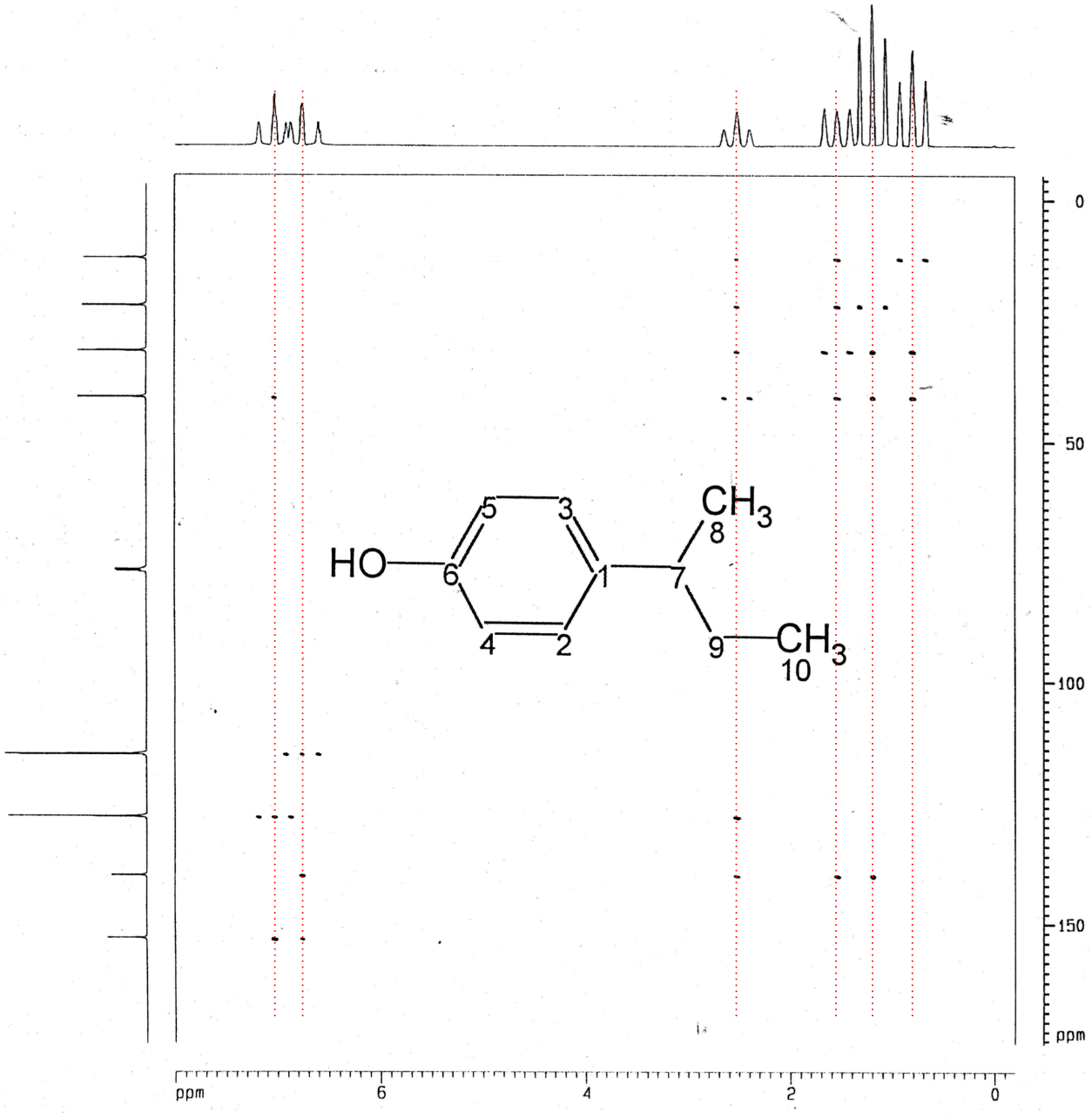
HMBC

correct settings of d1, d2 fo evolution of *J*-coupling necessary

- d1=$1/2 * {}^{1}J_{C-H}$ - (120-180 Hz)
- d2=$1/2 * {}^{2-5}J_{C-H}$ - (3-12 Hz)



^{1}H-^{13}C HMBC + ^{1}H-^{13}C HSQC



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