2D NMR

A correlation map between two NMR parameters

$\delta_I - \delta_I$

        COSY
        TOCSY
        NOESY
        ROESY, etc.

$\delta_I - \delta_S$

        HSQC
        HMBC, etc.

$\delta_I - J_{I,I}$

        homonuclear 2D-J

(and more… – only talking about the canned experiments here; more in Chm539 !)

COSY and TOCSY – based on resolved $J_{I,I}$ -coupling

COSY (COrrelated SpectroscopY):

Single-step magnetization transfer

Connectivities through 2 or 3 chemical bonds (rarely 4), depends on torsional angle

Antiphase cross-peak structure

TOCSY (Total Correlated SpectroscopY):

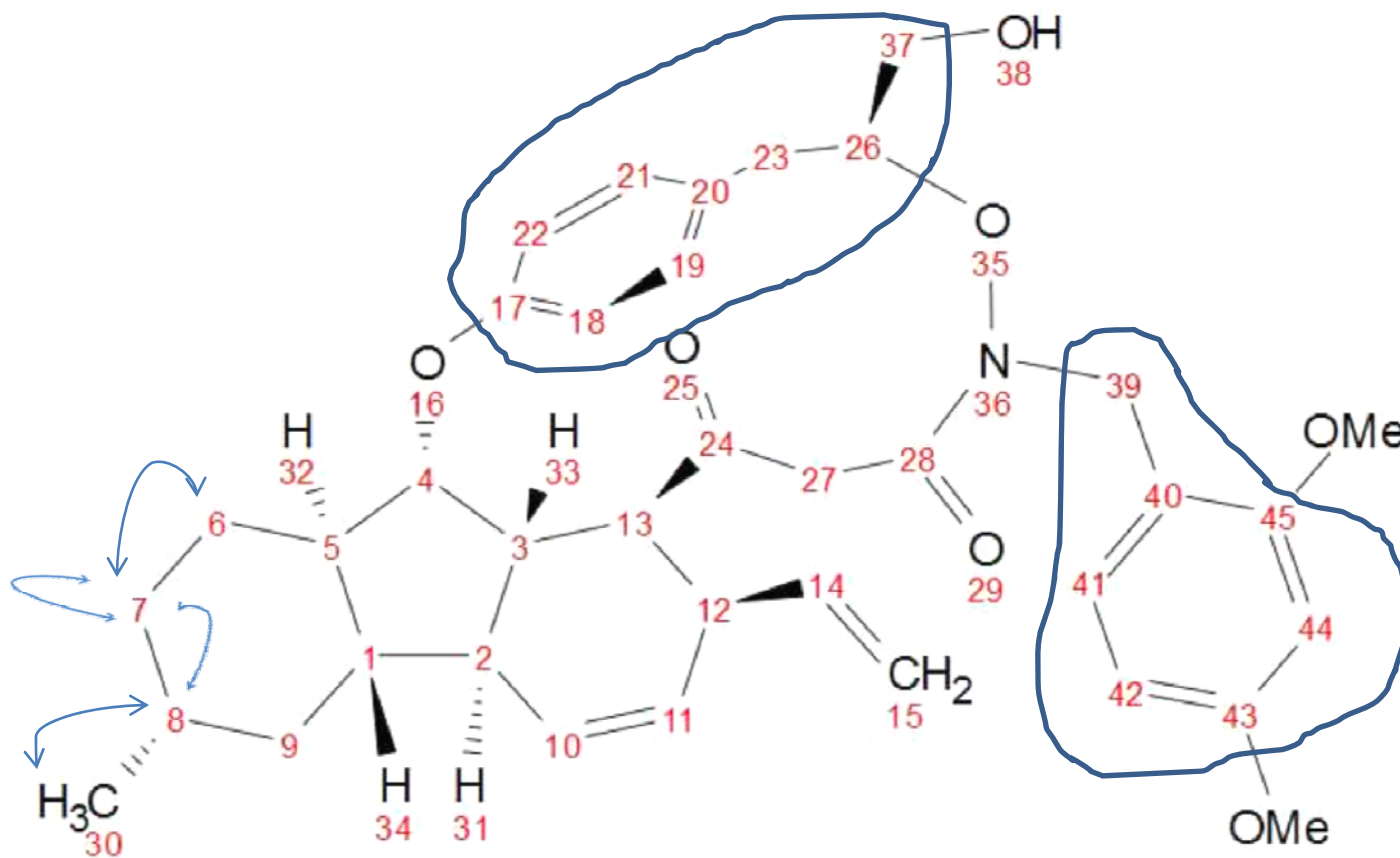Multiple-step magnetization transfer

Connectivities throughout the whole connected spin-topology (in ideal case)

In-phase cross-peak structure (enhances sensitivity)

COSY examples
TOCSY examples

NOESY and ROESY – based on spatial closeness (r^{-6})

NOESY (Nuclear Overhauser Enhancement
SpectroscopY):

Connectivities through distances up to 5-7 Å

+ or -, may be vanishing at certain mobility

ROESY (Rotating frame Overhauser
Enhancement SpectroscopY):

Same as NOESY, faster buildup, faster relaxation
losses

Always positive, may help to identify exchange

HSQC and HMBC – based on resolved $J_{I,S}$ -coupling

HSQC (Heteronuclear Single Quantum Coherence):

Connectivities through one chemical bond
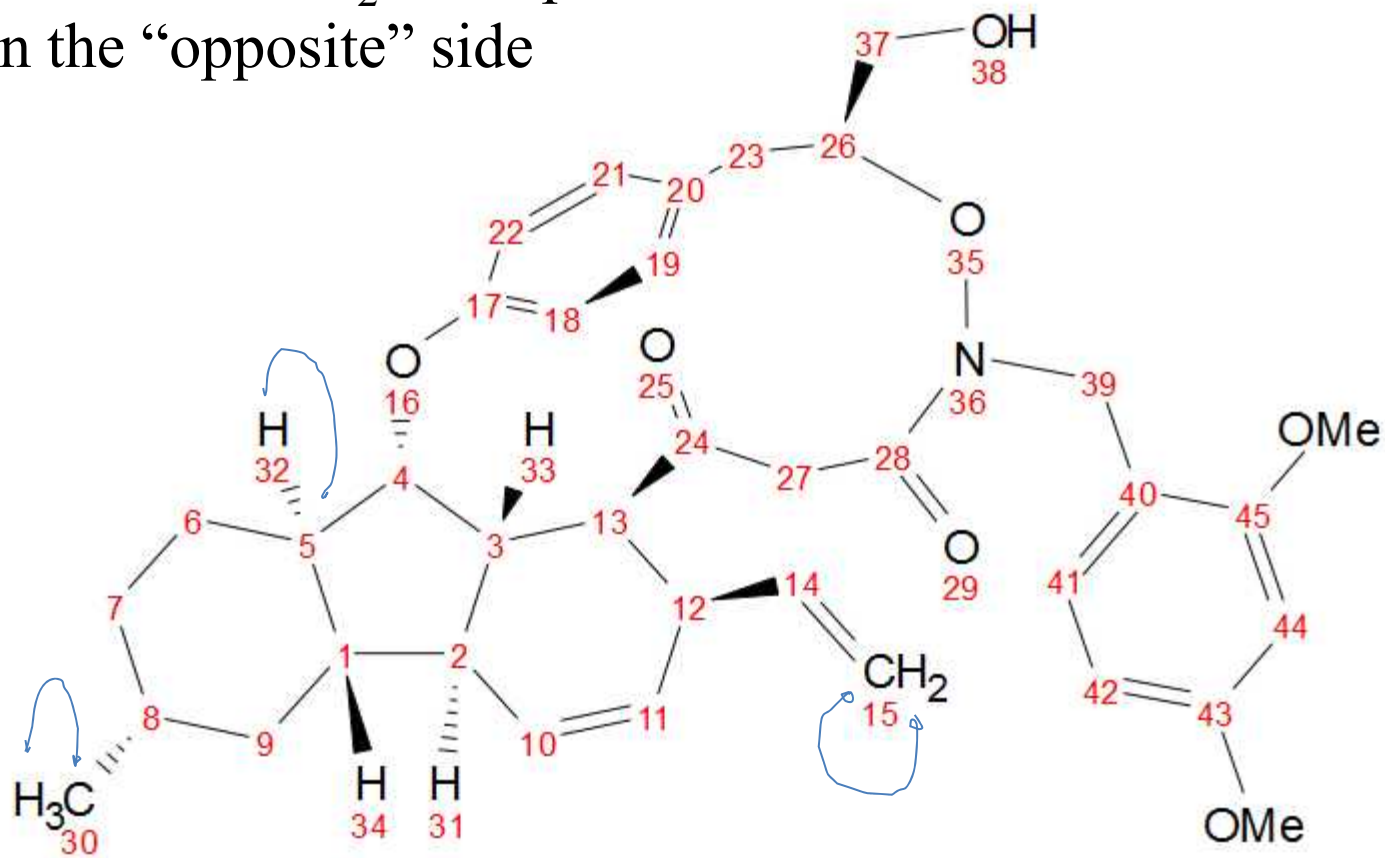
HMBC (Heteronuclear Multiple Bond Coherence):

Connectivities through 2-4(5) chemical bonds

[1]H-detected ; overall sensitivity depends on
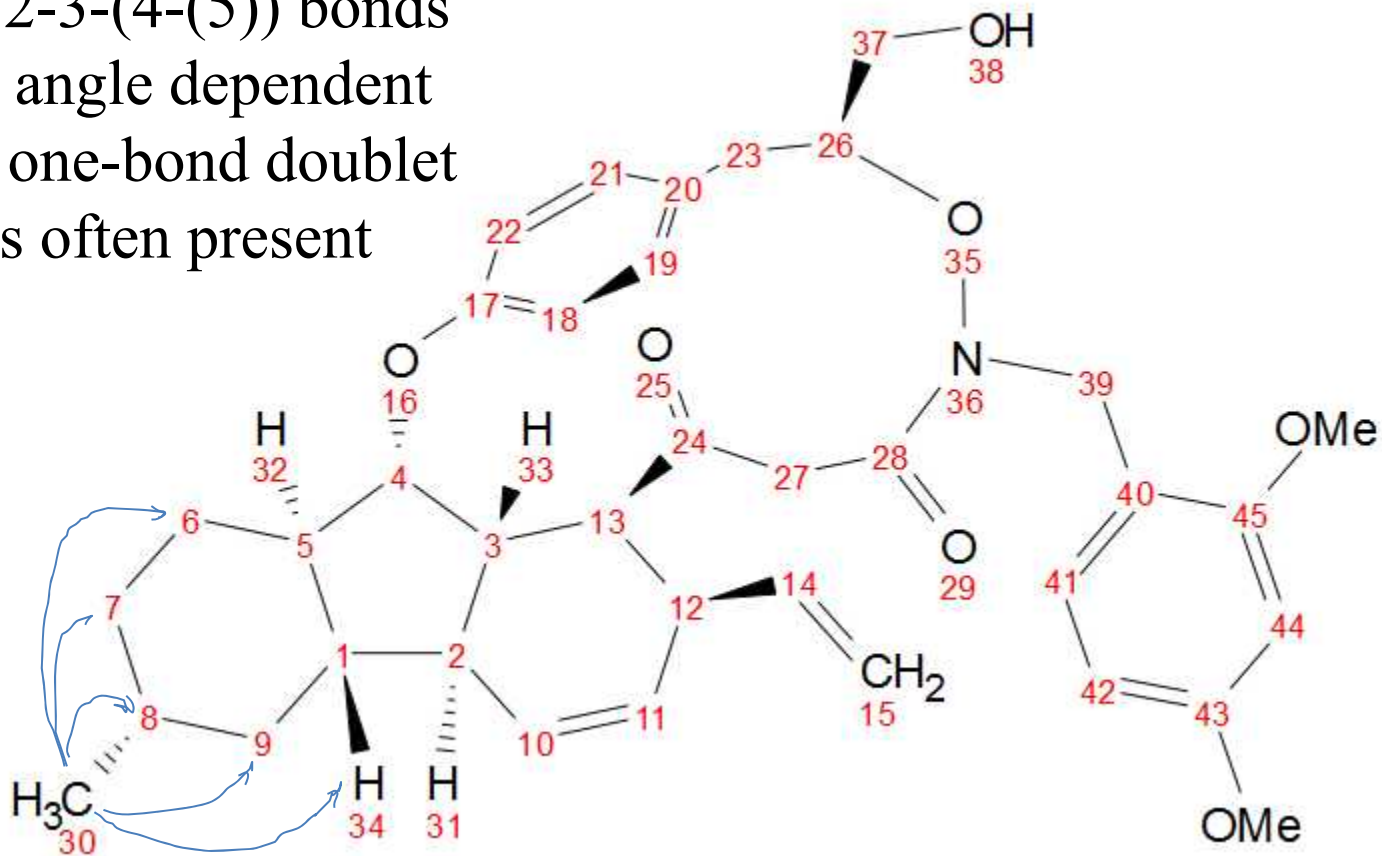[1]H sensitivity – A1 is best !

HSQC
DEPT-edited – CH$_2$ cross peaks
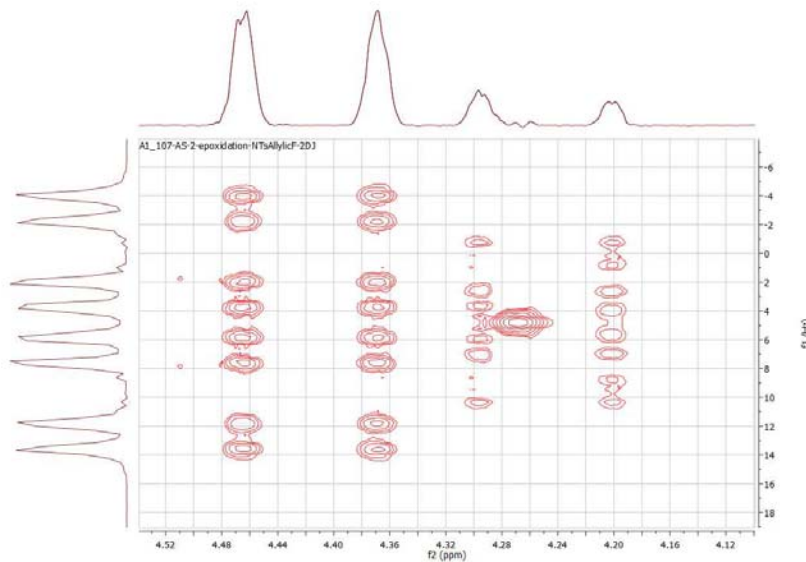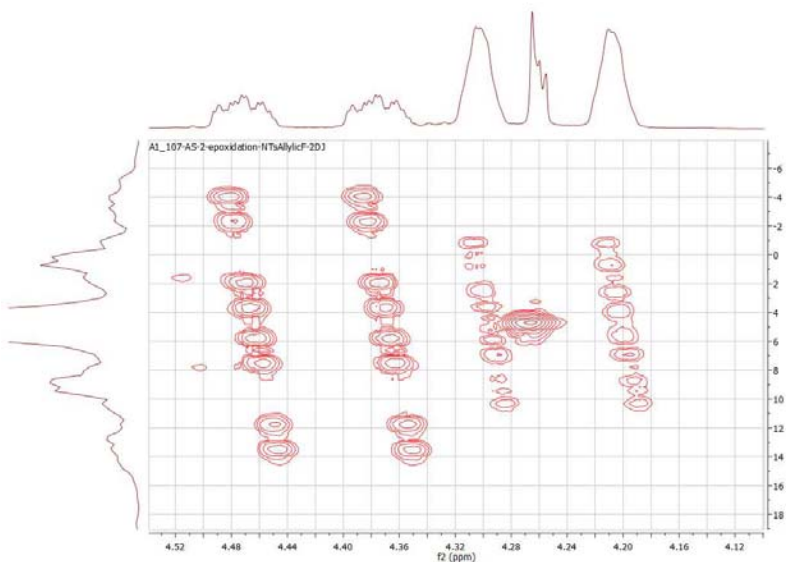are on the "opposite" side

HMBC
through 2-3-(4-(5)) bonds
dihedral angle dependent
residual one-bond doublet
      is often present

Homonuclear 2D-J-spectroscopy

Separates – after data processing – chemical shift and $J_{I,I}$ homonuclear coupling

Heterouclear coupling ($J_{I,S}$) remains in the f2 dimension

Default data structure, processing

	phase sensitive	magnitude
COSY		+
TOCSY	+	
NOESY	+	
ROESY	+	
HSQC	+	
HMBC		+
homonuclear 2D-J		+

(Gradient selected/assisted experiments.)

Default apodization parameters, first point correction

	f2	f1
COSY	Sin2, G5-15Hz	Sin, G15-25Hz
TOCSY*	Sin2/90, G5-15Hz	Sin/90, G15-25Hz
NOESY**	Sin2/90, G5-15Hz	Sin/90, G15-25Hz
ROESY**	Sin2/90, G5-15Hz	Sin/90, G15-25Hz
HSQC*	Sin2/90, G5-15Hz	Sin/90, G15-25Hz
HMBC	Sin2/90, G5-15Hz	Sin/90, G15-25Hz
homonuclear 2D-J	Sin2(/30), G1-3Hz	Sin, G1-1.5Hz

*   First point correction IS necessary in f1.

** NO first point correction in f1.

TOCSY, NOESY, ROESY – mixing/spin-lock time can be varied!

Phase correction

Automatic phase correction may work just fine…

In general:

>   move the Pivot point to the leftmost position
>   adjust PH0 (left mouse button) watching peaks
>   on the left only
>   adjust PH1 (right mouse button) watching peaks
>   on the right only
>   do it in few iterations…

Typical (approx.):	PH0	PH1
TOCSY :	90	0
NOESY :	90	180
ROESY :	0	180

Baseline correction

Polynomial, in both dimension

For phase sensitive spectra at the first place

Appearance, visualization options

In "Properties"

Contours, colors (red-blue)

Reference 1D-s, projections, slices