

# Table 1- Typical 2D NMR experiments used for molecular structure determination (\*)



Experiment	Typical quantity (mg)	Exp. Time*	What kind of information is obtained?
COSY	5 mg	5 to 30 min	Establishes correlation between spins <u>with scalar coupling</u> (J) but does not determine coupling constants.
Long-range COSY	5 mg	5 to 30 min	Emphasize correlation with <u>small Js</u> .
COSY-45	5 mg	5 to 30 min	Decreases the intensity of the diagonal peaks with respect to the correlation peaks, thus identifying correlation between strongly coupled spins.
DQ-COSY	5 mg	10 to 60 min	Establishes correlation between spins with scalar coupling (J); can be used to measure Js. Singlets are removed (CH <sub>3</sub> signals, for example) and solvent (H <sub>2</sub> O) by means of filtration of the correlation signals with a <u>double quantum</u> filter.
Relayed-COSY or RELAY	5 mg	5 to 30 min	Magnetization transfer to 1 or 2 chemical bonds beyond those of the COSY transfer to determine coupled spin systems (only via J).
TOCSY	5 mg	5 to 30 min	Show correlation among all the spins that have a common coupling partner, e.g., A → B → C, where J <sub>AB</sub> , J <sub>BC</sub> ≠ 0, <u>BUT</u> J <sub>AC</sub> = 0.
NOESY	10 mg	1 to 2 hours	Stereochemical information via <u>dipolar coupling</u> using cross-relaxation (longitudinal); determination of <u>chemical exchange</u> processes.
ROESY	10 mg	1 to 2 hours	Stereochemical information via <u>dipolar coupling</u> using cross-relaxation (transversal); adequate for molecules with average MW in the range of 1000-3000 and/or when $\omega\tau_c \sim 1.12$ (where $\omega$ is the spectrometer frequency and $\tau_c$ the correlation time).
HETCOR (1-bond)	20 mg	1 to 2 hours	Heteronuclear assignment
HMQC/HSQC (1 bond)	10 mg	0.5 to 2 hours	Heteronuclear assignment <u>using inverse detection</u> , i.e., using <sup>1</sup> Hs to detect heteronuclear frequencies (more often) or using a nucleus with larger ( to detect a low-( nucleus (e.g. use of <sup>19</sup> F to detect <sup>13</sup> C frequencies).
HETCOR (n-bond)	20 mg	4 to 12 hours	H-X <u>long range heteronuclear assignment</u> (via <sup>2</sup> J <sub>XH</sub> and <sup>3</sup> J <sub>XH</sub> ).
HMBC (n-bond)	10 mg	2 to 12 hours	H-X long range heteronuclear assignment (via <sup>2</sup> J <sub>XH</sub> and <sup>3</sup> J <sub>XH</sub> ) <u>using inverse detection</u> .
HMQC-TOCSY	10 mg	0.5 to 2 hours	H-X long range heteronuclear assignment (via <sup>2</sup> J <sub>XH</sub> e <sup>3</sup> J <sub>XH</sub> ) <u>using inverse detection</u> and <u>protonated Xs</u>
INADEQUATE	100 mg	24-72 hours	Establishes <sup>13</sup> C- <sup>13</sup> C connectivities. For structural elucidation of organic molecules, it is the most powerful experiment, but with the lowest sensitivity.

(\*) The acquisition times and quantities mentioned above are for phase-cycled 2D experiments, i.e., experiments that require a minimum number of transients in  $F_2$  to eliminate axial peaks and make quadrature detection. The experiments with pulsed field gradients tend to be faster than the phase-cycled ones.