NMR@Empa

Contact:

Daniel Rentsch

Mail: daniel.rentsch@empa.ch

Tel: +41 58 765 42 38

Roland Hany

roland.hany@empa.ch

+41 58 765 40 84

For related documents: make a search for “NMR@Empa”

A) Selection of NMR experiments in solution.

Experiment	**Amount of material required** [mmol]	**Comments**	
^{1}H; $^{13}C\{^{1}H\}$;^{19}F; $^{31}P\{^{1}H\}$ and many other NMR active nuclei	0.01 – 0.1	1D NMR spectra for the determination of chemical shift, of coupling constants, determination of purity, consistency with expected chemical structure.	1D
^{13}C DEPT-45/90/135	0.1	discrimination between signals from CH_2, CH and CH_3 groups	1D
^{1}H-^{1}H DQF-COSY	0.01	^{1}H,^{1}H correlation for the detection of binding in the neighbourhood network, assignment of signals	2D
^{1}H-^{1}H TOCSY	0.01	^{1}H-^{1}H correlation to identify related ^{1}H spin systems	2D
^{1}H-^{1}H J-RES	0.01	^{1}H-^{1}H correlation to separate chemical shift and coupling information	
^{1}H-^{13}C HSQC ^{1}H-^{15}N HSQC	0.05 – 0.2	^{1}H-^{13}C or ^{1}H-^{15}N correlation of ^{13}C or ^{15}N signals with signals of the directly bonded protons	2D
^{1}H,^{13}C-HMBC ^{1}H-^{15}N HMBC	0.05 – 0.2	^{1}H-^{13}C or ^{1}H-^{15}N correlation of ^{13}C or ^{15}N signals with signals of remote protons (coupled via 2-3 bonds)	2D
^{1}H-^{13}C HSQC-TOCSY	0.05 – 0.2	^{1}H,^{13}C correlation of all ^{1}H to all ^{13}C nuclei in the same spin system	2D
1D or 2D NOESY 1D or 2D ROESY	0.01	proof of spatial proximity. (NOE or ROE at distances < 5Å)	1D 2D
D_2O- und CD_3OD-exchange	0.01	detection of exchangeable protons (OH, NH, etc.)	1D
homonuclear decoupling	0.01	assignment of signals by simplifying the spectra, determination of coupling constants.	1D
temperature experiments	0.01 – 0.2	investigation of dynamic processes (conformational equilibria, chemical exchange).	1D

^{1}H diffusion-edited NMR-spectra	0.01	depending on the size of the diffusion constants of the individual components in the mixture ^{1}H NMR signals are selectively suppressed (proof whether functional groups are polymer-bonded or "free" in solution).	1D 2D
EXSY	0.01 – 0.2	detection of chemical exchange in the time zone of ms to s	2D
^{1}H-^{1}H ECOSY	0.01	^{1}H,^{1}H correlation for the determination of ^{1}H,^{1}H coupling constants	2D
^{1}H-^{13}C HSQC-HECADE ^{1}H-^{13}C J-HMBC	0.1	determination of ^{1}H,^{13}C-coupling constants	2D
1,1-ADEQUATE	> 0.1	^{1}H-^{13}C Correlation over ^{1}H-^{13}C- and ^{13}C-^{13}C 1J coupling	2D
^{13}C-^{13}C INADEQUATE	> 0.2	^{13}C-^{13}C Correlation ^{13}C-^{13}C 1J coupling	2D

B) NMR experiments on the HR-MAS NMR equipment.
For swellable material we have a special equipment to record “solution state like” NMR spectra.

- Required approx. 10-30 mg material
- The compound must be swellable in a deuterated solvent
- NMR spectra are recorded in 4 mm HR-MAS NMR rotors with deuterium lock under MAS rotation (up to 5 kHz)
- The probe is designed for ^{1}H and ^{13}C NMR experiments (X channel not tuneable to other nuclei far away from the carbon frequency)
- In principle all 1D and 2D ^{1}H / ^{13}C NMR experiments described in the “solution state NMR section” (see above) can be performed on the HR-MAS NMR probe
- In comparison to solution state NMR spectra, HR-MAS NMR spectra generally show a lower resolution

C) Selection of NMR experiments in the solid state.
For special problems further NMR experiments can be performed or implemented.

- Required amount of compound: 2.5 mm MAS probe (approx. 15 mg), 4 mm MAS probe (approx. 100 mg), 7 mm MAS probe (approx. 300 mg)
- Max. spin rates: 2.5 mm MAS probe (approx. 35 kHz), 4 mm MAS probe (approx. 15 kHz), 7 mm MAS probe (approx. 6 kHz)
- With our solid state NMR equipment we are NOT able to record ^{19}F NMR spectra

Experiment	probe (Outer Diameter [mm] of spinner)	**Comment**	
^{1}H, ^{7}Li, ^{11}B, ^{31}P… and all other nuclei with	2.5 / 4 / 7	single pulse experiments (ZG or HPDEC), CP-MAS, determination of relaxation time etc.	1D

high sensitivity			
^{11}B, ^{13}C, ^{29}Si… and other NMR active nuclei also with lower sensitivity	(ev. 4) / 7	single pulse experiments (ZG or HPDEC), CP-MAS, determination of relaxation time etc.	1D
^{11}B, ^{27}Al (and other quad-rupolar nuclei)	2.5	single pulse experiments, MQMAS	1D+2D