Probe Sensitivity - G-NMR Solid State Subgroup

Motivation:

Sensitivity is one of the largest problems in Solid State NMR. In addition to the external field, sensitivity depends crucially on the probe. Probe design itself contributes to this but it is often not clear how much. Even probes of the same design seem to differ considerably. To identify "bad" probes which are outperformed by many other is important to be able to address this problem.

Naturally, which sensitivity is important depends on the research of interest. One of the most used nucleus in Solid State NMR in ^{13}C. Therefore, here a protocol for determination of ^{13}C sensitivity is suggested. In addition, double CP performance (NCA in peptides) is a very crucial parameter for most biomolecular research and should be collected by the groups involved in this type of research.

Procedure:

Every participating lab should collect these test spectra for all their probes. The results should be submitted to

Johanna Baldus, BMRZ Uni Frankfurt, j.baldus@em.uni-frankfurt.de, 069 798 29930

After submitting the data, a list with all the available data will be provided on a confidential basis.

Protocol S/N on Glycine:

- Sample: α-Glycine (recrystallize from water), rotor as full as possible, no inserts, weigh the amount of sample used.
- Probe is well shimmed and MAS is set
- 10 kHz MAS, 290K set temperature
- ^{1}H 90: 2.5 µs
- Decoupling: 100 kHz Spinal64 decoupling optimized for length of decoupling pulses and ^{1}H offset
- Spectral width / Acquisition time: Use 300 ppm with 2k points as used by Bruker. (Or longer aq and truncate what would correspond to the above.) Centre near $^{13}C\alpha$.
- CP: 2ms contact time, 62.5 kHz on $^{13}C\alpha$ with a 80-100ramp on ^{1}H, ^{1}H power optimized to around 70 kHz
- If a probe cannot take any of the powers suitable reduced values should be used and commented
- 16 dummy scans 64 scans and 5 s recycle delay

- on Bruker instruments: AQ_mod: DQD, DIGMOD: digital
- Analysis: Do base line correction, FT without line broadening chose 20 ppm of noise, divide by 4 to get Signal to Noise with 4 scans as used by Bruker (does the way S/N is measured on Varian/Agilant/Joel spectrometers differ from Bruker?).
- report: S/N, $^{13}C\alpha$ -line width at half peak height, amount of sample used, things that were for some reason different to the standard protocol, probe used (Manufacture, rotor size, B0)

RESULTS TABLE FOR S/N MEASURMENTS ON GLYCINE

Facility	Field (MHz)	Console	Probe	Amount of Glycine packed	S/N of CA normalized to 4 scans	Line Width of CA in Hz	Comments (e.g. deviation from protocol)	Contact Person
BMRZ Frankfurt	600 WB	AV I	Bruker 4mm HX	122 mg				J. Baldus

Protocol: DCP efficiency:

- Sample: Any dry crystal with an amide bond with u^{13}C-^{15}N labelling, e.g. AcVal, AcValLeu, AGG, MLF, Fmoc-Gly ... (not Glycine, no wet protein)
- Optimize ^{13}C for $^{13}C\alpha$ -signal(s).
- Optimize DCP for NCA transfer (chose whatever pulse works best for you, ramp, adiabatic, ...)
- Use same acquisition parameters for ^{13}C and DCP Experiment
- Compare $^{13}C\alpha$ -signal intensity
- report: ratio $^{13}C\alpha$ -signal/^{13}C-signal, DCP parameters, probe used (Manufacture, rotor size, B0)

RESULTS TABLE FOR DCP TESTS

Facility	Field (MHz)	Console	Probe	Sample and packing (centre or full)	NCA signal/CA signal	DCP method (e.g. linear ramp, adiabatic)	Comments (e.g. deviation from protocol)	Contact Person
BMRZ Frankfurt	400 WB	AV II	Bruker 3.2mm HCN Cryo					J. Baldus