Acceptance Solid State NMR Test Procedure

for Avance NMR Systems

Manual P/N B92999

Acceptance Solid State NMR Test Procedures

for AVANCE systems

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1. Purpose

This manual describes the procedures for the NMR tests that are necessary to set up Solid State NMR MAS experiments in order to demonstrate the high performance of Bruker Avance Instruments. It includes basic specification tests such as ^{13}C resolution (line width at half height) and sensitivity tests, as well as some advice on shimming MAS probes. More advanced experiments and their setup can be found in the help menu of XWIN-NMR (other topics - solids users' manual) or using NMR-Guide.

All test procedures are subject to change without notice.

2. Area of application

This manual is for all Bruker Service personnel engaged in final testing, service and installation of NMR instruments, as well as for customers. The manual is a complement to the Acceptance and Test Procedures for Avance NMR systems, ZUEP0102. The manual complements the solids part of the ATP program and can be used only for systems with the software Release XWIN-NMR 3.1 patch level 11 and higher with AQS electronics.
Note: The CP pulse programs and related parameter sets do not work on systems with AQX/AQR electronics. Pulse programs for D*X systems are available in the software release under '`<Xwin-nmrhome>/exp/stan/nmr/lists/pp.dsolids'`. Appropriate adjustments to the parameter sets need to be done as well.

3. Referenced documents

ZUEP0102   Acceptance test procedures for Avance spectrometers
               and documents referenced in ZUEP0102
H9321   Solids user manual
Z31401   SB MAS Operation Manual
B3072   SB Wideline Probe Manual
Bxxx   HR-MAS Standard Test Procedures

4. NMR experiments for probe tests

4.1   Introduction

4.1.1 B_0-Field Adjustment, Referencing

Solid-state NMR probes have no ^{2}H lock channel. As a result, referencing is done for each nucleus with standard samples. For a general spectrometer setup one basic referencing is needed and one convenient standard sample is used. The built in frequency list - following R.K. Harris IUPAC recommendations - guarantees appropriate cross-referencing.

The general spectrometer referencing is done using the most convenient sample and related nucleus. If, for example, one uses the ^{1}H resonance of water or TMS if available, the liquid sample is centered in a MAS rotor as outlined below.

The parameter set **ZGSOLIDS** is loaded, using **rpar ZGSOLIS all** in the command line of XWIN-NMR. The nucleus ^{1}H is selected either in the **eda** or in the **edsp** editor. Before starting, make sure that in **eda** the parameter **locnuc** is set to *off*, and that neither the lock nor the sweep buttons on the BSMS keypad are lit. If no keypad is available, open the BSMS display/lock window and check the state of field sweep and lock. Both must be off.

The ^{1}H transmitter offset O1P is set to 4.7 ppm, temperature should be around room temperature, furthermore, a recycling delay **d1** = 0.5 seconds, **td** = 4096 and **sw** = 20ppm is chosen. For excitation one uses a power level of **pl1** = 10 dB and a pulse length **p1** = 5 us. The interactive experiment is started by typing **gs** into the command line. In order to observe the signal, switch to the acquisition window by typing **acqu**. Dial the field (BSMS keyboard or BSMS display / shim window) while pulsing, until the FID is on resonance. Then the experiment is stopped by typing **stop** and an acquisition is started with **ns** =1 and **ds** = 0, by typing **zg** into the command line. The signal is processed with **ft** and appropriately phased.   When TMS is used in the sample, change the BSMS field until the TMS signal is exactly on Zero the Zero frequency position.

Sample	Nucleus	Shift (ppm)	
Water	^{1}H	4.7	
TMS	^{1}H	0	
KBr	^{79}Br	About 60 ppm	R.K. Harris IUPAC recommendation, updated in TOPSPIN release from XWINNMR 3.1 where it was 41.1ppm
Adamantane	^{13}C	29.5 38.56	(Earl et al. JMR 48, 50 (1982))
TTMSS	^{29}Si	-8.9	
Glycine	^{13}C	176 (Carbonyl line)	

Table 1: Some chemical shifts that one can use for referencing.

The procedure has the advantage of allowing the spectrometer frequencies in the parameter sets to be used more easily on other spectrometers (if these spectrometers are referenced as well and use Bruker's frequency list). With O1 and O2 in ppm, different magnetic fields do not matter for parameter transfer.

4.1.2   Sample Preparation

A water sample should be made with a CRAMPS insert or, if that is not available, with some Teflon tape at the bottom of the rotor to center the sample. A drop of water with or without TMS is set on top of the insert or Teflon tape and the upper part of the rotor remains unfilled. Insert the rotor into the probe and do not spin. Some shimming may be done on this sample to improve the line width, using the **x** or **y** and **z** shims.

It is advantageous to prepare one rotor with a mixture of KBr and adamantine (except for the 2.5 mm rotors, for reasons of signal intensity). This sample is easier to pack than a pure KBr sample and a number of setup experiments can be done without changing the sample. The ^{79}Br resonance of KBr is used to set the magic angle. This process is described in section 4.2. Pulse lengths for ^{13}C and ^{1}H and the HH contact are determined on the adamantane ^{13}C (and ^{1}H) signals. A second rotor filled with Glycine is needed to determine the ^{13}C and ^{15}N sensitivity. A rotor filled with ammonium-dihydrogenphosphate is used to measure ^{31}P signals, determine pulse lengths and sensitivity (^{31}P has no official sensitivity specification but the experiment is useful for obtaining an overall impression of the probe).

 For SB CPMAS probes and 2.5 mm probes it is advantageous to have a sample with ^{15}N labeled glycine or ammonium chloride available to determine pulse lengths and power levels needed for the ^{15}N cross polarization (CP) experiment.

4.1.3   MAS monitoring

In order to monitor the spinning speed of the rotor, the **mascontrol** window must be opened. To do this, type **mascontrol** into the command line. Within this window the present probe needs to be selected, open the button **etc…** and select *Write log file ON*. This will write spin rates to log file so that spin stability can be determined. A printout of this file must be attached to the other test results.
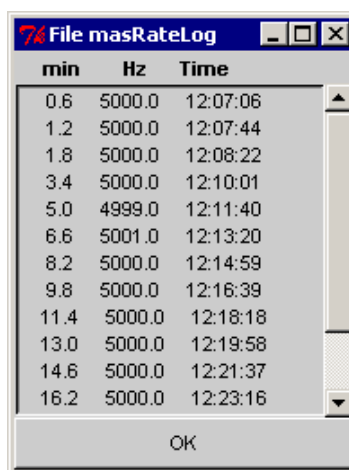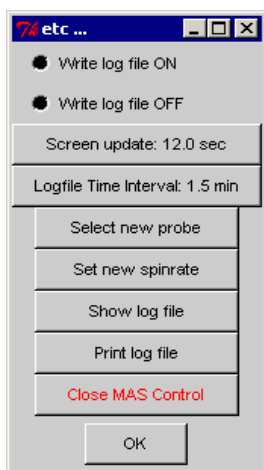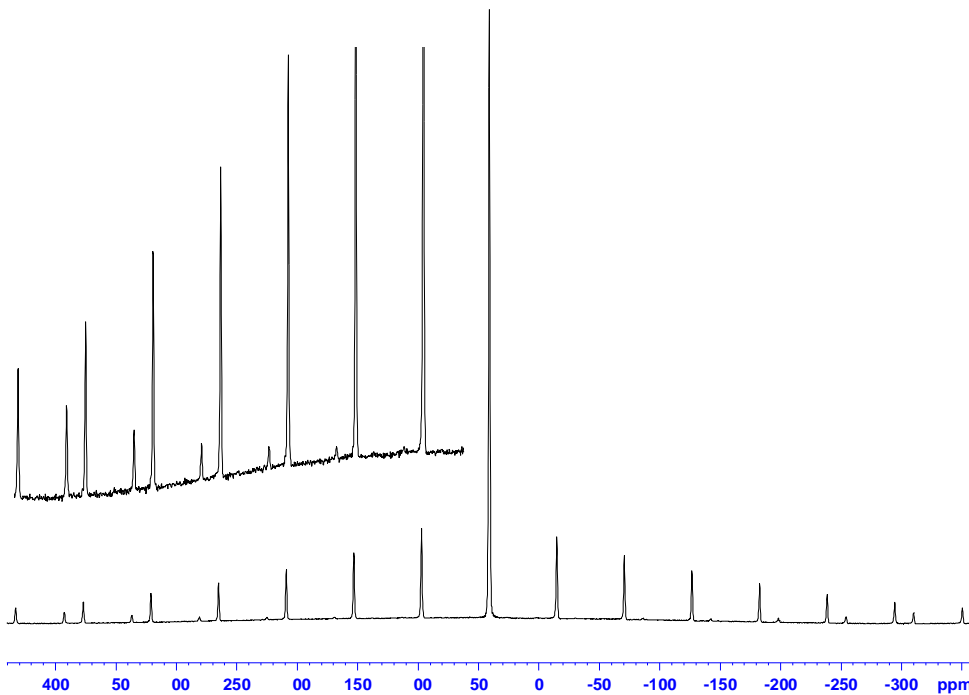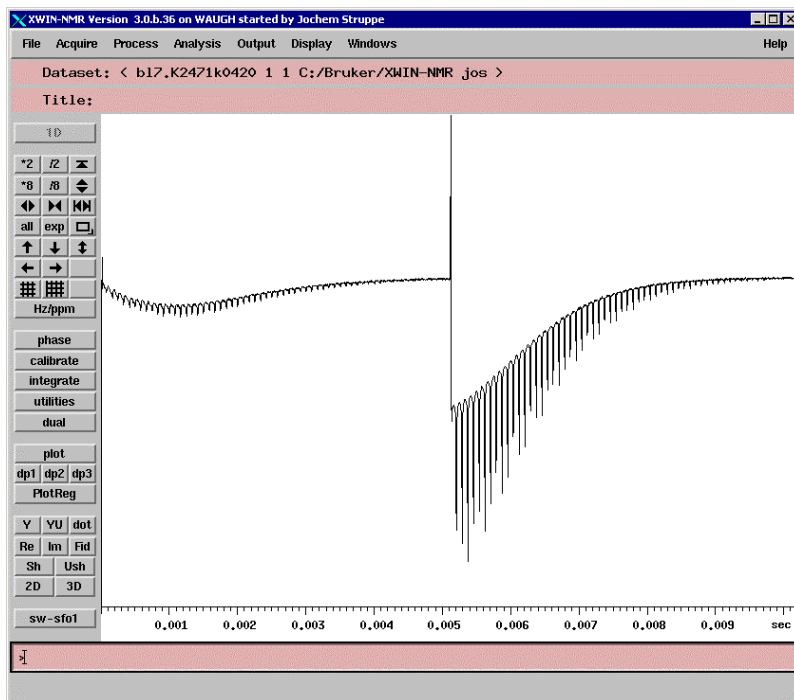
Spectrum with spinning sidebands at 7 kHz sample spinning using 100 kHz sweep width, **digmod** = ANALOG. Insert shows the folding back of spinning sidebands.

4.2   Setting the magic angle with KBr

For more information on how to adjust the angle using the FID in the gs mode, see manual Z31401 page 45 ff..

Test Sample:	KBr or KBr +Adamantane
Spinning rate:	7 kHz
Spectrometer *edsp***:**	NUC1 ^{79}**Br**
Parameter set :	KBr

Pulse program:

```
;zg
;#include <Avance.incl>
1      ze              ;pl1=power level f1 channel
2      d1              ;p1 =f1 channel high power pulse
       p1 ph1          ;d1 =relaxation delay = 1 - 5* T1
       go=2 ph31
       wr #0           ;write data to disk
exit


ph1  = 0 2 2 0 1 3 3 1
ph31= 0 2 2 0 1 3 3 1
```

Parameters: *Acquisition:*   PULPROG   zg

	NS	*4*
	P1	*4us*
	DW	5us
	D1	*500ms*
	PL1	as determined in the following:
	DIGMOD	analog
	O1P	41 ppm
	AQ	*about 8 msec*
	MASR	6000 Hz and higher
		NOT a multiple of 5 kHz

Setting Transmitter power: If the correct power level for the X-channel is not already known, start using 50% of the maximum power for the used channel using the power handling data sheet that comes with each probe. For the appropriate power level settings use 50% of the nominal transmitter power for the power level 0 if **CORTAB** has not been done and 25% if it has been done. Then use the fact that the power decreases or increases by 50% for a 3dB change above 0dB and below 0dB only if **CORTAB** has been done. As an example we want to use 75W on a 2.5 mm CPMAS probe with a linearized (**CORTAB** done) 1000W transmitter. Then we have 250W at 0 dB, 125W at 3 dB and 75 W at about 5.5 dB. As a reminder dB is defined as:

$$y\mathrm{dB} = 10 \cdot \log\left(\frac{power_1}{power_2}\right).$$

That is exactly what you can use to calculate the power level change for a certain change of the pulse width or a nutation frequency, but as you are using amplitudes and not power it changes to:

$$y\mathrm{dB} = 20 \cdot \log\left(\frac{pulsewidth_1}{pulsewidth_2}\right).$$

In order to find a new pulse width for a given power level it reads:

$$pulsewidth\ 2 = pulsewidth\ 1 \cdot 10^{(powerlevel_1 - powerlevel_2)/20}$$

1. Read in the parameter set **KBr**, by typing *rpar KBr all* into the command line.

2. Set the power level PL1 following the suggestions above; match and tune the probe.

3. Type *edasp* and check the default button, the routing should now go through the high power amplifiers.

4. Type *wobb* and toggle with *acqu* into the acquisition window.

5. Match and tune the probe.

6. Type *gs* in the command line and switch with *acqu* to the acquisition window.

7. Make fine adjustment of O1 in the gs-window until the signal is full on resonance and set the receiver phase, *phref*, so that the full signal is in one channel in the *ush* (unshuffle) mode.

8. Adjust the magic angle, observing the FID and dialing the MA adjustment so that the spinning sidebands extend as far as possible. The spinning sidebands are seen in the time domain signal, the free induction decay (FID), as spikes riding on the on resonance signal of the central transition. The sidebands should extend as far as possible to the end of the 8-10 ms acquisition time.

9. Stop *gs* and start zg to get a spectrum.

Plotting: Plot the whole spectrum

Note: A sweep width of 250 kHz can also be used to see the whole spinning sideband pattern. Such a sweep width can be achieved either in analog mode or in digital mode with DSPFIRM on smooth. At a spinning rate of 6 to 8 kHz, one can see the sidebands going almost all the way out to the end of the spectral window (depends on S/N! i.e. rotor size) and being reflected. Print the spectrum as proof for good setting of magic angle, use 16 to 64 transients to obtain a good enough intensity of the reflected signal. A sweep width of 250 kHz cannot be achieved on systems using SADC digitizers. For such systems use **swh** = 100 kHz and digmod=analog.

Final Test BH065700 Operator JOS
PH MASVTN 500 WB BL7 15N-31P/1H
Adamantane 1H
Spinning speed 7 kHz
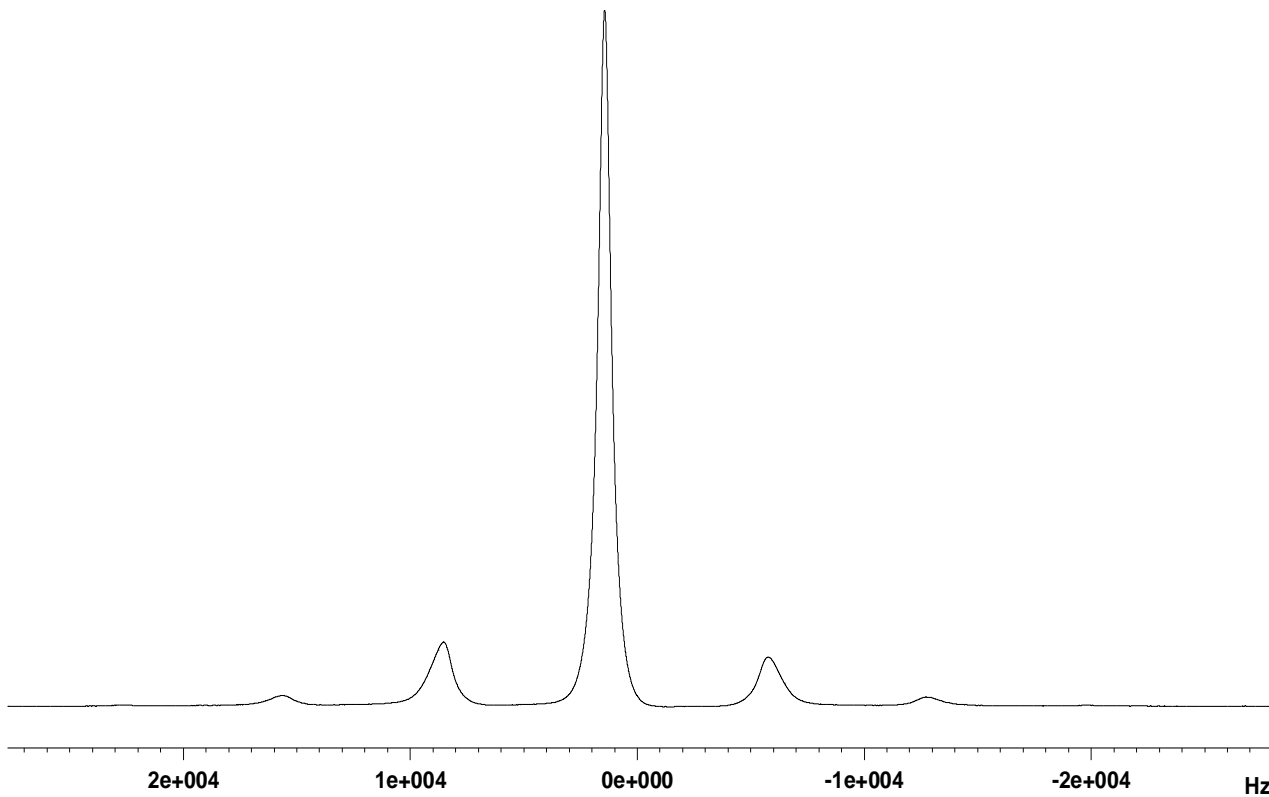
4.3   ¹H 90 degree pulse Adamantane

The Adamantane sample is used to determine the [1]H 90º pulse. While this is done a check of sample spinning needs to be performed order to check whether the probe spins well and to demonstrate stability of MAS controller. 7mm, 4mm and 2.5mm MAS probes need to run at the limit of 7 kHz, 15 kHz and 35 kHz respectively. A log file of the MAS controller must be created according to the procedures outlined in sections 4.1.3. Print this file as proof that stable spinning at the highest spinning speed was achieved. Make sure that the rotor is not too old if going to the fastest rotation speeds. With normal wear and tear I would suggest that for acceptance tests in the field the rotor should not be older than 50 high speed tests. It is advised to maintain a usage log for all rotors, speed, when filled last, trips and special issues (dropped etc.). Indeed, it is annoying to have a rotor crash in the field.

|			
Test Sample:		Adamantane	
Spinning rate		limit of probe	
Spectrometer *edsp*:		NUC1	**¹H**
Parameter Set		ZGSOLIDS	
Pulse program	**zg**		
Parameters	*Acquisition:*	PULPROG	zg
		TD	2048
		NS	4
		P1	4us
		DW	5us
		DE	6us
		D1	2s
		PL1	as determined in the following:
		DIGMOD	digital
		01P	2.3 ppm

Transmitter power: see 4.1

Plotting: Plot the whole spectrum. Choose Hz for x-axis units.

Note: Use the experiment to determine the pl1 for a 4 us 90-degree pulse on 1H, the power level for the decoupling pulse (e.g. 2.5 us for 4mm CPMAS probe) and write the obtained value for the high power decoupling pulse into edprosol 1H F1A1 in the field for **p90** (see appendix). Having those data available allows a fast setup of the CP as well as the sensitivity experiment. With CORTAB done, write the power level and for a 2, 3 and 4 us ^{1}H pulse into the prosol table on F1 use either all solvents or create a solvent solids following the instructions on the ICONNMR manual extension for solid state NMR.

Final Test BH065700   Operator JOS
PH MASVTN 500 WB BL7 15N-31P/1H
Adamantane 13C
Resolution in Hz
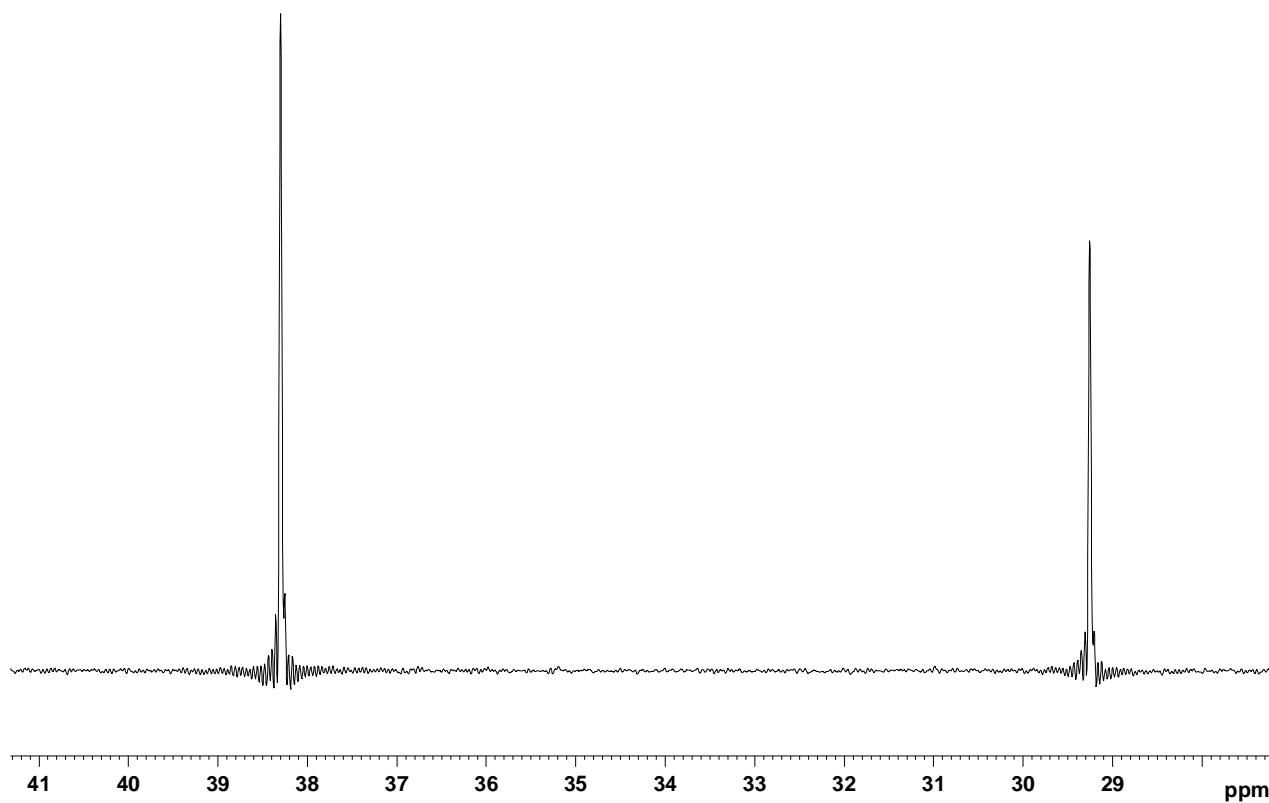
4.4 ^{13}C Direct Polarization on Adamantane

Direct detection of ^{13}C is done on Adamantane in order to determine the 90-degree ^{13}C pulse at various pulse lengths and power levels. The experiment can, in principle, be used for shimming, however, more signal is available through the CP experiment (see 4.5). Begin with **aq** = 100ms and adjust 90-degree pulses at various pulse lengths as deemed necessary. For shimming, **aq** can be increased up to 500ms if the ^{1}H decoupling is kept moderately low, see note. **Attention**: Be extra careful during that experiment: check **aq** if **sw**, **dw,** or **td** values are changed and make sure it stays below 0.1 s, otherwise the probe could be damaged. Use power levels that provide a 5us 90-degree pulse for a 4mm probe - see **note** below.

Test Sample:		Adamantane	
Spinning rate:		5 kHz or higher	
Spectrometer *edsp*:		NUC1	^{13}**C**
		NUC2	^{1}**H**
Parameter Set:		**DirectC13**	
Pulse program:		**hpdec.av**	

```
;hpdec.av
;acquisition on X with hp proton decoupling


1 ze                 ;set RCU to replace mode
2 d1 do:f2           ;recycle delay
  (p1 ph1):f1        ;transmitter pulse on F1 with power level pl1
  1u cpds2:f2        ;use cpdprg2 cw or tppm at power pl12
  go=2 ph31          ;make sure the adc is finished, turn decoupling off
  1m do:f2
  wr #0              ;save data in current data set
exit

ph0= 0               ;constant phase for acquisition
ph1= 0 1 2 3         ;simple pulse phase list
ph31=0 1 2 3         ;signal routing corresponds to pulse phase list
```

Parameters:	*Acquisition:*	PULPROG	hpdec.av
		TD	2048
		NS	4
		P1	4us
		DW	50 μs
		CPDPRG2	tppm15 (or cw at low field instruments)
		PL1	approach as determined in 4.2 and 4.3
		PL12	pl for 5.5us 90degree on ^{1}H (45 kHz B1 field) for 4 mm and 6.3 us (40 kHz) for 7 mm probes

P31	2*p3-0.2us as start value – optimize! Might require a decent shim already.
O1P	32 ppm
O2P	2.3 ppm
ZGOPTNS	-Dtppm

Plotting: Plot main part of spectrum and determine line width with the au program *hwcal*. Note the line width in title, if the experiment was used for shimming.

Note 1:   If no previous referencing has been done using the BSMS field or the BSMS display/lock window, set the right hand line to 29.5 ppm. If the spectrometer is referenced properly with the field and spectrometer frequency, use the values given in the parameter set. If the '**sr**' command is used to set the reference, use a frequency right between both lines as the O1 for the experiment. Then, measure the absolute frequency of the left line and multiply that frequency by 3.976787 in order to find the right SFO2 for the ^{1}H decoupling.

Note 2: This experiment can also be used for shimming the probe. Then it might be necessary to set **aq** to higher values, up to 500ms. In that case, we reduce the decoupling power as much as possible but try to stay high enough so that dipolar coupling cannot influence the line width. A maximum of 500 ms for **aq** can be used with reduced decoupling power. This is at the power level of a 5 us pulse, equivalent to a B_1 field of approximately 50 kHz. for 4 mm probes and 40 kHz for 7mm probes. Pay attention that in such cases the recovery delay D1 is 10 s. One must not exceed a maximum of 500 ms for **aq**.

Note 3: In the most recent release the au-program `au_cp`, changes the decoupler power level used for the experiment based on the acquisition time. The au_program uses and sets a hidden pulse programs permitting **aq** up to 1s by reducing the amplitude of the decoupler rf signal by 25% for 50ms < **aq** < 200ms and by 50% for 200ms < **aq** < 1s. The decoupler power in the prosol table must be 80% of the max. value, i.e. 80kHz for 4mm (3.1us), 56 kHz for 7mm (4.5us) and 125 kHz for 2.5mm (2.1us) CPMAS probes.

Decoupling programs:

The decoupling program **cw**:

> **0.5u pl=pl12**
> **1 100up:0 fq=cnst21**
> **jump to 1**

TPPM decoupling: TPPM (Two Pulse Phase-Modulation) has been invented 1995 by the Griffin group at MIT:
A.E. Bennett, C.M. Rienstra, M. Auger, K.V. Lakshmi, and R.G. Griffin; *Heteronuclear decoupling in rotating solids*, J. Chem. Phys. 103 (16); 6951 – 6958 (1995)

TPPM decoupling surpasses the traditional cw decoupling and is now standard in solid state NMR experiments. TPPM is not needed for the experiment with adamantane.

The decoupling programs tppm: (the frequency offset cnst21 can be set to 0 for these setup experiments)

tppm15	tppm20
0.3u fq=cnst21	0.3u fq=cnst21
0.5u pl=pl12	0.5u pl=pl12
1 p31:0	1 p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p31:15	p31:20
p31:0	p31:0
p30:15	p30:20
jump to 1	jump to 1

49 48 47 46 45 44 43 42 41 40 39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22 ppm
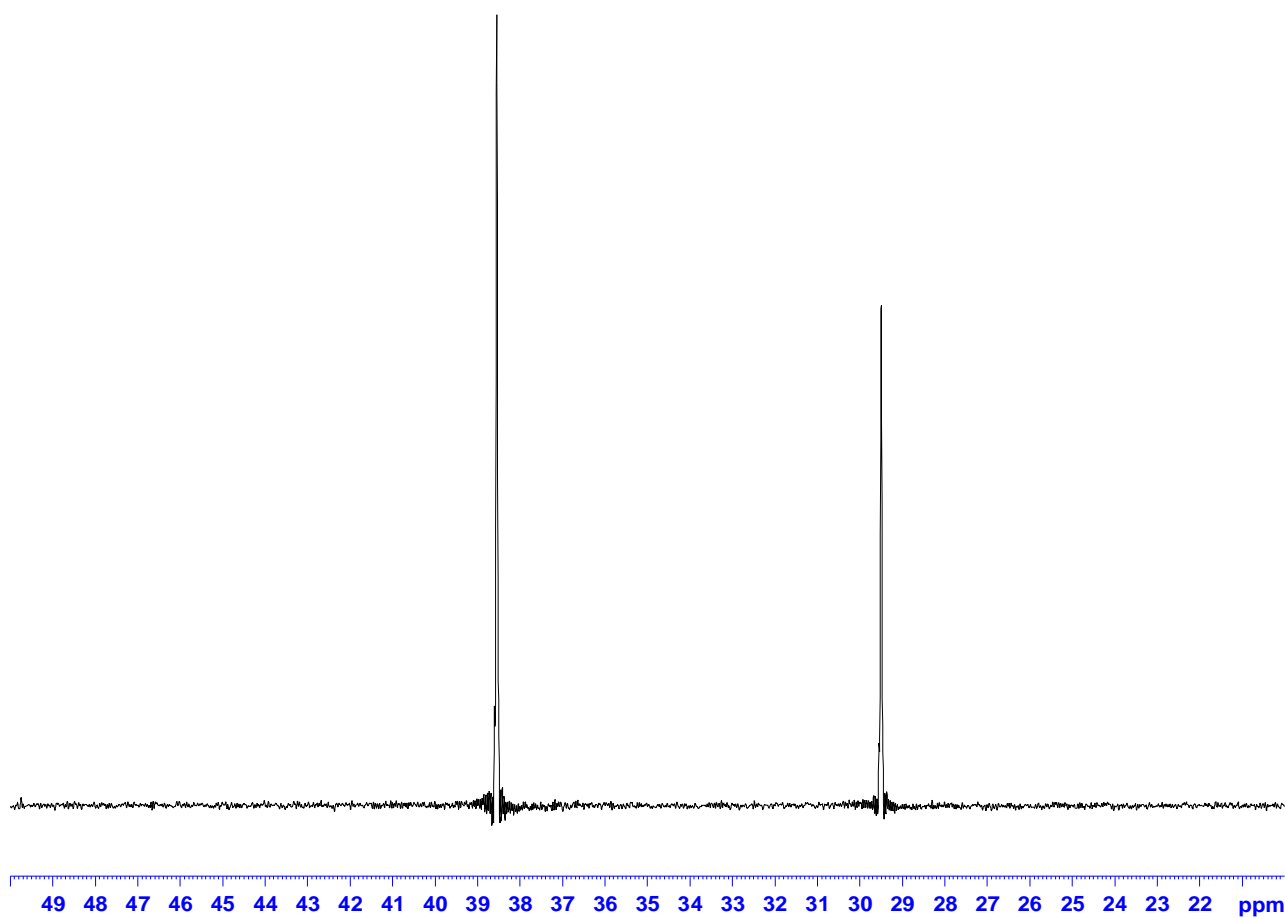
4.5 ^{1}H - ^{13}C Cross Polarization on Adamantane

NOTE: The following experiment uses the same power level PL12 for the 90º-pulse p3 and the decoupling pulse in order to allow control and direct measure of the nutation[1] frequency of the decoupling field. PL2 drives the power of the fast shape during the contact. The 3.1 us (80 kHz) pulse for 4 mm probes are known from the previous experiment (^{1}H on adamantane).
Use parameter set **CPADAM**, edit the pulse program and disable the *acquisition time* protection feature by commenting out the line #include <praq.prot> with a ";". Save the pulse program under the original name with your initials as extension, e.g. if xyz is your extension cp.av is renamed to cp.av.xyz. Subtract 3 or 6 dB from pl12 if it is set for the 80 kHz decoupling power level, i.e. if pl12 for 80 kHz is 5dB, use 8 dB for acquisition time 50ms $< aq < 200$ms and 11 dB if the acquisition time is 200ms $< aq < 1$s. The decoupler power in the prosol table must be 80% of the max. value, i.e. 80kHz for 4mm (3.1us), 56 kHz for 7mm (4.5us) and 125 kHz for 2.5mm (2.1us) CPMAS probes.

Set pl1 to the same power level achieved for a 5us 90º ^{13}C pulse from the previous hpdec experiment and adjust pl2 for best HH match using **popt**.

Test Sample:		Adamantane
Spinning rate:	5 kHz	
Spectrometer *edsp***:**	NUC1	^{13}**C**
	NUC2	^{1}**H**
Parameter set:	CPADAM	
Pulse program:		

Parameters:	*Acquisition:*	PULPROG	cp.av or cp90.av
		TD	1024
		P1	0
		P15	5ms
		SWH	10 kHz
		D1	5s
		PL1	for 4 or 4.5 us pulse as determine in 4.4
		PL11	120 dB
		PL2	power level of shaped contact pulse if one uses the

---

[1] The spin nutation frequency is the frequency at which the spins rotate around the rf-field; $f_{nut}=\gamma/2\pi*B_1$, B1 is the field strength of the rf field
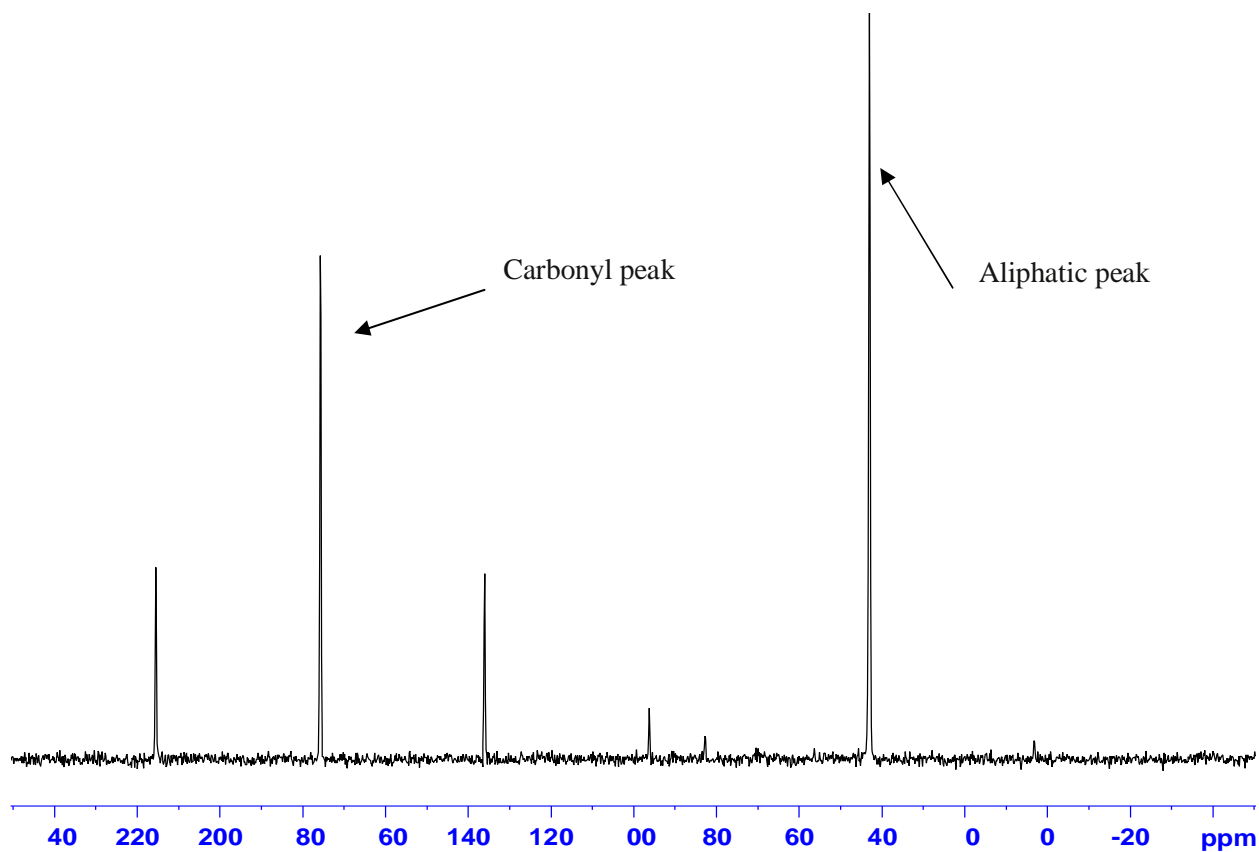
	ramp and pl1 for 4 us excitation pulse we need then the power level for 4 or 4.5 us 1H as determined in 4.3 minus 3 dB
SPNAM0	ramp.100
PL12	see text
P3	goes with pl12 – if pl12 was changed by 3 dB, multiply the original pulse with with 1.4, if it was changed by 6 dB double the original pulse width. If e.g. original pulse parameters were 3.1us at 5 dB, change to 8 dB gives 3.1*1.4=4.3us, and a change to 11 dB gives a 6.2 us pulse width.
CPDPRG	tppm15 (or cw for low field instruments)
P31	2*p3-0.2us (see above)
ZGOPTNS	-Dlacq (switches protection file off for long acquisition times – see note below for decoupling power limitations at long acquisition times)

Note: This experiment is used for shimming the probe and demonstration of power handling capability. It might be necessary to set *aq* to higher values. In that case, make sure that the decoupling pulse does not exceed 50 kHz (for 4 mm probes and 40 kHz for 7mm probes) and that the recovery delay D1 is 10 s. Save parameter setas **CPADAM**. Do not exceed a maximum of 500 ms for **aq**.

Attention: If **aq** >50ms, then the probe protection increases the attenuation (the power level) by 3dB for 50ms < aq < 200ms and by 6dB for 200ms < aq < 1s in recent pulse programs

Carbonyl peak

Aliphatic peak

40   220   200   80   60   140   120   00   80   60   40   0   0   -20   ppm

4.6 ^{13}C Sensitivity on Glycine

Having the best HH match from the previous experiment, it is necessary to optimize the HH match on for the glycine sample again if the spinning speed was changed. Spin at N kHz where N is defined by the 1H spectrometer frequency divided by 100MHz (e.g. for a 500.13MHz spectrometer N=5). Then p3 is measured in a nutation experiment (increment p3 using popt) in order to verify the measurements at the ^{1}H experiment. Then calculate the power level for a 2.5 us pulse, which is a 100 kHz B$_1$ field (4mm probe) or 75 kHz (7mm probe), using ***pulse*** in the ^{1}H adamantane experiment (section 4.3.) Remember, using the au program pulse requires values be placed in pl1 and p1, not pl2 and p3. Set to the obtained new values for p3 and pl12 and repeat the measurement. The obtained pl value in dB is then used for pl12. Measure p3 with ***popt*** in order to verify the calculation. A good p31, the pulse for TPPM decoupling, is estimated from p3 using p31=2*p3-0.2us.

The power handling test should be done at that stage of the test procedure. The experiment must be done with the pulse program **cp90.av** (**cp4c.98 for D*X** spectrometers) with the protection features implemented.

NOTE: In more recent releases, the pulse program uses the relations file ***solids_cp***. If this is not the case, the use of the relations file can be obtained by adding the line

`prosol relations=<solids_cp>`

between the comments and the first line with an include statement into the pulse program. This requires that the power levels and pulses are entered correctly into the prosol table (see appendix).

Test Sample:	Glycine
Spinning speed:	Spin at N kHz where N is defined by the 1H spectrometer frequency divided by 100MHz (e.g. for a 500.13MHz spectrometer N=5).
Spectrometer *edsp*:	NUC1 ^{13}C
	NUC2 ^{1}H
Parameter set:	CPGLY
Pulse program:	**cp.av**
Parameters: *Acquisition:*	**PULPROG cp90.av**

TD 2048
SW 300ppm
D1 5s
NS 4
PL1 for 4 or 4.5 us pulse as determine in 4.4
P15 2000 µs
PL2 power level for 4 - 4.5 us ^{1}H pulses minus 3 dB
SPNAM0 ramp.100
PL12 Power level for excitation pulse P3 and decoupling. Use **100% of the maximum values for sensitivity specs and 80% for the PROSOL**

table (56kHz, 80 kHz and 120 kHz for 7, 4 and 2.5 mm probes)!   Max. decoupling is 75 kHz, 100 kHz and 150 kHz for 7 mm, 4mm and 2.5 mm MAS probes, respectively. Acquire one experiment with maximum decoupling and aq=50 ms to document power handling.

P3	**Use the maximum values, 2.5us, 3.3us and 2us for 4mm, 7mm and 2.5mm MAS probes, respectively. Measure the 80% values and put those into the PROSOL table, i.e. use 3us, 4.5 and 2.1 us for 4mm, 7mm and 2.5mm MAS probes, respectively.**
P31	**2*P3-0.2us to be optimized**
O1P	**100 ppm**
O2P	**3.5 ppm (to be optimized in steps <=500 Hz**
CPDPRG2	**tppm15 or SPINAL64 (cw for 300MHz and Lower field spectrometers)**
Processing: **LB**	**0 Hz**

Note: If the carbonyl peak comes properly up at 176 ppm the O2 parameter above can be used. If not, set the carbonyl peak to 176 ppm. In order to get the right decoupler frequency, measure SFO1 at the carbonyl peak and multiply this number with 3.976245. Using *popt* with 400 Hz steps can also be used to find the best position for the decoupler frequency. For more advice, consult one of the references given above.

Sensitivity is determined by measuring the signal height of the aliphatic peak and noise over a 10 ppm wide region. Use the au program **sinocal**. *Signal region* 50 ppm to 30 ppm, *range for noise determination* from 250 ppm to – 50 ppm and *noise* 10 ppm.   When using ATP the appropriate parameters for sinocal are readily available and **sinocal** loads those parameters automatically.

Acceptance Test Procedure
Determination of 90 deg pulse for 13C

poptau for p1 finished.
ZERO at experiment 4.671153: p1 = 4.671153

NEXP=20
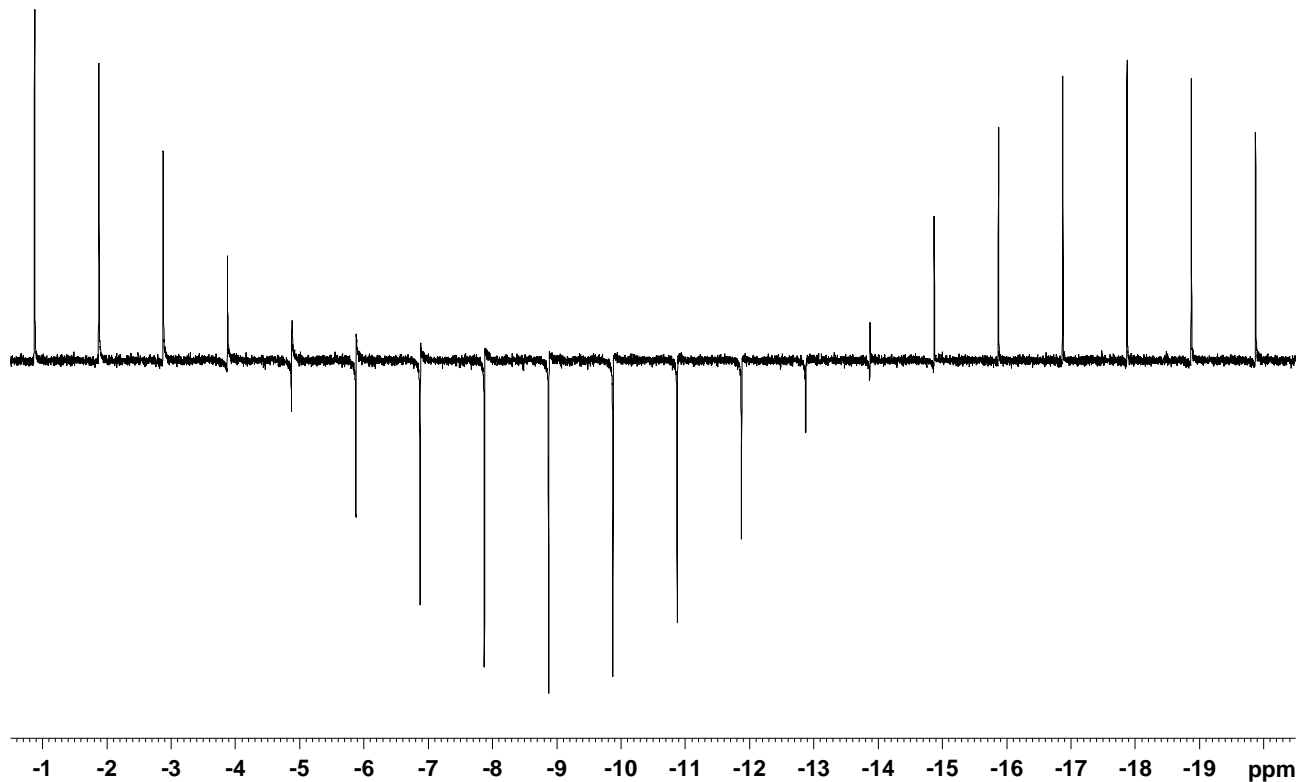
4.7 ^{13}C π/2 Pulse determination

Test Sample: Glycine

Spinning speed: Spin at N kHz where N is defined by the 1H spectrometer
                                    frequency divided by 100MHz
                                    (e.g. for a 500.13MHz spectrometer N=5).

Spectrometer *edsp***:**   NUC1 ^{13}**C**

                                    NUC2 ^{1}**H**

Parameter set: **CPGLY**
Pulse program: cp90.av
Parameters: *Acquisition:*   PULPROG   cp90.av
                   Set: PL11 = PL1
                                    all other parameters used as in 4.6
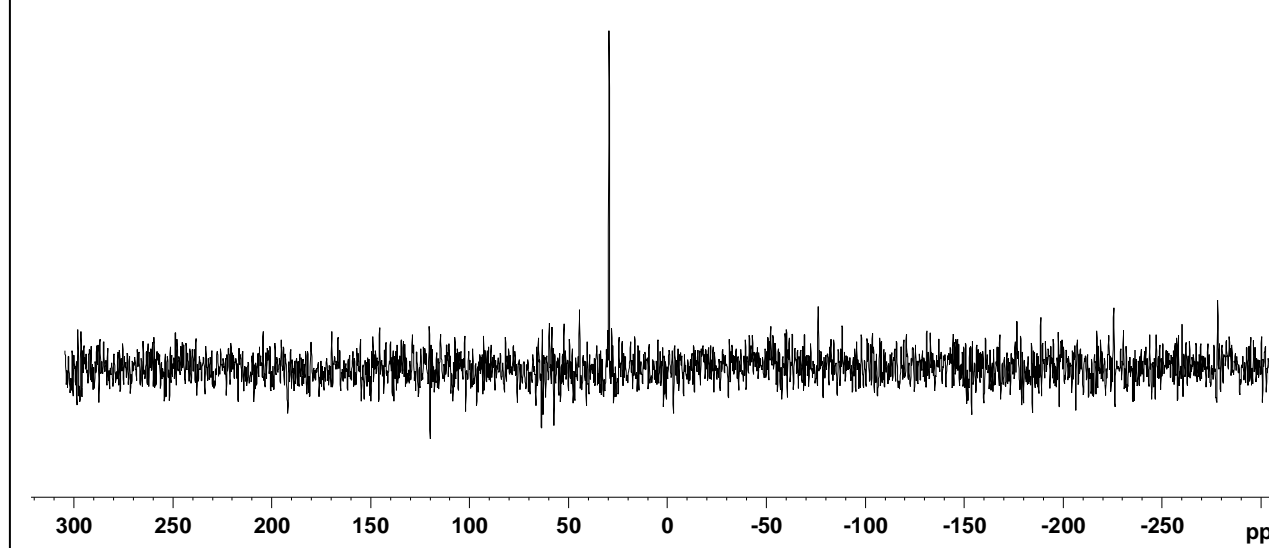
                   Processing   as above

Note: Use *popt* for this experiment.
A π/2 pulse on ^{13}C with the right phase after the spin lock gives no signal!
See figure.
Note: Enter the value pl11 and p1 into P90 of the prosol table.

```
        Final Test Probe B0628/101: PH MAS WB BL-4 1H/X/Y
                          Done by: JOS
                   Glycine 15N CP experiments
                    Spinning speed 11000Hz
                       Signal to Noise:
            Signal from      40 to  25 ppm
            Noise from      179 to 129 ppm
```

4.8 ^{15}N Sensitivity on Glycine

Test sample: Glycine, ^{15}N natural abundance
                          For parameter optimization: use ^{15}N enriched Glycine
Spinning speed: Spin at N kHz where N is defined by the 1H spectrometer
                          frequency divided by 100MHz
                          (e.g. for a 500.13MHz spectrometer N=5).
Spectrometer *edsp***:**   NUC1 ^{15}**N**
                                 NUC2 ^{1}**H**

Parameter set: CPN15
Pulse program: **cp90.av**

Parameters: *Acquisition:*

PULPROG	**cp90.av**
TD	**2024**
P1	**5.5us (goes with PL11)**
P15	**3 ms**
SW	**400ppm**
D1	**5s**
RG	**8096**
PL1	**Power level for 5 or 5.5 us pulse**
PL11	**120 dB**
PL2	**power level for 5 or 5.5 us 1H pulse + 3dB**
SPNAM0	**ramp.100**
PL12	**Use same decoupling as in previous experiment with ^{13}C**
O1P	**0 ppm**
O2P	**5 ppm optimize**

Note: A good starting point for the choice of power levels is to use the power level of the ^{13}C and subtract 3dB (double the power) and add 3dB to the proton power level *pl2* used for ^{1}H-^{13}C CP-MAS experiment. The ^{15}N peak is approximately at 33 ppm if the reference procedure was used following chapter 4.1 or 4.4. For **o2p** use **o2p** from Glycine sensitivity experiment and add 5.5 ppm. The best decoupler position can also be found using `popt` with 400 Hz steps. In order to setup HH and all other parameters it is advantageous to use a ^{15}N labeled glycine, if available. Another good and easy sample is ^{15}N enriched ammonium nitrate (allows direct polarization almost without any decoupling).
Sensitivity is determined by measuring the signal integral over the peak and noise over a 20-ppm wide region. Use the au program `sinocal`. *Signal region* = 35 ppm to 25 ppm, *whole spectrum* 300 ppm to -300 ppm and *noise* 50 ppm.
Then the program will look for the best noise region and return the values for noise region, signal region and S/N.
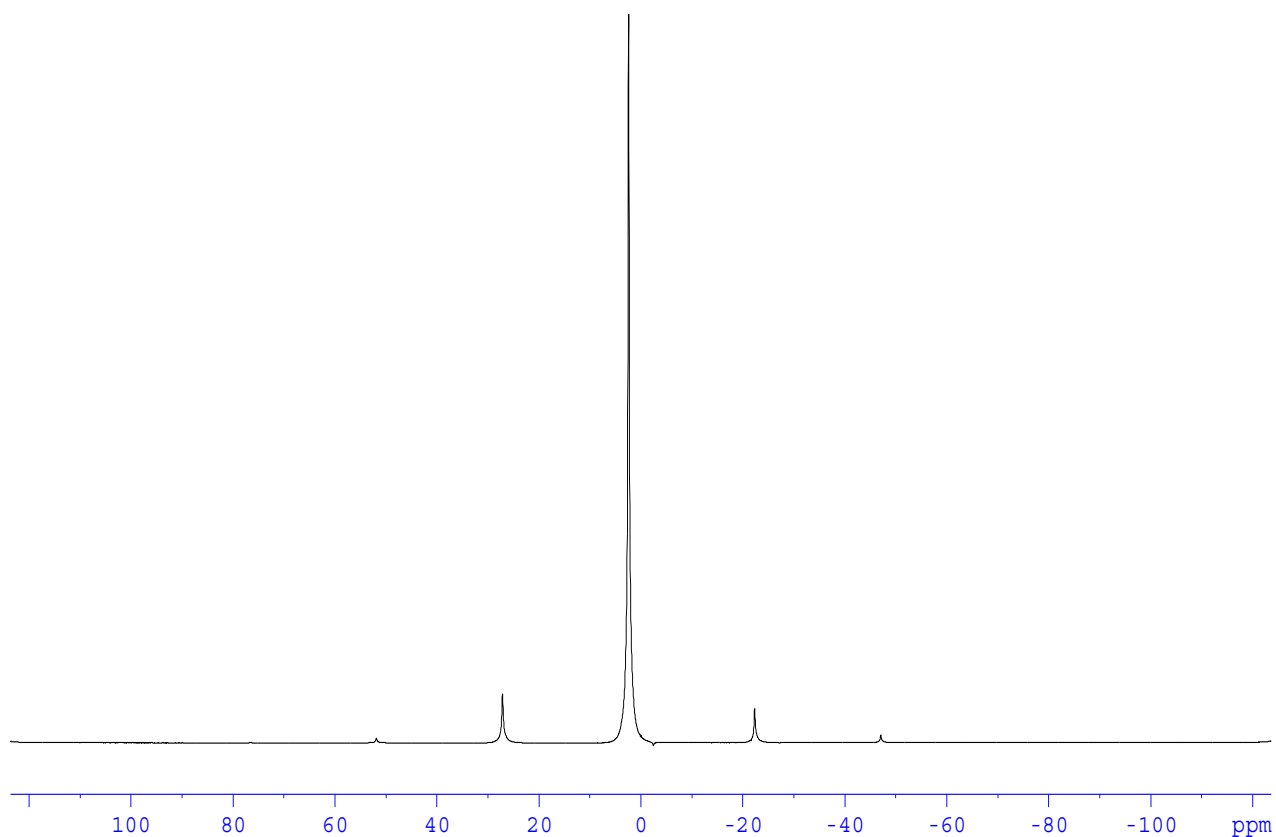
4.9 ^{15}N π/2 Pulse determination

Use same parameters as in 4.8 ^{15}N *Sensitivity on Glycine* and measure the pulse width as described in 4.7 ^{13}C *π/2 Pulse determination.*

Acceptance Test Procedures Solid State NMR

31P SINO

4.10 ^{31}P Sensitivity

Test sample:	Ammoniumdihydrogen Phosphate
Spinning speed:	See Glycine experiments

Spectrometer *edsp***:**	NUC1	^{31}**P**
	NUC2	^{1}**H**

Parameter set:	**CPP31**
Pulse program:	**cp.av**

Parameters:	*Acquisition:*	**PULPROG**	**cp90.av**
		AQ	**50 ms**
		P15	**5 ms**
		SW	**300 ppm**
		D1	**5s**
		PL1	**for 5 us pulse**
		PL2	**for 5 us 1H pulse + 3dB**
		PL12	**see ^{13}C sensitivity experiment**
		P3	**see ^{13}C sensitivity experiment**
		P31	**see ^{13}C sensitivity experiment**
		O1P	**0 ppm**
		O2P	**2.5 ppm**

Note: The ^{31}P peak is at 2.5 ppm. A good starting value can be obtained by adding 3 dB to the power level of HH match for ^{13}C (power reduction by 50%) , and using the same pl12.
.
Sensitivity is determined by measuring the signal level of the peak and noise over a 20-ppm wide region. Use the au program **sinocal**. *Signal region* = 15 ppm to -15 ppm, *whole spectrum* 125 ppm to -125 ppm and *noise bandwidth* is 20 ppm.
Then the program will look for the best noise region and return the values for noise region, signal region and S/N.

4.11 ^{31}P π/2 Pulse determination

Use same parameters as in 4.10 and measure the pulse width as described in 4.7 *^{13}C π/2 Pulse determination*

4.12 Double CP (DCP) experiment

Setup using ^{13}C – ^{15}N labeled glycine in center-packed rotor and rotate at 11 kHz
The Setup is done in three steps. Step one is CP parameter determination and measurement for ^{15}N,
Step 2 is CP parameter determination and measurement for ^{13}C at 11 kHz sample rotation and step
3 is the optimization of the second CP step in the DCP experiment.

In edasp the signal should be routed for each experiment as needed for the final DCP experiment.
The routing for the ^{15}N ^{1}H CP experiment is shown in figure 1. The DCP routing can be used for
the ^{13}C ^{1}H CP setup as well and is shown in figure 2.
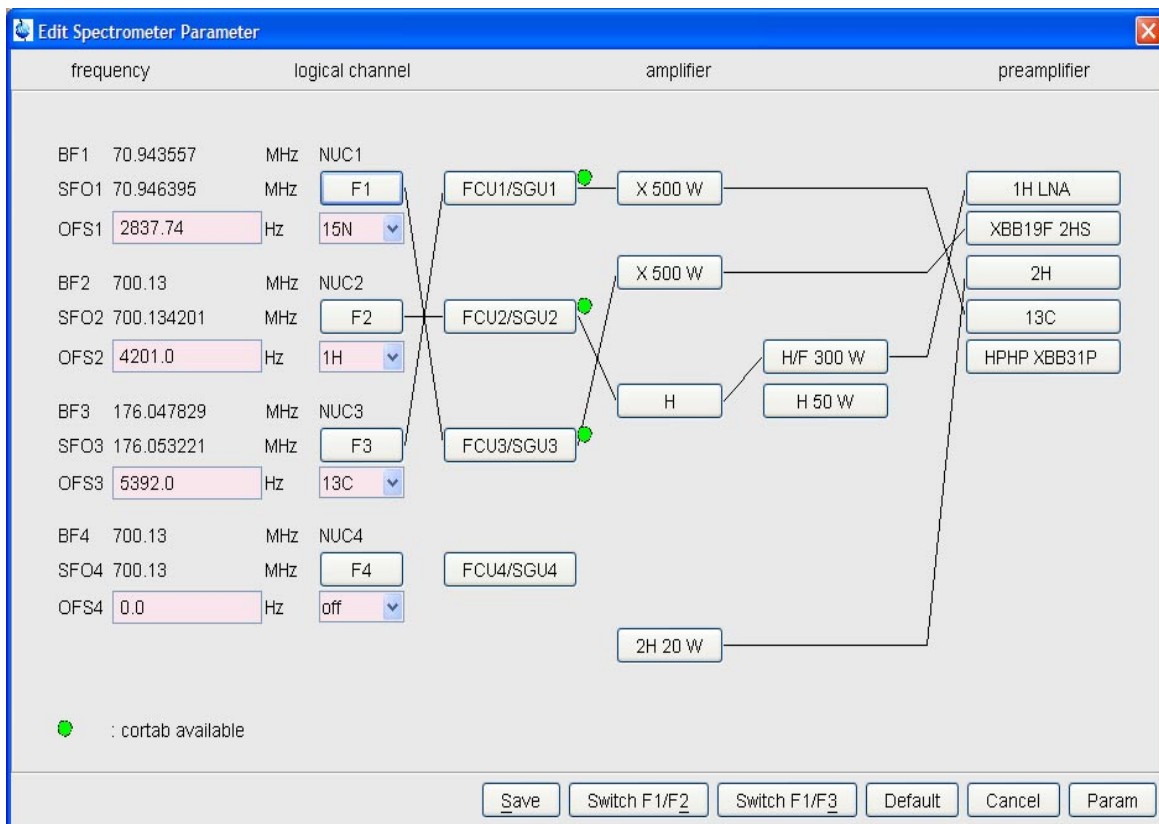
Figure 1: Setup for ^{15}N ^{1}H CP experiment, can be obtained from routing as in figure 3 by checking
the Switch F1/F3 button.
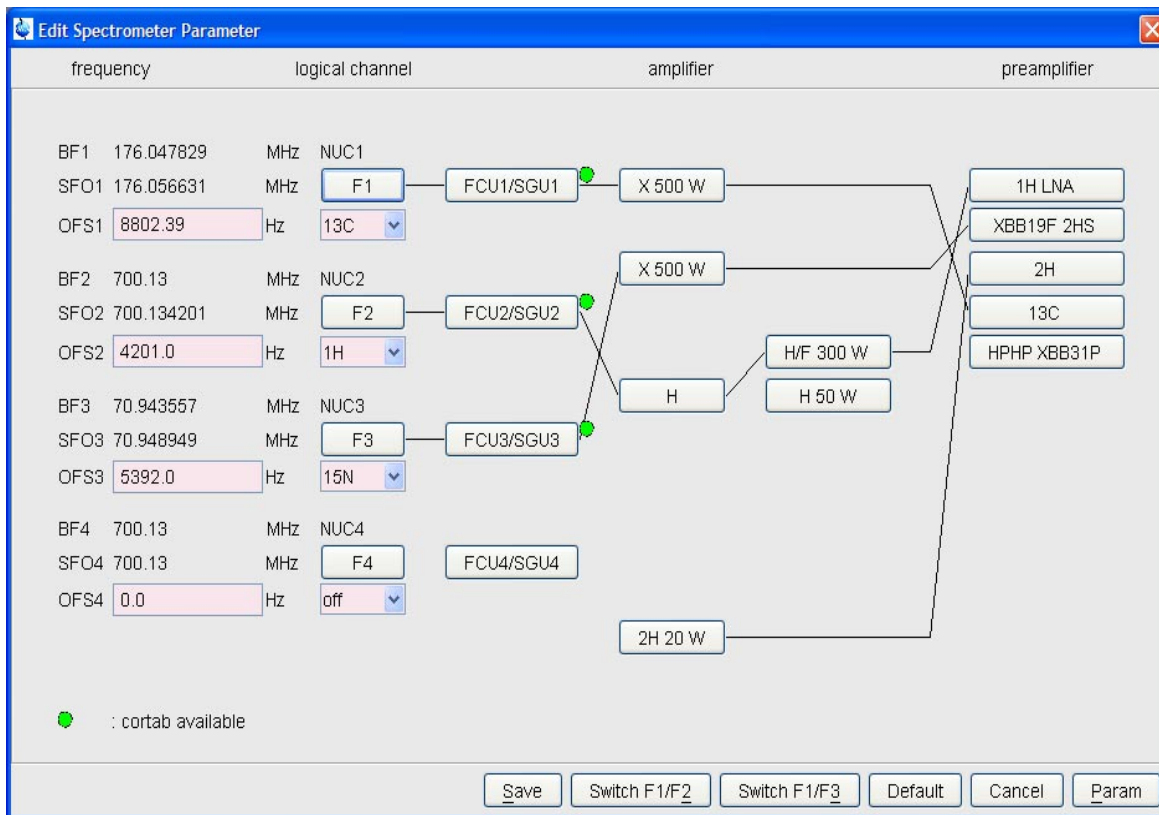
Figure 2: Routing for CP experiment with ^{13}C and the double CP experiment

Even if the CP conditions are known, it is advantageous to determine the 90 pulse of the X and Y nucleus using *cp90.av*. The 90 degree pulse is found, where the signal crosses Zero (see 4.7 and 4.9). The knowledge of the 90 degree pulse allows calculating the right HH condition by: $B1\_^{15}$N (Hz) = $B1\_^{13}$C (Hz) + spinning rate in Hz.

Run a CP experiment ^{13}C{^{1}H} with 16 scans as reference to measure DCP yield..

Call the pulse program *doubcp1.av*. Use all the parameters from CP experiments:

Test sample: center-packed U ^{13}C – ^{15}N enriched glycine
Spinning speed: **11 kHz**

Spectrometer *edsp***:**   **NUC1   13C**
                                     NUC2 ^{1}H
                                     NUC3 ^{15}N

Parameter set: **DCPNC**
Pulse program: **doubcp1.av**

Parameters: *Acquisition:*   **PULPROG   doubcp1.av**

TD	**2048**
P15	**2 - 3 ms**
P16	**4 – 10 ms optimize start with 4ms**
SW	**300 ppm**
D1	**5s**
PL1	**for 5 us pulse on ^{13}C**
PL2	**for best ^{1}H power level of HH match ^{15}N{^{1}H}**
PL3	**for 5 us pulse on ^{15}N (or best rf-field achieved)**
PL5	**for 35 kHz rf field on ^{15}N (calculate)**
PL12	**see ^{13}C sensitivity experiment**
PL13	**80 – 120 kHz cw decoupling (depending on specs)**
P3	**see ^{13}C sensitivity experiment**
P31	**see ^{13}C sensitivity experiment**
O1P	**50 ppm**
O2P	**3.5 ppm**
O3P	**40 ppm**

 Write all know pulse parameters into the parameter set, **pl1, pl2, pl3, pl12, pl13, p3 and p31**.
Then calculate **pl5** to achieve a rf field of 36 kHz and enter **pl5** into parameter set.

e.g. if **pl3** is 0 dB for 50 kHz (5us 90º pulse) we calculate: $20*\log 36/50$ =-2.85dB; **pl5=pl3-2.85 dB,** i.e. **pl5 = -2.8 dB** is the power level to use. Of course this calculation requires that **CORTAB** is done.

Note: pl1 is the power level for a 50 kHz carbon pulse. Loading the shape **spnam1** = **tancn** gives a tangential pulse at 50% of the full pulse amplitude, i.e. at 25 kHz which is the exact Hartmann Hahn (HH) cross polarization condition, $f_{rf}(^{15}N) = f_{rf}(^{13}C) + 1*f_{rot}$, with f_{rf} the ^{15}N and ^{13}C spin nutation frequencies and f_{rot} the sample rotation. This is called the +1 HH condition. If everything is setup carefully one should see a good double CP signal with 8 scans. Default is 16 scans, but for parameter optimization 4 scans suffice. Optimize **pl1** in steps of 0.1dB for $\pm$ 0.5 dB if a good signal was obtained, for $\pm$ 1 dB if a poor DCP signal was obtained. Do the optimization on the alphatic carbon resonance and use the full phase cycle **ns = 16** to exclude artifacts.
Run one experiment with 16 scans when all parameters are fully optimized and compare the intensity of the aliphatic resonance with the aliphatic resonance from the ^{13}C{^{1}H} CPMAS experiment.
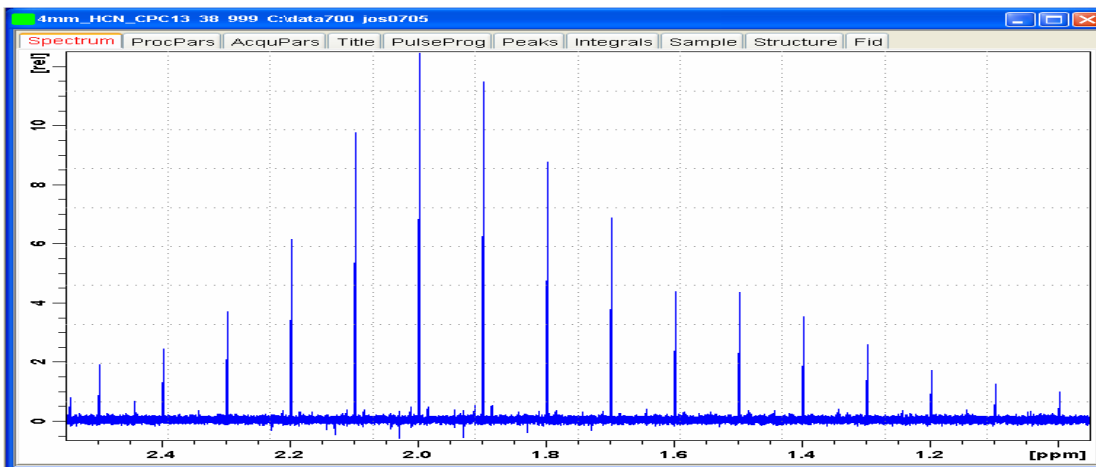
Figure 3: DCP optimization on PL1 in 0.1 dB steps.

4.13 Tuning Stability Test (optional)

Having setup DCP create a new experiment with **edc**, load pulse program **doubcp1_stab.av** change in **eda** to **2D** experiment and set **td** to 1 – 8k, set **pl12** to 100 kHz decoupling, **D1 = 5s, ns = 8** and start experiment for an overnight run.
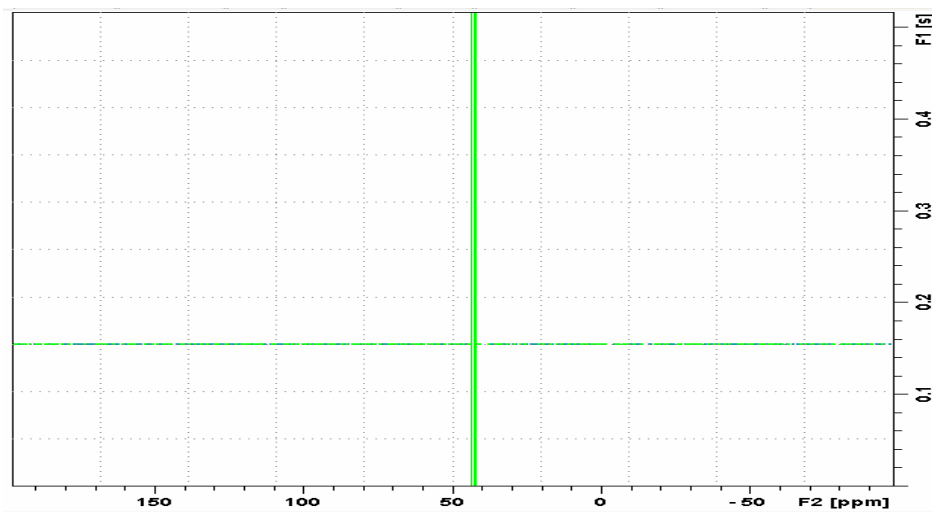

Figure 4: Double CP experiments in a hard instrument stability test over night with 8192 experiments 2 scans per experiment. Note the horizontal line from an arcing event.
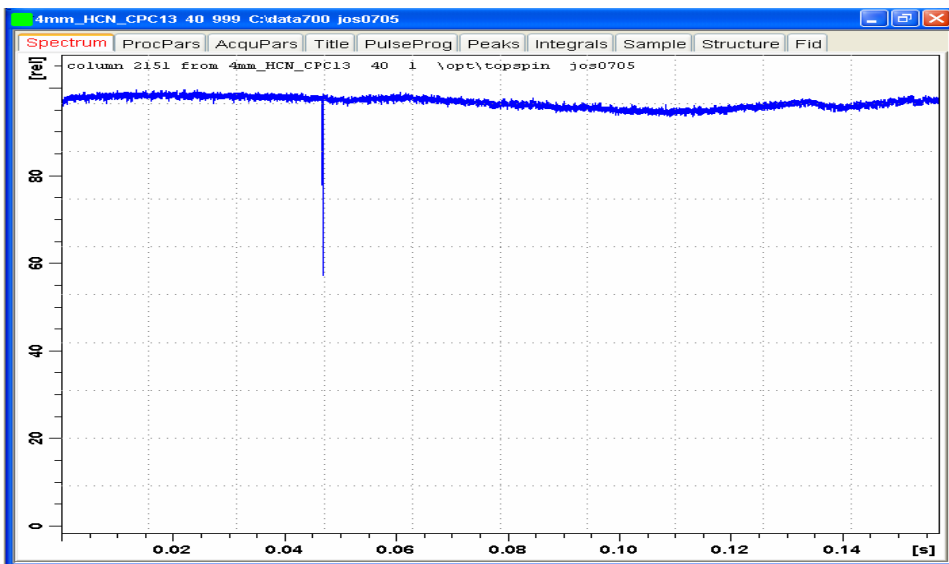
Figure 5: Double CP experiments column extracted from above 2D data set. The signal amplitude varies between 90% and 100% except for the arcing event. The tuning stability is well within the acceptance limits (20% instability).

4.14 HFX Probes: HF – Setup

General cannel setup for HFC experiments benefits from ^{19}F preamplifier module like for example the XBB19F 2HS module (figure 6)
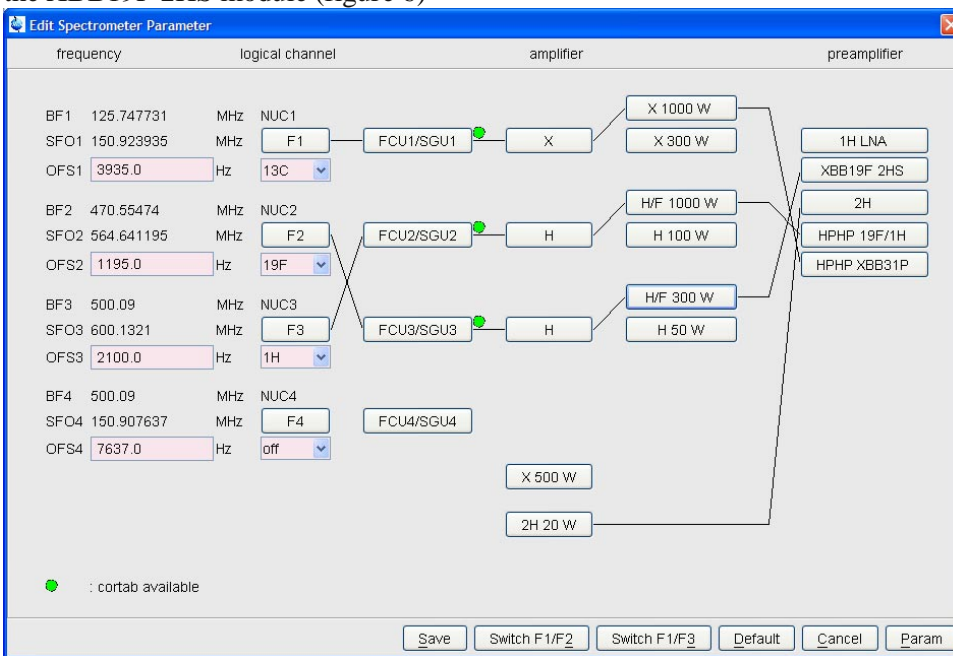

Figure 6: HFC experiment setup example with routing table and preamplifier selection.

4.14.1 FH – HF experiment

Use the pulse program *hpdec.av*. Use all the known parameters from CP experiments.
And do ^{19}F and ^{1}H detect experiment with ^{1}H and ^{19}F decoupling experiments.

Test sample:	**PVDF**
Spinning speed:	**15 kHz or 35 kHz if possible**

Spectrometer *edsp***:**   **NUC1** ^{19}**F**
                                        NUC2 ^{1}**H**

Parameter set:	**FH_dp**
Pulse program:	**hpdec.av**

Parameters: *Acquisition:*   **PULPROG**   **hpdec.av**
                                          TD   **2048**
                                          DW   **5us**
                                          D1   **5s**
                                          PL1   **for 3.1 us pulse on ^{19}F see probe specs**
                                          PL12   **for 80 kHz ^{1}H decoupling**
                                          O1P   **– 100 ppm**
                                          O2P   **3.5 ppm**

4.14.2 ^{13}C{ ^{19}F} CPMAS experiments

Use all the known parameters from CP experiments and from above HF – experiments.
With 2.5 mm probes at high spinning speed, > 20 kHz, use for the contact pulse **spnam0** =
ramp80.100.

Test sample:	**PVDF**
Spinning speed:	**15 kHz or 35 kHz if possible**

Spectrometer *edsp***:**   **NUC1** ^{13}**C**
                                        NUC2 ^{19}**F or ^{1}H**
                                        NUC3 ^{1}**H or ^{19}F**

Parameter set:	**CPCF**
Pulse program:	**cp.av**

Parameters: *Acquisition:*   **PULPROG**   **cp.av**
                                          TD   **2048**
                                          DW   **5us**
                                          D1   **5s**
                                          PL1   **CP power level for 4 us pulse f1**

PL2	CP power level f2
PL12	for 80 kHz ^{1}H decoupling / ^{19}F decoupling on f2
O1P	50 or 100 ppm
O2P	-80 – -100 ppm

Set ^{19}F carrier frequency on one of the ^{19}F resonances and optimized HH condition having ^{13}C contact pulse at 62.5 kHz for moderate spinning speeds < 20 kHz and for high spinning speed on 50 kHz. For ^{13}C{^{19}F} experiment with ^{19}F decoupling use cp.av pulse program. For ^{13}C{^{19}F} experiment with ^{1}H decoupling use cpfh1.av pulse program.

4.14.3 ^{13}C{^{19}F - ^{1}H} CPMAS experiments

Use all the known parameters from CP experiments and from above HF – experiments.
With 2.5 mm probes at high spinning speed use for the contact pulse **spnam0** = ramp80.100.

Test sample:	**PVDF**
Spinning speed:	**15 kHz or 35 kHz if possible**

Spectrometer *edsp:*	**NUC1**	^{13}**C**
	NUC2	^{19}**F**
	NUC3	^{1}**H**

Parameter set:	**CPCFH**
Pulse program:	**cpfh.av or cpfh1.av**

Parameters:	*Acquisition:*	**PULPROG**	**cpfh.av** or **cpfh1.av**
		TD	**2048**
		DW	**5us**
		D1	**5s**
		PL1	**CP power level for 4 us pulse f1**
		PL2	**CP power level f2**
		PL12	**for 80 kHz ^{1}H decoupling / ^{19}F decoupling on f2**
		PL13	**for 80 kHz ^{19}F decoupling / ^{1}H decoupling on f3**
		O1P	**50 or 100 ppm**
		O2P	**-80 – -100 ppm or 3.5ppm**
		O3P	**3.5ppm or -80 – -110 ppm**

Set ^{19}F carrier frequency on one of the ^{19}F resonances and optimized HH condition having ^{13}C contact pulse at 62.5 kHz for moderate spinning speeds < 20 kHz and for high spinning speed on 50 kHz. For ^{13}C{^{19}F} experiment with ^{1}H decoupling use cpfh1.av pulse program. For ^{13}C{^{19}F} experiment with ^{1}H and ^{19}F decoupling use cpfh.av pulse program.

4.14.4 ^{13}C{ ^{1}H – ^{19}F} CPMAS experiments

Use all the known parameters from CP experiments and from above HF – experiments.
With 2.5 mm probes at high spinning speed use for the contact pulse **spnam0** = ramp80.100.

Test sample:	**PVDF**
Spinning speed:	**15 kHz**

Spectrometer *edsp***:**	**NUC1**	**^{13}C**
	NUC2	**^{1}H**
	NUC3	**^{19}F**

Parameter set:	**CPCFH**
Pulse program:	**cpfh.av or cpfh1.av**

Parameters:	*Acquisition:*	**PULPROG**	**cpfh.av** or **cpfh1.av**
		TD	**2048**
		DW	**5us**
		D1	**5s**
		PL1	**CP power level for 4 us pulse f1**
		PL2	**CP power level f2**
		PL12	**for 80 kHz ^{1}H decoupling / ^{19}F decoupling on f2**
		PL13	**for 80 kHz ^{19}F decoupling / ^{1}H decoupling on f3**
		O1P	**50 or 100 ppm**
		O2P	**-80 – -100 ppm or 3.5ppm**
		O3P	**3.5ppm or -80 – -110 ppm**

Use optimized HH condition from ^{13}C sensitivity experiment. For ^{13}C{^{1}H} experiment with ^{19}F decoupling use cpfh1.av pulse program. For ^{13}C{^{1}H} experiment with ^{1}H and ^{19}F decoupling use cpfh.av pulse program. Set ^{19}F carrier frequency on one of the ^{19}F resonances.

7. Shimming a CP MAS probe

When shimming a high-resolution liquids probe there is a distinction between on-axis (Z) and off-axis (X, Y etc.) shims. Spinning the sample parallel to the magnetic field direction averages the off-axis inhomogeneity, which may result in spinning sidebands.

In MAS spectroscopy the spinner axis is at an angle, θ_m with the magnetic field direction and the distinction between the traditional on-axis and off-axis shims no longer holds. The spinning rates in MAS spectroscopy, however, are typically at least a few kilohertz; much larger than the magnetic

field inhomogeneity. As a result, the amplitudes of the sidebands are small and shimming may be done with a set of shims that is cylindrically symmetric about the MAS spinner axis.

Such a set of shims can be constructed from combinations of the standard laboratory frame shims, via a transformation to the tilted magic angle frame. In the simplest implementation, the MAS probe is aligned such that the spinner axis is in the laboratory YZ-plane which holds for WB probes in Figure 1 the configuration for SB probes is shown, with the alignment in the XY-plane.

The 'magic angle z-shims' are listed below for orders Z through Z^5 (Sodickson and Cory 1997). The simplest approach is to use the laboratory shims with the largest coefficient, so for instance the Y shim is used as the normal high resolution Z shim. Complete shimming can then in principle be accomplished using only Y, ZY, Z^2Y, Z^4 and Z^5 as the surrogate n^{th} order on-axis shims. Note, however, that the coefficients given for the magic angle shims do not take into account the efficiencies of the shim coils and the use of the other shims listed below may be needed.
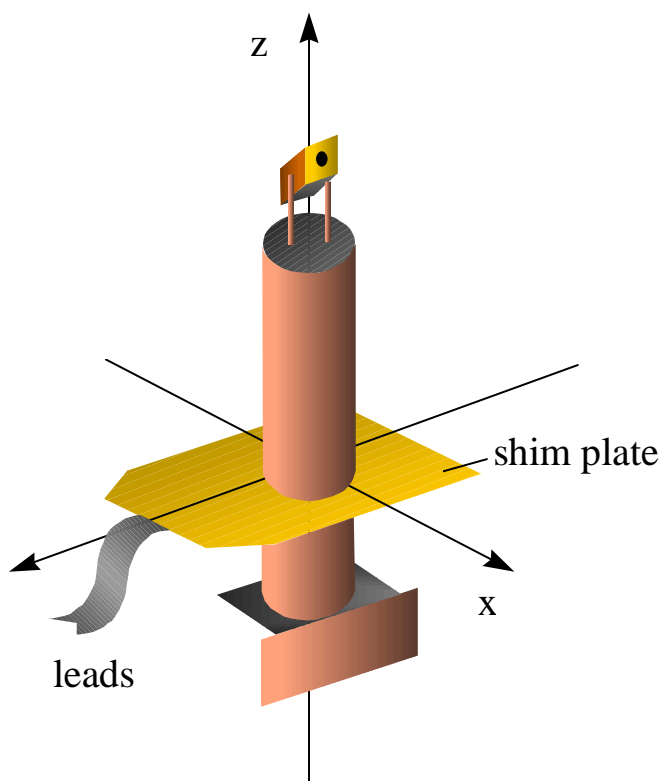
Figure 1: *TFor HRMAS probes and BioSolids Probes, the magic angle spinner axis is aligned with the xz-plane of the shims by positioning the front plate of the probe parallel to the direction of the shim leads. For WB probes, the positioning of the MAS –rotor is by 90º shifted, and the long axis is parallel to the front plate. The y-direction of the shim system is parallel to the direction of the leads / the long side of the RT-shim base plate.*

MAS shims	Laboratory shims
Z_m	$\dfrac{1}{\sqrt{3}}Z - \dfrac{\sqrt{2}}{\sqrt{3}}\mathbf{Y}$
Z_m^2	$(X^2 - Y^2) - 2\sqrt{2}\,\mathbf{ZY}$
Z_m^3	$\dfrac{-2}{3\sqrt{3}}Z^3 - \dfrac{1}{\sqrt{6}}\mathbf{Z^2Y} + \dfrac{5}{\sqrt{3}}(X^2 - Y^2)Z - \dfrac{5}{3\sqrt{6}}\mathbf{Y^2}$
Z_m^4	$\dfrac{-7}{18}\mathbf{Z^4}$
Z_m^5	$\dfrac{-1}{6\sqrt{3}}\mathbf{Z^5}$

To shim the MAS probe, a sample of adamantane is suggested. Make sure the magic angle is adjusted before shimming the probe. Spin the sample at a rate suggested in the following section. It is always best to shim up the probe already under the conditions used later for the real samples. See also chapter 4.4 and 4.5 for discussion regarding the use of direct polarization or cross polarization to shim. Cross polarization is needed if shimming a 2.5 mm CPMAS probe as it gives more signal intensity. Alternatively, make use of a lock control board if available, fill the rotor with D_2O and lock through the X channel on D_2O. Then, shimming on the lock signal is easily possible.

Lock the sample if D_2O is used, tune the probe and start shimming using the Y, ZY, Z^2Y, Z^4 and Z^5 shims as described above. If one of the shims requires an excessive shim current, reduce the current and continue shimming by adding current to another shim from the same group as shown in the table. For instance if the current in ZX is too high, reduce the value and optimize the line shape by adding current to the $(X_2-Y_2)Z$ shim. If the probe is not exactly aligned with the xz plane (see Figure 1), then a small amount of X and XZ may be needed for optimal shimming. A second shim set should be obtained for rotors with spacers (CRAMPS or *hr*-MAS rotors), and the optimal shim settings for these will be somewhat different. Under normal conditions the shim values obtained as described above, will be close to optimal for all other samples. When changing samples, only Y, Z (and ZY) need to be adjusted. Both shim value sets should be stored on disk. Make sure when the shim values are stored that the MAS probe is defined as the current probe in the EDHEAD command.

Note: The alignment of the spinner axis in *hr*-MAS probes is shifted by 90 degrees. The spinner lies in the XZ plane. Through symmetry, the appropriate shims to use are now X, Z, XZ, XZ^2 etc.. See table 3 in the reference.

Reference: A. Sodickson and D.G. Cory, *Shimming a High-Resolution MAS Probe*, J. Magn. Reson. 128, 87-91 (1997)

8. Prosol table and relations file for CP MAS experiments

[1]H Pulses and power levels entry:

Prepare the prosol table for the CPMAS use by entering the ramped pulse into the table to the decoupler channel (F2) with the shaped pulses for PSH16, last row, last column for nucleus [1]H (see figure).

Then return to the standard hard pulses.

Write pulses and power levels into the prosol table as usual for the ^{1}H experiment with adamantane into F1. Use for the decoupling pulse a pulse width of 120% as compared to the maximum possible decoupling field, i.e. for a 4mm WB CPMAS probe this would be 2.5us (100 kHz) for the maximum and then 3.1us (80kHz) for the entry into the field `PCPDP`.   Enter the obtained value for the power level also into the next field for `PLCPD2`. If P3 is measured – during the CP experiments with glycine, this value is entered as `P90` into the appropriate field on the right block, F2, with the power level from `PL12` of the experiment, "1H on channel F2 routed to amplifier A2". The power level for the contact pulse is calculated in the field PLNOE using 62500 Hz. The adjustment for the ramped contact pulse is done in the prosol file automatically during loading!

^{13}C pulses and power levels:

Enter the value for the 4us 90 degree pulse into the field for `P90` and calculate the HH match power level for 62500 Hz in the field `PLCW`.

9. Listings: Relations file, pulse program:cp.av.xyz

```
#****************************************************************
#
# $Source: /sc/CvsTree/sc/gen/src/prg/scripts/tix/prosol/lib/lists/solids_cp,v
$
#
# Copyright (c) 1999
# BRUKER ANALYTIK GMBH
# D-76287 Rheinstetten, Germany
#
# All Rights Reserved
#
```

```
#
# $Id: solids_cp,v 0.0.0 2003/04/26 14:48:33 bau Exp $
#
#****************************************************************

#../conf/instr/spect/prosol/relations/solids_cp
P[0]=P90[F1];
P[1]=P90[F1];               # 90 degree pulse at power level pl11 for x or y
                            # nucleus
P[2]=P90[F1]*2;             # 180 degree pulse at pl11
P[3]=PCPDP[F2];             # 90 degree pulse on 1H at pl12
P[4]=P90[F2]*2;             # 180 degree pulse on 1H at pl12
P[11]=P90[F3];              # 90 degree pulse on F3
P[12]=P90[F3]*2;            # 180 degree pulse for REDOR on F3
P[22]=P90[F2]*2;            # 180 degree pulse for pi decoupling
P[31]=PCPDP[F2]*2-0.2u;     # TPPM decoupling pulse width
PL[1]=PLCW[F1];             # power level for HH contact pulse F1 set to 62.5kHz
PL[2]=PLNOE[F2]-3;          # power level for HH contact pulse F2 62.5kHz -3dB as
                            # we use ramp.100 in parameter set which is a 6dB
                            # amplitude change
                            # set PLNOE to 62.5kHz
PL[3]=PL90[F3];             # power level for pulse on F3
PL[11]=PL90[F1];            # power level for hard pulse F1
PL[12]=PLCPDP[F2];          # set PLCPD to 80 kHz (3.1us pulse with for 4mm probe
                            #(80% of max. possible decoupling field
PL[13]=PLHD[F2];            # power level for homonuclear decoupling, e.g FSLG set
                            # to 80 kHz
SPNAM0=PNSH16[F2];          # set to ramp.100 as default for 1H on F2
P[15]=P_grad1*2;            # HH contact pulse width for 1H-X polarization transfer
```


    ;cp.av

    prosol relations=<solids_cp>

    #include <lgcalc.incl>
    ;cnst20=RF field achieved at pl13
    ;cnst24:additional LG-offset

    #include <trigg.incl>
               ;10 usec trigger pulse at TCU connector I cable 6
    #include <tppm.incl>

```
1 ze                        ;accumulate into an empty memory
  if "aq < 1.0s" goto 2
  print "acquisition time exceeds 1s time limit!"
  goto HaltAcqu

2 d1 do:f2            ;recycle delay, decoupler off in go-loop
#include <prp15.prot>
                     ;make sure p15 does not exceed 10 msec
                     ;let supervisor change this pulseprogram if
                     ;more is needed
#ifndef lacq/* disable protection file for long acquisition change decoupling power !!! or you risk probe
damage */

             /* if you set the label lacq (ZGOPTNS -Dlacq), the protection is disabled */

#include <praq.prot>
                     ;allows max. 50 msec acquisition time, supervisor
                     ;may change  to max. 1s at less than 5 % duty cycle
                     ;and reduced decoupling field
#endif
  1u fq=cnst21:f2
  10u pl12:f2 pl1:f1  ;preselect pl12 drive power for F2, pl1 for F1
  trigg                     ;trigger for scope, 10 usec
  p3:f2 ph1            ;proton 90 pulse
  0.3u
  (p15 ph2):f1 (p15:spf0 pl2 ph10):f2           ;contact pulse with square or ramp
                     ;shape on F2, at pl2 proton power level
  1u cpds2:f2

  go=2 ph31                 ;select appropriate decoupling sequence, cw or
                    ;tppm, both executed at power level 12, or lgs
                    ;executed at power level pl13
  1m do:f2            ;decoupler off
  wr #0                     ;save data to disk
HaltAcqu, 1m               ;jump address for protection files
exit               ;quit

ph0= 0
ph1= 1 3
ph2= 0 0 2 2 1 1 3 3
ph10= 0
ph31= 0 2 2 0 1 3 3 1
```