

a step by step guide

Use \rightarrow , \leftarrow to navigate through this set of instructions

version 2.2 for Topspin 3.x, August 20201 Johannes Leisen, Georgia Institute of Technology johannes.leisen@chemistry.gatech.edu please *do not distribute without my permission*

Sign up for instrument time and assistance is managed via: https://sites.gatech.edu/solidstatenmr/

Please request NMR time for the 300, 400 and 500 MHz instruments for the upcoming week before Friday/noon. You should do this by filling out the form at https://sites.gatech.edu/solidstatenmr/solid-state-nmr-signup/

Once approved your instrument time will be scheduled in SUMS.

The shortest time allocation for solid-state NMR time is one day. Usually, measuring times starts at 10 am and ends at 10 am the following day unless other arrangements have been made.

Solid-state NMR experiments at 700 MHz need to be requested. Requests will be waitlisted. As soon as the waitlist is long enough to warrant the hardware change of the cryoprobe experiments will be scheduled.

Pls send an e-mail to johannes.leisen@chemistry.gatech.edu for any unexpected changes and cancellation in your NMR schedule.

Expectations

In order to operate the spectrometer successfully it is important to have an understanding of the following physical principles/phenomena:

- Setting the instrument on resonance:
 What happens to an FID and peak if the sender frequency (sfo1) is changed?
 How can this be explained with respect to the rotating coordinate system (RCS).
- (2) Adjusting pulse lengths: effects of a π and $\pi/2$ pulse in the RCS.
- Basic solid state nmr pulse sequences: DP vs. CP
 Effect of repetition delays on signal intensities in DP
 Effect of contact time on signal intensities in CP

IT IS EXPECTED THAT ALL USERS OF THE SOLID-STATE NMR INSTRUMENTS HAVE AN UNDERSTANDING OF THESE PRINCIPLES.

Safety

No open-toed shoes are allowed in the NMR lab No food or drink is to be brought into the NMR lab

Chemistry lab in G143

You must follow the GT-dress code for laboratories when working in this lab. (lab goggles, lab coat, no shorts or skirts). Safety glasses are required even when just stepping into this laboratory.

Magnets:

Do not bring any ferromagnetic materials close to the magnet. Please ensure to remove all ferromagnetic materials from your body before coming closer than 9 ft to any of the magnets. This includes: your wallet, credit cards, cell-phones, hair pins and clips, ...

The laboratory is monitored for a safe oxygen concentrations. An alarm will sound if oxygen levels fall below a safe level. Please leave the laboratory immediately if the alarm sounds.

Some of the electronics in the NMR instruments use hazardous voltages. Unless you have not explicitly been instructed to do so: do not unplug any electrical connections.

Pregnancies?

data management

Data Export Data can easily be stored on memory sticks or USB hard drives. Please <u>do not insert memory-sticks directly into the acquisition computer</u>. Please insert your USB-storage medium in the data-station computer in the NMR lab. Then use win-SCP to transfer your data to your storage device. IP addresses are: 10.137.198.78 for the AV3-300 (obiwan) 10.137.198.79 for the AV3-400 (jaba) 10.137.198.76 for the AV3-500 (yoda) 10.137.198.87 for the AV3-700 (rey)

Files are stored in the directory /opt/"topspin-version"/user/nmr. Each directory corresponds to the experiment name (cf. IV.2.). Topspin has even the option of emailing you the archived files.

You may also safe your data on Prism or OneDrive. Instructions can be found at https://sites.gatech.edu/solidstatenmr

Processing using an External Computer

It is often not convenient to process your data on the acquisition computers of the NMR instrument. We recommend (but do not support) several software options:

- Topspin : Bruker-Biospin currently distributes free academic licenses.
- MestreNova: the Georgia Tech office of Information Technology has purchased a site license.
- Spinworks: free at http://www.umanitoba.ca/chemistry/nmr/spinworks/



Rates for the use of our instruments are published at: https://chemistry.gatech.edu/organization/nmr/rates.

NMR time is accounted and charged via Georgia Tech's Shared User Management System (SUMS). Starting the NMRsoftware (Topspin) will automatically start charging you via the SUMS system. A termination of Topspin will send a message to SUMS and your charges will be calculated.

IMPORTANT : Pls wait for a "pop-up message" from SUMS after terminating your Topspin session. Chances are that you will continue to be charged if you do not receive this message.

Changing incorrect charges in SUMS is tedious and will lead to a lengthy e-mail trail. Therefore we cannot guarantee that we are able to write off charges, which were caused by user mistakes.

The option of an auto-logout for SUMS and topspin after the completion of a data acquisition comes in handy for experiments, which run for several hours:

Instead of starting the experiment with "zg" type "SUMS_explo". You are asked to enter the number of experiments (1).

You may also queue several experiments by preparing them in subsequent experiment numbers. Go to the experiment with the lowest number, type "SUMS_explo" and enter the number of queued experiments. The macro will run all of the prepared experiments and then log you out. ("zg-safety" must be set to "off").



13C CP-MAS includes ¹H, ¹³C DP

so far there is only instructions for this experiment

CP-MAS for other nuclei ³¹P, ²⁹Si,...

MAS of quadrupolar nuclei (²⁷Al, ⁷Li, ²³ Na, ...)

1H: T₁,T₂ and Spin Diffusion

Diffusion NMR

NMR Microimaging



login			
Measure Reference S	ample	Measure Sample of interest	
spin up		¹ H	
¹ H	1	create file	
load reference file		probe tuning	
probe tuning		measure 1st spectrum	
measure first spectrum	adjust power level	set sender frequency	
process spectrum	measure first spectrum	measure spectrum	
set sender frequency	load reference file	measure spectrum	
adjust power level	¹³ C VACP	¹³ C DP measu	re spectrum
¹³ C DP	adjust power level	create file	tunina
lood reference file		probe tuning	file
load reference file	measure first spectrum	measure spectrum	
probe tuning	load reference file		³ C CP
measure first spectrum	¹³ C CP	'	
adjust power level		-	





login

- 1. Log into the LINUX computer.
- 2. Start Topspin: click on the Topspin icon.
- 3. Acknowledge that your instrument use is monitored and charged via SUMS.

Your reference sample is adamantane. A packed rotor with adamantane will be provided.



13C CP-MAS includes ¹H, ¹³C DP

MAS II as used on the AV3-400

MAS is controlled directly form the MASII unit. Use ONLY the automatic mode:

- a.) on the MAS unit: tap: stop, wait for spinning speed to reduce to 0, tap eject, (remove sample if any)
- b) tap *insert*, insert sample, put cap on transfer tube (push it down only halfway)

instead of the cap you can also use a removable plastic cage, which is used to collect ejected samples.

- c) tap auto
- d) on the MASII: tap on the number of the spinning speed to set the speed (VA) to 3000 Hz.
- e) tap go and watch the spinning speed. If the sample is nicely packed, the sample should easily spin up. If spinning is unstable, or sample is spinning noisily tap *stop*, *eject* remove the rotor and pack it again.
- f) if sample is spinning stable, the spinning speed can be increased in steps of 2000 kHz.

routine spinning speed: 10 kHz, max spinning speed 14 kHz

MAS III as used on the AV3-300,AV3-500 and AV3-700 steps are in principle identical to the MAS II, however the unit is now controlled via Topspin.

*type MASDISP to open a window, with control options of the MAS-unit.

4mm probes: routine spinning speed: 10 kHz, max spinning speed 14 kHz 3.2 mm probes: routine spinning speed: 18-20 kHz, max spinning speed 24 kHz 1.5 mm probes: routine spinning speed: 20-50 kHz, max spinning speed 64 kHz

Spectrometer adjustments using adamantane: do not spin faster than 3 kHz! actual samples: spin as fast as possible 10 - 14 kHz for 4 mm rotors)



Measure Reference Sample Measure Sample of interest spin up ¹H ad reference djust power level sure first spectrum asure first spectrum ad reference file ¹³C DP ¹³C VACP adiust power level create file ¹³C DP djust power level orobe tunina easure first spectrum load reference file ¹³C CP oad reference file probe tuning neasure first spectrun ¹³C CP

13C CP-MAS includes ¹H, ¹³C DP

load ¹H reference file

select and load reference file:

Drag and Drop the reference file from your user directory into the main window



check parameter sets:

It is advisable to check all parameters for correctness before starting an experiment. The tabs in the main window display provide options for viewing and editing: spectrum, processing parameters, acquisition parameters, title, fid

Tab-AcquPars: You will see a list of all possible acquisition parameters. (ased , \mathbb{R} will display only those parameters, which are relevant for the execution of the pulse program.

important parameters:

ns: number of scans

p1: 90 deg pulse length (typically 5 us).

pl1: power level of amplifier. This value is entered in dB and ranges from (-6 to 120). Atention: low values lead to higher amplifier outputs

d1: repetition time between scans: 3 - 5 s

reference files setup_adamantane should be available in your user directory.

load no. 1

(1)

(2) ¹³C DP

(3) ¹³C CP

(4) ¹³C VACP

Measure Reference Sample Measure Sample of interest spin up ^{1}H ¹H orobe tuninc robe tuning adiust power level neasure first spectrum bad reference file et sender frequenc ¹³C DP ¹³C VACP adjust power level measure spectrur create file ¹³C DP djust power level load reference file neasure first spectrum ¹³C CP bad reference file probe tuning neasure first spectrum ¹³C CP diust power level

13CCP-MAS includes ¹H, ¹³C DP

probe tuning

Probe tuning:

Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

For ¹H

At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude;

You need to switch several times between TH and MH in order to get a good result.

For ¹³C

At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;

Several iterations are needed to get a good result.

Switch between 1H and 13C and find tune both channels until both channels show a good tune.

Pay attention not to accidentally turn knob adjusting the magic angle!

correct tune





Measure Reference Sample Measure Sample of interest spin up ^{1}H ¹H create file load reference file djust power level asure first spectrur easure first spectrum ad reference file t sender frequency ¹³C DP ¹³C VACP just nower leve ¹³C DP djust power level load reference file easure first spectrum 13C CP probe tuning oad reference file easure first spectrum ¹³C CP



measure first spectrum for ¹H

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

Start the experiment:

(zg / 🕨)

If you observe any problems: immediately (stop/ .

There is a command (halt/ I), which performs a more defined "soft-stop".



Note: the shape of the fid will change with the sender frequency. Depending on the setting of the receiver phase real and imaginary part of the fid can be pointing up or down.

correct fid



login Measure Reference Sample spin up ¹H load reference file probe tuning measure first spectrum set seruice frequency adjust power level ¹3C DP load reference file probe tuning measure first spectrum ¹3C CP load reference file ¹3C CP ¹3C CP ¹3C CP ¹3C CP

13C CP-MAS includes ¹H, ¹³C DP

process the NMR spectrum for ¹H

Processing a FID into a spectrum:

involves the following steps, which can be called by typing the commands below:

em: exponential multiplication, apadozation function to reduce noise at the cost of

resolution. The effect of em is adjusted by the parameter lb (0-200). ft: fourier transformation

apk: automatic phase correction

If the spectrum is not phased correctly you should phase it manually:

Manual mouse on the 0-icon.

Hold the left mouse buttom and move the mouse up and down. Further corrections can be done in a similar way with the 1-icon.

🖳 when finished.

correctly processed ¹H spectrum of adamantane



the pl	nasing opti	ion can be s	selected fro	m the proce	essing statu	s bar
	he phasing option can be selected from the processing status bar Start Acquire Process Analyse Publish A Proc. Spectrum → Adjust Phase → Acalib. Axis → M Pick Peaks → J Integrate → Advanced →					
	Λ Pro <u>c</u> . Sp	ectrum 🚽 🔨	♦ Adjust Pha	se → 🕅 Advanced	ib. A <u>x</u> is ⊽	
	W/Y L K		j <u>i</u> ntegrate	Auvance		





set ¹H sender frequency

Adjust the sender frequency to excite the center of your spectrum:

,move the cursor on the maximum of the adamantane spectrum.

Click the left mouse button to define a new sender frequency.

Note down the value for the sender frequency

SF01 =

you can also save the sender frequency to the memory by typing ctrl-C

)● [[\$] *2 /2] \$} \$\$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$		
rowser Last50 Groups	1 Acquisition finished: setup_adamantane 1 1 /opt/topspin3.5pl5/data/hanno/nmr	5 ⁴ 1
a erkang Bhanno		
⊒ hanno_dsx ⊒ iwalton	adamantana, onepulse 1H	
acceptance	1.96 ppm / 46G.74 Hz / 300.200461 MHz	
	SET SEQUAL REQUEXIES FROM CORSER FASTION Define: Left-click initie data vindev	
	A	
	40 20 0 -20	- 40 [ppm
	Amplifier Control Acquisition information	MAS Spin Rate Probe Temperature 1





adjust the rf-power for ¹H

Adjustment of 90 degree pulse length:

- * set the pulse length p1 to 5 μs (you can do this fastest by typing "p1 5u")
- * gs: enters the setup mode. The FID is displayed for each scan, but data are not stored, parameters can be changed.
- * HI FT on the fly: spectra are displayed while scanning.
- * double value of p1 (type: "p1 10u")
- * adjust the power level plw1 (in Watts) to minimize the signal amplitude (the signal should approach a "flat" line).
- * change p1 back to $5 \mu s$ (type: "p1 5u"): the signal should jump to a maximum.
- * 🔍 when finished

Note down the power level for ¹H: plw1=

Note: you may also use the option "popt" to find the minimum power level at $p1=10 \ \mu s$



13C CP-MAS includes ¹H, ¹³C DP

load ¹³C DP reference file

select and load reference file:

Drag and Drop the reference file from your user directory into the main window



reference files are available in the directory setup_adamantane:



Enter frequency for ¹H channel:

sfo2: enter value previously determined as sfo1 for ¹H.

sfo1: sender frequency for observation channel sfo2: sender frequency for indirect channel.

Check/Enter Paramters

click on the tab: AcquPars, click parameters should be pulseprog: hpdec D1: 4s TD-SI=2k ns=4

power levels X-channel: plw1 ¹H channel: plw12

sfo1: sender frequency of 13C (detection frequency) sfo2: sender frequency of 1H (indirect fequency)

Measure Reference Sample Measure Sample of interest spin up 1<mark>Ĥ</mark> ¹H create file oad reference file orobe tuning probe tuning measure first spectrum adjust power level neasure first spectrum rocess spectrum bad reference file set sender frequency ¹³C DP ¹³C VACP adjust power level neasure spectrur ¹³C DP adjust power level neasure first spectrum oad reference file ¹³C CP load reference file probe tuning ¹³C CP

13C CP-MAS includes ¹H, ¹³C DP probe tuning

Probe tuning: Wobb: the instrument will dis

Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

For ¹H

At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude; Several iterations are needed to get a good result.

For ¹³C

At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;

Several iterations are needed to get a good result.

Switch between 1H and 13C and find tune both channels until both channels show a good tune.

Pay attention not to accidentally turn knob adjusting the magic angle!





login

Measure Reference Sample

spin up		spin up	
¹ H	I	'⊟ create file	
load reference file		probe tuning	
measure first spectrum	adjust power level	measure 1st spectrum	
process spectrum	measure first spectrum	measure spectrum	
set sender frequency adjust power level	¹³ C VACP	¹³ C DP	
¹³ C DP	adjust power level	create file	probe tu
load reference file	measure first spectrum	probe tuning measure spectrum	create file
probe tuning	load reference file		¹³ C
measure first spectrum	¹³ C CP		-

Measure Sample of interest

CP



measure first spectrum for ¹³C

acquire spectrum:

* rga, zg

* when finished measuring process data using em, ft, apk

Calibrate the ppm scale:

- * Calibrate Axis/ 🔥
- * move the cursor on the to pof the left peak, right click and enter the reference value (38.45 ppm).

calib. axis can be selected from the processing status bar								
	<u>S</u> tart	<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	P <u>u</u> blish			
	∫ Proc. Spectrum →] Adjust Phase →] 🍂 Calib. Axis →							
Mreck Peaks → Integrate → Advanced →								

Start Acquire Process Analyse Pu Analyse Pu Analyse Pu	ilish View Manage 🕢 trum → 🖓 Adjust Phase → 🗼 Calib. Axis → 🎊 Pick Peaks → 🥤 Integrate → Advanced →	11 [
I Proc. spec I Proc. spece I Proc. spece spe	Trum Adjust Phase Calib. Asis Mick Yeaks Integrate Advanced Image: Adjust Phase Image: Adjust Phase Image: Adjust Phase Image: Adjust Phase Adjust Phase Image: Adjust Phase Image: Adjust	
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		nghad far at the analysis for any
	300 200 100 0	[ppm
	Amplifier Control Acquisition Information MAS Spin Rate Pro no acquisition running 2999 Hz	21.6 °C

 login
 Measure Reference Sample
 Measure Sample of interest spin up

 1H
 Spin up
 1H

 load reference file
 rcreate file
 1H

 probe tuning
 adjust power level
 measure first spectrum

 set sender frequency
 adjust power level
 13C VACP

 13C DP
 adjust power level
 13C VACP

 load reference file
 13C VACP
 13C DP

 load reference file
 13C CP
 13C CP

 adjust power level
 13C CP
 13C CP

 adjust power level
 13C CP
 13C CP



adjust the rf-power for ¹³C DP

Adjustment of 90 degree pulse length:

- * set the pulse length p1 to 5 μ s (you can do this fastest by typing "p1 5u")
- * gs: enters the setup mode. The FID is displayed for each scan, but data are not stored, parameters can be changed.
- * H FT on the fly: spectra are displayed while scanning.
- * double value of p1 (type: "p1 10u")
- * adjust the power level plw1 to minimize the signal amplitude.
- * change p1 back to $5 \mu s$ (type: "p1 5u"): the signal should jump to a maximum.
- * 💷 when finished

Note down the power level for ${}^{13}C$: plw1=

The setup for DP is complete, acquire one final spectrum: zg, when finished process using: em,ft,apk





load ¹³C CP reference file

select and load reference file:

Drag and Drop the reference file from your user directory into the main window



reference files are available in the directory setup_adamantane:



Enter frequency for ¹H channel:

sfo2: enter value previously determined as sfo1 for ¹H.

sfo1: sender frequency for observation channel sfo2: sender frequency for indirect channel.

rf power settings:

plw1=power level for X (to be adjusted)plw2=power level for ¹H 90 degree pulsespw0 =power level for 1H contact pulse(often same as PIW2)power level for ¹H decoupling



13C CP-MAS Includes ¹H, ¹³C DP measure first spectrum for ¹³C CP

acquire spectrum:

* rga, zg

* when finished measuring process data using em, ft, apk

Calibrate the ppm scale:

- * Calibrate/ 👍
- * move the cursor on the top of the left peak, right click and enter the reference value (38.45 ppm).







adjust the rf-power for ¹³C CP

Fine-tuning of power for CP conditions:

The optimum conditions for CP-experiments can be found best by using the macro "popt". This macro records spectra while systematically varying a specified parameter.

Before running the macro you should run the CP-MAS experiment for ns=4 once. Process using "em,ft,apk".

Make sure that your spectrum is displayed properly. Type "dpl" and confirm the ppm, values, which will be displayed. This will ensure that "popt" will give a reasonable display.

Type *popt* and enter the parameters as displayed. (note: the range of PLW1 depends on the instrument and it needs to be centered +/-10 W around the expected value for the CP-condition)

Click "start optimize" to start the experiment. You should confirm both questions with (overwrite, and start) with "y".

You will now see a display CP-MAS spectra measured for varying values of PLW1 and it will be quite easy to identify the PLW1, which provides the highest signal-intensity.

It will take a few minutes to measure all of the CP-MAS spectra and complete the display.

Max. intensity at plw1=

Typing "re <expno> 1" switches back to the experiment used for the original setup (with the number <expno>). "re <expno> 999" switches back to he display produced by popt.

Optimize: step by step group: 0 parameter: plw1 optimum: posmax Startval: expected power level -5W Endval: expected power level +5W Nexp: will be calculated Varmod: lin Inc: 1

Note: The power level to achieve the maximum CPsignal depends also on the spinning frequency. Therefore 2 maxima are found for adamantane. Using the maximum found for the higher power usually give good CP-conditions for many samples spinning at MAS speeds of up to 10 kHz. However to absolutely make sure that you run your sample under optimum CP conditions you should run popt again on your actual sample. If this is not an option I recommend that you use ramped contact pulses for your CPexperiment.





load ¹³C VACP reference file

select and load reference file:

Drag and Drop the reference file from your user directory into the main window



Enter frequency for ¹H channel:

plw1= plw2= spw0 plw12=

sfo2: enter value previously determined as sfo1 for ¹H.

sfo1: sender frequency for observation channel sfo2: sender frequency for indirect channel.

power level for X (take from previous popt)
power level for ¹ H 90 degree pulse
power level for 1H contact pulse (to be adjusted)
power level for ¹ H decoupler

reference files are available in the directory setup_adamantane:

(1) 1 H (2) 13 C DP (3) 13 C CP (4) 13 C VACP



acquire spectrum:

* rga, zg

* when finished measuring process data using em, ft, apk

Calibrate the ppm scale:

- * Calibrate/ 🙏
- * move the cursor on the to pof the left peak, right click and enter the reference value (38.45 ppm).





Fine-tuning of power for CP conditions:

The optimum conditions for VACP-experiments can be found best by using the macro "popt".

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Make sure that your spectrum is displayed properly. Type "dpl" and confirm the ppm, values, which will be displayed. This will ensure that "popt" will give a reasonable display.

Type popt and enter the parameters as displayed (note: the range of SPW) depends on the instrument and it needs to be centered +/-30 W around the expected value for the CP-condition).

Click "start optimize" to start the experiment. You should confirm both questions with (overwrite, and start) with "y".

You will now see a display CP-MAS spectra measured for varying values of SPW0 and it will be quite easy to identify the value, which provides the highest signal-intensity.

It will take a few minutes to measure all of the CP-MAS spectra and complete the display.

Max. intensity at SPW0=



adjust the ¹H rf-power for ¹³C VACP

store as 2D dat	a (ser file)								
The AU program specified in AUNM will be executed			WDW= no						
Perform automatic baseline correction (ABSF)				PH_mod= pk					
Overwrite existing files (disable confirmation Message)				FT_mod= fqc					
Stop sample sp	inning at the e	end of optimization	n (mash)						
Run optimizatio	n in backgrou	nd							
No display of es	timated runni	ng time							
Calculate optim	um after POPT	has finished, but	do not store in d	ataset					
Correlate 2D Co	ontainer with e	xperiment							
OPTIMIZE	GROUP	PARAMETER	OPTIMUM	STARTVAL	ENDVAL	NEXP	VARMOD	INC	
Step by step	0	plw1	POSMAX	48	55	8	LIN	1	

Optimize: step by step group: 0 parameter: SPW0 optimum: posmax Startval: expected power level -30W Endval: expected power level +30W Nexp: will be calculated Varmod: lin Inc: 5W

Start optimize	Skip current optimi	Show protocol	Add parameter	Restore	Save	Read array file
Save array file as	Stop optimization	Delete paramet	Display Dataset	Update ProcPars	Help	

Note:compared to the regular CP experiment the dependency of the intensity on the power level SP0 is much more robust.



Congratualtions you did it! the instrument is tuned and you can now start measuring your sample



13C CP-MAS includes ¹H, ¹³C DP

spinup

MAS II as used on the AV3-400

MAS is controlled directly form the MASII unit. use ONLY the automatic mode:

- a.) on the MAS unit: tap: stop, wait for spinning speed to reduce to 0, tap eject, (remove sample if any)
- b) tap *insert*, insert sample, put cap on transfer tube (push it down only halfway)
- instead of the cap you can also use a removable plastic cage, which is used to collect ejected samples. c) tap auto
- d) on the MASII: tap on the number of the spinning speed to set the speed (VA) to 3000 Hz.
- e) tap go and watch the spinning speed. If the sample is nicely packed, the sample should easily spin up.
- If spinning is unstable, or sample is spinning noisily tap stop, eject remove the rotor and pack it again.
- f) if sample is spinning stable, the spinning speed can be increased in steps of 2000 kHz.

routine spinning speed: 10 kHz, max spinning speed 14 kHz

MAS III as used on the AV3-300, AV3-500 and AV3-700

steps are in principle identical to the MAS II, however the unit is now controlled via Topspin. *type MASDISP to open a window, with control options of the MAS-unit.

4mm probes: routine spinning speed: 10 kHz, max spinning speed 14 kHz 3.2 mm probes: routine spinning speed: 18-20 kHz, max spinning speed 24 kHz 1.5 mm probes: routine spinning speed: 20-50 kHz, max spinning speed 64 kHz

Spectrometer adjustments using adamantane: do not spin faster than 3 kHz! actual samples: spin as fast as possible 10 - 14 kHz for 4 mm rotors)

rotor:

- * balanced packing
- * undamaged, tight fitting cap
- * cap is all the way in
- * never enter just the highest spinning speed and press "go".
- * always spin up your sample in steps.
- * an oscilloscope may be used to check that the spinning is really stable.



13C CP-MAS includes ¹H, ¹³C DP

create file for ¹H

select and load reference file:

Drag and Drop the reference file from your user directory into the main window



reference files are available in the directory setup_adamantane:



Type "edc" (or "new") to create a new file using the existing ¹H parameter set.

New								
Prepare for a new experiment by creating a initializing its NMR parameters according to For multi-receiver experiments several datas Please define the number of receivers in the	new data set and the selected experiment sets are created. e Options.	type.						
NAME			provide experi	ment name	•			
EXPNO			provide experi	ment No				
PROCNO			usually 1					
Use current parameters								
○ Experiment		Select						
 Options 								
Set solvent	Acetic	-						
Execute 'getprosol'								
O Keep parameters	P 1, 01, PLW 1 🗸 C	Change						
DIR	hard disk loc	ation with	your username			-		
Show new dataset in new window								
Receivers (1,2,16)	1							
		your free	space to record san	nple informati	on etc			
IIILE								
					QK	Cancel	More Info	Help
					-			





probe tuning

correct tune



Probe tuning:

Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

For ¹H

At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude; Several iterations are needed to get a good result.

For ¹³C

At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;

Several iterations are needed to get a good result.

Switch between 1H and 13C and find tune both channels until both channels show a good tune.

Pay attention not to accidentally turn knob adjusting the magic angle!



Measure Reference Sample Measure Sample of interest spin up ¹H ¹H oad reference robe tuninc djust power leve ad reference file ¹³C DP ¹³C VACP 13C DP easure first spectrum load reference file ¹³C CP bad reference fil probe tuning ¹³C CP



measure first spectrum for ¹*H*

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

Make sure that a complete decay is observed for the FID. If you are "clipping" data" : double, quadruple,... the parameters SI and TD

Start the experiment:

(zg / 🕨)

If you observe any problems: immediately (stop/ .

There is a command (halt/ .), which performs a more defined "soft-stop".

Processing a FID into a spectrum:

involves the following steps, which can be called by typing the commands below:

em: exponential multiplication, apadozation function to reduce noise at the cost of

resolution. The effect of em is adjusted by the parameter lb (0-200). ft: fourier transformation a**pk**: automatic phase correction

If the spectrum is not phased correctly you should phase it manually:

. move the mouse on the 0-icon.

Hold the left mouse buttom and move the mouse up and down. Further corrections can be done in a similar way with the 1-icon.

🖳 when finished.



13C CP-MAS includes ¹H, ¹³C DP

set ¹H sender frequency

Adjust the sender frequency to excite the center of your spectrum:

,move the cursor on the maximum of the adamantane spectrum.

Click the left mouse button to define a new sender frequency.

Note down the value for the sender frequency

SF01 =



Measure Reference Sample Measure Sample of interest spin up ¹H ¹H oad reference file robe tuninc probe tuning diust power level easure first spectrum ad reference file ¹³C DP ¹³C VACP diust nower leve create file ¹³C DP djust power level probe tuning load reference file easure first spectrum ¹³C CP oad reference file probe tuning neasure first spectrum ¹³C CP diust power level



measure spectrum for ¹H

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

Start the experiment:

(zg / 🕨)

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🖳 when finished.



here comes the decision: CP or DP?

as a general rule: broad lines in 1H => CP, VACP for MAS > 10 kHz broad lines in 1H => DP

measure both if not sure, as you know, both experiments measure different things

login			
Measure Reference Sa	ample	Measure Sample of	interest
spin up		spin up	
1		¹ H	
'H Joad reference file		create file	
		probe tuning	
	adjust power lovel	measure 1st spectrum	
measure first spectrum	aujusi power lever	set sender frequency	
process spectrum	Ineasure hist spectrum	measure spectrum	
set sender frequency	Idad reference file	120 00	
adjust power level	¹³ C VACP	¹³ C DP	measure spectrum
¹³ C DP	adjust power level		probe tuning
load reference file	measure first spectrum		create file
probe tuning	load reference file	- mododio opeorium	¹³ C CP
measure first spectrum	¹³ C CP		·
adjust power level	·		

13C CP-MAS includes ¹H, ¹³C DP

create file for ¹³C

load your adamantane ¹³C reference file

reference files are available in the directory setup_adamantane:



Enter frequency for ¹H channel: sfo2: enter value previously determined as sfo1 for ¹H.

sfo1: sender frequency for observation channel sfo2: sender frequency for indirect channel.

itializing its NMR parameters according to or multi-receiver experiments several data lease define the number of receivers in th	the selected experiment asets are created. ne Options.	type.						
VAME	/		provide exp	eriment nam	е)	
EXPNO	r		provide exp	eriment No				
PROCNO			usually 1					
Use current parameters								
Experiment		Select						
Options								
Set solvent	Acetic	-						
Execute 'getprosol'								
O Keep parameters	P 1, 01, PLW 1 👻 C	Change						
DIR	hard disk loc	ation with	n your usernam	ne		-		
Show new dataset in new window						_		
Receivers (1,2,16)	1							
		vour free	space to record s	ample informa	tion etc			
ATLE								

Prepare for a new experiment by creating a new data set and

Measure Reference Sample Measure Sample of interest spin up ¹H ^{1}H oad reference file probe tuning probe tuning adjust power level asure first spectrum neasure first spectrum oad reference file ¹³C DP ¹³C VACP diust power level ¹³C DP adjust power level orobe tunina load reference file neasure first spectrum ¹³C CP bad reference file probe tuning ¹³C CP easure first spectrum djust power level



Probe tuning:

Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

For ¹H

At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude; Several iterations are needed to get a good result.

For ¹³C

At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;

Several iterations are needed to get a good result.

Switch between 1H and 13C and find tune both channels until both channels show a good tune.

Pay attention not to accidentally turn knob adjusting the magic angle!

correct tune





Measure Reference Sample Measure Sample of interest pin up ^{1}H ¹H bad reference file robe tunina djust power level reference file ¹³C DP ¹³C VACP ¹³C DP djust power level easure first spectrum oad reference file ¹³C CP probe tuning load reference file easure first spectrum ¹³C CP

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

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If the spectrum is not phased correctly you should phase it manually: . move the mouse on the 0-icon.

Hold the left mouse buttom and move the mouse up and down. Further corrections can be done in a similar way with the 1-icon. measure only a few scans (ns=32) to check your S/N.

C CP-MAS

spectrum for ¹³C

includes ¹H. ¹³C DP

measure

Then measure your spectrum with more scans.

Most ¹³C spectra need at least ns=1024=1k. Demanding samples need ns=10-20k.

You may follow long runs without stopping the expt by typing: "tr"

data will then be availabe for regular processing using em,ft, apk.

🖳 when finished.



Good luck with your Research