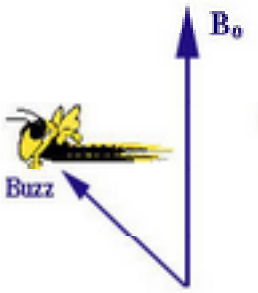


Georgia Tech
NMR Center

Solid-State NMR

a step by step guide

Use →, ←
to navigate through this set of instructions

version 2.1 for Topspin 3.x, August 2023
Johannes Leisen, Georgia Institute of Technology
johannes.leisen@chemistry.gatech.edu

Solid-State NMR

reserve NMR time

Sign up for instrument time and assistance is managed via:
http://pwp.gatech.edu/nmr-center/

Please request NMR time for the 300, 400 and 500 MHz instruments before Friday/noon will be considered for the upcoming week. Pls do this by filling out the form at https://sites.gatech.edu/nmr-center/solid-state-nmr-2/

Once approved your instrument time will be scheduled in SUMS.

The shortest time allocation for solid-state NMR time is one day. 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.

Solid-State NMR

Expectations

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

(1) 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.

(3) 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.

Solid-State NMR

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?

Solid-State NMR

data management

Data Export
Data can easily be stored on memory sticks or USB hard drives.
Please do not insert memory-sticks directly into the acquisition computer.
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:
192.168.0.3 for the AV3-300
192.168.0.4 for the AV3-400
192.168.0.184 for the AV3-500
192.168.0.7 for the AV3-700

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 OneDrive or Labarchives. Instructions can be found at pwp.gatech.edu/nmr-center.

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 https://home.cc.umanitoba.ca/~wolowiec/spinworks/

Solid-State NMR

Accounting

Rates for the use of our instruments are published at: https://sites.gatech.edu/nmr-center/charges/.

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 "exp_lo".
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 "exp_lo" 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").

Solid-State NMR

Experiments

^{13}C CP-MAS
includes ^{1}H, ^{13}C DP

so far there is only instructions for this experiment

CP-MAS
for other nuclei ^{31}P, ^{29}Si,...

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

^{1}H: T_1, T_2 and Spin Diffusion

Diffusion NMR

NMR Microimaging

Solid-State NMR

^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

login

Measure Reference Sample

spin up

^{1}H

load reference file

probe tuning

measure first spectrum

process spectrum

set sender frequency

adjust power level

^{13}C DP

load reference file

probe tuning

measure first spectrum

adjust power level

adjust power level

measure first spectrum

load reference file

^{13}C VACP

adjust power level

measure first spectrum

load reference file

^{13}C CP

Measure Sample of interest

spin up

^{1}H

create file

probe tuning

measure 1st spectrum

set sender frequency

measure spectrum

^{13}C DP

create file

probe tuning

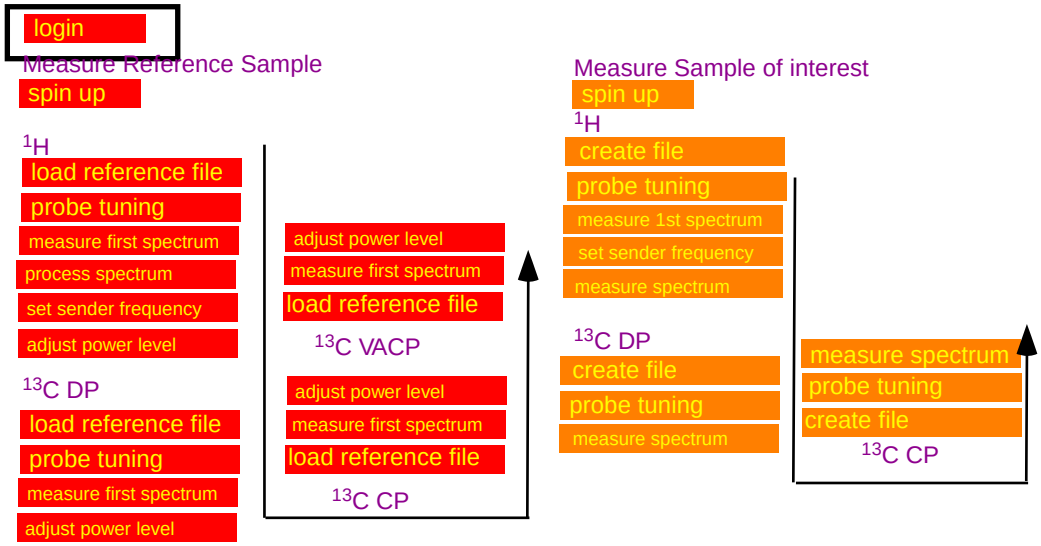
measure spectrum

measure spectrum

probe tuning

create file

^{13}C CP



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

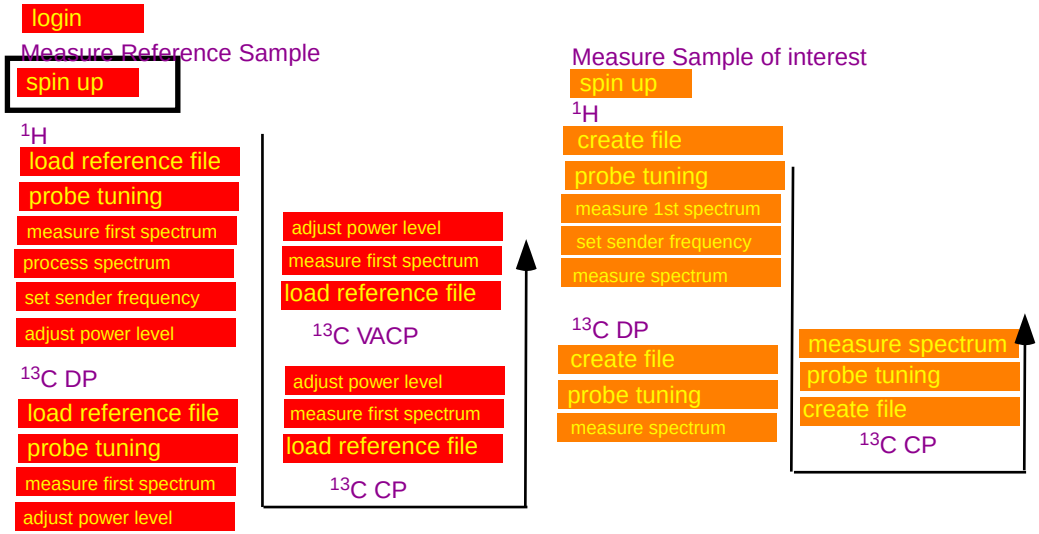
login

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.

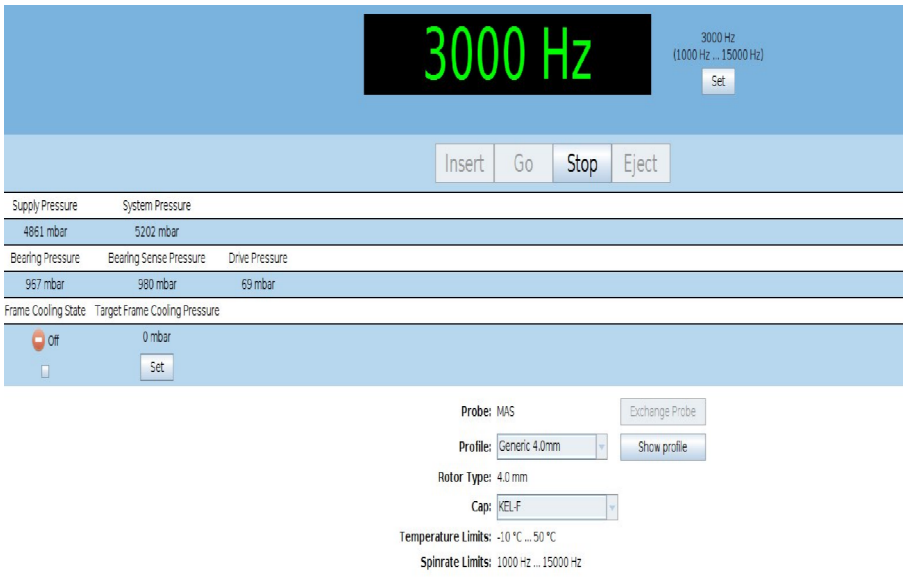


^{13}C CP-MAS

includes ^{1}H, ^{13}C 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),
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.

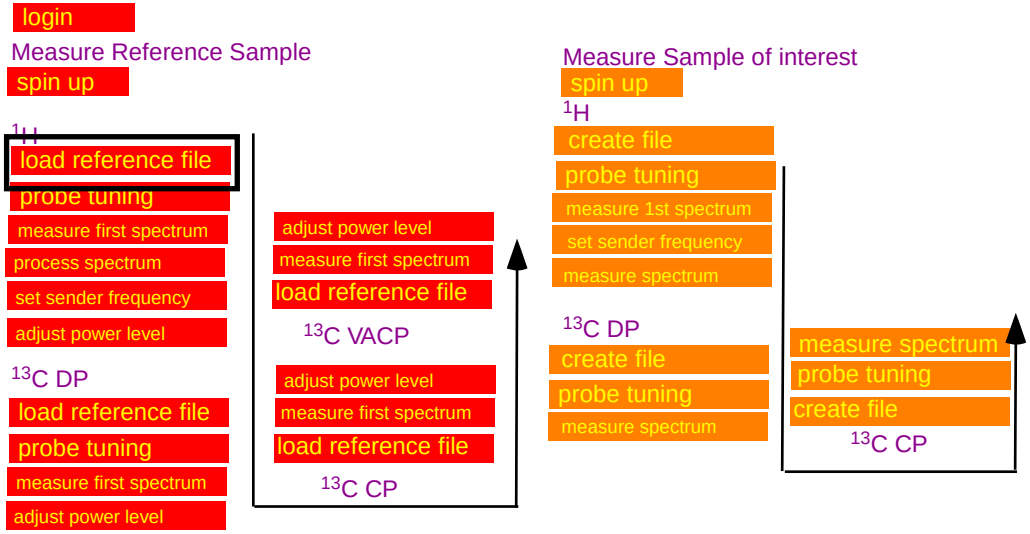
routine spinning speed: 10 kHz, max spinning speed 14 kHz



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)



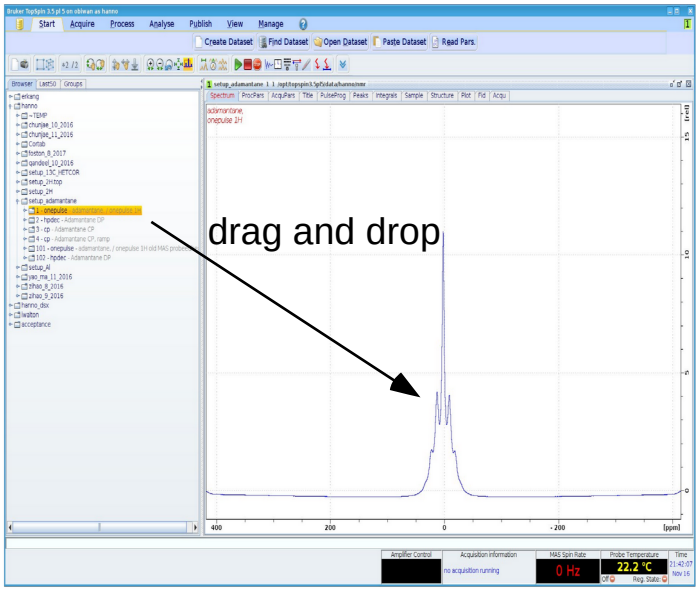
^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

load ^{1}H reference file

<u>select and load reference file:</u>

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



reference files are available in the directory setup_adamantane:

(1) ^{1}H

(2) ^{13}C DP

(3) ^{13}C CP

(4) ^{13}C VACP

load no. 1

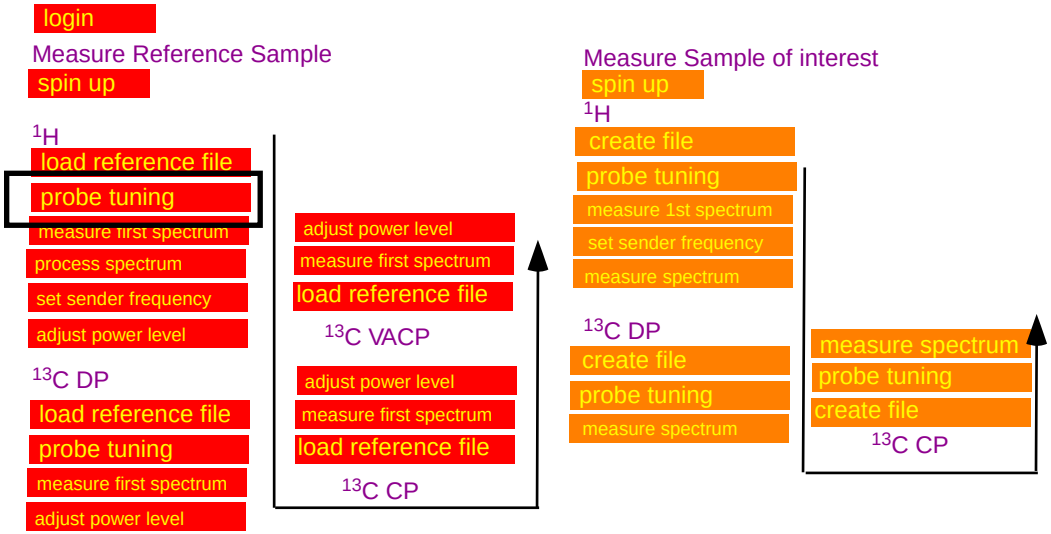
<u>check parameter sets:</u>

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 , will display only those parameters, which are relevant for the execution of the pulse program.

<u>important parameters:</u>

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



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

probe tuning

<u>Probe tuning:</u>
Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

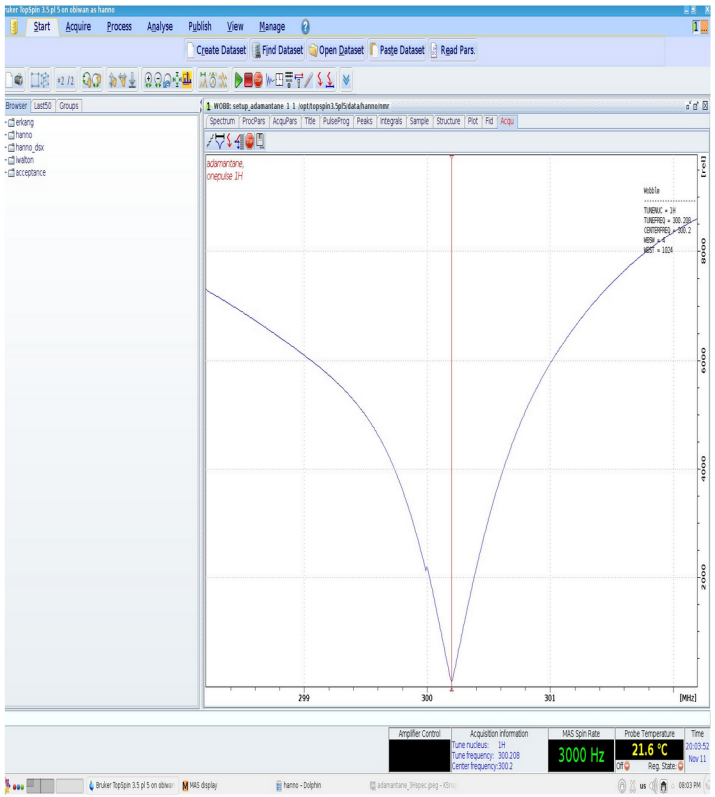
For ^{1}H
At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude;
You need to switch several times betweeh TH and MH in order to get a good result.

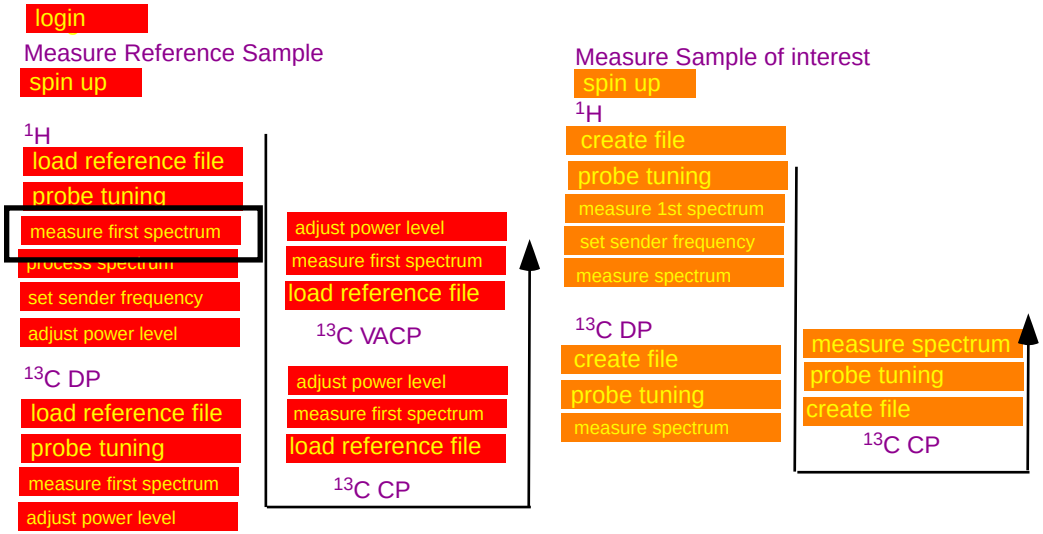
For ^{13}C
At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;
Several iterations are needed to get a good result.
Switch between 1H and 13C and find tune both channels until both channels show a good tune.

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

when finished

correct tune





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure first spectrum for ^{1}H

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

Start the experiment:

(zg / ▶)

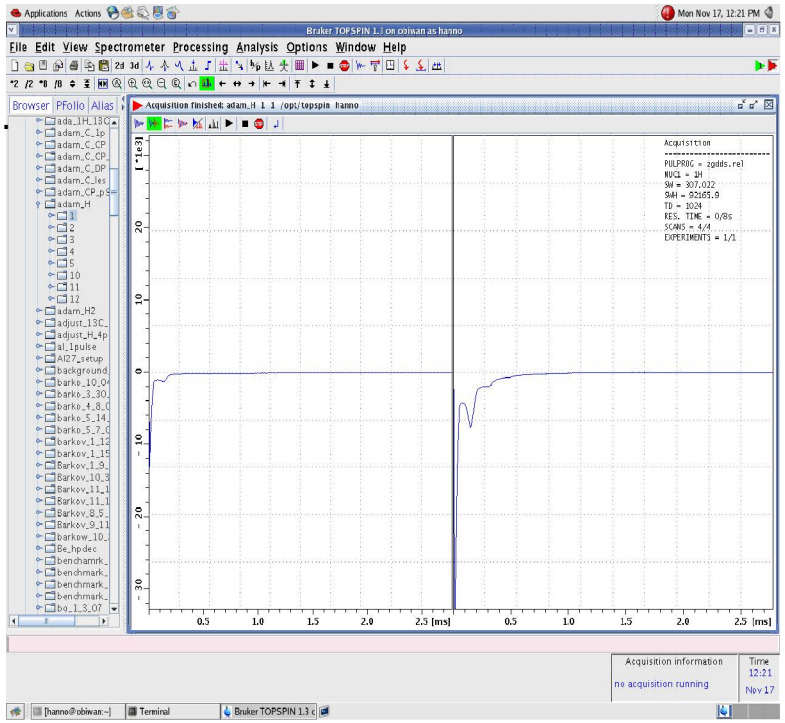
If you observe any problems: immediately (stop/ STOP).

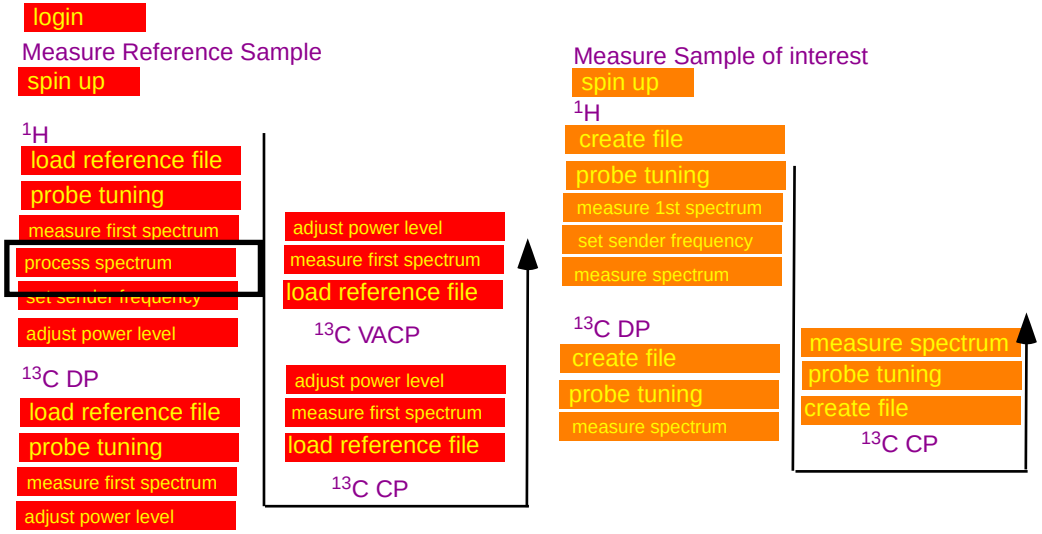
There is a command (halt/ ■), which performs a more defined “soft-stop”.

you can also click on these icons: 

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





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

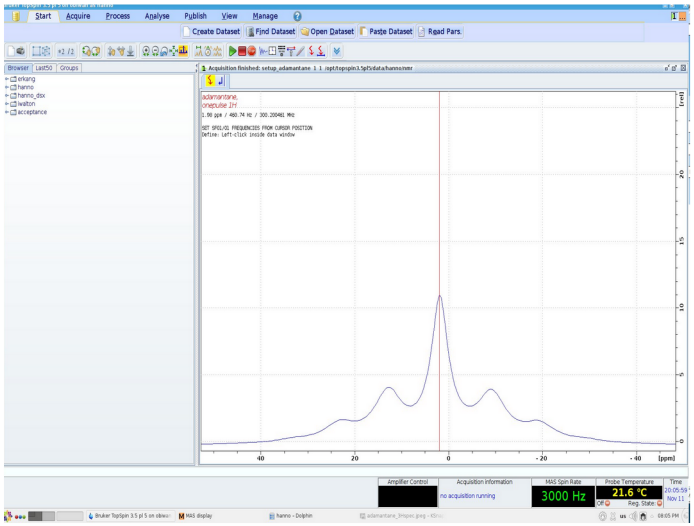
process the NMR spectrum for ^{1}H

<u>Processing a FID into a spectrum:</u>
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

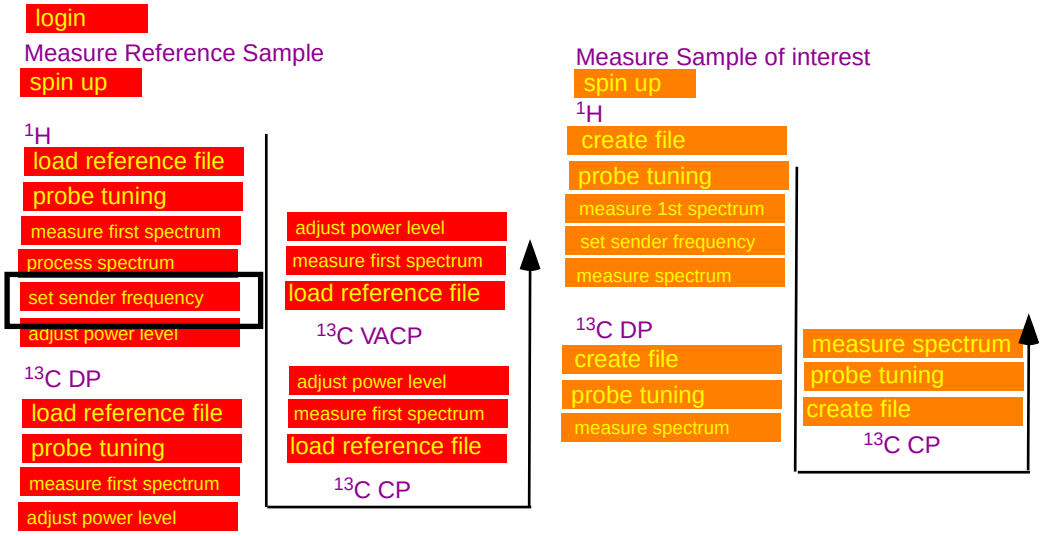
<u>If the spectrum is not phased correctly you should phase it manually:</u>
select the phasing option from the processing status bar
move the mouse on the 0-icon.
Hold the left mouse buttom and move the mouse up and down.
Further corrections can be done in a similar way with the 1-icon.

when finished.

correctly processed ^{1}H spectrum of adamantane



the phasing option can be selected from the processing status bar



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

set ^{1}H sender frequency

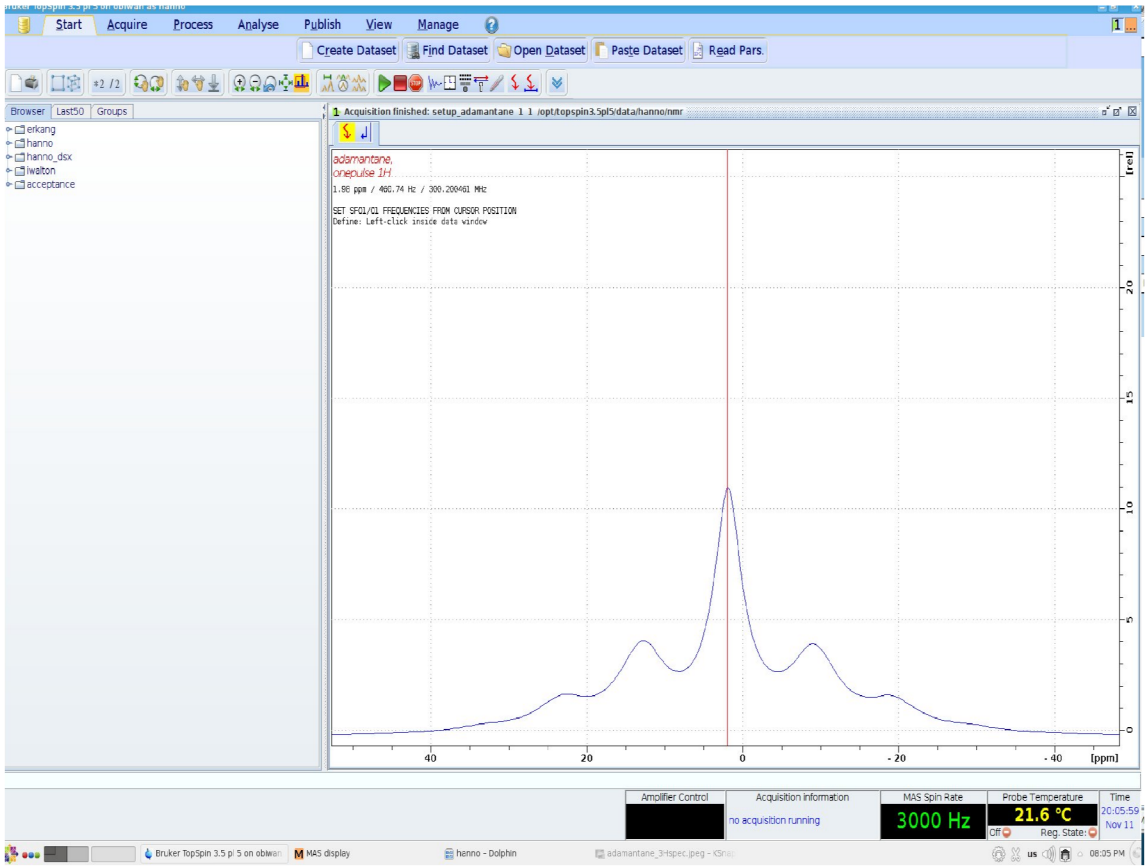
<u>Adjust the sender frequency to excite the center of your spectrum:</u>

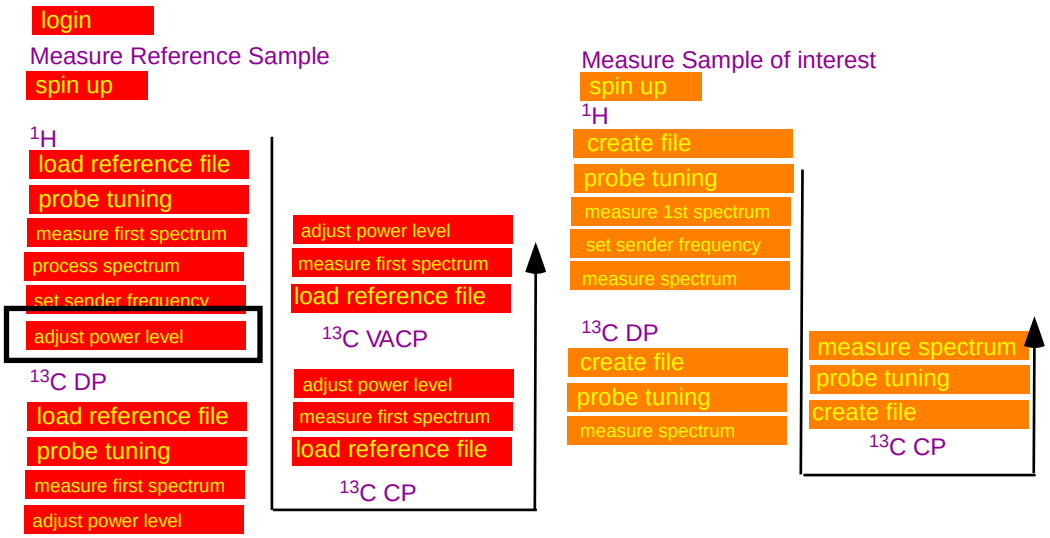

,move the cursor on the maximum of the adamantane spectrum.
Click the left mouse button to define a new sender frequency.

Note down the value for the sender frequency

SFO1 =

you may also use ctrl-c to copy and paste the value of sfo1 to the memory.
You will need this value later.





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

adjust the rf-power for ^{1}H

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.

*  FT on the fly: spectra are displayed while scanning.

* double value of p1 (type: “p1 10u”)

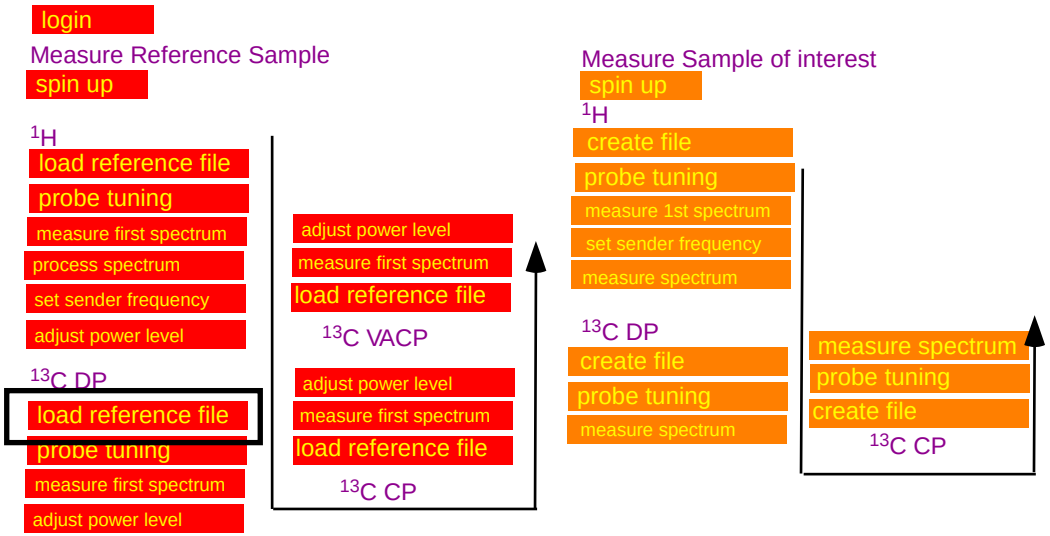
* adjust the power level plw1 (in Watts) to minimize the signal amplitude (the signal should approach a “flat” line).

* change p1 back to 5 µs (type: “p1 5u”): the signal should jump to a maximum.

*  when finished

Note down the power level for ^{1}H: plw1=

Note: you may also use the option “popt” to find the minimum power level at p1=10 µs



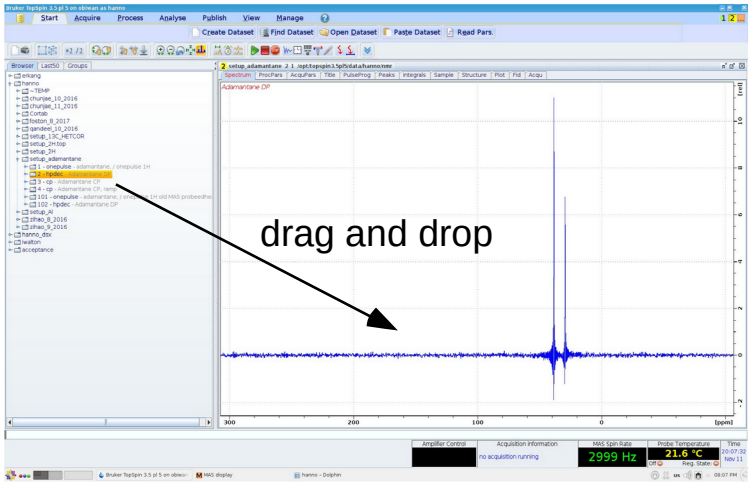
^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

load ^{13}C 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:

(1) ^{1}H

(2) ^{13}C DP ← load no. 2

(3) ^{13}C CP

(4) ^{13}C VACP

Enter frequency for ^{1}H channel:

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

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

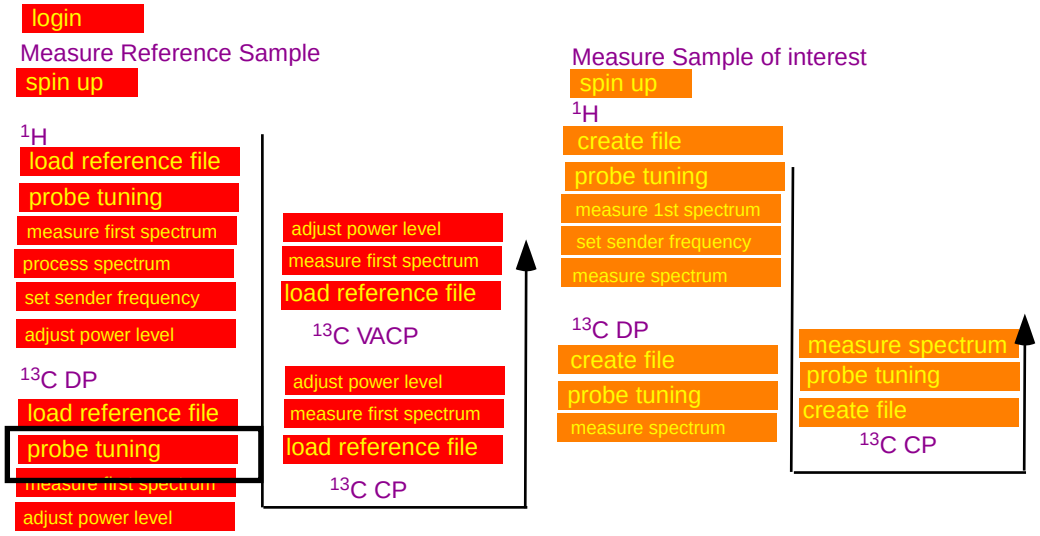
TD-SI=2k

ns=4

power levels

X-channel: plw1

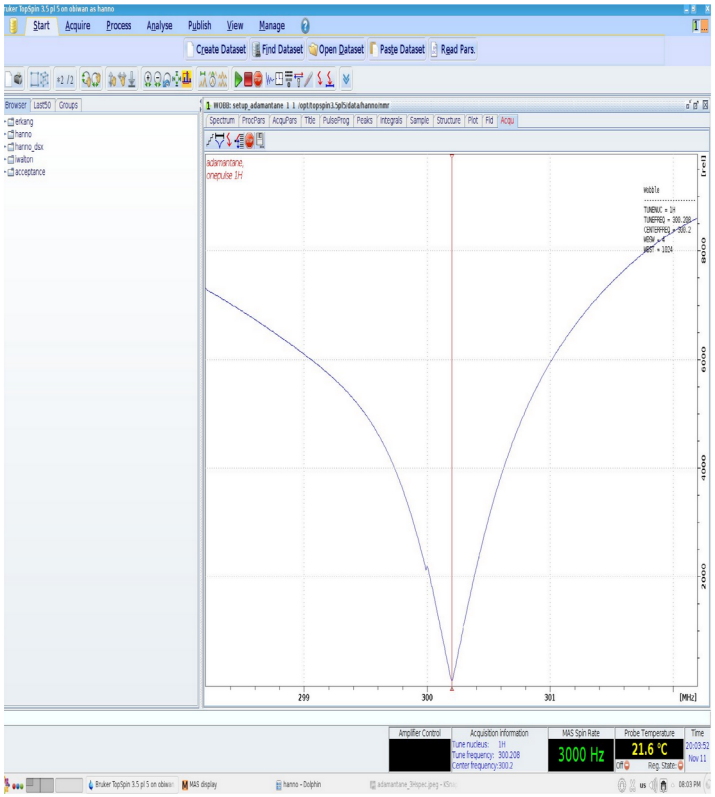
^{1}H channel: plw12



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

probe tuning



Probe tuning:

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

For ^{1}H

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.

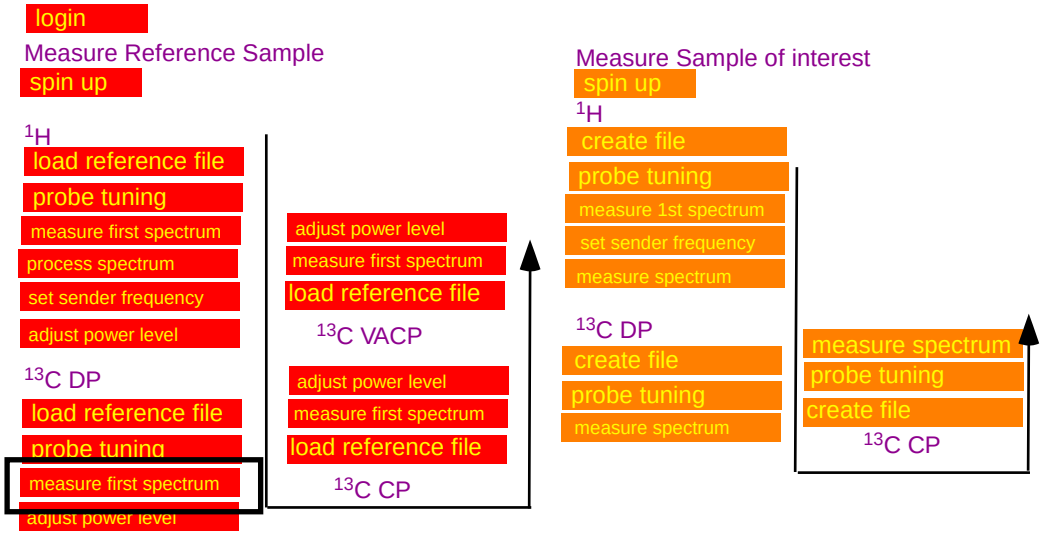
For ^{13}C

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!

when finished



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure first spectrum for ^{13}C

acquire spectrum:

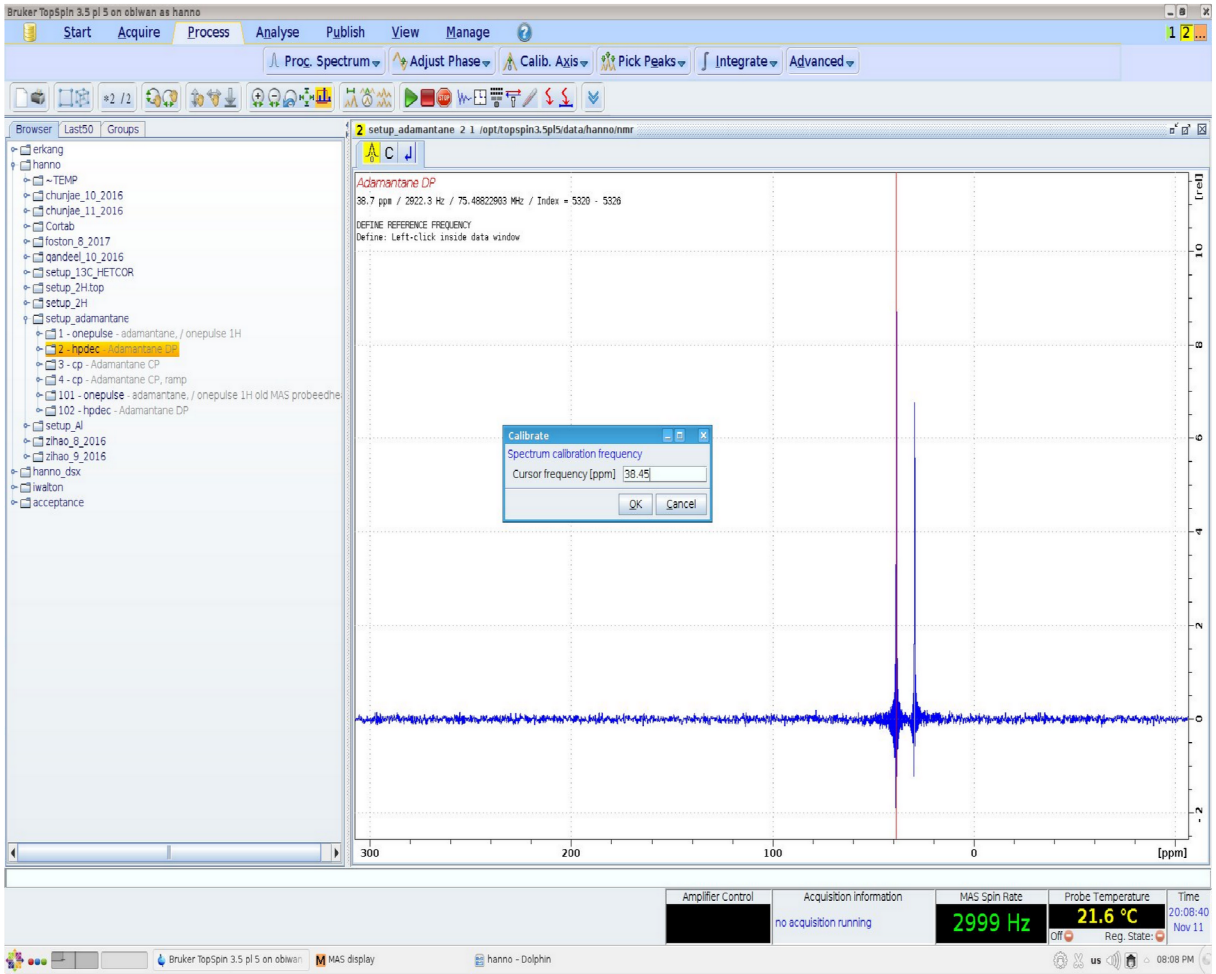
* rga, zg

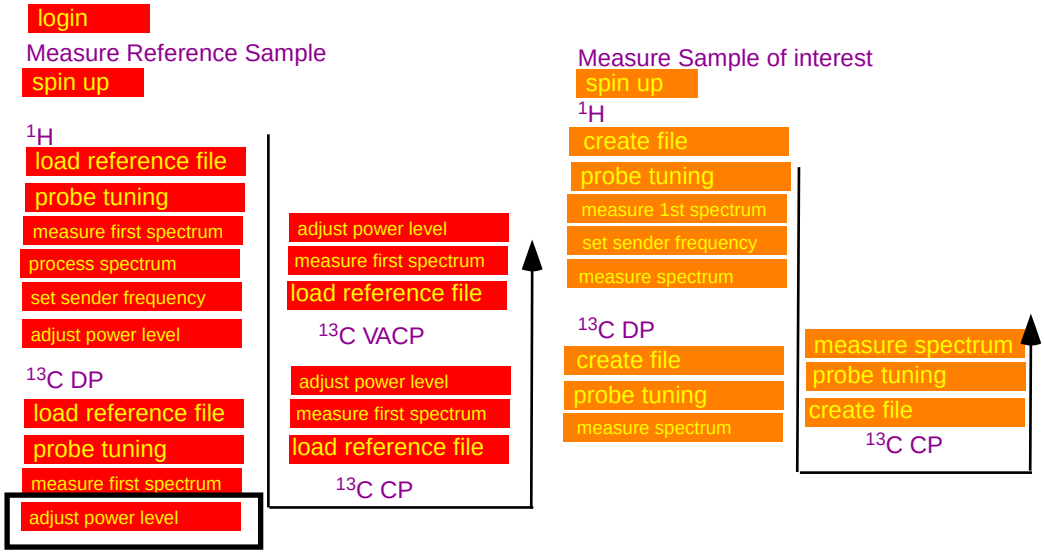
* when finished measuring process data using em, ft, apk

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).





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

adjust the rf-power for ^{13}C DP

<u>Adjustment of 90 degree pulse length:</u>

* 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.

*  FT on the fly: spectra are displayed while scanning.

* double value of p1 (type: “p1 10u”)

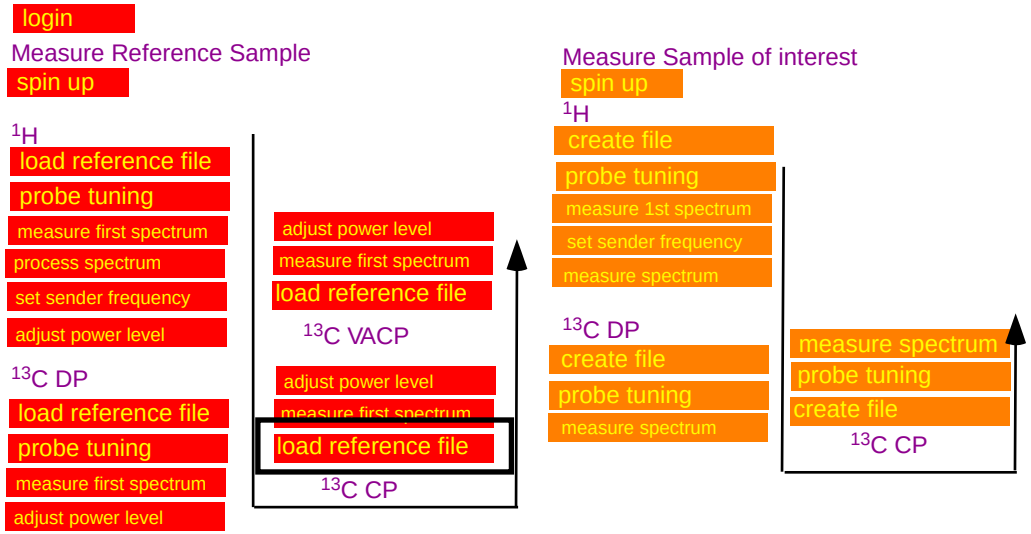
* adjust the power level plw1 to minimize the signal amplitude.

* change p1 back to 5 µs (type: “p1 5u”): the signal should jump to a maximum.

*  when finished

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

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



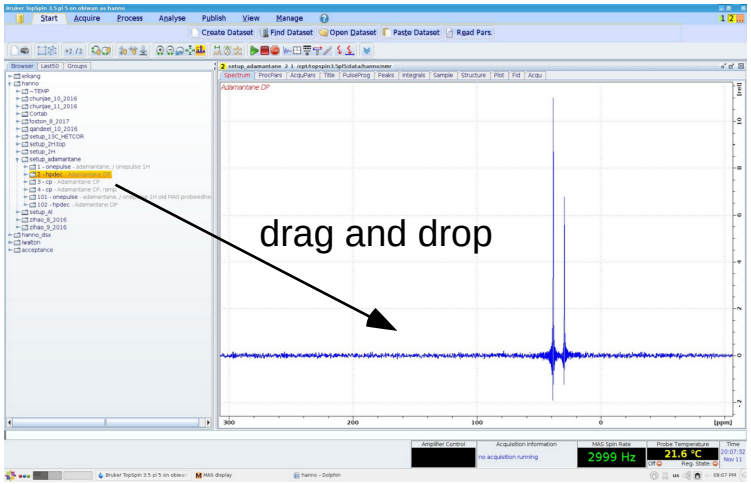
^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

load ^{13}C CP reference file

<u>select and load reference file:</u>

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



reference files are available in the directory setup_adamantane:

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

load no. 3

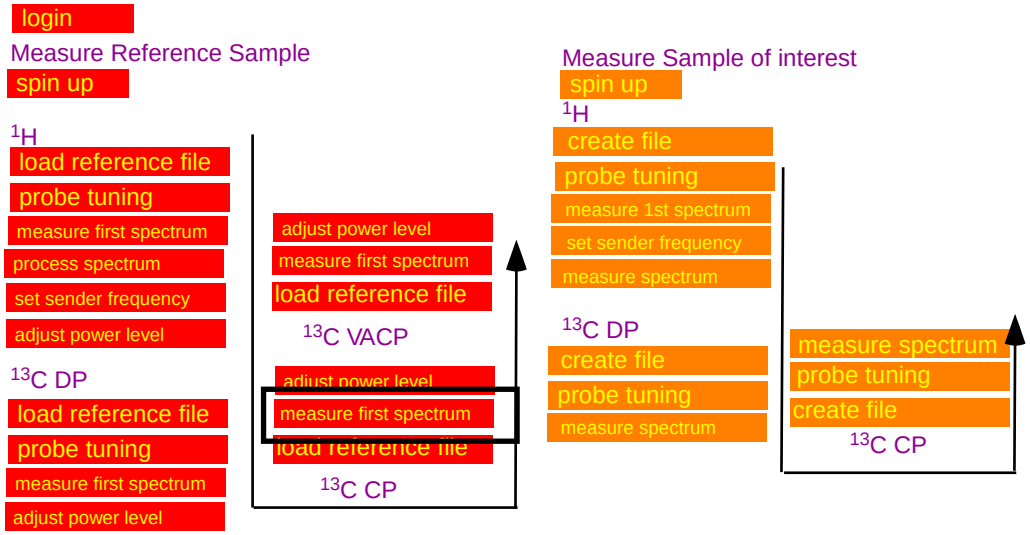
<u>Enter frequency for ^{1}H channel:</u>

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

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 ^{1}H 90 degree pulse***
sp0 =	***power level for 1H contact pulse (often same as PlW2)***
plw12=	***power level for ^{1}H decoupler***



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure first spectrum for ^{13}C CP

acquire spectrum:

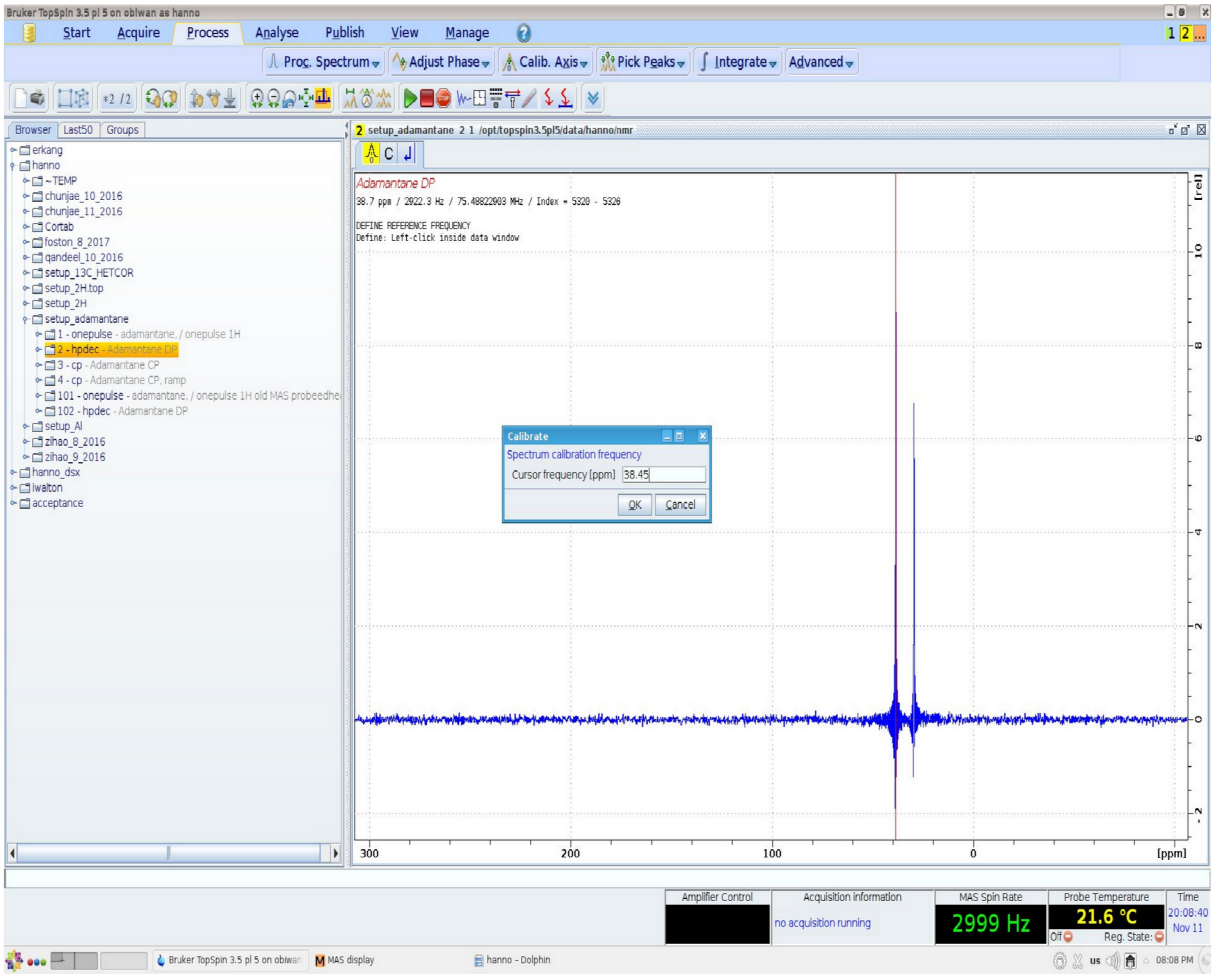
* rga, zg

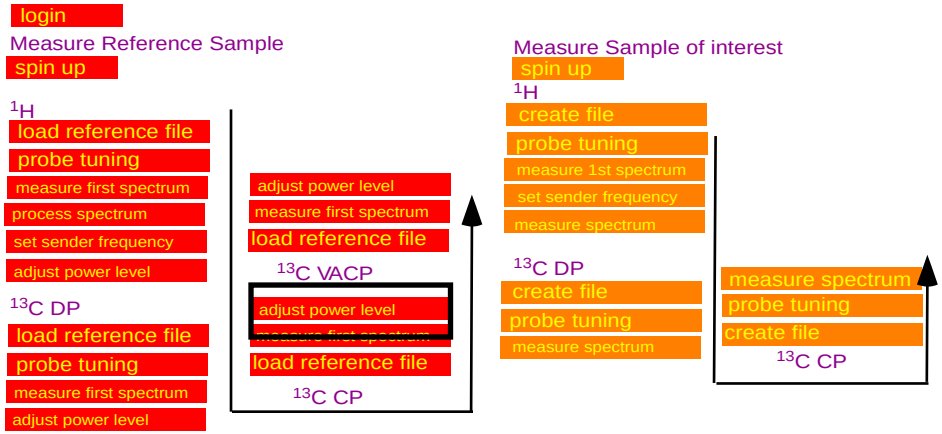
* when finished measuring process data using em, ft, apk

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).





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

adjust the rf-power for ^{13}C CP

<u>Fine-tuning of power for CP conditions:</u>
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.

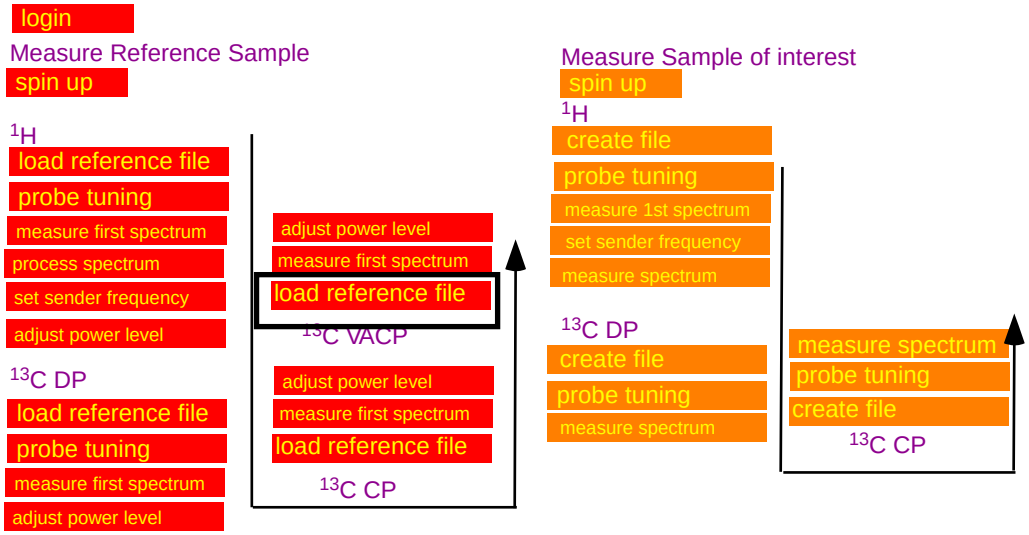
Type *popt* and enter the parameters as displayed. (note: the range of PLW1 depends on the instrument and it needs to be centered +/-10 W around the expected value for the CP-condition)
Click “start optimize” to start the experiment. You should confirm both questions with (overwrite, and start) with “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

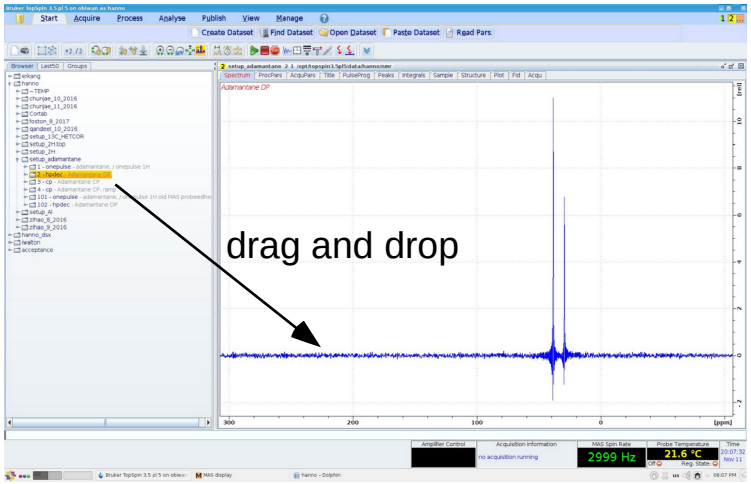
Note: The power level to achieve the maximum CP-signal depends also on the spinning frequency. Therefore 2 maxima are found for adamantane. Using the maximum found for the higher power usually give good CP-conditions for many samples spinning at MAS speeds of up to 10 kHz. However to absolutely make 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 CP-experiment.



^{13}C CP-MAS
includes ^{1}H, ^{13}C DP

load ^{13}C VACP reference file

<u>select and load reference file:</u>
Drag and Drop the reference file from your user directory into the main window



reference files are available in the directory setup_adamantane:

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

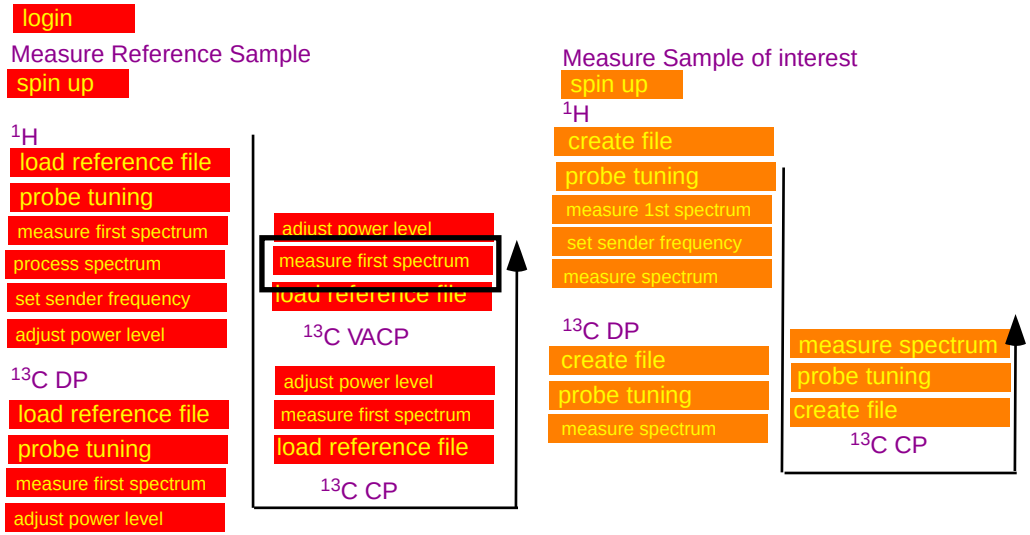
load no. 4

<u>Enter frequency for ^{1}H channel:</u>

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

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

plw1=	***power level for X (take from previous popt)***
plw2=	***power level for ^{1}H 90 degree pulse***
spw0	***power level for 1H contact pulse (to be adjusted)***
plw12=	***power level for ^{1}H decoupler***



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure first spectrum for ^{13}C VACP

<u>acquire spectrum:</u>

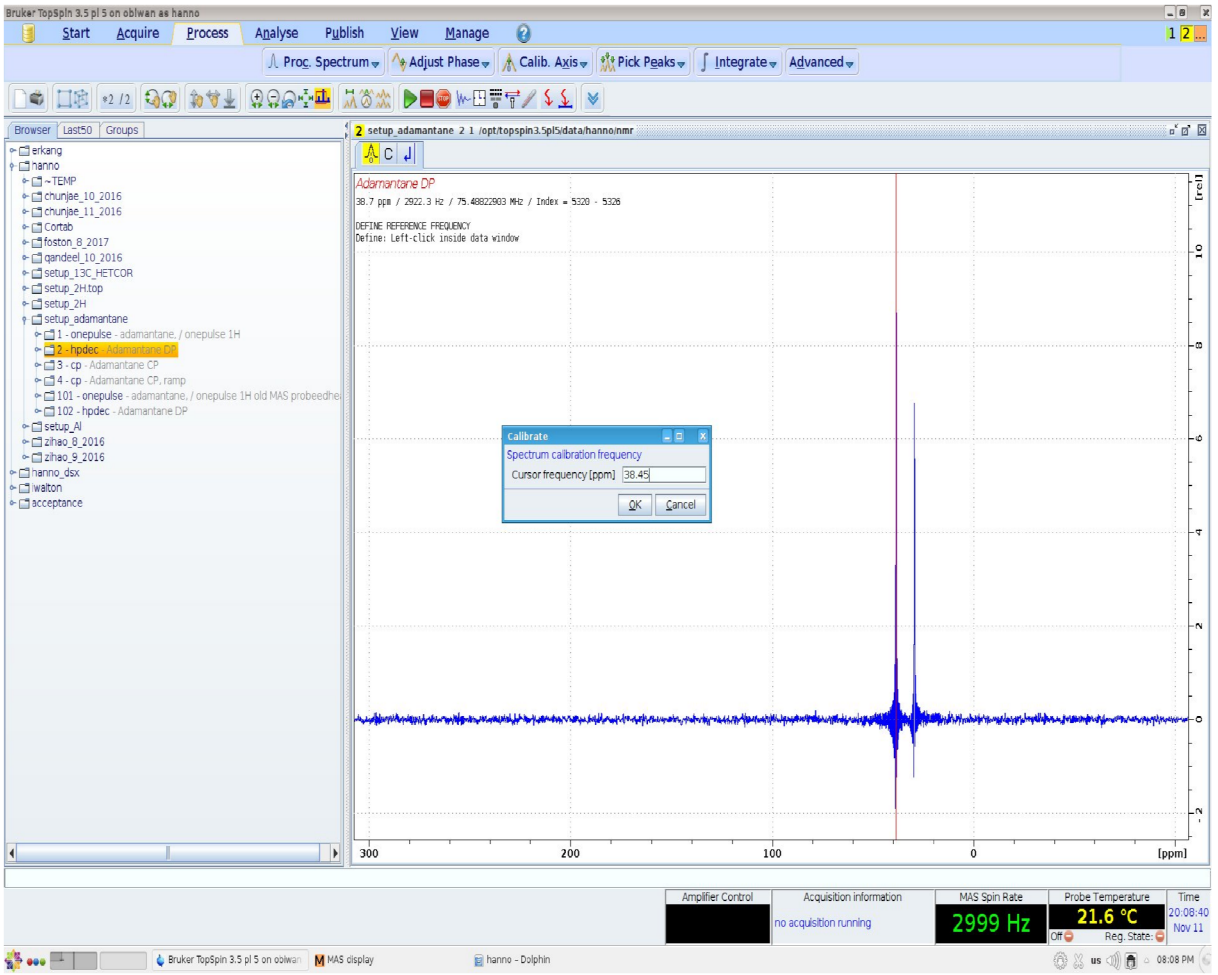
* rga, zg

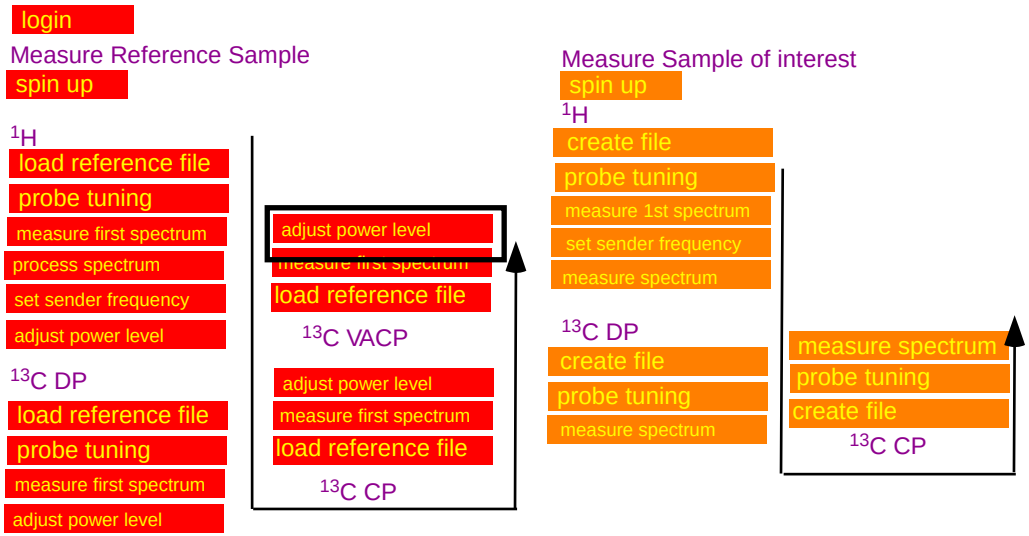
* when finished measuring process data using em, ft, apk

<u>Calibrate the ppm scale:</u>

* Calibrate/ 

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

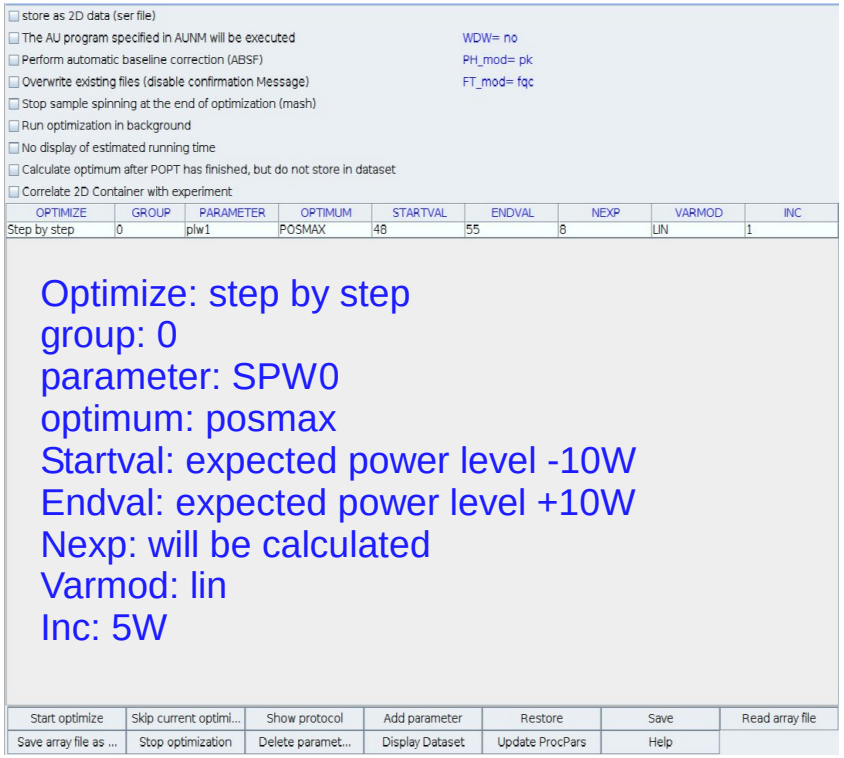




^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

adjust the ^{1}H rf-power for ^{13}C VACP



<u>Fine-tuning of power for CP conditions:</u>
The optimum conditions for VACP-experiments can be found best by using the macro “popt”.

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 SPW) 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 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.

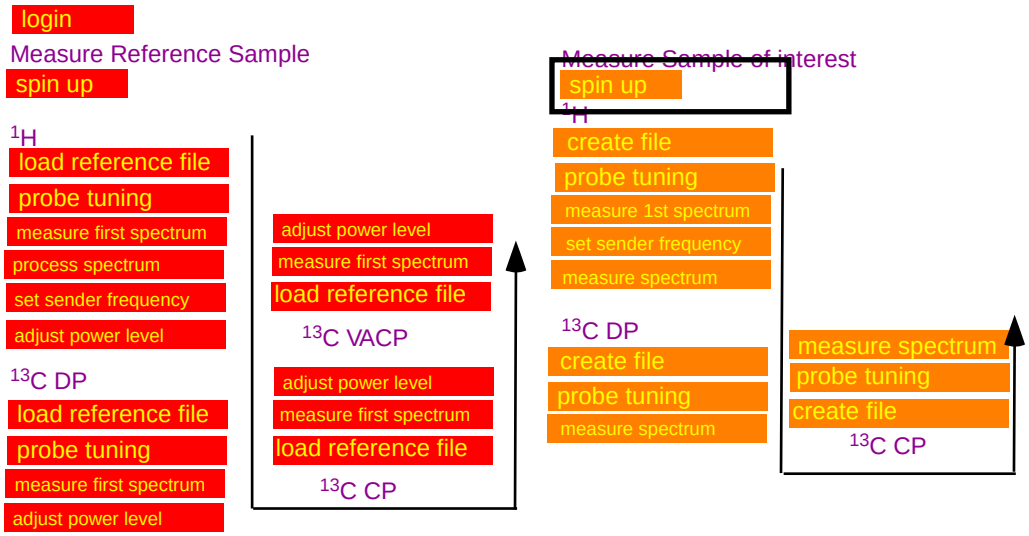
Max. intensity at SPW0**=**

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

Solid-State NMR

^{13}C CP-MAS
includes ^{1}H, ^{13}C DP

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



^{13}C CP-MAS

includes ^{1}H, ^{13}C 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),
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

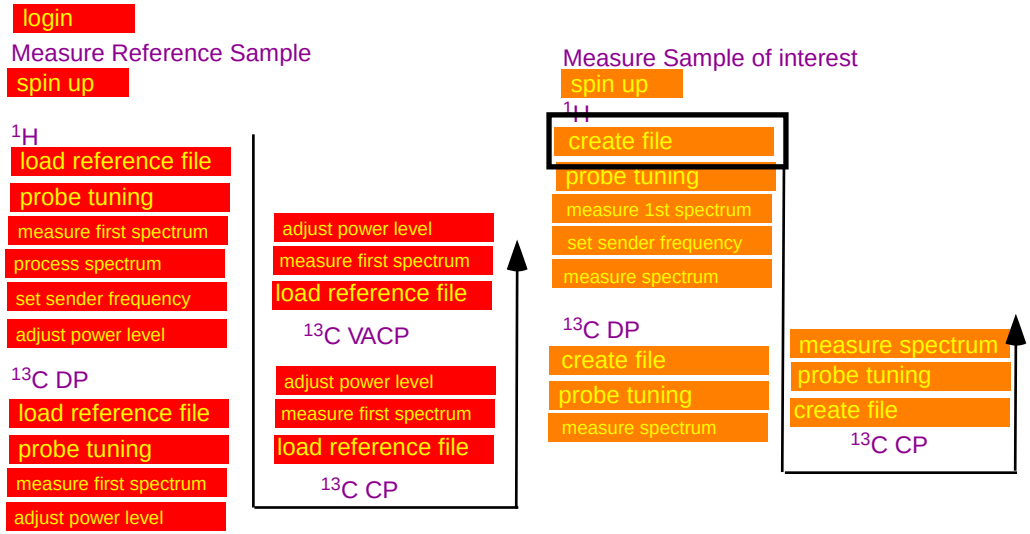
MAS III as used on the AV3-300,AV3-500 and AV3-700
steps are in principle identical to the MAS II, however the unit is now controlled via Topspin.
*type MASDISP to open a window, with control options of the MAS-unit.

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

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.

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)



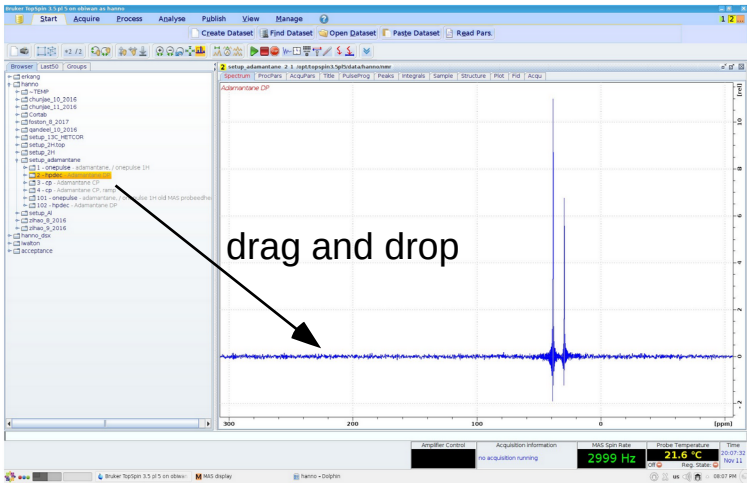
^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

create file for ^{1}H

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:

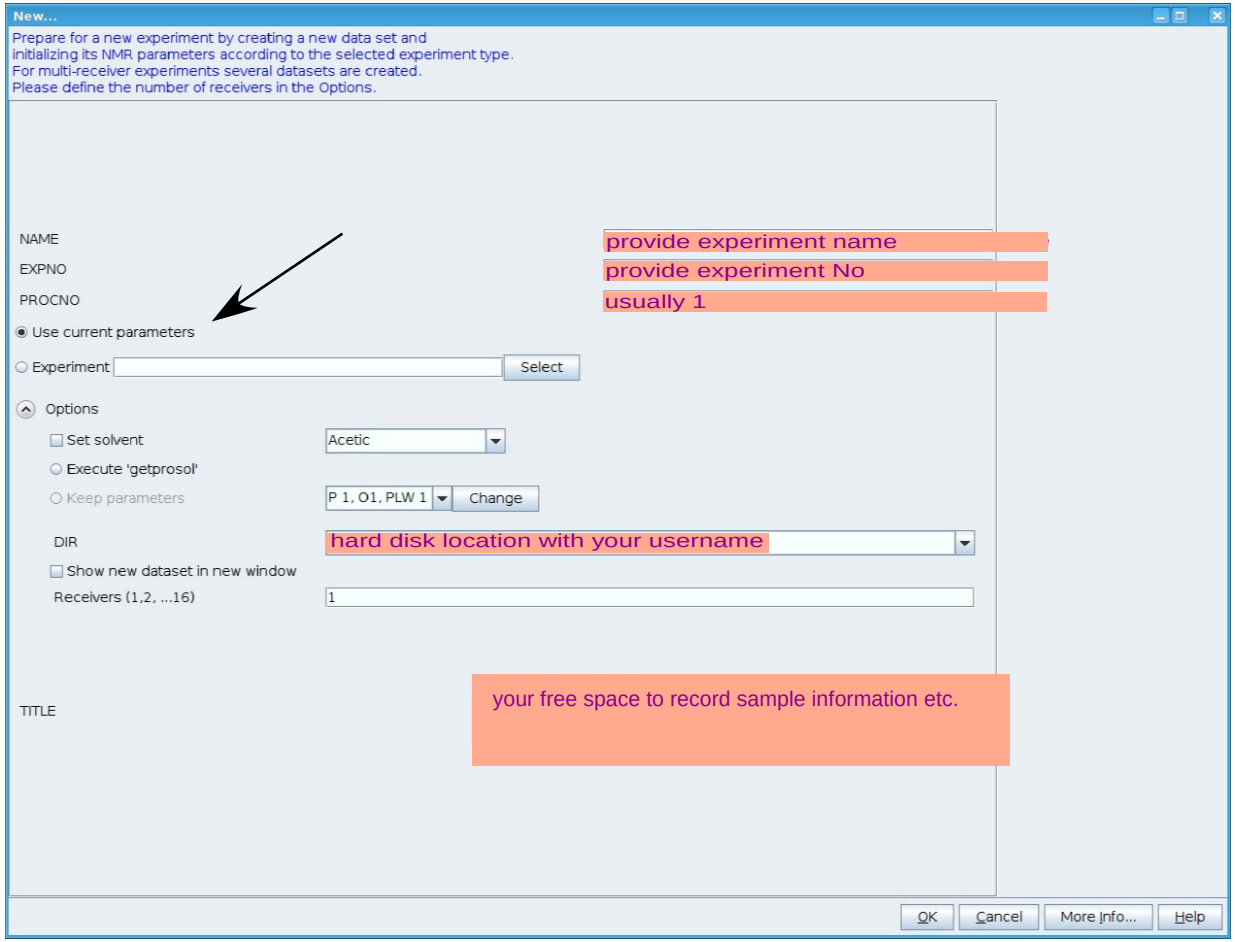
(1) ^{1}H ← load no. 1

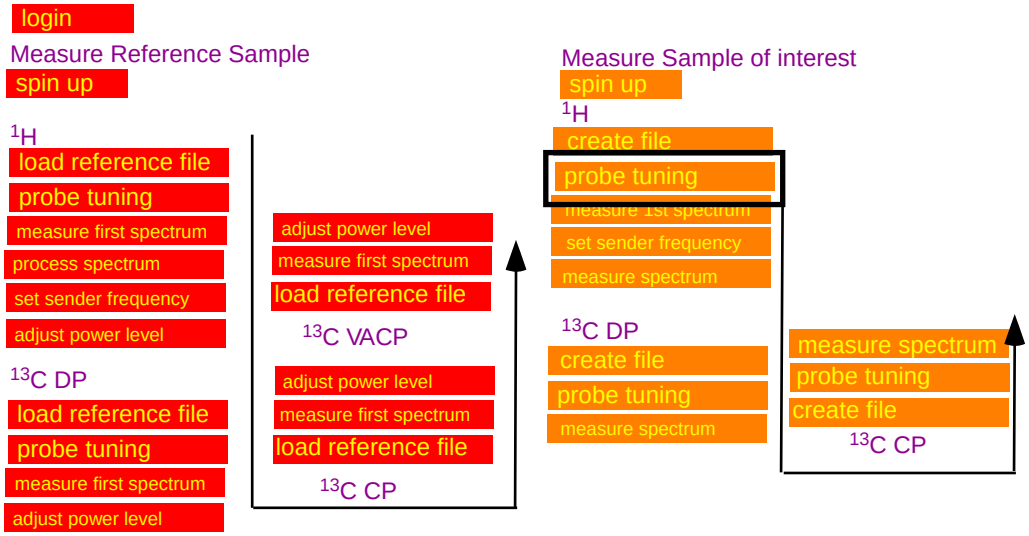
(2) ^{13}C DP

(3) ^{13}C CP

(4) ^{13}C VACP

Type “edc” to create a new file using the existing ^{1}H parameter set.





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

probe tuning

<u>Probe tuning:</u>
Wobb: the instrument will display the wobble curve for the nucleus selected for detection.

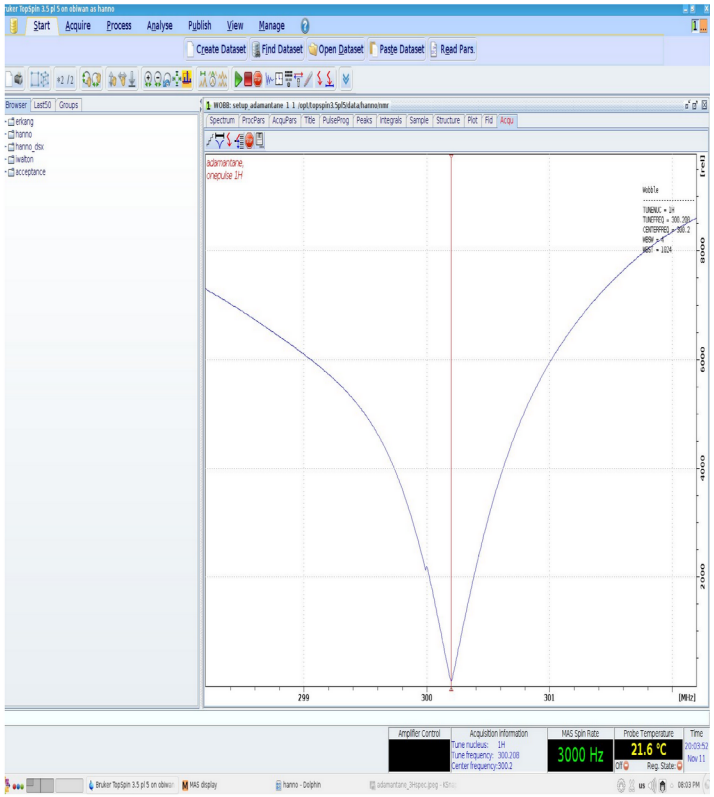
For ^{1}H
At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude;
Several iterations are needed to get a good result.

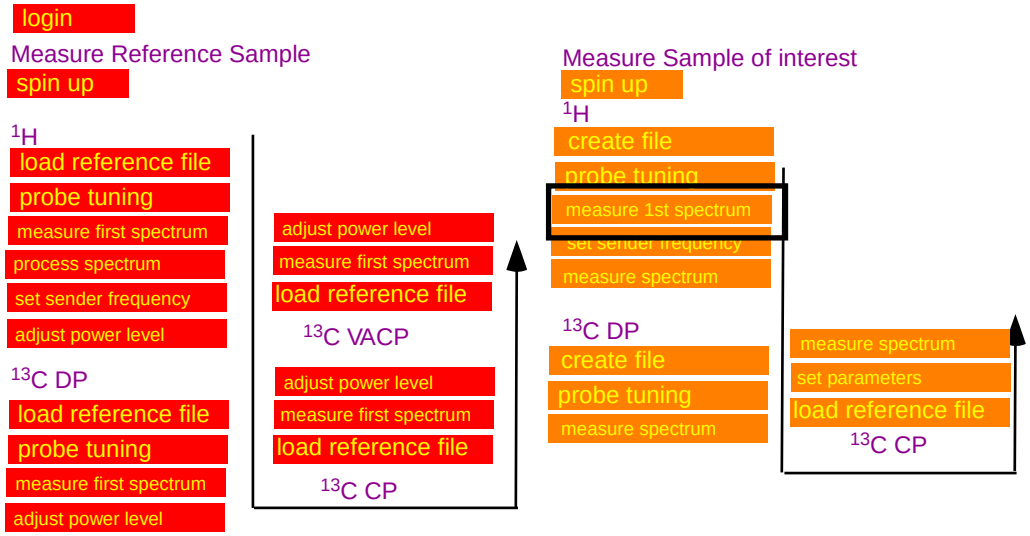
For ^{13}C
At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;
Several iterations are needed to get a good result.
Switch between 1H and 13C and find tune both channels until both channels show a good tune.

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

STOP when finished

correct tune





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure first spectrum for ^{1}H

Adjust the receiver gain:
rga for automatic adjustment of receiver gain.

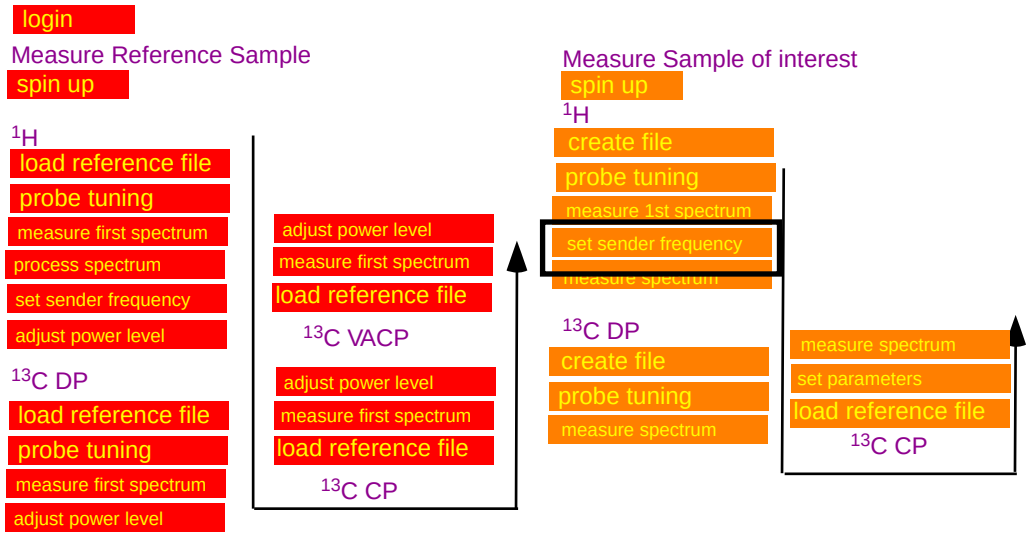
Make sure that a complete decay is observed for the FID.
If you are “clipping” data” :
double, quadruple,... the parameters SI and TD

Start the experiment:
(zg / ▶)
If you observe any problems: immediately (stop/ 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.



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

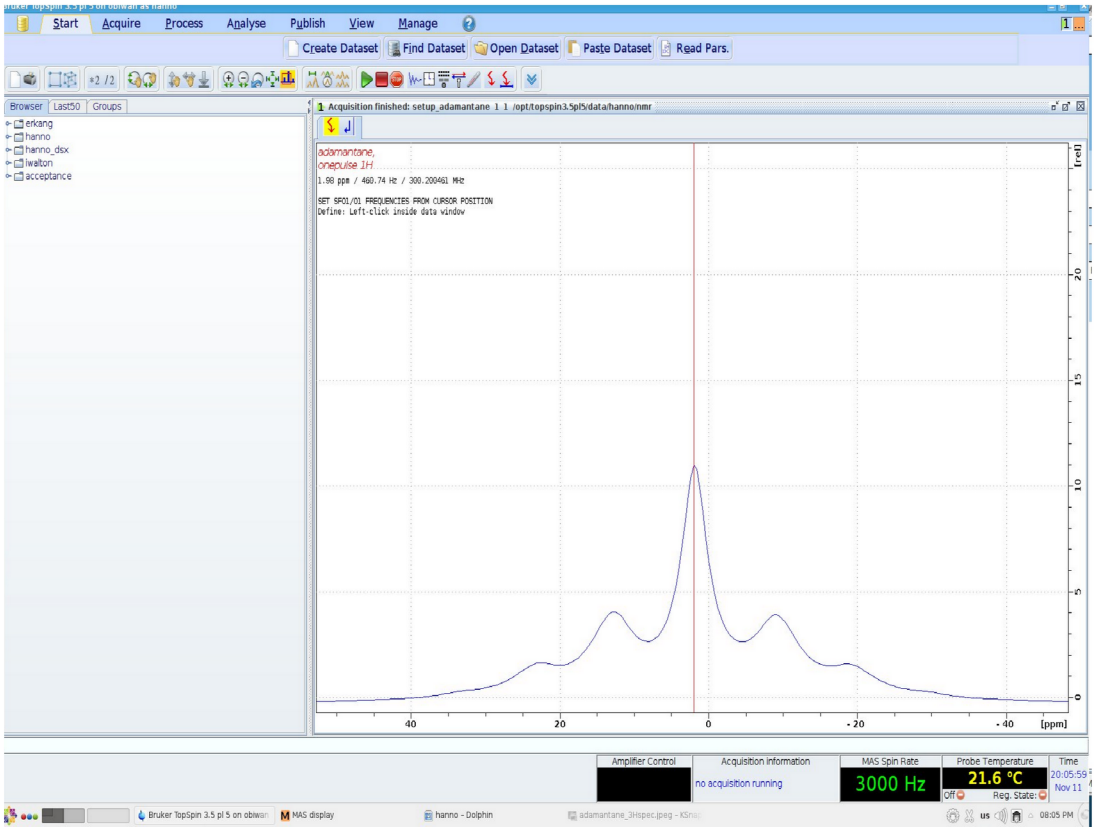
set ^{1}H sender frequency

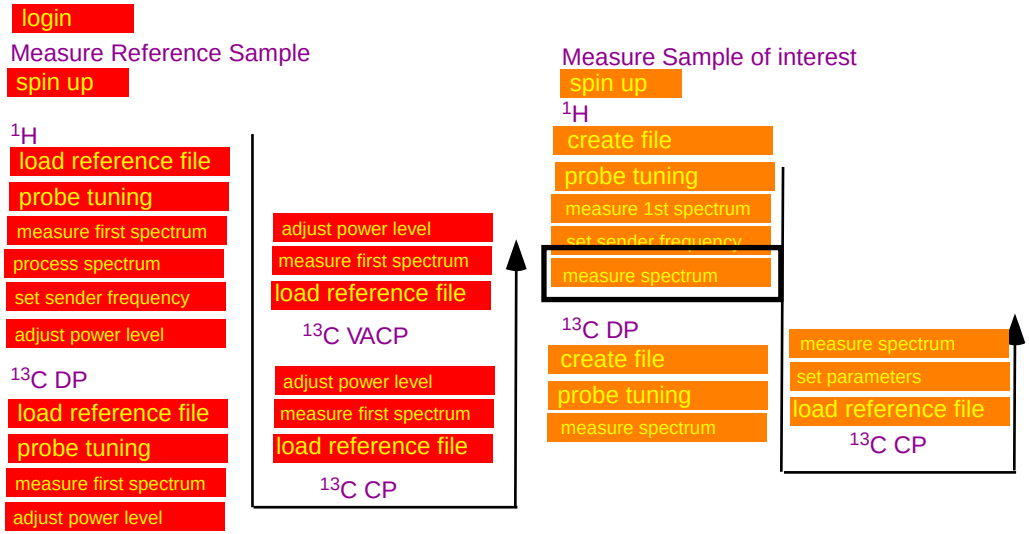
Adjust the sender frequency to excite the center of your spectrum:

,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

SFO1 =





^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

measure spectrum for ^{1}H

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

Start the experiment:

(zg / ▶)

If you observe any problems: immediately (stop/ 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.

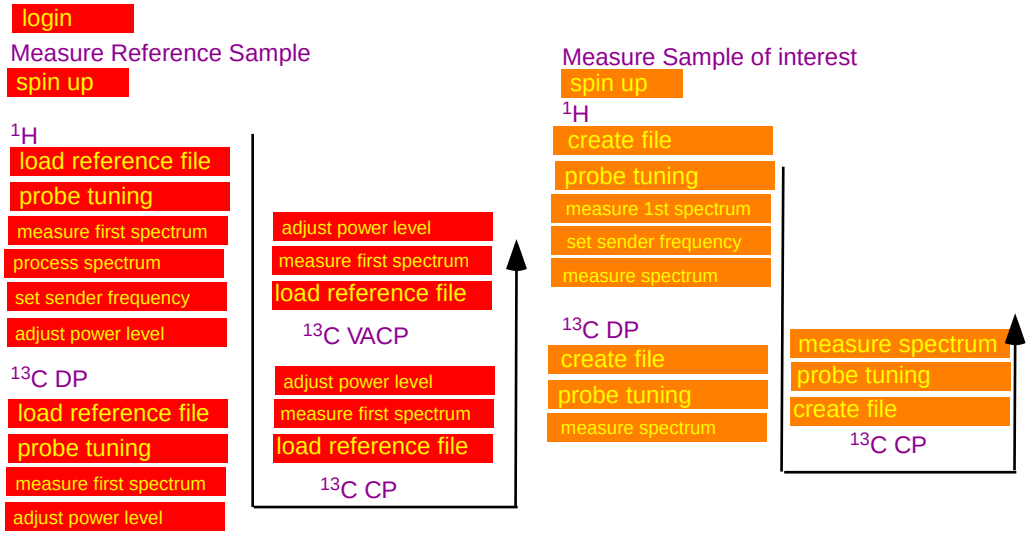
Solid-State NMR

^{13}C CP-MAS
includes ^{1}H, ^{13}C DP

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



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

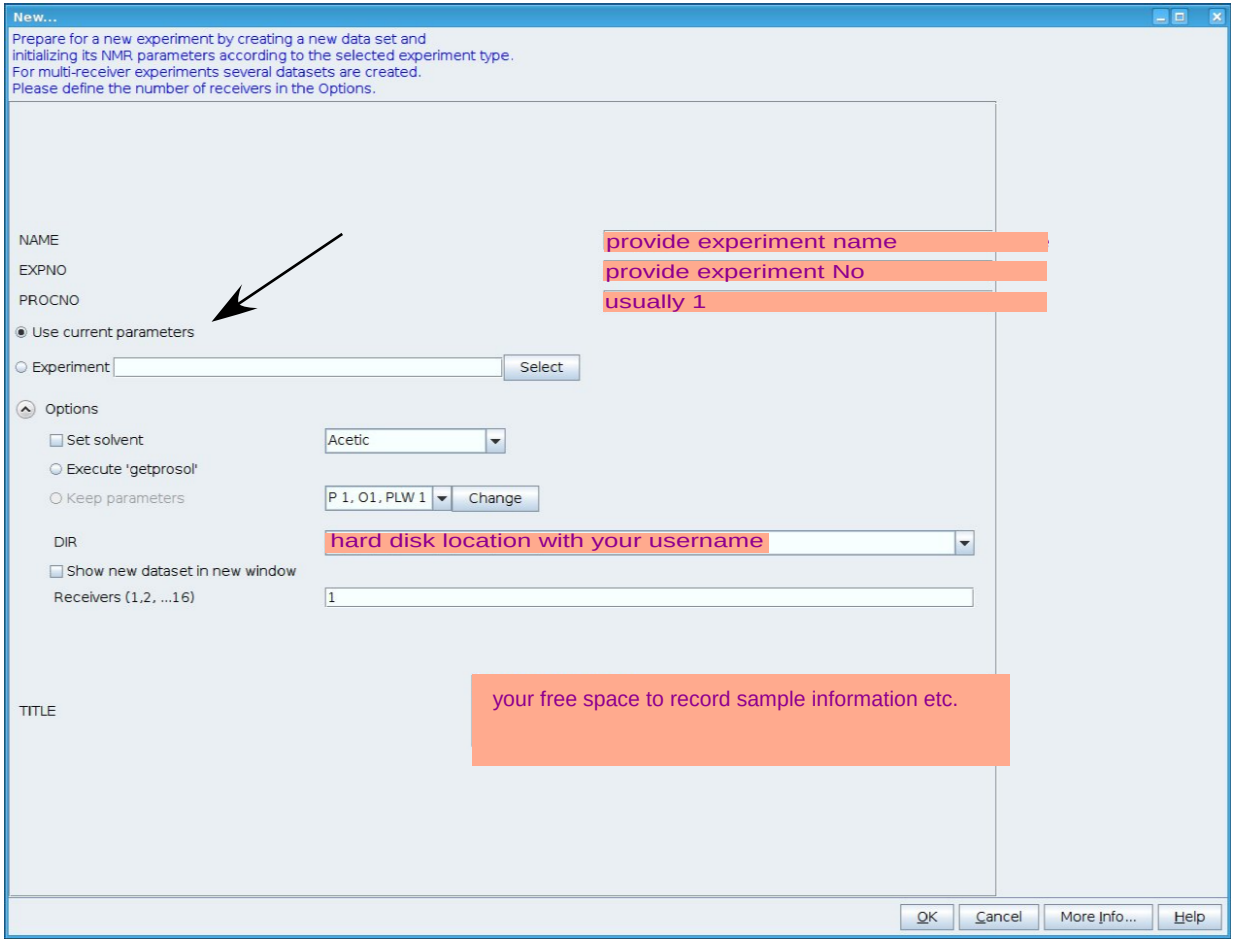
create file for ^{13}C

load your adamantane ^{13}C reference file

reference files are available in the directory setup_adamantane:

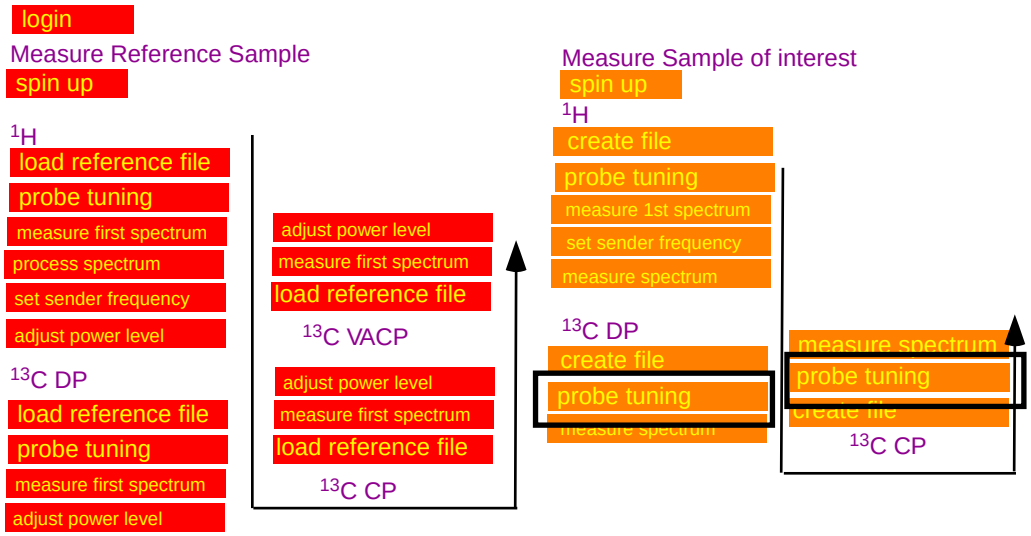
(1) ^{1}H
(2) ^{13}C DP
(3) ^{13}C CP
(4) ^{13}C VACP

select one



<u>Enter frequency for ^{1}H channel:</u>
sfo2: enter value previously determined as sfo1 for ^{1}H.

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



^{13}C CP-MAS

includes ^{1}H, ^{13}C DP

probe tuning

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

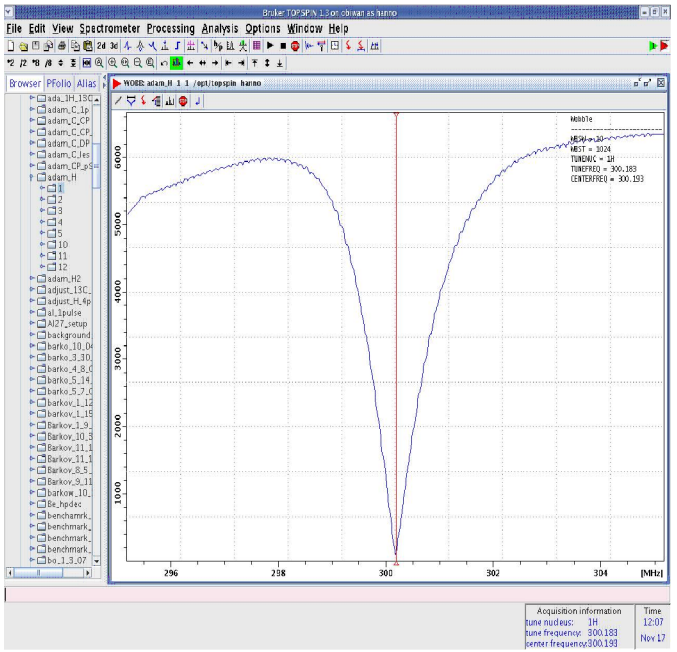
For ^{1}H
At the probe: adjust knob on probe labeled TH to move wobble curve to center of the screen, adjust knob labeled MH for maximum amplitude;
Several iterations are needed to get a good result.

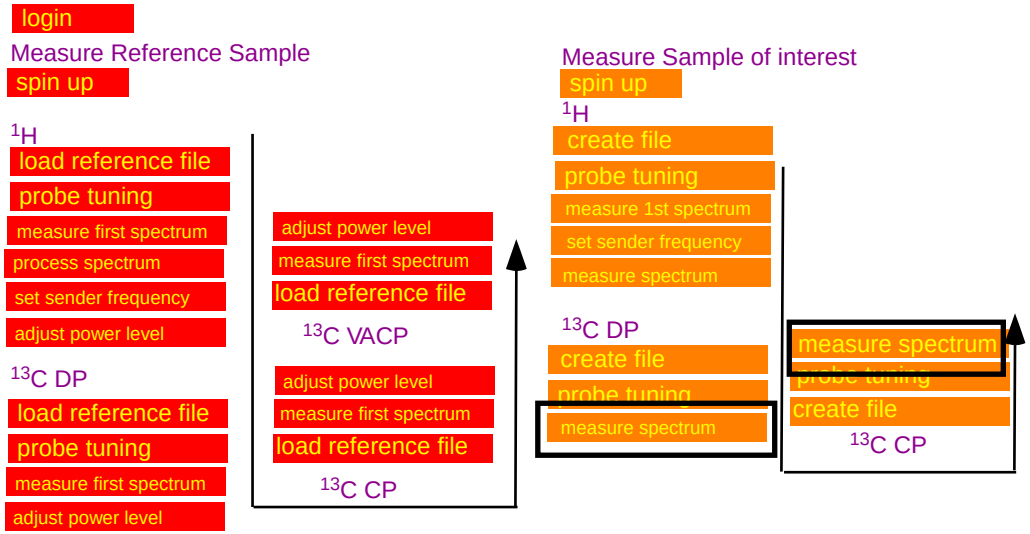
For ^{13}C
At the probe: adjust knob on probe labeled TX to move wobble curve to center of the screen, adjust knob labeled MX for maximum amplitude;
Several iterations are needed to get a good result.
Switch between 1H and 13C and find tune both channels until both channels show a good tune.

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

STOP when finished

correct tune





13C CP-MAS

includes 1H, 13C DP

measure spectrum for 13C

Adjust the receiver gain:

rga for automatic adjustment of receiver gain.

measure only a few scans (ns=32) to check your S/N.

Start the experiment:

(zg / ▶)

If you observe any problems: immediately (stop/ STOP).

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

Then measure your spectrum with more scans.

Most 13C spectra need at least ns=1024=1k.

Demanding samples need ns=10-20k.

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

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

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

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.

Solid-State NMR

^{13}C CP-MAS
includes ^{1}H, ^{13}C DP

Good luck with your Research