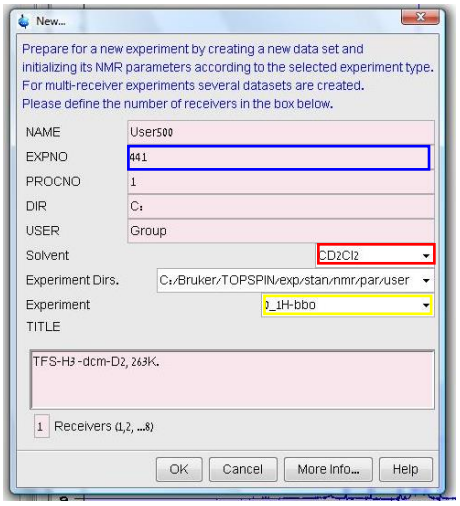
Bruker AV 200 / Topspin 1.3

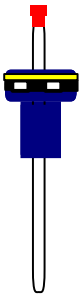
1. **Login and open the program**
   (a) Sign the logbook (name & phone number)
   (b) If Topspin is not open, open Topspin. 

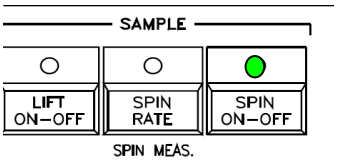


2. **Create a new file**
   (a) Click on  icon or type: new/edc

   - **EXPNO** – fill in the "exp number" only integers are allowed.
   - **Solvent** – choose your deuterated solvent
   - **Experiment** – always begin with **0_1H** choose a set of standard experiments,

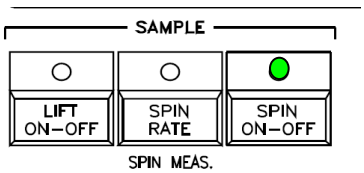
3. **Insert the sample** (sample preparation)
   (a) Make sure the spinner is intact. Place the spinner into the depth measure. Slide the tube down to the bottom (liquid level c.a. ≥ 3 fingers = 600µl).
   (b) Hold the tube above the spinner. Wipe the outside of the tube and the spinner.
   (c) BSMS keyboard: press the **"LIFT ON/OFF**" (green = on)





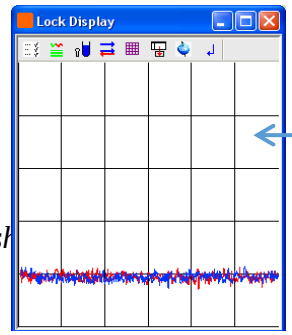
   (d) WAIT until you hear the air flow then set the spinner on the air cushion.
   (e) Press the "**LIFT ON/OFF**" button (no light = off).
   (f) Wait for the green "down" to light up.

4. **Spin** (possible only for standard tubes)
   BSMS keyboard:



   Press "**SPIN ON/OFF**" (green = on). Wait for steady green light.

5. Lock



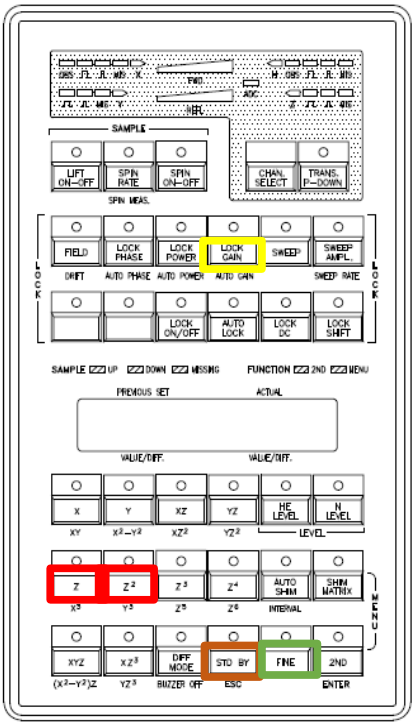
(a) Click  or type: lockdisp.
(b) Read a shim file by typing: rsh qnp.
(c) Click  or type: lock.
(d) Select your deuterated solvent -
this sets the chemical shift scale. Wait until you get "*lock finish*

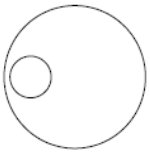
6. Shim

(a) Press "**LOCK GAIN**" (BSMS board) and use knob to place the locked signal on the second row from the top. Make sure "**FINE**" is on (lit).
(b) Maximize the signal: Press "**Z**" and move the knob. If signal is improved save the new shim current by pressing "**STND BY**", if not, return to the previous value by pressing "**Z**" again. Repeat for "Z^2". Repeat again for "**Z**", "Z^2",… until no further improvement is seen. (If you don't spin your sample shim also "**X**" and "**Y**").

- if lock signal too high, reduce "**LOCK GAIN**"
- pressing a lit button will undo changes
- pressing "**STD BY**" button will save changes

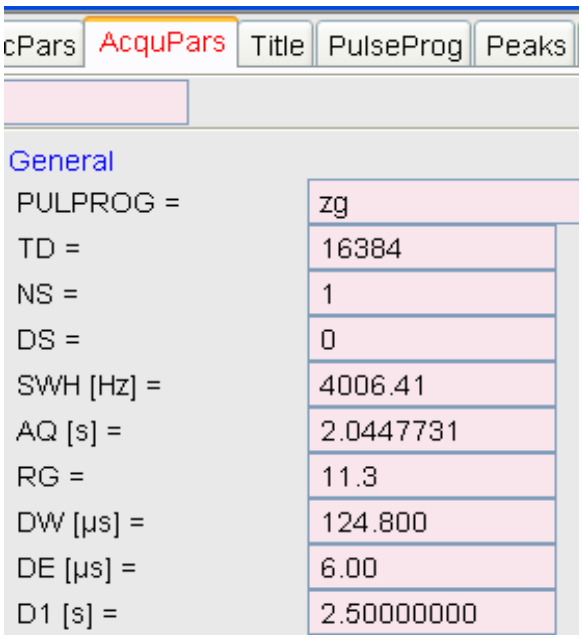
(c) Press "**STD BY**" when finished.





Control knob

7. Parameter setup



(a) Type ased: or click AcquPars tab.
(b) Type rga: wait till you see message "*rga finished*".

- large **RG** – diluted sample (more scans are required for good S/N)
- small **RG** – concentrated sample
- **RG** =1 too concentrated sample, should be diluted in order to avoid clipping

(c) Click ▶ or type: zg for preliminary acquisition.
(d) Examine your FID **(time domain)**and your spectrum and choose the right parameters. avoid clipping of the FID
Adjust **AQ** (acquisition time) to make sure that the signal vanishes at ~ 2/3 of the screen (x-axsis). Check that no clipping occurs at y-axis (reduce **RG** if required).

(e) Type: proc to process you data (FT, phase correction and baseline correction).
Spectrum (frequency domain):

- Adjust "sw" (spectral width in ppm) and "o1p" (center of the spectrum in ppm=resonance frequency) if required.
- Zoom in and check your shimming: if you observe unusual features, splittings or linewidths for ALL your peaks - do more shimming. You can use online shimming by "**gs**".
- Check again that your **AQ** is sufficient (it might have changed when you adjusted sw).
- Adjust **SI** (ProcPars tab) >=2***TD** to get optimal zero filling.

Set other parameters:

- set **NS** (multiple of 8 for a full phase cycle) and **DS** (=2)
- set **D1** (relaxation delay). Unless you have checked your T_1s (relaxation times) and set AQ+D1=5*T_1(slowest) the spectrum is NOT quantitative! And the integration is meaningless.

(f) Check the experimental time: click ⏲ or type: expt

8. Acquire a FID

(a) Type zg
Options during FID acquisition:

- tr ▶ saves the data and continues acquiring
- h ■ HALTs the acquisition and SAVEs your FID. *The FID is automatically saved once ns is reached*
- stop terminates the experiment – data will be lost
- kill terminates the experiment – data will be lost

9. Processing

Type proc (preforms Fourier Transform (ft), phase correction (apk), baseline correction (abs)).

10. Remove your sample

On the BSMS keyboard:

(a) TURN OFF: " **SPIN ON/OFF"** and "**LOCK ON/OFF "** (no light = off)
(b) press "**LIFT ON/OFF**" (green = on)
(c) gently remove your sample from the magnet – lift it up to clear the magnet
(d) press "**LIFT ON/OFF**" (no light = off)

11. Save your data onto another computer

(a) Open the nmr200 file icon on the desktop (double-click).
(b) Click "nmrlab4" network shortcut.
(c) Drag and drop your files from nmr200 to nmrlab4 and preserve the hierarchy:
<dir>/data/<user>/nmr/<name>/expno

DATA MAY BE **DELETED WITHOUT WARNING** at any time

Back up your data from **nmrlab4** to your lab computer. Nmrlab4 is not safe and not backed up. Your data is you responsibility!

12. Open your files on another computer

To open Topspin files on the remote computer:

(a) Open the program Topspin  .
(b) Navigate to your data using the Browser.
(c) Drag you data file into the display window.

PLEASE, report problems and bugs to Ira or Shifi (3748)

13. More Shimming (on lineshape)

(a) Shorten AQ and set D1=0sec; set NS=1 DS=0
(b) Run one scan: zg and process proc.
(c) Type: gs, choose spectrum view and zoom on solvent (or other narrow) peak.
(d) BSMS board: Shim slowly (let equilibrate) on **Z**, **Z²** (**X**, **Y** if not spinning) aiming to obtain narrower and higher peak.
(e) Press "STD BY" when finished.

14. More Processing

(a) ft fourier transform automatically applies the bc_mod and me_mod specifications
(b) window functions can optionally be applied to the FID
sensitivity-enhancement *(lb > 0)* ef
resolution-enhancement (lb < 0, gb > 0) gf
right-click to alter the display properties
(c) Automatic phase correction apk

Manual phase correction: **.**ph 

- "0" applies zero-order phase correction
- "1" applies first-order phase correction
- set the cursor with a right-mouse click ("set pivot point" left-click) save and exit: 

(d) chemical shift calibration .cal 
place the cursor at a known position (*e.g.* $CDCl_3$ = 7.26 ppm)
click the left-mouse button to define its position
(e) abs (automatic baseline correction and automatic integration)

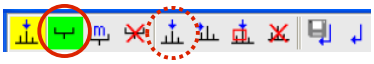
Manual baseline correction: **.**basl 

- highlight the triangle (green) to view differences save and exit.
- For more baseline correction options, type: bas

(f) ppf (automatic peak picking)

to toggle display units to relative: click  or type: .y
to change parameters for peak picking: pp
or type the values in the command line for mi, maxi, cy

Manual/interactive peak picking: **.**pp 



- d*efine the peak picking range (green box)*
- draw a box around the peaks to pick
- the symbol with the "m" allows you to change the box dimensions
- the peak picking icon allows you to select a single peak with the mouse
- save and exit

(g) abs (automatic integration and baseline correction)
for automatic integration without baseline correction, absg 0 then abs

Manual integration: **.**int 

- to delete all: “select all” with  and delete 
- to define new integrals 
- right-click on an integral to select or calibrate:
- save and exit.