

Simple Operation Guide for HFNMR Users

by Dr. Chi-Fon Chang for small molecules (2022.03.10 updated)

PART I: Preparation (Software & Hardware)

Step 1: Starting topspin

1. Login into computer
2. Double click on "topspin" icon (Fig.1)
(Topspin window will pop out)

Step 2: Temperature control

1. Type "edte" <enter>
2. The temperature control sub-window will pop out,
set up your target temperature (Fig. 2)

Step 3: Loading sample (no autosampler)

1. Put NMR tube into Spinner and adjust sample position using Sample Position Gauge (Fig.3)
2. Click "Lift" button on [BSMS] (Fig.4) , you should hear the air flow
3. Load your sample (make sure sample can "float" on the top of magnet before releasing the sample)
4. Click "Lift" button on [BSMS] again, the sample should go down to the probe

Step 3': Loading sample (with autosampler)

1. Put NMR tube into Spinner and adjust sample position using Sample Position Gauge (Fig.3)
2. Load your sample(s) to autosampler, and note your sample position(s)
3. Type "sx #" (# is your sample position number) to load sample into NMR probe

Note:

- Depends on hardware, it could be topspin2.x , topspin3.x or Topspin4.x

- Check temperature before loading sample is highly recommended.

- For regular 5mm NMR tube, fill in 450-500ul sample solution is recommended.

- For NEO 600 SampleCase, tube above spinner must less than 9.5cm

- In case autosampler couldn't load sample correctly, try to switch to other position. If error show up, ask facility members to help.

Step 4: Lock & Wobble (with ATM*)

1. Start a new data set :
type "**edc**" <enter> , or "**new**" <enter> (Fig.5)
 2. Select Standard Experiment (ex: **1GRC_1D_1H**) ;
Or, Type "**rpar 1GRC_1D_1H***"
 3. Type "**getprosol**" <enter> to read in standard pulses
 4. Type "**lock**" <enter> to choose your D-solvent
 5. Type "**atma**"<enter> to auto adjust 1H frequency or "**atmm**"<enter> to manually tune/match
- * ATM is the auto Tune and Match accessory installed on probe. For those probes without ATM accessory (ex: Dual probe on AV400 & TXI/QXI on AV600_CHEM) , if needed, type "**wobb**" to do wobble manually.

Step 5: Shimming

1. (**optional**) Type "**rsh** " (not necessary, but you can select previously saved shim file for specific probe/solvent)
2. Type "**topshim**" <enter>
or "**topshim tunea**" <enter> (recommended)
or "**topshim convcomp**"<enter> (for NEO 600 only)
3. Manually adjust shim if necessary

Note:

- Use 1H experiment to optimize hardware setting
- Why we should getprosol ?
- What is lock doing?
- When and why should we do wobble(atma) ?
- What is shimming?
- More options are available if typing "**topshim gui**"

By now, Lock & Shim has been optimized. **Unless you change temperature or sample, it's not necessary to lock/shim again during later experiment set up and data collection!**

However, **wobble (atma) might be needed depends on the experiments you set up.**

PART II: Experiment Set up & Data Collection

● Steps for Setting up 1D Experiment:

1. Type "edc" or "new" <enter> to set up new EXPNO
2. Select Standard Experiment (1GRC*);
Or, Type "rpar 1GRC* " <enter>
3. Type "getprosol" <enter> to read in calibrated pulses information from standard samples
4. Type "atma" <enter> or "atmm" <enter> to optimized nuclei frequencies (if no ATM probe, check it manually by typing "wobb")
5. (optional) For 1H experiment, type "pulsecal " <enter> to optimized 90 degree pulse for your own sample (recommend for sample in H2O/D2O, or 2D experiments)
6. Type "ns" <enter> to fill in scan number you like
7. (optional) Type "eda" or "ased" <enter> to fill in other parameters if needed (ex: O1, SW, TD,...)
8. Type "rga" <enter> to auto adjust receiver gain
9. Type "zg " <enter> to collect 1D FID
10. Type " efp" <enter> to do Fourier Transform
11. Type "apk" <enter> to auto-phasing the spectrum
12. Type "abs n" <enter> to adjust baseline

(Spectrum is ready for further processing/analysis: calibration, peak picking, integration)

● Extra notes:

- If using optimized pulses from "pulsecal" for other experiments (Fig.6)
Type "getprosol Δ 1H Δ <pulse in us > Δ <power level in dB or dBW>"
(ex: getprosol Δ 1H Δ 10.2 Δ -3.5) instead of "getprosol"

Note:

- Select most commonly used expts from our standard parameter sets
- May skip if already done for required nuclei
- Write down the corresponding power level (dB/dBW) and pulselength (us) for later usages
- ased only display parameter needed for specific experiment
- Can be combined as " efp, apk, abs n"

● Steps for Setting up 2D Experiment:

1. Type "edc" or "new" <enter> to set up new EXPNO
2. Select Standard Experiment (1GRC*);
Or, Type "rpar 1GRC* " <enter>
3. Type "getprosol" <enter> to read in calibrated pulses information from standard samples
4. Type "atma" <enter> or "atmm" <enter> to optimized nuclei frequencies (if no ATM probe, check it manually by typing "wobb")
5. (recommended) Use 1H pulse calibrated by pulsecal
"getprosol Δ 1H Δ pulse us > Δ level in dB or dBW >"
6. Type "ns" <enter> to fill in scan number you like
7. (recommended) Type "eda" or "ased" <enter> to double confirm all parameters
8. Type "rga" <enter> to auto adjust receiver gain
9. Type "zg" <enter> to collect 2D spectrum
10. After the first series data is done,
Type "rser 1" <enter> to call out the 1st FID
11. Do "efp, apk, abs n" to check spectrum quality (if signal not strong enough, stop the data acquisition, increase scan number (step 6), and rga, zg again.)
12. Type "xfb" <enter> to Fourier Transform 2D
(Spectrum is ready for further processing/analysis)

Note:

- Select most commonly used expts from our standard experiment sets
- Important but may skip if already done for required nuclei
- For other nuclei, you may use default values
- ased would be easier

PART III: Remove Sample and Logout

1. Click "Lock" botton on [BSMS] to turn off lock (Fig.4)
2. With Autosampler: type "sx #" where # is an empty position
3. Without Autosampler:
Click "Lift" botton on [BSMS] to turn on air and remove your sample
Click "Lift" botton again to turn off air
4. Exit Topspin and Logout Computer

● Notes on Useful Commands

1. > **edte** → edit temperature
2. > **lockdisp** → to open lock sub-window
3. > **lock** → to lock the field for selected D-solvent field
4. > **edc** → copy current data set to a new one
5. > **rpar** → to read in available parameter set and overwrite current data
6. > **getprosol** → read in default pulses and parameters (standard samples)
7. > **atma** → auto tune and match nuclei frequency
8. > **atmm** → manually tune and match nuclei frequency
9. > **pulsecal** → auto determine 90 degree pulse for current sample
10. > **ased** → display acquisition parameters needed for specific experiment
11. > **expt** → estimate experiment time
12. > **rga** → auto estimate receiver gain
13. > **gs** → start acquisition but no data saving (useful for optimization)
14. > **zg** → zero memory and start data collection (overwrite existing data)
15. > **tr** → transfer collected FID after current scan (save existing FID)
16. > **tr #** → transfer collected FID after # scan
17. > **go** → start data collection , add on NS to existing data
18. > **stop** → stop data acquisition immediately
19. > **halt** → similar to stop but after the current status
20. > **qu **** → submit commands (**) to spooler
21. > **qumulti** → submit multiple commands to multiple experiments
22. > **sx #** → to switch sample position (#) on autosampler
23. > **efp** → **em** (window function), **ft** (Fourier transform), **pk** (pick phase)
24. > **apk** → to auto pick phase
25. > **abs n** → to auto baseline correction without integration
26. > **rser #** → read series file # (ie. available FIDs)
27. > **xfb** → to do Fourier transform on both dimensions
28. > **dpa** → to display acquisition parameters
29. > **ii** → to initialize hardware connection
30. > **ii restart** → to initialize hardware connection

Fig 1. Topspin Icon

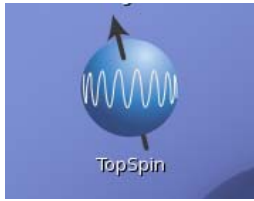
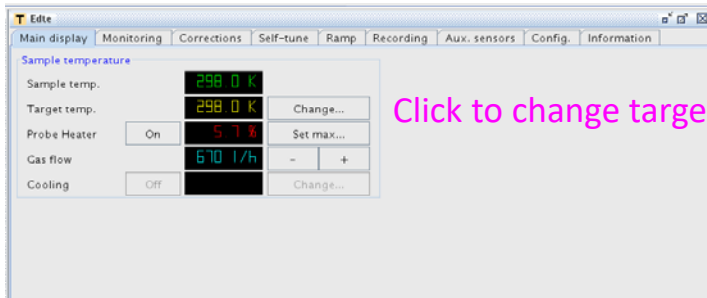


Fig 2. Temperature control window



Click to change target TEMP if needed

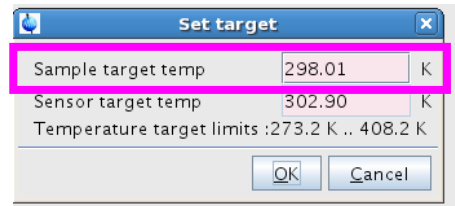


Fig 3. Sample and Spinner

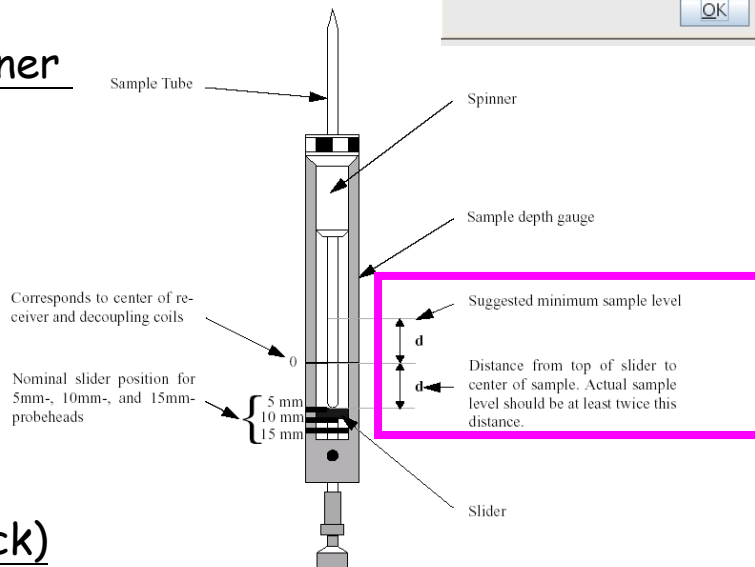


Fig 4. BSMS (Lift & Lock)

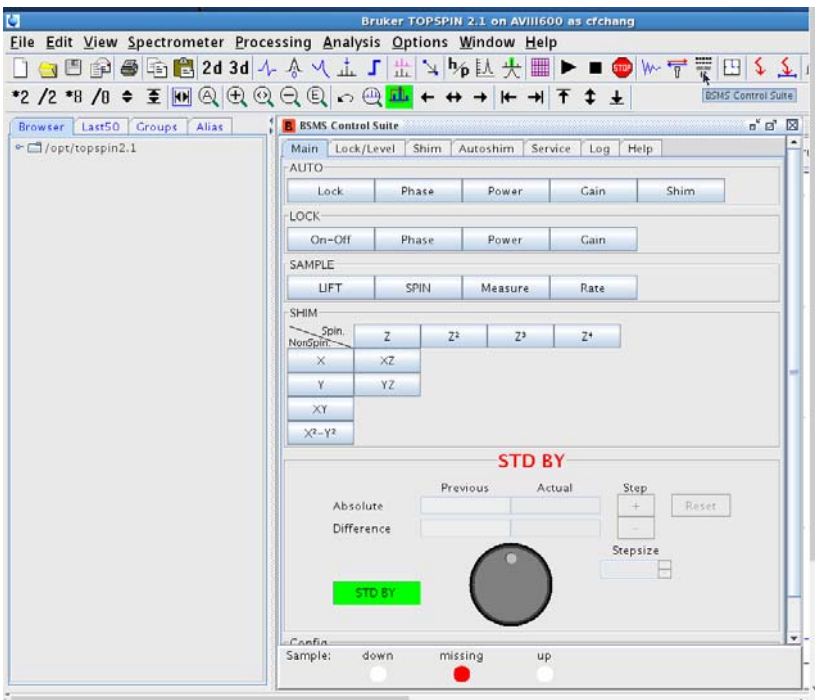
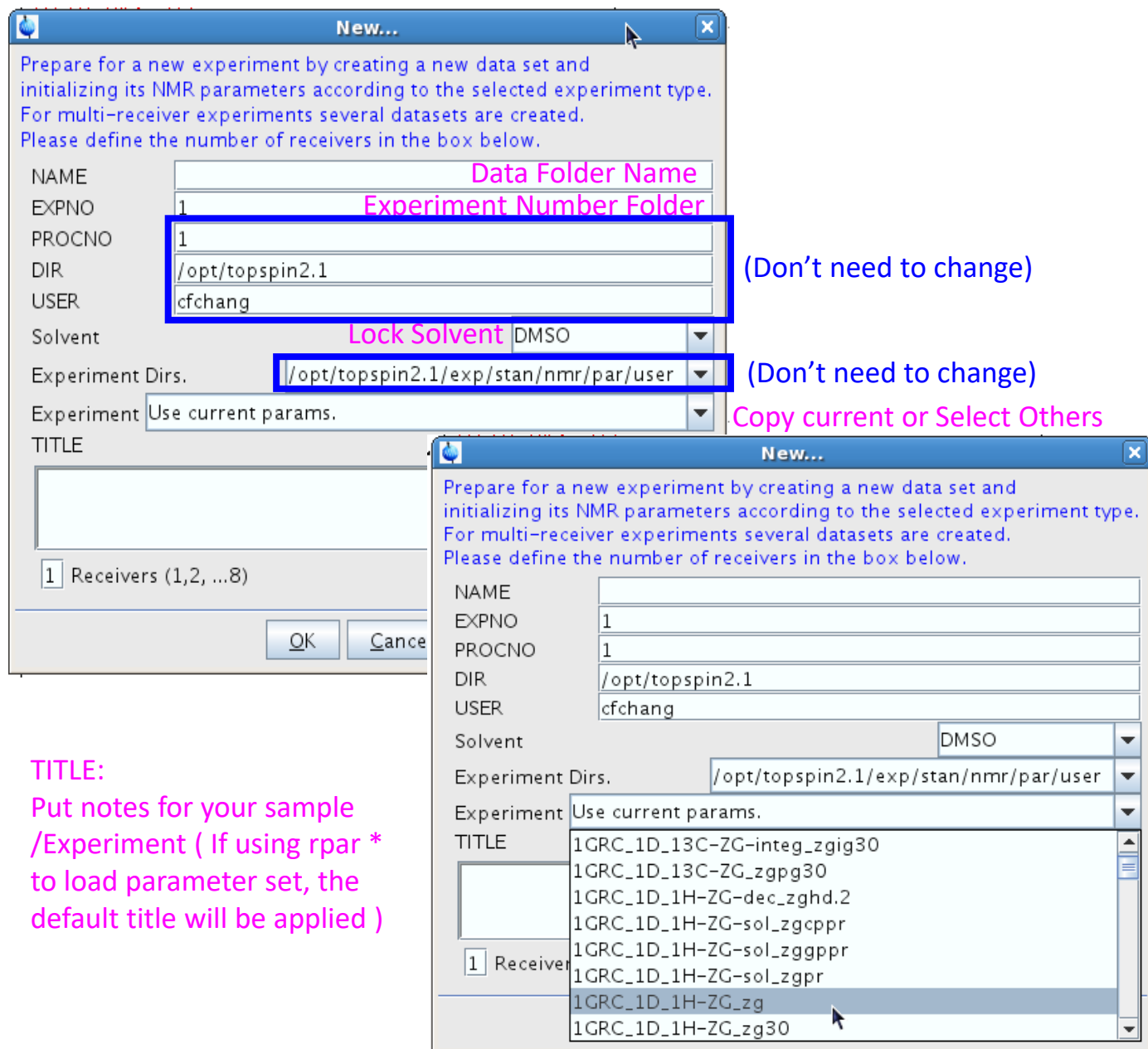
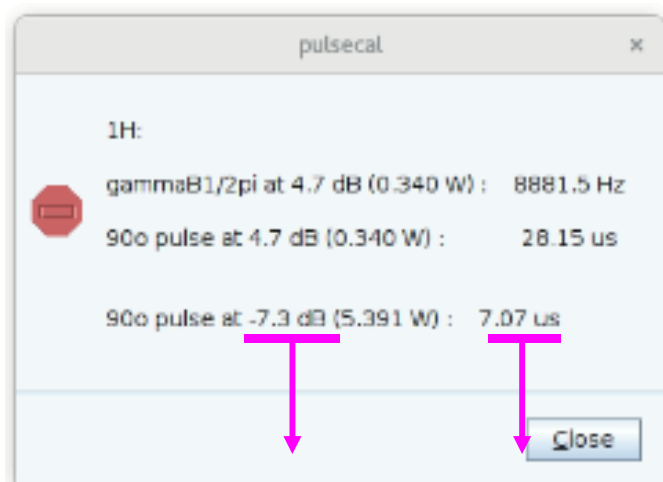


Fig 5. edc window



TITLE:
 Put notes for your sample /Experiment (If using rpar * to load parameter set, the default title will be applied)

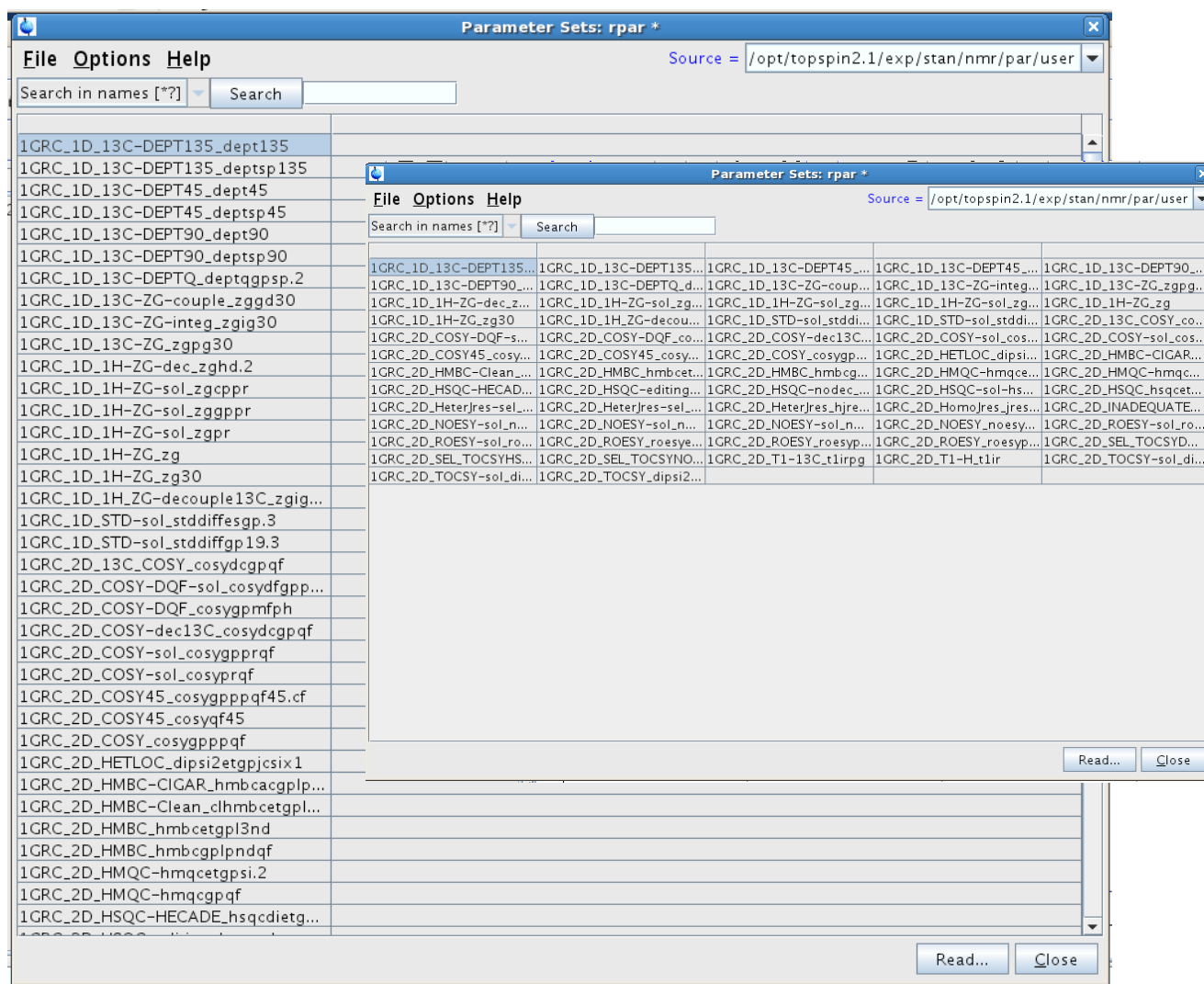
Fig 6. "pulsecal" output window



power level In "dB"
 Pulse length In "us"

Pulse length In "us"
 getprosol 1H 7.07 -7.3
 power level In "dB"

Useful Parameter Sets for small molecules in HFNMR



FTP <ftp.nmr.sinica.edu.tw>

/opt/topspin/(GroupAccount)/data/(your personal folder)

Mnova Campus License

<https://www.nmr.sinica.edu.tw/Mnova/>

Sinica Reservation System

<https://reservation.iis.sinica.edu.tw/servlet/SignInHandler>