Simple Operation Guide for HFNMRC Users

by Dr. Chi-Fon Chang for small molecules (2023.10.20 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.
- When using "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 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 H20/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 ....)

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"

● Extra notes:

- If using optimized pulses from "pulsecal" for other experiments (Fig.6)
  Type "**getprosol Δ 1HΔ<pulse in us >Δ<power level in dB or dBW>**"
  (ex: getprosolΔ1HΔ**10.2**Δ**-3.5**) instead of "getprosol"

● 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 1HΔ<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

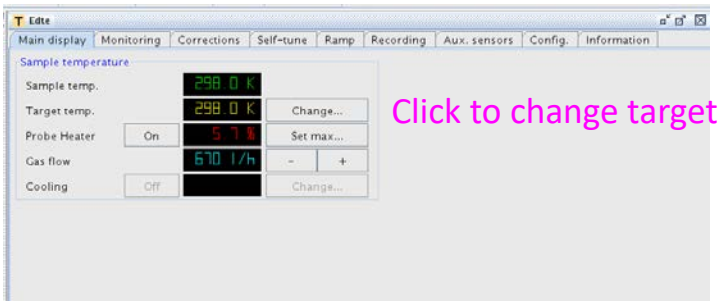
1. Click "**Lock**" botton on [BSMS] to turn off lock (Fig.4)
2. With Autosampler: type "**sx #**" where # is an empty position
3. Without Autosampler:
   Click "**Lift**" botton on [BSMS] to turn on air and remove your sample
   Click "**Lift**" botton again to turn off air
4. **Exit Topspin** and **Logout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

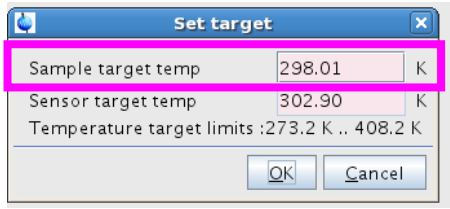


Fig 3. Sample and Spinner

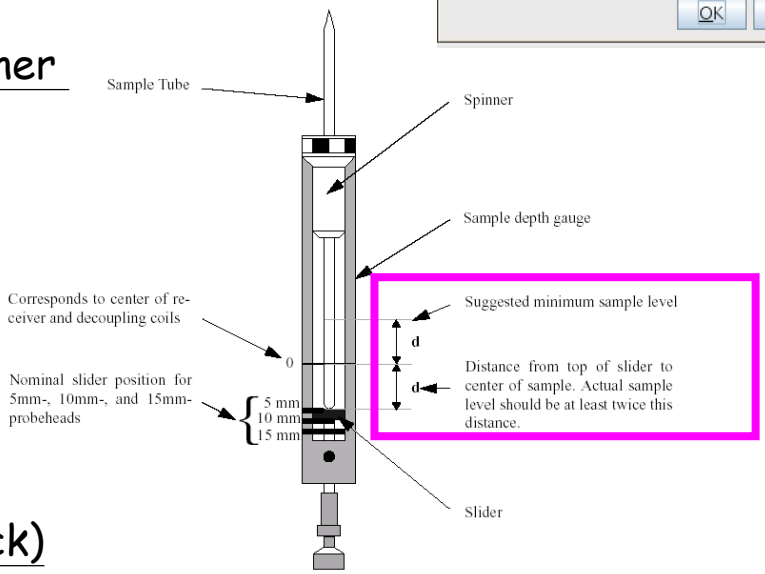


Fig 4. BSMS (Lift & Lock)

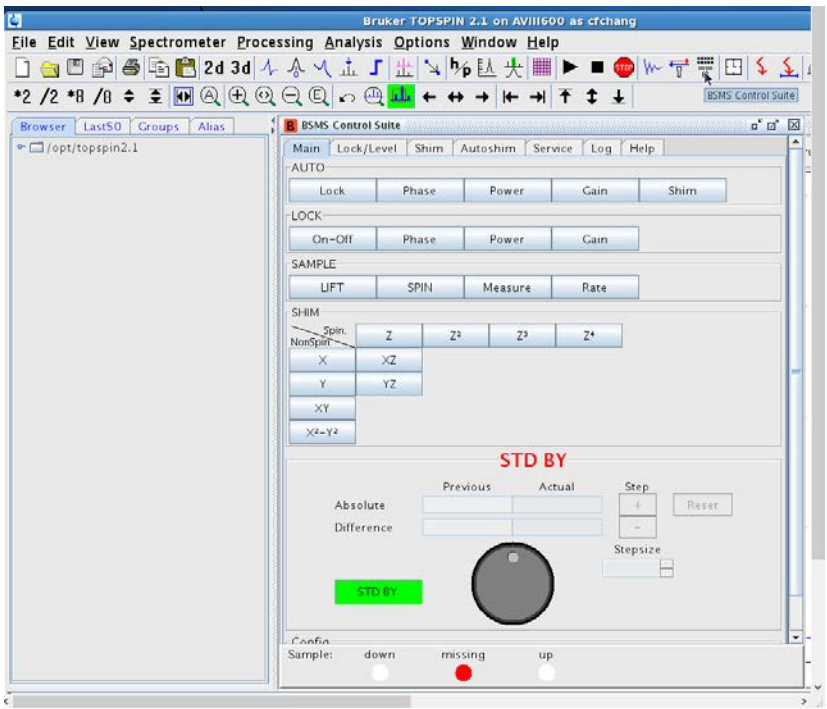
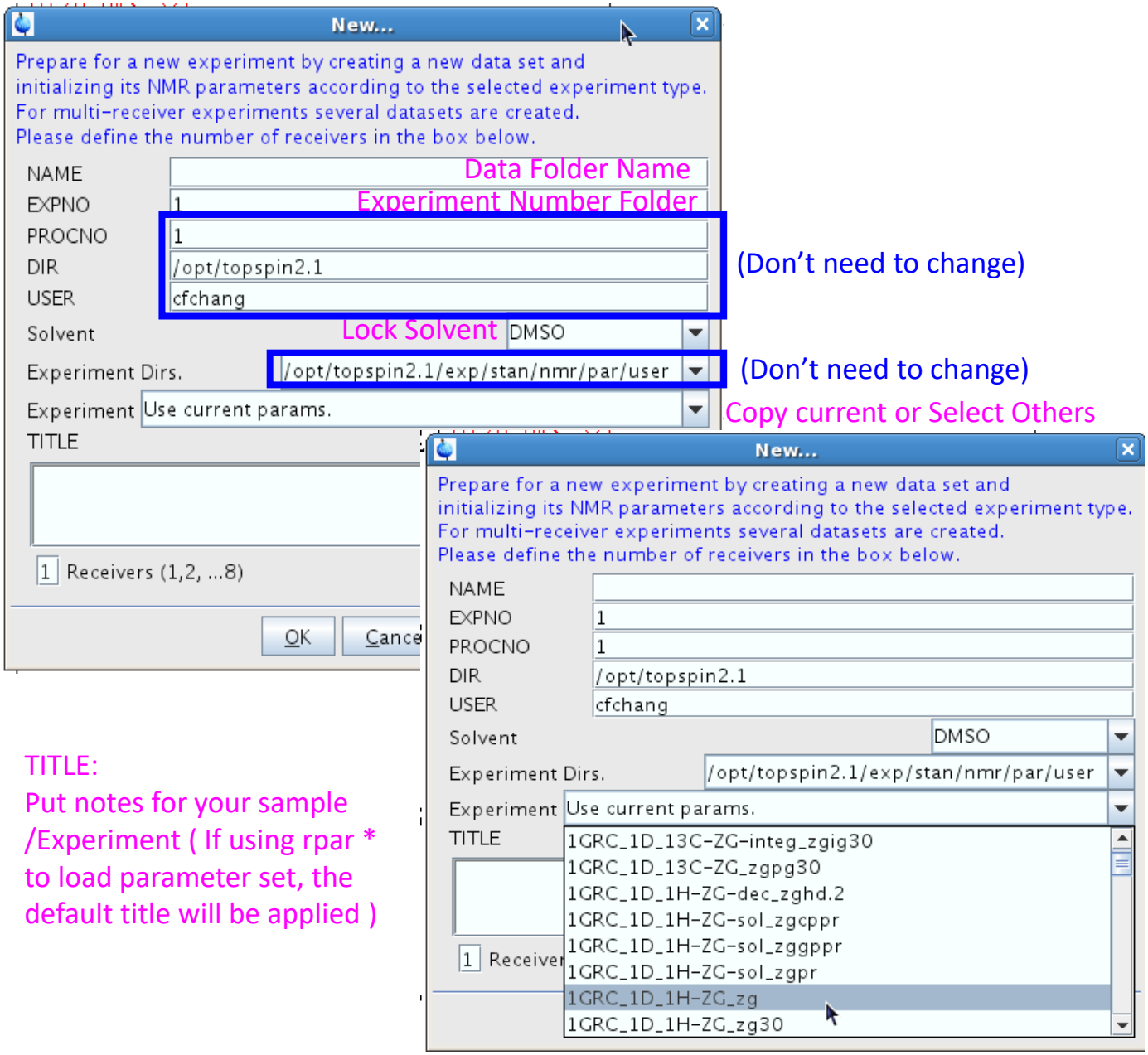
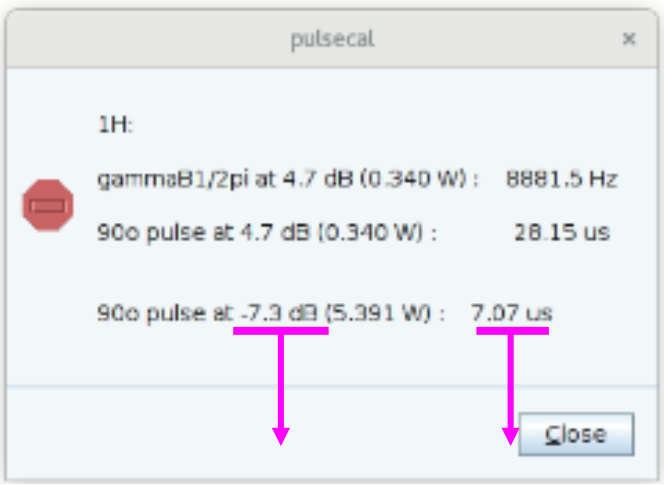


Fig 5. edc window



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"

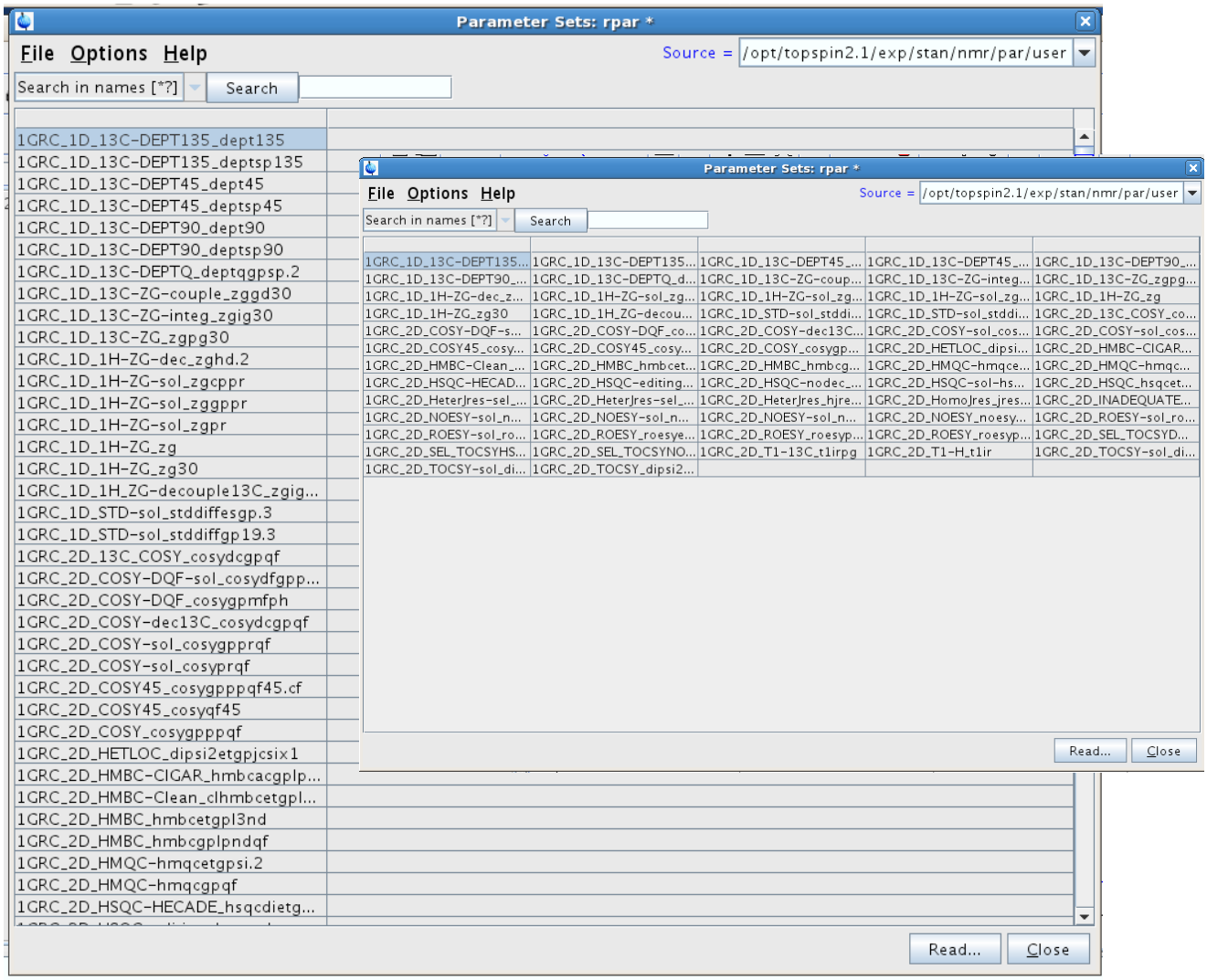
Pulse length
In "us"

Pulse length
In "us"

getprosol 1H 7.07 -7.3

power level
In "dB"

Useful Parameter Sets for small molecules in HFNMRC



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