NMR Data Acquisition and Processing Procedure

Dr. Jianfeng Zhu (Research Officer)

Please **DO NOT** remove from NMR lab!

Things to Avoid when Using NMR Lab

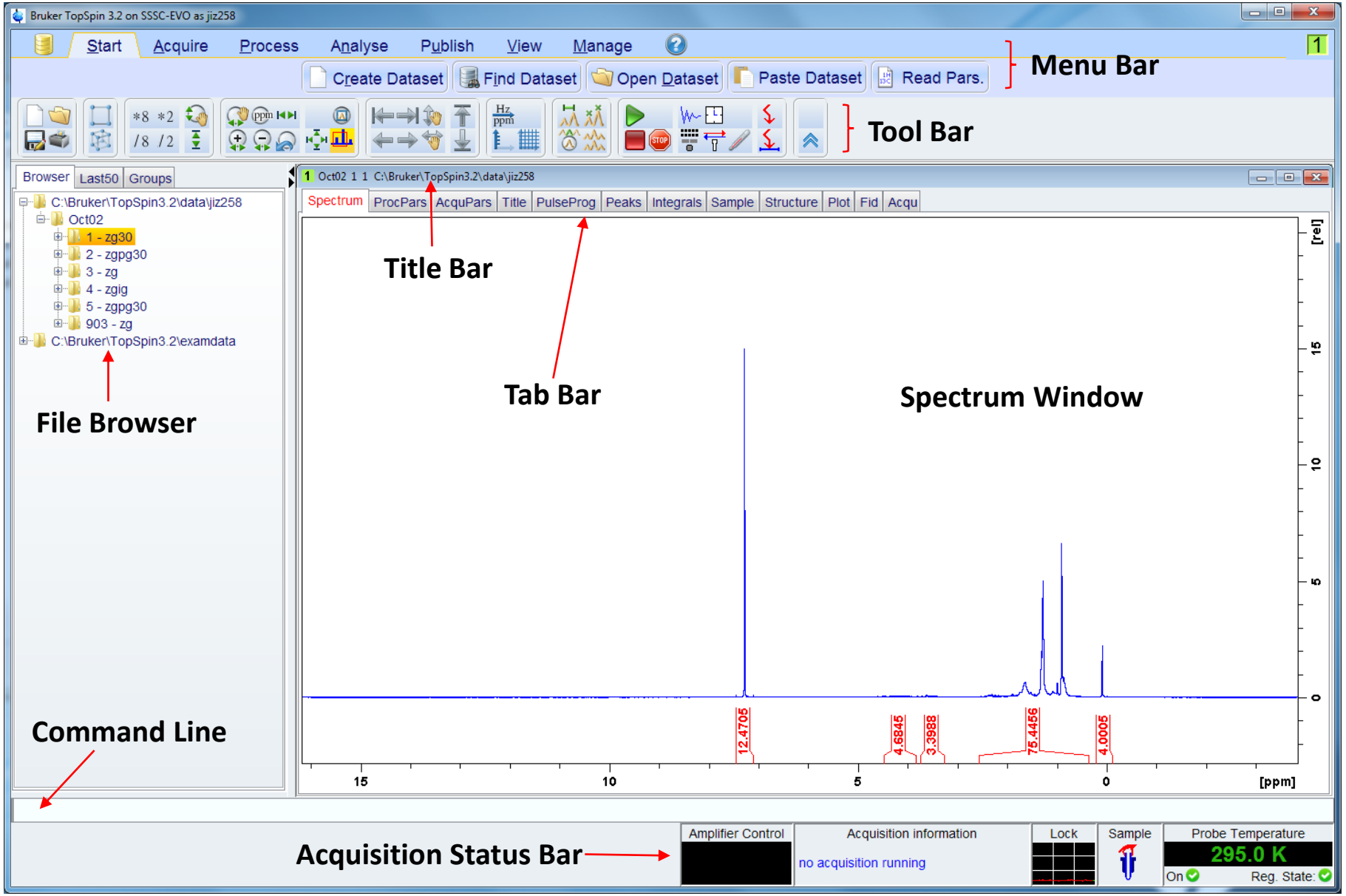
Here's a list of stuff that I have seen that you *should not* do:

1. Do not bring a magnetic substance near to the magnet ... after all it's a *magnet* and a very powerful one at that. Screwdrivers and wrenches are death tools around an NMR magnet ... keep them away!
2. Do not put little tape 'flags' with the name of your sample on your NMR tube. If the flag is left on the tube it will stick inside the magnet for sure (Murphy's Law).
3. Do not put your sample into a depth gauge and then try to put the whole thing, gauge and all, into the magnet.
4. Do not put the spinner on *upside down* and then put sample and spinner into the magnet. There *will* be problems.
5. Do not make the mistake of thinking that just because a sample in the autosampler carousel is past the sample injection point that it has already run. The *only* way to know if it has run is to look at the ICONNMR display.
6. Do **not** pull on the autosampler carousel in order to rotate it and retrieve your sample!!! Ever.
7. If you must take un-run samples out, PLEASE also cancel the samples in ICONNMR.
8. Do not be helpful and clean the NMR tube depth gauge with acetone. The gauge is plastic and will dissolve in acetone.
9. Do not be helpful and try to fix a broken depth gauge ... there are tiny springs in the stop mechanism that will fly out if you try to take it apart, never to be found again.
10. Do not put the sample into the magnet unless the lift air is turned on. Again, you risk damaging the probe if you simply drop the sample into the magnet.
11. Do not force the tuning/matching rods to turn further than their maximum or minimum positions ... this will cause serious internal damage to the probe and render it immediately useless until repaired.
12. Do not overreach when inserting or removing a sample ... you might lose your balance and injure yourself or you might break an NMR tube with sample in it.
13. Do not forget to shut off the lift air after you are finished with the machine.
14. Do not put a sample into the magnet and begin to take data when we are filling the magnets with cryogens.
15. *YOU* are responsible for the shimming. If you cannot shim your sample try starting from scratch by reading in the standard shim file (type 'rsh' and select 'currentshim') *before* complaining to the laboratory manager. If you still cannot shim the sample satisfactorily then contact one of the NMR facility staff.

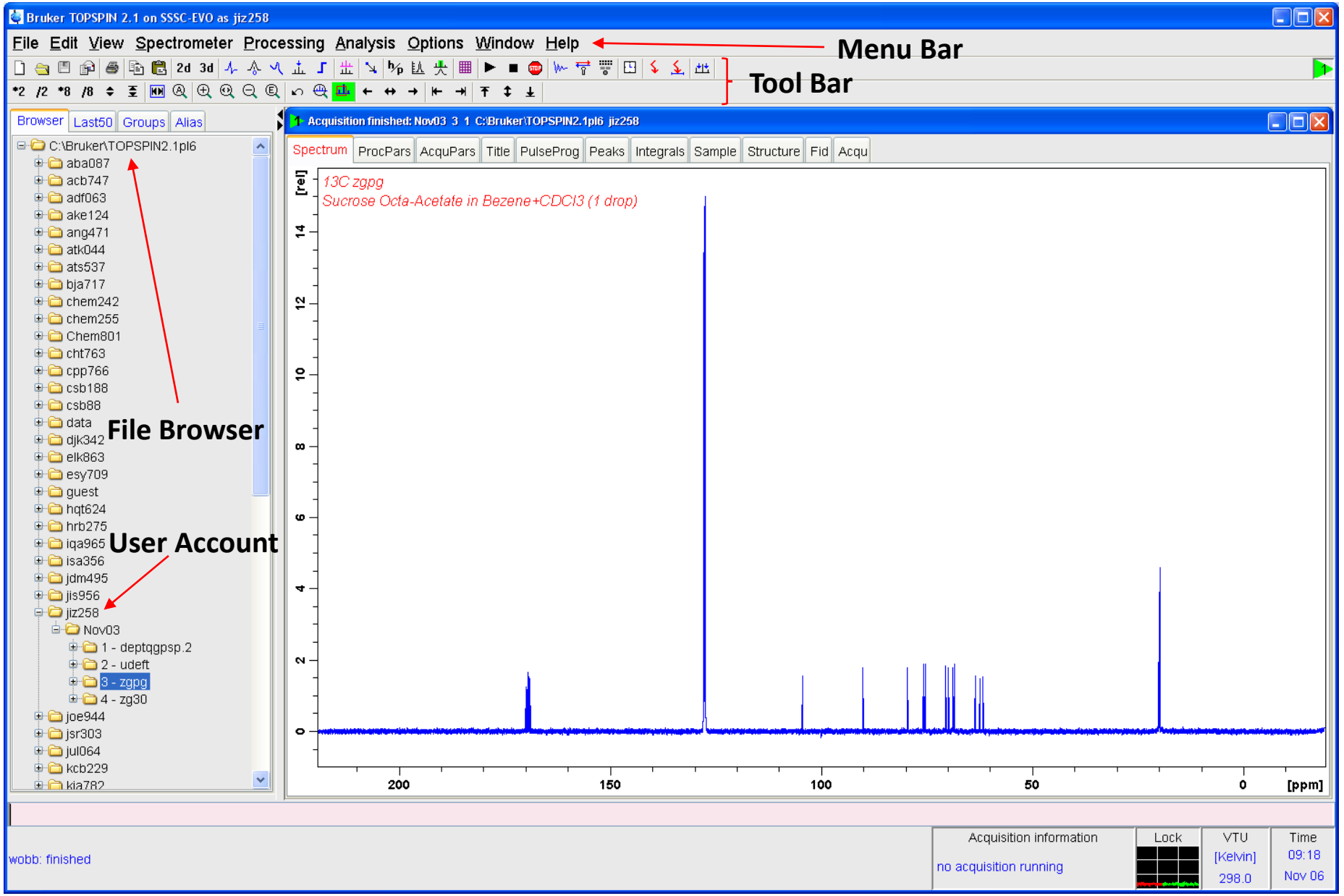
Procedures	Commands	Notes
1. Insert sample.	*ej* → load sample → *ij*	*sx #* (load sample using auto-sampler).
2. Create new data set.	*new* or *edc*	
3. Read in standard parameter set.	*rpar* → *choose …*	
4. Read in pulses and powers.	*getprosol*	*gpro* (Macro to shorten the command).
5. Check temperature setting.	*edte* → *set …*	
6. Lock the solvent.	*lock* → *choose …*	 (lock display window).
7. Tune the probe.	*atma*	
8. Shim the magnet.	*topshim*	*Topshim gui* (open Topshim interface). 
9. Check acquisition parameters.	*ased*	
10. Adjust receiver gain.	*rga*	
11. Start acquisition.	*zg*	*tr* (transfer/save data to workstation).
12. Processing the data.	*efp* → *apk* () → *absn*	*proc* (Macro combining the three commands).
13. Peak picking.	*pp*	 (open peak picking Interactive Bar).
14. Peak integration.	*int*	 (open integration Interactive Bar).
15. Plot the spectrum.	*plot*	

*** Refer to succeeding contents for detailed information of each step.**

Topspin3.2 Interface

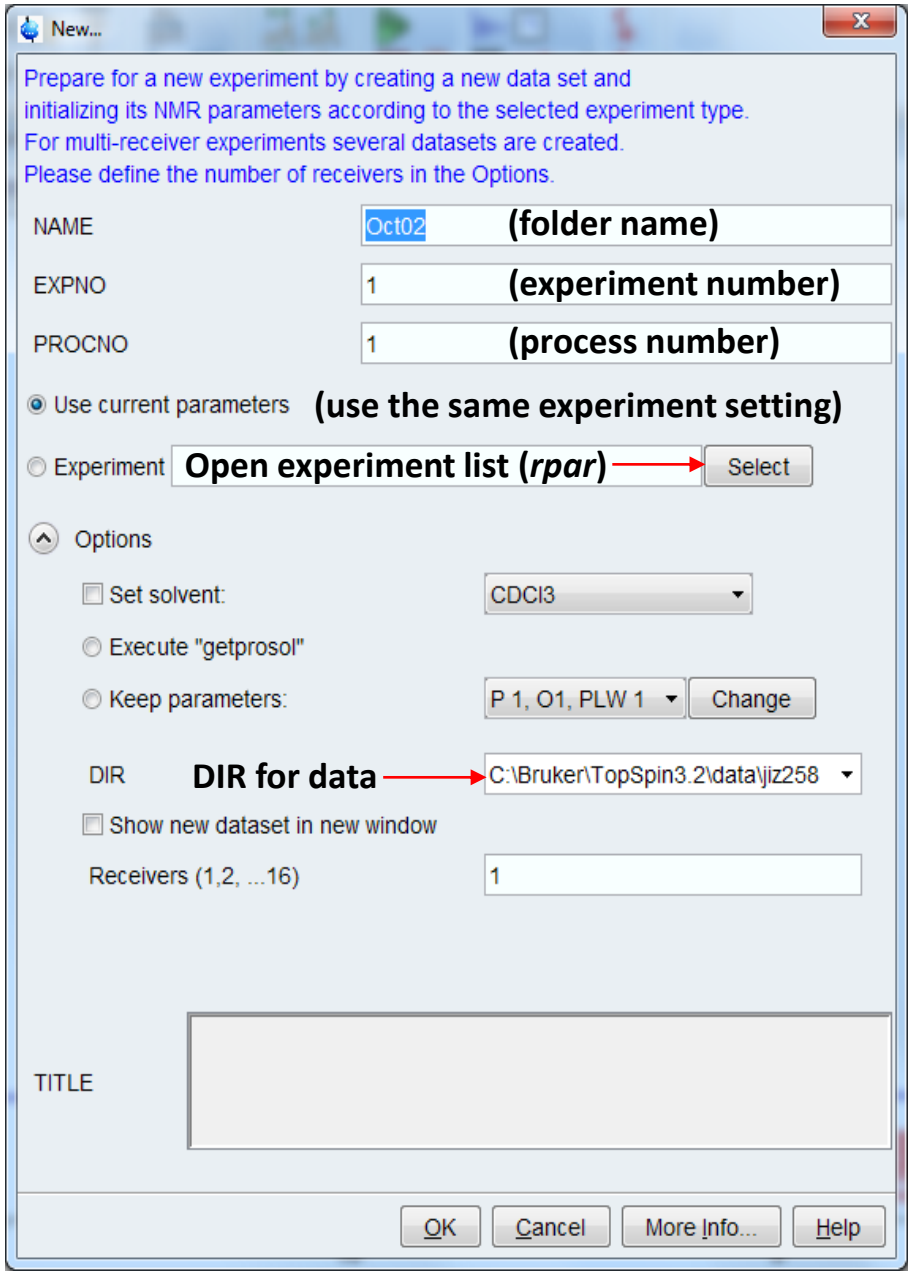


Topspin2.1 Interface

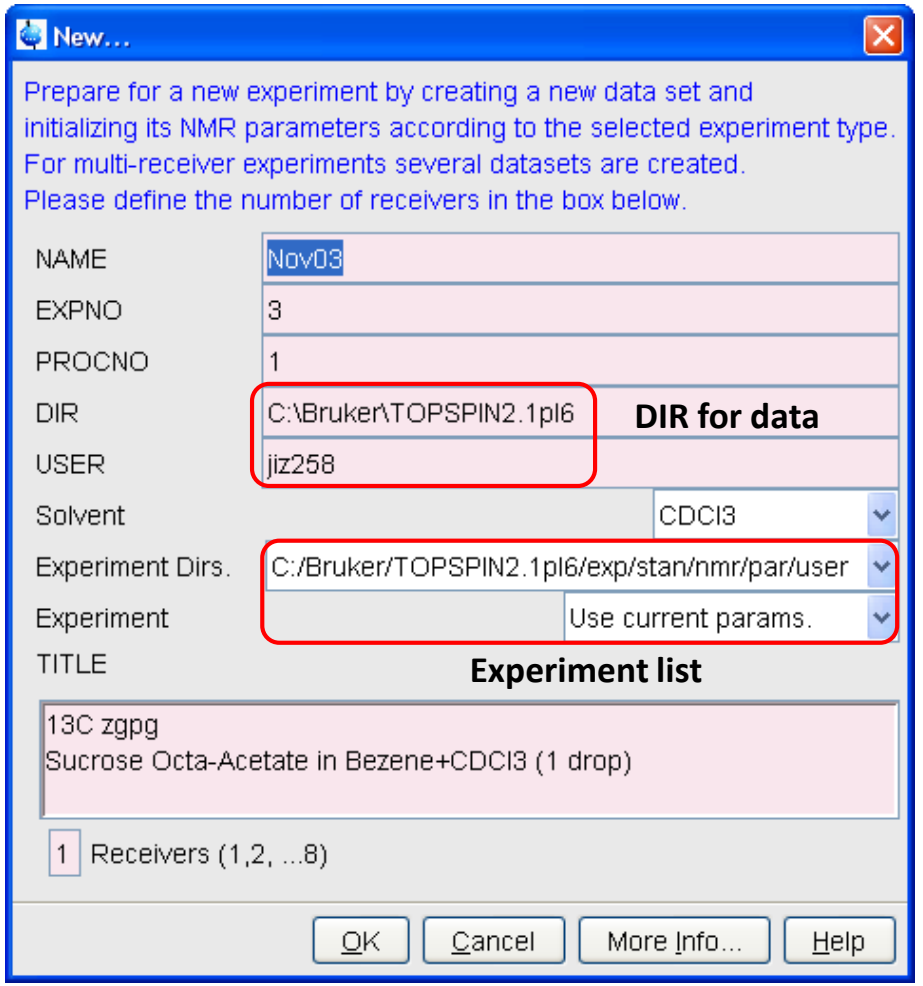


2. Create New Data Set

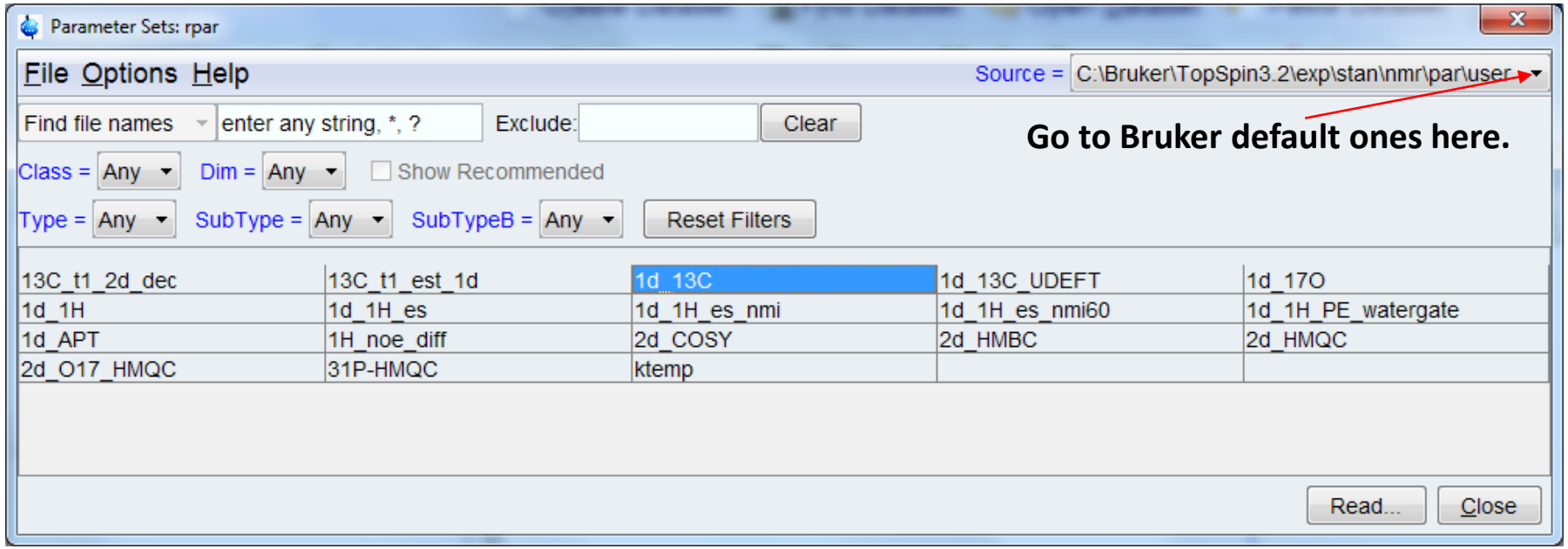
Topspin3.2



Topspin2.1



3. Read in Standard Parameter Set

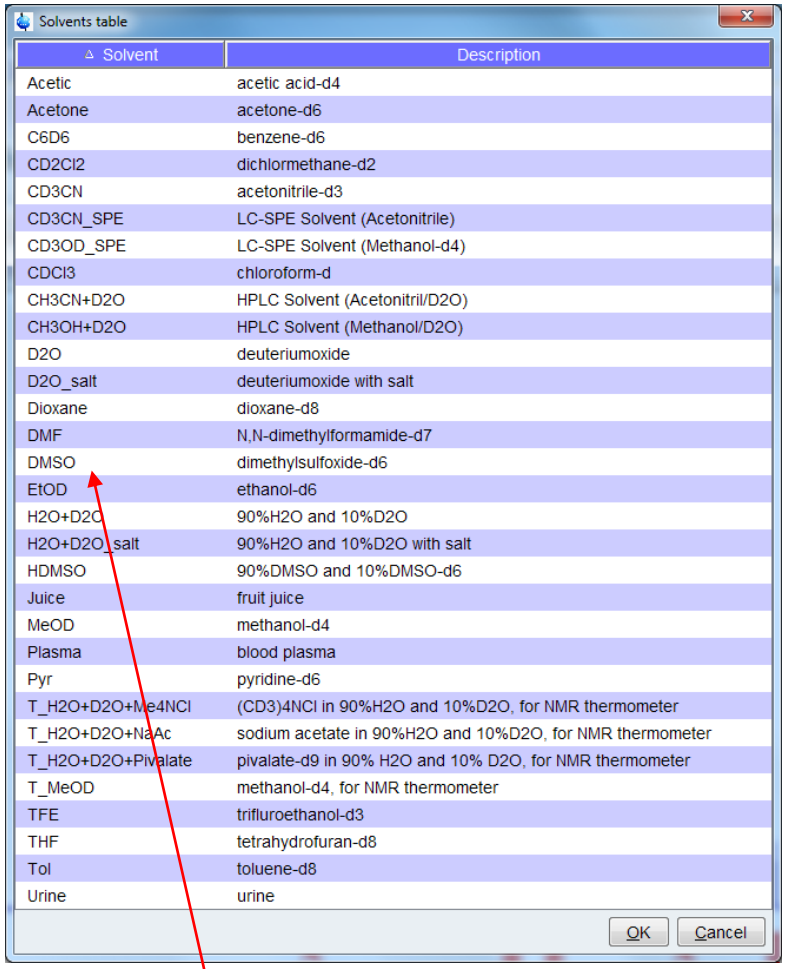


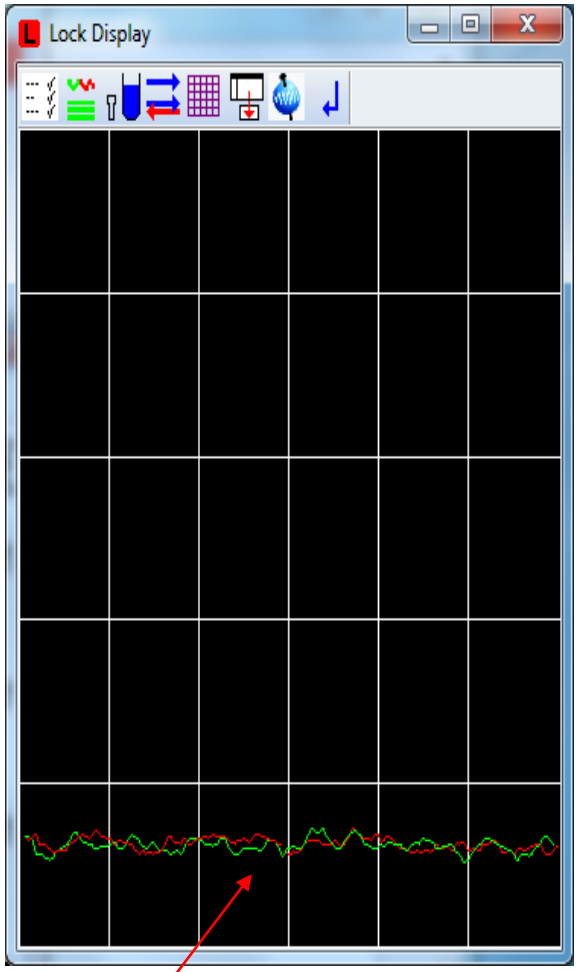
Existing Parameter Sets:

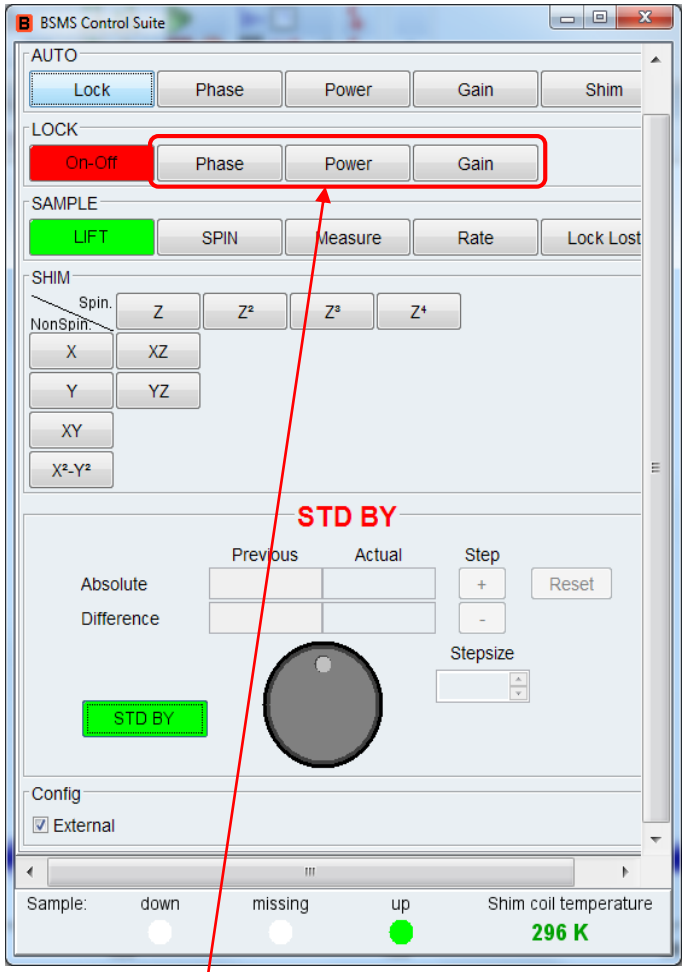
- Standard 1D: ^{1}H, ^{7}Li, ^{11}B, ^{13}C, ^{15}N, ^{17}O, ^{19}F, ^{27}Al, ^{29}Si, ^{31}P. (Format: ***1d_#X***, e.g. ***1d_13C***).
- Standard 2D: COSY, HMQC, HSQC, HMBC. (Format: ***2d_XXXX***, e.g. ***2d_COSY***).
- Other ^{1}H Expts: NOE, water suppression, T_1 estimate, homonuclear decoupling, etc.
- Other ^{13}C Expts: UDEFT, DEPTq, INADEQUATE, T_1 estimate, etc.

5. Set Up Variable Temperature

6. Lock the Solvent







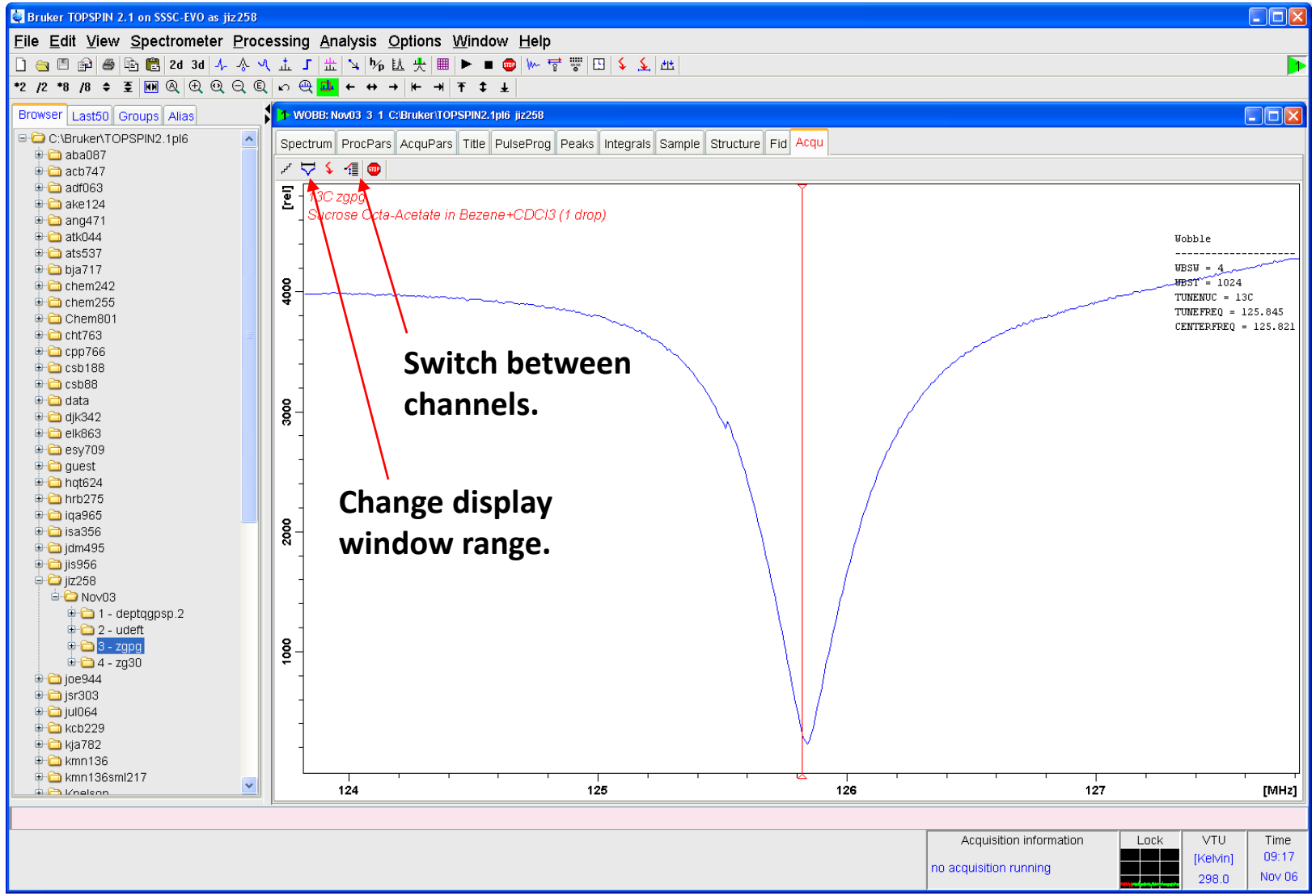
Choose the solvent from the list.

Check the lock level to get a sense of shimming.

Optimize these parameters if the lock signal is still weak after Topshim.

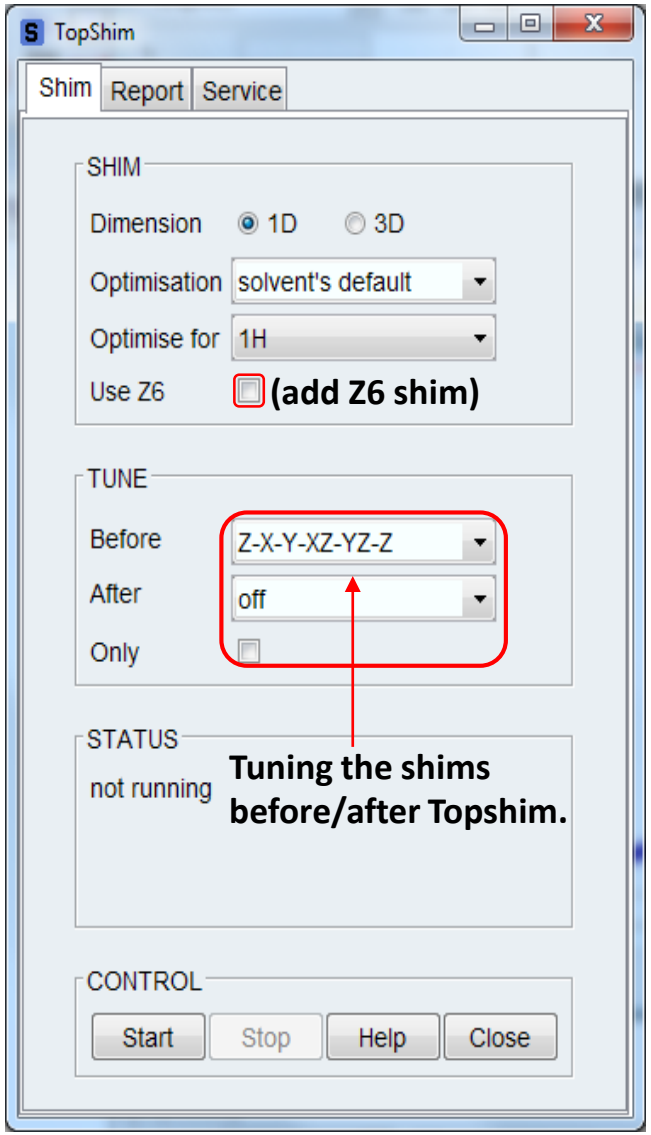
7. Tune the Probe

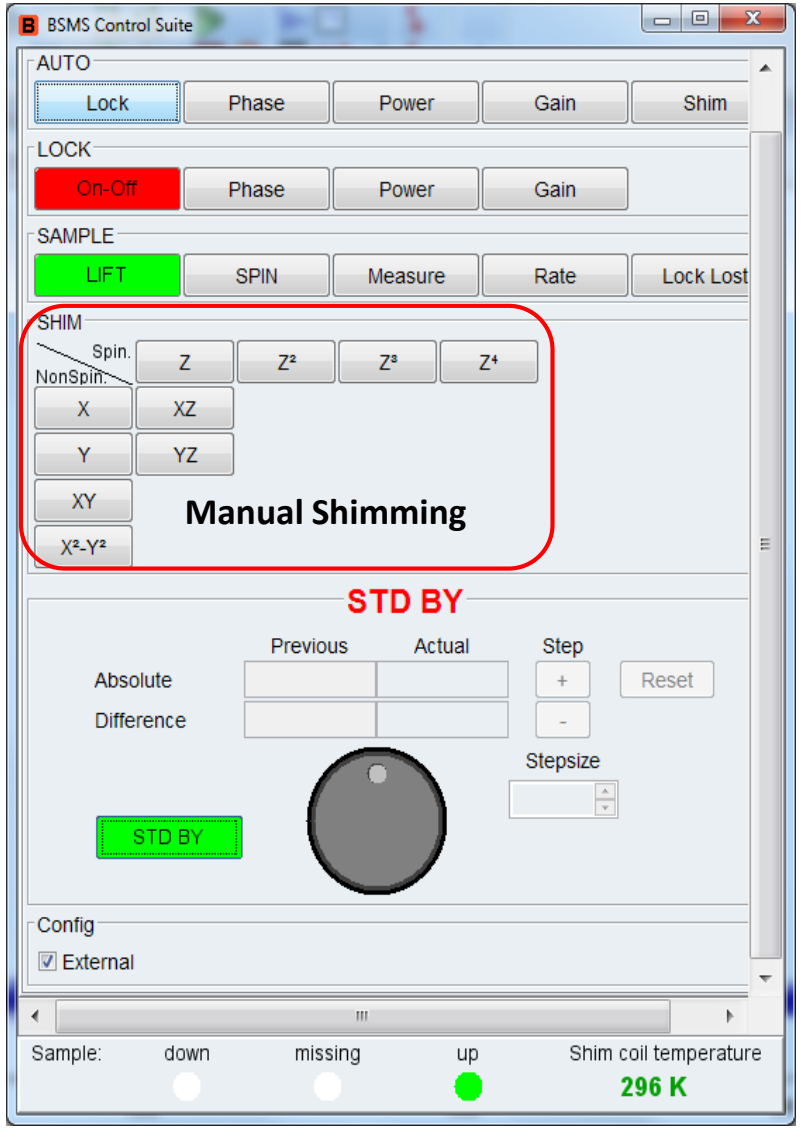
- **Tuning and Matching are done automatically for most probes (atma).**
- **Use ‘wobb’ and manual tuning/matching if necessary.**



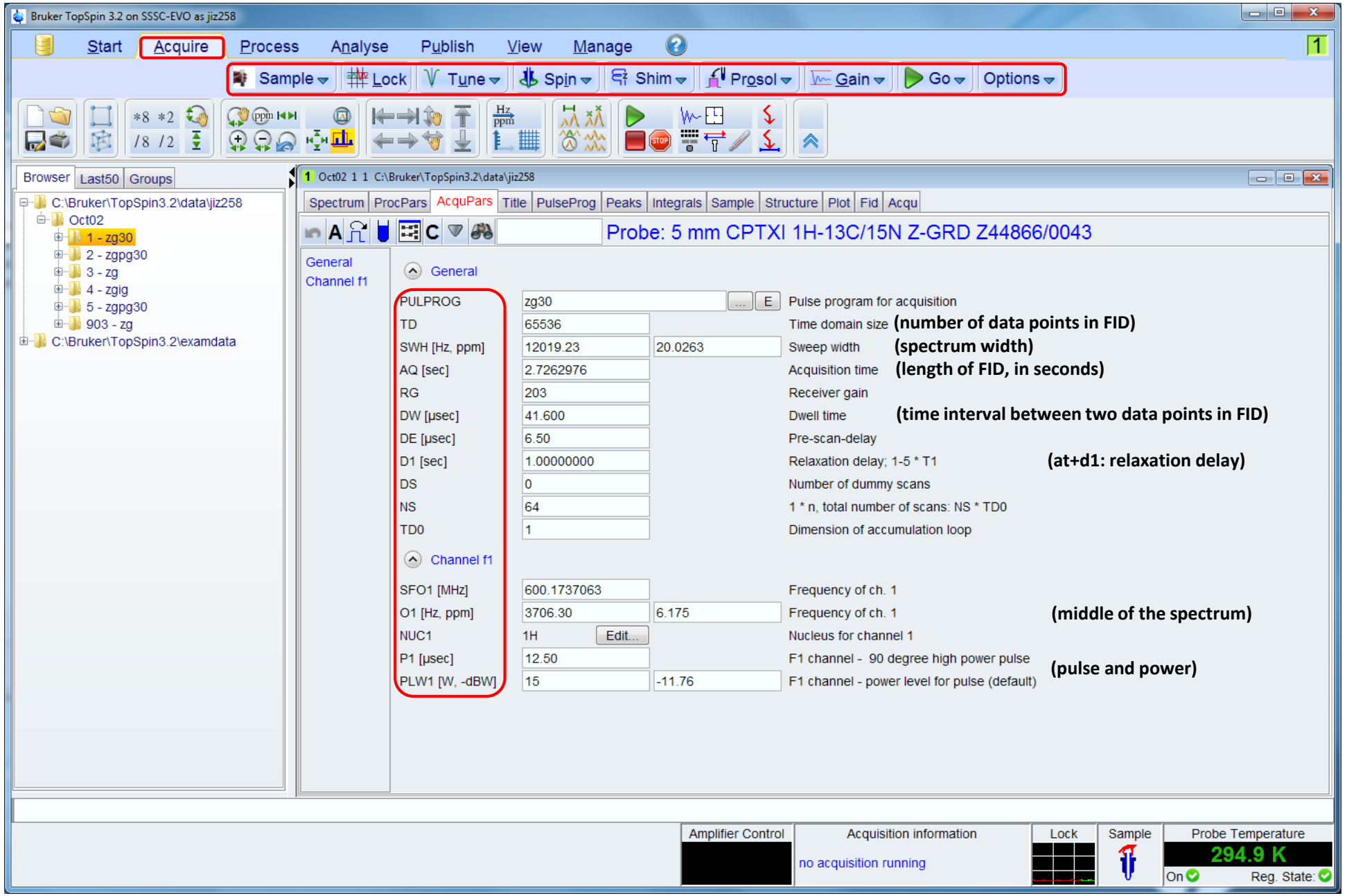
8. Shim the Magnet

- **Run ‘topshim’ alone is usually enough. If the shims are still not good, try ‘topshim gui’ as show below.**

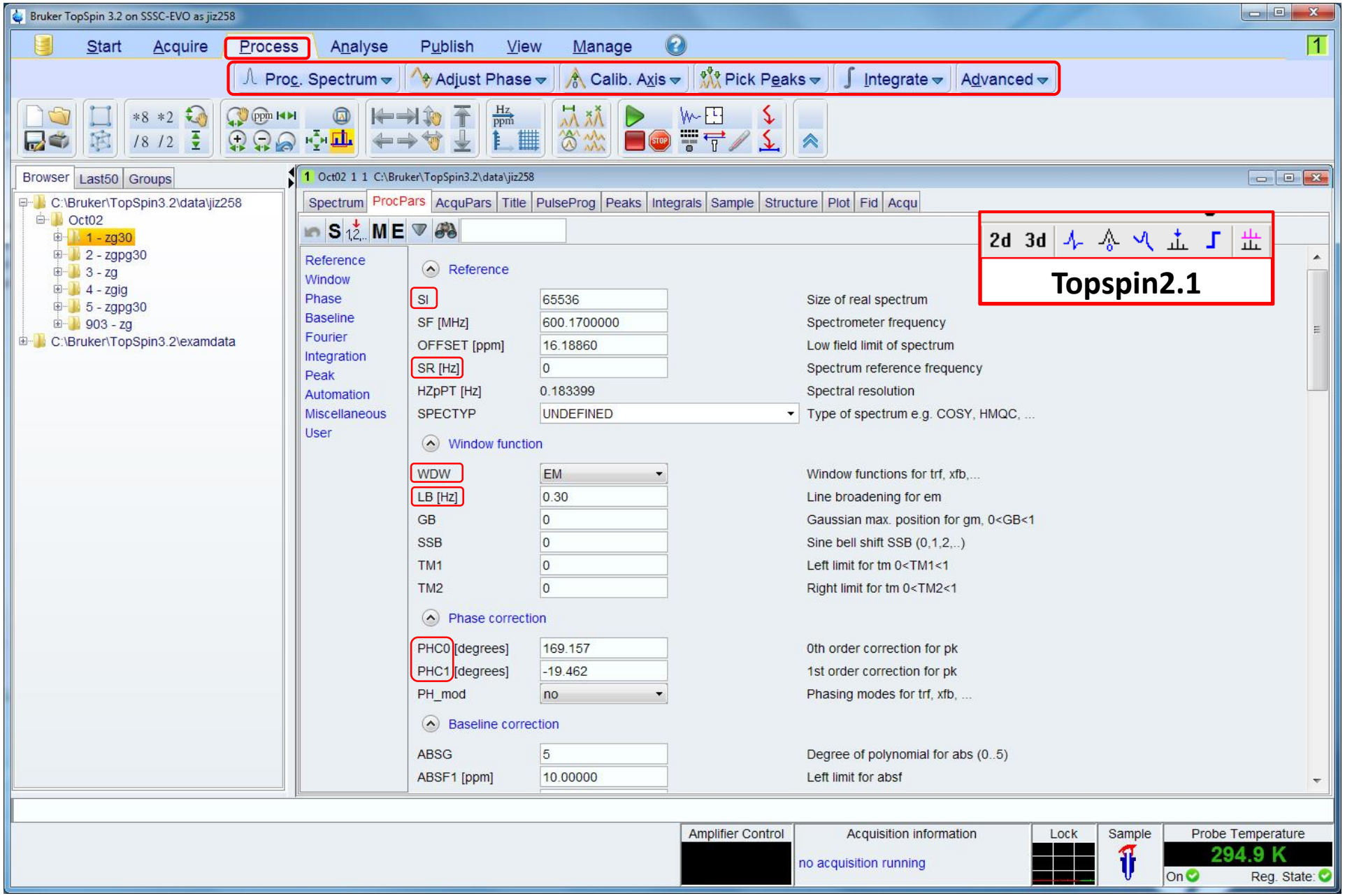




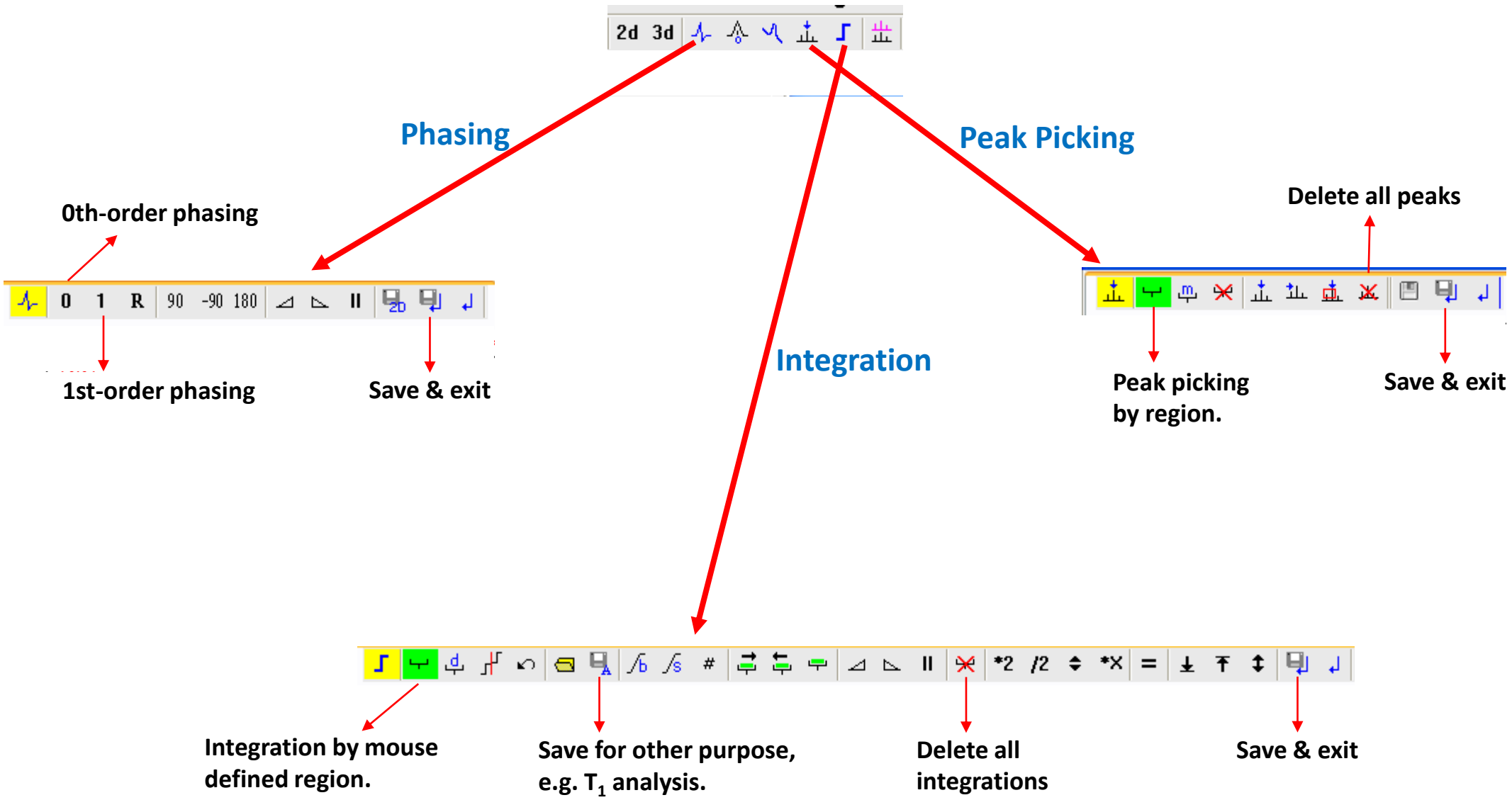
9. Acquisition Parameters



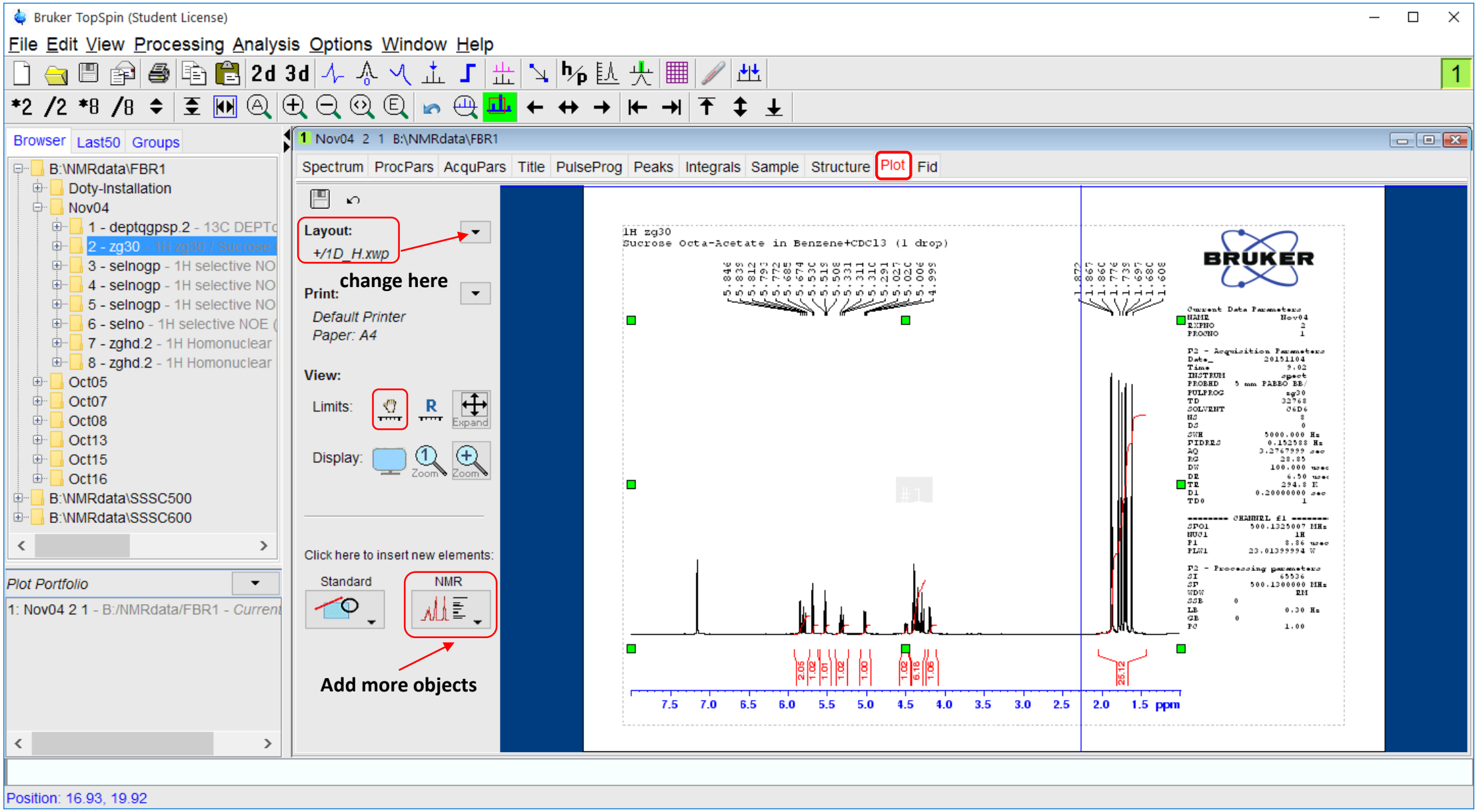
12. Processing the Data (1)



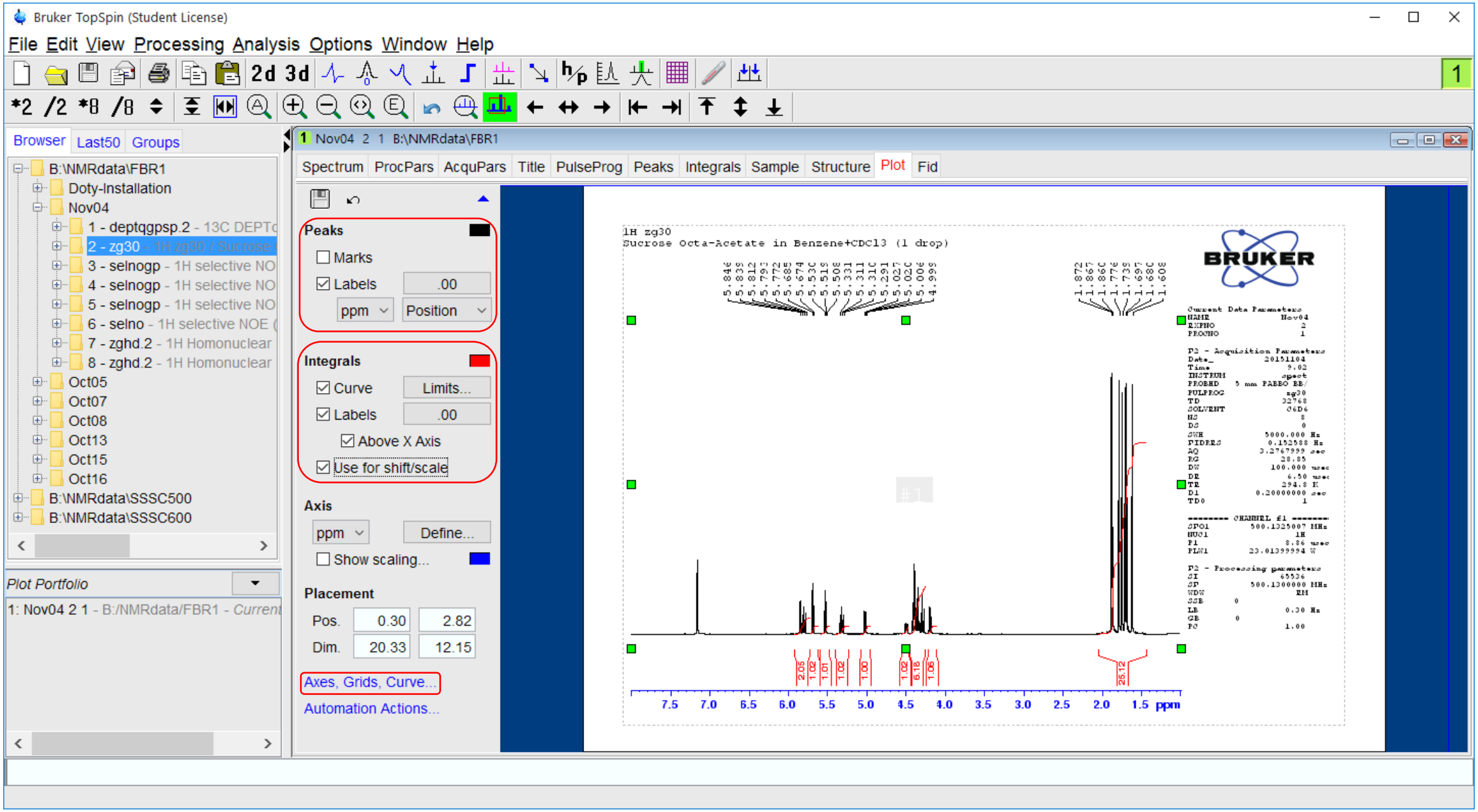
12. Processing the Data (2)



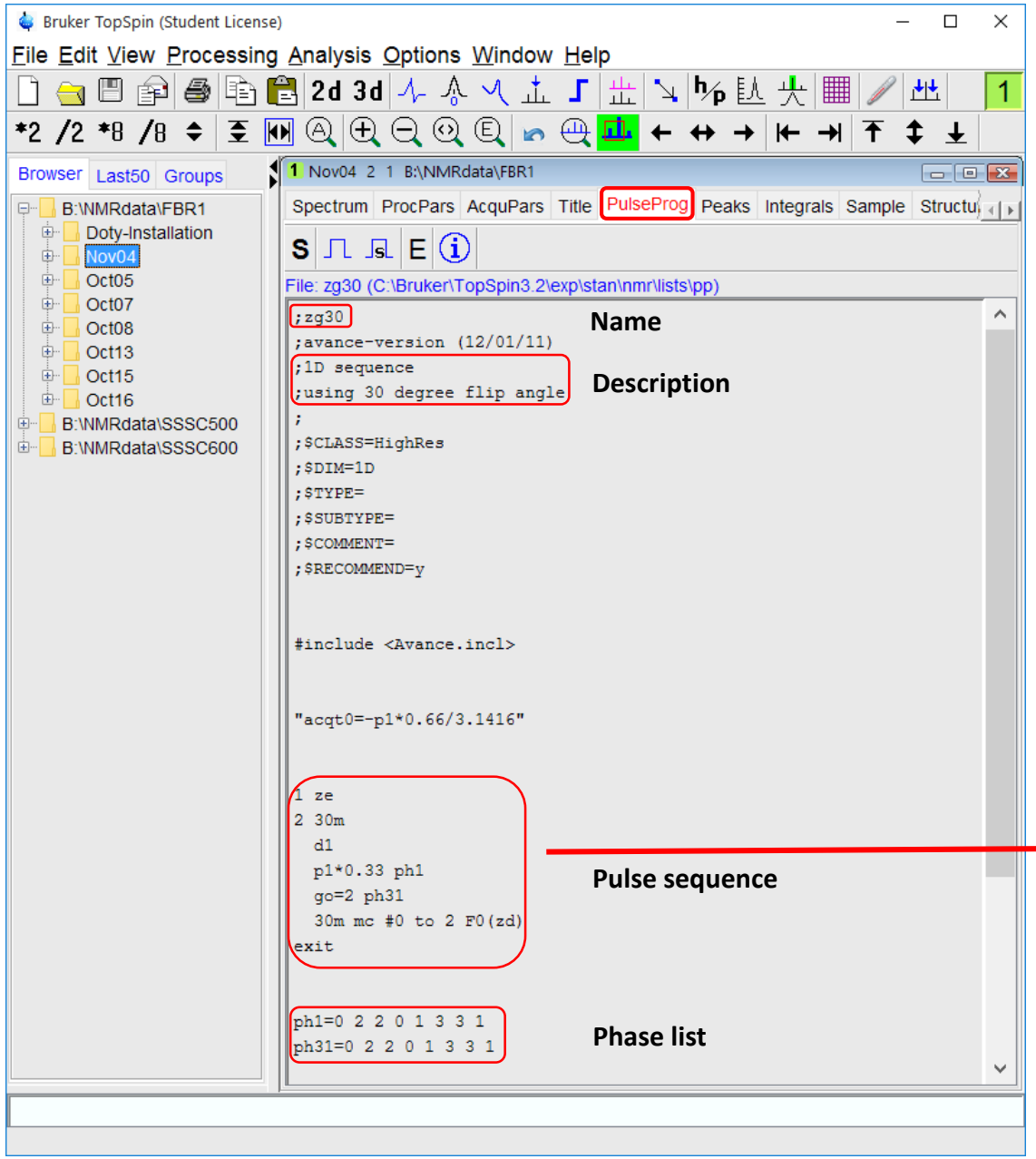
15. Plot the Spectrum (1)



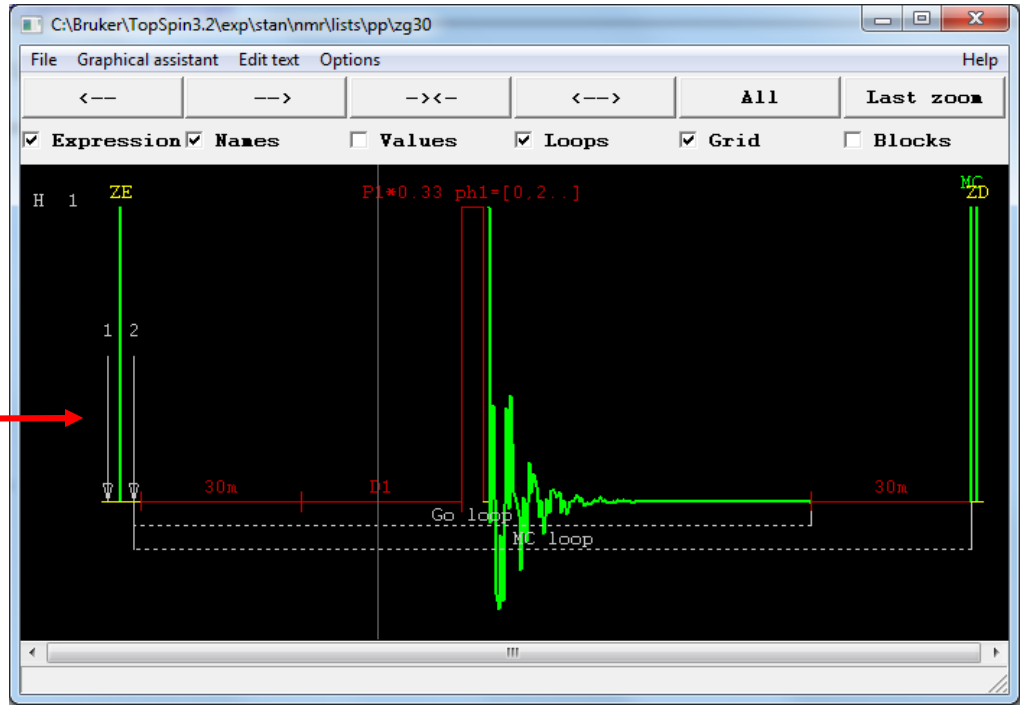
15. Plot the Spectrum (2)



Bruker Pulse Program (1)



1 ze (Reset scan counter and enable the execution of dummy scans.)
2 30m (Set up a label for loop.)
d1 (Apply a delay of 'd1'.)
p1*0.33 ph1 (Apply a 30° pulse on f1 with phase list 'ph1'.)
go=2 ph31 (Execute one scan and then loop to line '2' (NS-1) times.)
30m mc #0 to 2 F0 (zd) (mc macro to write data, including a disk writer (*wr*), a file increment (*if*) and memory initialization (*zd*).)
exit (The end of the pulse sequence.)



Bruker Pulse Program (2)

Bruker TopSpin (Student License)

File Edit View Processing Analysis Options Window Help

Browser Last50 Groups

B:\NMRdata\FBR1
B:\NMRdata\SSSC500
B:\NMRdata\SSSC600

Oct16 4 1 B:\NMRdata\FBR1

Spectrum ProcPars AcquPars Title PulseProg Peaks Integrals Sample Structure

File: zgpg30 (C:\Bruker\TopSpin3.2\exp\stan\nmr\lists\pp)

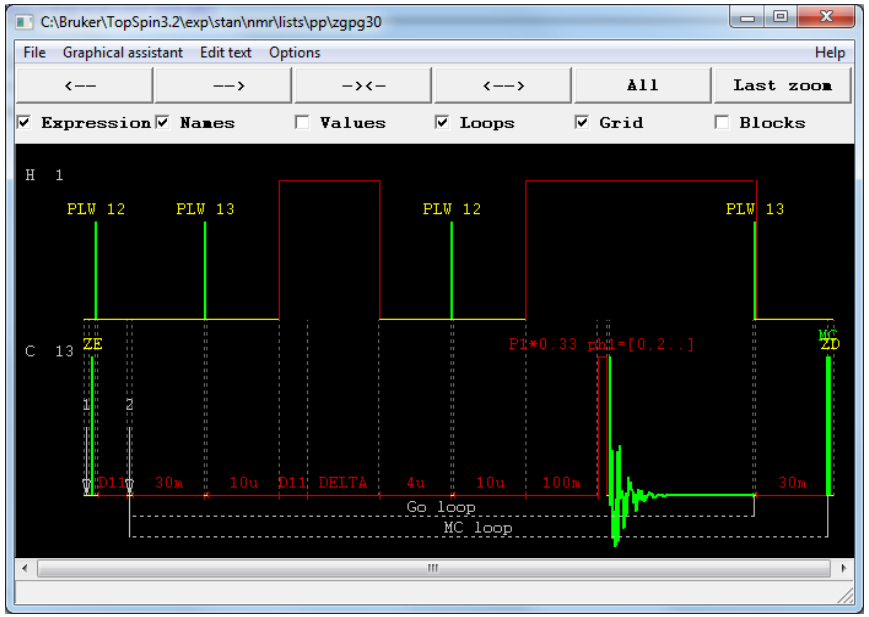
```
"acqt0=-p1*0.66/3.1416"


1 ze
  d11 pl12:f2
2 30m do:f2
  10u pl13:f2
  d11 cpd2:f2
  DELTA
  4u do:f2
  10u pl12:f2
  100m cpd2:f2
  p1*0.33 ph1
  go=2 ph31
  30m do:f2 pl13:f2 mc #0 to 2 F0(zd)
exit


ph1=0 2 2 0 1 3 3 1
ph31=0 2 2 0 1 3 3 1
```

Turn off decoupling on f2.

Turn on CPD decoupling on f2.

Turn off decoupling on f2.

Turn on CPD decoupling on f2.



Bruker TopSpin (Student License)

File Edit View Processing Analysis Options Window Help

Last50 Groups
Browser

Nov03 3 1 B:\NMRdata\SSSC500

Spectrum ProcPars AcquPars Title PulseProg Peaks Integrals Sample

File: zgpg (C:\Bruker\TopSpin3.2\exp\stan\nmr\lists\pp)

Description of Parameters

```
;pl1 : f1 channel - power level for pulse (default)
;pl12: f2 channel - power level for CPD/BB decoupling
;pl13: f2 channel - power level for second CPD/BB decoupling
;p1 : f1 channel -  high power pulse
;d1 : relaxation delay; 1-5 * T1
;d11: delay for disk I/O                              [30 msec]
;ns: 1 * n, total number of scans: NS * TD0
;cpd2: decoupling according to sequence defined by cpdprg2
;pcpd2: f2 channel - 90 degree pulse for decoupling sequence
```