

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 cryogenics.
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.

Automation Training Notes

1. Safety: 5 Gauss stray field (Earth magnetic field ~ 0.5 G) at about 1 m from the centre.

2. Sample preparations:

- Standard 5 mm NMR tube.
- Deuterated solvent.
- 600-700 μ l solution (^1H : 5 – 10 mg; ^{13}C : 20 – 50 mg).
- Clear solution, no insoluble particles (filtration).

3. Position your samples:

- Insert your NMR tube (cleaned) into the blue spinner at proper height (depth gauge).
- Check if carousel is busy. **Only if it's not**, press 'Add', choose 'Go to free' or choose 'Go to position'. Load your sample and remember the holder #.

4. Login: Change User \rightarrow Select/Type your NSID \rightarrow OK \rightarrow pw.

5. Setup experiments:

- Double click in the row of the **selected holder #**. (To free up the completed holders in IconNMR, click 'Holder' \rightarrow 'Delete Completed': this will not delete the data.)
- Edit **Name** (data directory) and **No.** (experiment #) as needed.
- Choose your **Solvent**, **Experiment**, edit **Par** and fill in **Title** if preferred.
- Adding more experiments for the same sample by click 'Add'.
- When ready, highlight the **holder row** (or all of your experiments) and click 'Submit'.
- Double check if your experiments are in queue (middle circle in *Yellow*).

6. Logout: Change User \rightarrow OK \rightarrow Cancel.

7. Spectra and Samples:

- **Experiments:** ^1H (1D, COSY, HSQC, HMBC), ^{13}C (UDEFT, CPD, DEPTq), ^{31}P (1D, CPD), etc.
- **Data:** Your data will be backed up into the SSSC Server 'FBR3' every 15mins. You can find your data in \\FBR3\spectra\fbr2\NSID. **Make your copy, PLEASE!**
- **Process:** PC for processing the spectra are available in G89. Login with your USask account.

ICON: Automation August 2016 0858 nmsu

File Run Holder View F10 Parameters Options Tools Help

Processing Skipped

Holder	Type	Status	Disk	Name	No.	Solvent	Experiment	Par	Title / Orig	Pri	Time	User
2			C:\Bruker\TOPSPIN2\1pl6	F7-143D-A-MA	1	CDCB3	PROTON128				00:26:01	nms027
3												
4												
5												
6												
7			C:\Bruker\TOPSPIN2\1pl6	Aug10-2016	1	CDCB3	chloroform-d	N PROTON			00:01:15	nmsu
8												
9												
10												
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20												

Submit Cancel Edit Delete Add 1 Copy 1

Change User

Preceding Experiments

#	Date	Holder	Name	No.	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title / Orig	Remarks
51	2016-08-09 13:01:49		W1209-18	1	PROTON								ch1763	C:\Bruker\TOPSPIN2\1pl6		
50	2016-08-09 15:45:11	20	F22-115H-CT	1	PROTON	✓	✓		✓	✓	✓		ch1763	C:\Bruker\TOPSPIN2\1pl6		
49	2016-08-09 15:37:37	19	F18-115H-CT	1	PROTON	✓	✓		✓	✓	✓		ch1763	C:\Bruker\TOPSPIN2\1pl6		
48	2016-08-09 15:32:39	60	ZH-33-59	4	COSYGPSW		✓				✓		zjh469	C:\Bruker\TOPSPIN2\1pl6		
47	2016-08-09 15:14:53	60	ZH-33-59	3	PROTON128	✓	✓		✓	✓	✓		zjh469	C:\Bruker\TOPSPIN2\1pl6		
46	2016-08-09 15:07:38	18	F11-16-115H-CT	1	PROTON	✓	✓		✓	✓	✓		ch1763	C:\Bruker\TOPSPIN2\1pl6		
45	2016-08-09 15:01:12	17	F6-10-115H-CT	1	PROTON	✓	✓		✓	✓	✓		ch1763	C:\Bruker\TOPSPIN2\1pl6		
44	2016-08-09 14:53:14	16	F2-3-115H-CT	1	PROTON	✓	✓		✓	✓	✓		ch1763	C:\Bruker\TOPSPIN2\1pl6		

Search Preceding ☐ include previous runs

Busy until: No Jobs! Day Experiments: 00:00 Night Experiments: 00:00 User: nmsu