

Weekly nitrogen fill

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Fills are performed weekly for all magnets. Without regular nitrogen fills the helium will boil-off rapidly causing the magnet to quench.

Typical fill schedule.

Day	Magnet(s)
Monday	Neo400, 400NB
Wednesday	Neo600, 500
Friday	B600

Prepare to fill

1. Start iLabs session.
2. Stop runs.

Magnets on manual mode:

- Verify an experiment is not running

Magnets on automation:

- **pause** automation in Icon.
- When all samples are complete, **stop** automation in Icon.

Fill

1. Move suitable liquid nitrogen dewar to magnet.
 - < 22 psi (vent for ~1 min. if pressure is high)
 - Install transfer line to the liquid value on the dewar (no ferreous tools within the 5 gauss line)
2. Place cover over magnet bore (**N/A: Magnets on automation**)
3. Lower magnet legs by releasing the nitrogen gas pressure. (**N/A: 400NB**)
4. Remove condenser from the nitrogen ports.
5. Install rubber tubes onto vent ports.
6. Flush transfer line with nitrogen.
7. Install transfer line to fill port.
8. Support the transfer line with a glove and slowly open value until liquid is flowing.
9. Adjust the liquid value on the dewar as nessecary to maintain flow rate.
10. When the magnet is full of nitrogen, close the liquid value on the dewar.
11. Replace condenser on the fill port.
12. Replace condenser(s) on the vent port(s).
13. Remove cover from magnet bore. (**N/A: Magnets on automation**)
14. Raise magnet legs by reconnecting the nitrogen gas. (**N/A: 400NB**)

Notes:

- Fills usually take 30-45 minutes.
- Stay nearby to monitor fill progress

Lineshape

1. Open TopShim
2. Inject lineshape sample (**1% CHCl₃ in Acetone**)

Magnets on manual mode:

- **ej** Eject sample.
- Check for air pressure
- load sample

- **ij** Inject sample.

Magnets on automation:

- load sample in sample changer and note position <#>.
- **sx** <#> Inject sample from position <#>.

3. **rsh** <probe> Read the shim file for the probe.

Probe	Magnet
bbfo	Neo400, 400NB, B600
bbo	500
cryoqnp	Neo600

4. Use the browser to locate the `\data\nmr\su` directory.

5. Right click the directory and **sort by date**

6. Drag the latest **lineshape-<DATE>** file into the main window.

7. **new** Create a new experiment.

8. Use the pop-up window to adjust experiment settings:

Field	Value
Filename	lineshape-<DATE>
Experiment number	1
Use current param	✓
Solvent	Acetone

9. **lock acetone** Lock magnetic field to solvent.

10. **atma** Automatically tune and match probehead.

- **Neo600**: **wobb** Manual tune and match.

11. **topshim gui** Shim magnet using graphical interface.

Field	Standard value	Neo400	Cryo or Neo probes
Dimension	1D	1D	1D
Optimization	Solvent default	Solvent default	Solvent default
Tune before	z-x-y-xz-yz-z	z-x-y-xz-yz-z	z-x-y-xz-yz-z
Tune after	z-x-y-xz-yz-z	off	z-x-y-xz-yz-z
parameters	N/A	convcomp	convcomp

- **Neo400**: perform tune after in topshim gui

Field	Neo400
Tune after	z-x-y-xz-yz-z
Only	✓

12. Close the topshim gui window.

13. **getprosol** Reads the probeheads and solvent dependent parameters.

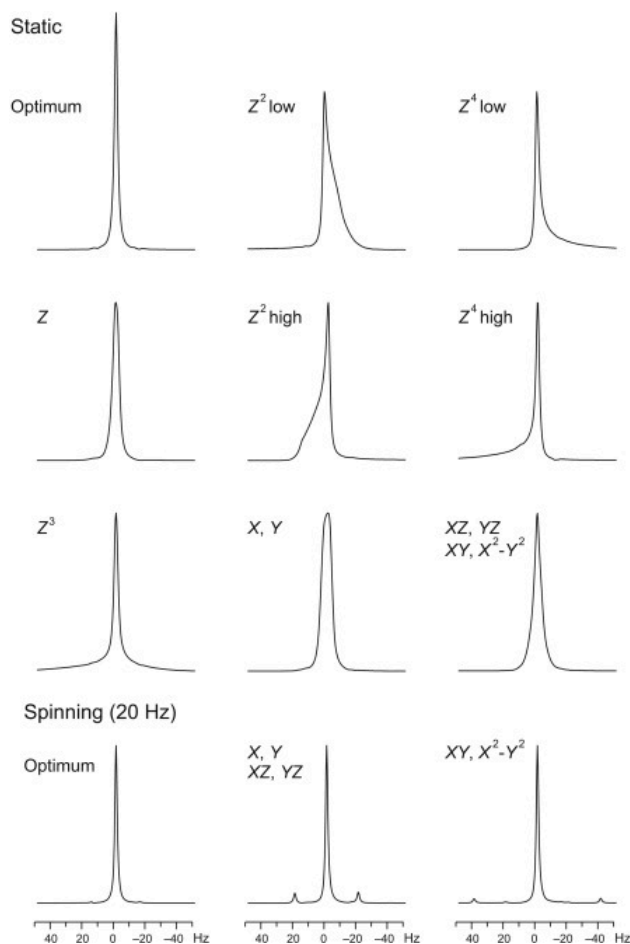
14. **rga** Optimize receiver gain.

15. **zg** Start acquiring raw data (**go zero**).

16. **fp** FT and phase correction.

17. **apk** Automatic phase correction.

18. Check spectrum for peak symmetry.



19. `humpcal` Perform hump test.

20. Use logbook to determine if hump test is within the normal operating window.

Pass hump test:

- Document hump test in logbook:

`<date> <initials> N2 fill <small#> <#> <large#> Hz`

Fail hump test:

- `wsh temp_<DATE>` Temporarily save the current shim settings in case you need to revert settings.
- `topshim gui` Rerun topshim.

Issue	Optimization
Peak wide at half-height	<code>lineshape width</code>
Peak wide at base (hump)	<code>lineshape hump</code>

Fail hump test multiple times:

- 3D shim using 10% H₂O in D₂O

21. `new` and copy experiment name.

22. `wsh <EXPERIMENT-NAME>`

Chloroform sample

1. Inject chloroform sample (1% Ethylbenzene in Chloroform)

Magnets on manual mode:

- `ej` Eject sample.
- Check for air pressure
- load sample
- `ij` Inject sample.

Magnets on automation:

- load sample in sample changer and note position `<#>`.
- `sx <#>` Inject sample from position `<#>`.

2. Use the browser to locate the `\data\numrsu` directory.

3. Right click the directory and `sort by date`

4. Drag the latest `CDCI3-<DATE>` file into the main window.

5. `new` Create a new experiment.

6. Use the pop-up window to adjust experiment settings:

Field	Value
Filename	CDCI3-<DATE>
Experiment number	1
Use current param	✓
Solvent	CDCI3

7. `lock cdc13` Lock magnetic field to solvent.

8. `atma` Automatically tune and match probehead.

- **Neo600:** `wobb` Manual tune and match.

9. `topshim` 1D shim.

10. `getprosol`; `rga`; `zg` Reads parameters; receiver gain; acquire data.

11. `fp`; `apk` FT; phase correction.

12. Check spectrum for peak symmetry.

13. Verify that the peak width at half-height is `< 0.8 Hz`

14. `wsh <probe>`

Probe	Magnet
bbfo	Neo400, 400NB, B600
bbo	500
cryoqnp	Neo600

15. Remove samples and resume normal operation.

Magnets on manual mode:

- `ej` Eject sample.
- Remove sample.
- `ij` Stop gas flow.

Magnets on automation:

- `sx ej` Eject sample.
- `start` Resume automation in Icon.

16. Finish ilabs session.