Variable temperature operation on 400 MHz NMR Verona

The regular blue plastic sample spinners can only be used near ambient temperature. For high or low temperature, use the white ceramic spinners instead.

* The SampleCase sample changer cannot handle the weight of the white spinners, and in particular, a very hot or cold spinner cannot be ejected into the sample changer.
* The sample changer should be turned off using the emergency stop with the magnet empty, so the “ej” and “ij” commands can be used for manual sample changes.
* The white spinners are brittle and expensive and likely to shatter if dropped on the floor.
* It can be somewhat difficult to start inserting an NMR tube into the white spinners; Bruker recommends that it works more easily if you twist the tube first and then transition into moving the tube up or down in the spinner.

High temperature experiments, up to 150 C, can be done with house air as normal. However nitrogen gas must be used for low temperature operation to avoid liquid oxygen condensing in the heat exchanger or water ice buildup inside the magnet. Make sure there is sufficient high pressure nitrogen gas in the tanks near the lab’s front door, then switch the instrument gas supply to nitrogen and wait for a few minutes to purge out the air lines.

The low temperature VT transfer line should be inserted into the 25 liter liquid nitrogen dewar, then the gas supply line should be unscrewed from the probe and connected to the back of the transfer line while the end of the line is connected to the probe. It does not take very much liquid nitrogen to do an experiment, and the dewar should not be completely full when you start because inserting the transfer line would cause liquid nitrogen to spill on the floor in that case. The dewar can be refilled during an experiment if you get a software message saying that the dewar is running low.

All of the controls for variable temperature operation are in the Topspin temperature panel which can be opened by double clicking on the temperature display in the bottom row of Topspin. When a set temperature below 0 C is turned on, the software automatically starts purge gas flowing through the probe and the shims. This protects the shims and the magnet from temperature extremes. The other main adjustment is the airflow. A flow of 400 liters per hour can be used at all times except when trying to get to very low temperature when it should be increased by the minimum amount (450 lph should give good regulation at -80 C).

The VT may regulate more smoothly if you let it “self tune” when the software says it’s out of tune.

At the end of the experiment, start bringing the temperature up towards ambient manually, then at about 0 degrees, quickly switch the gas line so that the low temperature line is bypassed. Then set the temperature back to 25 and allow the probe to warm up before switching back to house air. Remove the transfer line from the liquid nitrogen. It can be left on the floor until it defrosts, then it can be left on top of the console.

Operation of Bruker NEO 400 with iProbe

Capabilities: 1H/19F-31P-109Ag, 150 to -150 C

Samples can be loaded into verona in one of three ways:

* Under full automation, with ICON-NMR deciding when to change the sample. For this option, log in as an “additional user” under the nmrsu account, as on Florence.
* As a standalone user, using the manual controls on the SampleCase sample changer.
* As a standalone user, with the sample changer disabled with the emergency stop button. This option must be used for variable temperature operation, or for samples more than 8.5” tall which exceed the maximum height for the SampleCase.

Acquiring spectra as a standalone user can be done in two ways without using full automation:

* Start the “Routine Spectroscopy” dialog under ICON-NMR, which will prompt you for needed information and then run the sample automatically
* By manually carrying out each of the steps needed through the command line. The steps are the same on any modern NMR running any software. Here is a comparison of VnmrJ commands with Topspin commands:

Basic step Varian Bruker equivalent

Load parameters Click or type e.g. PROTON New dataset, rpar

Load pulse widths This step is implicit getprosol

Find lock Click find z0 Type lock, choose solvent

Gradient shim Tools -> standard calibration -> topshim [gui]

 Set up gradient shimming

Tune the probe Tools -> probe tuning -> auto atma

Find receiver gain Autogain box is checked in type rga

 acquisition window

Acquire data Click go type zg

There isn’t a save command as such in Topspin. Once you create a new dataset, and acquire data in it, that data remains there.

The default behavior for making a new dataset is that once you define a sample name, you will make numbered subfolders inside it. This may help organize your data on a single compound. Unfortunately, within Topspin, you can only use numbers for the subfolders instead of descriptive names. You should keep the numbers if you intend to return to the data within Topspin. However, if you copy the whole directory structure to your computer and only plan to process it in Mestrenova after that, you can rename the folders in your copy of the data and Mestrenova won’t care.