Operation of the BTN60 NMR Spectrometer
DL Olson; 21 May 2026

Overall Use

The spectrometer name on ChemFOM is the BTN60 (Benchtop Nanalysis 60-MHz).

- The location is A149 CLSL.

Operation of the spectrometer falls into three categories:

- NMR data acquisition
- T1 or T2 measurements
- Sample temperature control (range is +5 °C to +45°C)

Everyone who operates the instrument will need to know how to acquire NMR data. T1 and T2 measurements are an additional procedure, but begin with acquisition of an NMR spectrum. Temperature control can be added to any procedure, but is more involved.

Operational Notes

- Like all the other NMR spectrometers, use ChemFOM to activate the monitors.
- If you see something on the instrument or PC marked in red, please do not use or touch that area.
- You will need a USB stick to take NMR data from the spectrometer via the PC.
- There is no magnetic field hazard anywhere near the magnet. It is always kept on.
- On the magnet, if the LEDs are flashing blue, it's busy. If green, it's ready.
- The magnet tips the spins of protons at 62.69 MHz (1.472 Tesla).
- The default sample temperature is 32.4 °C.
- Please limit any lab work (wet chemistry, sample prep, etc.) to the desktop the magnet is on only.
- Where the computer monitors are located, desk work only please.

NMR Data Acquisition

Each instrument operation begins with an NMR spectrum. Here is the stepwise procedure.

- Initially, the instrument should already be:
    - Blue sample in the magnet (3% H_2O in D_2O).
    - Powered on and in Standby mode. It shims periodically in this mode.
    - Connected via the VNC on the PC
        - If not, on the PC, press the blue BTN60 Remote Connection icon, and select the BTN60 therein. It should connect, and the instrument control panel should appear.
        - Operation is always done from the PC

- To begin, press Standby in the upper left; Standby will turn off.
- Activate the Lock (press button in lower right).
- If the instrument was on Standby as described, you can proceed without more shimming.

- The only sample that can be shimmed is the blue sample (3% H_2O in D_2O). The instrument cannot shim on any other sample or solvent.
- If you want to shim more (only on the blue sample), press Status, and select an Autoshimming routine: 3, 6, 20, 40 minutes. Press Done when complete.

- **To Acquire NMR Data:**
  - Put sample in magnet
  - Declare solvent
  - Open Experimental Settings, make choices, press Done.
  - Check again that the instrument is locked.
    - Data will not acquire unless instrument is locked.
    - Lock needs to be re-established after every acquisition.
  - To initiate acquisition, press Go
    - Each transient updates the spectrum
  - If the spectrum seems satisfactory, press Done
    - Select Yes or No to save or not
    - When a file is Saved, keep in mind that periods are not allowed in file names. (This is unfortunate because if you want to include a number with a decimal, the file name will terminate at the decimal. You can use commas for decimal places.)
    - Type in a file name and comment as desired, and press Enter.
    - ALL the data sets are saved in the "spectra" file on the PC desktop.
      - Move each data file to your user directory and copy it to a USB stick to take it with you. Use the USB port under the curved monitor, not the PC itself.
      - Data not in a user directory could be deleted. Same with data left on the magnet itself (which will be deleted eventually by the NMR Staff).
      - Remove your USB stick when done.
    - Manipulation of the data is somewhat awkward using the spectrometer software. For an easier experience, use Mnova (on the PC desktop) instead. If you want to save an Mnova file, save it into your data directory in the "spectra" folder.

- **To Finish:**
  - Get back to the main control panel with the "Go" button.
  - Remove your sample.
  - Put the blue sample (3% H_2O in D_2O) in the magnet.
  - Press Standby, OK. The green peaks moving across the screen indicate periodic autoshimming.
  - Be sure to take your USB stick.

T1 Data Acquisition

T1 data acquisition is easy, but requires some care in choosing appropriate settings. Here, the T1 of H_2O is determined on the blue sample (3% H_2O in D_2O). The value is dependent on pH, temperature, and concentration of dissolved oxygen, but the value obtained will be typical.

- Please master this exercise before you attempt research samples.
- Overall, the goal is to get a good fit to the T1 curve, with little error in any region of the curve.

    - An example data set is named: BTN60_T1_1H_20260420_3%H2O
- Generally, the resultant T1 value (which appears in lower left of data plot) should:
    - Appear on the data plot in the region of ⅛ to ¼ of the x-axis time range. (Use the cursor to look; cursor position is upper right.)
    - Appear on the data plot in the upper 1/3 of the y-axis data range
- If you save the resultant T1 data set and import it into Mnova, the entire array of data sets will appear.
    - You can also analyze the data for T1 in Mnova.

- **To Acquire T1 Data:**
    - Watch this video: https://www.youtube.com/watch?v=riB3X6UjKBU
        - Several acquisition guidelines are given for successful T1 determination. These are summarized below.
    - First, acquire an NMR spectrum; this is used to set the autogain and select a peak on which to measure the T1 value.
        - Set Observe Nucleus, Lock Nucleus, Solvent, and Experiment correctly.
        - Adjust the Experimental Settings to yield a good spectrum.⅛
            - One transient will suffice if possible.
            - Press Done and Go to acquire. Phase as needed (Process, Phase).
            - Adjust Baseline as needed.
            - Press Analyze, Integrate. You may manually integrate if desired (Auto, Add, then **left click** on both the left and right integration limits of the peak of interest.
            - Press Done. Save if you want, but not necessary.
            - Change the experiment to T1 and go into Experiment Settings.
                - Set Scan Delay to 5-7 times the T1 value; you may need to guess, or just try 50 sec (which is likely plenty large), and corresponds to T1 = 10 sec.
                - Set Tau Stop to 4 times the T1 value (that is, 4/5 of the Scan Delay. Note that this value is in msec.
                - Press Done and Go to acquire. This will take some time, especially if NS is set to a larger value. But, with the Scan Delay = 50 sec, you will probably not need to do it twice.
                - If you want, use the resultant T1 value to compute again the Scan Delay () 5 X T1) and Tau Stop (4 X T1) values, and repeat the measurement.
                    - You have the option of determining T1 in Mnova.
                        - Scan Delay will appear in Mnova as Relaxation Delay.
                - Press Done. Save if you wish, or repeat. The Scan Delay and Tau Stop need to be big enough, but not so small as to affect the T1 determination.

Temperature Control

This would typically be employed for T1 studies. The approach requires some special equipment:

- Modified 8-inch, 5-mm NMR tube
    - Standard NMR 5-mm tube cap with 3-mm hole
- Sample is put into an 8-inch, 3-mm NMR tube.
- Filtration collar

- Nanalysis NMR sample tube holder
- FTS AirJet temperature controller and gas supply (dry air).
    - Allowed temperature range for NMR samples is +5 °C to +45 °C (-65 °C to +80 °C on the FTS).

- **Sample Apparatus Set-Up**
    - Newcomers need to be trained by an experienced user.
    - The apparatus for temperature control consists of:
        - One 8-inch, 5-mm OD NMR tube with two side holes about 2 cm from the top (done by SCS Glass Shop)
            - Same NMR tube has bottom ground off to create an opening
            - A cap for this tube with a 3-mm hole drilled in it (SCS Machine Shop)
            - An 8-inch, 3-mm OD NMR tube. (Note that the sample volume in a 3-mm NMR tube compared to a 5-mm tube is about 1/4 .)
        - One Nanalysis tube holder modified to have its "finger" openings sealed (1/2-inch to ¼-inch heat-shrink tubing)
        - One filtration collar
        - These three items are assembled in a particular manner and placed into the magnet in a way that seals the bottom of the tube from inlet air flow and forces the air
        - FTS AirJet temperature controller and gas supply (dry air).
            - Allowed temperature range for NMR samples is +5 °C to +45 °C
                - On the FTS, this is -65 °C to +80 °C
            - Air pressure set on the wall gauge to 1 bar (about 14.5 psi)
        - Custom made Teflon enclosure to contain the temperature-controlled air and direct it through the space between the inside of the 5-mm NMR tube and the outside of the 3-mm NMR tube to provide temperature control.
    - You will be given a demonstration on using the apparatus properly

- **FTS AirJet Operation for Temperature Control**
    - Open the gas valve on the wall first to supply air to the FTS
        - Pressure already set to 1 bar (about 14.5); adjust to 1 bar as needed
    - Power on by simply turning the power switch from Off to On (Breaker always On)
        - Only the fans turn on for about the first minute; remainder comes on soon thereafter.
    - Use the pencil erasure to set the temperature (green LEDs); use up or down arrow.
        - Press red Set button to set the temperature (red LEDs).
        - If the green digits are flickering, the temperature is not set to the green value.
    - You will be given a demonstration on using the FTS and sample apparatus properly

- **Supplies**
    - We recommend that groups get their own supplies
    - **For Standard Use:**
        - NMR Sample Tube Holders (for 5-mm OD tubes)
            - Order from Nanalysis
        - 5-mm OD, 7-inch Economy NMR Tubes (SCS Chem Storeroom)
    - **For Temperature Control:**

- NMR Sample Tube Holders (for 5-mm OD tubes)
  - Order from Nanalysis
- 5-mm OD, 8-inch NMR Tubes
  - Order via SCS Storeroom; VWR Scientific price = $20.36/5-pk (May 2026); no extra shipping cost via Storeroom; VWR part #
  - Or from Norell: https://shop.nmrtubes.com/ST550-8 but expect shipping cost
  - Get tube modified by SCS Glass Shop (two side holes, open bottom)
- 5-mm NMR tube caps, drilled with a hole to admit a 3-mm OD NMR tube
  - SCS Machine Shop
- 3-mm OD, 8-inch NMR Tubes
  - Order from Chemglass: $50.05/5-pk (May 2026)
  - Part #C-S-3-200-8
  - https://chemglass.com/norell-tubes-nmr-3mm-select-series-with-caps?sku=C-S-3-200-8
- Filtration collar
  - Used as a gas seal in temperature control work
  - SCS Storeroom item “Filter adapter neoprene size 2”