

NMR Spectroscopy using the Sample Changer

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Sample preparation

NMR Tube

- **Do not heat the NMR tube in an oven—the tube will warp and may cause probe damage**
- Clean the NMR tube with a detergent and dry with acetone and then nitrogen or air
- Tube caps are very cheap and disposable—dirty caps or old caps can contaminate your sample
- Do not rub tubes together—the glass will scratch and the tube will not shim
- Clean the NMR tube before bringing to the NMR lab
- Use only **EIGHT-INCH** tubes on the SMS sample changer!

Sample Preparation

- Use about 700 μL of deuterated solvent (5-mm tube)
- Filter the sample using glass wool or a kimwipe in the end of a pipette
 - Wash the glass wool with a small amount of deuterated solvent to remove glass particles
- The sample should be about 4.5 cm of liquid
 - Dilute samples do not benefit from a short liquid length
- Label your tube (on the cap or topmost 2 cm of the tube)

In the NMR lab

- Clean the tube with a *kimwipe* and isopropanol **prior** to putting into the spinner
- Put the tube carefully into the spinner so that it is inserted about 2 cm
- Put the spinner and tube into the depth gauge and position the tube to the bottom of the depth gauge
- Put the spinner with tube into the SMS changer at the position marked on the screen
- **Do not put the spinner (or anything else) under the robot fingers!**

Broken Samples

- **All broken samples must be reported!**
- Do not use a spinner that is dirty or has broken glass
- If the spinner is dirty, the inside must be cleaned with a cotton swab to ensure that all broken glass is removed
- Do not wipe the black line or white dots with any solvents

Safety

- Persons with pacemakers or other life sustaining implanted electronics must never enter the NMR laboratory
- Modern orthopedic implants are usually non-magnetic and should be safe near magnets. However, users must check with their physician **before** entering the NMR lab
- Magnetic objects (dewars, chairs, tools) must be kept away from the magnets—**severe damage to persons or magnets may occur if a metallic object is brought near the magnet**
- Floors are marked with blue and red dots or lines to indicate magnetic field—all metallic objects should be outside those lines
- Gloves should not be worn in corridors or in the NMR labs when handling the spinner or using the keyboard
- **Do not use the passenger elevator to carry NMR samples**

Walkup Operator Login and Logout

The Walkup operator logs in using the VnmrJ Walkup login screen. If Automation is active, the login screen shows the next available sample position.



Single-Sample Mode



Automation Mode



Next available
sample position

Operator list
Password field

Log In

1. Select an operator from the drop-down menu or enter an operator name in the field.
2. Enter the password for the operator.
3. Click OK or press Enter.

Log Out

1. Click **Utilities** menu.
2. Select **Switch Operators**. The login in screen appears.

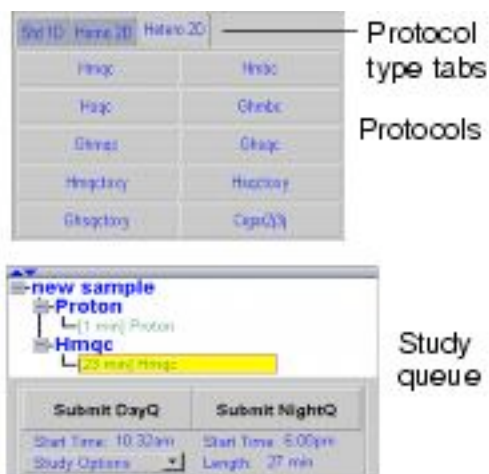
Starting an Experiment

Running an experiment requires the following:

- Defaults are set up by the VnmrJ Walkup account owner and will be used by each operator. Preset defaults include shimming, locking, saving data, and plotting but are not limited to these defaults.
- The VnmrJ Walkup Operator interface is active and the login screen is currently displayed.
- A new automation run is started.
- The sample tube is filled to the correct height (approximately 750 ml).
- The sample tube must be **eight inches high**.

Automation Mode

1. Place the sample in the spinner and adjust the position of the sample in the spinner. IMPORTANT: YOUR NMR TUBE SHOULD BE EIGHT INCHES IN LENGTH.
2. Place the sample and spinner in the location shown on the log in screen (or another available location in green, shown after logging in).
3. **Login.**
4. Click on a **gray sample position** - available sample positions are gray. Green sample positions can also be used..
5. The number displayed on the login window is the next available position but any grayed position can be used. Samples are run in the order in which they are submitted to the queue. The location of the sample in the sample changer does not determine when a sample will be run. The status of occupied sample positions is indicated by the color displayed at the position. Dimmed colors indicate the sample is owned or belongs to another operator, not the current operator.
6. An example is shown here with a SMS 50 sample tray.



Item	Description
<p>Available sample positions. The colors of the sample position indicate the status of that sample position as follows:</p>	
Gray	Available
Green	Completed

Item	Description
Blue(Cyan)	Active
Yellow	Submitted - Day queue
Purple	Submitted - Night queue
Red	Error
Faded or grayed out colored sample position	Sample submitted by a operate other than the operator that is currently logged in to the walkup interface.

7. Select the sample lock solvent from the drop down **Solvent** menu.
8. Place checks in either of the following options: **Find z0** and **Gradient Shim** to enable these options.
9. Place a check in the **Plot all data** to enable this option upon completion of the study or fid acquisition.
10. Optional - Fill in the information in the **Sample**, **Notebook**, and **Page** fields. Your FID will be saved in the format YYYYMMDD-sample-experiment-00n. Use only alphanumeric characters in the sample field; others (like "(") will cause your sample to be skipped and not run.
11. If E-mail is enabled, either or both of the **Email** options for notification upon completion.
12. Select a **Protocol Type** tab
13. Select a **protocol** or series of **protocols** from the display of protocols by single clicking on each desired protocol (or drag and drop the protocol into the study queue).
14. The study protocol for the current operators samples appears in the study queue window. Each time a protocol is clicked it is added to the study queue list.
15. To remove a protocol from the Study Queue, refer to "[Removing a Protocol from the Study Queue](#)".
16. Adjust acquisition parameters as necessary as described in "[Customizing Acquisition Parameters](#)".
17. Click on either the **DayQ** or **NightQ** to submit the study to the queue. The approximate time your sample will be run is given under the DayQ and NightQ button.
18. The study can be submitted to either the DayQ or NightQ provided the time allocated for data acquisition during either the DayQ or NightQ is longer then total experiment time. If either the DayQ or NightQ button is grayed out, the total experiment time exceeds the time allocation for that queue.
19. Add the next sample to the automation run.



Figure 1. Removing a Sample from the Study Queue

20. Repeat the steps beginning at [step 4](#) through [step 13](#) for each additional sample.
21. Log out as described in "[Walkup Operator Login and Logout](#)".

Customizing Acquisition Parameters

Customizing acquisition parameters *must be done before the experiment is submitted to the queue.*

1. Double click with the left mouse button on the time field for the experiment in the protocol - the text will change bold when it is selected.
2. Click on the **Acquire** tab.
3. Make adjustments to the acquisition as required.
4. Repeat the preceding steps for each experiment in the protocol.
5. If the sample is submitted and changes in the acquisition parameters are required, delete the sample from the queue, see "[Removing a Sample from the Queue](#)".

Removing a Protocol from the Study Queue

Must be done before the experiment is submitted to the queue.

Clicking on the **Study Options** drop down menu button
Select **New Sample**.

This will remove all the protocols for the sample and clear the study queue. A new study protocol can be created.

Drag an individual protocol for a sample in to the trash can.

Removing a Sample from the Queue

The only users that can remove samples from the queue are the owner of the sample (the VnmrJ Walkup operator that submitted the sample to the queue) and the VnmrJ Walkup account owner.

Individual parts of an experimental protocol for a sample can not be removed, that *entire sample must be removed from the queue*, a new experimental protocol list created and the sample re-submitted to the queue.

Remove a sample from the queue as follows:

1. Click on sample - it will become high lighted in yellow.
2. Hold the left mouse button down.
3. Drag the sample to the trash can and release the mouse button, see [Figure 1](#).

Due to a VnmrJ bug, the location may not appear grey until another sample is submitted.

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