

## Scheduling and Accounting

Please check and sign the reservation sheet provided. Instrument usage is first-come first-served, open-access, walkup mode. Accounting will be done electronically, based upon number of samples, log time and acquisition time.

### 1) CIF Main Lab NMR General Login info

NMR:	<u>Inova 300</u>	<u>Inova 400</u>	<u>Robot 400</u>
Username:	<i>(your EID)</i>	<i>(your EID)</i>	<i>(your EID)</i>
Password:	<i>nmr4CSU</i>	<i>nmr4CSU</i>	<i>nmr4CSU</i>



Operator login

### 1) Login

- If an operator is already logged in, please click the Logout button.
- At the Walkup login, enter your operator name or select from the pull-down.
- Click on password field, enter your password (see above if you did not change) and click OK.
- Please note that you may change your password if desired.

### 4) Sample Preparation

Use a deuterated solvent whenever possible (ask). You need about 0.650 ml total solvent volume or 3.5 cm in the NMR tube. The NMR is sensitive and good proton can be obtained on 5mg. You may want 10mg or more to get 13C in a reasonable time. You will have poor results if you try to use to short a column (less than 3cm).

### 5) Check and Gauge Sample:

Before putting your sample tube in the spinner, clean the tube and insert it into the glass gauge. If the tube is warped, it will not insert fully into the gauge, and must not be used, or else it will damage the probe.

If the tube is ok, place the spinner into the metal depth gauge, and insert the tube into the spinner. Center the sample volume around the dotted lines on the gauge.

Using a Kimwipe with a drop of ethanol, carefully wipe the sample tube and spinner. Be sure you are inserting a sample with a deuterated solvent (unless you have cleared otherwise with staff).

### 6) Sample Insertion

#### a) Important: The eject air must be ON before you insert the sample!

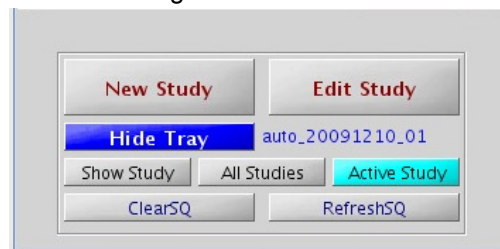
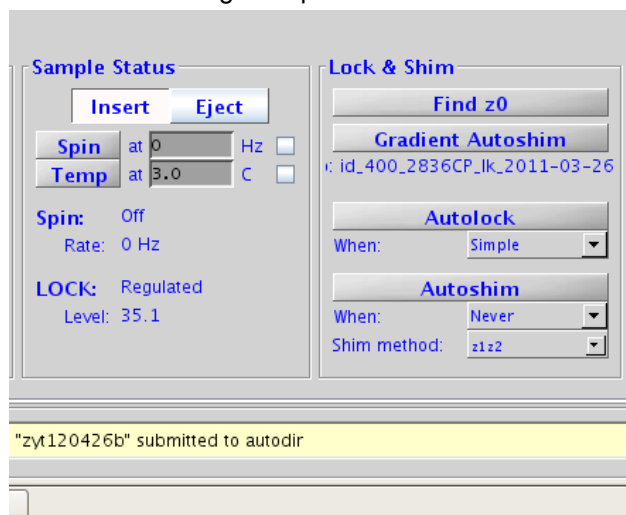
- Caution: The magnet produces a very powerful magnetic field. Observe all posted precautions when approaching the magnet. Do not approach the magnet if you have a pacemaker, other medical implant, or any metal objects in your body. Remove from your pocket and put in a safe place any magnetic media, such as computer disks, or magnetic strip ID cards or credit cards. Some watches may also be damaged. Do not bring any ferromagnetic objects near the magnet. If in doubt, please ask any of the CIF staff. Read the attached Magnetic Resonance (MR) Safety documents. The 5 Gauss limit is marked by a blue circle on the floor.
- In the Sample Status window click on Eject to turn on the eject airflow.
- Wait for the eject air to come on (it can be heard and felt rushing out the top tube of the NMR), and then insert the spinner with the sample into the NMR.
- Now enter **i** to insert and turn off the airflow. The sample will descend into the magnet.

### 8) Sample Status

- Leave Spin at 0 Hz and Off for the I400
- Leave Spin at 20 Hz and On for the I300

### 9) Study Queue

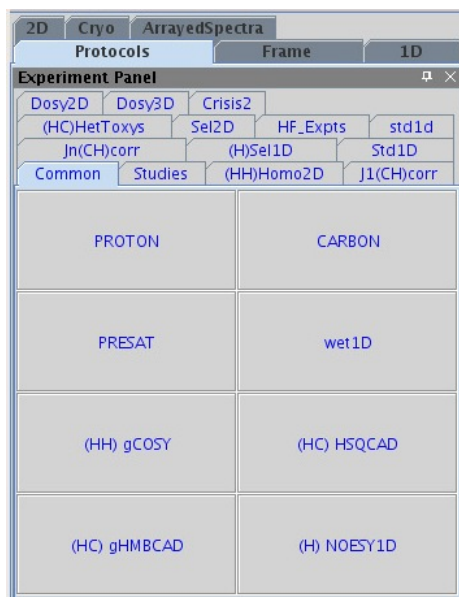
Ignore the data in the graphic window since it is not from your sample. You **must** click on **New Study**, in *New Study Queue panel*. If New Study is not available, click on Done first, then the New Study button should appear.



New Study Queue panel

**For each sample that you have:**

- Click on one or more desired protocols from those available, (e.g. Proton, Carbon and/or protocols on other tabs).
- If you select the wrong protocol, simply drag it from the study queue into the trash.



**Protocols tab panel**

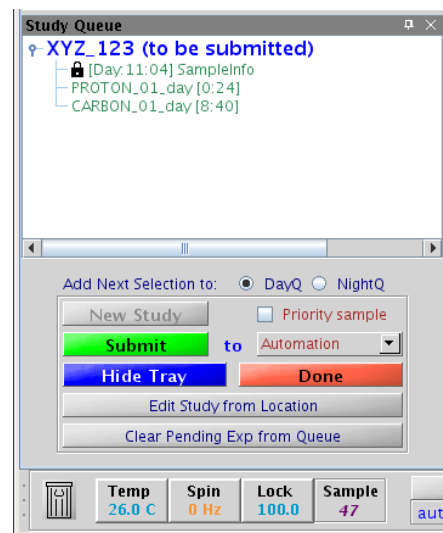
Enter sample info  
Enter sample name,  
select solvent,

Verify/enter correct  
email in Eaddr box.

**Submit Study Queue:** Click Submit.  
Wait for acquisition to complete.



**Enter sample info panel**



**Submit Study Queue panel**

**10) Retrieve your sample**

- When your sample is completed
- Click Eject to eject sample
- Remove sample from magnet, return spinner to tray.
- Click Logout button on *Enter sample info panel*.

**11) Data**

- Your data will automatically be saved
- A pdf survey spectrum will be emailed to you for each experiment.
- You will also receive data (FID) email for each one.
- If there has been an error, you will receive an error email.
- Please do not do data processing at the console.

**Data Retrieval**

- You will need to use an ftp program that uses SSH/SFTP security and be connected to the Chemistry Firewall. (Non-encrypted FTP will NOT work.)

NMR	IP Address	Username	Password
I300	129.82.76.81	i300	nmr4CSU
I400	129.82.76.87	i400	nmr4CSU
400MR	129.82.76.122	400mr	nmr4CSU

- Secure ftp for Win PC
  - CoreFTP LE v. 2.1 will work. It is free and can be found at <http://www.coreftp.com/download.html> Select the free version.
  - After installation, click on Sites – Site Manager
  - Example configuration for I400:
    - Enter the Site Name: CIF I400 URL: 129.82.76.87
    - Username: i400 Password: nmr4CSU
    - Be sure to check the box “Don’t save password”
    - Under Connection, select SSH/SFTP
    - To connect, click on “Connect”
- If you have a Mac, use Fetch version 5.5 or later.
- Your data will be found in `/home/_data/walkup/your-operator-name/`