BASIC OPERATION OF THE MERCURY 400

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1. INTRODUCTION

1.1. About these Notes and Related Notes

These notes describe the use of the Mercury 400 spectrometer to perform basic 1D and 2D NMR experiments. Several other worksheets are available and should be consulted in addition to this worksheet.

Table 1. Required and Recommended Notes for Performing NMR Experiments on the Mercury 400

If you would like to…	**Then you …**	
	…must consult these notes	**…should consult these notes**
Perform a 1D ^{1}H, ^{13}C ^{19}F, or ^{31}P experiment	• NMR Sample Preparation • Spectrometer Capabilities and Specifications • Retrieving NMR Data	• Processing 1D NMR Spectra using VnmrJ
Perform a 2D experiment	• NMR Sample Preparation • Spectrometer Capabilities and Specifications • Retrieving NMR Data	• Processing 2D NMR Spectra using VnmrJ

1.2. About the Mercury 400

The Mercury 400 has a Varian SMS Autosampler (robot) that permits the spectrometer to be used in a fully automated manner, making it ideal for beginners. Samples are submitted to a "queue" and the samples are run in the order in which they were submitted. For samples submitted during peak NMR hours, which are between 9:00 am and 6:00 pm on weekdays, the selected experiments must be shorter than 30 minutes in length, otherwise the experiment will be submitted to the NightQ, and will begin at 6:00 pm.

1.3. Capabilities of the Mercury 400

A brief summary of the capabilities of the Mercury 400, in comparison to the capabilities of the Inova 400 and Inova 600 are presented here. For a full discussion, please see the "Spectrometer Capabilities and Specifications" sheet.

Table 1. Capabilities of the Mercury 400, Inova 400 and Inova 600

Spectrometer	**^{1}H**	**^{13}C**	**^{19}F**	**^{31}P**	**Other nuclei**	**2D**	**VT**
Mercury 400	Yes	Yes	Yes	Yes	No	Yes	No
Inova 400	Yes	Yes★	Yes	Yes★	Yes	Yes	-80 °C to +130 °C[a]
Inova 600	Yes★	Yes	No	Yes[b]	Yes[b]	Yes	-20 °C to +80 °C

★Preferred spectrometer
[a]Requires the use of liquid nitrogen for cooling.
[b]Requires re-cabling of the probe.

1.4. Specific Mercury Policies

1) Samples must be clearly labeled with your supervisor's initials using a permanent marker on the cap itself, not the NMR tube. An additional sample label must be on the side of the cap.
2) Samples should be picked up within 48 hours of completion of the NMR experiment(s).
3) Do not move spinners from slot-to-slot, i.e. the spinner from slot 1 should not be placed in slot 3 and vice versa.
4) Do not place spinners or NMR tubes in slot 50 or the slots labeled "NO SPINNER HERE".

1.5. Training

In order to use the Mercury 400, all users must complete an NMR Facility Safety Sheet and receive spectrometer training from an NMR facility staff member. Training is regularly offered in September, January and May, but also upon request by contacting an NMR facility staff member.

1.6. Sample Preparation

Follow the directions outlined in the "NMR Sample Preparation" sheet.

2. LOGGING IN AND AN OVERVIEW OF VnmrJ

2.1. Logging In

1) Choose your group name from the drop-down list (see Figure 1).
2) Enter your group's password.
3) Press the "OK" button or hit the "Enter" key.

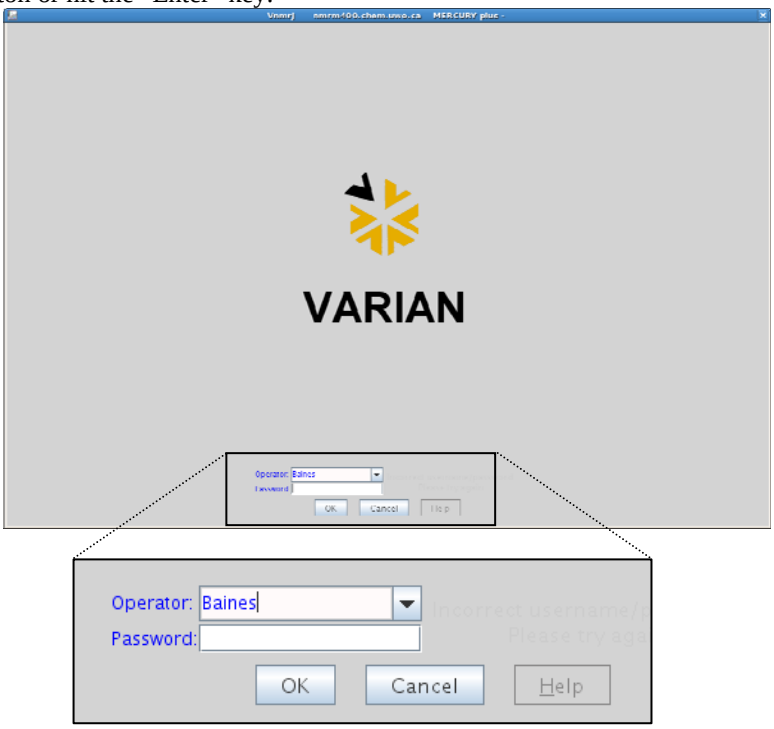

Figure 1. The Mercury 400 log-in screen. Select your supervisor's name from the drop-down list and enter the appropriate password to log-in.

2.2. Overview of the VnmrJ Software

1) The screen displayed upon logging into the Mercury 400 is displayed in Figure 2. The following sections and buttons are the most important:
   a) Experiment Panel: *Panel containing all possible NMR experiments. Used to select your experiment.*
   b) Study Queue Window: *Panel displaying all selected experiments and the time required for each experiment.*
   c) Sample Queue View: *Drop-down list that controls what details are displayed in the Experiment List window.*
   d) Sample Submission Window: *Contains buttons that initiates the study queue. The study queue allows the user to perform multiple experiments on the same sample in an automated fashion.*
   e) Queue check window: *Contains buttons used to check the sample queue.*
   f) Status Indicators: *Windows that display the spin-rate (in Hz), lock level and sample changer number of the sample in the magnet. The values will be blue when regulated, purple when trying to regulate, and orange when not-regulated (or off).*
   g) Spectrometer Status Window: *Indicates whether the spectrometer is idle or performing a task. Turns blue when an experiment is running and indicates the time remaining in the experiment.*
   h) Information Panel: *Displays a variety of useful messages and also error messages (if any).*
   i) "Action" Buttons: *A few of the most commonly used buttons. The buttons change depending on whether you are submitting a sample, modifying the acquisition parameters or processing your data.*

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j) Panels: *Panels containing pages that are used to operate the spectrometer. There are two main panels;* ***Start*** *(which contains the pages to set the conditions before the experiment can begin), and* ***Process*** *(which contains pages that set all the processing parameters and plots your spectrum).*

k) Graphics Display: *Window displaying your spectrum or FID.*

l) Graphics Controls: *A variety of buttons that control what is displayed in the graphics display and the appearance of your spectrum or FID in the graphics display. Allows you to zoom-in and out, perform integration, etc.*

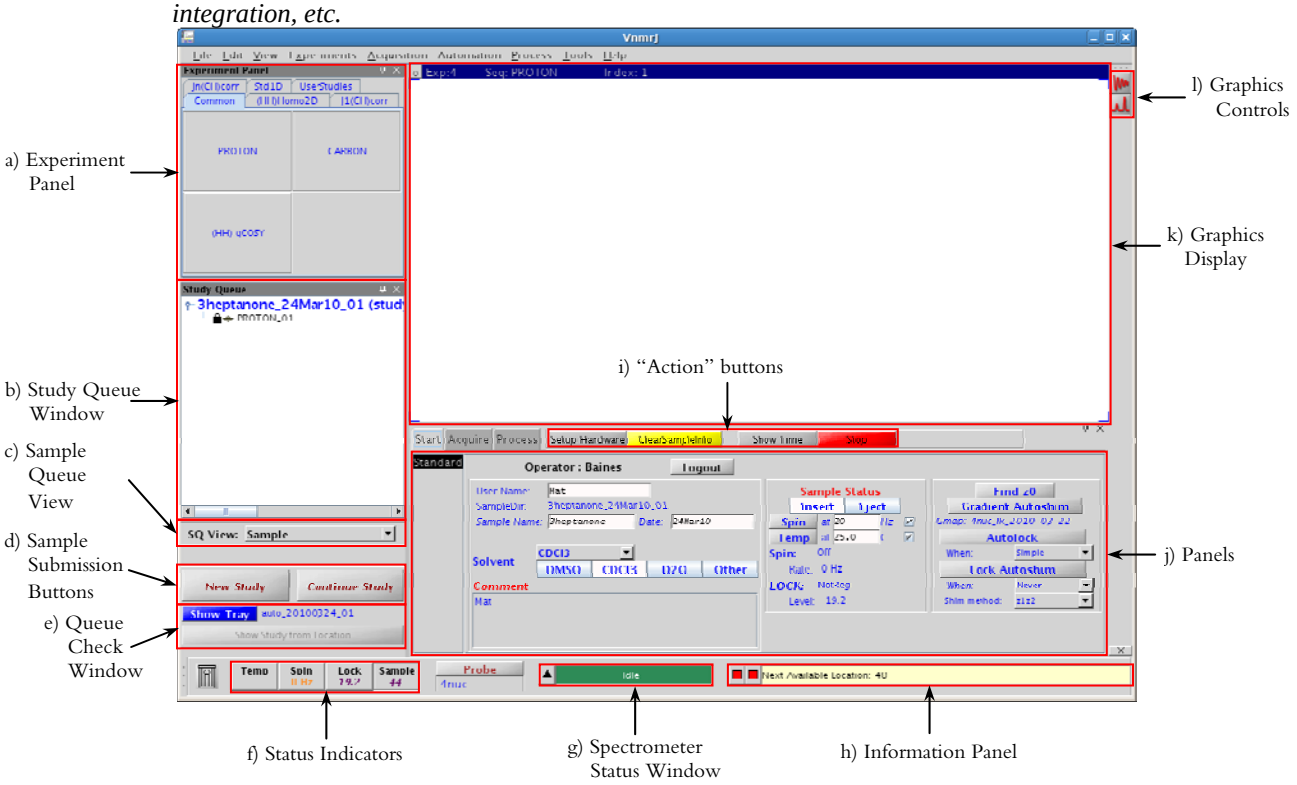

Figure 2. The VNMRJ screen displayed after log-in. The most important buttons and panels are labeled.

3. CHECKING THE QUEUE *(OPTIONAL)*

If desired, you may check the queue to determine how many samples are ahead of you and to determine when your experiments will be performed. There are 2 ways in which you may check the queue: Method 1 shows you how many samples are queued and Method 2 displays the time remaining to complete the experiments on the queued samples. The relevant buttons are shown in Figure 3.

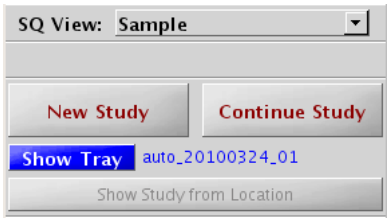

Figure 3. A close-up showing the Show Tray button and SQ View drop-down list that may be used to check the sample queue.

3.1. Method 1: Using the Study Tray

1) Click on Show Tray, which is found on the bottom left-hand side of the screen. The screen shown in Figure 4 will appear.

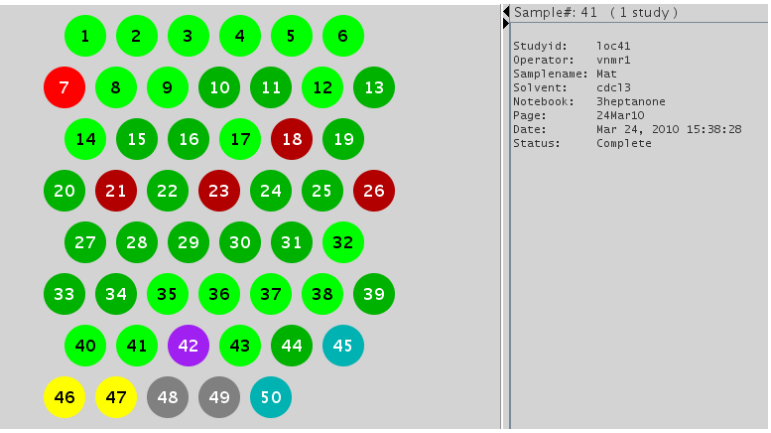

Figure 4. The Study Tray, which shows all samples submitted to the study queue. By clicking on a particular location number, the sample information for that location number will be displayed on the right-hand side of the screen.

2) The study tray displays all 50 spinner locations and each location is colour-coded:
   - 10 = completed with no error
   - 18 = completed with an error
   - 42 = queued nightQ sample; will begin after 6:00 pm and after the dayQ samples are complete
   - 46 = active sample
   - 47 = queued dayQ sample
   - 49 = location not yet used
3) In this example there are 2 samples in the dayQ and 1 sample in the nightQ. Unfortunately this method does not provide the time in which your sample will be submitted to the queue, only the number of samples ahead of you in the queue.

3.2. Method 2: Using the Drop-Down List

1) If you would like to see the time required to complete the experiments on the queued samples, select "Spectrometer" from the drop-down list on the left-hand side of the screen. Information similar to Figure 5 will be displayed in the Study Queue Window on the left-hand side of the screen:
   a) Active Study: *Refers to the sample currently in the spectrometer.*
   b) Completed Studies: *Refers to samples that have been completed.*
   c) Errored Studies: *Refers to samples that have completed but with some error.*
   d) Studies in day queue: *Refers to the samples currently in the day queue.*
   e) Studies in night queue: *Refers to the samples currently in the night queue.*
2) Click on the node next to "Studies in day queue" to show all samples in the day queue and the estimated time that each sample will start. The same can be done for "Studies in night queue".

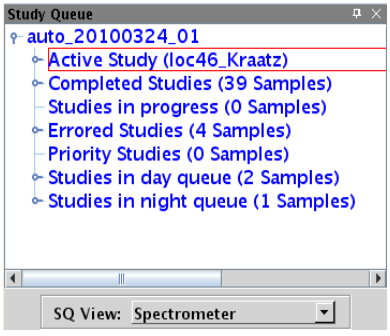

Figure 5. The information displayed in the Experiment List after selecting "Spectrometer" from the drop-down list on the left-hand side of the screen. By clicking on the appropriate node, information about each sample will be displayed.

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4. SUBMITTING SAMPLES TO THE QUEUE

1) Press New Study, which is found on the bottom left-hand side of the screen. The screen pictured in Figure 6 will appear.

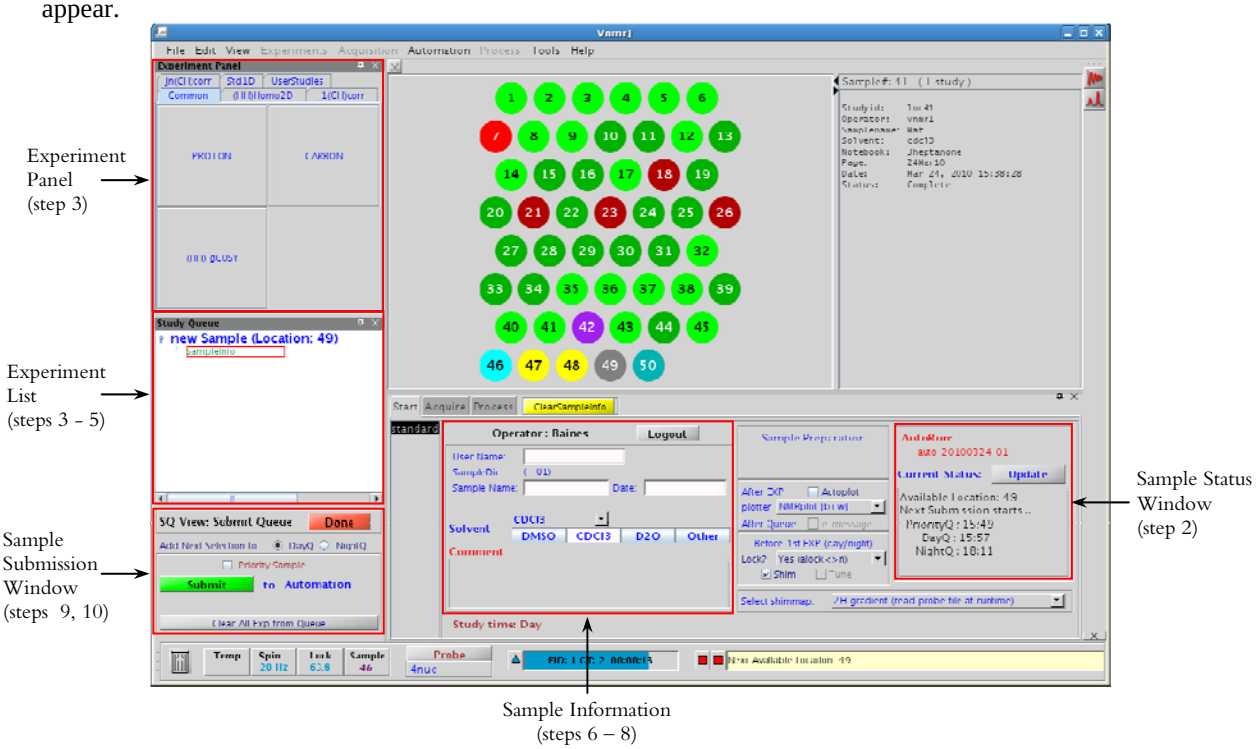

Figure 6. The VNMRJ screen displayed after hitting the "New Study" button. The outlined windows contain information or buttons required to submit your sample. The step number corresponds to the step listed below in which the outlined window is used.

2) Insert your sample into the correct slot.
   a) The slot number for your sample is displayed in the Sample Status Window, and is dictated by "Available Location: #", where # is the correct slot for your sample (see Figure 7). The slot number will also be displayed in the information panel, which is on the very bottom of the screen, and in the Study Queue Window.
   b) Walk up to the spectrometer, up the ladder, and remove the spinner from the appropriate slot.
   c) If there is a sample in the spinner, remove it and place it in the tray labeled "Current Week".
   d) Place the empty spinner into the brass depth gauge.
   e) Insert your NMR tube into the spinner and push down until the tube until it hits the bottom of the depth gauge.
   f) Remove your NMR tube and spinner and wipe the bottom of the NMR tube clean with a Kim-wipe.
   g) Place the NMR tube and spinner into the correct slot.

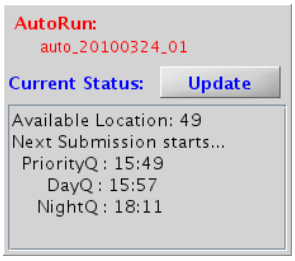

Figure 7. A close-up of the Sample Status Window used in step 2. The estimated time that your experiment will start and slot number for your sample are displayed in this window. Separate times are displayed depending on whether you will submit to the DayQ or the NightQ. Note: The PriorityQ is disabled for all users.

3) Select your experiments.
   a) The experiments are listed in the "Experiment Panel" found on the left-hand side of the screen (see Figure 6).
   b) Click the experiments you would like performed, in the order you would like the experiments to be performed. Each tab contains several experiments of that experiment type (see Figure 8 for more information).
   c) The chosen experiments will be listed below the "Experiment Panel". The "Experiment List" displays the length of each experiment, whether the experiment is part of the DayQ or the NightQ, and the total time required to complete all experiments. Experiments longer than 30 minutes long are automatically added to the NightQ.

Figure 8. A close-up of the Experiment Panel used in step 3 and Experiment List used in steps 4 - 5. In addition to the common tab, the (HH)Homo2D tab contains homonuclear ^{1}H–^{1}H 2D experiments such as gCOSY, TOCSY, NOESY, the J1(CH)corr tab contains 1-bond ^{13}C–^{1}H 2D correlation experiments such as gHSQC and gHMQC, the Jn(CH)corr tab contains multiple-bond ^{13}C–^{1}H 2D correlation experiments such as gHMBC, and the Std1D tab contains ^{19}F and ^{31}P 1D experiments. The UserStudies tab is not currently used. As experiments are selected, they will appear in the experiment list along with the time required for each experiment and the total time for the sample will be updated.

4) ***(Optional)*** Customize your experimental parameters.
   a) Double-click on the experiment in the list of chosen experiments (not the Experiment Panel). This reads in the experiment.
   b) In the main panel, click on the "Acquire" tab, a screen similar to what's displayed in **Error! Reference source not found.** will appear.
   c) Modify any parameters that you wish.
   d) If desired, click Show Time. The experiment time will be updated and displayed in the experiment list.

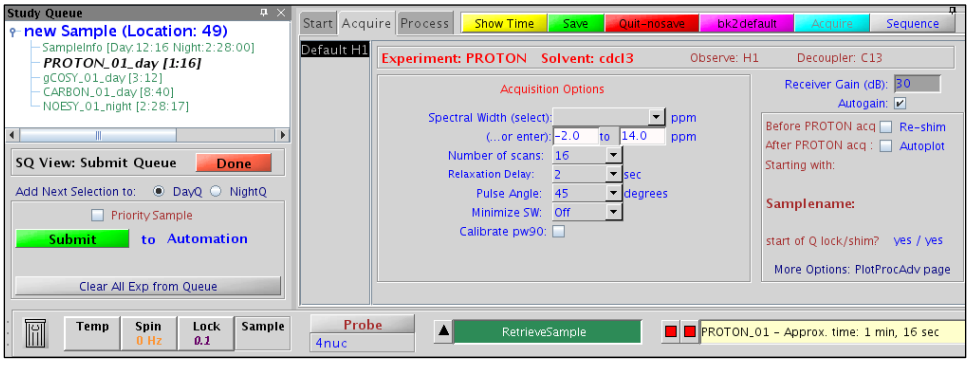

Figure 9. The screen displayed after double-clicking on the PROTON_01 label in the Experiment List, changing the number of scans to 16 (from 8) and the relaxation delay to 2 (from 1), and clicking on the "Show Time" button. Note that the exact parameters that can be modified depend on the chosen experiment.

e) Then choose one of the following options:

i) Save to save the modified parameters and return to the previous screen.
ii) Quit-nosave to cancel the modified parameters and return to the previous screen.
iii) bk2default to reset the modified parameters back to the default parameters. You must then hit "Save" or "Quit-nosave" to return to the previous screen.

5) ***(Optional)*** Delete unwanted experiments by selecting the experiment from the Experiment List and dragging it into  on the bottom left corner of the screen. The total sample time will be updated.
6) Fill out the information needed to save your data (see Figure 10). Your data will be saved in the folder "/home/data/group name/UserName/SampleName_Date"
   a) Complete the "User Name:" section.
   b) Complete the "Sample Name:" section.
   c) Complete the "Date:" section.
7) Select your solvent from the drop-down list (see Figure 10).
8) ***(Optional)*** Enter any information in the "Comment" section. This section is free-form and can contain any information you want (see Figure 10).

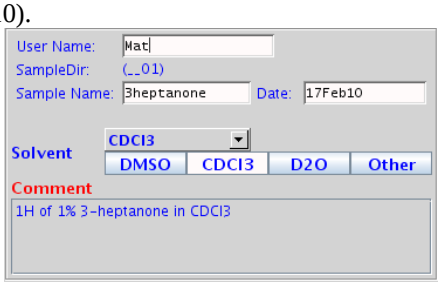

Figure 10. The Sample Information Window used in steps 6 – 8. In this window, the information needed to save the experimental data is entered, the solvent is chosen and any desired comments about the sample are entered.

9) Click on Submit to submit your sample (see Figure 11). The message, "Sample will be submitted to location ##" should appear and the location number in the Study Tray Window should turn yellow if submitted to the DayQ and purple if submitted to the NightQ.

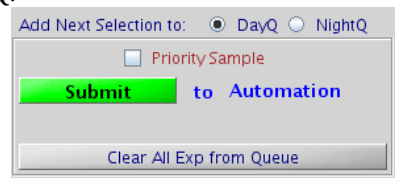

Figure 11. The Sample Submission Window used in step 9 - 10. The Submit button is used to send your sample to the queue, while the Clear All Exp from Queue button may be used when submitting additional samples.

10) If you have additional samples to submit, and:
   a) The same experiment(s) will be performed:
      i) Click on the ClearSampleInfo action button. This clears the User Name, Sample Name, Date, and Comments.
      ii) Put your sample in the appropriate location within the Sample Tray (see step 2).
      iii) Repeat steps 6 – 9.
   b) Different experiments will be performed:
      i) Click on Clear All Exp from Queue (see Figure 11), which clears all fields.
      ii) Repeat steps 2 – 9 for each sample.
11) When you have no more samples to submit:
   a) Click on Logout, which is in the middle bottom of the screen, next to the text displaying the current Operator.

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5. RE-SUBMITTING YOUR SAMPLE *(OPTIONAL)*

There are several instances in which you may want to re-submit your sample to queue. The most common instances are that your sample has reported an error, you want to perform additional experiments on your sample or you want to re-run an experiment but with modified parameters.

1) Log-into your group's account.
2) If the study tray is not displayed, click on Show Tray, which is found on the bottom left-hand side of the screen.
3) Find your sample in the study tray and click on the location.
4) In the top main menu, select **Automation → Tray Actions.. → Recall & resubmit Study from a location**.
5) Put your sample in the appropriate location within the Sample Tray (see step 2 of Section 4 for more information).
6) Make any desired changes to the experiments and experimental parameters.
7) Click on Submit to submit your sample.

6. VIEWING YOUR SPECTRUM *(OPTIONAL)*

1) Log-into your group's account.
2) If the study tray is not displayed, click on Show Tray, which is found on the bottom left-hand side of the screen.
3) Find your sample in the study tray and click on the location.
4) Click on Show Study from Location, which will read in the completed study. All experiments completed for that sample will be displayed in the experiment list.
5) Double-click on the experiment that you would like to see. The spectrum is automatically processed and displayed.
6) If you would like to analyze or re-process the spectrum, please see "Processing 1D NMR Spectra using VNMRJ" and/or "Processing 2D NMR Spectra using VNMRJ".