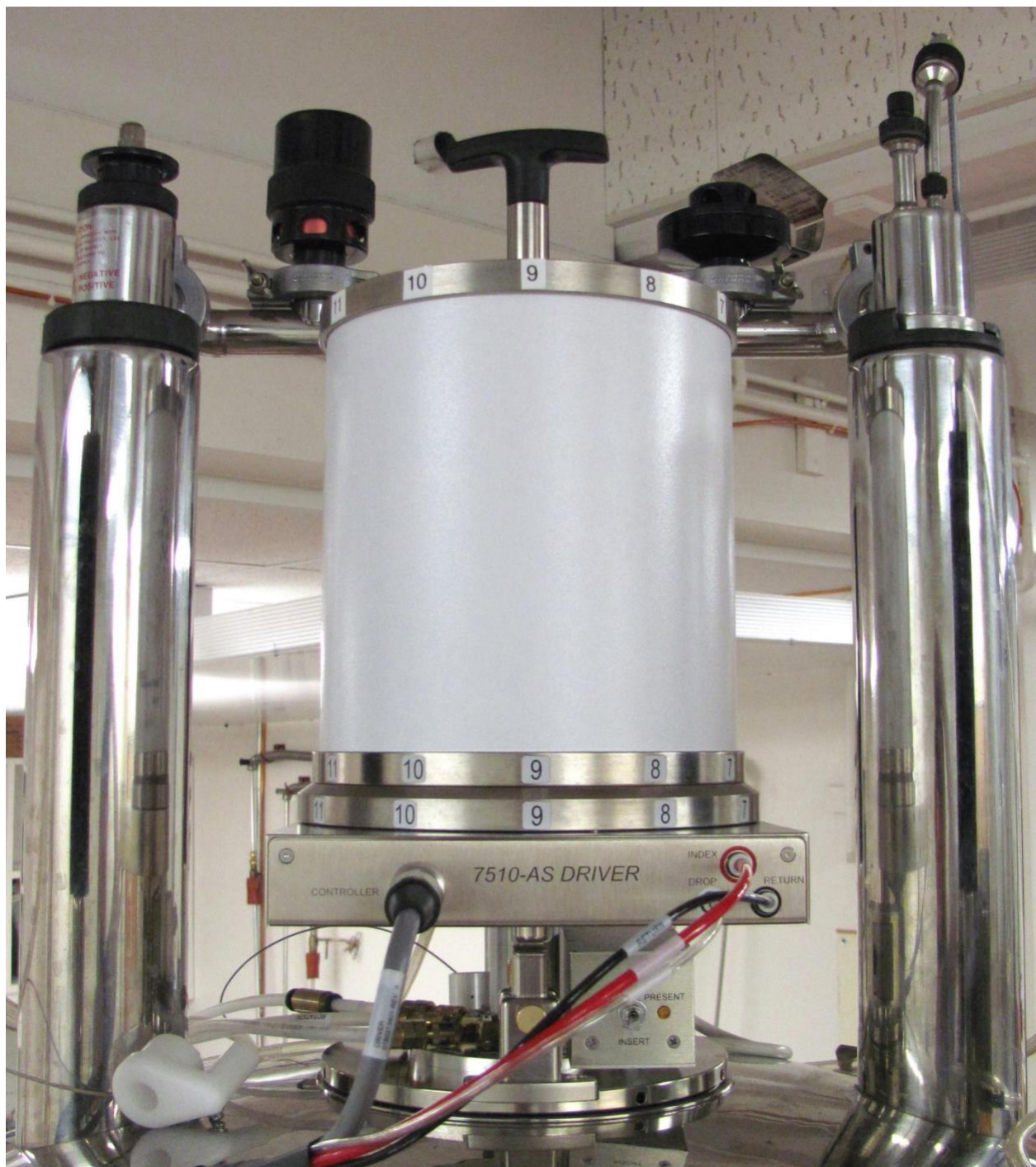




# Operation of u500 with 7510-AS Autosampler



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## OverView

The usage of the u500 with 7510-AS autosampler (robot) is intended to be as similar as possible to the i400 with SMS autosample handling robot (see associated i400 manual). There are differences in usage between these two systems mostly governed by the hardware/autosampler type of each system. Both systems have had their software modified to look and behave as similar as possible to each other.

### Chapter 1 - General Considerations

- Operation is intentionally **simple** and **strict** due to the attached robotic sample handling system.
- No manual tuning, locking, or shimming are required. The software and hardware perform these operations, including inserting and ejecting the sample. All that is required of the user is to select the desired experiment(s), make adjustments to their experiment(s), and supply a sample name.
- The same experiments are available on the u500 as other instruments.

- **No “J-Young” tubes**

#### WARNING

No “J-Young” tubes on the u500 7510-AS robotic system  
The top is not compatible with the sample “catcher” at the  
top of the instrument.



- **No data processing/plotting** at u500. Please use the NMR data station(s).
- **An e-mail address is required for usage.** The u500 will send an **e-mail** when experiments for a sample are complete, or when a sample fails to run due to an error.
- **Files are saved automatically** in a standard format based on the date the sample was run, spectrometer name, time acquired, user name, research group and sample name. The time stamp ensures that all file names are unique. For example, the user *Testuser* in the research group *demo* measures a H1 1D on the sample named *robot demo* on the u500. The text in blue italics, *robot demo*, was entered by the user. The resulting file name:

```
/home1/demo/nmrdata/DATA_FROM_NMRSERVICE/Testuser/2012.05/2012.05.07.u5_rob  
ot_demo_13.45_H1_1D.fid
```

- Data are copied to the user’s regular research account every half hour on the half hour and all data can be found in the directory **DATA\_FROM\_NMRSERVICE** in the users regular research account. An example of when files will appear in a user’s research account: a file saved at 11:25 am is copied to a research account at 11:30 am, moreover, a file saved at

11:35 am is written to a research account at 12:00 pm. In the above [robot\\_demo](#) example, the spectrum was recorded at 13:45 and the user will find their data in their research account in the directory specified above at 14:00.

- **Samples not picked up within 24 hours will be disposed of** without further notice.
- **In case of problem**
  - Place the “**Out of Order Sign**” up in front of the keyboard
  - **Contact the NMR staff** - Phone: 780.492.2573, room WB-13, and/or email the NMR Facility Manager after hours.
  - **Do not attempt repairs yourself.**
  - **When in doubt, stop.**
  - **Be safe.** Never do anything that will endanger you or others. Remember we are never in a rush.

## Chapter 2. Time management (Queues).

- Holidays are treated like regular days.
- The start/end of queues and maximum study length for each sample are controlled by the software. These times are periodically reviewed based on user input and feedback.
- Studies must fit within the desired queue. No time is carried forward from one queue to the next. The day queue (DayQ) starts at 8:00 am and ends at 8:00 pm. The night queue (NightQ) starts at 8:00 pm and ends at 8:00 am.

**Table 1-** Summary of DayQ and NightQ Maximum Study Times

|   | Monday-Friday | Saturday & Sunday |
|---|---------------|-------------------|
| <b>DayQ</b>   | 08:00 – 20:00 | 08:00 – 20:00     |
| <u>max.</u> study time  | 0.5 hours     | 2 hours           |
| <b>NightQ</b>   | 20:00 – 08:00 | 20:00 – 08:00     |
| <u>max.</u> study time  | 4 hours       | 4 hours           |
| <b>Sample submission to the NightQ is available daily, starting at 16:00.</b> |               |                   |

All study times indicated in Table 1 include the **total experimental time**, plus all overhead required by the spectrometer to insert, lock, tune and shim a sample, approximately 5 min.

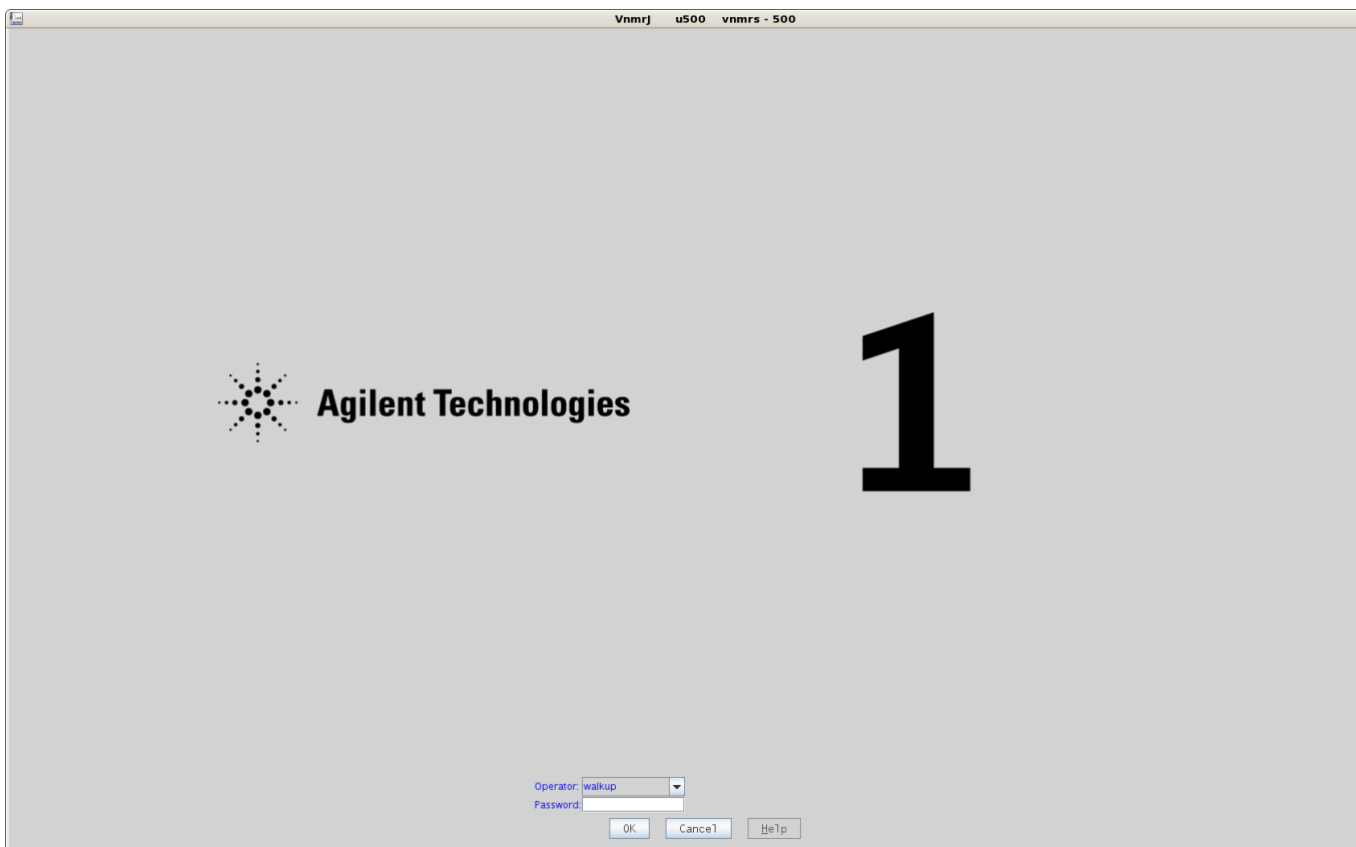
- The software will prevent the following:
  - Sample submission to the study queue when the combined time of all experiments for one sample is over the study time limit for the selected queue.

- Changes or additions to a study once submitted for acquisition.
- Sample submission when the DayQ or NightQ is full.

### Chapter 3. Step-by-Step Operation.

It is assumed throughout these instructions that carousel **position 1** is the next available position in the autosampler but any **Grey**, **Green**, or **Red** completed position may be available (see below).

1. The login screen as shown in Figure 1 should be present. If someone has forgotten to logout, please click the “Switch Operator” button (Figure 6) which should return the system to the Login Window. The very large number (**1** in this example) indicates the next suggested available position in the carousel. Select your research group from the **Operator** drop-down list and enter the **Password** for the operator/group selected. Generally, the same password is used as on other spectrometers, however the unique password *prefix* will be different for each instrument.



**Figure 1** - Login window.

**Hint:** Having a problem with the password? Check for an accidental “spacebar” keystroke before the password from trying to wake up the sleep display.



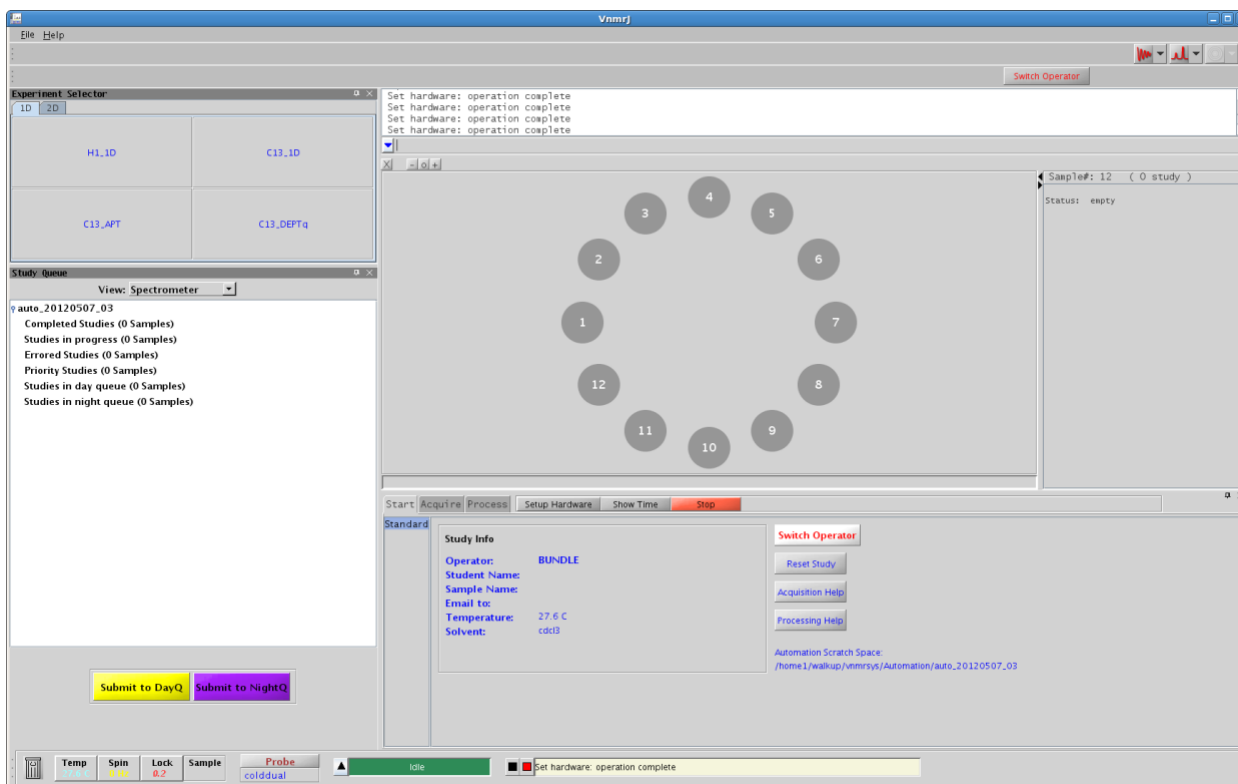


Figure 2 - VNMJR interface when used with the 7510-AS autosampler.

- The graphical user interface for the sample changer software is shown in Fig. 2. Tray locations shown in Grey, Green, or Red are available for use. Tray positions that are shown in Cyan, Yellow, or Purple are busy and **NOT** to be used.
- Remove spinner #1 from the table spinner tray (Fig. 3). If your sample has a removable label, attach it where spinner #1 was located on the table spinner tray so that the sample can be found again. Spinners for two different sizes of NMR tubes are available, 5 mm NMR and 3 mm NMR tubes.

### Why 3 mm NMR tubes?

To be brief, salt. Salty samples can be aqueous samples containing salts, acidic or basic samples, even charged compounds with a counter ion dissolved in an organic solvent.



Figure 3 – Table spinner rack for u500

The u500 uses a “Cold Probe” with very high signal to noise (*i.e.* high Q-factor) which is extremely sensitive to sample salt content. Salty samples can be un-tunable on cold-probes with a 5 mm NMR tube. In some cases, while the sample might be tunable the signal to noise for the sample will be greatly reduced. We recommend **3 mm NMR tubes for salty samples**. The shape of the tube reduces and can even remove the effects of salt. **Spinners 11a and 12a** are for **3 mm NMR tubes**. Please place these spinners in carousel positions 11 and 12 on the autosampler.

4. Insert your sample into spinner #1 and adjust the sample height in the spinner using the depth gauge as shown in Figure 4.
5. Place your sample inside spinner#1 into the carousel position #1 in the autosampler (Fig. 5).

**Spinners are NEVER placed in the carousel without a sample.**

Figure 5 - Top of autosampler.

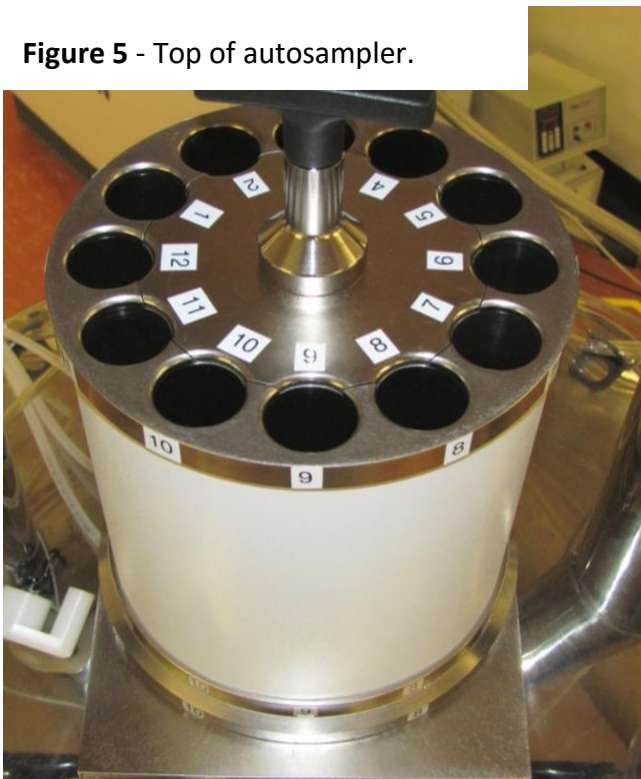
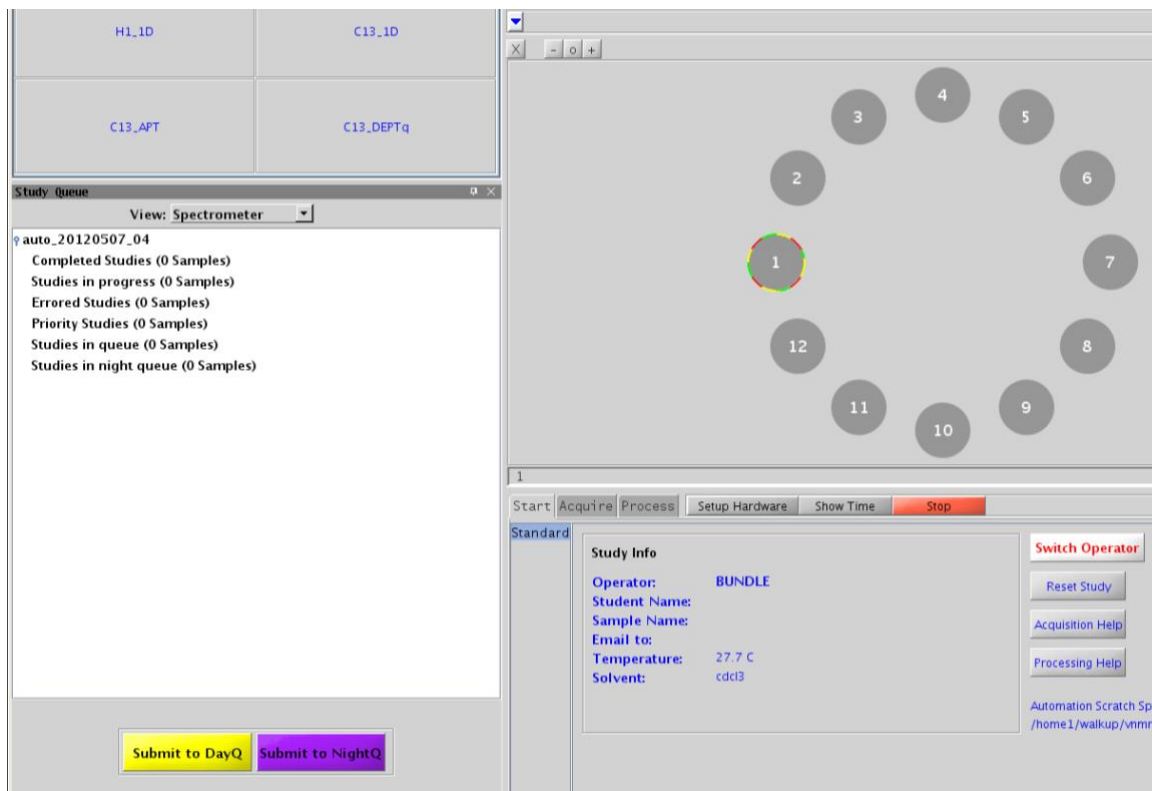


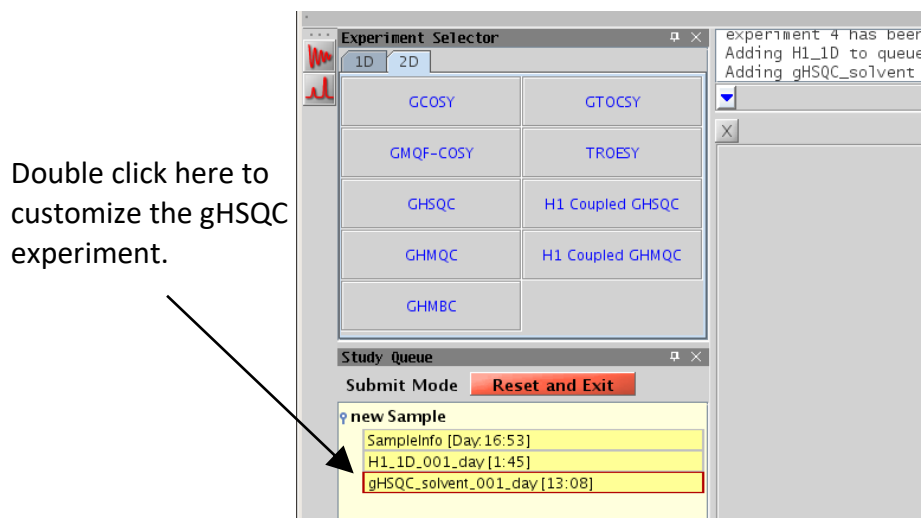
Figure 4 - Sample depth gauge

6. Click on **virtual carousel position #1** in the software panel (Fig. 6), and then click on the button **Submit to DayQ** or **Submit to NightQ** to enter **submit mode** for either the DayQ or NightQ (Fig. 7).



**Figure 6** - Click on virtual carousel position number 1 to start the submission process.

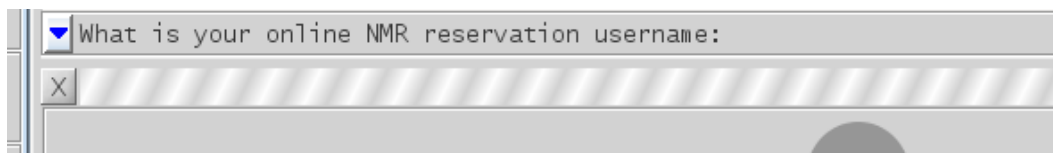
- If needed and time permits, more experiments can be added to the study (see Fig. 7, where a "H1 1D" and "GHSQC" have been added to the study).



**Figure 7** - DayQ submit mode. A H1\_1D and GHSQC have been added to the study.



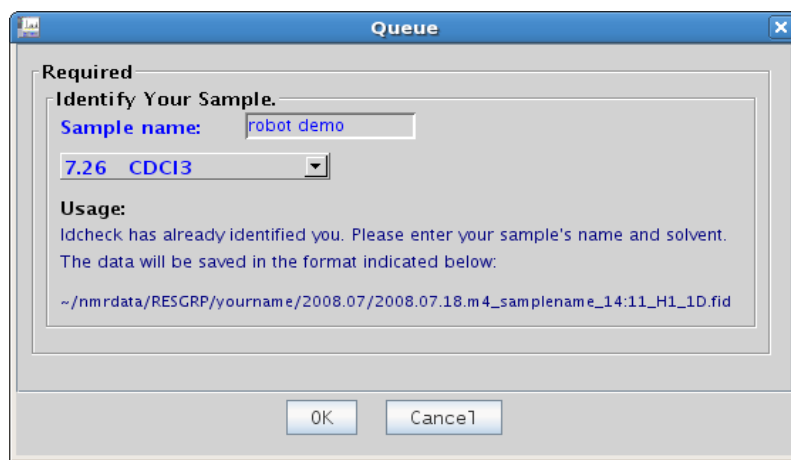
8. The study length will increase as more experiments are added until the maximum time limit is reached for the StudyQ. The study length also contains “overhead” for robot movement, locking, shimming, etc.
9. Make sure the study has all desired experiments and changes to those experiments. Changes to an experiment must be made before submitting the study. Simply double click on the experiment that you would like to change, *e.g.* H1\_1D in Fig. 7, and then make changes to the experiment as desired (Chapter 4, Fig. 13).
10. When all desired experiments have been added, click on the **Submit** button (Fig. 7) to submit the study for acquisition. **NO further changes are possible once the study has been submitted to the desired StudyQ.**
  - If desired the entire study can be reset by clicking on the button **Reset Study**. All experiments will be removed, and new experiments can be added. The button **Reset and Exit** will remove all experiments and exit out of submit mode.
11. The system will ask you to **identify yourself** by entering your on-line reservation username (Fig. 8). Sample submission will be allowed to continue:
  - **if** your username and email address are registered with the NMR Service group.
  - **if** you belong to the same research group as the Operator that is logged in.

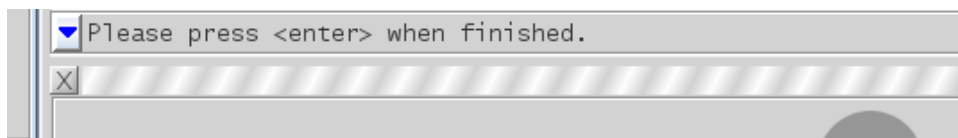


**Figure 8** – Identify user for file saving.

12. A new window will appear (Fig. 9) to identify your sample.
  - **enter a name** for your sample
  - **select the solvent** from the drop-down menu
13. Click on the button OK to complete your sample submission, or click on Cancel to reset the study.
14. Finally, complete the submission process by pressing the Enter key (Fig. 10) on the VNMRJ command line.

**Figure 9** - Identify your sample.





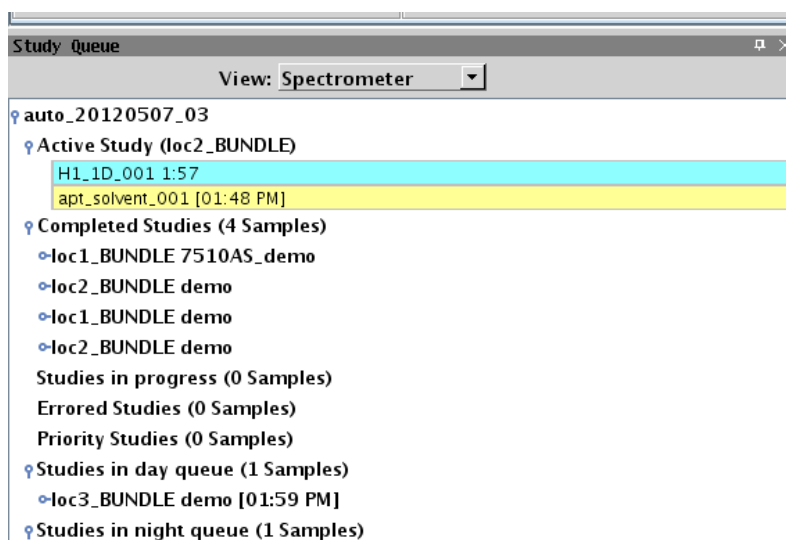
**Figure 10** - Press Enter on the VNMRJ command to complete the submission process.

15. The system will insert your sample, tune and match via ProTune, find the lock, and shim. The StudyQ will display your study as active or submitted to the queue of choice, the sample location, Operator name and sample name (Fig. 11). If the study is active, the experiment in progress and the experiments waiting in study are highlighted (**Cyan** - the current running experiment, **Yellow** - the experiments waiting to be acquired during the DayQ and the time the experiment will start).

16. If no other samples are to be submitted, click on the **Switch Operator** button to return the system to the login screen (Fig 2.).

17. Relax! Wait for the system to send you an email and pick up your sample.

18. If **another sample** is to be measured, select the next available carousel position in autosampler and software panel (for example carousel position #2, Fig. 2 and 5) and repeat steps 3 to 17 above for that sample.



**Figure 11** - StudyQ summary panel. Information regarding the current StudyQ, samples that are: active, completed, pending acquisition in the DayQ or NightQ, etc.

19. **Wait for an e-mail** from the system when your study is complete

20. **PLEASE pick up your sample** as requested in the e-mail. **The last sample measured by the spectrometer will be removed. All completed samples will be sitting in the autosampler carousel.**

21. Notes about colors in the virtual carousel panel (Fig. 12).

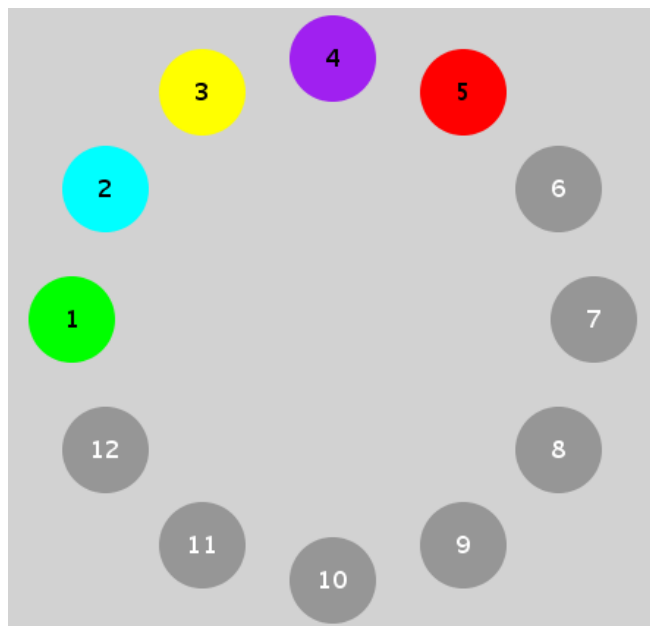


Figure 12 - Virtual carousel sample colors.

#### Positions **OK** to use

**Grey** A tray position that is available to use, no sample is present. **PLEASE use** these tray positions **first**.

**Green** The sample was completed successfully and an email has been sent to the user. **Ok to re-use**. If a sample is in the spinner please remove the sample, **re-attach the sample's label**, and place in a tray beside the spectrometer host computer.

**Red** The sample encountered an error and did not complete successfully. An email has been sent to the user. **OK re-use**. If a sample is in the spinner please remove the sample, **re-attach the sample's label**, and place in a tray beside the spectrometer host computer.

#### Positions **NOT** ok to use

**Cyan** The sample is currently being measured by the system. **DO NOT use**.

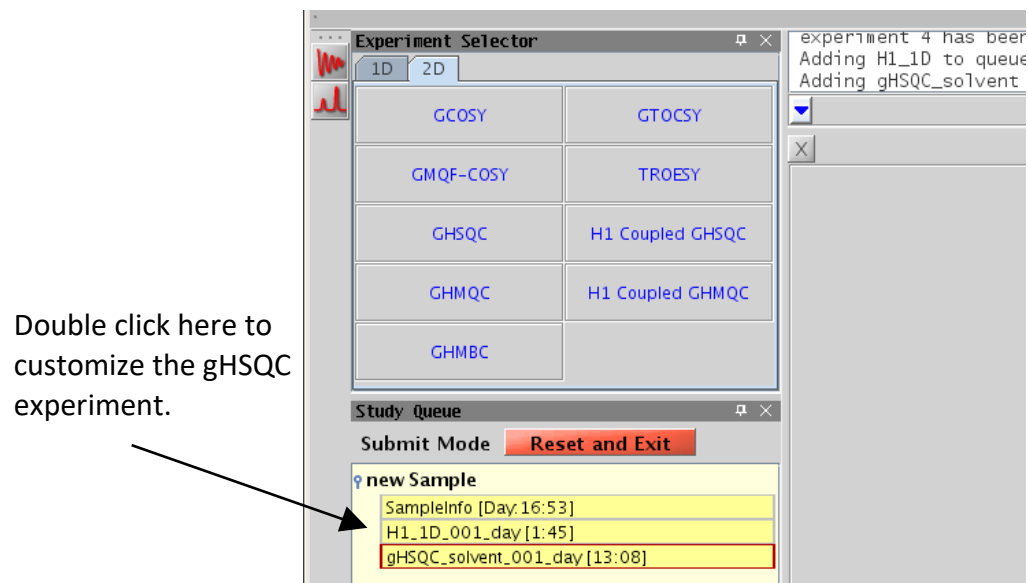
**Yellow** A sample has been submitted to the DayQ and is waiting in the DayQ for measurement. **DO NOT use**.

**Purple** A sample has been submitted to the NightQ and is waiting in the NightQ for measurement. **Dark purple** (not shown) can indicate that a sample is in prescan (*i.e.* the system is loading the sample, finding the lock, shimming, tuning, *etc.*). **DO NOT use**.

## Chapter 4. Customizing experiments.

All experiments can be customized however these must be done **BEFORE Submission** to the StudyQ. Please be sure you know what effect your planned changes will have on the spectrum. Please note

- Time limitations (Chapter 2) cannot be changed
- Double-click the experiment that you would like to customize (Fig. 13).



**Figure 13** - Double click on the experiment of interest to customize.

- Enter your changes on the command line or in the Acquire tab
- Click on the **button** Save or Show time to check time requirements
- Submit to desired StudyQ when the system allows sample submission
- Remember changing **nt** (number of transients) directly affects the experimental time and the signal-to-noise ratio but does not change resolution. Changing **ni** (*number of increments*) in 2D experiments also changes the experimental time and indirect dimension digitization.
  - The standard Agilent macro **time** can easily be used to adjust the experimental time. The **time** macro will change **nt** to fit the requested amount of time.

Usage of the macro `time(x,y)` where x is in hours and y is in minutes, entering

### Example 1

```
time(4,0)
```

This used on the VNMRJ command line will set **nt** such that the experimental time will be approximately 4 hours.

### Example 2

`time(y)` where y is in minutes, entering

time (30)

on the VNMRJ command line will set **nt** such that the experimental time will be approximately 30 minutes<sup>1</sup>.

- Individual experiments can reordered (left click the experiment and drag up or down in the study) and even deleted (drag to the trash) in submit mode.

Click on the **Reset Study** button to start over or, if one wishes to cancel everything and exit out of submit mode click on the button **Reset and Exit**.

## Chapter 5. Space on the autosampler.

- There is no need to free up space in the StudyQ (in fact you can't\_). All sample positions can be used/reused if a **virtual tray position** is **Red**, **Green**, or **Grey**

If a sample is present in the desired spinner position, remove the sample from the autosampler sample tray, re-attach the label from the spinner tray, and place it in the sample tray beside the spectrometer host computer so the user can reclaim.

## Chapter 6. Quick Autosampler Instructions.

### Chapter 6 - Quick Autosampler Instructions

1. Select Operator from the login screen and enter password. Note the large number that appears on login screen. The number indicates the next available sample position for use. For example, a large number 5 indicates that sample position number 5 is the next available sample position.
  - a. After logging into the system, ensure that the recommended sample position or virtual tray position is colored grey, green or red in the virtual sample tray. **DO NOT** use sample positions that are colored cyan, yellow, or purple.
2. Select the spinner from the spinner tray with the same number as the virtual tray position that you have chosen (*e.g.* spinner 5). Attach your sample label to spinner position on spinner tray.
3. Insert your sample into the spinner and set the sample height using the sample depth gauge adjacent to the autosampler.

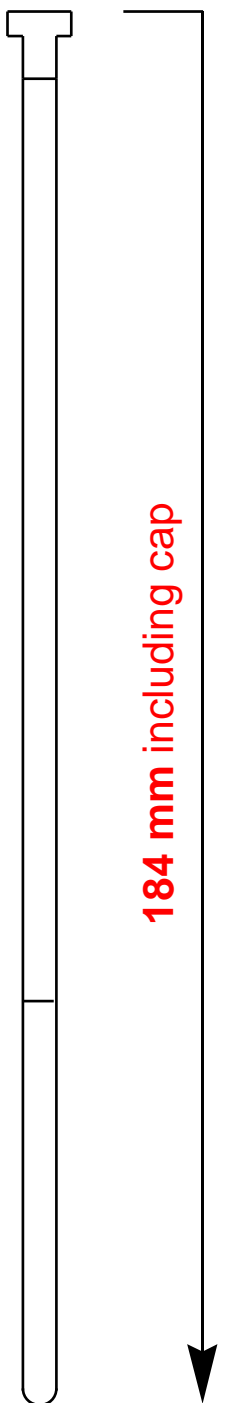
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<sup>1</sup> Note that a single number will be interpreted as minutes, only X,Y is hours,mins



4. Place your sample with spinner in the sample tray adjacent to the autosampler using the corresponding sample tray position and spinner number (*i.e.* sample tray position 5, spinner number 5 corresponding to virtual tray position number 5). **DO NOT** place your sample directly below the autosampler's fingers.
5. Return to the spectrometer host computer and click on the corresponding virtual tray position such that it becomes highlighted (*i.e.* virtual tray position 5).
6. Click on the button Submit to DayQ or Submit to NightQ to enter submit mode.
7. Select desired experiments and make changes to the experiments as needed.
  - a. To make changes to the experiment double click on the experiment of interest, *i.e.* H1\_1D, and use the Acquire tab or the VNMRJ command to make changes.
8. Click on the button Submit to submit the study for acquisition.
9. Answer question that appears on the VNMRJ command line; enter you online reservation name or student ID.
10. A new window will appear, enter your sample name and select your solvent, click on the button OK to proceed or Cancel stop the submission process.
11. Click on the VNMRJ command line and press the enter key when finished.
12. Additional samples can be submitted for acquisition by repeating step 1 to 11.
13. Click on the button Switch Operator to return to the login screen.
14. Wait for email from system indicating successful completion of your sample and pick up your sample.
15. If your sample was the last sample that was measured by the i400 autosampler then it will remain in the magnet. To remove your sample click on the button Retrieve Sample. Your sample will be return to its original location.

Do NOT use sample tubes shorter than shown here!

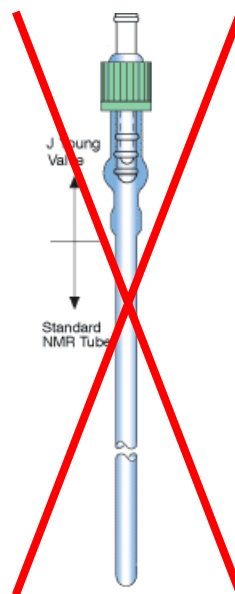


Use only **standard** 5 mm NMR tubes (\*).

Too short sample tubes

- will break and hence the content is lost
- **will damage the system severely**

(\*) **NO** J Young tubes!



What is shown on the left side is the standard length of a 7" NMR tube. Provided the tube did not break previously, it should be this length.