

# Operation of i400 with SMS Autosampler



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## Overview

The usage of the i400 with “SMS autosampler” robot is intended to be as similar as possible to the u500 with “7510-AS” auto sample carousel and the v700 with “7620-AS” robotic arm system. There are of course physical differences in usage between these systems, mostly governed by the autosampler specifications, but all system’s software interfaces have been programmed 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 is required. The hardware and controlling software perform these operations, including inserting/ejecting and moving samples to and from the bore of the instrument from the stand-by racks on top of the support table. Users are required to: insert the sample in a spinner, place the sample/spinner in an appropriate position, start the submission software, select the desired experiment(s), adjust number of scans, click “submit”, then select their solvent, and lastly supply a sample name for storage.
- The same experiments are available on the i400 as other instruments.
- **Full length 7 inch (184 mm) NMR tubes MUST be used** (please see last page).

\*NMR tubes that are cracked, chipped, or broken will be too short and may damage the instrument and/or sample.

**WARNING**  
No “J-Young” tubes on the i400 SMS robotic system  
The robot fingers grab the tube not the spinner and will  
break the sample.



- **Avoid the use of excessive parafilm.**
  - Use parafilm sparingly and only if sealing your sample is necessary. **One single** layer of wrap around the tube is plenty. Too much parafilm may damage the instrument and/or sample.
- **No data processing/plotting** at the instrument. Please use one of the NMR data station(s).
- **An e-mail address is required for usage.** The instrument will send an **e-mail** when all experiments for a sample are complete or when a sample fails to run due to an error.
- **Retrieve your sample(s) when done.**
- **Files are saved automatically** in a standard format and location 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 i400. The text in blue italics, *robot demo*, was entered by the user.

- The resulting location and file name:

### Example Research Lab Data

*group/nmrdata/DATA\_FROM\_NMRSERVICE/Testuser/2021.06/2021.06.13.i4\_robot\_demo\_13.45\_H1\_1D.fid*

For students taking classes and using the i400, data will be saved by their course number, student ID, and sample name.

Example: As above the text in blue italics was entered by a student in Chem 461/561.

### Example Chem 461/561

*.../ch461/nmrdata/studentID/2021.06.07.i4\_robot\_demo\_13.45\_H1\_1D.fid*

- **NMR Sever Backups** - 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 user's regular research account.
  - Examples 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 while 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 (*i.e.* 1:45pm) 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 and Queues

- Holidays are treated like regular days for schedules.
- 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, repairs, 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, while 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	1 hour	4 hours
<b>NightQ</b>	20:00 – 08:00	20:00 – 08:00
<u>max.</u> study time	8 hours	8 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, and shim a sample (*i.e.* approximately 3 min per sample).

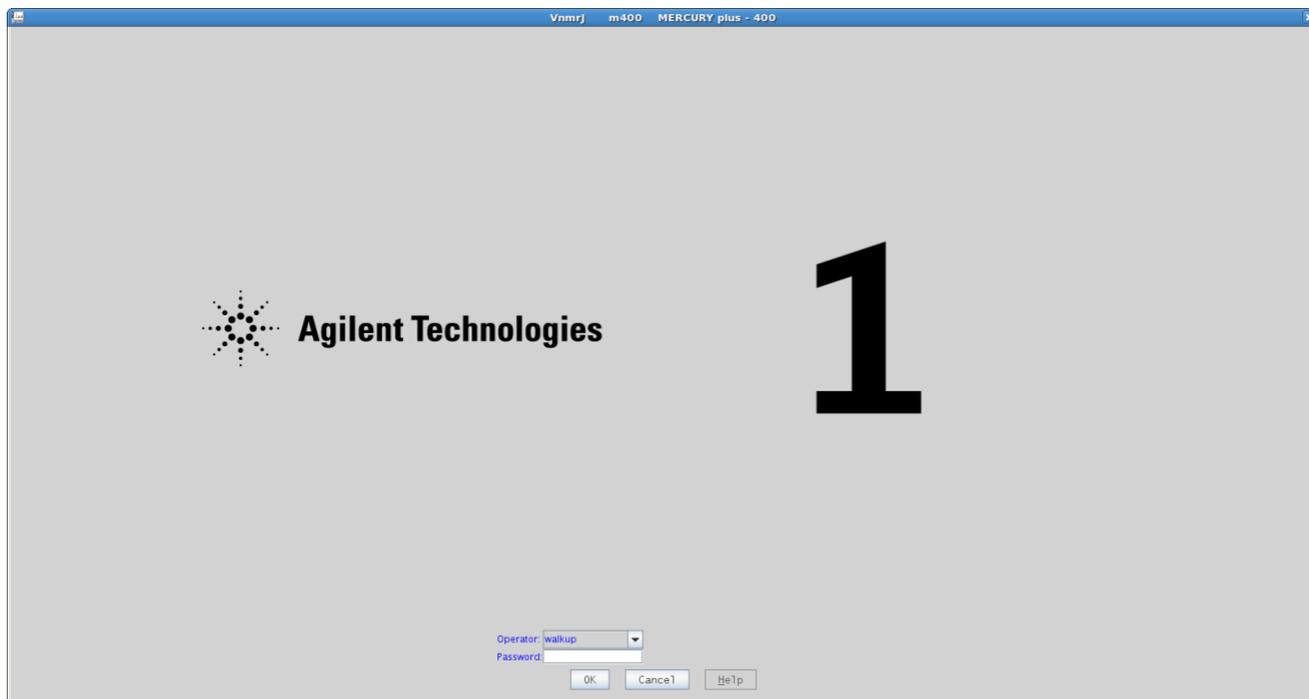
The software will prevent the following:

- Sample submission to a 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.
- Additional sample submissions when the DayQ or NightQ is already full.

## Chapter 3 - Step-by-Step Operation

It is assumed throughout these instructions that tray **Position 01** is the next available position in the autosampler, but any **grey**, **green**, or **red** completed position may be available (see below).

1. The Login Window (Fig. 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 tray. Select your research group from the **Operator** drop-down list and enter the **Password** for your operator/group. Generally, the same password is used as on other spectrometers, however the 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.

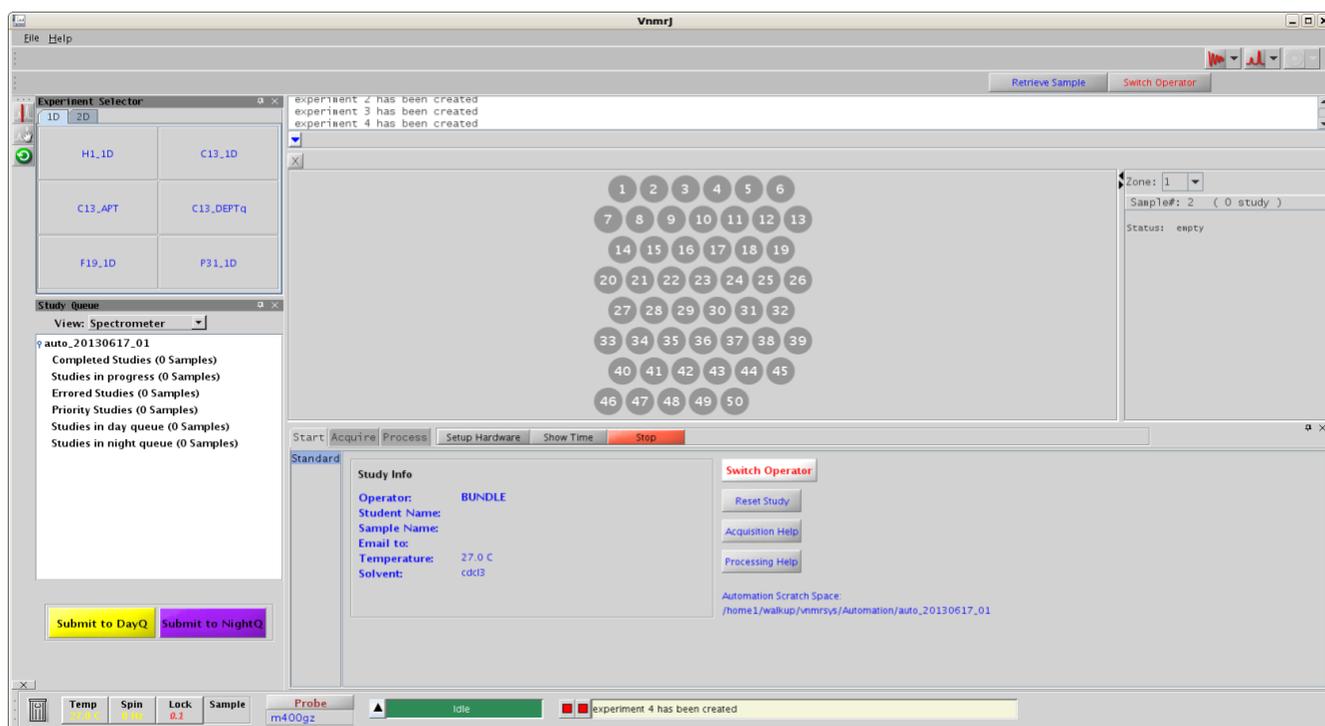


Figure 2 - VNMJR interface when used with “Walkup” style autosamplers.

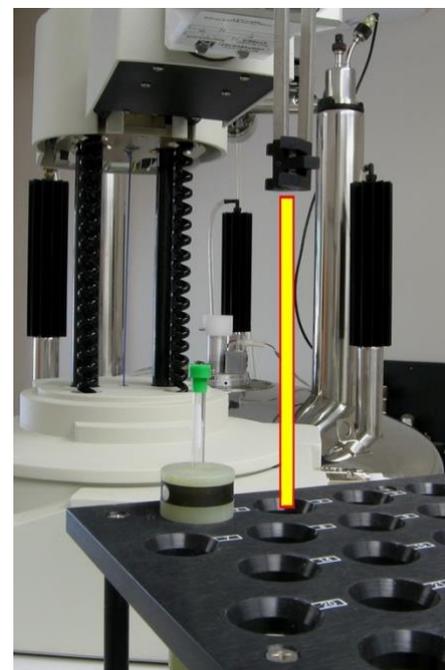
2. The graphical user interface for the sample changer software is shown in Fig. 2. Tray locations shown in Grey, Green, or Red are available for use. Tray positions that are shown in Cyan, Yellow, or Purple are busy and **NOT** to be used.
3. In our example, remove Spinner 01 from the spinner tray (Fig. 3). If your sample has a removable label, attach the label to the spinner tray where Spinner 01 was located so that your sample label can be found again and attached to your sample if/when removed.



Figure 3 - Spinner tray for the i400. Similar trays are at the u500 and v700 but with different tray sizes, etc.



**Figure 4** Sample depth gauge. These are at all spectrometers. If a sample breaks contact staff for help, especially if it breaks in the depth gauge.



**Figure 5** – Example of the SMS sample tray with a sample in tray position 01. The yellow line indicates the sample position below the fingers of SMS autosampler (*i.e.* sample tray position number 02) or the sample position that is currently in the magnet.

**WARNING**

**DO NOT** place a sample below the fingers of the autosampler or add/remove samples when the arm is moving.

4. Insert your sample into spinner 01 and set the sample position in the spinner using the depth gauge as shown in Fig. 4.
5. Place your sample-in-spinner into the sample tray position 01 on the autosampler (Fig. 5). Remember: **Never put your sample directly under the autosampler's fingers** (highlighted by the line, Fig. 5). Doing so will lead to sample loss and equipment damage. The robot arm position indicates the sample that is/was active, and is still in the magnet. The sample will eventually be returned to that tray position.
6. Click on the software **Virtual Tray Position #1** (Fig. 6), and then click on the **Submit to DayQ** or **Submit to NightQ** button to enter **submit mode** for either the DayQ or NightQ, respectfully, depending on the time of day and which queue is desired (see Table 1, and Fig. 7).
7. Select the experiments you wish to run from the buttons in the top left of the software and note that 1D and 2D experiments are in separate groups (Fig. 7).

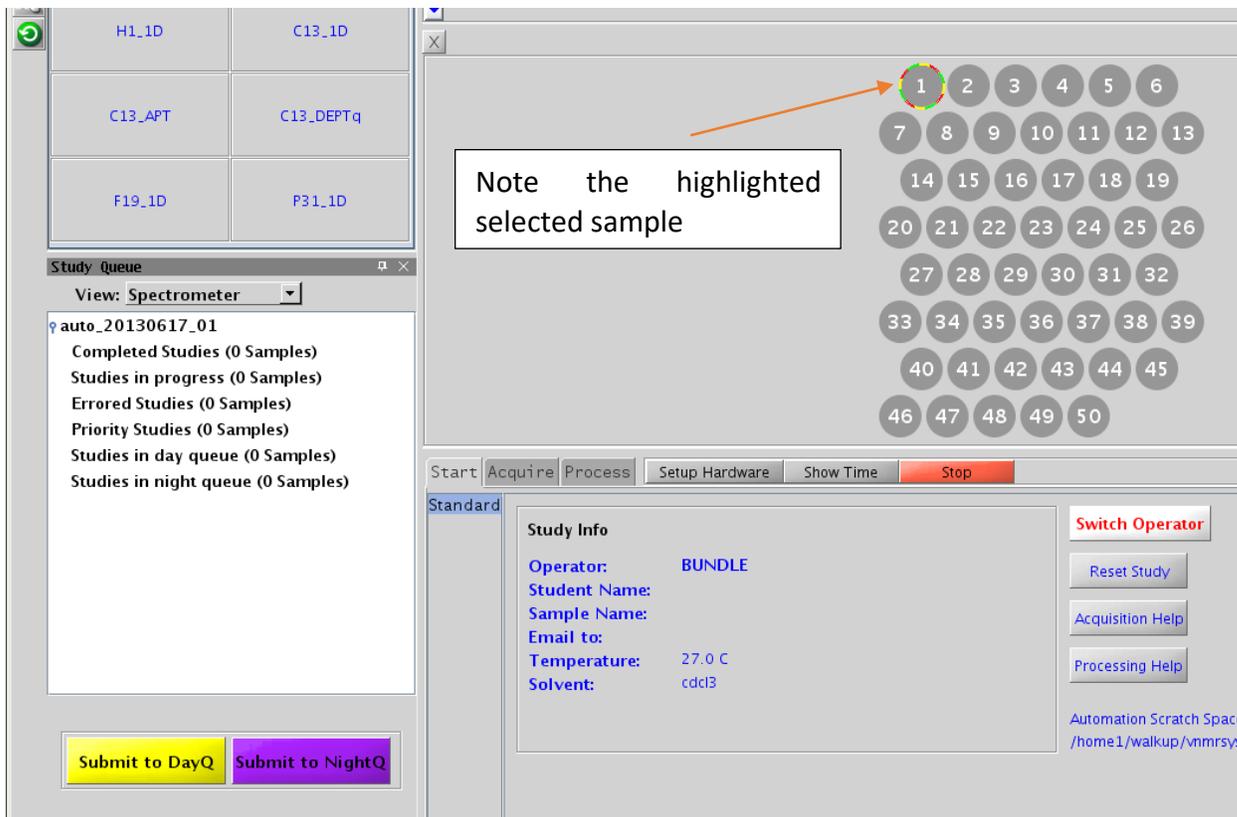


Figure 6 – Example clicking on virtual tray position number 1 to start the submission process.

8. If needed and time permits additional experiments can be added to the study.
  - See Fig. 7 example where a H1\_1D and GHSQC have been added to the study.

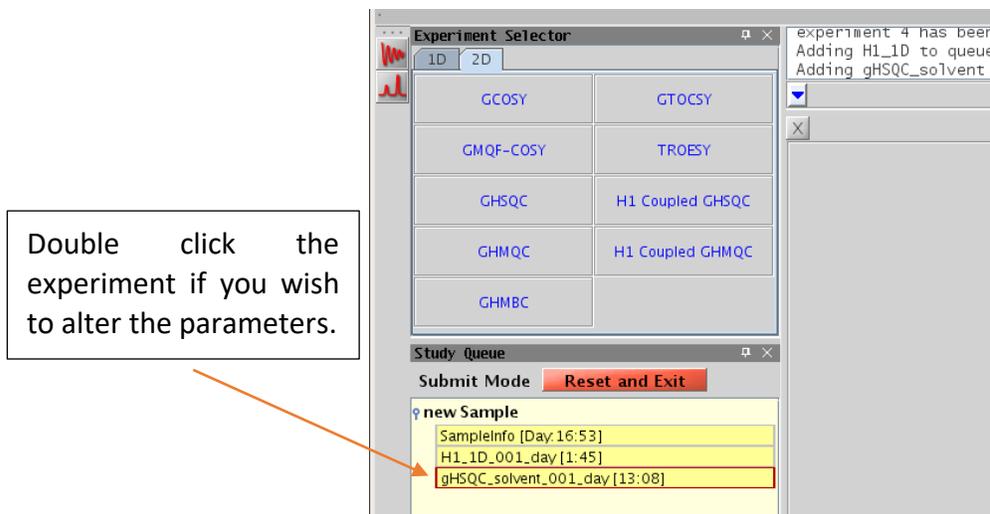
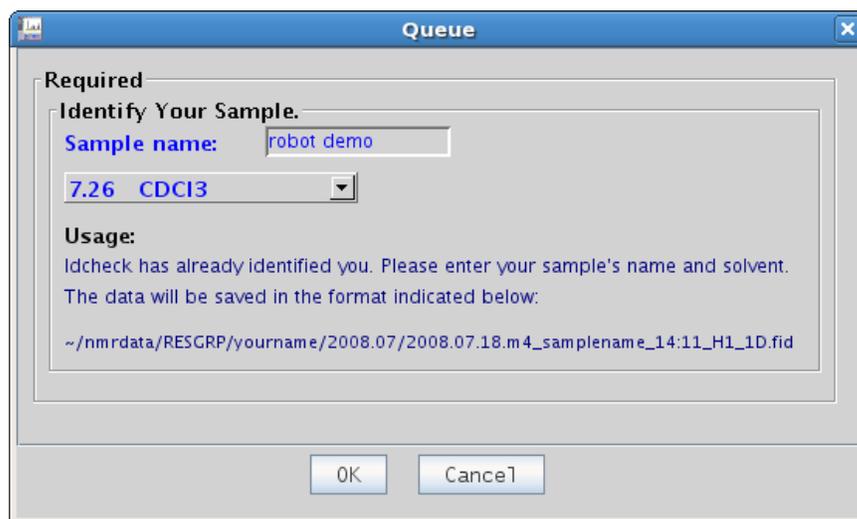


Figure 7 – Example of DayQ submit mode with two experiments selected showing completion times.

9. The study length will increase as more experiments are added until the maximum time limit is reached for the StudyQ. The study length contains “overhead” for robot movement, locking, shimming, *etc.*
10. 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, *i.e.* H1\_1D in Fig. 7, and then make changes to the experiment as desired (Chapter 4, Fig. 13).
11. When all desired experiments have been added and/or modified, click on the **Submit** button (Fig. 6) 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 cancelled/reset by clicking on:  
**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.
12. The system will then 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.
13. A new window will appear (Fig. 9) to identify your sample.
  - **enter a name** for your sample
  - **select the solvent** from the drop down box



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



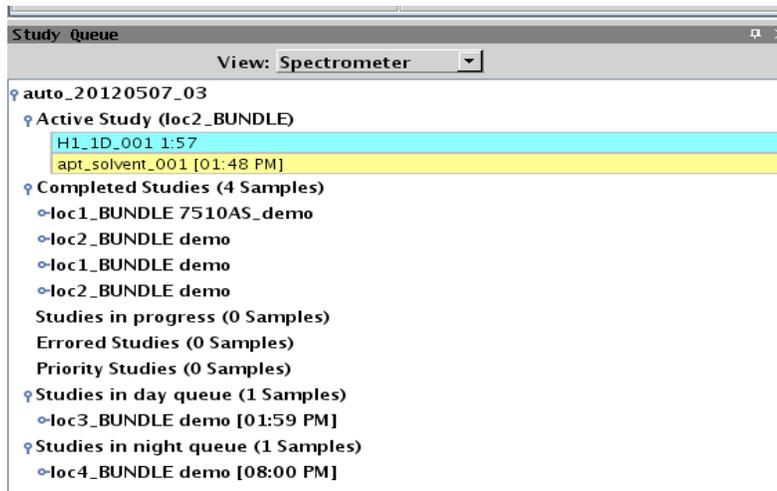
**Figure 9** - Identify your sample.

14. Click on the “OK” button to complete your sample submission or click “Cancel” to reset the study.
15. Finally, complete the submission process by pressing the Enter key (Fig. 10) on the VNMRJ command line.



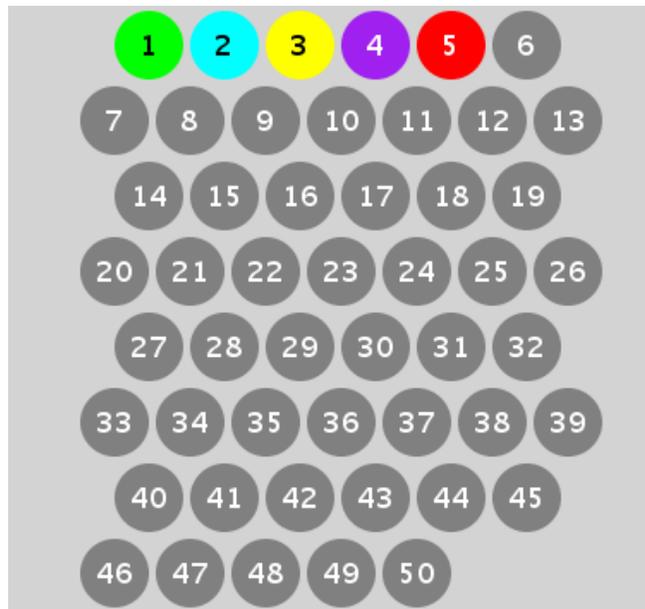
**Figure 10** - Press Enter on the VNMRJ command to complete the submission process.

16. The system will insert your sample, find the lock, tune and match, and shim your sample. 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, experiments in progress and experiments waiting in the study are highlighted (Fig. 11) as follows: **Cyan**- the current running experiment, **Yellow** - the experiment(s) waiting to be acquired during the DayQ, and the approximate time the experiment will start.



**Figure 11** - StudyQ summary panel. Information is displayed about the current StudyQ, samples that are: active, completed, pending acquisition or encountered an error in the DayQ or NightQ.

17. If no other samples are to be submitted, click on the button **Switch Operator** (Fig.2) to return the system to the login screen (Fig 1.)
18. **Relax!** Go back to work and wait for the system to send you an email. Then pick up your sample.
19. If **another sample** is to be measured, select the next available tray position in autosampler and software panel (for example tray position #2, Fig. 2 and 5) and repeat steps 3 to 18 above for that sample.
20. When submitted and accepted, the StudyQ displays the Operator name and sample position as shown in Fig. 11.
21. Notes about colors in the virtual sample tray see Fig. 12 (next page).



**Figure 12** - Virtual sample tray colors and their explanation.

#### 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**.

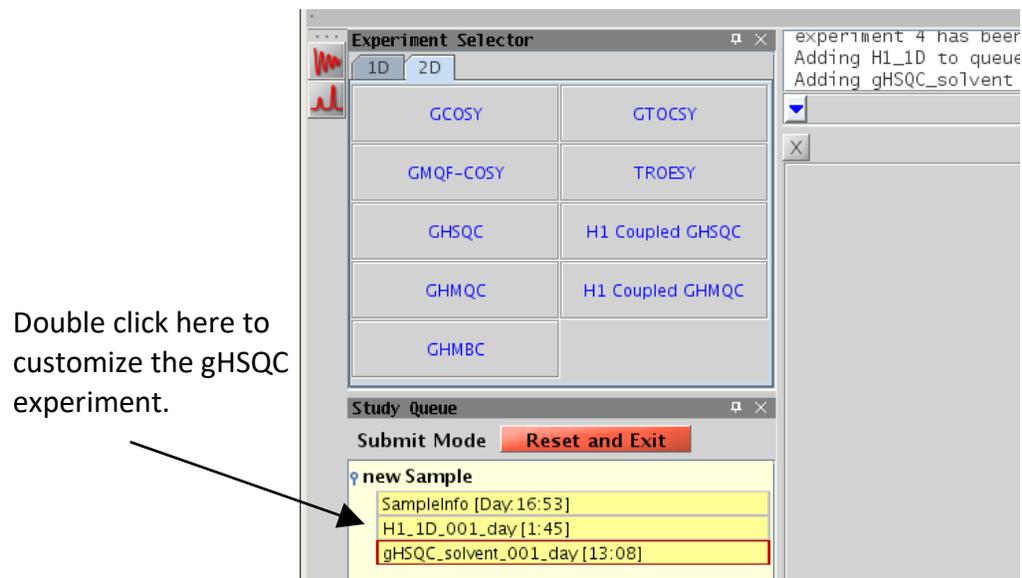
**Yellow** A sample has been submitted to the DayQ and is waiting in the DayQ for measurement. **DO NOT use**.

**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 be 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

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.
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

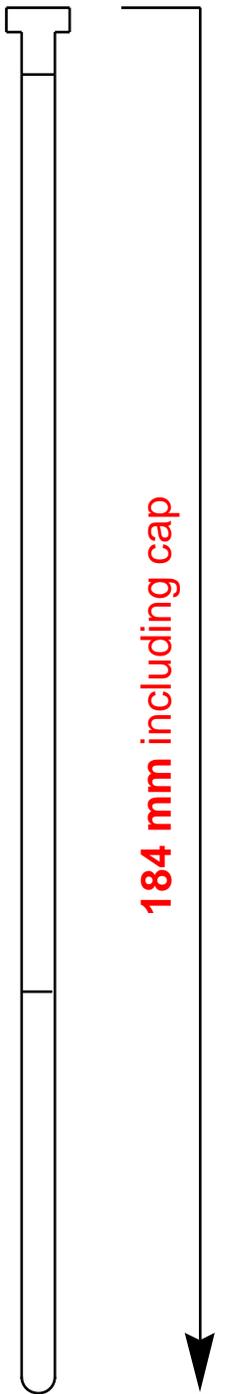
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<sup>1</sup> Note that a single number will be interpreted as minutes, only X,Y is hours,mins

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!**

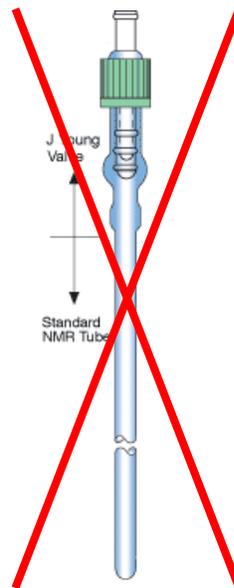


Use only **standard** 5 mm NMR tubes (\*).

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.