

# Training Notes for the Bruker AVANCE 400 with Sample Changer (version 2)

## How it works:

Users put their samples into numbered holders on the carousel of the sample changer and submit their experiments to a queue. The samples are run on a first-come-first-serve basis with all experiments longer than 30 minutes run at night. Immediately after the experiments are completed, the data are stored automatically on disk and can be accessed by way of FTP. Every morning the NMR lab staff will take all completed samples off the carousel and place them in the NMR tube rack in a slot corresponding to the carousel holder in which the sample was placed. If you require your sample tube before 9:00 am the next morning, you should remove it from the carousel and log in to delete the completed sample from the list.

## Before you Start:

1. Your NMR tube **MUST be at least 17.5 cm (6.9 inches)** in length and **MUST have a top that is neither broken nor cracked**. If your tube is shorter than 17.5 cm, it will not be grabbed fully by the sample changer and may fall and break on the floor or on the top of the magnet. A depth gauge is provided with a plastic guide on the side. If your tube is not taller than the guide while touching the bottom of the depth gauge, then do not submit it to the queue. Tubes with chipped tops are weakened and may be broken by the sample pincers which grab the tube very firmly close to the top. **Any tube shorter than 17.5 cm or with a chipped top submitted to the sample changer will be removed by the NMR Facility staff and cut to an unusable length (i.e. destroyed)**
2. Your NMR tube must **not have any label (paper, tape, sticker etc....) attached to it**. This will stop the sample pincher from working properly and may result in sample breakage and equipment damage. **Tubes can be labelled on the glass or the cap with permanent black marker**. Any tube with an attached label submitted to the sample changer will be removed by the NMR Facility staff
3. Sample tubes with screw caps or vacuum valves will not be handled by the sample changer. They **WILL cause equipment damage**.
4. The magnet and sample changer are suspended on air pistons. Never lean or exert any force on the sample changer or magnet. This may cause a magnet quench resulting in many weeks of down time and tens of thousands of dollars being spent.

**Users are asked to report improper sample tubes to the NMR Facility staff immediately in order to avoid needless down time and equipment damage!** Improper sample tubes are:

- a. sample tubes shorter than 17.5 cm (6.9 in)
- b. sample tubes with chipped tops
- c. samples tubes with screw caps or vacuum valves
- d. samples with attached labels

## Collecting Data:

1. Check that your tube is in good shape (no chips or cracks), is at least 17.5 cm in length, is free of paper or tape labels and has a standard NMR tube cap.
2. Put your tube in a rotor and set the depth with the sample depth gauge. Check that your tube is at least as high as the plastic guide on the side of the depth gauge. If it is not, then put your sample in a longer tube.
3. Gently place your tube in any available holder on the sample changer. **DO NOT LEAN OR EXERT ANY FORCE ON THE SAMPLE CHANGER.**
4. At the computer, if the login window is not being displayed, click the "Change User" button

- located near the bottom right of the display.
5. Double click on your supervisor's name.
  6. Enter your password.
  7. Double click on the holder number in which you placed your sample. (If there is a red traffic light beside the holder number, put your sample in another holder without a red traffic light beside it).
  8. Click in the "Name" field and give a name for your data set. Make sure the name does not contain any spaces or "/"s. The name is case sensitive.
  9. Click the down arrow to the right of the "Solvent" field and click the solvent used to prepare your sample.
  10. Click the down arrow to the right of the "Experiment" field and click the desired NMR experiment.
  11. If you want to add an additional experiment to the same sample then click on the "Add" button near the bottom of the display. An experiment will be added with the same data set name with an incremented experiment number.
  12. Click the holder number to highlight it and then click the "Submit" button near the bottom of the display. The traffic lights should turn yellow indicating that your sample is now queued to run. All experiments longer than 30 minutes will be run only at night and all experiments shorter than 30 minutes will be run at any time (day or night).
  13. Click the "Change User" button to log out.

### **Accessing and Processing Your Data:**

All data will be processed offline. Your data is available to you as soon as it has been acquired. Use your FTP program to access 137.122.148.57. Log in with the user name "bloch" and password "nmr". Your data is in a directory with your supervisor's name. For example if you are from Dr. Fogg's lab and you submitted a sample called "reactionproduct" for proton and carbon NMR, the data would be stored as follows:

D:\data\Fogg\nmr\reactionproduct\1	(Proton data)
D:\data\Fogg\nmr\reactionproduct\2	(Carbon data)

It is the responsibility of the user to back up and archive their own data. Data will be removed from the disk at the beginning of every month.

We now have a 5 user network licence for Bruker's new NMR software called "TOPSPIN" which you can use to process all of your NMR your data offline. You are free to install the software on your computer and process your data from any university computer. You will require a licence file from the NMR Facility. Your computer must have a DVD drive to install the software, a TCP/IP enabled ethernet card and at least 512 Mbytes of RAM (! Gbyte recommended). See the NMR Facility manager for assistance in installing the software on your computer.

If you process your raw (FID) data using MESTReC, you will have to phase your spectrum manually and enter the value of -24550 in the "PH" (first order phase correction) field.

If you process your raw (FID) data with XWINNMR in DRO room 416 you will have to type "topspin" on the command line before carrying out a Fourier transform.

If you do not have a suitable computer in your lab, one is available to all NMR users in Biosciences room 411. This workstation is capable of connecting to all of the NMR spectrometers via FTP and runs Bruker TOPSPIN and MestReC for data processing. The computer is equipped with a laser printer and a CD/DVD burner for data printing and archiving. All users are welcome to log in with the username, "bloch" and password, "nmr".

**Retrieving Your Sample:**

The morning after you submit your sample to the queue, your sample will be placed in the NMR tube rack in a slot corresponding to the carousel holder in which the sample was placed.. If you require your sample tube before 9:00 am the next morning, you should remove it from the carousel and log in to delete the completed sample from the list. Samples left in the lab for more than 2 days will be disposed of.