Organic TA's Guide to using Icon NMR

As a TA, you will acquire NMR data for the organic students on the Bruker 400, but the students will process the FID and print the spectra themselves. Once the acquisition is set up, the automation can run without supervision, so data collection should not necessitate your leaving lab for more than the ten minutes needed to load the tubes and enter parameters in the Icon-NMR window.

Before leaving the lab with samples, verify that:

1. All samples are tagged in a way that will not interfere with the sample rotation or leave any residue in the instrument. Removable paper tags are fine. A tape flag is not.
2. All samples are a reasonable depth (1 – 1½").
3. All samples are clear and well mixed — uniform color and no droplets or suspended particles.
4. The exterior of each tube is clean.
5. All tubes are a sufficient length (6½" minimum; see the guide in the organic prep room).

If any samples are not properly made, the student should fix the problem or make a new sample.

To set up the Data Collection

1. Log into the Organic Lab account of the NMR computer.
2. TopSpin should open automatically; if it does not, click the TOPSPIN icon (lower left).
3. A login window for the automation (Icon-NMR: Identify User) should eventually open. If not, type icon in the main TopSpin window and click the AUTOMATION button.
4. Log into Icon *as the lab day* (Mon, Tue, etc.). Sorting by Full Name will help you find the day.
5. For each sample:
   - *i.* Wipe the sample with a Kimwipe and insert it into a spinner, using the depth gauge.
   - *ii.* Place the sample in the first available slot of the carrousel, noting the slot number.
   - *iii.* If students are using paper tags, write the slot number on the tag so that the correct tube may be returned to each student.
   - *iv.* Fill in the table row for the sample (see conventions below!).
   - *v.* If two spectra are desired for the sample (eg, ^{1}H and ^{13}C), click the ADD button to add a second line for the sample, and change the experiment to "c13_32scan".
6. Highlight the rows you filled in Step 5, and click SUBMIT. The rows should change from "available" to "queued." To change anything after this point, CANCEL the row, EDIT it, and re-SUBMIT it.
7. Place the ethyl benzene standard as the last sample of the run, and acquire its spectrum as "EtBz". It will remain in the magnet when you are done.
8. Click the gear-icon at the top left.
   - The first sample will generally be in slot #1, but enter a different value if it is not.
   - Other settings should not need changing.
9. The run should start:

- Any sample in the magnet will be ejected and the first sample injected.
- Verify that the automation is starting properly.
- If additional samples need to be added, enter them as above and click SUBMIT.

10. Note the "Busy Until" time at the bottom of the screen and return to lab.

Shutting Down

11. Stop the run (stop-sign icon).
12. Close the automation window.
13. Verify that the standard is indeed in the magnet and the lock-level is reasonable (stable red/green traces about $^2/_3$ the way up the black window.)
14. Close TopSpin and exit the window-server (right-click on desktop/LEAVE).
15. Bring the samples back to the lab.

Datafile Naming Conventions

- The sample name should be the student's first name and last initial, with no space (eg, "JohnR"). Please maintain this convention to help students to find their spectra and to keep the data folder manageable.
- The Bruker EXPNO parameter ("No." in the setup-table) should correspond to the lab experiment: data from the first week involving NMR should go in the 10's, the second in the 20's, and so on. Within the experiment, EXPNO 20 and 21 might be the ^{1}H and ^{13}C data for the same sample, or the first and second distillation fractions.
- Include a title with the student's name and the experiment name: eg, "John R -- Grignard product."
- To maintain the numbering for partner labs, save the data into one partner's directory, then, click the NMR_DIR button at the left of the desktop and copy the data folder (eg. "20") into the other partner's directory.

Troubleshooting

Panic Button: If anything goes haywire, press the big red button on the front of the sample changer. Contact Dr. Trujillo or Mencer to reset the changer.

Pincers Can't Grab Tube: Is the tube too short? Was the turbine set at the right height?

Frozen Sample-Changer: If the sample changer gets stuck and the red LED on the changer begins blinking, press the RESET button on the changer.

Failed Samples: Verify that the sample was properly made and loaded (appropriate sample depth, appropriate positioning in the spinner, a lock signal is near the middle of the black window). If things seem ok, put it back in the carousel, CANCEL the row, and re-SUBMIT it.

BSMS Errors: If messages regarding the BSMS unit appear, ask Dr. Trujillo or Mencer for help.

RG Errors: This message appears regularly, but is not a problem.