Operation of Siena: Varian 400 MR

NMR Facility Director:	David VanderVelde	x3004	davidv@caltech.edu
GLA:	Clint Regan	x6050	cregan@caltech.edu
NMR Facility Website:	http://mangia.caltech.edu/NMR.html		

Safety Considerations

Compliance with these rules is required the NMR facility!

WARNING

Accidents caused by carelessness near strong magnetic fields can cause serious injury or death and significant damage to personal property, equipment and data

- **1.** Individuals with medical devices that can be disrupted by magnetic or radio fields (e.g. cardiac pacemakers and metal prostheses) must remain outside the 5-gauss perimeter.
- 2. In the event of a "magnet quench" (a sudden, violent release of cold helium gas from the magnet), evacuate the facility immediately, and contact NMR personnel.
- **3.** Ferromagnetic metal objects must remain outside the 5-gauss perimeter, marked on the floor around each magnet. This includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Only non-ferromagnetic materials should be used near the instruments.
- **4.** Do not look directly down the upper barrel of an NMR spectrometer while trying to eject the sample. Refer any problems with sample ejection to facility staff.
- 5. Do not exceed the boiling or freezing points of your sample; approach these temperatures cautiously, as the instrument may overshoot the selected temperature or the temperature display may be somewhat inaccurate. This can cause excessive pressure to build up and break the tube.
- 6. If you have a sample that is expected to have or generate internal pressure, test all operations with it in a hood and with suitable safety equipment before inserting it into an instrument. Cold samples will warm up significantly while they are being inserted into the instrument, even if the probe is precooled.
- 7. Keep your hands out of the path of the robotic arm of the SMS sample changers. Broken NMR tubes and cuts to your hands could result.
- 8. Wipe down NMR tubes with a Kimwipe before they go in the magnet and <u>after</u> you remove any lab gloves you have worn from your own lab. Don't wear lab gloves while handling spinner turbines or typing on shared keyboards.
- **9.** Don't pipet or mix chemical solutions on the computer work tables—use the fume hood. Lab gloves and eye protection should be worn for those operations, but remove the gloves before returning to the spectrometer.
- 10. Cards with magnetic strips, cellular phones, laptops and mechanical watches should remain outside the 5gauss perimeter. Strong magnetic fields surrounding the NMR spectrometers can damage the strip of magnetic media found on credit cards, ATM cards, driver's licenses, and other kinds of cards, disk drives, and other susceptible devices.

The magnet on Siena is internally shielded and has the smallest fringe field of any magnet in the lab. The 5 gauss line only extends out a couple of inches beyond the magnet itself. However, a fringe field is still present and you should be as careful around the magnet with ferromagnetic objects as you would with any other NMR magnet.

The magnet is supported by anti-vibration legs, which increase spectrum quality. Do **NOT** lean on or bump the magnet when you are changing samples! The magnet can be set into rocking motion very easily, which will negatively affect your spectra.

Siena's specs

The instrument is a 400 MHz Spectrometer with multinuclear capabilities from 31P to 15N and 1H to 19F, computer controlled probe tuning, a variable temperature range from -80 to +130 C, and a 12 position sample changer for automated operation. You may use any of these features during your reserved time, but it is your responsibility to ensure that your planned experiments fit within your time slot.

Scheduling

The instrument has an online calendar, accessible through WebCal (http://chemex.che.caltech.edu/cgi-bin/webcal-3/webcal.cgi), called "Siena."

Time limits are:

Mon-Fri 8 am – 5 pm: 30 minute maximum

Sat-Sun 8 am – 4 pm: 2 hour maximum

All other times: unlimited

The calendar software does not enforce these limits, so it is your responsibility to stick to them.

Please sign the logbook whenever you use the instrument.

Sample submission

Use the 7510 sample changer to load and unload samples. This changer can accept tubes of varying length, including J. Young tubes. Please use tubes that are at least 7" in length so that they are easy to put into and pull out of the sample changer.

Insert your NMR tube into a spinner and set it to the correct height using the depth gauge. For best results, the sample volume should be ~ 600μ L which corresponds to about a 5 cm solvent height. Make sure the solvent height covers the depth gauge window. **PLEASE DO NOT ADJUST THE DEPTH GAUGE** (it should be set at 70 for Siena). Reduction of sample height below 4 cm can lead to longer shimming times and degrade peak shape.

When loading a sample, put your sample(s) in the chute(s) to the left of the one over the magnet bore (it will have the next higher number(s)). Do **NOT** put your sample in the chute directly over the magnet and type "i." This would be asking for a broken sample and a contaminated probe.

You can have samples loaded automatically by using automation, or you can load them manually. If the chute currently over the magnet is #4, and your sample is in #5, type

loc=5 change

to load your sample. If you want to retrieve the sample in #5, type

loc=6 change

There is an empty tube with a green cap that should be left in the magnet after your experiments. This avoids a number of errors that may otherwise pop up.

Manual operation

You can run any of the predefined experiments manually just by dropping and dragging a protocol into the main window. Then it is your responsibility to do a find z0 and shim (gradient shimming is highly recommended). It is also an excellent idea to tune the probe on your sample, as you may not know what nuclei were used previously and where the probe is currently tuned. Under manual operation, tune the probe by going to the Tools menu and choosing Auto tune

probe. (If doing a multinuclear experiment, tune to the low gamma nucleus first.) Then you can click the relevant buttons under Quick Tune.

Automated operation

Each time you want to run automation, select Automation -> Automation Run -> New Automation Run. You will see all grey circles for when viewing the sample changer on your screen. This avoids a number of errors that may otherwise pop up.

You can submit a sample queue of one or more samples through your own account. Experiments of any length will be accepted, but it is your responsibility to be sure they will fit within your time reservation. As with manual operation, it is recommended that you have the probe tuned as part of your experiment. The "tune" box is checked by default on the submit panel—all you need to do is to leave it that way. Load your first sample manually before you start an automation run (the sample number display window in VnmrJ should have a number in it, not be blank).

On Siena only (do **not** do this on Indy or Hg3), you can submit your samples to a queue, log out, and leave, so you don't have to return to log out later. Your experiments will continue; your last sample will remain in the magnet until you or the next user enters a "loc" command. However, if another user has a queue running, it is not possible for you to submit samples ahead of time by "queuing queues."

Multinuclear operation

You can run manual or automated multinuclear experiments using any combination of double resonance that is valid on the hardware that we have. Basically you can combine one high gamma nucleus (H or F) with one low gamma nucleus. We cannot combine two high or two low gamma nuclei. (Although there is a tab for H-F experiments in the software, these won't run.) For a nucleus you are unfamiliar with, consult with staff to verify that a 90 degree pulse calibration exists for that nucleus.

Variable temperature operation

For the time being, please alert Dave or Clint if you plan to run VT on Siena.

The probe limits are 130 to -80 C. For temperatures above 100 or below ambient, use nitrogen gas instead of air. We don't have an FTS air bath connected to the instrument at this point, so below ambient operation is only possible by using the heat exchanger bath. You can use liquid nitrogen in the bath, or other cold liquids, although the Styrofoam bucket is NOT compatible with a dry ice/acetone slush.

The nitrogen gas pressure in the lab is not high enough when more than one instrument is using nitrogen gas. Therefore, if someone is using nitrogen to do VT on Daytona, it should not be used concurrently on Siena. Please consider this limitation in your scheduling and make sure to communicate within the lab.

When it comes to VT gas pressure, if you would like to adjust it when at lower temperatures, please only do so through the buttons on the VnmrJ interface and do **not** adjust any of the toggles on the instrument hardware. Also be sure to set the VT gas flow back to "10" when you are done.

DOSY experiments

We have a full set of pseudo-2D and pseudo-3D DOSY experiments on the instrument, plus some DOSY data analysis options not available on any of the other instruments. These are enhancements introduced with VnmrJ 3. There is a full PDF manual describing the experiments and how the data from them is analyzed.

Some common error messages or instrument issues

- "robot arm didn't move" first, try again. If that doesn't work, try typing sethw('robot', 'index') in the command line, if that doesn't work, talk to Dave or Maddy.
- Your spectrum has a rolling baseline use "region bc" in the VnmrJ command line or try linear prediction under processing. In MestreNova use any of the baseline correction methods.
- If there is no sample in the location that is chosen, this will give an error try to avoid this by using the empty tube as a placeholder. If someone left the magnet with no sample loaded, just load yours as usual by placing your tube in the next space to the left and typing loc=# change.
- "Motor#0 does not seem to be doing anything" ignore this error. It should not affect your experiments.
- Any other issues please talk to Dave or Maddy instead of trying to fix them on your own. Thanks!

Your account

Because you have your own account on the instrument, you can customize it as you wish.

- e-mail options you can have a plot or a fid e-mailed to any e-mail account you provide. I don't recommend e-mailing a fid because it is a very large file with a poor file type
- auto-plot only if you want it. If you don't plan to keep these plots, please save paper by turning this option off.
- save a plot to the "plots" folder in your account. Recommended.
- automated save data setup defined the same way it is on Indy or Hg3 (sample name becomes the main directory, pulse sequence becomes the subdirectory), but you can define it in many other possible ways if another scheme would work better for you.
- submission mode can be exited automatically after you finish submitting a sample, or you can do this manually