

NMR Safety Guidelines

Compliance with these rules is required in 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 and spinners with a Kimwipe before they go in the magnet and after 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 5-gauss 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.

I have received a copy of these safety guidelines, read them, and will abide by them.

Name of Facility User: _____ Date: _____

IMSS Username: _____ PTA #: _____

Email Address: _____ Advisor's name: _____

Status: Postdoc____ Graduate Student____ Special Student/Visitor____ Other____

Undergraduate, here this summer only: ____ or continuing Caltech student: ____

I have trained the above user in the proper use of the facility instruments and proper safety guidelines.

Name of GLA: _____ Instrument: _____ Date: _____ Account made: _____

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Scheduling and logging in

- Siena is scheduled through a Webcal page. Normally you can readily find instrument time available that same day, although you might want to make a reservation an hour or so in advance of when you want the instrument.
- On weekdays, the maximum length of a reservation between 8 am and 5 pm is 30 minutes. After 5 pm, the amount of time you can reserve is unrestricted until 8 am the next day.
- You can reserve more than one time block in a day, but you should not reserve consecutive time blocks. For instance, don't reserve the instrument from 4:30 to 5 pm, then from 5 pm to 9 pm. We don't want the instrument already tied up in a long experiment as early as 4:30 pm.
- Don't put multiple names down on the reservation schedule to create a block of time longer than 30 minutes for the use of one person during weekday daytimes. Run the longer experiments in the evenings or on weekends, or use another instrument (for instance, daytona or fid, which you can reserve for up to 24 hours starting any time, or florence, which does a lot of the same experiments but much faster).
- You may use the instrument without a reservation when it is not reserved by anyone else. For instance, if you reserve and use the instrument for 30 minutes, but no one else has made a reservation after you, you may keep using the instrument.
- When you arrive to use the instrument, you should see the Linux login screen. Log in as yourself, start VnmrJ, do your experiment, then close VnmrJ and log out of the Linux desktop. Choose the option to log out, **not** the option to change user. Strange things may happen the next time you use the spectrometer if you do not close VnmrJ before logging out of Linux.
- There should be a tube of just D₂O in the probe when you arrive. Put this sample back in the probe when you are done. Make sure that when you leave, you take your own samples, not the D₂O standard. If you break the standard tube, get a new tube, and fill it with D₂O which you can find in the NMR lab fume hood.
- Normally you will be charged for all of the time the VnmrJ window is open, whether you are acquiring data or not. This could lead to a large bill if you start an experiment and leave the window open until you come back. For a long experiment, you may set up your sample, start your experiment, close VnmrJ, and log out of Linux. The experiment will continue running without VnmrJ being open, and you will only be billed for the time the spectrometer is running. However, if you do that, you should leave a note on the keyboard stating that the spectrometer is in use and give the time when your experiment is set to finish so other users know when it would be safe for them to change the sample and start their own experiment.

Nuclei and experiments on siena

- Siena has two probe channels. The high frequency coil is for H1 and F19. The low frequency one is for all the nuclei between P31 (at 161 MHz) and N15 (at 40 MHz) with the exception of some nuclei close in frequency to deuterium, which won't work (for example, Li6).
- The standard experiments provided in VnmrJ potentially all work with the exception of the low frequency nuclei around deuterium and the proton-fluorine double resonance experiments. Proton and fluorine are mutually exclusive choices.
- Sample spinning almost never works properly on siena. It should be disabled in software—on the Spin/Temperature tab, check the box that says “control spinning from this tab only” with the spin turned off.
- In order for a nucleus to be observed with a standard setup, a pulse width and power calibration must exist for this nucleus in the probe file. Ask before you try an oddball nucleus. The current list of calibrated nuclei is H1, F19, B11, Pt195, Sn119, Si29, Cd113, Li7, O17, Ga71, C13, N15, P31.
- You can run a sample without a deuterated solvent being present, a.k.a. no-D NMR. For nuclei other than H1 and C13, this is straightforward, as there normally won't be any solvent peak. Normal locking and shimming

should be disabled if there isn't a deuterated solvent. For H1, a solvent suppressed experiment will typically be used, and special gradient shimming procedures may be required.

- In standard 2D experiments e.g. HMBC, you can substitute F19 for H1 and other spin ½ nuclei for C13, for instance N15, P31, or Si29, and the experiment should work. You may need to adjust the value of the expected J coupling constant.
- You can get a reasonable deuterium spectrum on siena by loading e.g. carbon parameters, changing the observe nucleus to lk (a.k.a. lock, or H2 observe), changing tpwr to 45 and pw to 100. However, the version of this experiment available on florence is many times faster because of the cryoprobe on florence.
- Variable temperature operation on siena may work for temperatures not very far above ambient, for instance 50-60 C to check for amide group rotamers. You need to watch that the variable temperature unit does not error out. Allow time for the probe to warm up and also to cool back down to ambient temperature before the next user arrives. Low temperature operation is not recommended on siena.

Submitting a new study

- Using the automation queue is how Agilent recommends running the instrument. Refer to the Agilent graphic “6 Steps to an NMR Spectrum.”
- Normally the probe tune option will be selected in automation (or probe tuning will be executed manually). We routinely run many nuclei on siena, so tuning the probe is the only way to be sure it is tuned to your desired observe nucleus. But if you run multiple samples in a row in the same solvent and observing the same nucleus, you can omit the probe tuning step and save a couple of minutes per spectrum.
- It is a good practice to load the default shims before gradient shimming your sample, or to enable auto-loading of the default shims in automation.
- The default shims will work best if you make a sample as long as Agilent recommends that it should be (5 cm or “three fingers” of liquid height). If you choose to make your samples shorter, then center the liquid in the tube around the probe center line marked in the sample depth gauge, rather than just pushing the tube to the bottom of the gauge. The correct position for the siena depth gauge is 70 mm.
- Pay attention to the lock phase—it may stay constant for long periods of time if there aren't any power outages or instrument shutdowns, but after either of those, the lock phase will settle in a new random value. The current value is noted on a piece of paper taped to the computer monitor.
- Manual operation, or a workflow combining manual and automated steps, is also possible. With manual data acquisition, spectra are not normally auto-saved, so be sure to save your spectra manually.

Scheduling and logging in on hg2

- Hg2 is scheduled through a Webcal page. Normally you can readily find instrument time available within minutes, or just start your experiment and then check the schedule to make sure you aren't using someone else's reserved time.
- The maximum length of a reservation is 10 minutes
- You can reserve more than one time block in a day, but you should not reserve consecutive time blocks.
- You may use the instrument without a reservation when it is not reserved by anyone else. For instance, if you reserve and use the instrument for 10 minutes, but no one else has made a reservation after you, you may keep using the instrument.
- When you arrive to use the instrument, you should see the Linux login screen. Log in as yourself, start VnmrJ, do your experiment, then close VnmrJ and log out of the Linux desktop. Choose the option to log out, **not** the option to change user. Strange things may happen the next time you use the spectrometer if you do not close VnmrJ before logging out of Linux.
- There should be a tube of just D₂O in the probe when you arrive. Put this sample back in the probe when you are done. Make sure that when you leave, you take your own samples, not the D₂O standard. If you break the standard tube, get a new tube, and fill it with D₂O which you can find in the NMR lab fume hood.

Nuclei and experiments on hg2

- Hg2 has a fixed frequency 4 nucleus probe, for H1, C13, F19, and P31. Compared to our other instruments, C13 sensitivity is low, and you should only use it if your sample is quite concentrated. It will do simple, high sensitivity 2D experiments such as a COSY.
- You can run a sample without a deuterated solvent being present, a.k.a. no-D NMR. For F19 or P31, this is straightforward, as there normally won't be any solvent peak. Normal locking and shimming should be disabled if there isn't a deuterated solvent. For H1, a solvent suppressed experiment will typically be used, and special gradient shimming procedures may be required.
- In contrast to siena, sample spinning works on hg2, and should be used for optimal spectrum quality. The box "control spinning from this panel only" should be un-checked on the Spin/Temperature tab.

Submitting a new study

- Using the automation queue is how Agilent recommends running the instrument. Refer to the Agilent graphic "6 Steps to an NMR Spectrum."
- It is a good practice to load the default shims before gradient shimming your sample, or to enable auto-loading of the default shims in automation.
- The default shims will work best if you make a sample as long as Agilent recommends that it should be (5 cm or "three fingers" of liquid height). If you choose to make your samples shorter, then center the liquid in the tube around the probe center line marked in the sample depth gauge, rather than just pushing the tube to the bottom of the gauge. The correct position for the siena depth gauge is 70 mm.
- Manual operation, or a workflow combining manual and automated steps, is also possible. With manual data acquisition, spectra are not normally auto-saved, so be sure to save your spectra manually.

Vnmrj



6 Steps to an NMR Spectrum

- 1 New Study
- 2 Choose Experiment in Selector
- 3 Provide Sample Information
- 4 Double-Click Experiment in the Queue
- 5 Set Parameters
- 6 Click Submit

1 New Study

2 Experiment Selector

3 Sample Information

4 Experiment Queue

5 Acquisition options

6 Submit

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