

**Standard Operating Procedure (SOP): High field Nuclear Magnetic Resonance**  
**(Oxford NMR AS400 MHz) NMR Training and Reservation**

1. NMR-Training is required prior to independent use Please contact Dr. Syed Kaleem Ahmed ([ahmedsy@usc.edu](mailto:ahmedsy@usc.edu)) to schedule training.
2. All users of NMR are required to use the FBS scheduling ([FBS - Scheduling](#)) system to reserve the equipment.
3. Hours of operation: Monday to Friday 9am to 5pm, except holidays.
  - a. Maximum reservation during normal business hours is two hours.
  - b. If a new user requires to schedule NMR for more than 2 hours after 5 pm (Monday to Friday) please contact Dr. Syed for assistance ([ahmedsy@usc.edu](mailto:ahmedsy@usc.edu))
4. NMR is in PSC 402E. Users need key card access to 402B to enter the room. Manual keys to 402E can be checked out from PSC B4 by signing the log form. Keys need to be returned to PSC B4 immediately after use of NMR.
5. 402B has equipment that are part of TRLab core. Please do not touch any equipment in 402 B.
6. **USERS MUST ENTER PSC 402E WITH APPROPRIATE PPE.**

**Being in the NMR laboratory:**

1. No one must enter the NMR room (PSC 402E) with magnetic implants like a pacemaker (heart rate stabilization device). A person who has been in a war zone and been exposed to, for instance, a grenade which has left iron shrapnel in the body must not enter the premises. A person with other implanted magnetic material must not enter the NMR areas. Magnetic materials in the body might move upon exposure to the large magnetic fields. Not obeying these rules can result in severe harm or death.
2. Do not use and dispose any magnetic materials such as a staple, a screwdriver, and other small tools in the NMR rooms. A lost staple can stick to a magnet and be impossible to remove.
3. The NMR rooms are equipped with oxygen (O<sub>2</sub>) sensors. These sensors will beep loudly when the oxygen level falls below life sustaining level due to a slow magnet quench. The rooms must be left immediately when these sensors start beeping, and please inform Dr. Syed (406-546-4238) or DPS (323-442-1000)
4. A fast magnet quench occurs very seldom. A fast magnet quench is seen by a large plume of gas coming out of the top of the magnet. Evacuate immediately. Press the fire alarm to evacuate the building. In case of an extreme evolution of Helium or Nitrogen gas from one of the NMR magnets, a safety plate at the bottom of the magnet will fall out (to avoid explosion of the magnet vessel) with release of very cold liquid helium and/or nitrogen. In this case the gas pressure in the rooms will rise considerably and the people in the laboratory will be exposed to a triple hazard. The high gas pressure will blow open the aluminum safety windows to release the high gas pressure. In extreme cases the doors will be pushed open into the outside corridors. The wooden frames might be removed from the wall. **Firstly**, the liquid is extremely cold, and the individuals present can sustain frostbites.

**Secondly** the individuals will not be able to take up sufficient oxygen and must leave immediately to avoid suffocation. **Thirdly** the high pressure might inflict bodily harm.

5. No documentation indicates harmful effects upon the fetus or mother in pregnancy from the permanent magnetic fields around NMR magnets. The policy of the NMR lab is to inform about this and at the same time inform about the recommendation for anyone to avoid being inside the industry norm of 5 Gauss of magnetic field for prolonged periods.

Moving close to the NMR magnets with credit cards, ATM cards and similar cards erases these cards. Watches might be destroyed.

### **Preparing sample:**

1. Use the Correct Quantity of Material. For one dimensional  $^1\text{H}$  spectra of organic compounds (except polymers) the quantity of material required is often about 5 to 20 mg. It is possible to obtain spectra from smaller quantities, but at very low concentrations, the peaks from common contaminants such as water and grease tend to dominate the spectrum.  $^{13}\text{C}$  is six thousand times less sensitive than  $^1\text{H}$ , and a good rule of thumb is to provide as much material as will give a saturated solution. (If about 0.25 mill moles can be dissolved in 0.5 ml, the  $^{13}\text{C}$  spectrum will take no more than about half an hour to acquire. If the quantity of material is halved, the data accumulation time will be quadrupled. You should be aware that if you make up a sample at high concentration for  $^{13}\text{C}$ , and then record a  $^1\text{H}$  spectrum from it, the increased solution viscosity may result in a spectrum that has broader lines than you would get from a more dilute solution.
2. Make Samples to the Correct Depth. The samples must be 4 cm high in NMR tube or 0.7 ml solvent. Shorter samples are very difficult to shim and cause considerable delay in recording the spectrum. Samples that are too long are also difficult to shim and are a waste of costly solvent. You should check your sample depth using a ruler. After preparation, you should ensure that the cap is pushed fully onto the tube to minimize solvent loss through evaporation.
3. Remove Solid Particles. Solid particles distort the magnetic field homogeneity because the magnetic susceptibility of a particle is different from that of the solution. A sample containing suspended particles thus has a field homogeneity distortion around every single particle. This causes broad lines and indistinct spectra that cannot be corrected. So that there are no solid particles in your samples, you must filter ALL samples into the NMR tube. You should filter samples through a small plug of glass wool tightly packed into a Pasteur pipette. If the plug is not tight enough, the filtration will be ineffective; if it is too big, some of your sample will remain trapped in it. Do not use cotton wool, since most NMR solvents dissolve material from the wool, which can easily be seen in  $^1\text{H}$  spectra. After filtration the sample should be as clear as water though, of course, not necessarily colorless.
4. Use Clean Tubes and Caps. NMR-users Tubes must be capped. You must not use NMR tubes with a chipped or broken top because they are dangerous, and very likely to splinter lengthwise.

5. Use Deuterated Solvents. Samples must be prepared using solvents that contain deuterium in place of hydrogen. The NMR signal from the deuterium nuclei is called the NMR lock and is used by the spectrometer for stabilization. Deuterated solvents (CDCl<sub>3</sub>, D<sub>2</sub>O, DMSO-d<sub>6</sub>, CD<sub>3</sub>OD, Acetone-d<sub>6</sub>, Acetonitrile-d<sub>3</sub>, Benzene-d<sub>6</sub>, etc.,).
6. Transport the NMR tube in a suitable secondary container that will contain any spills and prevent injuries to the individual transporting it.

**Equipment Use: Inserting and removing samples:**

1. Label your sample: This is best done with a permanent marker directly on the top of the tube, or on the cap. If you use a sticker or a piece of tape, your label must stick smoothly on the tube. Remember that the tube must spin (1200rpm) while it is in the magnet. If a sticker is lost in the magnet a lengthy procedure has to be performed to remove it.
2. **Depth Gauge.** Do not put the spinner into the depth gauge before inserting the tube into the spinner. It is very easy to break off the bottom of the tube if the spinner is held too rigidly. Put the tube and spinner into the depth gauge and push the tube to the bottom. If you break a tube while inserting into the spinner, please clean the spinner with isopropanol. Use no other solvent. Wear gloves if necessary. Check that there is no glass inside the spinner. Do not put the depth gauge alone or together with the spinner/tube into the magnet. If you by accident, do it, please report the incident by sending an email to [ahmedsye@usc.edu](mailto:ahmedsye@usc.edu) immediately. Please leave a note in front of the computer screen describing the incident so other users can plan for not using the NMR instrument.
3. **Do not lean on magnets:** Do not lean on the top of the magnet. It might fall over.
4. **Remove your samples:** Remove your samples from the NMR when finished. Sample tubes left in the lab will be discarded.
5. **<sup>31</sup>P, <sup>19</sup>F-NMRs:** If you are recording <sup>31</sup>P, <sup>19</sup>F-NMRs manual tuning is required, for assistance please email [ahmedsye@usc.edu](mailto:ahmedsye@usc.edu)

**Contact person:** Dr. Syed K Ahmed, Ph.D.  
 Associate Director- Medicinal Chemistry Core  
[ahmedsye@usc.edu](mailto:ahmedsye@usc.edu), PSC-B-4  
 Phone: (office) 213-749-7618  
 (cell) 406-546-4238

**By typing your name below, you agree with the NMR policies and procedures to tightly follow them.**

Name of User

\_\_\_\_\_  
 Signature

\_\_\_\_\_  
 Date

**By signing below, the Principal Investigator agrees to assume responsibility for the proper use of equipment and to be fully liable for the cost of repairs/replacement due to damage resulting from improper use by his/her research personnel.**

\_\_\_\_\_  
 Name of PI

\_\_\_\_\_  
 Signature

\_\_\_\_\_  
 Date