



# Chemistry Department

## NMR/Instrumentation Facility

### *Users Guide - Rules, safety and system information*

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The NMR/Instrumentation facility at the Chemistry Department at Kansas State University is equipped with two Varian NMR spectrometers:

- Gemini 200MHz
- Unity Inova 400MHz

These instruments are devoted to the structural characterization of organic and inorganic compounds, and to the study of chemical reactions and chemical interactions between molecules.

This manual provides users of the NMR Facility at **K-State University** on rules, safety and system information:

- Lab rules
- Sign-up rules
- Safety: cryogenics and magnet quench
- System components

This information must be read and understood by **all NMR users**

# K-State NMR Facility

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## Lab Rules

- All users must be trained by the lab manager (at CB 126 or by email [leila76@ksu.edu](mailto:leila76@ksu.edu) before using the instruments.

### **No unauthorized training.**

- Each user will be given a user name and password for scheduling time and for using the instruments.
- All users must sign the log book when using the instruments.
- Do not leave your NMR tubes in the NMR lab, they will be tossed in the trash, or become lost.
- Please do not leave Kimwipes, unwanted spectra, sample tubes, etc., lying around.
- Do not bring cell phones, headphones, iPods or the equivalent of these into the NMR lab.
- No gloves while using the NMR instrument. Do not contaminate keyboard, mouse, etc.
- If a sample breaks in a magnet, contact the NMR manager immediately, at CB 126 or by email [leila76@ksu.edu](mailto:leila76@ksu.edu) - *Do not attempt to remove the probe.*
- If a sample is stuck in a magnet, contact the NMR manager immediately, at CB 126 or by email [leila76@ksu.edu](mailto:leila76@ksu.edu) - *Do not change any of the pressure regulators.*

## NMR sign-up

- Reservations to use the instruments are done online at <http://faces.cerc.uga.edu>
- Login as Group: **KSUNMR** and use your user name and password
- Use the “Choose a schedule” list to select the instrument you wish to reserve.

### Sign-up Rules

- Experiments that require more time should be performed overnight; exceptions will be made by the manager for students that need help to set up the experiments.
- Standard **Carbon-13** experiments should not be performed during the day. If a student needs to use the instrument after 30 minutes of the carbon acquisition, your experiment will be stopped.
- You can schedule time slots up to 48 hours in advance of the starting time of the reservation.
- **You cannot reserve consecutive time slots.**
- You are limited to 2 standing reservations per instrument per day, and a total of 3 future reservations per instrument.
- **Weekdays:** from **8:00 AM** to **6:00 PM** is for walk up experiments or you may reserve 30 minute time slots.
- **Weekends and overnight:** you may reserve time slot of up to 10 hours. On weekends the time between 12:00 PM (noon) and 4:00 PM is for walk-up experiments.
- **Overnight:** slots are divided in two:
  - 6:00 PM to 10:00 PM
  - 10:00 PM to 8:00 AM
- You should not reserve the time slots during the same night. You should reserve just the time necessary to complete your experiment.
- You should delete any reservations that you are not going to be using ahead of time, to allow someone else to use the instrument.

You may leave instructions to the next user to save your data by writing on a sheet a paper and placing it on top of the keyboard the following information: your name, time and date the experiment is expected to finish. The file name and directory where you want the file to be saved.

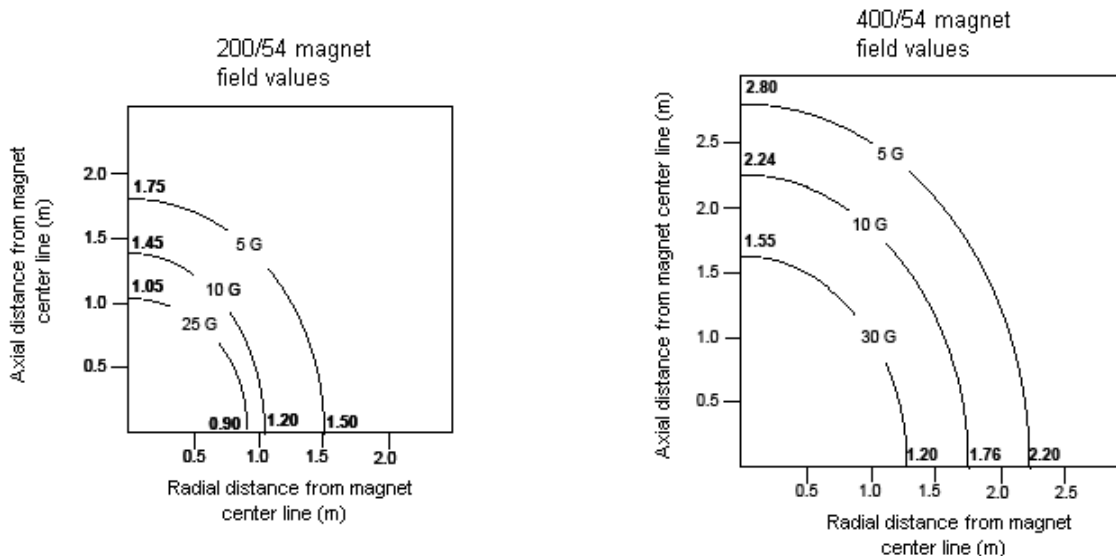
**Remember!! Your experiment and your sample are your responsibility!!!**

**If you have special needs please talk to the lab manager in CB 126 or by email [leila76@ksu.edu](mailto:leila76@ksu.edu)**

# SAFETY

## MAGNETIC FIELD HAZARD

The gauss lines describe the distance from the center of the magnet where a magnetic field strength is experienced. At the **5-** and **10-gauss** lines a strong magnetic field can act upon ferromagnetic objects. Unless restrained, these objects can suddenly fly toward the magnet, causing possible personal injury and significant damage to the NMR instrument.



System	5-gauss line	10-gauss line
<b>Gemini 2000</b> (200MHz magnet)	1.5-1.75m (4.9-5.7ft)	1.0-1.5m (3.3-4.9ft)
<b>Unity Inova 400</b> (400MHz magnet)	2.0-2.8m (6.6-9.2ft)	1.8-2.2m (5.9-7.2ft)

## WARNINGS

Laboratory access only to **authorized users**.

### The magnetic field is always on!!



Keep all metal objects outside the **10-gauss** perimeter from the centerline of the magnet.

The magnetic fields can affect the cardiac pacemakers and prosthetic parts. Persons with implanted medical devices must remain outside the **5-gauss** perimeter.

Metal objects (e.g. chairs) must remain outside the **5-gauss** perimeter.

Keep magnetic media, credit cards, watches, cellular phones, laptops outside the **5-gauss** line.

Minimize time within the **5-gauss** line.

Do not operate NMR spectrometers in the presence of flammable gases or fumes. Flammable gases or fumes create the risk of injury or death from inhalation, fire and explosion.



Be aware of your sample's potential chemical or biological hazard.



In addition to the computer hardware, the NMR lab contains electrical equipment operating at high current and voltages. These are contained within the cabinets/consolas and are connected to the magnets via cables. The risks with electrical/RF hazards are similar to those in the use/maintenance of other lab equipment. Changes to cables and wiring should only be made by the NMR Facility manager.

**Do not walk behind consoles or around magnets.**



The exposed cables close to the magnets are a potential trip hazard. Only authorized users should be within these areas.

**Use care around platform/legs of magnets; do not use magnets as a support.**

Only carbon dioxide fire extinguishers should be used to avoid equipment damage and exceptional care is needed to ensure that fire extinguishers are not used near the magnet.

## CRYOGENS



The main risks in the NMR lab are burns when handling cryogenics and asphyxiation if a magnet quenches.

**Cryogenics** in the facility are liquid nitrogen and liquid helium:

Temperature: nitrogen:  $-196^{\circ}\text{C}$  (77K) and helium:  $-269^{\circ}\text{C}$  (4.2K)

**Only individuals who have had special training should transfer cryogenics to the instruments.**

### Cryogen fills

The **liquid nitrogen** fills are done *once a week*, while the **liquid helium** fills is done *every 5 weeks*.

*You won't be able to run any experiment during the fill.*

It takes approximately 45-60 minutes to completely fill each magnet.

When liquid helium fill is being performed, a sign will be placed outside the NMR lab. Users should not enter the lab during the liquid helium fill.

The possibility of a **quench** is higher when filling magnets wait for the fill to be done; and the Dewar and hoses removed away from the magnet in order to use the instrument.

## MAGNET QUENCH

A quench refers to the sudden release of gases from the magnet Dewar. This happens when a portion of the superconducting wire becomes resistant, generating heat causing the cryogens to boil.

The causes for a quench can be problems during liquid helium fill and the attraction of a large ferromagnetic object, such as a chair or ladder against the magnet.

*The quench will be obvious by the noise of the escaping gas and clouds of vapor.*

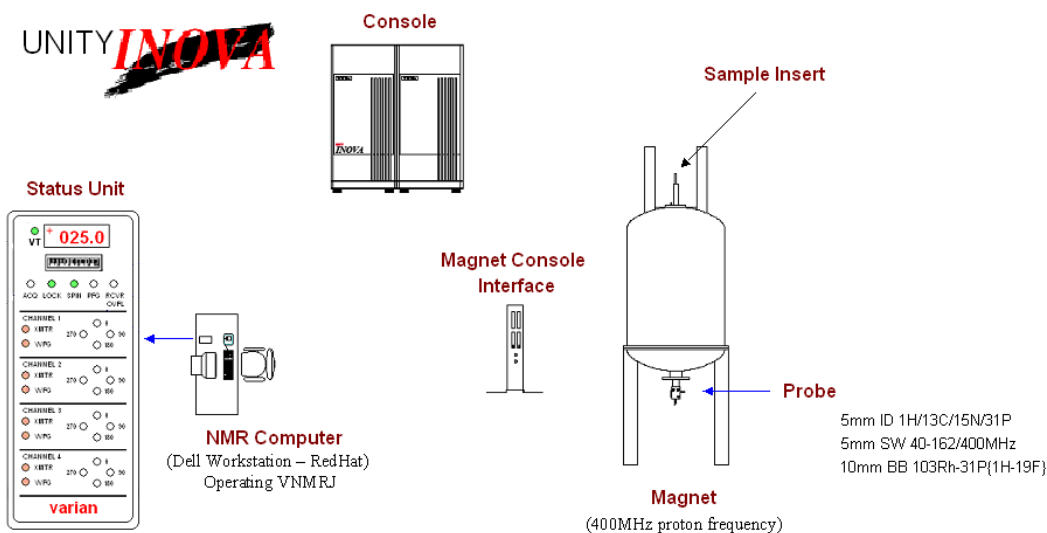
The rapid expansion of liquid helium or nitrogen gas can displace breathable oxygen in an enclosed space. This is extremely dangerous condition since exposure to atmospheres containing 8-10% or less oxygen will cause unconsciousness without warning and the possibility of asphyxiation.

**In the event of a quench leave the NMR lab immediately.**

## Inova 400 Information

The instrument is configured with a two-channel detection system, with pulse field gradients and a variable temperature controller.

The Inova 400 (400MHz) instrument consists of six major parts:



### NMR workstation

The NMR instrument is controlled by a computer workstation running Red Hat operating system. The spectrometer is controlled by the **VNMRJ** software. The software directs the NMR experiments from start to finish, controlling radio frequency (**rf**) pulses for acquisition and the signal processing to convert the FID into a spectrum.



### Status Unit

The **Status Unit**, which is placed next to the workstation, contains a number of system status indicators. These display activity in each radio frequency channel, lock system, gradients, receiver, spinning and temperature. The instrument should always be left on a “standby” mode where the lock and spin lights are **green**.

### NMR console

The console contains electronic components, including transmitters, amplifiers and receivers. It controls the pulses and acquisition process, signal digitization and magnet homogeneity.

## Magnet

The magnet operates at 400MHz frequency for protons (9.4 tesla). It is a superconducting magnet, which has an electromagnet, made of superconducting wire. It contains an outer Dewar filled with **liquid nitrogen** and an inner Dewar with **liquid helium**, where the solenoid coils are immersed.

## Magnet Console Interface

The **Unity Inova 400** has an additional unit – **magnet console interface** that contains the tune control, air supply controls and preamplifiers.

## Probe

It is the part that accepts the sample, sends radio frequency (**rf**) energy into the sample and detects the signal emanating from the sample. It is placed inside the magnet, bore – a hollow tube at the center of the magnet.

Three probes are available for the 400MHz unit:

- 5mm [ $^1\text{H}/^{13}\text{C}/^{15}\text{N}/^{31}\text{P}$ ] indirect detection probe
- 5mm 40-162/400MHz switchable probe
- 10mm  $^{103}\text{Rh}-^{31}\text{P}\{^1\text{H}-^{19}\text{F}\}$  broad band probe

The probe typically in use for the Inova system is the [ $^1\text{H}/^{13}\text{C}/^{15}\text{N}/^{31}\text{P}$ ] ID (**indirect detection**) probe designed for more demanding applications in high-field NMR, such as heteronuclear and multidimensional experiments. This probe is equipped with Z-gradients. The probe has highest sensitivity for  $^1\text{H}$  experiments and it is the best probe for 2D homonuclear (COSY, NOESY) and 2D heteronuclear inverse experiments (HSQC, HMBC)

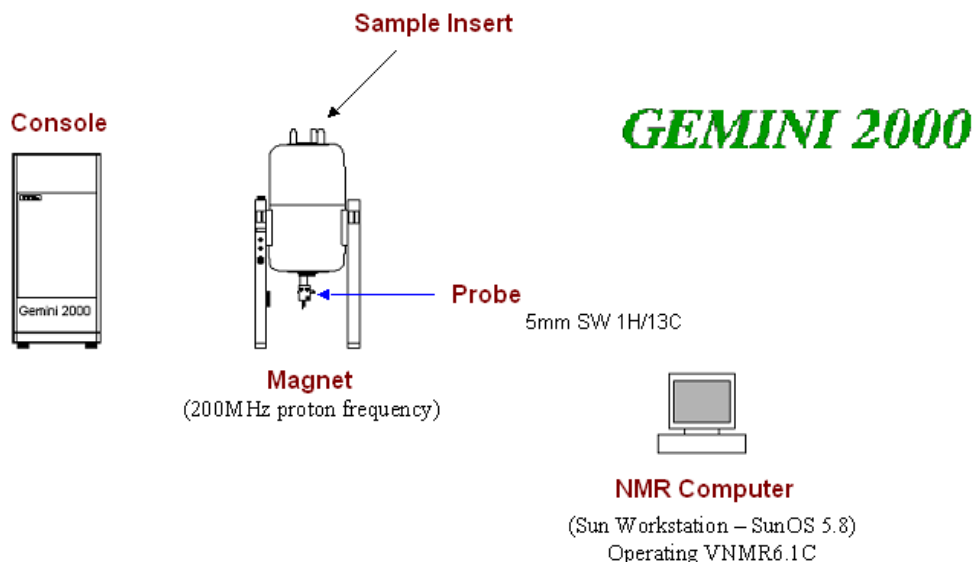
The 40-162MHz SW (**switchable**) probe enables observation and decoupling of high-frequency and low-frequency broadband nuclei, such as  $^{15}\text{N}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ .

The 10mm for  $^{103}\text{Rh}-^{31}\text{P}\{^1\text{H}-^{19}\text{F}\}$  BB (**broadband**) probe is a multinuclear probe optimized for improved performance for the lower frequency nuclei (14-162MHz).

## Gemini 2000 Information

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The Gemini 2000 (200MHz) instrument consists of four major parts:



### NMR workstation

The NMR instrument is controlled by a Sun computer workstation, running **VNMR 6.1C** software. The software directs the NMR experiments from start to finish, controlling radio frequency pulses for acquisition and the signal processing to convert the FID into a spectra.



### NMR console

The console contains electronic components, including transmitters, amplifiers and receivers. It controls the pulses and acquisition process, signal digitization and magnet homogeneity.

### Magnet

The magnets operate at 200MHz frequency for protons (4.6 tesla). It is a superconducting magnet, which has an electromagnet, made of superconducting wire. It contains an outer Dewar filled with liquid nitrogen and an inner Dewar with liquid helium, where the solenoid coils are immersed.

### Probe

It is the part that accepts the sample, sends radio frequency (**rf**) energy into the sample and detects the signal emanating from the sample. It is placed inside the magnet, bore – a hollow tube at the center of the magnet. The **Gemini 2000** system uses a 5mm  $^1\text{H}/^{13}\text{C}$  SW (**switchable**) probe, which enables observation of  $^1\text{H}$  and  $^{13}\text{C}$  and decoupling of  $^1\text{H}$  in  $^{13}\text{C}$  experiments.