

NMR Service Rules

Before you start using the NMR-Service

Register yourself at the NMR service mailing-list:

<https://dlist.server.uni-frankfurt.de/mailman/listinfo/nmrservice-nutzer>

Read the article on safety in the NMR rooms.

Each authorized user of the spectrometers is given a personal user code and a password for his workgroup.

Instruments may be used by anyone who has been trained by the NMR supervisor and who has a current four-character code.

Do not share your code or password with anyone. Violators may lose access privileges.

Interns and bachelor students are only allowed to use the 250MHz spectrometer under the responsibility of their supervising PhD student. They use the 4 letter code of their supervising PhD student or postdoc supplemented by a two letter code from their initials.

General policies

Do not bring backpacks or metallic / magnetic stuff near the spectrometers.

After completion of the experiments fetch your samples from the carrousell as early as possible. Otherwise your sample is removed next day and stored for max. 1 week.

Any removing of a sample whose experiments are not completely done is allowed only with deleting the queued experiments from the setup table at the same time to avoid shut-down of the samplechanger.

If a sample breaks in a magnet, contact the NMR Manager immediately, so the probe has to be cleaned. This will minimize probe damage from the solvent and sample. Repairs of damage to probes, or other equipment, arising from user error will be paid for by the user group.

All users must sign in the elog supplying all information asked for.

Scheduling NMR Time

In NMR service routine the spectrometers are running under TOPSPIN / ICONNMR. The samplechanger machines are configured for fast access on daytime. Most longer experiments should be run over-night or over the weekend.

Spectrometers

All spectrometers have their own specialty- while it is possible on some of them to run more than the specified experiments, this is what is recommended:

DPX250: short ¹H spectra.

AV300: direct detected heteronuclear spectra

AV400: 2D spectra , homo and heteronuclear (e.g. COSY, NOESY, HSQC)

AV500: highest sensitivity for protons, 2D spectra , homo and heteronuclear, direct detected heteronuclear spectra

DRX600: highest resolution, 2D spectra , homo and heteronuclear (e.g. COSY, NOESY, HSQC)

Note: spectrometer time is only half the price at 250-400MHz compared to 500 and 600MHz

Service work – special samples

Samples may be submitted for the NMR administrator to run for you. Please fill out a sample submission form.

If you want to measure without deuterated solvent please contact the NMR manager.

If the contents of your tube is known to be explosive or very dangerous to health you must not use the sample changer.

Other non-routine work, e.g. neat liquids, variation of temperature or special tubes (10mm or different types of microcells) can be done only outside the sample changer mode. In such cases please contact the NMR administrator.

Data filenames

Each service user is requested to use the data filename convention as follows:

<machine>-<year>-<month>-<day>-<usercode>.<elog-number>

e.g. AV500-2017-12-05-jwhs.1234

Archiving data

All data files from ICONNMR-users are stored automatically and can be download from server “neon” Z:\
<machine>/data/<groupname>/nmr/<filename>

NMR Sample Preparation and placing it in the magnet

NMR tubes and caps

Each working group uses a differently colored cap for the NMR-tube and different color for writing on the sample tube.

Each tube has to be labeled with water proofed signs, the user code is an obligatory part of the label.

You can buy NMR tubes and caps in the NMR-service department

Drying of NMR tube should be performed in horizontal position not exceeding 50 degrees centigrade to avoid bending of tube and damaging the NMR probe head.

Minimum tube length is 18cm. NEVER use a chipped or cracked NMR tube.

Sample preparation

Using the Sample changer your samples must contain at least 15% deuterated solvent and a total of 0.6ml solvent. The optimal filling height of the 5 mm tube is 4 cm. For realization of good signal shapes the solution inside the tube has to be of low viscosity, clear and free of paramagnetic or suspended matter or gas bubbles.

If your sample is sealed, be sure that there is no pressure developing inside and perform a burst test before coming to the NMR-service.

Placing the sample in the magnet

First: check in the elog system which is the next free place in the sample changer for your sample.

The NMR tube has to fit tightly to the rotor. If you are in doubt try another rotor. Loose rotors will cause operational faults. Center the tube in the rotor using the provided device.

Please hold the rotor at the top only. Always wipe the rotors body and the outside of your tube with a clean tissue before placing it into the carrousel. This step will keep the turbine clean and free from contaminants and skin oils.