

NMR Guidelines

Safety

DO NOT ENTER THE NMR LABS IF YOU HAVE A PACEMAKER OR ANY METAL SURGICAL IMPLANTS

- The NMRs are super conducting magnets so any metal must be removed before approaching the magnet and left either on the work up or computer benches
- All the magnets are cooled using liquid helium and there is a separate liquid nitrogen Dewar around this to reduce boil off
- There is a risk of a quench which is when all the cryogenics are ejected from the magnet. This will fill the air with helium and nitrogen gas making the air unbreathable
- All the labs are equipped with oxygen sensors which alarm when the oxygen levels go below 19%
- Evacuate the labs immediately if these alarms sound or you see a white plume erupting from the top of the magnet
- DO NOT ENTER THE LAB WHEN THE CRYOGENS ARE BEING REFILLED
- No lab coats or gloves should be worn in the NMR labs. Safety specs are also not required
- Keep the labs tidy by putting any rubbish including tissues in the bin
- Collect your samples and any sample carriers or anything else you bring to the lab

Sample preparation

- Make sure your tubes are clean and have no cracks or chips
- All samples must be run in deuterated solvent

- Samples must be completely dissolved with no solid left in the tube.
- If your sample has low solubility, you may need to filter your samples
- The solvent must be of a sufficient level (two fingers)
- Make sure that the tubes are tight in the spinners without having to use excess force to insert the tubes
- Always use the depth gauge to ensure the tubes are sitting properly in the probe
- Wipe the probe with a tissue before putting it in the sample changer
- Always put your sample in the correct position of the sample changer before submitting your experiments.
- Please collect your samples once your experiments have run
- Any samples left in the labs will be disposed of at the end of each month

NMR TUBES ARE NOT DISPOSABLE AND CAN BE REUSED

Only use the NMR tubes from GPE which can be obtained from chemistry stores.

Experiments

All spectrometers have multichannel probes and can run 1D experiments for the nuclei; ^1H , ^{13}C , ^{15}N , ^{14}N , ^{11}B , ^{31}P , ^{29}Si , $^{\text{K}}39$, $^{\text{Li}}7$, ^{51}V and ^{19}F . The probes can have a wide range of nuclei, but these must be mapped first. NMR service manager if you require other nuclei.

There are also a wide range of 2D experiments which can give more information about the structure of your samples as well as a range of other uses.

Some of the most common 1D and 2D are;

- COSY: 1H-1H through bond interactions for directly bonded protons
- NOESY: 1H-1H through space interactions works well for small or large molecular weights
- ROESY: 1H-1H through space interactions works well for mid-sized molecules
- HSQC: 1H-13C used to determine single bond correlation
- HMBC: 1H-13C used to determine bond correlations that are up to 4 bonds with single bonds being suppressed
- TOCSY: 1H-1H through bond interactions for protons within a given spin system which can be over multiple bonds as long as they share a spin correlation
- INADEQUATE: 13C-13C used to observe direct interactions of 13C nuclei with single 13C signals being suppressed
- DOSEY: 1H-1H used to determine diffusion coefficients of polymers or sample mixtures this cannot be done I automation so please contact the NMR service manager
- DEPT-135: 13C where CH and CH3 signal are positive and CH2 is negative, quaternary peaks are suppressed
- Solvent suppression: 1H spectroscopy with solvent suppression uses NOESY presat to suppress the largest peak
- There are standard 1D experiment for B11, 19F, 31P and 14N which can be run with or without proton decoupling
- F19HOESY: NOESY experiment for fluorine
- F19COSY: COSY experiment for fluorine
- Multinuclear HMBC: HMBC experiment replacing 13C with 19F, 31P, 11B and 29Si

- T1 experiments: Used to determine the relaxation time of samples
- Q_Proton: Quantitative proton NMR used for reaction monitoring or determining the concentration of mixtures

NMR is versatile and capable of a wide range of techniques, and this list is not exhaustive. If you have an experiment from literature you want to set up or are unsure how to collect the data you require, please contact the NMR service manager.

If you wish to book out one of the spectrometers for longer experiments during the day queue, then please contact the NMR service manager with at least 2 days' notice.

Contact

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