Open-Access NMR Spectrometers

These notes introduce the Chemistry Research Laboratory's open-access automated NMR facilities which comprise a Bruker DPX200 spectrometer (first floor), and four Bruker automated AV400 spectrometers; the AVG400 and AVH400 on the ground floor, the AVF400 on the first floor and Hg400 on the second floor. The purpose of the open-access facilities is to allow researchers in the CRL rapid and simple access to routine spectra of proton, carbon, fluorine, phosphorus and selected other nuclei. They are geared towards fast turnover and hence rapid sample throughput. These notes cover both safety aspects and sample preparation and should be read before attending a training session.

Important safety precautions

Superconducting NMR magnets generate a substantial magnetic field in the surrounding area, and should be treated with respect. The most serious hazard is the possibility of metal objects being attracted to the magnets. The force exerted by the magnets increases very rapidly as separation decreases, so that large objects, such as metal dewars, may be wrenched from your grasp almost immediately after you experience any force at all. So:

1. Never enter the spectrometer room when carrying **anything** made from ferrous metal.
2. No lab. coats are to be worn in the spectrometer labs. (as it is easy to forget metal objects in your pockets).

A second area of concern relates to heart pacemakers. There is a known risk to pacemakers from static magnetic fields and radio-frequency pulses. Anyone in the department required to use a pacemaker **MUST** notify Tim Claridge or Nick Rees and consult with their doctor on this matter. There are many high-field magnets throughout the department whose "stray-fields" extend into corridors and therefore constitute a hazard for pacemaker users. It may not be possible for people in this situation to have access to the NMR facilities.

More minor problems relate to effects of the magnetic fields on personal possessions. Magnetic-strip cards such as cash machine, credit cards or University ID cards are liable to be erased if you are carrying them on your person when close to the magnet. Watch dials may run erratically after repeated exposure to the static field (as their components become magnetised), so should be kept away from magnets. Those with digital displays are not affected.

Introduction to the facilities

The open-access spectrometers are designed in such a way that they are very easy to use for routine ^{1}H, ^{13}C, ^{19}F and ^{31}P 1D and 2D spectroscopy; the capabilities of the various instruments are summarised below. Samples that require more complex analysis should be submitted to the NMR service or you may be trained by the NMR staff to do this yourself. You will be shown how to run spectra when you attend a basic training session, **which you must do before using these instruments**.

Bruker DPX200 *[Organic]:* This operates automatically but with manual sample insertion, providing 1D ^{1}H NMR only.

Bruker AVIII400 (AVF400) *[Organic]:* This spectrometer operates automatically and provides: 1D ^{1}H, ^{13}C, DEPT, ^{19}F and ^{31}P; 2D ^{1}H-^{1}H COSY and ^{1}H-^{13}C HSQC. Between 1pm and 6pm proton, phosphorus and some fluorine experiments have priority. The current status of the automation run and whether your sample has been completed can be checked through its automation history web page: http://flounder-crl:8015/ (log in using avf400 as log-in and password).

Bruker AVIII400 (AVG400) *[Organic]:* This spectrometer operates automatically and provides quick ^{1}H, ^{19}F and ^{31}P spectra during the day (8am – 7pm) and also ^{13}C, DEPT, COSY and HSQC experiments overnight. The current status of

the automation run can be checked through its automation history web page: http://turbot-crl:8015/ (using avg400 as log-in and password).

Bruker AVIII400 (AVH400) *[Organic]:* This spectrometer operates automatically and provides quick ^{1}H spectra during the day (9am – 9pm) and also ^{13}C, ^{19}F and ^{31}P, DEPT, COSY and HSQC experiments overnight. The current status of the automation run can be checked through its automation history web page: http://halibut-crl:8015/ (using avh400 as log-in and password).

Bruker AVIII400 (Hg400) *[Inorganic]:* This spectrometer operates automatically and provides quick ^{1}H, ^{7}Li, ^{11}B, ^{19}F, ^{27}Na, ^{27}Al, ^{31}P and COSY during the day (8am – 7pm) and also HSQC and HMBC overnight. The autosampler will accept both normal and Young's tap NMR tubes.

Sample preparation and handling

The open-access spectrometers attempt to automate some of the more troublesome aspects of spectrometer operations. The automatic lock and shim systems are quite effective, but in order for these to be reliable, to make subsequent operations straightforward and to obtain a good quality spectrum, care must be exercised when preparing samples. This is of utmost importance! Remember: Junk in, Junk out! The following points are most important:

1. Precision 5 mm tubes must be used (available from stores, as are *deuterated* solvents). These should be periodically checked for cracks and for scratches around the bottom of the tubes, and discarded if necessary.
2. Tubes that are shorter than 7 inches are already broken and should be discarded.
3. **Samples must be made up to a solvent depth of 4.0-4.5 cm.** This is an **extremely** important point! If you allow the liquid column to fall below 4 cm, you will find it impossible to lock and shim. Making samples too deep, although less detrimental, will dilute your sample and waste solvent.
4. Care should be taken to ensure that sample tubes are clean on the outside before placing them in the instrument. Contamination of the probe, either by transfer of external dirt or by a sample breakage in the probe, is a very serious matter. You will be shown how to insert a sample correctly into a turbine and then into the magnet or sample changer during your training session. If you break a tube whilst loading a turbine, *clear up the mess* with some methanol. DO NOT USE chlorinated solvents as these destroy the depth gauge plastic.
5. When preparing samples you will typically require the following sample quantities for acceptable results:

200 MHz:	ca. 5 mgs for a proton spectrum
	ca. 50 mgs or more for a carbon spectrum
400 MHz:	ca. 2 mgs for a proton spectrum
	ca. 20 mgs or more for a carbon spectrum

When handling oils, the quantity drawn up into a pipette by capillary action should be sufficient for a proton spectrum. It is good practice to filter your samples before placing them in the NMR tube as floating debris or a cloudy solution can be guaranteed to give a poor result.