

NMR Walk-Up Systems Submission Guidelines

General safety

Remove lab-coat and gloves before entering the NMR laboratories.

No one may enter an NMR lab whilst a cryogenic refill is in progress.

Sample Preparation

Samples must be prepared using deuterated solvent.

Samples must be clear with no significant pieces or substantial precipitate.

Tubes must be 8" long (for use on any system) or 7" long (for use on systems with a sampleXpress autosampler), 5 mm diameter. Tubes fitted with a J. Young, or any other, tap must be less than 10" long including the tap.

Tubes must be undamaged and free from scratches.

Samples must have a 5 cm solvent depth, this corresponds to 0.60 mL solvent in a standard tube.

Labelling

Samples must bear the initials of both the PI (supervisor) and user, have a reference that can be easily traced back to a laboratory notebook which contains full details of the contents of the sample tube, and, if there is space, the solvent.

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'Proper' NMR tube labels, or a permanent marker pen must be used.

Labels must be attached to the top 1.5 cm of the tube, immediately below the cap and must be flush with no 'flag'.

Login

If the Red 'Experiment in Progress' sign is placed in front of the keyboard the system is in use and not available for walk-up. Please use another system.

Users must log into the system using the login their own details. A verbose copy of the label on their tube must be included in the sample details.

Samples must be placed in the carousel before clicking submit.

Always submit your sample in the next available slot. This is the first green slot after the currently running sample.

Adding samples to the carousel

Users submitting samples fitted with J Young type taps must reserve 'Dummy' slots either side of their sample. Users must not submit samples to a slot next to a tube fitted with a J Young tap. If the sample in the tube with the J Young tap is completed it should be removed from the carousel before using the Dummy slots. RO should be set to 0 for tubes with J Young taps so that they do not rotate inside the instrument.

Samples can be added to holders coloured in green (Available) or yellow (Completed). Samples must not be added to holders coloured pink (Queued) or pink with a cross (Failed).

If you remove a sample from a spectrometer before it has run then you must delete it from the queue.

Service samples must not be removed from the carousels. They can be identified by the red and white striped sticker at the top of the tube under neither the label.

Day Queue

A user may not submit more than 3 samples in a row, and no more than 6 in total at any one time. If a user needs to submit 4 or more samples then every fourth, fifth and sixth position in the carousel must be left empty.

No more than 30 mins of experiments may be in the day queue for a single user on a single spectrometer at any one time, with a maximum of 15 minutes for a block of 3 samples.

Experiments are run in order of holder number. I.e. sample in holder 4 will run after sample in holder 3.

Night Queue

The night queue times are detailed on the white boards for each spectrometer. Once the night queue is full no more experiments may be submitted.

Experiments are run on a first come, first served basis.

Booking

When you wish to book a spectrometer you must request permission from CB/DB/RA at least 24hrs before you intend to use it.

Priority

To book a priority slot:

1. At least 1 hour before you run your sample: go to <http://nmrbooking.ch.man.ac.uk/> from any computer.
2. Log in. User: *priority* Password: *27AlNMR* (That's 27Al as in Aluminium)
3. Click Schedule -> Bookings
4. Click the time-slot for the spectrometer you wish to book and enter your name in the 'Title of the Reservation' box.
5. Click Create.

6. Log out

Experiments run with *priority* must submit their sample to a holder *before* the currently running sample. I.e. if the sample in holder 7 is running submit to holder 6.

In general, only 1 priority booking is allowed at any one time - i.e. one instrument at a time.

If a priority booking is required on a second instrument then permission must be granted by a member of NMR staff as there must always be at least one fully-available walk-up instrument in the basement.

If a priority booking is in place then multiple users can exploit that booking, provided that this is agreed with the person who made the booking.

Priority can only be used between 9 am and 6 pm.

Instruments that have a priority slot booked will appear with a 'yellow and black halo' and 'PRIORITY BOOKING IMMINENT' will also be displayed by the queue length on the booking screen outside B14. There will be no indication at the spectrometer itself.

Removing samples from the carousel

To avoid robot failure, users must wait at least 1 minute after the robot has returned their sample to the carousel before removing the sample.

Removed samples must be placed in the relevant sample holder I.E. 'today's samples' or 'yesterday/last night's samples'.

Tubes should be collected by the end of the next working day after the sample is run.

Tubes **MUST** be collected by the end of the following Tuesday.

To place a sample in the carousel users should not lean or reach over other samples. The < and > arrows on the autosampler should be used to access the required position only while the robot is not in motion.

Under no circumstances should a sample be removed from the position where the robot picks samples up from. Use the < and > arrows to move the carousel before removing the sample.

Endnotes

Any failures or errors should be reported to the NMR service staff.

The penalties for noncompliance will be:

1st occurrence: Warning (email).

2nd occurrence: Caution (email cc'd to Supervisors).

3rd occurrence: Minimum 1 week ban.

If a user shares their login details they may be banned indefinitely.

If these guidelines are not adhered to the service staff reserve the right to remove samples from the queue and, where continued violation occurs, restrict access to the NMR systems. If in doubt consult Daryl Bamber, Carlo Bawn or Ralph Adams.