

ICON-NMR

User Guide



Important information!

- Never use broken NMR tubes or any tubes shorter than a full length 7 –inch tube!
- Use at least 500 µL of solvent, otherwise locking and/or shimming may fail
- Guidelines for day-time queue (8am – 7 pm):
 - Try to keep the total experimental time for each sample below 20 mins during the day
 - Avoid queueing several samples in a row if the total time will exceed 20 minutes
 - Remember that you can switch a day experiment to the night queue if you need to run, for example a proton or an HSQC experiment longer than 20 minutes (see IconNMR manual)
- Guidelines for night queue:
 - Avoid experiment times longer than 3 hours
 - For carbon experiments, 3 hours is (in most cases) sufficient to get a ^{13}C spectrum for 3mg of a small molecule ($M_w < 500$), see example spectrum
 - If 3 hours is insufficient, increase the concentration (if possible) or run it on the 600 MHz instrument, see example spectrums for ^{13}C sensitivity comparison
 - Contact Mattias or Tobias if you need time on the 600 MHz instrument. We aim to make it available at least one day per month
- Keep the area clean!
 - **You are responsible for your own sample!** Remove it from the sample changer after it has been analyzed.
 - Put the blue spinners back in the box
 - Samples left too long in the sample changer will be moved to the rack labelled “old samples” in the fume hood
- Avoid using the “Priority” function, especially during the day, unless you have an unstable compound

If you have questions or problems, please contact:

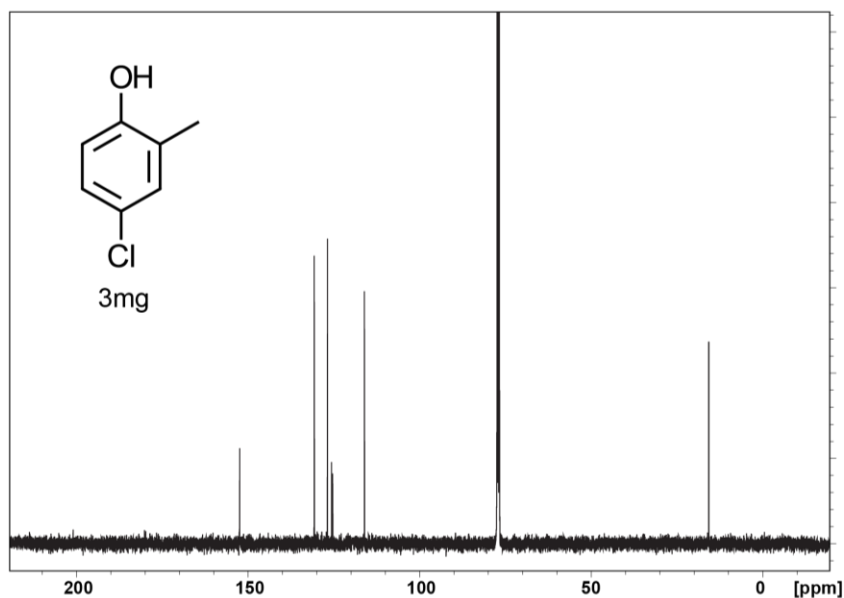
Mattias Hedenström (mattias.hedenstrom@umu.se)

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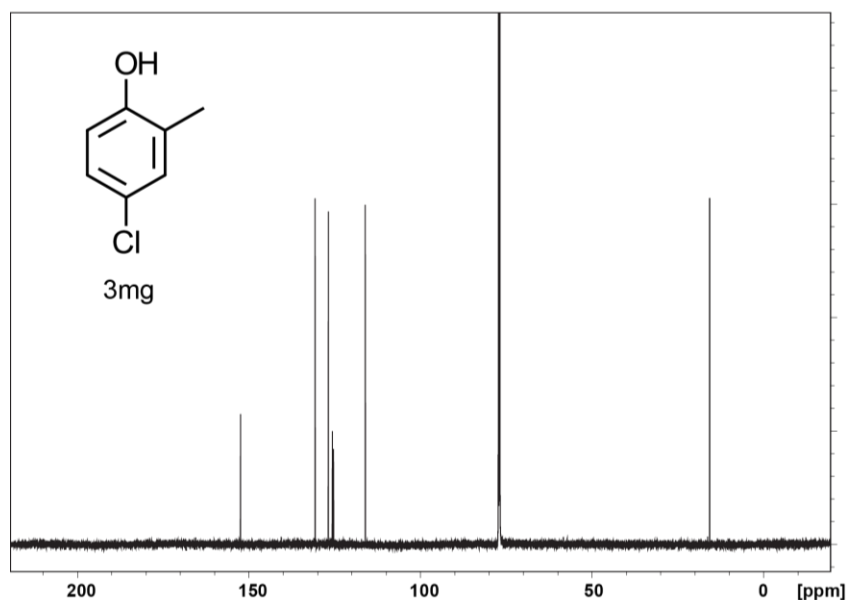
Comparison of ^{13}C sensitivity on 400 and 600 MHz spectrometers

- With our setup, you get 3 times higher S/N-ratio in 6 mins (128 scans) at 600 MHz than 2 hours (2048 scans) at 400 MHz!

400 MHz, 2 hours (ns = 2000), S/N = 32



600 MHz, 6 mins (ns = 128), S/N = 104



Automation window

The Icon-NMR automation window should always be open and is the only window that requires user input for routine measurements together with the BACS sample changer.

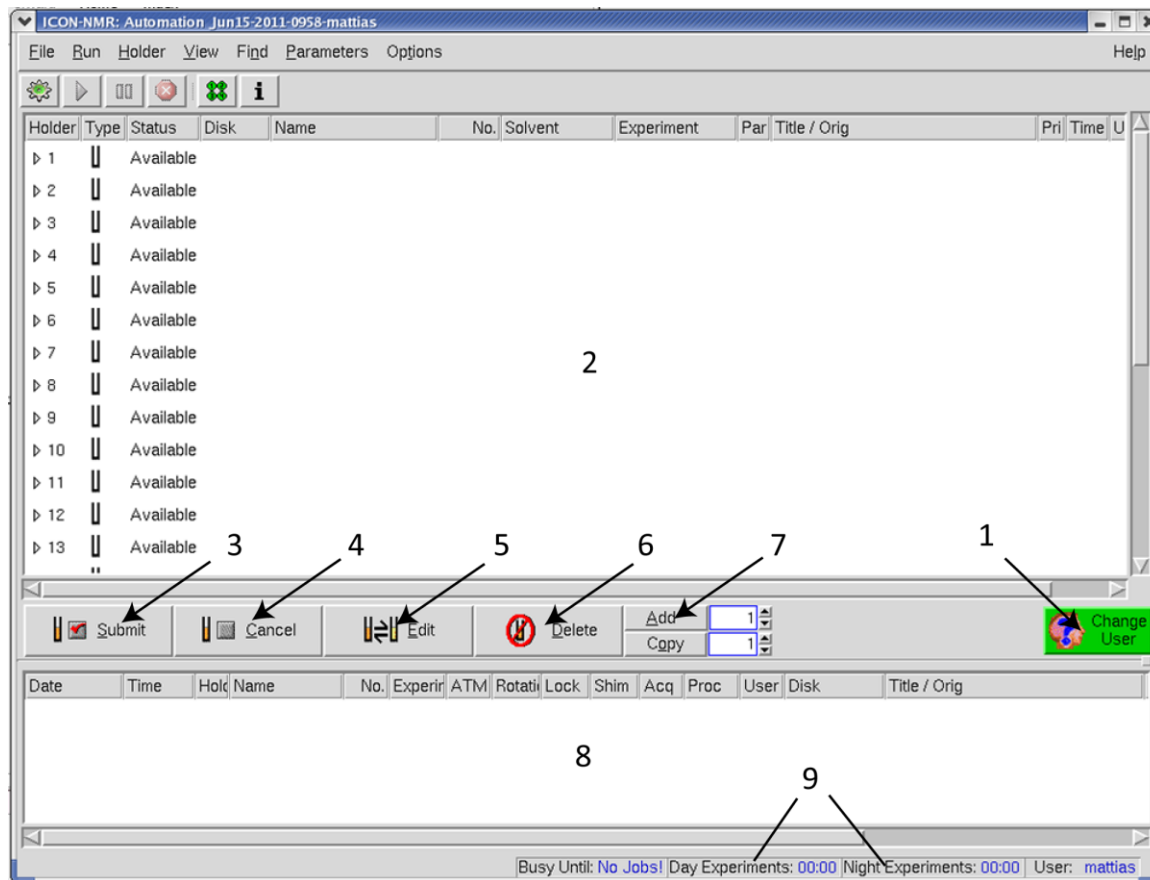


Figure 1. The automation window

1. Switch user
2. Main window where you can see which of the 60 sample positions that are available.
3. Click here to submit an experiment to the queue after you have entered all required fields.
4. Removes the experiment from the queue.
5. Enables you to edit the experiment if you want to change anything to an experiment already in the queue. You have to click the cancel button to remove it from the queue before clicking edit! Don't forget to re-submit it after the change.
6. Deletes an experiment..
7. Click here if you want to run several experiments on the same sample.
8. History. Here you can see the experiments that have been acquired and eventual error messages.
9. Current queue times for day and night experiments.

Adding experiments

To create an experiment, first locate an available position on the sample changer that also is available in the list in the automation window. Double-click on that position in the list to open it, see figure 2. Then simply follow the steps outlined below.

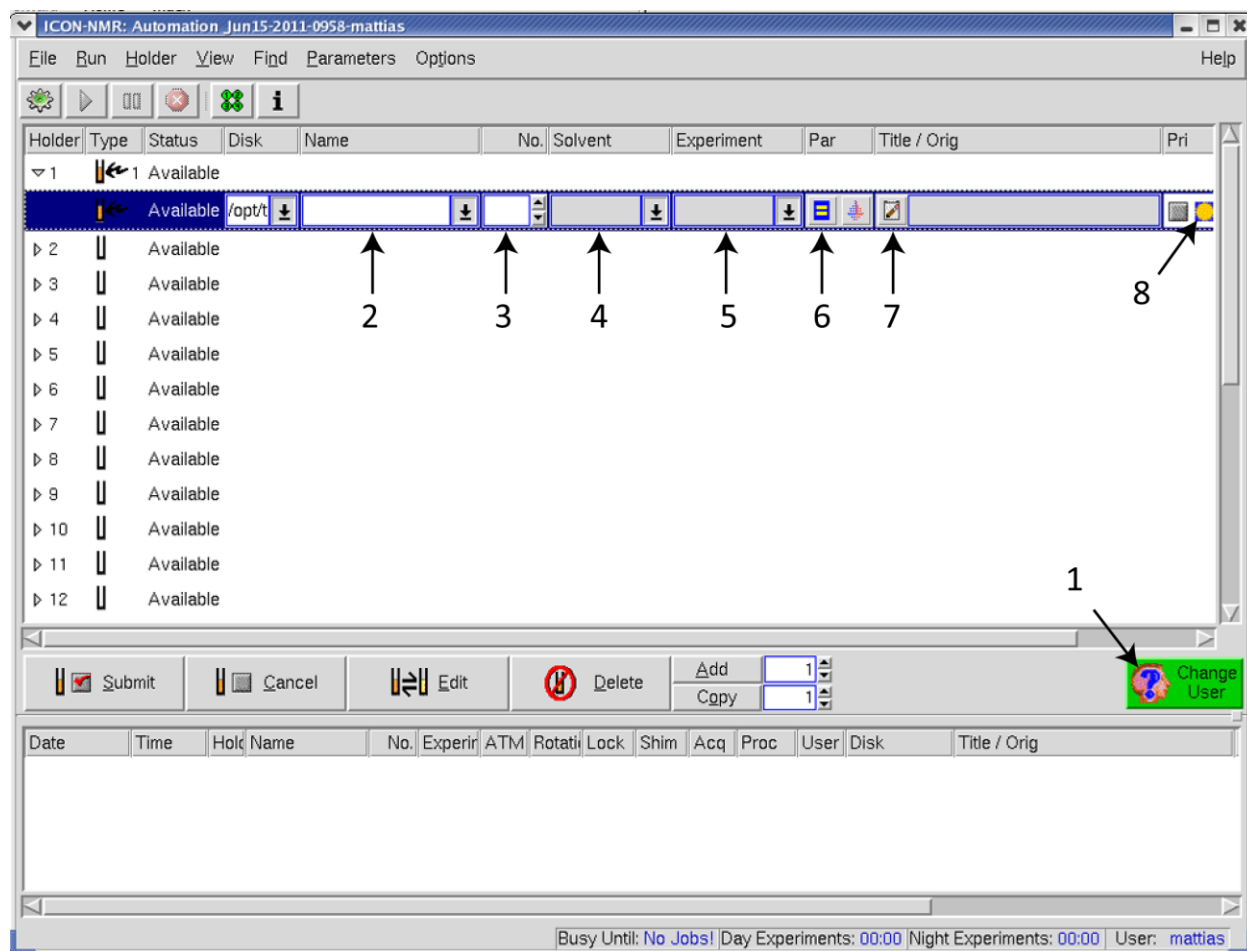


Figure 2. How to add an experiment

1. First you must switch to your user. This is important, otherwise your spectra will end up in another user's directory!
2. Enter the desired experiment name, for example the name of your compound.
3. Set the experiment number, this will be 1 by default for the first experiment for each sample and you shouldn't need to change it. However, if you already have an experiment with the same name and experiment number you must change this or the old experiment will be overwritten.
4. Choose the correct solvent from this drop-down menu.
5. Choose the type of experiment you want to run. Only use one of the experiments at the top of the list with names that start with BACS (see below for details)!
6. **Optional!** Here you can change some parameters such as number of scans and temperature.

7. Optional step but recommended! Give a title to the experiment.
8. **Optional!** Here you can see if your experiment will be run during the day or night. Some experiments, will always end up in the night queue but you can force your experiments to be run during the night by clicking this button. Experiments that are pre-set to be night experiments cannot be changed to day experiments.

If you want to run multiple experiments on the same sample you can click the Add button (see Figure 1) and then repeat steps 3-8. Click the Submit button to add your experiment to the queue.

If you want to change anything to an experiment already in the queue, you need to press *cancel* then *edit*. Remember to submit the experiment after any changes.

Experiments

Only use the experiments listed below

Day-time experiments:

BACS_1H	Normal one-dimensional proton experiment (8 scans)
BACS_19F	19F experiment without proton decoupling (16 scans)
BACS_19Fcpd	19F experiment with proton decoupling (16 scans)
BACS_31P	31P experiment (32 scans)
BACS_C13CPDshort	Carbon spectrum, proton decoupled. (15 min)
BACS_cosy	2D COSY experiment (5 min)
BACS_hsqc	2D 1H-13C correlations (15 min)
BACS_dept135	DEPT135 (17 min), CH2 negative, CH and CH3 positive
BACS_dept45	DEPT45 (15 min), all carbons with protons attached are positive
BACS_dept90	DEPT90 (15 min), only CH-groups

Night experiments:

BACS_TOCSY	2D TOCSY, (30 min)
BACS_roesy	2D ROESY experiment, same information as in a NOESY, (35 min)
BACS_hmbc	2D Long-range 1H-13C correlations (43 min)
BACS_C13CPD	Carbon spectrum, proton decoupled (1 hour)

Remember that you can change the number of scans in all experiments (see step 6 in the previous paragraph) but keep all day time experiment shorter than 20 min if possible and night experiments shorter than 3 hours.