

NEO 300 INSTRUCTIONS - SOP

SAMPLE PREPARATION

- Dissolve your sample (5-20 mg) in 700 μ l of deuterated solvent and transfer it to a 5mm NMR tube
- Write your name/sample code on the top part of the NMR tube
- Clean your NMR tube using a kimwipe.
- Insert the NMR tube in a blue spinner, adjusting its position using the sample depth gauge available
- Place the spinner(s) in the carousel of the autosampler, noting its position(s).

DO NOT:

- Use broken NMR tubes
- If the NMR tube slides too easily in the spinner, change the spinner

AUTOSAMPLER

CAROUSEL

The carousel has 60 positions, numbered from 1 to 60. It moves around the center post in a clockwise manner, by incrementing the holder numbers. Each holder contains a blue spinner.

- Do not put any weight on the carousel
- Do not reach OVER the carousel to put or take a sample
- Current sample position is indicated on the front screen
- Always place your sample in the first available from the center position
- Wait until the arm stopped moving to retrieve your sample.

ICON SOFTWARE (see picture)

A: Login window. If **A**'s not there, click on **B**

C: Preceding experiments. The history of what experiment ran before you logged in. If the top line is red, there was a problem. Check with us that the error has been cleared.

D: The experiment table. Should always match the carousel. This is where you will enter the sample/experiment details.

Traffic light system:

Green: the sample is in the magnet and the highlighted experiment is currently running. It cannot be edited or canceled.

Yellow: the experiment is submitted and waiting to be run. The entry can be edited or canceled by its owner only.

Red: the experiment is completed successfully. The entry can be deleted by anyone.

Black: the experiment had a major failure. It wasn't completed. Do not delete the entry until cleared with staff.

The screenshot shows the ICON software interface. At the top, there's a menu bar with 'Menu Disabled' and 'Do Acquisition'. Below is the 'Experiment Table' with columns: Holder, Type, Status, Name, No., Solvent, Experiment, Pri, Par, Title/Orig, Time. A dialog box titled 'IconNMR: Identify User' is open, showing a list of users with their full names and User IDs. A 'Change User' button is visible in the bottom right. A 'Busy until' field is highlighted with a red arrow labeled 'E'. Other red letters A, B, C, and D point to various elements in the interface.

EXPERIMENT SETUP

- Login in ICON using your username
- Select the holder # corresponding to your sample's position
- Enter the details of your sample and choose the experiment you want to perform
- Enter a title
- You can add more experiments for the same sample using the **add** button
- You can copy the same experiments into another holder using the **copy** button
- Select the holder line and click submit.

You can get an idea of when your sample(s) will be finished by checking “busy until” (E). When your sample is done, you will receive an email notification as well as a PDF of the spectrum. You have then 24hrs to pick up your sample from the carousel and clearing the entry from the software.

EXPERIMENTS AVAILABLE

Starter package includes:

- PROTONRO: ^1H NMR with rotation, 20 ppm
- C13CPD_256: ^{13}C experiment with 256 scans. Ideal for concentrated samples (>20mg/ml)
- C13CPD_overnight: ^{13}C experiment for low concentration (<5mg/ml)
- C13DEPTQ135: ^{13}C DEPT with quaternary. Useful for multiplicity determination.

Other experiments are added upon request.