

Bruker Avance III HD 500 MHz NMR User Guide

The Bruker Avance III HD 500 MHz NMR spectrometer is operated under automation by the interface of Topspin 3.6 ICONNMR. The instrument is equipped with a 5 mm BBFO probe which is capable of performing $^1\text{H}\{^{19}\text{F}\}$ and $^{19}\text{F}\{^1\text{H}\}$ experiments. As for sample preparation, the ideal sample volume is ~ 0.6 mL. To submit your experiments, please follow the step-by-step procedure as below:

Step 1: To log in your account, click “Change User” (circled in red) at the lower right corner of the ICONNMR interface.

The screenshot displays the ICONNMR interface. At the top, the title bar reads "IconNMR: Automation Feb03-2020-1354-cem415". Below the title bar, there is a menu bar with "Menu Disabled" and a toolbar with various icons. The main area features an "Experiment Table" with the following columns: Holder, Type, Status, Disk, Name, No., Solvent, Experiment, Pri, Analysis Type, Analysis S..., Par, Title/Orig, Time, User, and Start Time. The table contains 10 rows, all with a status of "Available". A dialog box titled "Automation Locked" is centered on the screen, with the text: "To unlock the automation window click on the change user button at the right hand side of the Automation Setup window." At the bottom of the interface, there is a toolbar with buttons for "Submit", "Cancel", "Edit", "Delete", "Add", "Copy", and "Change User". The "Change User" button is circled in red. Below the main interface, there is a section for "Preceding Experiments" with the following data:

#	Date	Holder	Name	N...	Experi...	Load	ATM	Lock	Shim	Acq	Proc	User	Disk	Title/Orig	Remarks
2	2020-02-03 15:25:30	1	test proton	10	PROTON	✓	✓	✓	✓	✓		cem 415	/opt/topspin3.6.2/data/cem415/nmr		
1	2020-02-03 13:56:46		Start-Up												Automation was stopped by user

Step 2: Double click the first available Holder number (e.g. Holder 1) to pop up the entry line, as shown below. The **Disk** column shows where your data will be saved and has been already set up by default. Next, type your sample name in the **Name** box (e.g. test), enter a number for your first experiment (each experiment you run in a given **Name** should have a unique number), choose your solvent (deuterated) in the **Solvent** dropdown menu, and then choose the NMR experiment you would like to run in the **Experiment** dropdown menu (e.g. N PROTON for ^1H experiment). Next, click the = sign (circled in red in the picture below) to pop up the experimental parameter dropdown menu where you can edit important parameters such as number of scans (NS) and relaxation delay (D1) if needed. To submit another experiment, type 1 in the **Add** box (circled in blue) and then click Add, then a new entry line will pop up, simply repeat to add another experiment. Additional samples should be run in new **Holder**.

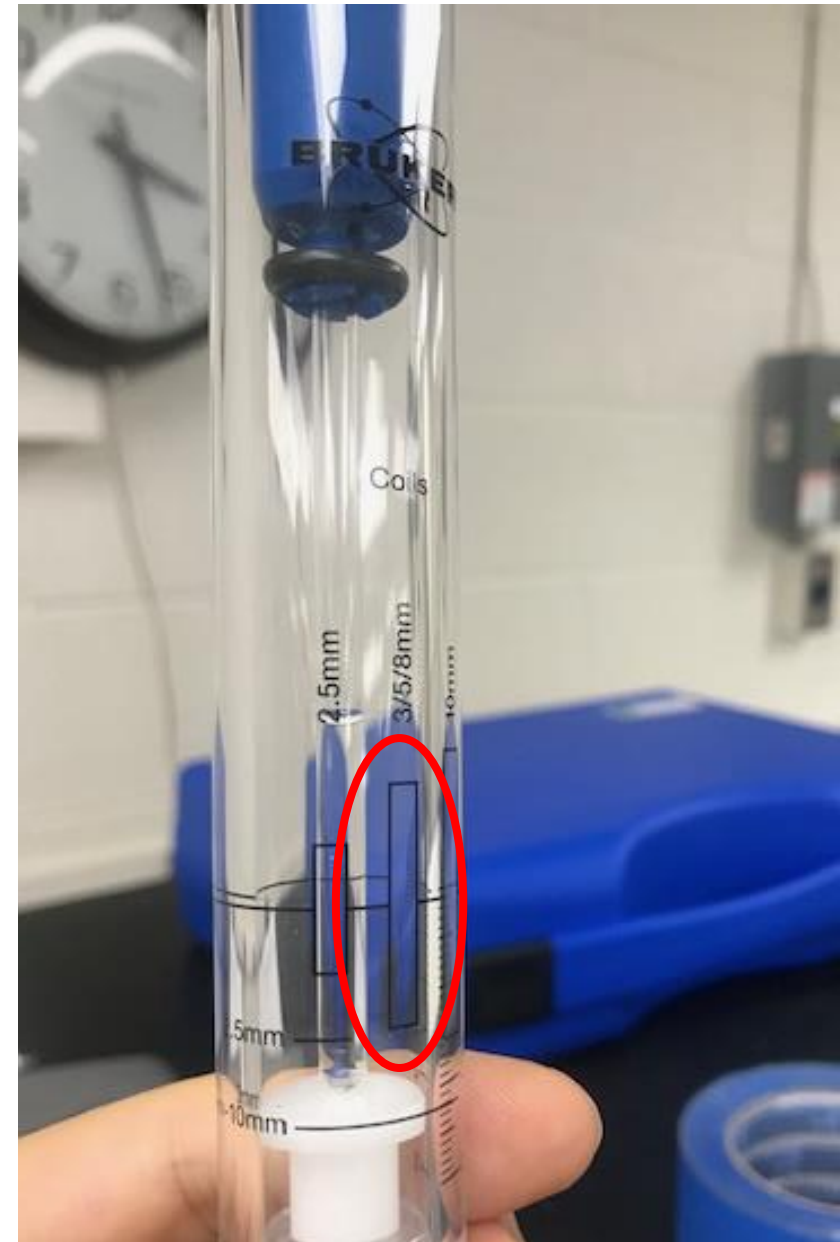
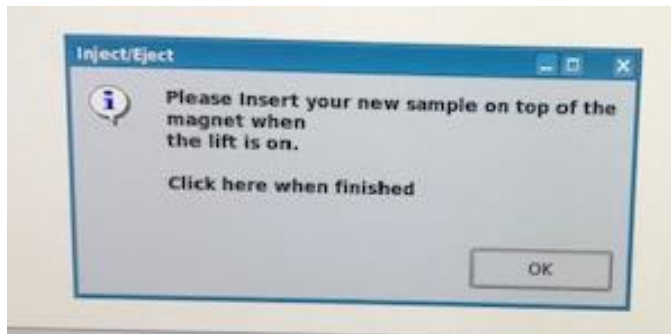
The screenshot shows the IconNMR software interface. The main window title is "IconNMR: Automation Feb03-2020-1021-cem415". The menu bar includes File, Run, Holder, View, Find, Parameters, Options, Tools, and Help. The toolbar contains icons for Start, Stop, and other functions. The "Processing started" status is visible in the top right.

The "Experiment Table" is the central component, displaying a list of experiment entries. The table has columns: Holder, Type, Status, Disk, Name, No., Solvent, Experiment, Pri, Analysis Type, Analysis S..., Par, Title/Orig, Time, User, and Start Time. The first entry (Holder 1) is selected, and its details are shown in a row below the table. The "Solvent" dropdown menu is open, showing a list of solvents including Acetic acid-d4, Acetone-d6, cyclohexane, benzene-d6, dichloromethane-d2, acetonitrile-d3, and others. A red circle highlights the "=" button in the row's action area.

At the bottom of the interface, there is a control bar with buttons for Submit, Cancel, Edit, Delete, Add, Copy, and a numeric input field. The "Add" button and the input field containing "1" are circled in blue.

Holder	Type	Status	Disk	Name	No.	Solvent	Experiment	Pri	Analysis Type	Analysis S...	Par	Title/Orig	Time	User	Start Time
1	1	Available	/opt/topspin3.6.2/da	test	20				No Analysis			cem415			

Step 3: After you have finished adding all experiments, click the Submit button at the lower left corner of the interface to submit your sample. Next, click the OK button in the pop-up Inject/Eject window, wait for a few seconds before the instrument ejects the empty standby NMR tube from the magnet, then take the standby tube and the spinner out of the magnet and remove the tube from the spinner, then put your sample tube in the spinner and gauge it properly. The sample volume in your NMR tube should be centered about the 3/5/8mm rectangle box (circled in red) on the gauge. After gauging, place the spinner on the top of the magnet, then click OK in the Inject/Eject window (see picture below) to insert the sample in the magnet.



Step 4: At this point, you have successfully submitted your sample. The instrument will automatically perform locking, tuning, shimming, and then start acquiring data. Once the experiment is finished, the NMR data will be sent to your email.

When all experiments are done, click OK in the pop-up Inject/Eject window to eject your sample from the magnet, then remove your sample tube from the spinner and put the standby tube in the spinner and then place it back on the top of the magnet. At last, click OK in the Inject/Eject window to insert the standby tube in the magnet.

Step 5: Lastly, click “Change User” at the lower right corner of the ICONNMR interface. Click Lock IconNMR to log out of your account.