

ICON NMR

BRUKER AVANCE-400 NMR INSTRUCTIONS

Introduction:

These instructions cover an introductory level of use for our NMR spectrometer via ICON-NMR. In the instructions below, if you are asked to press a key on the keyboard, or type a value from the keyboard, the name of the key(s) will be typed in brackets [acetone]. Mouse clicks are typed with the correct selection listed in curly brackets {OK}. The default mouse click is with the left mouse button.

Preparing to use the NMR

1. Remove all ferromagnetic objects from your pockets and place them on top of the black-rubber mat on the NMR console.
2. Remove the black cap on the magnet bore. Be careful not to bump the magnet. If you need to use the stairs, carefully position them next to the magnet.
3. Sign your name, date, and experiment that you are going to run in the log book. When you have finished, sign out with OK, or note any errors that occurred. The log keeps track of day to day usage of the NMR, and most importantly notes any problems or errors that need to be corrected.

Logging into ICON-NMR

1. If the windows screen saver is activated, move the mouse or hit a key to deactivate the screen saver.
2. If there is no one logged into the computer, Press [ctrl-alt-del] and enter your login name and password. Ie: [ochem] with a password of [ochem].
3. Once windows has opened, wait ~15 seconds and then double click on {ICON-NMR 3.0.b}
A BRUKER ICON NMR window should appear with four ICONS
4. Click on the ICON above Routine Spectroscopy.
5. Click on the appropriate user name {ochem} and click {OK}
6. Enter a password. [ochem]
7. A window with four vertical icons should appear

Following the ICON Routine Flow Chart.

1. Click on {Inject/Eject} icon, and then click on {Insert New Sample} button.
A flow of air will raise the sample to the top of the magnet.
2. Remove the sample by grasping the NMR tube and place it into the depth gauge.
DO NOT touch the sides of the spinner.
 - a. Place your index finger on top of the spinner, and remove the tube.
 - b. Insert your sample into the spinner and adjust the depth to the 5 mm line.
 - c. Remove the tube from the depth gauge and wipe with a Kim-wipe.
 - d. Place the spinner into the bore of the magnet so the sample floats on the air stream.
 - e. Click on {OK}The sample should gently lower into the magnet.

The Filename icon should be displayed

3. Click on the downward arrow to the right of File: {? }

- a. Select a filename from the list.
- b. Select the default experiment number No: 10

Note that all expts. start at 10 and are incremented to prevent overwriting of data.

c. The default disk for saving data should already be set Disk: D:\u

d. Click on {OK}

4. Click on {Set Solvent/Experiment }

a. Click on the downward arrow {? } under the Set Solvent/Experiment ICON and then select the solvent that your compound is dissolved in from the list.

b. Click on the downward arrow {? } under the Set Experiment ICON and then select the desired experiment from the list.

c. Click in the {open field} above "commands" and then click in the window that opens

d. Type in the title that will be printed on your spectrum

e. Click on {Set Title}

f. Click on {OK}

5. Changes in Experimental Parameters.

****NOTE THIS STEP IS OPTIONAL****

Most routine data collection will work fine with the default parameters.

Follow these commands only if you need to obtain more precise integrals or if you need to change the number of scans for a dilute or concentrated sample. If you are ready to collect data move to step 6.

For more precise integrals.

a. Click on {Parameters} at the top of the flow chart window

b. Click on Acquisition Parameters Editor {ased}

c. Click in the {D1} field

d. Enter a value of [20] followed by [ENTER]

e. Click on {Save}

For dilute or concentrated samples.

a. Click on {Parameters} at the top of the flow chart window

b. Click on Acquisition Parameters Editor {ased}

c. Click in the {NS} field

d. Enter a value four times larger for dilute samples or four times smaller for conc. samples

ie: For ¹H NMR, [64] for a dilute sample or [4] for a concentrated sample followed by [ENTER]

e. Click on {Save}

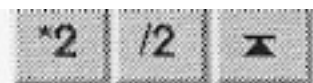
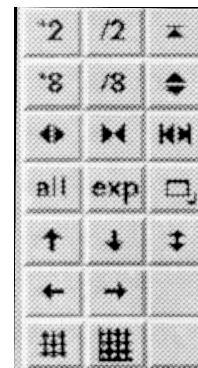
6. Click on {Start} icon

a. Six ICONS should appear at the top of the screen and an informational panel will be displayed in the routine flow chart on the left side of the screen.

- b.** The instrument will sequentially step through each of the ICONS and messages will change under each ICON if the step was successful. If a step was unsuccessful, the routine will stop.
- c.** You can follow what the instrument is doing by clicking the buttons that are not "dimmed" in the informational panel.
- d.** To follow the locking and shimming process, click on the {lock} button. If the lock screen does not appear, check to see if it has already been minimized at the bottom of the screen. If it has already been minimized, you can select it by clicking on the minimized window at the bottom of the screen.
- e.** To follow the data collection, click on the {FID} button. This button takes you to an XWIN-NMR screen that shows you the free induction decay (FID) of your NMR data. The FID shows how your NMR signals decay over time. At the end of the experiment, the FID is converted from units of time into the resonance frequencies you observe in the final spectrum by applying a fourier transformation (FT) function to the FID.
- f.** The final spectrum will be displayed in XWIN-NMR and a default region (1 ppm to -1 ppm) will be integrated and plotted on the laser printer.
- g.** Shrink the XWIN-NMR screen down to an icon. To change the sample, or collect another NMR spectrum on the same sample select the {Continue} button in the ICON NMR Window.
- h.** To collect another NMR spectrum on the same sample select the {Use Same and Continue} button and return to step 3 above.
- i.** To change the sample or to finish using the instrument, select the {Insert New Sample} button.
 - ?? For a new sample return to step 2 above.
 - ?? If you are finished with the spectrometer, insert the reference standard sample as described in step 2 above. NOTE THE SOLVENT USED IN THE REFERENCE SAMPLE!
 - ?? After the standard has been inserted into the magnet. In the ICON NMR Window click on the {File} pull-down menu and select {exit}
 - ?? In the command line at the bottom of the XWIN-NMR window type [lock] followed by [ENTER]. Select the solvent that matches the standard ie: {Acetone}
 - ?? The instrument should lock on the acetone solvent in the standard.
- j.** Sign out of the log book.
- k.** Replace the black cap on top of the magnet bore

Changing the Display of Data in XWIN-NMR

The upper left corner of the XWIN-NMR screen contains a series of buttons shown below that control the data display. For more information on these commands see the XWIN-NMR Processing Software Manual p. 9-14.



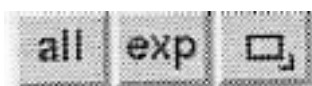
- ?? Multiply Data by 2
- ?? Divide Data by 2
- ?? Scale the biggest peak to the top of the screen



- j.** Multiply data by 8
- ?? Divide data by 8
- ?? Interactive vertical scale when left mouse button is depressed



- ?? Expand data by factor of 2 along X-axis
- i.** Contract data by factor of 2 along X-axis
- ?? Display the full spectrum along X-axis



- ?? Display the complete spectrum along X-axis
- ?? Display the last expanded region
- ?? Interactive zoom of region. Draw a box around the region to be expanded by keeping the left mouse button depressed. Click right mouse button to zoom.



- ?? Draw the zero point of the spectrum in the middle of the screen
- a.** Draw the zero point of the spectrum on the bottom of the screen
- ?? Interactive zero point when left mouse button is depressed



- ?? Shift display 1/2 screen width to left
- ?? Shift display 1/2 screen width to right



- ?? Display a grid with a fixed width on the screen
- ?? Display a grid that will change with expanded or contracted data

Plotting Data

1. Once you have manipulated the data on the screen, if you want a plot of that region, click the {PLOT} button.
2. If you want to display a specific chemical shift region, select the {DP1} button. Enter a downfield chemical shift ie: [10.0] for 10ppm, followed by an upfield chemical shift ie: [0.0] for 0ppm. Enter [n] when asked to change the scaling.
3. Click the {PLOT} button.

Changing the Display Scale

Change from hertz (useful for J-coupling constant measurement) to ppm by selecting the {hz/ppm} button.

Manually Setting a Reference

1. Select the {Calibrate} button and a cursor will appear on the screen.
2. Position the cursor on top of the reference peak and click the middle button.
3. Enter the chemical shift for the reference. Ie for TMS [0.00] [ENTER]

Manual Integration of Peaks

1. Click the {Integrate} button on the left side of the screen.
2. Click the left button of the mouse, and a cursor will appear on the screen.
?? Move the cursor to the left side of the peak to be integrated and click the middle mouse button.
?? Move the mouse and a white arrow will appear.
?? Move the cursor to the right side of the peak to be integrated and click the middle mouse button. The integral will be displayed on the screen.
?? Repeat these steps for all peaks to be integrated.
3. To set the area of one integral to a specific area.
?? Click the left mouse button and a white arrow will appear.
?? Move the arrow on top of the integral to be used as a reference and click the left mouse button. A small white arrow should appear on top of the integral.
?? Select the {Calibrate} button from the left side of the screen. Enter the desired area into the data field followed by [ENTER].
4. To exit the Integration routine select the {return} button in the lower left corner, followed by the {Save as 'intrng' & return} button to save the new integrals for plotting.