Basic NMR Operation  
Guide for the Bruker AV-III 400 MHz NMR Spectrometer  
using ICON NMR in Automation

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Before You Start:

NMR tube must be at least 7 inches in length and must have a top that is neither broken nor cracked. If your tube is shorter than 7 inches, it will not be grabbed fully by the sample changer and may fall and break on the floor or on the top of the magnet. Tubes with chipped tops may be broken by the sample pincers which grab the tube very firmly close to the top. Any tube shorter than 7 inches or with a chipped top is not allowed.

NMR tube must not have any label (paper, tape, sticker etc....) attached to it. This will stop the sample pincher from working properly and may result in sample breakage and equipment damage. Tubes can be labeled on the glass or the cap with permanent marker. Any tube with an attached label submitted to the sample changer is not allowed.

Sample tubes with screw caps or vacuum valves will not be handled by the sample changer. They will cause equipment damage.

Approximate amount of middle-size compound (MW ~ 500) advisable for running following experiments:

- $^1$H NMR, $^{19}$F NMR, $^{31}$P NMR experiments - about 3-5 mg
- $^{13}$C NMR short run experiment (0.5-1 hr)-about 20-50 mg; long run experiment - about 5-10 mg
- 0.6-0.7 ml of NMR solvent is appropriate for the right solvent level in NMR tube. Unsuitable solvent level can lead to a bad shimming result and horrible looking spectrum.

Never lean or exert any force on the sample changer or magnet. This may cause a magnet quench resulting in many weeks of down time and hundreds of thousands of $$ being spent.

Users are asked to report improper sample tubes to the NMR Facility staff immediately in order to avoid needless down time and equipment damage!

The 400 NMR uses the Sample Xpress Automatic Sample Changer, and it is extremely important that you pay attention to the holder number containing your sample, and to only remove your samples from your holder number, from the sample rack. It is possible that someone might have samples in the rack with longer experiments submitted in the Night Queue. You must be sure to not disturb samples left by other users. Labeling your NMR tubes (on the glass or the cap with permanent marker.) is STRONGLY recommended.

1. Start

(1). Log-In
Click on your account then enter password

(2). Start ICON NMR
Click on the TopSpin launch TopSpin
In the command line, type icona, start ICON NMR
2. Setup Experiment

(1). **Sample tube/s**, Wipe-off NMR tube with kimwipes 1. Insert sample tube in the spinner 2. Set the sample depth using Bruker depth gauge 3. Then, place sample tube/tubes in the holder position at the SampleXpress Lite Sample Changer carefully (place all samples once at the beginning).

(2). Setup ICON NMR, steps 1-8

1) Double-click on sample **Holder #**

2) Click on Name ▼, then put /write file name

3) Click on **Solvent ▼**, then select solvent on drop-down menu

4) Click on **Experiment ▼**, then select experiment on drop-down menu

5) Click on **Par ▼**, then check/change parameter on drop-down menu, then click on **Ok**

6) Click on ▼, type in title/remark, then Click **Set Title**

7) Click on **submit** to send job
Click **Start**, and **Start** on popup box (start to run now or after all samples/experiments are setup)

1). **Double-Click** in the row of **Holder #**. The row will “open up”.

2). **Click** on the **Name**, then put /write file name.

3). **Click** on the **Solvent**, a pull-down menu will appear, then select a solvent from the pulldown Menu.

4). **Click** on the **Experiment**, select the experiment from the pulldown Menu.

5). **Click** on the **icon**, a dialog box popup, enter the new value to change basic parameters, then click [OK].

   - NS is the number of scans;
   - D1 is the relaxation delay
   - SW is the spectral width in PPM;
   - O1P is the center of the spectrum

6). **Click** on **Submit**, the dialog box popup, add “title or remark”, then Click [Set Title] when everything is entered.

7). **Double-Check** that everything in the row is correct, confirm that sample is properly placed in the correctly numbered holder in the sample-change. Then click the **Submit**, the status indicator will turn yellow.

8). For the first sample click on **Start** on the left top corner button, then click on **Start** on popup box. The status indicator light will turn green.

   **For steps 8, click on Start when the first sample is setup or skip step 8 now to setup all samples/experiments, then do step 8.**

   Once green, SampleXpress Lite Sample Changer will insert the sample, tune the probe, lock on the solvent, shim, and proceed with your experiment.

   * **Add another experiment for the same sample:**

   Select the row # containing a previously defined experiment, then click the **Add** button. Go through step 2-7 procedures above, then **click** **Submit**.
3. Finish. After experiments are done, there are only **red** and **dim** lights, no **green** and **yellow** lights showing on **ICON NMR**, then remove all sample tubes from the **SampleXpress Sample Changer**, take sample tubes out from blue spinner, and place blue spinner in the box.

4. Data Processing

(1). **Open Dataset**, Dataset can be opened by **a)** or **b)**.

a). Clicking on the **Start** in the TopSpin Menu bar, then click on **Open Dataset**, your dir/files will appear select dir/file which you want to process, then click on **Open**

b). click on the **Browser**, locate your data and right-click on a dataset name, and choose **Display** from the drop down menu

(2). Process Spectrum,

Click on the **Process** in the TopSpin Menu bar, then click on **Proc. Spectrum** there are a few options to choose from the drop-down menu
a). Auto Process, for easy and quick, Select *xaup* to auto process spectrum with peak picking and integration.

b). Standard Process, Select *proc 1d* (2d, 3d), then fill in drop-down table to process spectrum.

c). Manual Process, type *efp apk* in the command line to process data and execute automatic phase correction.

(3). Phase Correction. The simplest procedure is automatic phase correction which is implemented using the command *apk*. For more corrections, click on Adjust Phase for manual phase by 0 and 1 (0 and first order). Highlight on 0 or 1, then press and hold the left mouse button move up/down to adjusted phase.
Click on disc icon to save phase correction, then click on return icon to get out the phase section.

(4). Optimizing the Spectral Width. To display a specific region of your spectrum hold the left mouse button and drag your mouse over the region of interest.
For exact chemical shift range, click and specify the range you wish to display. For example, to display the region of the spectral window from 9 to -1 ppm, put 9 and -1 in the From and To fields, then click OK.

(5). Peak Picking. Click on Process, then Pick Peaks in the TopSpin Menu bar. Click on Process Peak picking range by highlighting to define range.

Press and hold the left mouse button over the peaks of interest. A green highlight will form, release the left mouse button, all peaks in green area were picked. Repeat this process to pick all peaks of interest. Then click to save and to get out the mode.
(6). Manual Integration. Click on **Process**, then Click on **Integrate** and **highlighting** to define integration region.

Set the cursor line, starting at the left of the spectrum, to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button.

* will set the first integrated peak as 1.

Repeat this process to integrate all peaks of interest.

Click on it to delete old integrations.

Click on to save and get out the mode.
Option for set integral reference
Place the cursor within the integral label peak and press the right mouse button. Select **calibrate Current Integral** on popup menu. Enter the desired value of the selected integral in popup box, then **OK**.

*chemical shift auto set to lock solvent
Option for set peak chemical shift reference
Click **Calib. Axis**, then click the peak to reference. Enter the desired chemical shift of the peak, click **OK**.

Click on **`*`** then click to save and out the mode
(7). Plotting  a). Quick print, click on printer icon, then click ok on drop-down box, another Print box popup, click on to select **HP-Laserjet-P2035** in Name, then click on Print

b). Plot Editor, click on **Publish**, then click on **Plot Layout**. Editing by a panel at the left (active/select menu)
Print, click on ▼ and select Print... on drop-down list, click on ▼ to select HP-Laserjet-P2035-Series in Name on popup Print box, then click on Print.

Or type `prnt` in the command line, it will automatically print your spectra as displayed (what you see is what you get).

5. Close Icon NMR & TopSpin

(1). Close Icon NMR, Click Stop, select yes on popup, then place pointer on File and choose Close All to logout Icon NMR.

(2). Close Topspin, place pointer on Close and select Close on popup menu, then click OK on popup to close Topspin.
6. Copy/Transfer Files to USB Flash Drive

(1). Open USB, insert your USB flash drive to PC, icon appear, click on it to open.

(2). Copy files, click Home folder on desktop, select the file/files to be copied, right-click and select Copy from the drop-down menu. Move the mouse to USB DISK window, right-click and select Paste from the drop-down menu.
(3). **Unplug USB**, After copy files, right-click on and select Unmount Volume on drop-down menu to unmount the USB flash drive, an Error may popup, click on OK. **Wait ~30 sec** for writing files to USB (not do so, the copy files are empty, have name only), then the USB drive can be removed.

7. **Logout**
Place pointer on **System** on the top menu bar, select **Log Out** on drop-down menu to Log Out the account. The screen shows as