Bruker Avance DPX 300MHz NMR Spectrometer

***The superconducting magnet will erase magnetic strips on IDs and credit cards. Leave wallet, keys, watches, phones etc. on console table before you approach the magnet.***

To Get Started:
When asked to “click” on an icon, left-click (if not specifically designated). From “ICON-NMR” screen with small “Identify User” icon in center
Left-click on icon: Highlight “NMR Super-User”  User ID: bruker300!
If there are multiple boxes to choose from, then click on “Routine Spectroscopy”.

The Routine Flow Chart takes you through a series of automated steps that will result in a phased, albeit poorly framed and integrated, spectrum (see Optimizing Spectrum Output below).

“Sample” Dialog Box
Click on the blinking “Inject/Eject” icon, then click on “Insert New Sample”
If another sample is in the magnet, it will be ejected with compressed air.
Remove sample – Lift it straight up and KEEP FINGERS OFF OF WHITE & BLACK ring.
Insert sample collar into the depth gauge (set at 5mm for standard tubes), remove previous tube if necessary and insert your new sample tube until tube hits white platform of the gauge. Remove sample tube from depth gauge with sample collar attached.

WAIT until separate “OK” dialog box appears (do not click “OK” yet!)
After you see “OK” box (i.e. have full airflow) then gently insert tube and with sample collar on top of the magnet. **Be sure not to put depth gauge into the magnet as well!**
Click on “OK” button – sample should go down and then you will hear a click.

“Data Set Filename” Dialog Box
File name drop down list – select today’s date with “NMRSU” suffix attached
Experiment number – start with 10, then increase by 10 for subsequent samples
Disk Unit - select “u”
Click “OK”

“Set Solvent” Dialog Box
Select the solvent being used from menu (left-click), then click “OK”.

“Set Experiment” Dialog Box
Choose type of experiment to be run, e.g. proton vs. Carbon-13.
If doing standard proton NMR then choose “PROTON” from list;
If doing standard C-13 NMR (composite pulse decoupled), choose C13CPD (1024 scans) or C13CPD32 (32 scans) for very concentrated (> 100mg) samples.

Click on Set Plot Title – move cursor to pink box at top and type title that will appear at the top of your spectrum – click “Save”, then “OK”
“Start” Button
This will start instrument on preset series of steps: **Initialize the interface, lock** the sample, **shim** the sample, set the **receiver gain**, **Acquire** the appropriate number of scans, **Fourier transform** and **phase** the spectrum and finally **plot**. You can watch progression of this process via the blinking boxes at top.

Click “Start” icon and then click on **View / Lock**.
This will allow you to watch as instrument **locks** on your sample and then **shims** it.
The shimming process changes the current (in “shim coils”) in small increments surrounding your sample to fine-tune the alignment of the field with the sample. You will see the lock signal getting progressively less noisy during shimming. This process may take up to 5 minutes.

When “Acquire Data” begins to blink, the box becomes “ZG in Progress” (meaning Zero previous experiment and Go!) then click on **View / FID** to open the acquisition window and quickly minimize the previous display page (small dot at top right corner).

This NMR spectrometer is a Fourier-Transform (FT) instrument – you can watch the free induction decay (FID, time-domain spectrum) grow as the number of pulses is accumulated. By default, the proton experiment collects 16 scans.

When complete – click on “Seen” button. The spectrum will be seen in a frequency domain (ppm) and will print with default x and y axes that will likely need to be adjusted. Note: If the spectrum is off-scale in either domain (X- or Y-axes) click on |◄►| and ▲.

**Optimizing Spectrum Output – XWINNMR**

Now you can **frame, calibrate, integrate, peak pick, expand** and **plot** the spectrum as needed. The left mouse button activates commands and the center button works within that command. Assume “left” click if not specified.

**Framing the Spectrum (along X-axis):**
This process can be used to expand regions of the spectrum as well.
With the cursor in the spectrum field, left-click to attach the cursor to the spectrum baseline. Place the cursor at your desired downfield (left) limit and without moving it, center-click. Move the cursor to the upfield limit and center-click again.
Once you have the frame you want, click **dp1** (opens a dialog box) to set F₁ (downfield limit) for your printed spectrum <enter>; set F₂ (upfield limit) <enter>, then change Y-axis scaling, answer “y” <enter>. Essentially, after clicking on **dp1**, strike “enter” 3 times.
Calibrating the X-axis on the Spectrum:
Left click on “Calibrate” button on left panel. Place cursor on top of internal standard or solvent peak and center click. A dialog box will open and type in the known chemical shift (type 7.26 for CHCl₃). You may need to temporarily expand the region where the spectrum is prior to calibrating (see “Framing…” above).

Integrating the Spectrum:
You will need to re-integrate the spectrum or parts of the expanded spectrum. The arrows at the top of the left panel allow you to move the spectrum left or right as needed.
Left click “Integrate” button and left-click to attach the cursor to the spectrum. Move the cursor to the left of the downfield-most signal that you wish to integrate and center-click. Now center-click on the upfield side of that peak. Continue to the next signal and use the center mouse button to define the start and stop of each integral trace. Continue moving upfield from signal to signal until finished.
Assigning absolute integral values: Choose an integrated signal for which you know the absolute number of H’s (a methyl group for example). Once the spectrum is fully integrated, place the cursor on the chosen integral and left-click to place the cursor at the top of the integral trace – an arrow appears and selects that integral for adjustments. Under “current” (left panel) select “calibrate” and a dialog box appears with an area value; change it as you desire (e.g. 1 or 3). Your areas will then be set relative to this signal. Click on “return” (bottom of left panel) and “save as intrng & return” (or “cancel” if you want to try again).

Peak height adjustment and Peak Picking:
Type “pscal” <enter> and select global. YU (Y axis units) toggles between absolute and relative values. This must be set on relative to adjust CY (tallest peak ht in cm) and MI (minimum intensity). Click utilities menu from the left panel. CY will be set so that the tallest peak is at the top of the spectrum. To adjust this, click CY and move the horizontal bar to where you want the top of the spectrum to be, then center-click. Now left-click and type a value for CY (a good number is 12) which is the distance from the baseline to the horizontal bar in cm.
To set the minimum intensity for peak picking, click MI (YU still in rel units) and move horizontal bar just below the top of the smallest peak you want annotated. (Note: you may have to click MI twice to get the horizontal bar.) Left-click to set threshold (only peaks above this line will be “picked”) and click “seen”.

Printing the Spectrum:
Before printing, move the cursor to the pink command box at the bottom and type “view” <enter>. This will show the spectrum as it will appear on paper. [If the spectrum appears too tall, (off-scale), lower CY (type “CY” in pink box and enter new CY); if it appears too short, increase CY.] Click Quit. If you type “plot” in pink box at the bottom, spectrum will print according to the plot parameters you set earlier (dp1). If you click on “plot”, the spectrum displayed on the screen will print.
Printing the peak picking separately:

At the top of the screen click Analysis, Peak Picking, List peaks on screen, and click print. Then “OK” to exit out of that display.

To customize your spectrum, before plotting, type “edg” <enter> (edit graphics) in the pink box:

For peak picking on the spectrum, “draw peak labels?” answer “yes”
To remove integral trace lines, “draw integral trails?” answer “no”
To remove the parameters from right side of spectrum, “draw parameters on plot?” answer “no”, then set CX = 25 (spectrum width in cm) once you have clicked “Save”

Type “view” to see how things look. Print spectrum as above.

When done:

Minimize display (dot in upper right).
If small icons across top then click on “Routine”
Once you get to the “Routine Flow Chart” box, then click on “Continue”
You can now select whether to continue with same sample (e.g. you may wish to run a C-13 spectrum), continue with new sample, or to Eject and Terminate. In the latter two cases, your sample will be ejected.