Avance III 400 MHz NMR Basic Operating Instructions

If the operating program is not running, double click on the TopSpin 3.2 icon.

Insert the sample as follows:

1) Never handle the spinner (blue NMR tube holder) with your fingers. Always use a Kimwipe®, not a tissue, because Kimwipes® are lint-free.
2) Remove that spinner closest to the feed tube on the left from the circular tray. Insert your sample into the spinner, use the depth gauge to seat the tube, and replace the spinner in the tray in the same slot.

The software works off of a series of tabs & sub-tabs that you use in sequence. In each case, click on the relevent tab and following the instructions that follow.

1) Start tab –
    a. Create dataset – Choose a file name which has no spaces or special characters. Set EXPNO and PROCNO to 1. "Experiment" should be set to either PROTON (for ^{1}H) or C13CPD32 (for ^{13}C). (Click "set selected item in editor") Check "Set Solvent" and choose the solvent for your sample. Choose the appropriate directory to save your spectrum. Do NOT just use default directory. Click on OK.
    b. READ PARS – either PROTON or C13CPD, then click on the READ button, then OK.
    c. Unless you are opening a spectrum that has been saved, move to the Acquire tab.
2) Acquire –
    a. Sample – To insert the sample, on the command line (located on the left about an inch from the bottom of the screen) type SX followed by a space and then the number of the space into which you placed your sample. The lights will blink green and yellow. When the light stays yellow, move to the next step.
    b. Lock – Click on this tab, then select the solvent and click on OK. The sample is locked when "lockn: finished" appears in the lower left box.
    c. Tune – Click, but not on arrow, to start. "Job succeeded" tells you this is done.
    d. Spin – Choose "Turn sample rotation on." "ron: finished" tells you this is done.
    e. Shim – click to start (not on arrow), "Job succeeded" tells you this is done.
    f. Prosol – click to start(not on arrow), "get prosol finished" tells you this is done.
    g. Gain - click to start, do NOT click on the down arrow, "Job succeeded" tells you this is done.
    h. Go – click to start, do NOT click on the down arrow, "checklockshift" tells you this is done.

At this point, you should have collected the data from your sample. Now you must process the data to obtain a usable spectrum.

3) Process –
    a. Proc. Spectrum – click to start, do NOT click on the down arrow. At this point you should have a usable spectrum. The following bullet points will help you with typical refinements to the spectrum.
    b. If the spectrum is not at the bottom of the screen, click on the  icon.
    c. To zoom in on a region of the spectrum, click on the  icon and enter the spectral range you which to view. To go back to the full spectrum, right click and choose "show full spectrum." Or Depress the left mouse button and drag to the right and let go when you have highlighted the region you wish to expand.
    d. To expand the vertical range, roll the mouse wheel way from you (increase peak size) or toward you (decrease peak size).
    e. To show peaks, click on "Pick Peaks," then draw a box around each group of peaks holding the left mouse button down. If you have too many peaks, draw a box above the spectrum and release the button and all picks will erase. To keep your selection, click on the  icon.
    f. To integrate peaks, click on the integrate tab. Just above the spectrum is a toolbar with many icons.
        i. Click on the second one from the left "Define new region using cursor. then to the left of each multiplet/region you want to integrate, place the arrow, hold the left mouse button down and drag right until you reach where you want to stop integrating and release. Repeat for each multiplet.
        ii. To set the integration value, pick one multiplet whose value you think you know. Place the arrow over the number at the bottom of the spectrum, right click, and choose "Calibrate current integral." Set the value and click ok.
        iii. Click on the  icon.
4) Publish –
    a. To print the spectrum,
        i. Make sure the region you wish to print is correct (i.e. ppm range).
        ii. Adjust the position of the spectrum above the integration values at the bottom of the page using the icon of the double vertical arrow with the hand. Left click over the icon and drag up/down to move the spectrum.
        iii. Make sure the peaks remain on screen. Use the mouse scroll wheel to expand/shrink the spectrum vertically.
        iv. Use the mouse wheel to adjust spectrum height.
        v. Click on print.