Topspin Protocol for Buker 500 NMR

I. Sample Introduction

a. Login to the NMR workstation using the proper username and password
b. On the desktop open "Topspin 3.0".
c. Type "**ej"** in the bottom window of Topspin.
    i. Note: Wait for the air to turn on before inserting sample.
d. Insert the sample into the magnet
e. Type **"ij"** to lower sample into the magnet
f. Type **"ro"** to spin sample

II. Experimental Setup

a. At the bottom of topspin type **"rsh latest"**
b. Click **File** then **New** on the top menu.
    i. **Name** – Define a file name for your sample
    ii. **Expno** – This is the experimental number and should begin with **1**
    iii. **Procno** – this is the process number, normally set to **1**
    iv. **Dir** – This should be set to C:\Bruker\Topspin 3.0\Data
    v. **User** – This should be the same username you are logged in under
    vi. **Solvent** – Select the deuterated solvent used.
    vii. **Experiment** – Select **Proton** from the dropdown menu.
    viii. **Title** – Enter a title for the spectrum. This will be displayed in the printout.
c. Click on **OK** in the window to close

III. Locking, Shimming, and Wobbing

a. Type **"lock"** and choose the appropriate lock solvent
    i. When lock is finished, as noted in the bottom window, proceed to the next step.
b. Type **"getprosol"** in the bottom window of Topspin
c. Type **"atma"**, in the bottom window to wobb the sample.
d. When wobbing is finished, as indicated at the bottom of topspin, then type **"topshim"** to shim the sample.
    i. Note: Wait until shimming is completed before going on.

IV. Data Acquisition

a. Type **"rga"** and wait for finished to appear
b. Type **"zg"**. You should see an FID signal after the dummy scans are completed

V. Data Processing

a. Once Data Acquisition is complete, type **"ef"** to perform exponential multiplication and a Fourier transform.
b. Type **"apk"** to perform automatic phase correction.
c. Type **"abs"** to perform automatic baseline correction.

VI. Spectrum Calibration

a. Click  on the top ribbon to begin calibration.

i. Zoom in on the desired peak by adjusting:  or by clicking, holding, and dragging the cursor around the region desired.
ii. Center the cursor on the peak to be calibrated, and click once.
iii. Type the calibration frequency and click **OK**

VII. Automatic Peak Picking

a. Click  then type "**pps**" to perform automatic peak picking.

VIII. Integrating Peaks

a. Click  to begin integration.
   i. Click  to select all integrals.
   ii. Click  then **OK** to delete the automatically calculated integrals.
   iii. Click  to begin defining new regions.
   iv. Click, hold, drag, and release the cursor around the peak to be integrated. Repeat for remaining peaks.
   v. Once finished with integration, click  to save changes.

IX. Plotting

a. Type **"plot"** at the bottom of Topspin
b. Double click on the center of the screen (green boxes should appear around the spectrum).
c. Change Xmin / Xmax to the desired frequencies (in ppm).
   i. Click **OK** when finished.
d. Right mouse click on the center of the spectrum and select **1D/2D-Edit …**
   i. Adjust the vertical height using  and then click **Close** when finished.
e. Click **File… Print** or press Ctrl+P to print.
f. Close the Topspin Plot Editor and click **No** if a dialog asks if you wish to save changes.

X. Running a New Sample

a. To run a new acquisition, start at step II, Experimental Setup

XI. To Eject a Sample

a. To eject the sample type **"ej"** at the bottom of Topspin.
b. Log off when finished