TOPSPIN quick guide to record routine 1D ^{1}H, $^{13}C\{^{1}H\}$ and $^{13}C\{^{1}H\}$ DEPT135 NMR

Penn Chemistry NMR Facility

Getting Started:

- Login using your group ID and password.
- In **Desktop**, double click on **TOPSPIN icon**.

BSMS keypad Operation:

- press **LOCK ON/OFF** button to switch off lock hold.
- press **SPIN ON/OFF** button to stop spinning (if spin indicator is **ON**).
- press **LIFT ON/OFF** button to eject sample.

Gently slide NMR tube into the spinner and adjust tube height in depth gauge before inserting sample.

- On BSMS keypad, press **LIFT ON/OFF** button to insert sample.

Creating New Dataset:

- Type **new** ↵
- Enter ***sample_id*** (**maximum 13 character**) in **NAME** box, type a numeric value for **EXPNO** (**1-999**) and a numeric value for **PROCNO** (**1-999**).
- Click the down-arrow of the **Solvent** box to select a solvent
- Click the down-arrow of the **Experiment** box to select **use current params**
- Type **rpar** ↵ and select parameter set
- Parameter Name: proton = **1_protonstd**
  carbon = **1_carbonstd** DEPT135 = **1_dept135**

Locking and Shimming:

- Type **lopo** ↵. Select deuterated solvent from the list.
- Type **lockdisp** ↵.
- If lock ring down pattern is normal, press **LOCK ON/OFF** button to start lock hold.
- Optimize **X** and **Y** shim iteratively without spinning the sample.
- Press **SPIN ON/OFF** button to start sample spinning (wait for spin indicator to stop blinking).
- Optimize **Z** and **Z2** shim iteratively (make sure **FINE** indicator is **ON**)
- Press **STDBY**.

Acquiring Spectrum:

- Type **rga** ↵ and wait for ***rga:finished*** message.
- Type **zg** ↵ to start acquisition.
- After **4-8** transients (**only for ^{1}H**), type **tr** ↵ to transfer data to host computer.
- Type **efp** ↵ to perform Fourier transformation including apodization and default phasing.
- Type **apk** ↵ to perform automatic phase correction.
- If signal-to-noise of the spectrum is satisfactory, then type **halt** ↵, wait for ***zg:acquisition finished*** message.
- Type **efp** ↵ to transform rest of the acquired transients.

Processing Spectrum:

Phase correction:

If phase of the spectrum is fine, go to next step, otherwise click on the button, click-hold the 0 and 1 buttons to get correct phase. Click

Chemical shift calibration:

Horizontally expand solvent peak region and click the button, click on top of the solvent peak, enter **correct ppm value** of the solvent from the chart.

Integration:

Click on the button, with the green highlighted button, select integration region by dragging the mouse to cover the whole region of the peak(s) [***Adjust bias and slope if necessary***]. Select the known integral by right-clicking and choosing **Calibrate** from the popup menu while cursor is under the integral region, enter value for the integral in the popup box. Click **OK** button.

Peak picking:

Click on the button, with the green highlighted button, select peak picking range, and with the button, modify the peak picking range.

Printing Spectrum:

- Click **Title** tab and enter plot title
- Type **layout** ↵ and select **desired layout** by clicking down-arrow button in **LAYOUT** box
- Type **plot** ↵, if necessary, modify layout in TOPSPIN plot editor
- Click **File→print** to print the spectrum in TOPSPIN plot editor.

Logout Steps:

- On BSMS keypad:
  - press **LOCK ON/OFF** button to switch off lock hold.
  - press **SPIN ON/OFF** button to stop spinning.
  - press **LIFT ON/OFF** button to eject sample.

- Insert standard sample tube back into the magnet.
- Start Lock hold by typing **lopo cdcl3** ↵ (***don't spin standard sample***)
- Optimize **Z1** and **Z2** shims without spinning.
- Click **quit** tab in **Lockdisp** window to close.
- Type **exit** ↵ to close **TOPSPIN** window.
- In **desktop**, Click on **start**, select **Log Off**, click **Log Off** tab to log out.
- Fill out log book with all necessary info (your ID, group ID, solvent, time, experiment).

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