

Standard NMR Procedures

Prepping the sample:

- Wipe NMR tube with a chem wipe
- Place NMR tube in blue spinner using depth gauge to set the tube to the correct depth
- Only touch the blue spinner with a chem wipe
- Place sample into a numbered slot
- Take note of number

Taking NMR:

-Open Top Spin 3.0

1. sx # → changes sample to the correct numbered slot (from above)
2. ro on → turns spinner on
3. edc → creates new data set
 - Name
 - Change directory to your own
 - Exp/proc are typically 1
 - Change title to represent sample
 - Click OK
4. lock → find solvent being used
5. rpar → read parameter file
 - Click what NMR is being run
 - Click READ
 - Parameter window (acquisition, processing, output data)
 - Click OK
6. atma → automatic tune and match
7. rsh → read shim file
 - Read most recent date (lastbest)
 - Click READ
8. topshim 1d → shims sample
 - Lock signal disappears and sample still spins
 - The magnet shifts along z axis
 - Already averaged along x and y
9. eda → edit acquisition parameters
 - Click blue test tube (will not highlight)
 - NS: number of scans → left at 16
 - DS: dummy scans → changed to 0 for fast scans
 - Changed to 4 for carbon
10. rga → pulses to change gain
 - If the sample is concentrated then the number will be low

11. zg → zero go: zeros out waiting commands
 - Wait for all scans to finish
 - Y axis is relative intensity
12. fp → Fourier processes the .fid file
13. apk → automatic phase correction

Editing Data:

1. Find and reference solvent
 - Click Process → Calibrate axis
 - Find and reference middle of peak
2. Integrate peaks
 - Integrate Tab
 - Yellow highlight button (goal post button)
 - Click and drag to integrate
 - Click floppy disk button to save
3. Pick peaks
 - Click and highlight with box
4. Plot
 - Plot tab
 - Click double folder button
 - Select H+pp+info
 - Click paper sun icon to edit graph
 - Print

LAST:

- sx 1
 - Places the standard “ethyl benzene in chloroform” back in instrument

TRACS Site: Dept. of Chemistry and Biochemistry NMR

Use the calendar feature to reserve time.