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NMR User Guide

<u>Login to the computer:</u>

Use the "nmr" account, the password is nmr.

> *Topspin commands are shown in* **bold**

<u>Preparing and loading the sample:</u>

- Solvent height in the NMR tube should be at least 5 cm.
- Use only good quality NMR tubes. Do not use cracked NMR tubes or tubes that do not fit well in the spinner.
- Place your NMR tube into the spinner and use the depth gauge to adjust the spinner height on the tube.
- Clean the outside of the tube using a Kimwipe. Hold the tube and not the spinner.
- Type "**ej**" to turn the lift air on. Take the previous sample out of the magnet if there is one.
- Carefully place the spinner with the NMR tube into the magnet bore. *<u>Make sure airflow is ON before dropping the sample!</u>*
- Type "**ij**" to turn the lift air off and lower the sample into the magnet.

Experiment Setup:
(a) Create Dataset

Type "**new**" on the command line to create a new dataset. The dialog below will appear.
(*Tip: Start from an open dataset to have all the data path set correctly*)

Complete ONLY the fields circled:

NAME: Your sample ID
EXPNO: 1 for the 1st experiment,
         2 for 2nd and so on

Make sure the "Read parameterset" radio button is selected.

Type PROTON for ^{1}H NMR
Type C13CPD for ^{13}C NMR
Type COSYGPSW for ^{1}H-^{1}H COSY
Type NOESYPHSW for ^{1}H-^{1}H NOESY
Type HSQCEDETGPSISP_ADIA for
                    ^{1}H-^{13}C HSQC
Type HMBCGP for ^{1}H-^{13}C HMBC

You can also select these by clicking the "Select" button.

Make sure the "Execute getprosol" radio button is selected.

Finally, you can give a title for your dataset in the Title window.

For those who use water suppressed experiments, please use the parameter sets that end with "_WS".
(For example, COSY_WS, NOESY_WS, etc).

(b) Instrument setup and data collection

- Read in the shimming presets by typing "**rsh temp**" at the command line.
- Click the lock icon to open the lock window.

- Then click on the "test tube" icon on the lock window to select the solvent.

Select the appropriate solvent and click "OK"

- On the command line, type "**atma**" to automatically tune and match the probe.
  (*will take about a minute to complete*)
- Type "**ts**" to automatically shim the sample
  (*takes about 3-5 mins to complete*)
- Type "**rga**" for automatic receiver gain adjustment
- Type "**ns**" to set the desired number of scans.
- Type "**zg**" to acquire data.

Solvents table	
△ Solvent	Description
Acetic	acetic acid-d4
Acetone	acetone-d6
C6D6	benzene-d6
CD2Cl2	dichlormethane-d2
CD3CN	acetonitrile-d3
CD3CN_SPE	LC-SPE Solvent (Acetonitrile)
CD3OD_SPE	LC-SPE Solvent (Methanol-d4)
CDCl3	chloroform-d
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)
D2O	deuteriumoxide
D2O_salt	deuteriumoxide with salt
Dioxane	dioxane-d8
DMF	N,N-dimethylformamide-d7
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
H2O+D2O_salt	90%H2O and 10%D2O with salt
HDMSO	90%DMSO and 10%DMSO-d6
Juice	fruit juice
MeOD	methanol-d4
Plasma	blood plasma
Pyr	pyridine-d5
T_H2O+D2O+Me4NCl	(CD3)4NCl in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+NaAc	sodium acetate in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+Pivalate	pivalate-d9 in 90% H2O and 10% D2O, for NMR thermometer
T_MeOD	methanol-d4, for NMR thermometer
TFE	trifluroethanol-d3
THF	tetrahydrofuran-d8
Tol	toluene-d8
Urine	urine
	OK Cancel

> Locking and Shimming (steps in blue) only needs to be done <u>once</u> per sample.

(c) Data Processing:

- When the acquisition is completed, type "**ef**" to do Fourier Transform of the FID.
  (For 2D data, type "**xfb**" for Fourier Transformation)
- Type "**apk**" for Automatic Phase Correction
  (For 2D spectrum, use "**apk2d**")
- Type "**abs**" for Automatic Baseline Correction
  (For 2D spectrum, use "**abs2d**")

Tip: You can also process the data when it is being acquired. Type "**tr**" to transfer the data (Only for 1D) to disk that has been collected so far and then you process as mentioned above.

(d) Data Analysis:

Perform Spectrum Calibration, Peak peaking and Integration as shown during the training.

All data must be stored only under the current year and your group directory in the path.
(D:\<u>2021</u>\data\<u>Group</u>\nmr\)

Data stored only in this path will be backed up. Any data\files stored on C:\ will be deleted periodically.