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### *Dissimilar*

*This manual is intended to be only a very brief introduction for using the Bruker 400 MHz NMR spectrometer (PS751) at the California State University LA NMR Facility. For complete information about the automation program ICON-NMR or running experiments, please refer to the documentation that Bruker provides. Most of the information in this manual was taken from different Bruker manuals and modified and edited to fit our needs in this facility.*

*Dr. Ali Jabalameli  
November 03, 2008*

## **Short instructions for the basic operation of the AVANCE II 400 MHz Bruker NMR Using ICON-NMR (PS751)**



# Table of Contents

Table of Contents.....	2
1. Introduction to the ICON-NMR: .....	3
2. Sample Loading:.....	4
3. Creating a new data set and loading standard parameters: .....	5
4. Acquisition & Processing. ....	8
5. Exiting the ICON-NMR. ....	10
6. Command list ( <i>TOPSPIN</i> ). ....	11
7. Frequently asked questions:.....	12

# 1.Introduction to the ICON-NMR:

*Icon-nmr was designed to make the execution of routine NMR experiments easy and straightforward by providing a state of the art icon based user interface. The steps necessary to acquire, process, and plot a spectrum are reduced to inserting a new sample, defining the data file name, the solvent, and the experiment. No other knowledge about the instrument, the parameters, or the software is necessary. Although running on top and under the control of the computer's Windows operating system and Bruker's Topspin program, providing the acquisition and processing functions, Icon-nmr shields the user entirely from both. Accessing Topspin commands is only possible if permission has been granted by the laboratory manager. As such, Icon-nmr is particularly suited for open access spectrometers with a large number of users.*

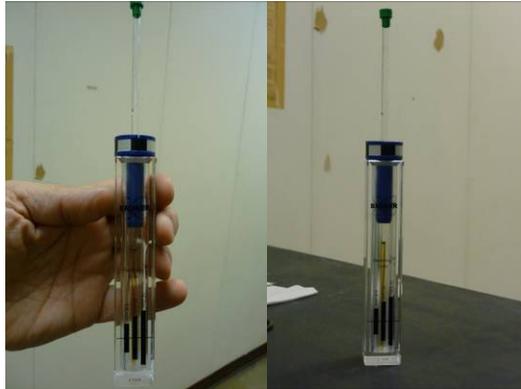


**WARNING:** Indicates the possibility of severe personal injury, loss of life or equipment damage if the instructions are not followed.

## 2. Sample Loading:

**STEP 1** Gently place your sample inside the spinner turbine. Use the depth gauge to measure the sample height as shown below.

**CAUTION: Do not use excessive pressure to insert your sample inside the spinner turbine**



**CAUTION: Do not forget to remove your sample from the depth gauge prior to inserting it into the magnet.**

**STEP 2** Carefully climb the stepladder and place your sample in a numbered holder. Be sure to note the sample holder number. Try to use whatever position is available from the beginning (#1).



**!!!WARNING! Be absolutely sure that no sample is in the magnet and the sample holder contains your sample before proceeding to the next step.**

# 3. Creating a new data set and loading standard parameters:

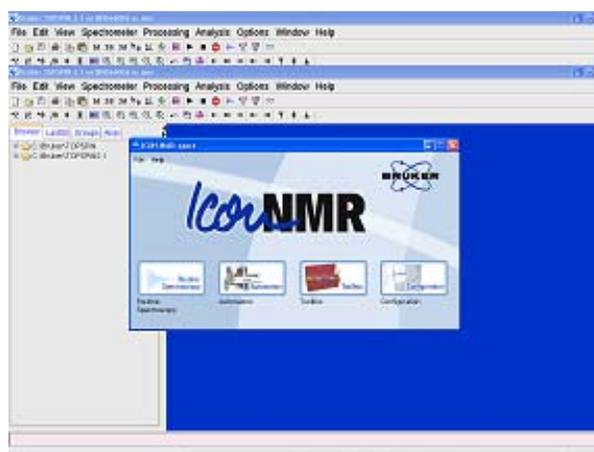
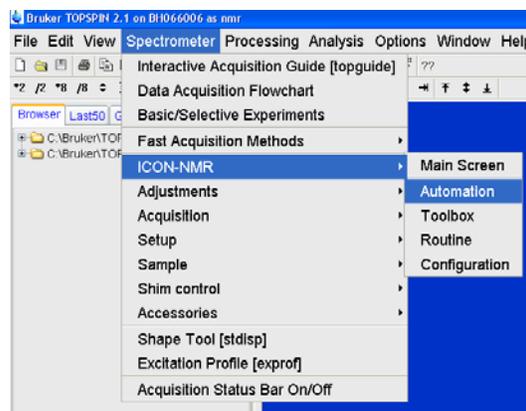
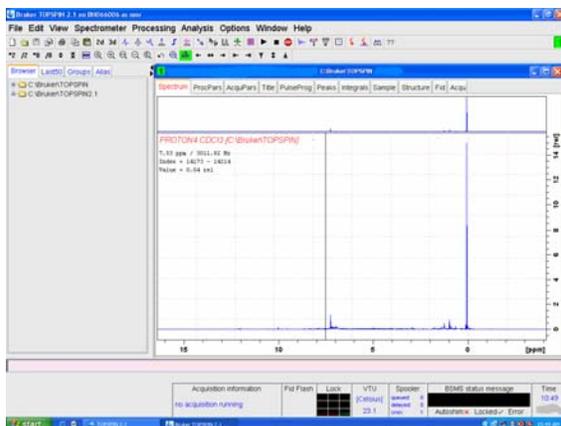
**STEP 1** Log into the nmr account on the spectrometer's workstation.



**STEP 2** Double click on the TOPSPIN icon  on your desktop.



**STEP 3** Once the TOPSPIN window appears, on the menu bar under Spectrometer/ ICON-NMR, click on "**Automation**" or at the prompt in the bottom left corner of TOPSPIN window type "**icon**" and then click on the automation icon.



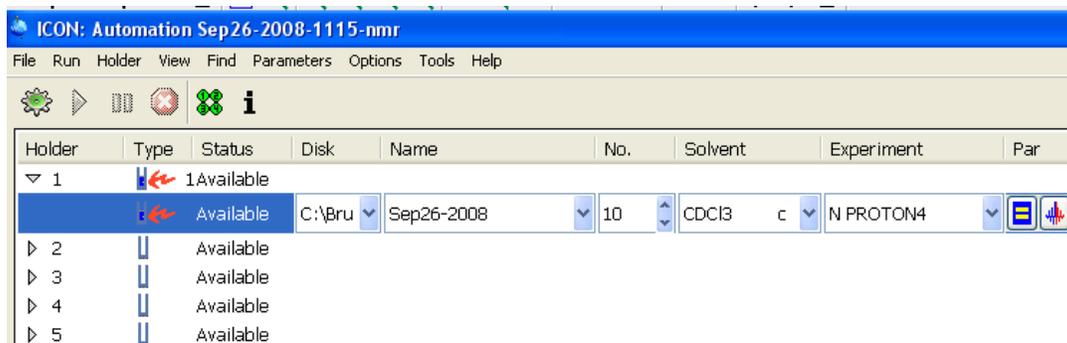
**STEP 4** Log into your ICON-NMR account by typing your user name in the “**ICON-NMR: Identify user**” window and then type your password in the next window.



**STEP 5** In the “**ICON: Automation**” double click on the sampler holder number that corresponds to the holder position which contains your sample.



**!!!WARNING! Be absolutely sure that no sample is in the magnet and the sample holder contains your sample before proceeding to the next step.**



**STEP 6** In the “**ICON: Automation**” window from the pull down menu select or type the required information; “**Name**” “**No.**” “**Solvent**” “**Experiment**” “**Par**” “**Title/ Org**” as shown below. You may leave some of the default information unchanged according to your experiment requirements.

**Name** (name of the data set)

Disk	Name	No.	Exp
C:\Bru	Sep26-2008	10	C

- Sep26-2008
- Sep26-2008-nmr
- 09262008
- 1-26-09-nmr
- 26092008-nmr

**Solvent** (choose the solvent used)

Solvent	Experiment	Par	Title / Or
CDCl3	c	N PROTON4	

- Acetic acetic acid-d4
- Acetone acetone-d6
- C6D6 benzene-d6
- CD2Cl2 methylenechloride-d2
- CD3CN acetonitrile-d3
- CD3CN\_SPE LC-SPE Solvent (Acetonitrile)
- CDCl3 chloroform-d

**Experiment** (N PROTON for proton and N C13CPD for carbon experiments)

Experiment	Par	Title / Orig	P
c	N PROTON4		

- N PROTON4 PROTON CDCl3 {C:\Bruker\TOPSPIN} test
- N C13CPD C13 exp. comp. pulse dec. 1024 scans
- N C13DEPT45 C13 dept all positive
- N Ali-13C(CPD) 13C(CPD) - 13C NMR Experiment
- N Ali-195Pt 195Pt - PROTON D20 {C:\Bruker\TOPSPIN} test
- N Ali-19F(CPD) 19F(CPD) - 19F NMR of 0.05 Tri
- N Ali-31PNMR(CPD) 31PNMR(CPD) - 31P NMR of 10% Phosj
- N Ali-cosy cosy - Cosy Experiment
- N Ali-hmbc hmbc - HMBC Experiment
- N Ali-hmgc hmgc - HMQC Experiment
- N Ali-noesy noesy - Noesy Experiment
- N Ali-roesy roesy - Roesy Experiment

**Par** (Important parameters; NS = number of scans, D1= delay between pulses)

Par	Title / Orig
TD	65536
NS	4
D1	1 [sec]
SW	20,5489 [ppm]
O1P	4.000 [ppm]
O2P	4.000 [ppm]
1TD	65536
1SW	20,5489 [ppm]

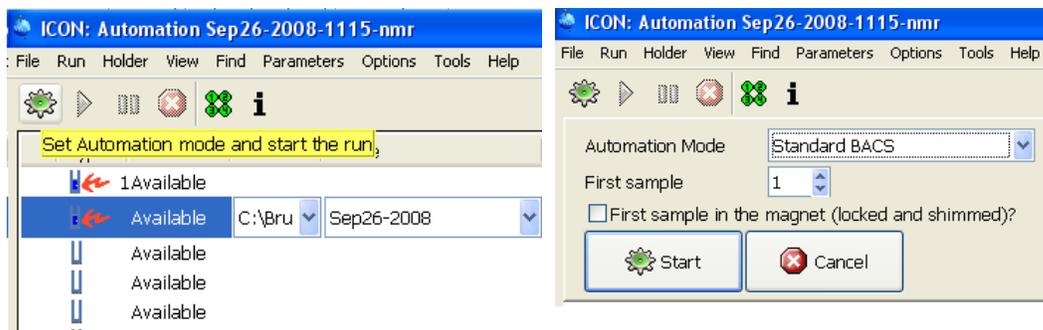
OK

**“Title/ Org”** (Type the title for your data)

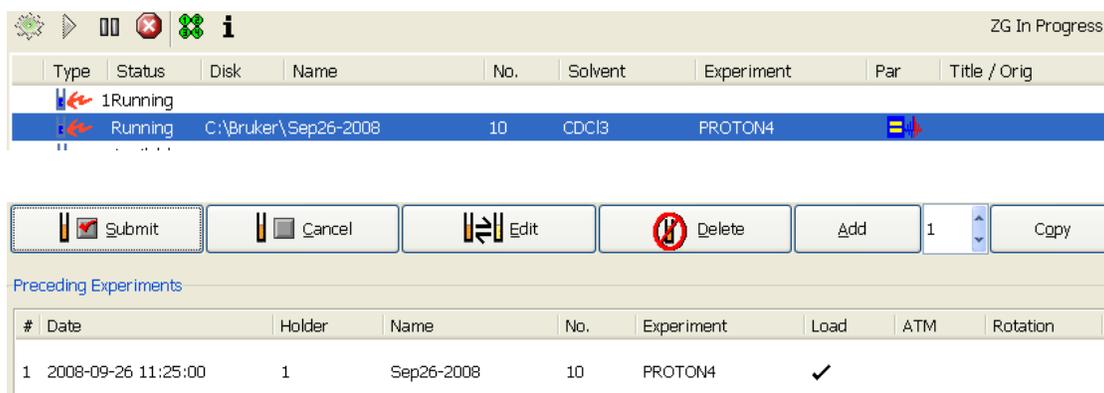
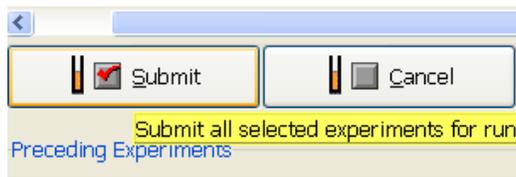
## 4. Acquisition & Processing.

**STEP 1** To start your experiment you need to start the automation by

clicking on  in the menu bar and then by clicking on 



**STEP 2** Once the automation starts click on submit, to submit and start your experiment. Check the status of your experiment and make sure it is under “Running” status and monitor the progress under “Preceding Experiments” window. **Your sample will be loaded automatically by the sampler changer.**



**STEP 3** When the experiment is finished, click on the experiment information under the “**Preceding Experiments**” window in order to display the processed spectrum. **Sample will be ejected and put back in the sample holder.**

Submit Cancel Edit Delete Add 1 Cgpy 1

Preceding Experiments

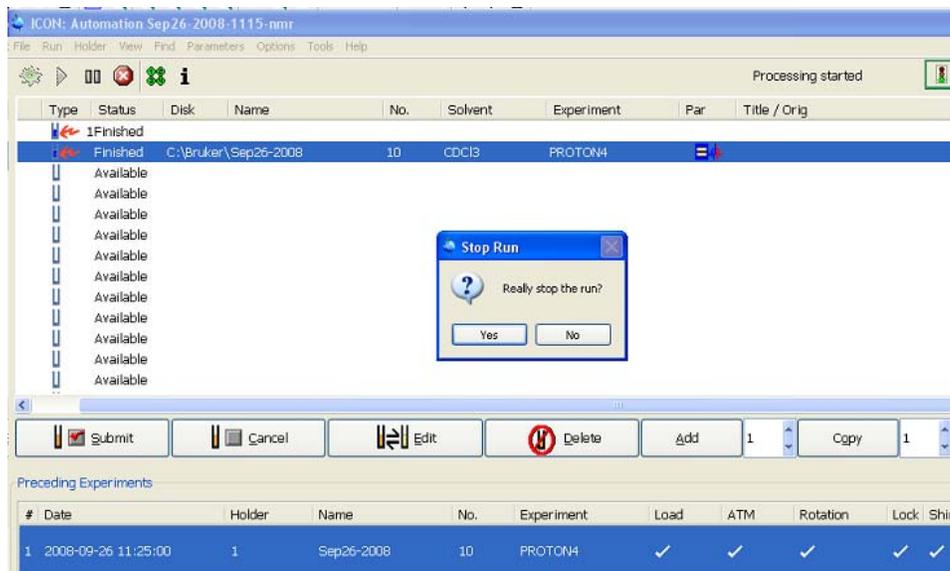
#	Date	Holder	Name	No.	Experiment	Load	ATM	Rotation	Lock	Shim
1	2008-09-26 11:25:00	1	Sep26-2008	10	PROTON4	✓	✓	✓	✓	✓

Acquisition information: no acquisition running  
 FID Flash: Lock [Cetus] 23.1  
 Spooler: queued 0, delayed 0, error 1  
 BSMS status message: Autoshim X Locked ✓ Error  
 Time: 10:49



## 5. Exiting the ICON-NMR.

**STEP 1** Wait until the last sample has finished running and has been ejected then stop the ICON-NMR program by first clicking on  (stops the automation program), and then by clicking on  as it shown below.



**STEP 2** Click on “Exit” to exit the ICON-NMR program under “File” under the “ICON-NMR spect” window.



You may process, print and manipulate your data using TOSPIN (refer to the TOPSPIN manual)

## 6. Command list (TOPSPIN).

*(All the command (bold words) must be typed at the prompt as they appear)*

### Loading the sample Commands:

**bsmsdisp** - starts BSMS windows

**ej** - lift the air up (eject sample)

**ij** - lift the air off (insert the sample)

### Locking, tuning and shimming Commands:

**lockdisp** - it generates the lock window.

**Lock solvent-name** – it will lock on the named solvent after several seconds. Picking the wrong solvent may result in the lock failure

**topshim** – starts automatic shimming

**gradshim** – invokes gradient shimming windows in which gradient shimming routine can be started.

**atma** - automatic tuning and matching

**atmm** – invokes manual tuning and matching window in which automatic routine can be started

**wobb**- invokes tuning and matching window.

### Creating a new data set Commands:

**new** – creates new data set

**edc** – creates new data set

**re filename** – read the file

**wr filename** – write a file

### Acquisition parameters Commands:

**eda** – it will display acquisition window

**ased** – display short list of acquisition window

**sw** – display spectral width

**o1p** – display transmitter center (center of spectrum)

**ns** – display number of scans

**rg** – display receiver gain

**rg** – automatic receiver gain adjustment

**d1** – display relaxation delay

**p1** – display pulse width (for simple 1D acquisition)

**aq** – FID acquisition time

**td** – display number of points used to define FID

**si** – display number of points used during FT (can be different from td)

**zg** – zero and go

### Processing Commands:

**edp** – it will display processing window

**em** - exponential multiplication on the FID, uses the parameter LB. This improves signal to noise at the expense of resolution.

**lb** - this controls the degree of broadening added and affects your signal-to-noise. To see its effect, simply change its value and re-Fourier Transform with **ef**.

**ft** - Fourier transform

**ef** - combines **em** and **ft**

**pk** - phase correct, applies the last phase correction to the spectrum. Useful when you have phased a preliminary spectrum, (with only a few scans) and wish to apply the same phase correction to the final spectrum.

**efp** - combines **em**, **ft**, and **pk**.

**abs**- automatic baseline correction

**apk** – automatic phase correction

## **7. Frequently asked questions:**

**Q1. How should I prepare my NMR sample?**

**Q2. Can I run more than one sample at a time?**

**Q3. Can I run more than one experiment for one sample?**

**Q4. Why is the sample changer not loading my sample, or why is my sample not ejected?**

**Q5. How do I improve the resolution for my spectrum? The peaks look awful!**

**Q1. How should I prepare my NMR sample?**

Dissolve your compound in a suitable deuterated solvent. You may have to filter the solution because there should not be any undissolved particles. Place the solution in an NMR tube with a cap. The sample height in the tube should be around 5.0 cm. Wipe and clean the outside of the sample tube.

[Click to go back to the menu](#)

**Q2. Can I run more than one sample at a time?**

Yes, you can run more than one sample at a time as long as you place your samples in different sampler holders and submit them all. They will be queued and run according to the numbers.

[Click to go back to the menu](#)

**Q3. Can I run more than one experiment for one sample?**

Yes, you can specify as many experiments as you wish as long as it is done prior to submitting your sample(s). Make sure to define the new experiment.

[Click to go back to the menu](#)

**Q4. Why is the sample changer not loading my sample or why is my sample not ejected?**

- 1- There is no air.
- 2- Air flow is too low.
- 3- Sample changer crashed and needs to be reset.

[Click to go back to the menu](#)

**Q5. How do I improve the resolution for my spectrum? The peaks look awful!**

1- Bad sample, wrong sample depth, not enough sample volume. Sample preparation is important. Make sure that you have enough sample solution; the sample height in the tube should be around 5.0 cm. Sometimes metallic impurities in the sample could cause that. Make sure your sample looks clear; any undissolved solid floating in your sample may have an undesirable effect on your spectrum.

2- Poor quality NMR tube could cause bad shimming.

[Click to go back to the menu](#)