FIVE steps to use IMSERC NMR

1. **Login to NUCORE with your netid and logon to the instrument:**
   a) turn on the computer monitor at instrument,
   b) your usage count starts

2. **Login to instrument with your operator id (usually same as netid)**

3. **Load your sample and run your experiment**

4. **Logout from the instrument**

5. **Login to NUCORE and logout your instrument session:**
   a) turn off the computer monitor at instrument,
   b) your usage count stops

Common Commands/Parameters in TopSpin

Setting up experiments & Processing

sx 10	to put #10 (could be any position) sample on the autosampler into magnet.
sx ej	to put sample inside magnet back to the autosampler
rga	automatically set receiver gain
zg	start acquisition
tr	transfer data (while acquisition is in progress)
multizg	start multiple acquisitions starting from current dataset
go	submit experiment to acquisition
stop	abort an acquisition, losing all the FID data recorded so far
halt	halt the running acquisition, saving the recorded FID data to hard disk
efp	weighted Fourier Transformation for 1D dataset
apk	do automatic phase correction
abs	automatically optimize baseline
xfb	weighted Fourier Transformation for 2D dataset

Important parameters for acquisition

P1	F1 channel 90° pulse width, micro seconds
P2	F1 channel 180° pulse width
RG	Receiver gain
D1	relaxation delay, 1 to 5 times T1
2TD	Time domain data points for F2 (direct dimension)
2SW	spectral width in ppm for F2 (direct dimension)
1TD	Time domain data points for F1 (indirect dimension)
1SW	spectral width in ppm for F1 (indirect dimension)
AQ	Acquisition time in seconds
NS	Number of scans
DS	Number of dummy scans
NUC1 – NUC8	Nucleus observed (1H, 13C, 31P, 19F, etc.)
O1 – O8	Frequency offset for channel 1 – 8 in Hz
O1P – O8P	Freq. offset for channels 1 – 8 in ppm
SFO1 – SFO8	Freq. for channels 1 – 8 in MHz

Workflow to interactively setup experiment with TopSpin
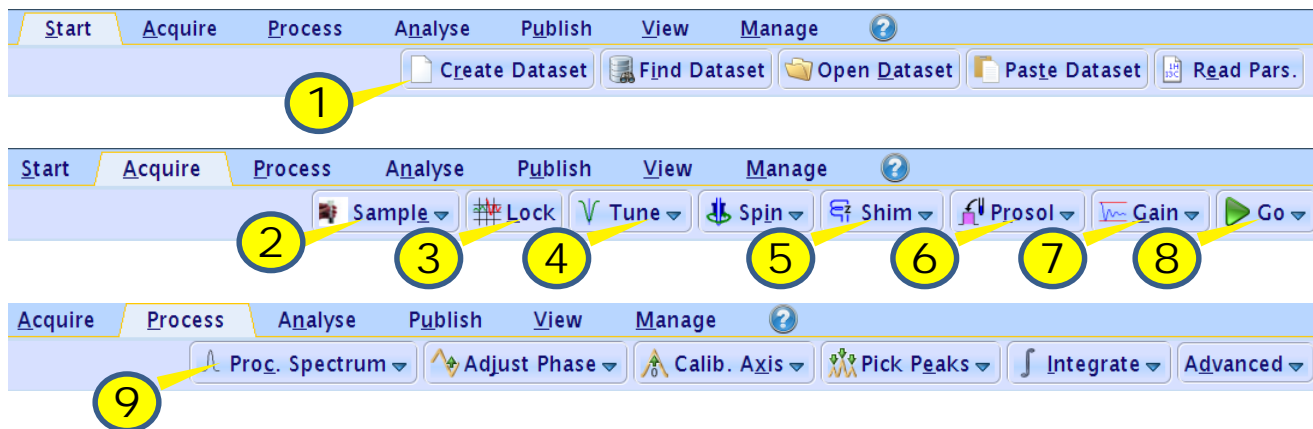
1. **Create new dataset and setup initial parameters**

2. Insert/Eject Sample

3. Lock on your selected Solvent

4. Tune/Match the Probe to the nucleus of your expt

5. Shim

6. Load probe related parameters

7. Auto set receiver gain

8. Acquire fid

9. Process data

The **red arrowed** fields below need to be filled, **red circled** parameters in next page can be changed based on your need

Create New Dataset - new

Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments several datasets are created. Please define the number of receivers in the Options.

Sample name → NAME `yw1-042`

Experiment number → EXPNO `3`

PROCNO `1`

○ Use current parameters

Experiment to run → ● Experiment `13C` [Select]

⌄ Options

Solvent used → ☑ Set solvent `C2D2Cl4`

● Execute 'getprosol'

○ Keep parameters `P 1, O1, PLW 1` [Change]

Directory for the dataset (your folder under your group name) → DIR `/home/walkon/data/Marks/ygq763`

☑ Show new dataset in new window

Number of additional datasets: (1,2, ...16) `1`

TITLE

[OK] [Cancel] [More Info...] [Help]

Parameters setting for 1D and 2D experiments

1 presat 1 1 /home/walkon/data/zhang

Spectrum	ProcPars	AcquPars	Title	PulseProg	Peaks	Integrals	Sample	Structure	Plot	Fid	Acqu

A ⌂ ▮ ▦ C ▽ 🔍   Probe: PA BBO 600S3 BB-H-D-05 Z BTO

General
Channel f1

⌃ General

PULPROG	zgpr	... E	Pulse program for acquisition
TD	32768		Time domain size
SWH [Hz, ppm]	9615.38	16.0212	Sweep width
AQ [sec]	1.7039360		Acquisition time
RG	203		Receiver gain
DW [µsec]	52.000		Dwell time
DE [µsec]	6.50		Pre-scan-delay
D1 [sec]	2.000000000		Relaxation delay; 1–5 * T1
d12 [sec]	0.00002000		Delay for power switching [20 usec]
DS	0		Number of dummy scans
NS	4		1 * n, total number of scans: NS * TD0
TD0	1		Number of averages in 1D

⌃ Channel f1

SFO1 [MHz]	600.1678208		Frequency of ch. 1
O1 [Hz, ppm]	2820.78	4.700	Frequency of ch. 1
NUC1	1H	Edit...	Nucleus for channel 1
P1 [µsec]	12.700		F1 channel – 90 degree high power pulse
PLW1 [W, dB]	31.623	–15.00	F1 channel – power level for pulse (default)
PLW9 [W, dB]	0.00020402	36.90	F1 channel – power level for presaturation

1 tp35 12 1 /home/walkon/data/zhang

Spectrum	ProcPars	AcquPars	Title	PulseProg	Peaks	Integrals	Sample	Structure	Plot	Fid	Acqu

∏ S ▮ ▦ 1,2 ▽ C 🔍   Probe: PA BBO 600S3 BB-H-D-05 Z BTO

Experiment
Width
Receiver
Nucleus
Durations
Power
Program
Probe
Lists
NUS
Wobble
Lock
Automation
Miscellaneous
User
Routing

	F2	F1	Frequency axis

⌃ Experiment

PULPROG	hsqcetgp	... E	Current pulse program
AQ_mod	DQD ▼		Acquisition mode
FnTYPE	traditional(planes) ▼		nD acquisition mode for 3D etc.
FnMODE		Echo–Antiecho ▼	Acquisition mode for 2D, 3D etc.
TD	1024	256	Size of fid
DS	16		Number of dummy scans
NS	8		Number of scans
TD0	1		Loop count for 'td0'
TDav	0		Average loop counter for nD experiments

⌃ Width

SW [ppm]	20.0264	165.0000	Spectral width
SWH [Hz]	12019.230	24902.283	Spectral width

2 RR 3 1 /home/walkon/data/zhang

Spectrum	ProcPars	AcquPars	Title	PulseProg	Peaks	Integrals	Sample	Structure	Plot	Fid	Acqu

∏ S ▮ ▦ 1,2 ▽ C 🔍   Probe: PA BBO 600S3 BB-H-D-05 Z BTO

Experiment
Width
Receiver
Nucleus
Durations
Power
Program

⌃ Nucleus 1

NUC1	1H	Edit...	13C ▼	Observe nucleus
O1 [Hz]	2820.78		11318.37	Transmitter frequency offset
O1P [ppm]	4.700		75.000	Transmitter frequency offset
SFO1 [MHz]	600.1678208		150.9229277	Transmitter frequency

Setup experiment using ICONNMR w/ SampleXpress

1. Login with your operator ID
2. Load your sample to SampleExpress
3. Click the Holder # where you sample is loaded.
4. Click Add
5. Fill in following fields: Name, No., Solvent, and Experiment
6. Change parameters if needed
7. Click Submit
8. Logout ICON by clicking on "change user"

Message display ←

Displays the cassette with its 60 positions.
- Gray positions are empty positions.
- White positions are loaded with samples.

Information about Operator Access Position and Sample inside magnet.

Touch screen buttons to **Add** and **Remove** samples.
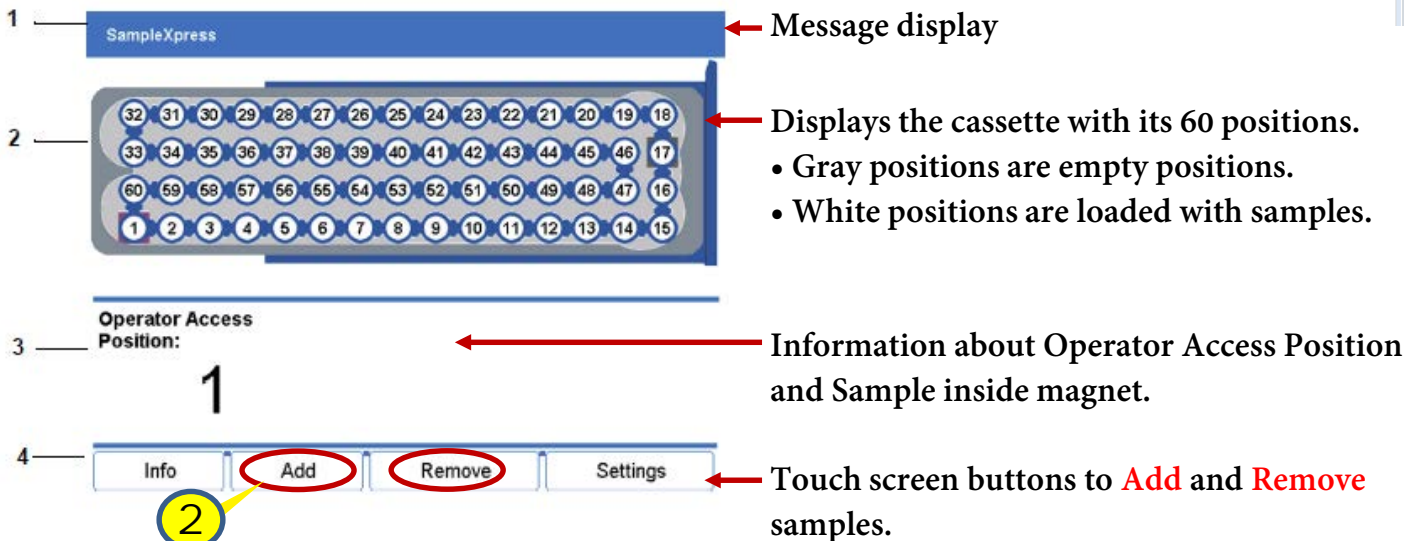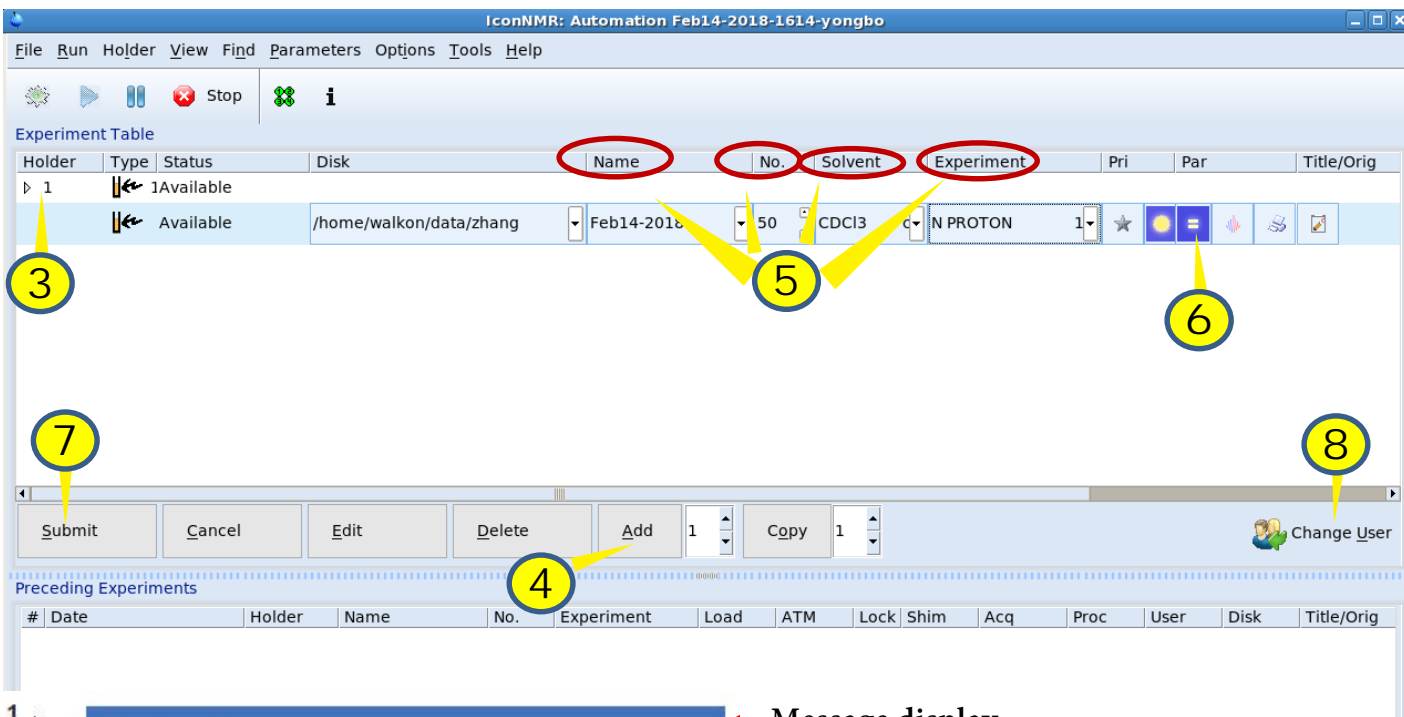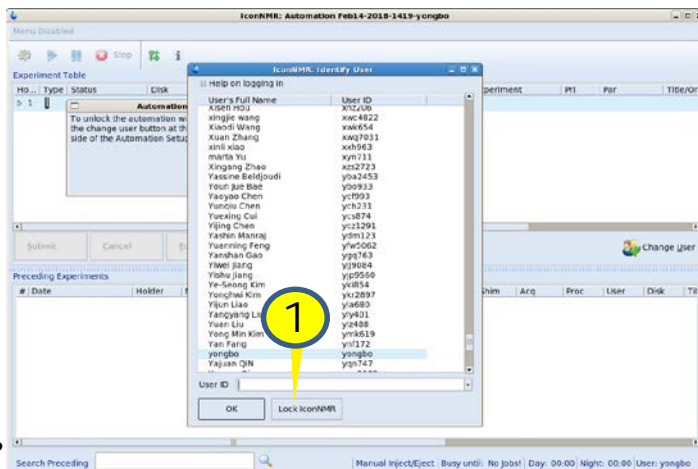
Setup experiment using ICONNMR w/o autosampler

1. Login with your operator ID
2. Click the Holder *1*
3. Click Add
4. Fill in following fields: Name, No., Solvent, and Experiment
5. Change parameters if desired (add additional experiments if needed)
6. Click Submit
7. Insert your sample when promoted
8. (optional) If you sample is not ejected automatically after finishing, switch lock sample in