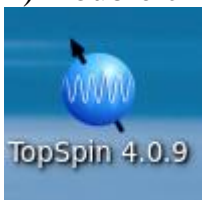


## Bruker 500 Tutorial

### Acquisition of 1D $^1\text{H}$ and $^{13}\text{C}$ spectra

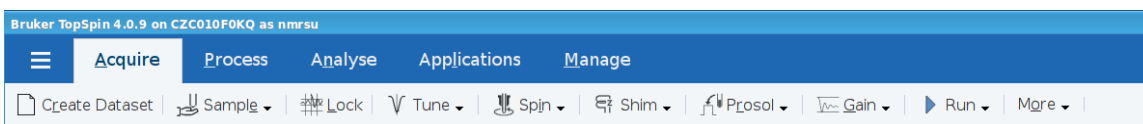
**DO NOT PUT J. YOUNG TUBES ON THE AUTOSAMPLER. ASK THE OPERATOR FOR HELP.**

- 1) Log in the linux account.
- 2) Double click on the TOPSPIN icon to start the program.



- 3) Put your sample into a spot in the AutoSampler and remember the number of the spot. Use a spot to the left of the insert tube. **DO NOT USE J. YOUNG TUBES!!**

- 4) Click on tab “Acquire”.



The row below Acquire that starts with “Create Dataset” has all the setup steps that you have to follow, one by one, until “run”, running the sample.

5) Click on “Create Dataset”. The window shown below pops up;

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 Advanced field.

Dataset

NAME Test1

EXPNO 1

Directory bin4.0.9/data/Operator/Carlos

Open in new window

Parameters

Use current parameters

Read parameterset  Select

Set solvent Acetic

Additional action

No additional action

Execute getprosol

Keep parameters P 1, O1, PLW 1 Change

Advanced

Number of datasets (receivers) 1

Title

OK Cancel More Info... Help

6) In the Dataset section of the “Create New Dataset” window;

- a) NAME; type a name for the Dataset.
- b) EXPNO; type “1”
- c) Directory: /opt/topspin 4.0.9/data/(group name)/username

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 Advanced field.

Dataset

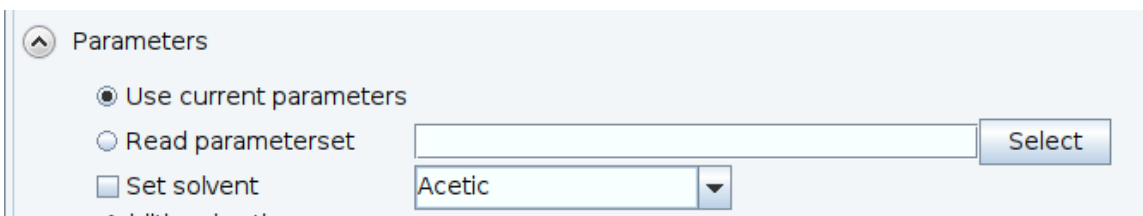
NAME Test1

EXPNO 1

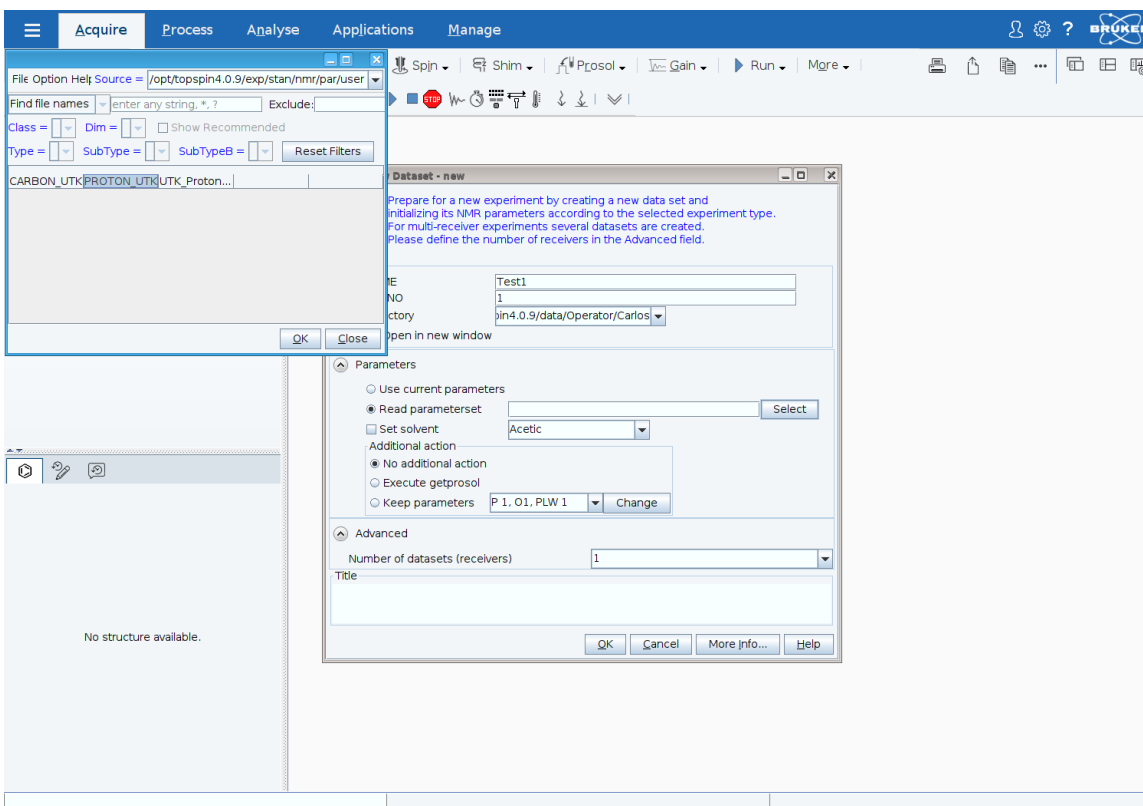
Directory bin4.0.9/data/Operator/Carlos

Open in new window

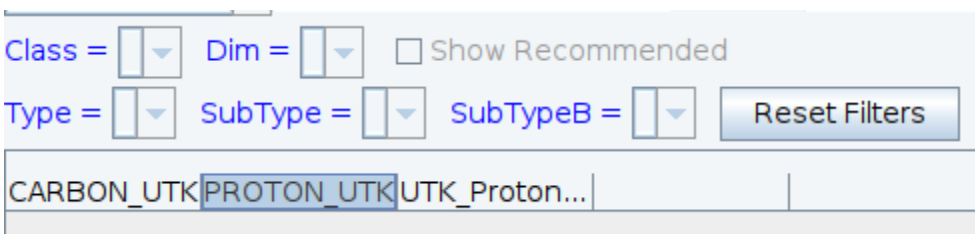
7) On the Parameters section of the window;



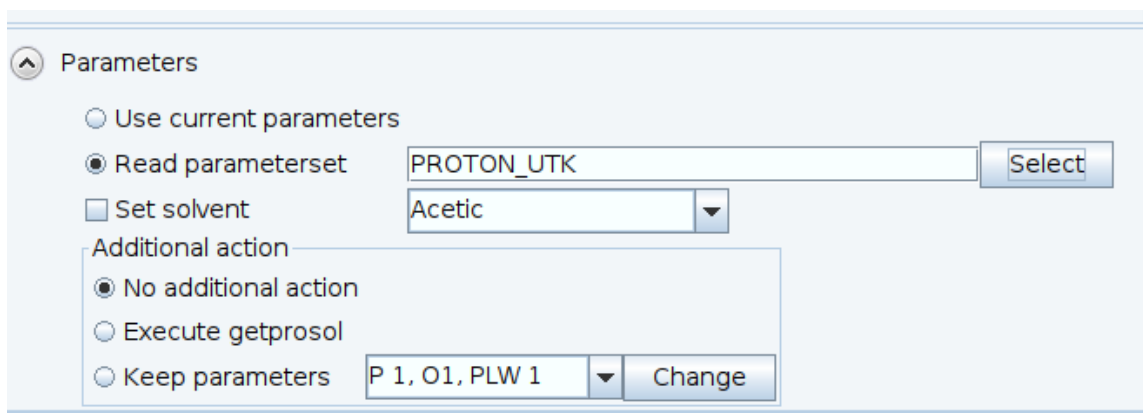
a) “Read parameterset”, click <Select>. The window shown below pops up.



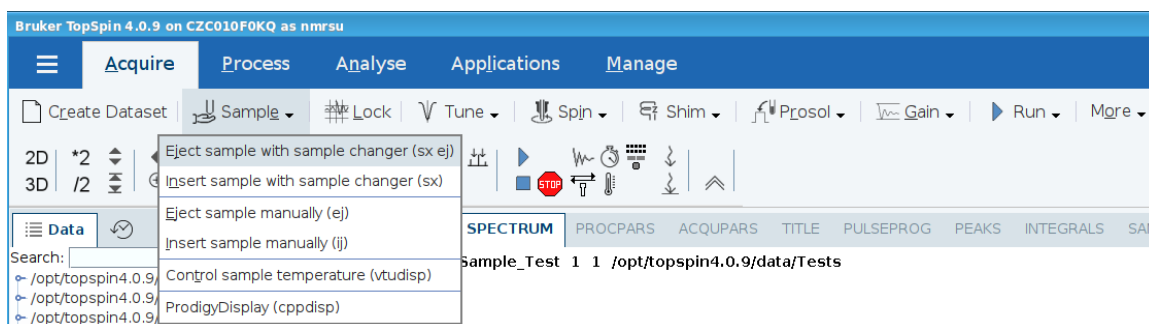
8) Select “PROTON\_UTK” and click <OK> to close the window.



9) Select the solvent and click <OK> to close the window.



10) Click on “Sample” and select “Insert sample with sample changer (sx)”, as shown in the picture below.

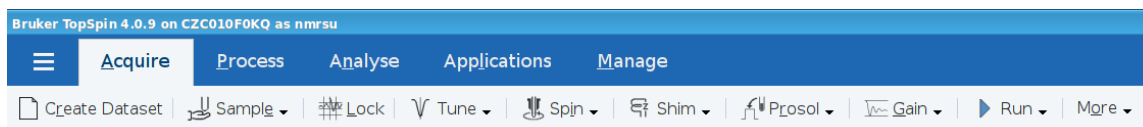


11) In the window that pops up enter the number # of the position of your sample.

(Alternatively, type sx # to insert your sample.)

**To recover your sample, type “ej” in the command line.**

12) click on “Lock”. A window with the solvents will pop up.



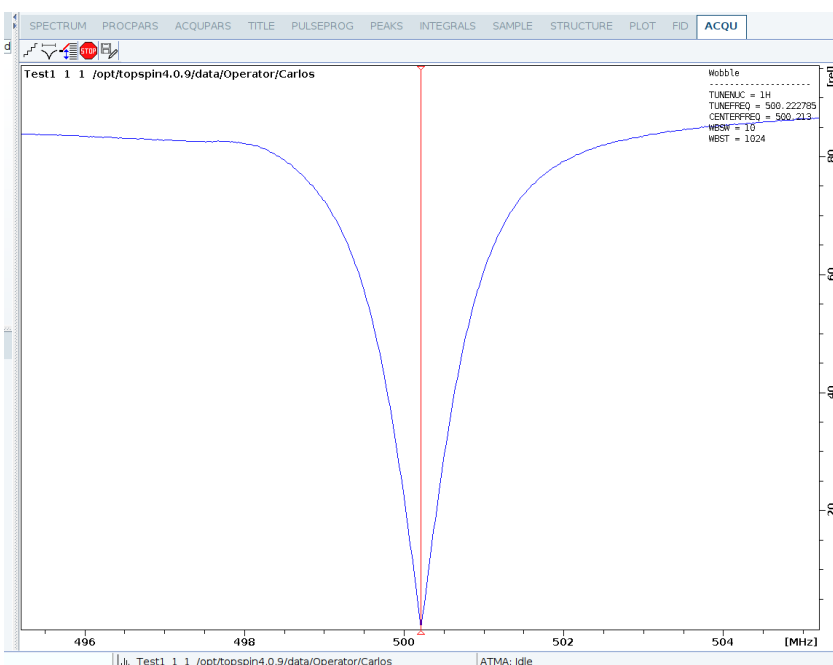
13) Select the solvent and click <OK> to close the window.

Δ 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)
<b>CDCl3</b>	<b>chloroform-d</b>
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)

Wait until the lock process is finished. At the bottom left, above the task bar, “lockn: done” is displayed, as shown in the figure below.

lockn: done						
Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler	Time
				<b>Corr. 300.0 K</b>	queued: 0 delayed: 0 cron: 0	<b>15:34:50</b> <b>Sep 29</b>
				On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>		

14) click on “Tune”. It is slow to start. Then, the probe mode shown below shows up.



Wait until the tuning process is finished; “ATMA: successfully finished”.

ATMA: successfully finished						
Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler	Time
			✓	<b>Corr. 300.0 K</b> On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>	queued: 0 delayed: 0 cron: 0	15:40:28 Sep 29

15) click on “Spin” and wait until the spin process is finished. Sample show spinning. **DO NOT spin J. YOUNG TUBES!!**

Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler
			✓	<b>Corr. 300.0 K</b> On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>	queued: 0 delayed: 0 cron: 0

16) click on “Shim” and wait until the shim process is finished; “topshim: completed successfully”.

topshim: completed successfully						
Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler	Time
			✓	<b>Corr. 300.0 K</b> On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>	queued: 0 delayed: 0 cron: 0	15:36:09 Sep 29

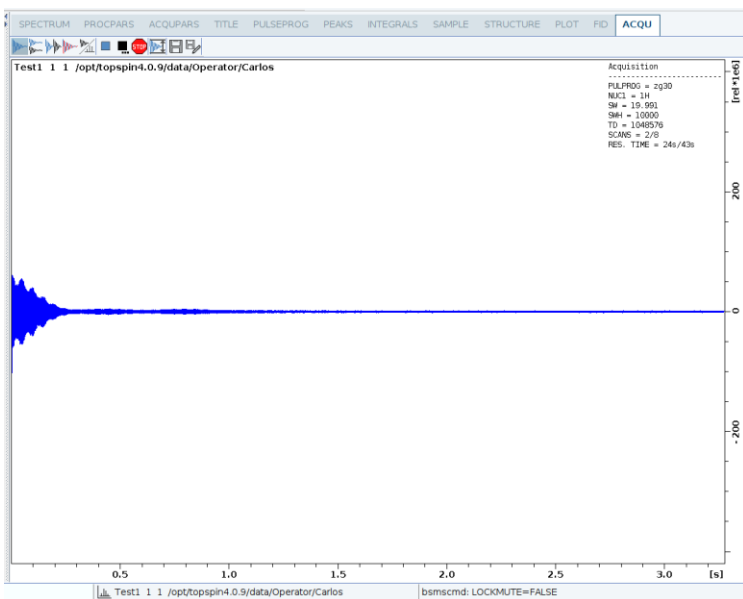
17) click on “Prosol” and wait until the getprosol process is finished; “getprosol finished”.

getprosol finished!						
Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler	Time
			✓	<b>Corr. 300.0 K</b> On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>	queued: 0 delayed: 0 cron: 0	15:36:25 Sep 29

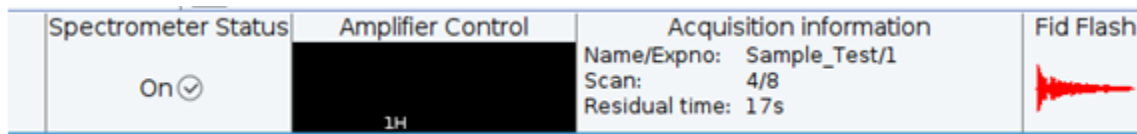
18) click on “Gain” and wait until the gain process is finished; “rga finished”.

rga finished						
Fid Flash	Lock	Sample	POWCHK	Sample Temperature	Spooler	Time
			✓	<b>Corr. 300.0 K</b> On <input checked="" type="checkbox"/> Reg. State: <input checked="" type="checkbox"/>	queued: 0 delayed: 0 cron: 0	15:36:48 Sep 29

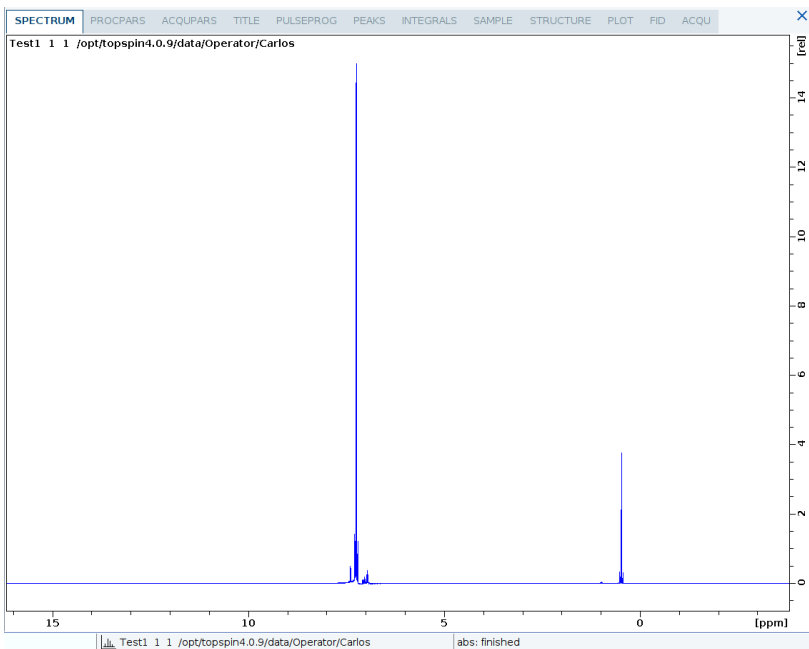
19) click on “Run”. After the dummy scans, the FID starts to be acquired.



Acquisition information, including residual time are displayed on the task bar.

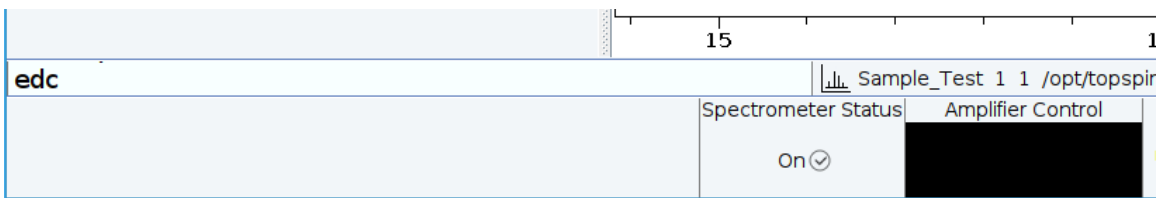


20) Once the acquisition is finished, the spectrum is displayed.

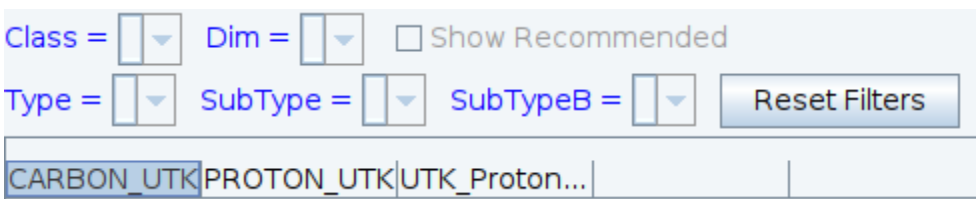


## Running a carbon experiment.

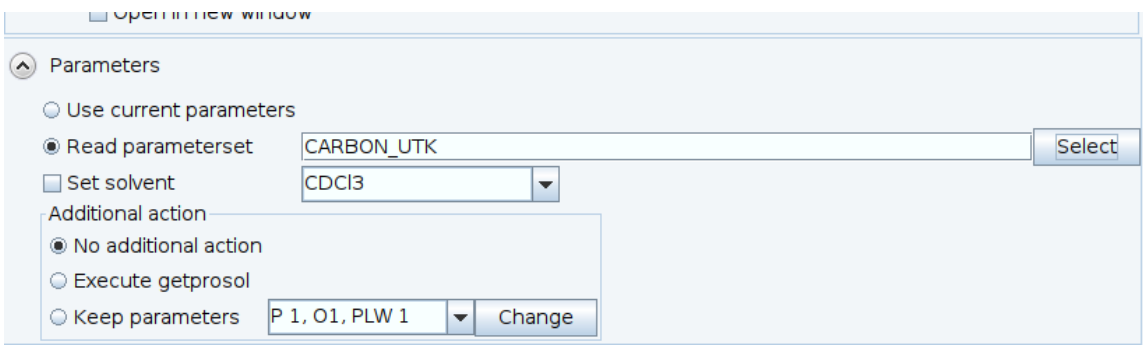
1) Type “EDC” in the command line, then press <Enter>



2) In “Read parameterset”, click on <Select> and select CARBON\_UTK.



3) click <OK> to close the window



4) click <OK> to close the EDC window.

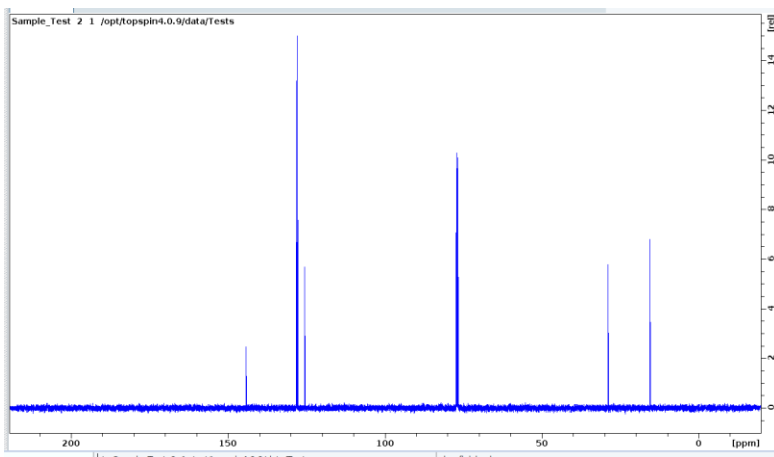
5) click on “Tune”

6) click on “Prosol”

7) click on “Gain”

8) click on “Run”. Spectrum is displayed after the acquisition is finished.





### **tr command**

To observe a  $^{13}\text{C}$  spectrum while is running;

- a) type “tr” on the command line.
- b) type “efp” on the command line.

**When you are done with your work, type “ej” in the command line to recover your sample.**

### **Usefull commands:**

stop : stops the experiment but doesn't save the data.

halt : stops the experiment and save the data acquired at to that point.

ej : ejects the sample inside the magnet.

sx # : insert sample in position # in the autosampler.

ns : number of scans

d1 : delay between scans

tr : allows to observe the spectrum while acquiring it.

ased : displays the parameters of the NMR experiment.

efp : automatic line broadening, Forier Transform and phase correction.

apk : automatic phase correction of the spectrum.

lb : line broadening value

## **Retreiving data from the spectrometer computer using SSH**

- 1) Open the WinSCP program.
- 1) click on <NEW> .
- 2) Fill in the windows that pops up: host name: romeo.chem.utk.edu, user name: .... , and password:.....
- 3) click on <save>.
- 4) click <OK> on next window.
- 5) when trying to connect for the first time, a windows pops up. Just click on <yes>.
- 6) the right panel is the host computer, change the directory to /opt/topspin/data/groupsaccount/user/.
- 7) download the datasets.
- 8) once you are done, disconnect from the host computer.