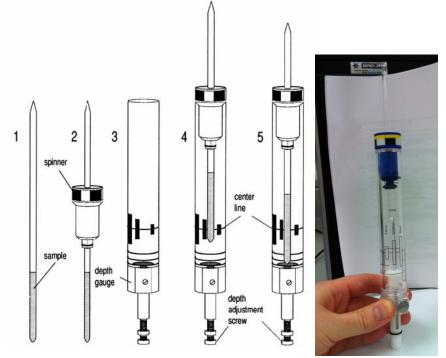
Bruker Fourier 300 Guide

Loading a sample

- Check your solvent height. For best shimming homogeneity, the sample height should be 5 cm.
- Wipe the exterior of the tube clean using a Kimwipe.
- If present, remove the cap from the top of the magnet bore.



• Type "ej" in the command line at the bottom of the screen to turn the lift on.



- •
- Use the depth gauge to adjust the spinner height on the tube.
- Carefully place your sample into the bore. Make sure air flow is on before releasing sample!
- Type "ij" to turn off the lift.

Experiment Set-up

• Click on the 'Start' tab in the TopSpin Menu bar

<u> </u>	<u>A</u> cquire <u>P</u>	rocess A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0
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Select Create	e Dataset by o	clicking on it.				
🖕 New	by	chenning off he.	×			
Prepare for a new exp						
For multi-receiver exp	periments several d		ent type.			
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PROCNO	1					
O Use current param	ieters					
Experiment 1H (16)	Scans)		Select	-	Select t	ype of NMR experime
(a) Options						
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 Set solvent. Execute "get 	procol!	CDCIS			Select S	orvent
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O Reep parame	21013.	F I, OI, FLW I	Change			
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Receivers (1,2	,16)	1				
				-		
test 9 ◄					Title (wil	I appear at top of spec
TITLE						
	OK	Cancel More I	nfo Help	1		

- Give your experiment a NAME and an experiment number (EXPNO)
- Make sure your data directory (DIR) is set to C:\data\student\nmr
- Press the "Select" button next to Experiment, and choose which NMR experiment you will carry out.

Find file names enter any string, *, ? Exclude Clear Class = Any Dim = Any Show Recommended Can change pa Type = Any SubType = Any SubTypeB = Any Reset Filters Can change pa C13APT C13CPD C13CPD32 C13CPDSN C13DEPT90 C13HUMP C13IG C13RESOL C13SENS COSY90SW COSYDQFPHSW COSYGPFHSW COSYGPFIXSW COSYOPSW COSYDQFPHSW COSYGPPH gradshim1d1h_f MBCGPND HMQCGP HSQCEDGPPH HSQCEGPPH MLEVPHSW napper NOESY NOESYGPPHSW PROTON128 PROTONCONLF PROTONEXP PROTONLF PROTONNR PROTONNREXP PROTONNRLF PROTONT1 ROESYPHSW standard1D standard2D WATER	rameter folder her
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PROTONNR PROTONNREXP PROTONNRLF PROTONT1	PROTON
	PROTONLFEXP
ROESYPHSW standard1D standard2D WATER	ROESYPHPR
Click here once you select your experiment	

- Once you have clicked on the experiment you wish to run, be sure to click the "Set selected item in editor" button.
- Note: If you change the parameter folder to nmr\par\user, you will be presented with a much more manageable set of experiments!

🖨 Parameter Sets: rpar			×
File Options Help	Source = C:\Bruker\TopSpi	n3.2.2\exp\stan\nmr\par	\user •
Find file names venter any string, *, ? Exclude: Clear			
Class = Any Dim = Any Show Recommended			
Type = Any SubType = Any SubTypeB = Any Reset Filters			
1H (128 Scans) 1H (16 Scans) C13 (1024 Scans)	C13 (32 Scans)	C13DEPT135	
C13DEPT90 gradshim1d1h_f gradshim1d2h_f KetoEnol - 1H (4 Scans) KetoEnol - 1H (4 Scans)	Keto-Enol 1H (16 Scans)	KetoEnol - 1H (4 Sca	ins)
	Set sele	cted item in editor	Close

 \geq

- Choose the correct solvent from the drop down menu next to "Set solvent".
- Enter a Title for your experiment.
- Click on OK
- Click on the '**Acquire**' tab in the TopSpin menu bar.

	<u>S</u> tart	Acquire	Process	A <u>n</u> alyse	P <u>u</u> blish	⊻iew	<u>M</u> anage	2	
	💐 Sa	mple 🚽 🏥	Lock 🕹 S	Sp <u>i</u> n → 🗧 S	Shim 🚽 🔏	Pr <u>o</u> sol →	<u>Main</u> ▼	▶ Go →	Options 🗢

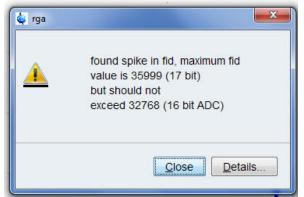
• Type **lock** in the command line at the bottom of the screen, or click on the "Lock" button. Select your deuterated solvent from the list that appears.

Solvents table	×		
△ Solvent	Description		
Acetone	acetone-d6		
CD3CN	acetonitrile-d3		
CDCI3	chloroform-d		
D2O	deuteriumoxide		
DMSO	dimethylsulfoxide-d6		
H2O+D2O	90%H2O and 10%D2O		
	OK Cancel		

- - Click on the "OK" button, and wait for the lock routine to finish. At the bottom left corner of the screen, a message "lockn: finished" should appear.
- Select \square shim \square by clicking on the icon.
- Wait for the shimming to finish before continuing. It should take about 1-minute to shim. The message "gradshimau: finished" will appear in the lower left corner of the screen once gradient-shimming is complete.
 - Note: shimming only needs to be performed once per sample. If you run a 13C experiment after a 1H experiment, this step can be omitted.
- Select **A Prosol** by clicking on it
 - NOTE: This will load the pulse width and power levels in to the parameter set.

Acquisition

- Select **Gain** by clicking on it.
- Click on **Close** if the following window appears:



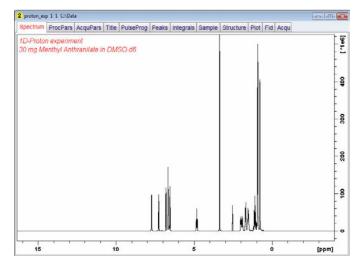
- Wait for the receiver gain auto-adjust (rga) procedure to finish before continuing. The message "rga: finished" will appear in the lower left corner of the screen once the receiver gain has been automatically set.
- Select **Goo** by clicking on it.
 - NOTE: The 1H (16 scans) experiment will take \sim 2 minutes.

Processing

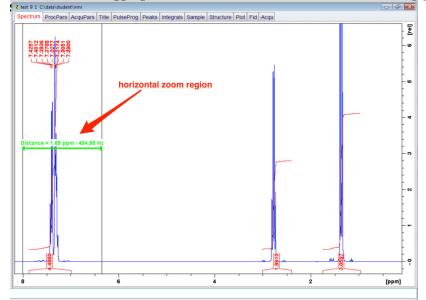
• Click on the '**Process'** tab in the TopSpin Menu bar.

	<u>S</u> tart	<u>A</u> cquire	Process	A <u>n</u> alyse	e P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0	
	∿ Pro <u>c</u> . S	Spectrum 🔫	Adjust P	hase 🗢	Å Calib. A <u>x</u> is 🚽	N P	ick P <u>e</u> aks –	∫ <u>I</u> ntegrate →	Advanced 🗢

- Click on <sup>∧ Pro<u>c</u>. Spectrum →
 </sup>
 - NOTE: This executes a processing program including commands such as an exponential window function 'em', Fourier transformation 'ft', an automatic phase correction 'apk' and a baseline correction 'abs'.



• Expand the spectrum to include all peaks. This can be done by left clicking inside the spectrum window, and dragging from one side to the other, and then letting go of the mouse button.



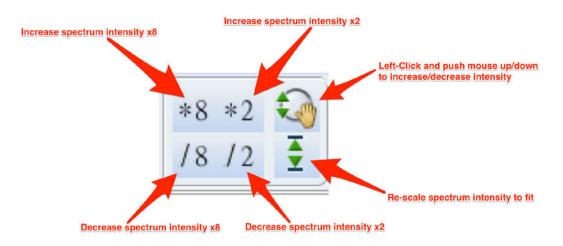
2 test 9 1 C/\data\student\nmr	
Spectrum ProcPars AcquPars Title PulseProg Peaks Integrals Sample Structure Plot Fid Acqu	
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7.8 7.6 7.4 7.2 7.0 6.8	6.6 [ppm]

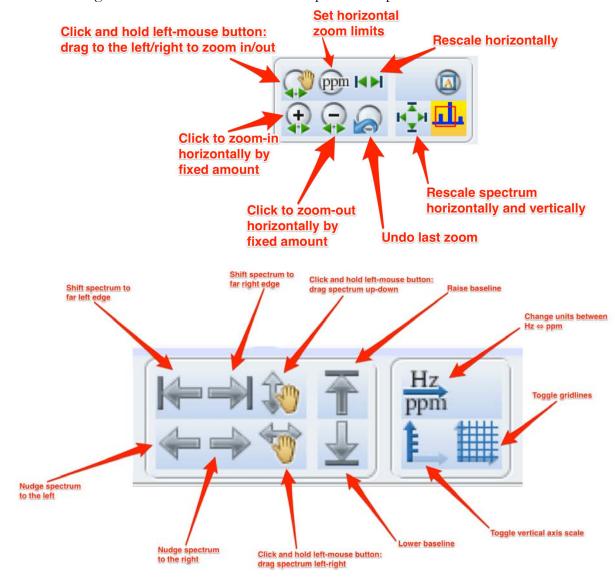
• Another option is to click the button, and type in the desired left and right ppm values.

Please enter to of the desired	the exact coordinates Lexpansion.
	[ppm]
From	10

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- Spectrum intensity (vertical-zoom) can be increased/decreased by pushing the mouse scrollwheel forwards/backwards.
- You can also use the icons below:

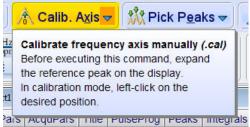




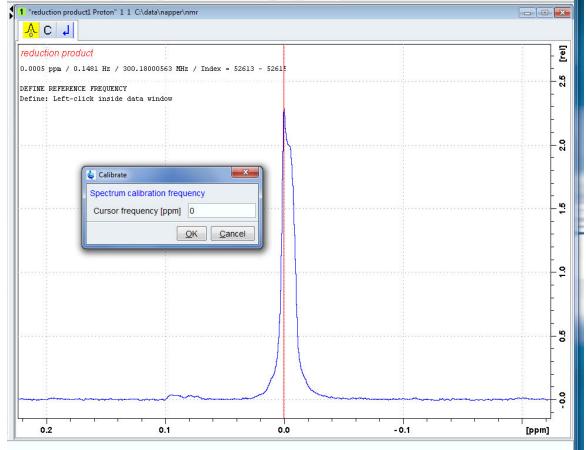
• The following toolbar icons can be used to manipulate the spectrum.

Axis Calibration

- If you are using an internal standard such as TMS (tetramethyl silane), you can set the position of a particular peak quite easily.
- Zoom in on the peak whose position you wish to set.
- Click on the Calib. Axis. Button.



- - Left click on the peak you wish to set.

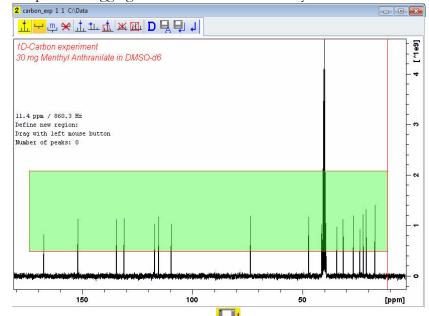


•

• Type in the peak position in the dialog box the appears.

Peak Picking

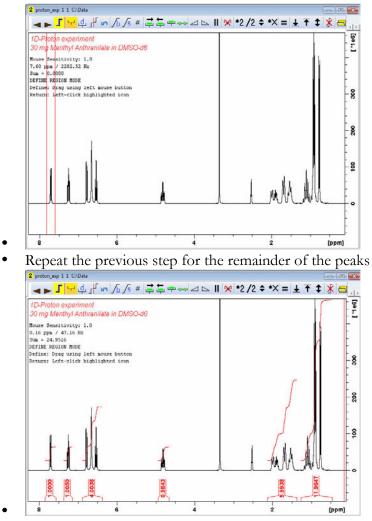
- Click the peak picking button **Pick Peaks** on the toolbar.
- Drag the cursor over the tip of a peak to select it. The green box only needs to enclose the tip of the peak—dragging the box to the baseline may cause noise to be identified as peaks.



- Click the save and return button when you are done picking peaks.
- If you wish to pick additional peaks, be sure that the select region icon $(\overset{\frown}{})$ is highlighted.

Integration

- Click on ∫ Integrate
- Set the cursor line, starting at the left of the spectrum, to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button.



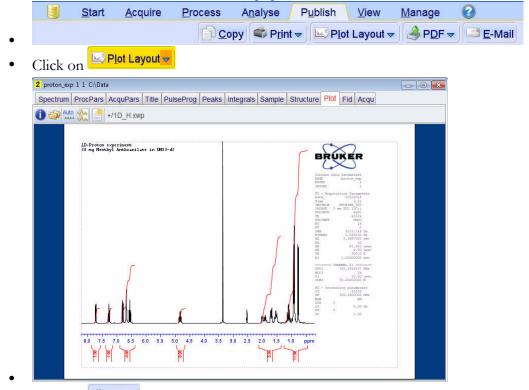
- Note: the first peak integrated will be assigned an area of 1.000.
- To change the integration values, right click on the integration mark below the peak, and select **Calibrate Current Integral** from the menu that appears. Type in the new value of the integral from the dialog box that appears.



• Click on 🔁 to save the integration regions.

Printing

• Click on the '**Publish**' tab in the TopSpin Menu bar.

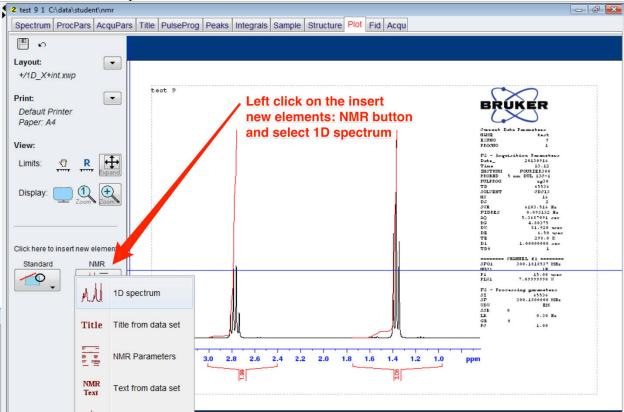


• Click on **Print** to plot the spectrum.

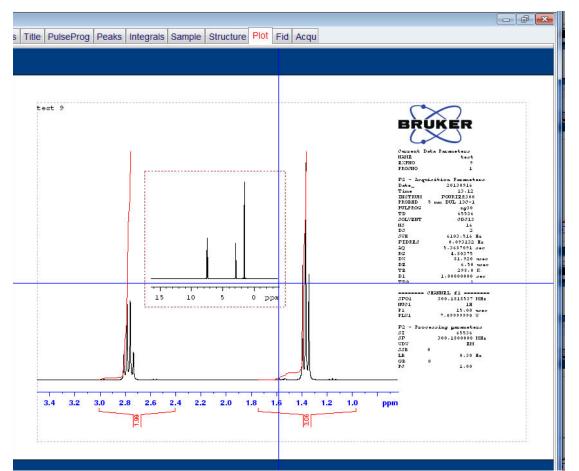
Advanced Topics

Creating insets

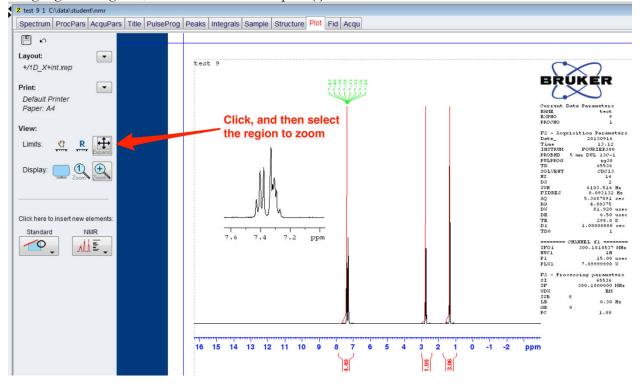
While in the Plot Layout mode (Publish \longrightarrow Plot Layout), click on the Insert New Elements, NMR icon, and select 1D spectrum.



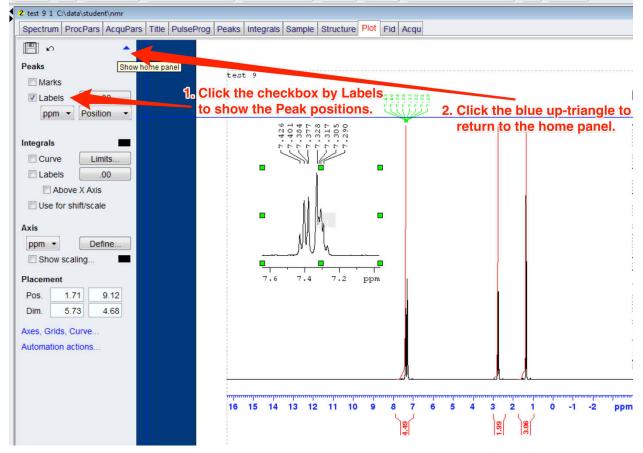
By clicking and holding the left-hand mouse button, draw out the region where the insert will be placed.



To zoom in on the insert, click on the Expand icon, then hover over the insert window until you see it highlighted in green, then zoom in on the peak(s) of interest.



To show peak positions, etc. in the inset, select the inset region, then click the checkbox next to Labels under the Peaks sections. Click the blue up-triangle once you are finished. If the Browser window has been hidden from display, press Control-D to toggle its visibility.



Some Useful TopSpin Commands

Instrument Setup

Create dataset	edc or new
Turn on eject gas	ej
Insert sample and turn off eject gas	ij
Turn spin gas on	ro on
Load shims	rsh
Open lock display	lockdisp
Turn lock on	lock

Experiment Setup and Acquisition

Adjust parameters (ns, sw, d1, etc.)	ased
Set receiver gain	rga
Start experiment	zg
Transfer acquired FIDs	tr
Add more scans to FID	go
Change number of scans to 32 (say)	ns 32
Change pre-acquisition delay to 20s (say)	d1 20

Data Processing

ft
apk
apks
halt
efp
abs

Next Experiment on Same Sample

Increment the experiment number	iexpno
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When Finished

Turn spin offro offEject sampleej