Vanderbilt NMR Facilities
Instructions for Setup of 1D Experiments Using TOPSPIN 3.x

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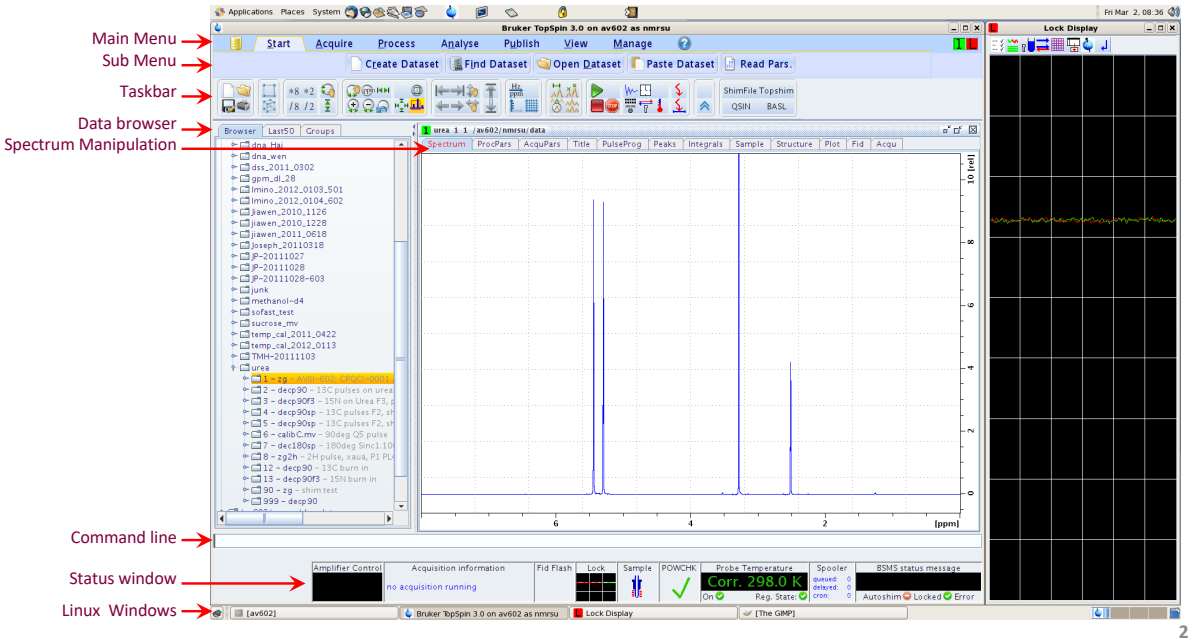
June 2018

Acquisition - Step by Step TS 3.x

General

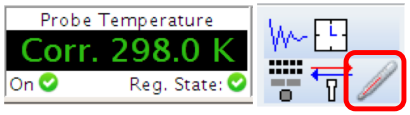
A more complete menu structure was implemented in Topspin 3.x that guides you through the setups.



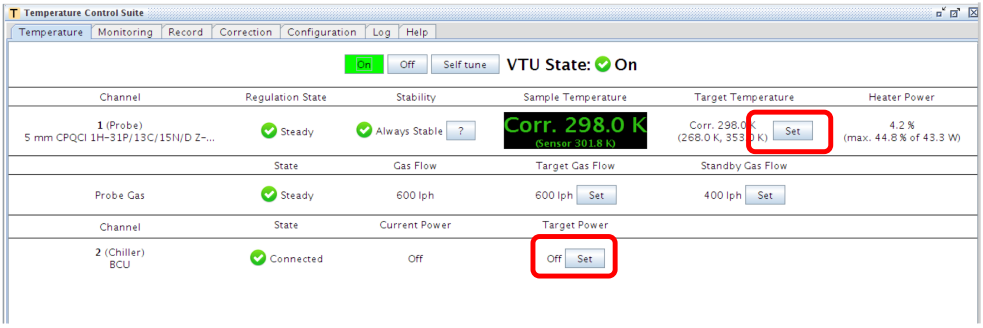
Acquisition - Step by Step TS 3.x

1. Set / check Temperature

- ***edte,*** double click on window



- For low temperature (<298K), turn on the BCU chiller, when done, please turn off again
- Turn chiller off when your experiment is done!!



2. Insert Sample

- ***Make sure you clean the tube and adjust the height in the spinner properly!***
- *Hit the lift button on either the BSMS-keyboard or BSMS-software tool*
- *Insert sample*
- *Turn lift off*

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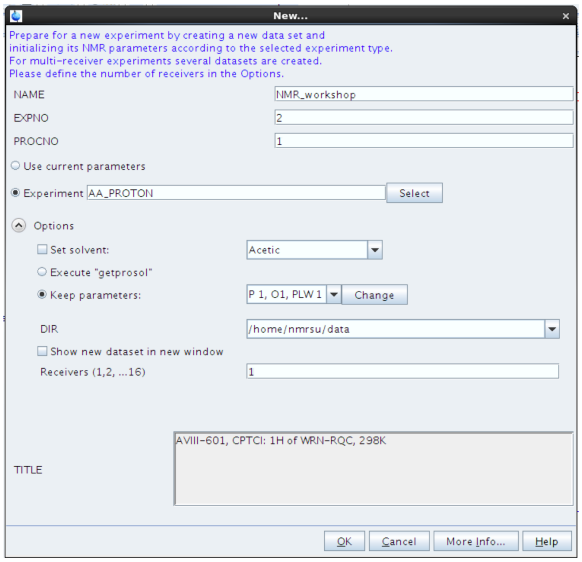
Acquisition - Step by Step TS 3.x

Basic (1D) NMR

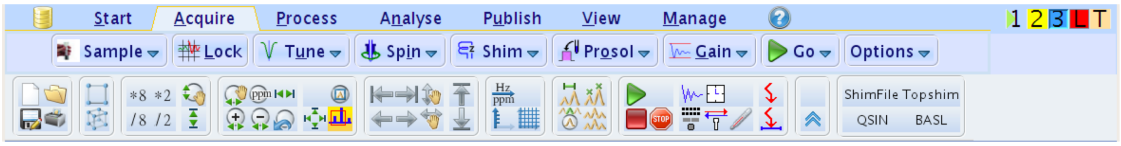
3. Create new data set:

1. ***edc***
2. *iexpno*
3. Set title: use Title tab (the title is only saved if you change the tab after setting!)

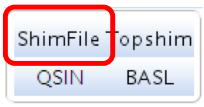
Selecting “Use current parameters” gives you a **copy of ALL current parameters** in the new data set, while under “Experiment” you read in a **NEW** standard parameter set!



Acquisition - Step by Step TS 3.x



4. Read-in standard shim file — ***rsh chloshim.600***



5. Lock sample — *lockdisp,* ***lock <solvent>***

6. Read in parameters (opt) — ***rpar*** *(example: AA_proton.mv)*

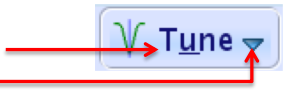
This step is only needed, if no proper parameter set was selected in EDC (step 1)

7. Make proper routing (opt) — ***edasp****, edsp (on 800 after field change)*

Make sure all necessary nuclei are activated, click "***Default***" followed by "***Save and Close***".

8. Tune

- ***atma*** (automatic tuning, click center of tune button or type command)
- ***atmm*** (manual interaction with ATM)
- *w1, w2, w3* (shortcut), *wobb f3, f2, f1* on older probes



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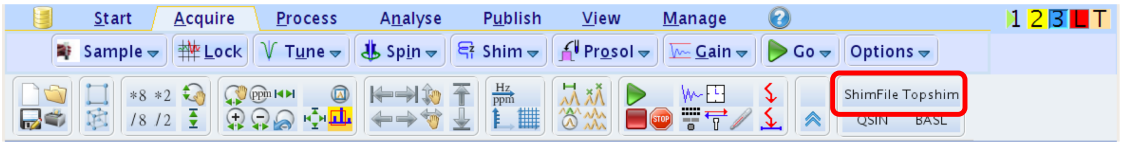
Basic (1D) NMR

Acquisition - Step by Step TS 3.x

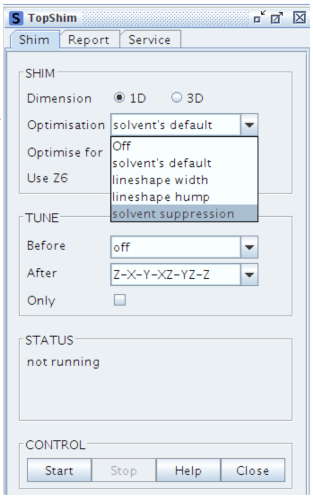


9. Shim

1. if not done in step 4, read in standard shimfile "***ShimFile***"
2. Click on "***Topshim***" to open the Topshim gui.

In Shim Tab:

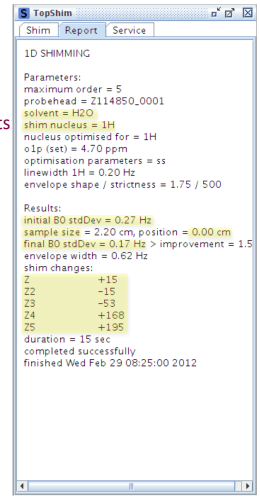
1. Select Dimension (1D for deuterated or H_2O solvents, 3D for H_2O only)
2. Optimization: either solvent's default or solvent suppression (or H_2O)
3. TUNE After: only in 1D mode, will touch up lock phase and off-axis shims
4. **Start**



Report Tab:

Here you find the shim results

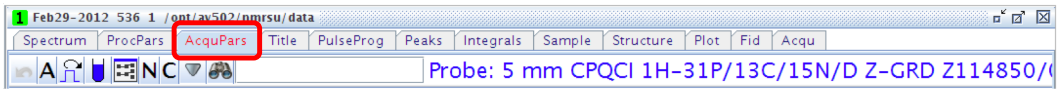
- Solvent shimmed
- Nucleus shimmed
- Initial B0
- Sample position (0.0 cm)
- Final B0 (~0.2)
- Shims changes



10. Check Parameters

Facility approved, e.g. parameter sets starting with AA…, or Bruker parameter contain good starting parameters and only the “test tube” icon or getprosol command (see next page) will be required for proper pulses.

To optimize any other acquisition parameter, choose either the full parameter list (EDA) or experiment specific parameter list (ASED) in the AcquPars tab.



eda vs *ased*

- In any case click on the test tube icon to assure proper pulses – or
- Type ***pulscal*** to determine the actual pulse values

- If you have determined the proton 90 degree pulse execute the *getprosol 1H <p1> < pldb1>* command to adjust all proton and X-pulses!

ASED List:
Most important parameters for 1D acquisition pointed out

Probehead Information

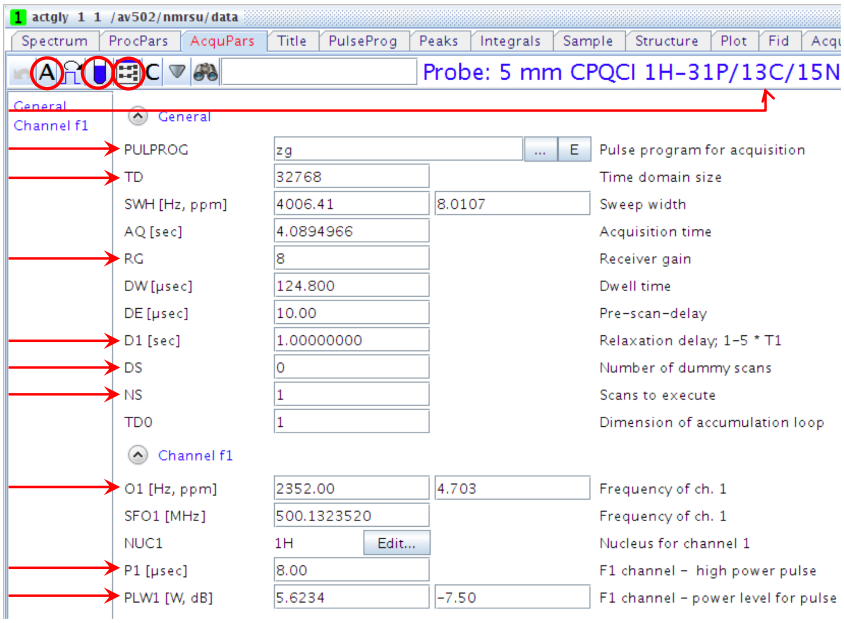
Experiment, see *PulseProg* tab
Number of points in FID ***TD***

Receiver Gain ***RG***

Relaxation delay ***D1***
(time between scans)
Dummy scans ***DS***
Number of scans ***NS***

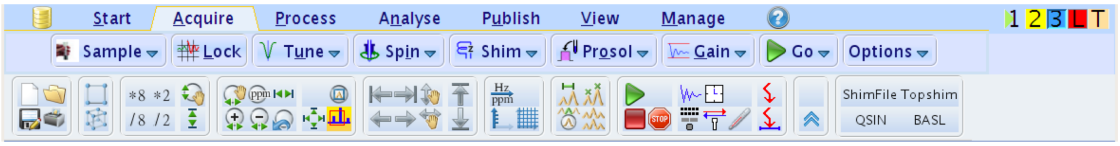
Transmitter offset ***O1P***
(center of spectrum in ppm)

Pulse length ***P1***
Pulse power ***PLdB1***



Each single parameter in ased is important and needs to be verified!!

Acquisition - Step by Step TS 3.x

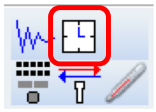


11. Receiver gain adjustment

- ***rga*** (automatic, only good for ^{1}H)

10. Experiment time (opt)

- ***expt*** (if too long, adjust NS or TD)



11. Start Acquisition with *ZG*

- Watch Lock
- Watch temperature
- Watch first fid
- Watch insert temperature on cryoprobe!!

If any of those indicators is unstable, stop immediately (*stop*)

Acquisition

Summary: Black Box 1D ^{1}H Acquisition Approach

1) Set Temperature (***edte***)
2) Lift on, insert sample (cleaned and height adjusted), lift off
3) Create new experiment with appropriate parameters (**edc**)
4) Read standard shim file (***rsh chloshim.600***, click on "**ShimFile**" button)
5) Lock sample (***lock <solvent>***)
6) Tune probe for all channels used (***atma*** *or manual with* ***atmm***)
7) Shim sample by clicking on "**Topshim**" button
8) In *AcquPars* tab hit test-tube icon to read in standard pulse length and power
   - If you know proper 1H pulse, use "***getprosol 1H <p1> <pldb1>***
9) Double check acquisition parameters
10) Adjust receiver gain on ^{1}H spectra only **rga, rgacryo** (all other typically: rg=1k, maximum)
11) Start experiment with **zg**
12) Make sure lock remains stable, otherwise **stop** immediately and check parameters
13) For long experiments, use **tr** to transfer data to computer for processing
14) Basic processing: ***efp, apk, abs***

Acquisition – Most Important Commands

Summary of Acquisition Parameters Requiring Optimization :

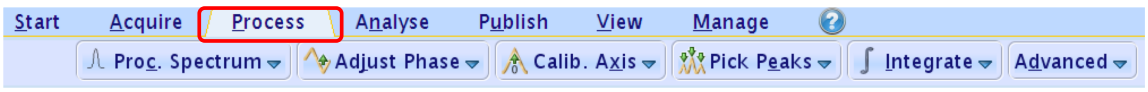
rpar:	read in parameter set
getprosol:	read in parameters from probe table (example: getprosol 1H 10.0 5.0)
topshim:	start gradient shim routine
edte:	Temperature unit
lock:	lock sample
atma, atmm:	Automatic tune probe
p1…31:	Pulse length [µsec] !! (determine, from standard parameter set, www page)
pldb1…31:	Pulse power [dB] !! (attenuation, the bigger the value, the less power !) (*gpro*)
o1, o1p:	Transmitter frequency (center of spectrum)
sw, swh:	spectrum coverage
td:	number of points in FID, typically 8k, 16, 32k for a 1D spectrum
aq:	acquisition time [sec] : AQ = TD / (2 * SW)
d1…31:	Relaxation delay (recycle delay, repetition rate) [sec]:
ns:	Number of scans: $S/N = NS^{1/2}$
ds:	scans without data acquisition to equilibrate system
rg:	Receiver gain (*rga* for automatic adjustment)

Basic Steps for Processing a 1D Spectrum

What are the processing steps:

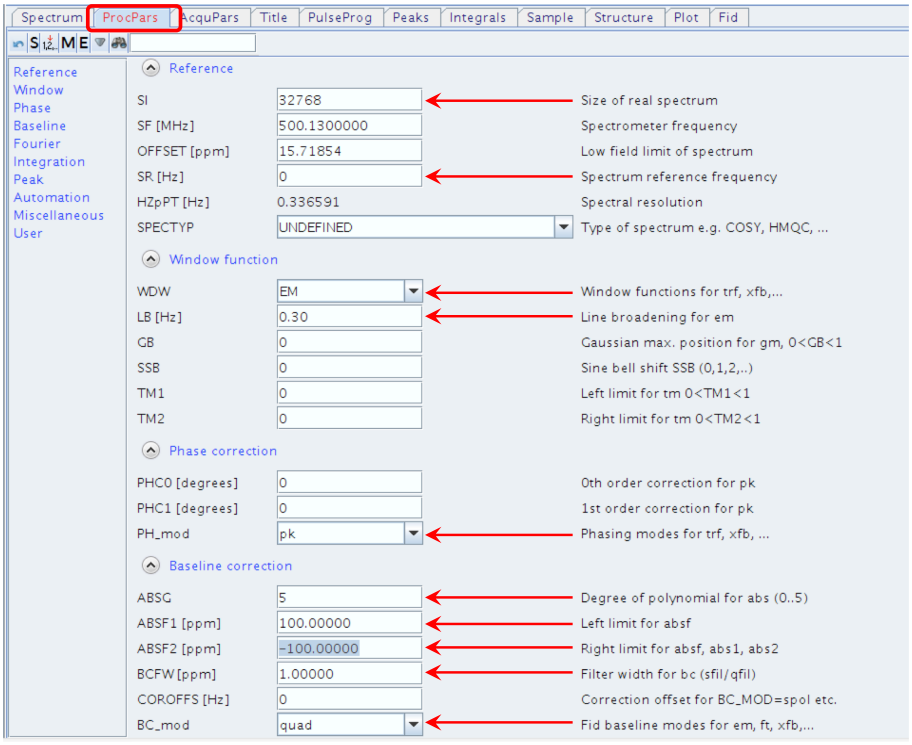
1. List all Processing Parameters (edp)
2. Set most common parameters in edp
3. Fourier transformation (t --> ν)
4. Phasing
5. Referencing
6. Baseline correction
7. Peak picking
8. Integration
9. Plotting spectrum
10. Compare spectrum (dual display)

Topspin 3.x menu bar for Processing



Basic Steps for Processing a 1D Spectrum

1. *List all Processing Parameters: edp*



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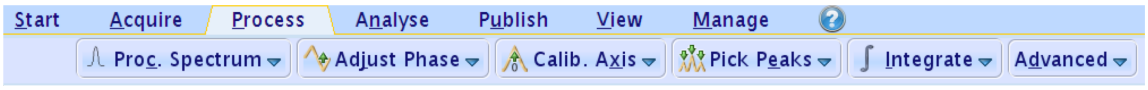
Basic Steps for Processing a 1D Spectrum

Basic (1D) NMR



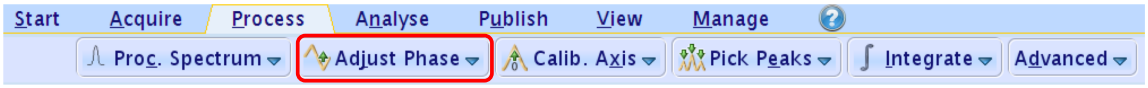
2. *Set most common parameters in edp (ProcPars tab)*

a. The number of points in a spectrum is defined by the parameter ***SI***, typical values: SI = 2 x TD (16k or 32k points)
b. Spectrum reference (***SR***), typically =0 (approximate reference)
c. Window function:
   Typical values:
   a. ^{1}H: ***wdw*** = EM, ***lb***=0.3
   b. ^{13}C: ***wdw*** = EM, ***lb***=2.0
d. Baseline correction values
   a. ***absg*** is the polynomal order to use for baseline correction (typical 5)
   b. ***absf1*** (dowfield limit for baseline correction)
   c. ***absf2*** (upfield limit for baseline correction)
   d. ***bc_mod*** for baseline correction on FID (typical quad)

3. *Fourier Transformation:*

ft: simple fourier transformation
fp: combination of ft and phase correction
*ef, **efp**:* apply exponential window function, followed by ft, phase correction
gf, gfp: apply gaussian window function, followed by ft, phase correction

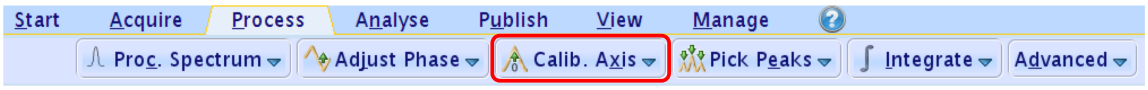
Basic Steps for Processing a 1D Spectrum



4. Phase Correction:

1. Place red pivot line on peak to left or right of spectrum *(right-click followed by "Set Pivot Point" selection*
2. Phase that peak using the 0-order correction 

3. (initial inversion of the spectrum by using the 180 tab, or adjust with 90, -90)
4. Phase the other side of the spectrum using the 1st order correction
5. Save and return from phase module
6. Alternatively, use ***apk*** for auto phase correction (works best if a lot of baseline is around the signals

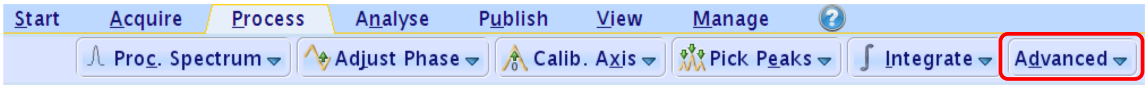


5. Referencing Spectra :

sr, sref: If set to zero and lock command was used, choose "Calib. Axis"

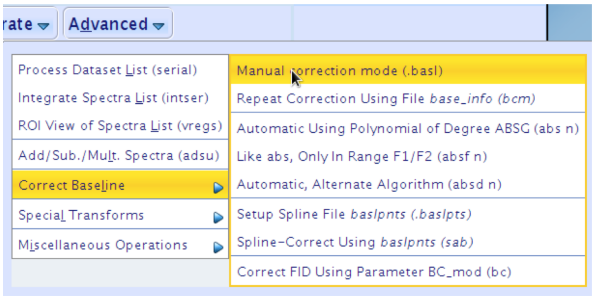
a. Move cursor to top of peak to be calibrated
b. Click left mouse button, enter the reference value followed by return.

Basic Steps for Processing a 1D Spectrum



6. Baseline correction:

There are many options under the "Advanced" tab
Alternatively type the following commands:

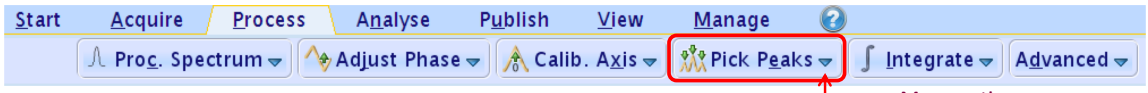


absn: automatic baseline correction, uses the following parameters in edp:
absf1: high field ppm limit
absf2: low field ppm limit
absg: degree of the polynomial (0 … 5)

bas: fit correction line manually to the spectrum from left to right with increasing order of the polynomial A, B, C, D, E

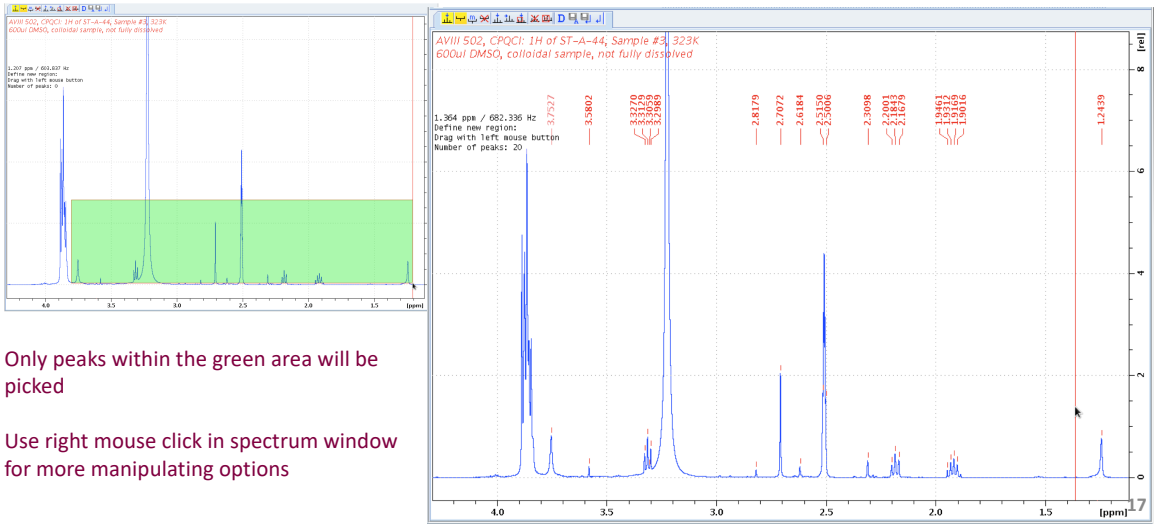
save and return from baseline module

Basic Steps for Processing a 1D Spectrum



7. Peak Picking (main menu):

- select area in which peaks should be picked, **deselect to expand or zoom on spectrum**!
- delete all picked peaks
- save and return from peak picking module



Only peaks within the green area will be picked

Use right mouse click in spectrum window for more manipulating options

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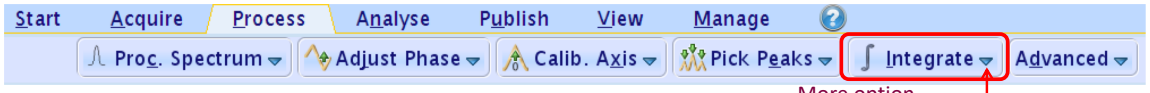
Basic (1D) NMR

Basic Steps for Processing a 1D Spectrum



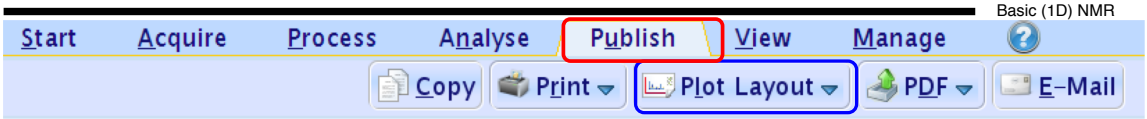
8. Integrate

abs: this command also defines integral regions automatically

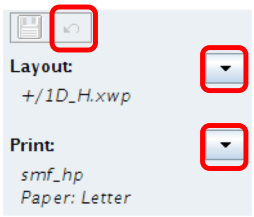
Integral tab: shows the integral list analog to peak list.

- select area in which integrals should be picked, **deselect to expand or zoom on spectrum**!
- cut integral at point selected with cursor following a left click.
- delete selected integral (select integral hovering over respective area in spectrum and right click)
- delete all integrals
- save and return from integral module

Basic Steps for Processing a 1D Spectrum



9. Plot spectrum: *Plot Editor: WYSIWYG (type **plot**, old version **plot0**)*



See full Tutorial on NMR Web site under the "User Info → Tutorials" tab

Layout: select any layout available (example: H1.A.xwp, 1D_H+pp.xwp, …)
Print: Page setup, Size "letter", "Landscape" → OK

As you click on individual components of the spectrum (e.g. spectrum, parameter, title window), the menu on the left will change so you can adjust the parameters appropriate for that window. Click on ***return arrow*** on top left to go back to main menu.

Cursor snap in setting: Layout → Properties → Cursor snap-in setting

Display more then one spectrum for print

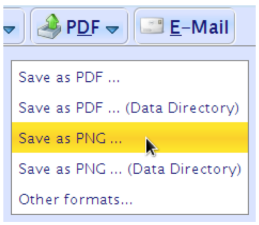
1. Drag all the spectra you want to plot (procno) from the browser to the Plot Portfolio window
2. Open Layouts for 1-3 spectra (1D_H+pp.xwp, 1D+1D+pp.xwp, 1D+1D+1D.xwp)
   Or
3. Right click on existing spectrum and duplicate spectrum (keeps only one title and parameter set)
4. To change the content of any window (spectrum, parameter, title, …), select the desired spectrum and drag it over the existing window. This will change the content of the window to the selected one.

Basic Steps for Processing a 1D Spectrum

9. Plot spectrum *(contd.)*

Print: Same as before, but select "***Print…***"
set proper printer options and ***Print***

Output as file:
PDF tab - click on main tab to save spectrum as pdf
- click on down arrow in PDF tab to select other file formats like png, jpg, bmp, tiff, …
- select target directory

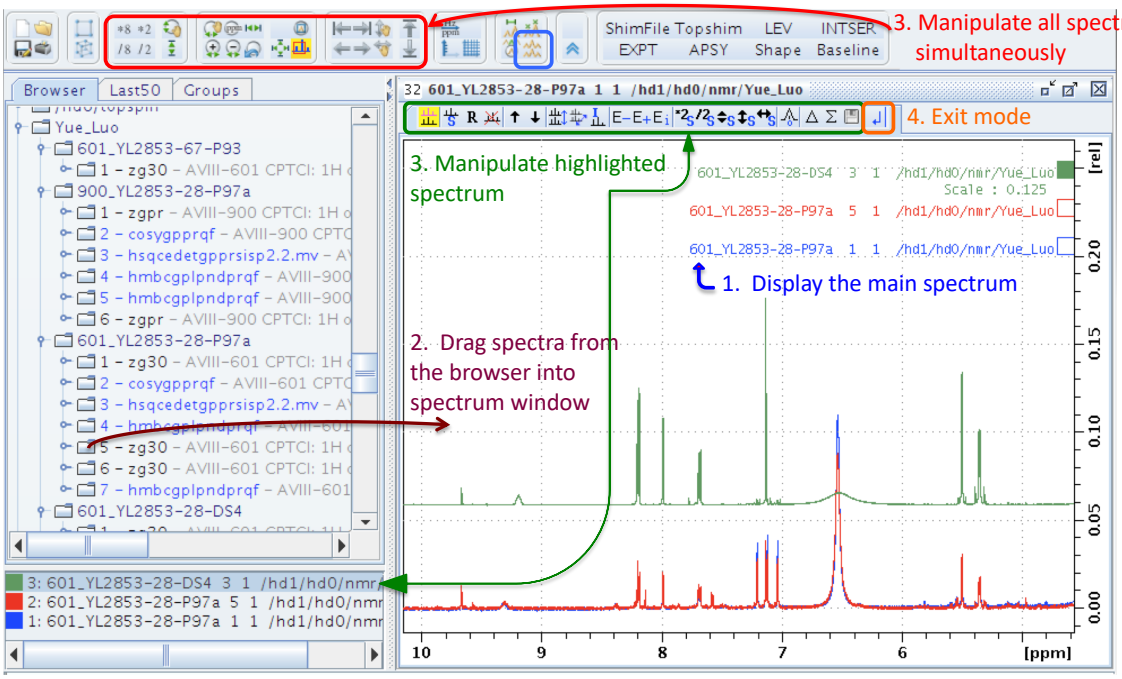


Return to spectrum:

Select spectrum tab in menu bar of current window

Basic Steps for Processing a 1D Spectrum

10. Dual Display, overlay spectra (can' t be printed from here)



Same applies to 2D spectra overlay