

Basic Operating Procedures for 1D Spectrum

Italic indicates command you type on the command line
[] and { } indicate a mouse click selection on top and bottom icons

1. LOGON

Log on to the Sun station using your “**Login ID**” and “**password**” (you have to be authorized to use the instrument).

2. EXCHANGE SAMPLE (see R1 for SAMPLE PREPARATION).

□ Click on the Spectrum icon at the bottom of the screen on the CDE toolbar, or right click the background and select VNMR.

□ Click [Acqi], it will bring the ACQUISITION window up, then click [LOCK]; click spin: [off], lock: [off], SAMPLE: [eject]; then:

a). REMOVE LOCK SAMPLE (or PREVIOUS SAMPLE)

There is ALWAYS a sample (sealed NMR sample tube) in the magnet with the lock ON at a level of 50%-70%. The purpose of using a lock solvent is to minimize magnetic field drift.

Remove the sample from the top of the magnet by holding onto the top of the spinner. Then remove the previous sample from the spinner with a firm pull (twisting the sample relative to the spinner also helps) on the top end and place it at NMR samples holder.

b). INSERT THE SAMPLE INTO MAGNET

Push the sample tube through the spinner using a twisting motion to avoid breaking the sample tube. The bottom of the sample should be inserted into the wide end of the spinner first. Adjust the spinner's position *using the gauge*. Wipe the outside of the tube and the spinner with a soft tissue. When moving the sample/spinner assembly, always hold the top of the spinner to avoid repositioning of the spinner. Place the sample tube and spinner into the transfer tube opening in the center of the magnet. Make sure that there is enough air to support the sample *before* letting go of the sample tube and spinner. Then:

□ Click SAMPLE: [insert], wait until you hear two click sounds, then click spin: [on] (spin rate is set to 20)

3. ESTABLISHING LOCK MANUALLY

The lock signal appears as a sine wave if the lock signal is off-resonance, and as a dc signal if the lock signal is on-resonance.

□ Increase the **lockpower** to 30 and the **lockgain** to 30 or higher until you see sine wave signal, then adjust **Z0** to get a dc signal (clicking the left mouse button while the cursor is on an [-N+] button decreases the value by N; clicking the right mouse button while the cursor is on an [-N+] button increases the value by N), when you see a dc signal, click lock: [on] to turn on field-frequency regulation

□ Adjust the lock phase so that the dc signal is highest at the above setting of the **lockpower** and **lockgain**.

□ Adjust the **lock level** to 60-70% by decreasing **lockpower** and **lockgain**.

4. SHIMMING MANUALLY

□ Click [SHIM], Adjust the **Z1C** and **Z2C** (start from -4+ to -1+), then **Z1** and **Z2** (start from -64+ to -16+) to maximize the lock level.

□ Click [LOCK], adjust **lockpower** and **lockgain** to leave the **lock level** at 60-70%, then click [CLOSE].

5. SET-UP EXPERIMENT

□ Click {Setup exp}, then click on solvent:□ and hold down the left mouse button to select the solvent you are using, click on Experiment selection at Basic 1D experiment: □ and hold down the left mouse button to select Proton1D.

□ Click {Flags cond.}, select auto lock no and auto shim no.

6. COLLECT SPECTRUM

□ Type *dg* ↵ (display the parameters you are using); type *nt=XX* ↵ (nt is the number of transients or scans, default to 16 for proton), then type *ga* ↵ to collect the spectrum.

7. DATA PROCESSING (see R2 and R3 for alternative commands)

□ Type *aph* ↵ (automatically phase the spectrum); type *vsadj* ↵ (adjust the vertical scale vs, you can click [Phase] to manually adjust the phasing); type *ds* ↵ (display the spectrum again and get you out of phasing mode).

To reference the spectrum: Locate the solvent resonance or some other reference line.

Clicking with the left mouse button on one side of the peak and with the right mouse button on the other side of the peak will allow you to then click [Expand] to expand this region of interest. Place the cursor on top of this resonance and type *nl* ↵, then type *rl(7.27p)* ↵ (for chloroform) or whatever ppm shift is appropriate for that resonance. Click [Dscale] (display the entire scale). Type *f full* ↵ (display the full spectrum).

To integrate the spectrum: Click [Part Integral] to display the integral line; type *cz* ↵ (clear all resets (zeroes) already in memory). Click [Lvl/Tlt] to make the integral line level. Use the left mouse button and click and hold on the left-hand side of the spectrum, adjusting the level up or down, then move to the center of the spectrum and click with the left mouse button and adjust the tilt up or down. Click [Resets] and use the mouse button to click once for each reset. The clicks toggle the integral trace on and off. The parameter is the integral scale *vs* controls the vertical scale of the spectrum, or you can use the middle mouse button (roll up or down to change the vertical scale).

To peak picking: Click [Th] (the peak picking threshold) and adjust the threshold with the left mouse button to move the horizontal line.

Type *dpir* ↵ to display the peak integral regions and type *dppf* ↵ to display the peak frequencies

To add text to the spectrum: Type *text('blah blah blah&')* ↵ to put in whatever text you want to have displayed with your spectrum.

To plot the spectrum: Type *vp=12* ↵, type *pl pscale pir ppf pap page* ↵

pl = plot spectrum

pscale = plot scale

pir = plot integrals

ppf = plot peak frequencies

pap = plot parameters

page = eject paper

vp must equal 12 to plot the integral ratios

8. SAVE SPECTRUM (see R4 for LOADING SPECTRUM)

Click [Main menu], then [DATA], select your directory by marking on it, then click [Set Directory], then type *svf* ↵; type *file name* ↵

9. PUT STANDARD SAMPLE BACK

- Click [**Acqi**] (type **acqi** if the button is not present); click [**LOCK**], then Spin: [**off**]; Lock: [**off**]; SAMPLE: [**eject**], then take your sample out and insert the sealed sample into spinner, place the sample on top of the magnet bore tube, then click [**insert**].
- Repeat **Step 3** to lock the sealed sample.

10. LOGOFF

- Click [**Main menu**], [**More**] and [**Exit VNMR**]
- Click on **EXIT** on the CDE toolbar and confirm the logout. Please write a note if there are any problems you may have had with the instrument.

Refer to the following for detail

R1. SAMPLE PREPARATION FOR ¹H NMR

- Use about 3-10 mg of sample dissolved in ~0.7 mL of deuterated solvent. Filter the solution if there are any solid particles present.
- The recommended amount of TMS added to the deuterated solvent is 0.1% (v/v). If too much TMS is added to the sample, spurious peaks may appear in the spectra.

R2. DATA PROCESSING

Click [**Autoprocess**] or type command **wft**↵

The following are a few possible commands to further process the spectrum if needed:

Display the scale: Click [**Dscale**] or type **dscale**↵, if the scale is in hertz but you need ppm, you can type **axis='p'**↵

View the full spectrum in the full screen: Type **f full**↵

Adjust the vertical scale: Use the middle mouse button directly on the spectrum or type **vs=number**↵ wanted

Zoom in on the region within the cursors: Left-click on the left and right-click on the right of a region in the spectrum then click [**Expand**]

Change the limits of the spectrum to specific values: For example 1.0ppm and 6.0ppm, type **sp=1.0p wp=5.0p**↵

Baseline correction: Type **dc bc**↵ (after integration)

Reference a peak: For example, set acetone to 2.05ppm (in organic solvent) or 2.225ppm (in D₂O), click as near as possible to the center of the acetone peak, type **nl**↵ then **rl(2.225p)**↵

Integrate: Click on [**Part integral**], and type **cz**↵ then click [**Resets**], and use the left mouse to cut at the beginning and the end of the peaks.

– Type **vp=12**↵ to move the spectrum allowing to write the value of the integrals.

– Click on middle button to adjust increase/decrease the scale of the integrals.

– Type **dpir**↵ to display the values of integrals.

Set the value of an integral: Type **ds**↵, move cursor on the peak, click [**Set Int**], then type **1** or **2** or **3** (which you think there's one proton or two or three)

– Type **ds**↵, click [**Th**] and adjusting the threshold with the left mouse button to move the horizontal line.

– Type **dpf**↵ displays the peak frequencies.

R3. PLOT DATA

Click [**Main Menu**], [**Autoplot**]

Or click [**Main Menu**]; [**Display**]; [**Plot**]; [**Plot**]; [**Scale**]; (more options can be selected by clicking on them), then click [**Page**] (Page is the command that sends the information to the plotter. Every option you click before page will be included in the plot).

R4. LOADING SPECTRUM

Click [Main menu], then [DATA], select your directory by marking on it, then click [Set Directory], selecting the fid file which you want to load by marking on it, then click [Return], [Load], type **wft**↵.

☺ Gradient Shimming (only for INOVA-400)

1. Click {Setup exp}, then click on solvent:□ and hold down the left mouse button to select the solvent you are using.
2. Click on Lock: |find z0|, wait until rmdir: /home/.../findz0tmp appear, then type **lock**↵, wait until it shows Idle in the status Icon box, then adjust lock level around 70-80%.
2. Type **jexp2**↵
3. Access the Gradient Shimming System menu
 - Type **gmapsys**↵
 - Click [Autoshim on z]
4. If the shim does not work, find the transmitter offset frequency before autoshimming
 - Type **gmapsys**↵
 - Click [Set Params], [find tof]
 - Repeat **Step 3**
 - Type **jexpN**↵ Join the experiment you will be working in and run your spectra (N is work space number, type **jexp1** if it is 1) .



Retrieve standard shim set if you have problems on locking or shimming (follow the instructions which are posted on the wall).