

Varian NMR Training

Login into NMR facility computers

If your NMR account has been created use your university NetID and Password to login into any NMR facility computer, otherwise contact NMR facility personnel.

Measurement of 1D 1H spectra

Type the command:

```
setexp_H1_solvent
```

Some examples:

```
setexp_H1_cdcl3, setexp_H1_d2o, setexp_H1_cd3od, setexp_H1_dmsol, setexp_H1_acetonitrile,  
setexp_H1_acetone
```

To display the current experiment parameters, type:

```
dg
```

1. Open the acquisition window by clicking on the ACQI button (the last button in the first row).
2. Open the LOCK window.
3. Turn off spinning and lock.
4. Eject the standard sample
5. Remove the standard sample from the spinner and replace it with your sample.
6. Adjust sample height in the spinner using the gauge attached to the console door.
7. Place spinner with your sample on the top of the magnet. Before you do so make sure the air is flowing.
8. Press the insert button to insert your sample. Wait until the sample reaches its correct position inside the magnet.
9. Turn on spinning and lock.
10. Open the SHIM window.
11. Adjust Z1C, Z2C, Z1, Z2, and if necessary Z3 shims by maximizing the lock signal level. Current shim values shown in the square brackets next to the shim labels can be changed in steps of 1, 4, 16, and 64. Clicking the left button will decrease the current shim value. Clicking the right mouse button will increase the current shim value. See the section "**Shimming on the Lock Signal**" for a detailed description of the shimming procedure.
12. Close the acquisition window when shim adjustment is finished.
13. Set number of transients (parameter nt) according to the concentration of your sample, for example:

```
nt=16
```

The data acquisition can be started using the commands

```
go - (Acquire FID only)
```

```
ga - (Acquire FID and do FT)
```

```
au - (Automatic, acquire, process, print)
```

Homo Decoupling

1. Place a cursor in the middle of the multiplet you want to irradiate. The vertical red cursor will move in the position of the arrow cursor. Type the command:

```
sd
```

2. Type:

```
dm='yyy' homo='y' dpwr=20
```

3. Type the command:

```
ga
```

Measurement of both homodecoupled and nondecoupled ¹H spectra in one experiment

1. Set the values of parameters dof, homo, and dpwr as described in the preceding section "Homo Decoupling".

2. Type:

```
dm='nnn','yyy' gain='y'
```

3. Set number of transients (parameter nt) according to the concentration of your sample, for example,

```
nt=16
```

4. Start the data acquisition using commands:

```
ga ai
```

Measurement of 1D $^{13}\text{C}\{^1\text{H}\}$ spectra

The following set up procedure assumes that you intent to measure $^{13}\text{C}\{^1\text{H}\}$ spectrum right after ^1H spectrum was measured. There is no need to change lock and shim values, because locking and shimming was done before as a part of the measurement of ^1H spectrum.

1. Open the **Main Menu**→**Setup**→**Nuc,Solv**.
2. Select C13 and your solvent. The necessary acquisition, processing and plotting parameters will be loaded.
3. Set the number of transients (parameter nt) according to the concentration of your sample, for example,

```
nt=64
```

4. The data acquisition can be started using command:

```
ga
```

- The following set up procedure assumes that ^1H spectrum was not measured right before measurement of $^{13}\text{C}\{^1\text{H}\}$ spectrum.

1. Type command:

```
setexp_C13_solvent
```

```
setexp_C13_cdcl3, setexp_C13_d2o, setexp_C13_cd3od, setexp_C13_dmsd, setexp_C13_cd3cn,  
setexp_C13_acetone, etc
```

Follow the steps 2-13 in “Measurement of 1D ^1H spectra”.

Measurement of APT spectra

1. Set parameters to measure $^{13}\text{C}\{^1\text{H}\}$ spectrum as described in the preceding paragraph "Measurement of 1D $^{13}\text{C}\{^1\text{H}\}$ spectra".
2. Open the **Main Menu**→**Setup**→**Sequence**→**APT**

Alternatively, type command

```
apt
```

3. Set the number of transients (parameter nt) according to the concentration of your sample, for example:

```
nt=32
```

4. To start the experiment, type command:

```
ga
```

The data acquisition can also be started using commands:

```
go
```

```
au
```

Measurement of DEPT spectra

The measurement of DEPT spectrum requires you acquire a regular $^{13}\text{C}\{^1\text{H}\}$ spectrum first. It is adequate to measure this spectrum with only one transient (nt=1). You do not need to see any carbon signals corresponding to the solute molecules. Detecting just the solvent signal is satisfactory. After you measured a regular $^{13}\text{C}\{^1\text{H}\}$ spectrum do the following.

1. **Main Menu**→**Setup**→**Sequence**→**DEPT**

Alternatively, type command

```
dept
```

2. Adjust number of transients (parameter nt) corresponding to the concentration of your sample. The number of transients must be multiple of 8, e.g., nt=8, 16, 24, etc.

```
nt=32
```

3. To start the experiment, type command:

```
au
```

Shimming on the Lock Signal

1. In the SHIM display window, try a change of +4 or -4 in the setting for Z1C. If the lock level goes up with one of these, continue in that direction until the level is maximized (It no longer increases, but instead begins to fall.). If the lock level drops when using +4 or -4 step try a change of +1 or -1.
2. Change the setting for Z2C by +4 or -4 and continue in that direction until the level is maximized. Try a change of +1 or -1, if the step ± 4 seems to be excessive.
3. Readjust Z1C for maximized lock level. Change the Z1C in steps of 1.
4. Readjust Z2C for maximized lock level. Change the Z2C in steps of 1.
5. Repeat steps 3 and 4 until the lock level goes no higher.
6. Adjust Z1 and Z2 shims (the fine Z shim controls) in the same way as Z1C and Z2C until the lock level is maximized. Use the step +4 or -4. You also may try a change +16 or -16.
7. After Z1 and Z2 have been adjusted for maximum lock signal, write down the lock level, adjust Z3 in one direction, say by +4, and then iteratively re-optimize Z1 and Z2 until lock signal is at its maximum. Note this level of the lock signal. If the lock signal is higher than it was before (when you first wrote it down), continue changing Z3 in the same direction. If the lock signal is lower than it was before, Z3 must be changed in the opposite direction. Every change in Z3 must be followed by optimization of Z1 and Z2 until the lock level is at a maximum.
8. If the lock level increases to 100, stop shimming, select z0/pwr/gn/ph from the SHIM menu (Put arrow cursor on the axial z button and press the right mouse button.) and decrease lock power. The reduced lock level should be ~ 60 . Return to shimming by selecting axial z from the same SHIM window.

Archiving and Retrieving NMR Data

1. To store the acquired data in user afs space type command:

```
connect_afs
```

2. Type command:

```
svf('name_of_file_to_be_stored')
```

To reload NMR data stored in user afs space into any NMR facility Sun computer type command:

```
connect_afs
```

It is important to disconnect from the afs drive before you logout, otherwise your account can become corrupted.

To disconnect type command:

```
cd
```

Processing

Data acquired with the command go (FID signal) must be processed using the command ft or wft to obtain a spectrum. To convert (Fourier transform) the FID signal into a spectrum, type the command:

```
ft or wft
```

If the command wft is used, the FID signal is multiplied by a weighting function, which corresponding processing parameter has non zero value, prior Fourier transformation. The command ft performs only the Fourier transformation and skips the application of weighting function. To enhance signal to noise (S/N) ratio in 1D proton spectra, set the value of parameter lb in the range of 0.1 to 0.5, by typing:

```
lb=x
```

where $0.1 \leq x \leq 0.5$, and pressing the Return button on the keyboard. For resolution enhancement of 1D proton spectra, set the lb value in the range of -0.5 to -0.1, by typing:

```
lb=x
```

where $-0.5 \leq x \leq -0.1$, and pressing the Return button on the keyboard. To enhance S/N ratio in 1D carbon or other heteronuclei spectra, type:

```
lb=1
```

To remove a particular weighting function from use, the value of its corresponding parameter should be set to 'n', for example type:

```
lb='n'
```

If the command ga is used for data acquisition, the FID signal is converted into a spectrum by performing Fourier transformation automatically, immediately after the acquisition is completed. Provided some processing parameters have non zero values, the FID signal is also automatically multiplied by corresponding weighting functions prior Fourier transformation.

Phasing spectra

Automatic phasing

1. Type the command:

```
aph0
```

Manual phasing

1. Consecutively click the buttons Main Menu, Display, Interactive and Phase using the left mouse button.
2. Put the arrow cursor above the far right peak of displayed spectra approximately to the middle of the display window in vertical direction.
3. Press and hold down the left mouse button and slide the mouse on the mouse pad until the right part of spectra is phased correctly.
4. Release the left mouse button and move the arrow cursor above the far left peak of displayed spectra approximately to the middle of display window in vertical direction.
5. Press and hold down the left mouse button and slide the mouse on the mouse pad until the left part of spectra is phased correctly.
6. Release the left mouse button and click the Phase button using the left mouse button to exit the phasing procedure.

Referencing spectra

1. Open the **Main Menu**→**Display**→**Interactive**→**Cursor**(the 1st button in the second row)
2. A red vertical cursor will appear in the display window. If the first button in the second row has the label Box, double click on this button.
3. Put the cursor on the top of the peak you want to use for referencing, usually TMS or solvent signal, and click the left mouse button. The red vertical cursor will move to the position of the arrow cursor. Type the command:

```
nl
```

4. Click the button Ref, type the correct chemical shift value for the chosen signal and press the Return button on the keyboard.

Displaying the Scale

1. To display a scale below the spectrum, type the command:

```
dscale
```

You can change the scale from PPM or Hz by typing:

```
axis='h'
```

```
axis='p'
```

Adjusting Zoom

To expand a spectral region which spans from the chemical shift value of x ppm (right margin) to the chemical shift value of y ppm (left margin), set

```
sp=xp (Start Point)
```

```
wp=Dp (Width)
```

```
sp=2.8p wp=0.6p
```

Adjusting vertical scales of the spectra

To adjust the heights of the peaks in 1D spectra, type the command:

```
vsadj(x)
```

Alternatively, the peak heights in 1D spectra can be adjusted by consecutively clicking **Main Menu**→**Display**→**Message**→**Adj VS**

The peak heights in 1D spectra can also be adjusted putting the arrow cursor above a selected peak and sliding the mouse on the mouse pad with the middle mouse button continually pressed.

Peak Picking

To set a threshold for display and printout peak frequencies, click the buttons

Main Menu→**Display**→**Interactive**→**Th**

A yellow horizontal cursor will be displayed. To adjust its vertical position, put the arrow cursor on the yellow horizontal line, press and hold down the left mouse button and slide the mouse on the pad.

You may display the peak picks by typing the command:

```
dpf
```

To plot the peak picks use the command:

```
ppf
```


Integration of Spectra

1. **Main Menu**→**Display**→**Interactive**→**Part Integral**

If the second button has the label Full Integral, click three times on this button. If the second button has the label No Integral, double click on this button. Previously defined integral regions will be displayed.

2. Type the command:

3. If the integral curve is tilted, click the button **Lvl/Tlt**

Put the arrow cursor on the right side of the integral curve about halfway vertically up the screen, press and hold down the left mouse button and slide the mouse on the mouse pad until the integral curve over the regions without peaks is parallel to the baseline. Release the left mouse button and click the Lvl/Tlt button to exit the tilt adjusting procedure.

To integrate spectral regions containing peaks

1. Click the button **Resets**.
2. Start with the far-left region. Put the arrow cursor on its left side right behind the last peak and click the left mouse button.
3. Place the arrow cursor on the right side of this region right in front of the first peak and click the left mouse button.
4. In the same way integrate all other spectral regions going successively from left to right.

Alternatively, the spectral regions containing peaks can be integrated by consecutively clicking the buttons **Main Menu**→**Display**→**Message**→**Adj IS**.

To assign the integral value to a chosen integral region

1. Place the red vertical cursor over this region and click the **Set Int** button.
2. In the command window enter the desired value and press the Return button on the keyboard.

The position of the integrals with respect to the spectrum is governed by the parameter *io*. Its values must be entered in units of millimeters. For example, typing:

The height of the displayed integrals can be adjusted by using the parameter *is*. Its values can be set in the range of 1 to 700. For example, type:

Plotting spectra

Plotting spectra by using commands

1. Type the commands:

```
pl pscale pap pir ppf page
```

and press the Return button on the keyboard.

- pl - plots spectrum
- pscale - plots scale under the spectrum
- pap - prints all parameters
- pir - prints integral values under the scale
- ppf - prints peak frequencies
- page - submits the current plotter file, which has been created by all previous plotter commands, and prints the actual page.

Plotting spectra by clicking buttons

Click consecutively the buttons

Main Menu→**Display**→**Plot**→**Plot**→**Scale**→**All Params**→**Peaks**→**Page**

If you also want to print the integral values you must type the command:

```
pir
```

The parameter **vp** positions the whole spectrum vertically on the screen and consequently on the paper when plotted. To move the spectrum up by 50 mm, type:

```
vp=50
```

Its values must be entered in units of millimeters.

To plot the scale at the bottom of the paper when the spectrum is moved up by *xy* millimeters (**vp=xy**). Use the command:

```
pscale(vp-xy)
```

Plotting two spectra stacked vertically

1. Display the first spectrum by typing the command

```
ds(1)
```

2. Adjust vertical scale and position of the first spectrum by typing the command:

```
vsadj(85)
```

```
vp=0
```

3. Type the commands:

```
pl pscale pap
```

4. Display the second spectrum by typing the command:

```
ds(2)
```

5. Type:

```
vp=85
```

6. Type the commands:

```
pl page
```