

Varian NMR Instructions - 1D Quick Reference

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Loading a Sample, Locking, and Shimming

Loading a Sample

- Select [**Change Sample**] on the top menu line. [**Eject**] the dummy sample and [**Insert**] your sample. Don't forget to use the sample gauge to center your sample in the magnet.
- **OR** Select [**Acqi**] on the top menu line. Select [**Lock**]. Turn spin [**off**]. [**Eject**] the dummy and [**Insert**] your sample. Turn spin [**on**].

Locking

- Choose [**Change Sample**] - [**Set Solvent**] and select the proper solvent for your sample.
- Choose [**Autolock**] which finds the **Z0** of your solvent and locks on it.
- Select [**Set Standard Shims**] to load the standard shims. Wait for the spectrometer to beep.
 - If the autolock does not find the lock, select [**Acqi**] - [**Lock**].
 - Turn spin [**on**] and Lock [**off**]
 - adjust **Z0** in steps of 1 or 4 (300MHz) or 16 or 64 (500MHz) until a sine wave appears and the wavelength gets longer.
 - Once approximately one wavelength is seen, turn Lock [**on**].
- In [**Acqi**] window:
- Reduce **Lock Power** and **Lock Gain** until the lock level is <100%.

Shimming

- Select [**Change Sample**] - [**Set Standard Shims**] to load the standard shims if you have not already done so. Wait for the spectrometer to beep.
- In [**Acqi**] window: select [**Shim**]
- Iteratively adjust **Z1C** and **Z2C** (coarse) in steps of 1 or 4 to maximize the lock signal (300MHz)
- Iteratively adjust **Z1** and **Z2** (fine) in steps of 4 or 16 to maximize the lock signal (500MHz).
- If the lock signal goes above 100%, reduce **Lock Gain** and/or **Lock Power**.
- For short samples, you may also need to adjust **Z3** and **Z4**.
- If necessary, adjust **Lock Phase** in steps of 1 to maximize the lock signal.
- Type **su** in the command line and the [**Acqi**] window will close automatically.

Acquiring a Proton Spectrum

Setup and Acquisition

- Select [**Main Menu**] - [**Setup**] - [**Nucleus, solvent**] - [**H1**] - [**solvent**].
- OR** if you used the [**Change Sample**] menu for locking, select [**Setup Exp**] - [**H1**]
- Change appropriate parameters (e.g., use **nt=1** to check shims)
- Type **ga** or select [**Acquire**] - [**Go,wft**].
- When spectrum is displayed, type **aph** to autophase it.
- Save data: [**Main Menu**] - [**File**] - [**Data**] - [**Save FID**] or simply type **svf** [return] in your data directory. Supply a filename when prompted. **ds** will redisplay your spectrum.

Acquiring a Carbon Spectrum

Setup and Acquisition

- Select [**Main Menu**] - [**Setup**] - [**Nucleus, solvent**] - [**C13**] - [**solvent**].
- OR** if you used the [**Change Sample**] menu for locking, select [**Setup Exp**] - [**C13**]
- Change appropriate parameters (e.g., to change the number of scans, type **nt=128**).
- Type **ga** or select [**Acquire**] - [**Go,wft**].
- After every 16 scans (if **bs=16**), you can update your spectrum by typing **wft**.
- When spectrum looks good enough, select [**Abort Acq**] to stop experiment or type **aa**.
- Save data: [**Main Menu**] - [**File**] - [**Data**] - [**Save FID**] or simply type **svf** in your data directory. Supply a filename when prompted.

Parameters

- Set **dm='nyn'** for proton-coupled carbon spectra.
- Set **dm='nny'** for decoupled carbon spectra without NOE enhancement.

Acquiring a DEPT Spectrum

Setup and Acquisition

- Setup a C13 spectrum first: **[Main Menu] - [Setup] - [Nucleus, solvent] - [C13] - [solvent]**
(or recall a carbon spectrum that you ran previously *on the same instrument.*)
- Type **dept**.
- Change appropriate parameters (e.g., to change the number of scans, type **nt=128**).
- Type **ga** or select **[Acquire] - [Go,wft]**.
- Type **ds(1)** to display the first spectrum.
- Phase all peaks positive and type **dssa**. Use **pl('all')** instead of **pl** to plot DEPT spectra.
- Save data: **[Main Menu] - [File] - [Data] - [Save FID]** or simply type **svf** in your data directory. Supply a filename when prompted.

Saving and Loading Data

Saving Data

- **[Main Menu] - [File] - [Data] - [Save FID]**. Or type **svf**. Then type a filename -
any alphanumeric string with no spaces and only "-" (dash) and "_" (underline).

Loading Data

- **[Main Menu] - [File] - [Data]**
- Highlight filename and select **[Load FID]**.
- Type **wft**.

Data Processing and Analysis

Fourier Transform

- Automatic processing is available by typing **process**.
- Alternatively, type **wft** or select **[Main Menu] - [Process] - [Weight, Transform]**.

Apodization (optional)

- Sensitivity Enhancement: **[Main Menu] - [Process] - [Select Params] - [Broaden]**.
Type **wft** to apply the apodization.
- Resolution Enhancement: **[Main Menu] - [Process] - [Select Params] - [Resolve]**.
Type **wft** to apply the apodization.

Interactive Display Mode (ds command)

- Start this mode by typing **ds** or by selecting **[Main Menu] - [Display] - [Interactive]**.
- Left and right mouse buttons control the red vertical cursors.
- Middle mouse button controls vertical scale of spectrum (**vs**) or integral (**is**), if displayed.
- Type **vsadj / isadj** to automatically adjust vertical scale of spectrum / integral.
- **[Cursor]/[Box]** buttons toggle between 1 and 2 vertical cursors.
- **[Expand]/[Full]** buttons allow zooming in and out on regions of the spectrum.

Phasing

- Type **aph** for automatic phasing.
- For manual phasing in the interactive display mode, select **[Phase]**.
- Click on a peak near the right edge of the spectrum and drag up and down with the left
(coarse) or right (fine) mouse button to phase this peak (adjust **rp**).
- Click on a peak near the left edge of the spectrum and drag up and down with the left or
right mouse buttons to phase this peak (adjust **lp**).
- Select **[Box]/[Cursor]** to get out of phase mode.

Referencing

- Find reference peak (TMS or solvent) and click on it to move the red vertical cursor to that position.
- Type **nl** to move to the top of the nearest line.
- Type **rl(7.24p)** if your solvent is CDCl₃. Or select **[Ref]** and give the ppm value when prompted.

Peak Picking

- Select [**Th**] button to display yellow horizontal line.
- Use left mouse button to drag yellow line to set threshold for peak picking.
- Select [**Th**] again to get out of threshold adjustment mode.
- Type **dpf**, **dpf('pos','top')** or **dll** to list peaks.

Integration

- Select [**Int**] to display integral trace (green line).
 - Select [**resets**] to start integral reset mode.
 - Click with the left mouse button on both sides of each peak of interest to cut integral regions.
 - If you make a mistake, click with the right mouse button to remove the nearest integral reset.
 - To start over, type **cz** to clear all reset points.
 - Select [**Box**]/[**Cursor**] to get out of integral reset mode.
- An alternative way to set integrals is to move the red cursor to each reset point and type **z <ret>**.
- Type **dpir** or **dpirn** to display integrals below spectrum. (**vp** must be =12).
 - (optional) Type **bc** for baseline correction. *All* peaks must be integrated for this to work properly.
 - (optional) Select [**lv/ltf**] button to adjust slope and bias of integral:
 - Click on integral on left side of spectrum and drag up or down to level the integral.
 - Click on integral on right side of spectrum and drag up or down to level the integral.
 - Select [**Box**]/[**Cursor**] to get out of lv/ltf mode
 - Repeat if necessary.

Plotting

- Set **plotter='LaserJet_300R'** (inner room) or **'lj4m_S168'** (outer room) or select [**Main Menu**]
 - [**Change Plotter**] and cycle through plotter choices until the desired one is displayed.
- Use **text** command to set title for plot: **text('title of plot')**.
- Automatic plotting is available by typing **plot**.

Commands for manual plotting

- pl** - plot spectrum and, if currently displayed, the integral.
- plww** - plot a series of spectra taken in an arrayed experiment in whitewash mode.
- pl('all')** - also plots a series of spectra from arrayed experiments, not whitewashed.
- pscale** - plot axis under spectrum. To change the units on the axis type **axis='h'** for Hertz or **axis='p'** for ppm.
- pap** - plot all parameters in the upper left of the page.
- ppa** - plot a short list of parameters in the upper left corner of the page.
- ppf** - plot peak frequencies above spectrum with a line going down to the peak.
- ppf('top')** - same, but peak frequencies are all plotted along the top of the plot.
- ppf('pos','top')** - same, but plot only positive peaks.
- vp=vp+80 ppf('top')** **vp=vp-80** - same, but plot with short lines that don't go all the way down to the top of the peak.
- pir** - plot integrals under the spectrum. **vp** (vertical position of the spectrum) must be at least 12 for this to work.
- pirn** - plot normalized integrals under the spectrum. Again, **vp=12**
- page** - send plot to the plotter. The output from all plotting commands go on the same plot until the **page** command is typed.
- page('clear')** - delete everything that has been plotted so far instead of printing it.
- pll** - plot a list of peak frequencies in ppm and in Hz. Do not do a **pap** or **ppa** or **page** if you do this command.
- pli** - plot a list of integrals. Do not do a **pap** or **ppa** or a **page** if you do this command.
- text('title of plot')** - set plot title.
- pltext** - plot the title set by the **text** command in the upper left of the plot. The text will also be plotted with the **pap** and **ppa** commands.

Parameters

- wc** - width of chart in mm. Determines the width of the spectrum on the page.
- sc** - start of chart in mm. Distance of right edge of spectrum from right edge of page.
- wp** - ppm range of the plot.
- sp** - start of the plot in ppm.
- vp** - vertical position of spectrum in mm. Moves spectrum up and down on the page.
- wc2** - height of chart in mm. Usually set equal to **wc2max**. Determines where top is in **ppf('top')** command.
- vs** - vertical scale of the spectrum.

Printing

-- Anything output to the VNMR text window can be printed: **printon commands printoff**, eg: **printon dg dg1 dg2 dgs printoff** will print all the parameters to a page.

Other Nuclei and Special Experiments

On the VXR-300, ¹⁹F and ³¹P are also available on a routine basis.

- Select [Main Menu] - [Setup] - [Nucleus,solvent] - [P31] - [D2O].
- Change appropriate parameters (e.g., to change the number of scans, type **nt=128**).
- Type **ga** or select [Acquire] - [Go,wft].

For other nuclei, variable temperature work, or other non-routine experiments, contact Letitia a week in advance to schedule time for a probe change.

Exiting VNMR

- *Reinsert the dummy sample and lock and shim on it!!*
- Type **exit** on VNMR command line or use permanent menu button: [Exit VNMR].
- Sign the logbook.