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 **[lvl/tlt]** 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 lvl/tlt 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, ^{19}F and ^{31}P 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.