



## I. INTRODUCTION

VNMR has menu buttons that can be used to operate the spectrometer and manipulate spectra. When you click on a VNMR menu button, the program executes NMR commands. You can alternate using menus and typing commands at any time. This section will show how to acquire, process, display and plot spectra solely by using menus. . Examples of some of the most useful commands executed by the menus are shown in bold in the margin. The handout on manual operation discusses the individual commands that are used to carry out the same functions.

## II. INSERTING, EJECTING SAMPLES.

You must first eject the standard sample (CDCL<sub>3</sub>) and then place your sample in the magnet to acquire a spectrum. Please, use Wilmad 528-PP 5 mm NMR tubes (other tubes will damage the probe). **Please take your time and handle tubes with care. NMR tubes are extremely fragile. Broken tubes and spilled samples may cause expensive damage to the Gemini.**

Eject Sample  
Insert Sample

**e**  
**i**

Type **e** to eject the CDCL<sub>3</sub> sample.

Place your sample in the spinner and use the depth gauge to position the nmr tube. Make certain your sample column =>5cm. Carefully wipe the spinner and clean the nmr tube. Reposition if needed. Put the spinner in the magnet bore, making certain that the spinner is freely floating on the air column, then type **i**. Follow the same steps to remove your sample when you are finished. Leave the sealed CDCL<sub>3</sub> sample in the magnet and be sure to sign both the log and the cards before you go.

## III. OPERATION USING MENUS

### A. Menu Overview

The **vnmr** menu bar shown below consists of a fixed menu and a menu that changes according to the functions currently active. The fixed menu is the upper row of menu buttons. From left to right, these buttons can abort an acquisition (**Abort Acq**), cancel a command in progress (**Cancel Cmd**), activate the glide menu (**Glide**), display the main or primary floating menu buttons (**Main Menu**), display a description of the current active menu buttons (**Help**), flip or hide the text window behind the graphics window (**Flip**), and resize the graphics window from small to large (**Resize**).



The lower row of main menu buttons brings up menus that let you manipulate the experiment workspace (**1:Workspace**), setup the spectrometer for different experiments (**2:Setup**), and acquire, process and display spectra (**3:Acquire**, **4:Process**, **5:Display**). Functions are also available for analysis of spectra by simulation and deconvolution (**6:Analyze**) and for saving and manipulating data files (**7:File**). The button **8:More**, will display additional functions for selecting plotters, configuring hardware, creating a Unix window, etc.



You can activate any on the menu functions by putting the mouse arrow on the function and clicking the **LEFT** mouse button. You can use the function keys (**F1 to F8**) on the keyboard to activate the menu functions, instead of using the mouse. Click **Main Menu** whenever you are in doubt about where you are in the floating menus. All menus are within six clicks of the **Main Menu**.

## B. Experiment Setup Using Menus (Button 2: Setup on Main Menu).

Put your sample in the spectrometer as described earlier (and i). Then click **2: Setup** from the **Main Menu**. The floating palette will show the following choices:

**1:H1,CDC13** **2:C13,CDC13** **3:Nucleus,Solvent** **4:Sequence** **5:App Mode** **6:Acquire**. You can setup for a proton or carbon experiment optimized for CDG by choosing buttons 1 or 2. If you want to choose a different solvent and nucleus, click on **3:Nucleus,Solvent**. This will display a choice of nuclei:

**1:H1** **2:C13** **3:Return**.

Choose **1:H1** or **2:C13**. You will then be given a choice of solvents:

**1: CDC13** **2: D2O** **3: Benzene** **4: DMSO** **5: Acetone** **6: Other** **7: Return**

Choose a solvent. At this point, a parameter table for the experiment will be displayed. You can change any of the parameters at this point, e.g., number of transients, sweep width, etc., before starting the experiment.

Equiv. Command:  
**setup('H1','CDC13')**

## C. Menu Acquisition (Button 3: Acquire on Main Menu)

To acquire a spectrum using only the menus, click **Main Menu** and click on **3: Acquire**. This displays a menu of acquisition choices:

**1: Show Time** **2: Go** **3: Go, Wft** **4: Go, Periodic Wft** **5: Automatic**

**1: Show Time** will display an estimate of the time required to obtain the spectrum. **2: Go** will simply acquire the spectrum, but carry out no further processing. **3: Go, Wft** will acquire the spectrum and then perform a fourier transformation. **4: Go, Periodic Wft** will periodically transform the spectrum as it is being acquired. **5: Automatic** will carry out user-definable procedures to process and plot spectra automatically after acquisition is completed.

For basic  $^1\text{H}$  and  $^{13}\text{C}$  spectra, use **3: Go, Wft** or **4: Go, Periodic Wft** to start the experiment. You should now wait until acquisition is complete before proceeding. When acquisition is finished, the spectrum will be automatically fourier transformed and interactively displayed.

**3: Go, Wft**  
**ga**  
**4: Go, PeriodicWft**  
**bs=16 ga**

## D. Menu Processing and Display (Button 5: Display on Main Menu)

The **5: Display** menu button on the **main menu** display buttons that let you select menus to display, message, reprocess and plot spectra. This menu is the best starting point for choosing between interactive display, processing and plotting of spectra. You can return to this menu at any time by clicking **Main Menu** and then **5: Display**.

**1: Interactive** **2: Message** **3: Size** **4: Reprocess** **5: Labels** **6: Plot** **7: More**

**1: Interactive** displays the transformed spectrum (if present) and shows a menu for interactively manipulating spectra. **2: Message** gives a menu to phase-correct, integrate, baseline correct and scale the spectrum. **3: Size** changes the physical size of the spectrum on the output page. **4: Reprocess** retransforms the spectrum. **5: Labels** gives you a menu to show peak, integral and text labels. Finally, **6: Plot** gives a menu for plotting the spectrum, peak labels, parameters and text.



Interactive display:  
ds

You are put in interactive display mode as soon as acquisition is completed, which is the equivalent of typing **ds** or clicking on **1:Interactive**. The display menu shown below is active anytime you are interactively displaying spectra:

**1:Box** **2:Integral** **3:Full** **4:Th** **5:resets** **6:Phase** **7:Next** **8:Return**

Some buttons in this menu will change, depending on the state of the displayed spectrum. This menu can be used to switch between a single cursor or two cursors (**1:Box** or **1:Cursor**), display an integral (**2:Integral** or **2:No Integral**), display the full spectrum or expand a region defined by two cursors (**3:Full** or **3:Expand**), display a minimum threshold for peak-picking (**4:Th**), add and remove reset or break points in the integrals (**5:resets**), and phase the spectrum (**6:Phase**).

At this point, the spectrum will need to be phased, integrated and scaled for plotting. Many of these functions can be carried out using the **Message** menu. Click **8:Return**. Now click on **2:Message**

Autophase:  
aph  
Calc int. regions:  
region

The message menu shown below will appear.

**1:Autophase** **2:Region** **3:BC** **4:DC** **5:Adj VS** **6:Adj IS** **7:Adj WP** **8:Return**

Click **1:Autophase** to phase the spectrum. Click **2:Region** to calculate integrate regions. You can carry out a spline baseline correction (**3:BC**) or a linear DC offset correction (**4:DC**). Use **5:Adj VS** to adjust the vertical scale of the spectrum and **6:Adj IS** to adjust the integral scale. Finally, click on **7:Adj WP** to display only that region of the spectrum that has any peaks. Note that you can only adjust the width of the plot AFTER you have calculated integrals. Now click **8:Return**, to get back to the display and processing menu. You are now ready to plot the spectrum.

### E. Menu Plotting (5:Display on Main Menu, then 6:Plot)

The Plot menu buttons correspond to:  
pl pscale  
pap ptext pir  
ppf and page

After you have finished massaging the spectrum, click **Main Menu**, then **3:Display**, and **6:Plot**. A menu of plot choices will be shown:

**1:Plot** **2:Scale** **3:Params** **4:TextOnly** **5:Int.Values** **6:Peaks** **7:Page** **8:Return**

Click **1:Plot** to plot the spectrum and integral (if displayed) **2:Scale** to add a scale to the plot, **3:Params** to plot a table of parameters, **4:TextOnly** if you want to output your sample description without parameters, **5:Int.Values** to plot numeric values under each integral, and **6:Peaks** to label peak frequencies above each peak. Finally, click **7:Page** to output all the information to the plotter or laser printer.

## III. REMOVE YOUR SAMPLE AND EXIT

When the experiment is finished, you should remove your sample and insert the standard CDC<sub>3</sub> sample. Hide the **glide** interface by clicking **glide** on the menu button bar. Type **e** to eject your sample. Place the CDC<sub>3</sub> standard in the spinner, adjust its position using the gauge and place the sample in the magnet bore. Type **i** to insert the standard back into the magnet. Exit the NMR program and logout of the workstation as described on the first page.

Type **exit** in the NMR command window. The NMR windows will disappear. Next, place the mouse arrow anywhere on the background. Click and release the **RIGHT** mouse button. A pop-up menu will appear. Click on the word **EXIT**. You will be asked to confirm your choice. Click on **EXIT**. After a few seconds, the graphics environment will stop and you will be logged off the Unix workstation.