

Reminder Sheet for Bruker DRX-500 Operation

abbreviations:

L : click left mouse button	M : click middle mouse button
LL : double click left mouse button	LH : click and hold left mouse button
MH : click and hold middle mouse button	Menu Buttons in Bold Face
C : red diagonal arrow cursor	V : vertical arrow cursor
> XwinNMR prompt	% UNIX prompt

I. Insert Sample, Lock and Shim.

1. Login: **LL** on icon with your name, enter password
2. Start NMR Software: Position **C** in winterm (blue window), **% xwinnmr LH** on top (grey) bar, move XWIN-NMR window to upper left of screen, **L** on **Windows**, **L** on **Lock**, re-size lock window to fit space to right of XWINNMR window.
3. Change Sample (BSMS keyboard): **Lock Off**, **Spin Off**, **Lift On/Off**, remove and replace sample at top of magnet bore. **Lift On/Off**, **Spin On**
4. Auto Lock: **>lock**, select solvent from list (**DO NOT** lock manually)
5. Shim (BSMS keyboard - **On-Axis** and **Fine** buttons must be selected): Adjust Z1, Z2, Lock Phase; Reduce **Lock Gain** if necessary. Press **Stdb** when finished. If necessary, **>rsh current-n3** to recall starting shims (**>rsh current-dual** for dual probe), then shim Z1 and Z2.

II. Acquire an FID.

1. Create Data Area: **L** on File, **L** on New, edit fields for Name (your sample ID) and EXPNO (experiment); e.g., **NAME**=menthol and **EXPNO**=1 (¹H), 2 (¹³C), 3 (APT), 4 (COSY), etc., **L** on **SAVE**
2. Read in Parameters: **>rpar**, use slider on right to move to end of list, highlight parameter set for desired experiment: std1h-dual, std13c-dual, std_apt-dual, etc. "Dual" refers to dual ¹³C-¹H probe, "n3" refers to Nalorac triple-res. probe, etc.
3. Move to FID Area: **>a** (**L** on **Return** to go back to Spectrum Area). For all but 1D ¹H spectra: **>wobb**, adjust tune and match at probehead, **>halt**.
4. Adjust receiver gain: **>rga**

5. Begin Data Acquisition: **>zg** (info window shows scans and remaining time)
For ^{13}C , use **>tr** to process data during acquisition, **>halt** to stop acquisition

III. Processing Your Data.

1. Check LB: **>lb** (window shows current value, edit with **L** and keyboard)
2. Fourier Transform: **>ef** (automatically switches display to spectrum window)
3. Adjust Spectrum Display: **L** on buttons in upper left corner of display: **|<>|** to see full spectrum; ***2**, ***8**, **/2**, **/8** to change vertical scale; **<>** to expand and **><** to compress horizontally; up and down arrows to move spectrum up and down. Interactive adjustment of vertical scale: **LH** on (vertical position) or (vertical scale) and drag up or down.
4. Manual Phase Correction: **L** on **Phase** to enter phase correction mode, set pivot peak by **L** on **biggest** (to use biggest peak) or **cursor** (to position **V** on upfield peak and **M**). **L** on ***2** or ***8** to exaggerate phase errors, **LH** on **PH0** and drag up/down to correct pivot (dotted vertical line) peak, **LH** on **PH1** and drag up/down to correct "other" peak. **L** on **Return**, **L** on **Save and Return** to exit phase correction mode and apply correction to spectrum.
5. Expand Region of Reference Peak: **L** anywhere on spectrum, move **V** to left edge of desired region, **M** and move second **V** to right edge, **M** and then **L** to get back **C**.
6. Set Reference Chemical Shift: **L** on **Calibrate**, move **V** to top of reference peak and **M**. Enter Reference Shift in ppm (no need to specify units).
7. Baseline Correction (for accurate integration and peak picking): **>absf1** and enter 15 (ppm) for ^1H or 250 (ppm) for ^{13}C , **>absf2** and enter -5 (ppm) to indicate entire spectral region. **>abs** to automatically correct baseline.
8. Integrate: **L** on **|<>|** to display full spectrum, **L** on ***2** or **/2** to adjust vertical scale, **L** on **Integrate** to enter integration mode. **L** anywhere on spectrum and position **V** at left edge of first integral, **M**, move **V** to right edge and **M**. Repeat **M**, **M** for each integral. To highlight one integral: Position **V** within integral region and **L**. Under *Current* bar **L** on **Calibrate** to enter normalized area of this integral or **Delete** to remove it. Under *All* bar **L** on ***2** or **/2** to adjust height of all integrals on screen. To change color of integrals displayed on screen: **L** on **Display, Options, User Interface, integral** and adjust Red, Green and Blue sliders. **L** on **Apply** and **LL** on upper left corner of *User Interface Settings* window to remove it. **L** on **Return** and **Save as 'intrng' & Return** to exit integration mode and save the integral regions for your spectrum plot.

9. Peak Picking: >pscal, select **global**. **L** on **Utilities**, **L** on **Y** and / or **YU** as required to get a vertical scale in cm, **L** on **Return**. **L** on **Analysis**, **Peak Picking** and **Define Region**, set **F1** and **F2** values to desired ppm range and accept defaults for everything else. **L** on **Analysis**, **Peak Picking** and **Adjust minimum intensity**, move horizontal line up to desired threshold level and **L**. >psign and **L** on **pos**, **neg**, or **both** to select type of peaks to be picked. To print out a list, **L** on **Analysis**, **Peak Picking** and **List peaks on screen** and **L** on **Print**.

IV. Plotting Your Spectrum.

Do Not Use the Plot Button in XWIN-NMR!

1. Enter plotting program (XwinPlot): **L** on **Windows** and **Plot editor**
2. Set up Printer (first time only): **L** on **Options** and **Printer Setup**, under *Printer Type* highlight HP LaserJet 5L (very important!). For thesis margins, set *Left Offset* and *Bottom Offset* to 3.81 (cm) and *Right Offset* and *Top Offset* to 2.54 (cm). **L** on **File**, **Save as Default** and then **Apply** and **OK**.
3. Get List Information from Spectrum: **L** on **XWIN-NMR** and **XWIN-NMR Interface**, **L** on **Create Parameter List**, **Create Peak List**, and **Edit Title** (**Do not** select **Create Integrals**). Enter sample description into Title editor (e.g., "Oxime Methyl Ester in CDCl₃ 25C") and **L** on **Save** and **Quit** (blue). This title will appear above the spectrum any time in the future when you return to look at this data. **L** on **Close**.
4. Put Parameters on Plot: Use picture icons on left. **L** on Parameters icon (list of text) and move **C** to upper left of desired region of plot, **L**. **L** on Mark Objects icon (square of 8 green dots) and **L** on parameter list to highlight it with green squares and dotted lines. **L** on **Attributes** and set *Font Size* (8.00) and *Font Type* (Times Roman). **L** on **File** and **Save as Default** to always use this font. **L** on **Set** and **OK**. **LH** on highlighted parameter list and drag to position in Plot.
5. Put Spectrum on Plot: **L** on Spectrum icon and position "+" cursor at upper left of desired spectrum region. **LH** and drag to the right and downward to expand spectrum into desired space on plot. To expand a spectral region, **L** on **Expand** and hold down the *SHIFT* and *CTRL* keys on keyboard while you position vertical blue line at left edge of desired spectral region and **LH** and drag to right edge.
6. Adjust Spectrum: **L** on Mark Objects icon (square of 8 green dots) and **L** on spectrum to highlight. **L** on **1D/2D Edit** and use buttons in *Edit Display Object* window as you do in XWIN-NMR to adjust vertical scale (*2 and /2), position (**LH** and drag on double arrows) vertical and horizontal expansion (**LH** and drag on double triangles) and **L** on **Show Peaks** and/or **Show Integrals** to display chemical shifts and/or integrals on spectrum. **L** on **Close** to exit this window. If necessary, **LH** and drag on green squares at corners of spectrum to re-size the spectrum and **MH** and drag on spectrum to re-position.

7. Plot Spectrum: **L** on **File** and **Print**, **L** on **Print**. **L** on **File** and **Close**, **L** on **OK** to exit Plot Editor and return to XWIN-NMR.

8. Exit XWIN-NMR and Log Out. Your data is already saved and you can return to it at any time by **L** on **File**, **Open** and **Data Sets by Name** and selecting your dataset by **L** on the sample description (Name). If there is more than one experiment for this sample (e.g., ^1H , ^{13}C , APT...) you can select experiments within a sample name by **>re #** where "#" is the experiment number (1, 2, 3...). **L** on **File** and **Exit**, **L** on **OK** to leave XWIN-NMR.

Do Not Logout From UNIX Without First Exiting XWIN-NMR!!!
Do Not Exit the Lock Window!!! **Do Not Exit XWIN-NMR Using the UNIX Double-Click on the Upper Left Corner!!!** Any of these actions can leave processes "hung" and the next user will be unable to use the spectrometer!!!

Position **C** on the background ("wallpaper") of the graphics screen ("desktop") and click and hold the Right mouse button, drag down to **Log Out**, **L** on **Yes**. Your research group will be billed for the entire time you were logged in.