Delta NMR software

The Delta NMR processing software used by our Delta and Eclipse spectrometers is available free on Windows 2000 and Windows xp computers.

When Delta is installed, you need to register with Jeol. Go to File / License Key to get the "machine_id". E-mail the ID to Paul Lawrence P.G.Lawrence@bristol.ac.uk who will get the License key from Jeol. When entering the key, set the key as "global" - you will need to run delta with admin rights to do this (right click on the delta icon and "run with admin rights").

To reprocess FIDs (normally a ".1" file) you will need to set up processing lists or use the ones available from:

The chemistry filestore under: T:\ob\nmr\chpgl\shareall\processlists\ or, nmr-master under: files / Processlists

These lists should be copied to My Documents / delta / process_lists on your PC.

Viewing and Plotting 1D data using Delta

Start Delta on your PC. In Delta, open the Data Slate tool (the one with a picture of an NMR spectrum). In the Open File window, select the 1D file you want to view. It should have a suffix H, C, D135, P, D2 etc. Check that the version selected has dimensions in ppm or KHz. (This will normally be a .3 file, less commonly a .6 file. For 31P spectra it will probably be a .5 file.) Click Open to open the Data Slate window. This can be resized with the mouse as required. The Data Slate window opens with the 1D data with X and Y axes. These can be removed with Alt-X and Alt-Y, but are normally required. Similarly, integrals (if present) can be removed (and recovered) with Alt-I, peak markings with Alt-P. The cursor has twelve modes, each of which has several functions. The mode can be selected either with the cursor itself, or with the function keys (F1 to F12). A full description of all cursor functions cannot be given here. Use the floating help feature of the cursor to see a brief explanation of each cursor function.

- **F1 Zoom.** This is generally the most useful cursor mode, especially the Zoom Data function. Within the data area it works as an X-Y Zoom (draw a box), on the X or Y axis it works as X-Zoom or Y-Zoom respectively. Note that there are always two extra cursor functions immediately available, by pressing the Shift key (usually gives the Zoom Data function) or by pressing the centre mouse key (this changes Zoom Data to Pan Data, for example). If your mouse only has two buttons, get a three-button mouse. If your supervisor says (s)he can't afford it (about £5), get a new supervisor.

- **F2 Select.** Used for selecting individual spectra (geometries) when several are displayed, but this can be equally well done with the right mouse key when in other modes.

- **F3 Region.** Used for creating new spectra (geometries) from regions of the existing spectrum. These can be displayed either side by side (vertical view), one above the other (horizontal) or up to 4 x 4 (box). Each geometry can then be individually scaled.

- **F4 Cursor.** Used for selecting positions in a spectrum, for example during baseline correction.

- **F5 Reference**. Two functions here are most useful. Grab Reference takes a reference position from the spectrum, and Paste Reference applies this to another, selected, position in the spectrum. Alternatively, the reference position can be set in Tools Reference from the Menu Bar.

- **F6 Peak.** This mode has many useful functions for working with peaks. To work with Baseline, Noise Level, and Threshold, which determine which peaks are selected by the peak-picking software, you must first press Alt-T to display them. When Baseline, Noise Level, and Threshold have been set, the Peak-Pick icon at the top of Data Slate will pick all peaks above Threshold. Another useful function is j-picking. Select two peaks (with the Select More function), then press j on the keyboard. The separation, J, of the two peaks is displayed (and plotted), and a new peak at the mid-point is generated, from which the chemical shift of the doublet (if that is what it is) can be determined.

- **F7 Pick**. Used in the Data Processor (see below) for selecting the PP position for phasing, or in 2D spectra for selecting slices. Less useful in Data Slate.

- **F8 Integral.** Another complicated mode, with several functions for creating and adjusting integrals. The vertical gain functions in particular are rather awkward at times.

- **F9 Text.** Can be used to write text annotations on the spectrum, which can be useful since the Comment cannot be changed. Exact positioning relative to the Filename and Comment can be difficult, since the display is not truly WYSIWYG.

- **F10 PIP.** This means Picture in Picture, and allows a portion of a spectrum to be drawn as an overlay. Otherwise it works very like Region (F4).

- **F11 Offset.** Used with overlaid spectra (use Open Overlay) to change the offset between them.

- **F12 Molecule.** Can be used to draw molecular structures on the spectrum, either using imported structures drawn, e.g., in Chemdraw, or using the built-in molecule drawing package. As for Text, exact positioning relative to the Filename and Comment can be difficult, since the display is not truly WYSIWYG.

When you have finally got the screen display as you want it, simply press the printer icon, then hit OK to print on the selected printer. Before plotting you might want to check whether certain plotting options have been set, e.g. Plot Params or Plot Processing List. These can be set in the File menu of Data Slate, or set in Preferences (see below).

Preferences

Many features of the display can be turned on or off with Alt-key combinations, or accessed with a right-mouse click. Some have been mentioned above, e.g. Alt-X. The default behaviour can be set in the Preferences file, which can be edited from the File menu in the main Delta window. Functions affecting the data display are on the Data page.

Some of the more useful options are given below.

- Alt-Shift-C Comment
- Alt-F File name
- Alt-G Grid
- Alt-I Integrations*
- Alt-N Data Points
- Alt-P Peaks*
- Alt-T Statistics (Baseline, Noise Level, and Threshold)
- Alt-X X-Axis
- Alt-Y Y-Axis *

These functions cannot be switched off if the appropriate cursor mode is selected.

Viewing and Plotting 2D data using Delta

Start Delta as normal. Use ssh to copy all the relevant spectra from the spectrometer computer (or cho097) to your own PC. "All" includes the high-resolution spectra which will be drawn on the sides of the 2D spectrum.

In Delta, open the Data Viewer tool (the one with a picture of a video camera) In the Open File window, select the 2D file you want to view. It should have a suffix HH, PP, CH, HMQC, or HMBC. Check that the version selected has dimensions in ppm or KHz. Click Open to open the viewer window.

The Data Viewer window opens with the 2D data and two places for slice/projection data in both X and Y dimensions. On opening both will contain projection data. In the Display menu select Row Slices, then Column Slices, which will remove the space for the projection data. Then under Display High-Res Load X Slice select the high resolution spectrum for the X dimension (make sure it is dimensioned in ppm, usually a .3 spectrum) and under Display High-Res Load Y Slice select the high resolution spectrum for the Y dimension (again make sure it is dimensioned in ppm, usually a .3 spectrum).

Note that the high-res spectra may not align perfectly with the 2D spectrum due to differences in calibration. See the item Referencing in 2-D spectra on Delta systems

Control of the contours displayed is done with the Level Tool. Select this with the right mouse button. The Level Tool has two sets of controls. At the left you can select up to 24 levels to be plotted (the left hand set are for negative levels, only used in phase-sensitive 2D spectra). Normally 6 to 12 will be quite sufficient. On the right are two slider bars labelled

Bounds and Bias . As you raise the Bias slider, you will see the green line in the centre move to the left, i.e. to lower thresholds. The Bounds slider can be used to raise the lowest threshold, but is less useful.

After any change in Bounds or Bias, you need to click Apply to recalculate the contours. Levels which are too low, and therefore produce too much noise, are not calculated, and shown in grey. This limit can be removed in Level Tool with the right mouse button then Options. After calculation, levels can be removed from the display by clicking on them, then restored.

The Level Tool can easily become disconnected from the 2D spectrum. To restore the connection, use the right mouse button in the 2D data area.

In the 2D spectrum, most cursor functions work as they do in 1D spectra. The Zoom tool is probably the most useful. Slices, if required, can be selected with the Pick tool. The slice will then replace the High Res spectrum.

Referencing in 2-D spectra on Delta systems

2-D and high-resolution spectra on the Delta are aligned by chemical shift, not by the observed frequencies (probably an error). High-resolution spectra are referenced by the Autoref function, which looks for TMS, then for the solvent, finally reverts to a calculation from the absolute frequency. 2-D spectra are only referenced by the third method, so if Autoref has found a TMS or solvent peak, there will usually be a small difference between the two references.

The solution is to reference the 2-D spectrum from a TMS or solvent peak BEFORE loading the high-resolution spectrum into the slice (or projection) area. After you have loaded the 2-D spectrum, expand the peak you want to use as a reference (TMS obviously preferred). You will normally see that it is not exactly at 0.0 ppm (in either dimension). To correct the reference, select the Reference cursor mode, then the first option (Grab Reference). Click the mouse cursor on the position which is shown as 0.0 in x, 0.0 in y, then move the cursor to the centre of the TMS peak and press the centre mouse button (the Paste Reference function).

Now that the 2-D spectrum is "correctly" referenced, you can load the high-resolution spectra and they will be properly lined up with the 2-D spectra. Note that you must load (or reload) the high-resolution spectra AFTER you have recalibrated the 2-D spectrum.

Processing 1-D spectra on Delta systems

Old data is only stored as FIDs, normally a ".1" file. Processing is done with the Data Processor (top left of Delta main window). Opening this gives the Open Data window, in which the file for processing can be selected. Check that the dimensions of this file are seconds [s] before opening it. The Processor window which then opens will automatically detect whether the data is 1-D or 2-D, and for 1-D data will show the FID twice. This is because no processing has been applied (right hand side of window). You must either create or read from disk a Processing List. By clicking on Open Processing List you will get a list of pre-written lists, which should include a suitable one for the spectrum you are processing

(more can be added on request, or downloaded from cho097 (delta / process_lists).

Having loaded a suitable list, click on Process to convert the FID to the frequency-domain spectrum. The 1H list supplied on cho097 also does Automatic Integration and Peak Picking. The 13C list omits the Integration, and is suitable for other nuclei like 31P, 29Si, etc.

Possible easy amendments to a processing list are Zerofill (from Pre-Transform menu, insert this after the window function. x 2 is normally adequate) or adjustments to the Gaussian window. Decreasing Line Width or increasing Shift will improve resolution, the reverse will improve signal/noise ratio. The automatic phasing is quite good, but may occasionally need refinement. The PP marker for the largest peak is an inverted yellow T under the spectrum, which can be moved with the Pick cursor function. Then adjust P0 and P1 as you would in SpecNMR. When you have finished phase adjustments hit Apply. This will put those phase values into the processing list. The position of the Phase correction in the processing list may, however, need adjustment with cut and paste.

Baseline Correction (from Post-Transform menu) requires re-processing the spectrum, and must be inserted after phasing, but before Peak Pick/Integration. Several modes are available, both Automatic and Interactive (preferred), easiest is probably Piecewise Linear. Baseline positions are selected with the mouse in Cursor mode, a Preview is available, the correction is only applied when Accept is clicked. The spectrum may now be expanded as normal, and plotting works just as from Data Slate. Alternatively you can Put Processed Data into Data Slate (fifth icon from left in top row) and proceed from there.

Processing 2-D spectra on Delta systems

2-D data is automatically recognised by Delta, and the Processor window opens up as the nD Processor, but with no spectrum displayed. There are now two processing list, one for F2 (rows) on the left, one for F1 (columns) on the right. These are stored together in one processing list on disk, so you should open 2d_cosy.list or 2d_chshf.list as appropriate.

These should work well in most cases. Choose the icon Process File and Put in Data Viewer, which will transform the data in both dimensions and put the transformed spectrum into Data Viewer.

Amendments to these lists are possible just like 1-D lists. Suggestions might be Symmetrisation and Ridge reduction. Be careful to select the X or Y sub-lists before inserting (Y for symmetrisation or ridge). A preview of the results is available via the Toggle icon (top right). The Sinebell window used for 2-D processing is unsuitable for broad lines, so some amendments (changing shift to –2 or –5) can sometimes help.

Note that when the 2-D processing is completed, the nD processor window remains open. It is helpful to close it when it is no longer required.