Fluid Inclusion Data Reduction:

Nuclear Microprobe analysis of fluid inclusions is a sophisticated and well-characterised standardless method. This means that analytical uncertainties that arise from depth estimates, geometry corrections, density estimates etc. cannot simply be swept under the carpet with a "internal standard" fiddle factor. Moral of the story: garbage in – garbage out. Before you embark on meaningful data analysis consider the following:

1. All measurements (size, depth-to-midplane etc.) you make have to be as accurate as possible - "guesstimates" just won't do.
2. If you are unsure on the day that you analysed the inclusion you thought you were analysing, don't bother reducing the data. If it's not the inclusion you think it is the measurements (and results!) are meaningless.
3. Think about the depth of inclusions and the resulting meaningfulness of the data. For example, good data for Cl in saline inclusions can only be obtained for inclusions less than 20 microns depth, dilute inclusions have to be less than 10 microns. S numbers are rarely meaningful, firstly the flincs can't be deeper than 5 microns and there can't be much Cl (because Cl lines usually swamp S lines – that always makes the S images look exactly like the Cl images).
4. A number of 200% Cl is not evidence of a dubious technique, it's evidence that the software is probably over-correcting for depth, i.e. wrong inclusion or bad depth measurement (dubious analyst).
5. Think about spectral overlaps – e.g. As and Pb. In most cases GeoPIXE is smart enough to resolve these, but if one element occurs in very high concentrations (e.g. wt% As), you have to look closely at the PIXE spectrum to convince yourself that the Pb is real.
6. Think about detection limits. Sometimes big numbers appear for some elements that occur in the "grassy" regions of the spectrum (e.g. Sn, Ag, Sb) but when you look at the spectrum there's nothing there. The warning signs usually is when you start imagining your Nature paper – talk to Chris immediately.

Building the Directory Tree

1. Transfer all the raw data from the data acquisition to an appropriate directory on one of the PC disks.
2. In Explorer, go into this directory, click on the "Size" column and delete all the files that have a file size of "0".
3. Run GeoPIXE
4. On the "Sort EVT File" window, make sure the correct data format is shown in the top drop-list, and then click "B"
5. A window opens called "Batch Sort EVT", click the "Dir" button, and select the directory of interest.
6. All the files should load into the table
7. Click "Build Tree"
8. Click "Save" and save the .sbatch file with the default name given.

Doing image analysis as a batch job

The principle of doing the imaging as a batch job involves doing one analysis the long way that GeoPIXE can then use as a template for all the other analyses.

Doing one analysis as a template:

1. On the "Sort EVT File" window, click on "EVT File:" button and a select a typical file to be used as a template by browsing the directories until you find it.
2. Enter the charge as noted in the logbook in the text field provided
3. Load a .DAM file by clicking on the "File" button ¾ way down the window– it is recommended that you use a well-tested template such as "flinc-d10-t10.dam", copies of which can found in most of the project directories (perhaps make a copy in the current directory you are working on for future use).
4. Click "Start" on the "Sort EVT" window (not the batch window yet).
5. The image will then appear in the main "GeoPIXE II 3.x Image" window
6. On this window, go to the "Window" menu and open the "Image Processing" window.

7. Go through all the images and apply image-processing operations to each as you think appropriate from both the “Image Processing” window and the colour buttons below the image.
8. Go to the “File” menu and select “Save” – save the .dai file (usually it will be the original file with “-m” added to the filename.

Doing the rest of the images as a batch job:

1. Go back to the “Batch Sort EVT” window. Delete any rows that represent any aborted analyses and save the information as the default “.sbatch” file.
2. Using the logbook, carefully enter the preset charge (Q) for each analysis; take care to note that they are listed in the table in alphabetical order, not the order in which they were analysed. Periodically save the information as the default “.sbatch” file as you go.
3. Select from the options below the table how you would like the batch processing to proceed – the normal is to select all.
4. Click “Start”.
5. If you chose “Apply digital filters” you will be required to tell the software where the template file is (as prepared above) – a file requestor will automatically appear. Browse the directories until you find the template file, remember to select the file with the “-m” added to the filename.
6. You will then be asked to select the elements for which the template file will be consulted – default is to select all.
7. Next you will be asked which elements to export as HTML (if you selected this option on the “Batch Sort EVT” window) – choose the elements you want. This may happen twice if you also requested the black-and-white export option.
8. Go have coffee - this will take a while.
9. Completed HTML files (+ all the associated .png files) can be zipped (select all files + right click + “Add to Zip”) one by one in Explorer to be written to CD or dispatched via email.

Quantitative Fluid Inclusion Analysis

Welcome to quantitative fluid inclusion analysis. Make yourself comfortable and lets begin …

Quantitative data analysis is done on an inclusion-by-inclusion basis, so you will be reading the next few steps several times:

1. On the main "GeoPIXE II 3.x Image" window (just called the main window from now on), select "File" then "Load"; select the .dai file of interest, usually one with "-m" added to the file name.
2. From the droplist below the image, select "Ellipse" and then drag an ellipse to completely encompass you flinc. Use various images that may show inclusion shape (e.g. Cl) and your notes in the logbook to help you position the ellipse in the right place.
3. Watch the ellipse size in the text box – it should be a little bigger than your flinc dimensions. If it's smaller, or more than a factor of 2 bigger, you have the wrong inclusion.
4. Click the "∑" (sigma) button – numbers will appear in the "Image Regions" window (these are NOT corrected for flinc geometry so ignore).
5. On the "Image Regions" window, click the "Save" button and save the file with the default filename given.
6. On the same window click the EVT button, and select the defaults as they are requested (two pop-up requesters).
7. Spectrum should appear in the "Spectrum Display" window.
8. You can zoom in on the spectrum be setting the 2 "View" markers left and right of the area you are interested in and then selecting "Widen".
9. On the "Spectrum Display" window, click on "Window" menu, then "PIXE fit".
10. On the "PIXE Fit" window, select ONLY the elements you want. The colour legend at the top of the periodic table will tell you which lines will be fitted for each element (green for K lines, yellow for L lines, blue for both K and L, …).
11. MAKE SURE THE "**CSIRO #34"** DETECTOR IS SELECTED, NOT THE **#34-out**, change if necessary.

12. Turn “Boost” to “On”.
13. Next to the empty field called “Yields” is a button called “New”, click “New”.
14. The “Layer II – PIXE Yield Calculation” window appears. In the middle of the window is a box entitled “Target Layer Selection”.
15. Next to #Layers, select “3”, and make the “unknown” layer “2”.
16. Sequentially enter the composition of each layer in the large text box by first selecting the layer you are defining from the droplist next to “Define Layer”. As an example, the simplest case for flincs in qtz would be (don’t use subscripts):
    - Layer 1: (SiO2)100.0
    - Layer 2: (H2O)75.0(NaCl)25.0
    - Layer3: (SiO2)100.0

Follow the rules for entering matrix information as given in the “Help” below the text field, and select “weight %” from the droplist on the right.

17. Each layer requires a thickness, this “sandwich” yield model will be a generic starting point, so just make layer 1 and 2 both “10”. Layer 3 is infinite as far as the beam is concerned so make this some big number like “500” Enter the density for Layer 1 (if qtz then 2.6) and 2 (if water then 1.0) Density for Layer 3 doesn’t matter because it’s infinite.
18. Below the “Target Layer Selection” click on “output” and give the yield file a name – usually something generic like “flinc-d10-t10”. Later, you can load these yields into “PIXE Fit” using the “Load” button.
19. At the top of the window click on the “Save” button to save this set-up, and finally click on “Calculate Yields” – confirm the filename for the yield file when the file requested pops up.
20. Close this window and go back to “PIXE Fit”
21. Next to the word “Fit” click on “One”. The fit should appear on the green spectrum as a red line, and the background fit appears as a purple line. This is the point where you have to think about the quality of the data, goodness of fit, spectral overlaps and all those good things I talked about at the start. This part of the training you will have to sit down with Chris or Tin Tin to interpret the spectrum.

22. When you are happy with the spectrum, go to “Window” again and select “Fit Results”.
23. On the “Fit Results” select the inclusion of interest and then click “Properties”.
24. Fill in the missing details – flincs dimensions, depth and density
25. Click “Apply”
26. Look at the data and decide whether it makes sense (e.g. no 200% Cl?) – look at the elements in the grassy part of the spectrum. They may have numbers in the table that you will have to veto by clicking the “Veto” button – this will replace phoney numbers with the detection limit for that element.
27. That’s it. Save the data by clicking on the save button – it will be saved in GeoPIXE format as a .pfr file.
28. Then export the data using the “Export” button as a .csv Excel file. Select the “Concentration”, “Uncertainty”, “Detection Limits”, “Veto light shallow elements” output options. The latter will veto data for light elements from deep inclusions, where the uncertainties grow too large. Click “OK” to output the CSV file.

The first inclusion require all these steps, if the second one is in a similar matrix then steps 10 to 20 are not required again, just keep using the same PIXE-Fit set-up. Don’t forget to save the PIXE_fit file for tomorrow.

All data are collated for your use and our database in a single spreadsheet file, which also contains all the geometry and thermobarometry info you have already done. This means carefully adding each inclusion’s results to the final spreadsheet as you go, taking care not to get columns mixed up. The spreadsheet file is saved with your name and the approximate date of your run, e.g. “BinFu_September2003.xls”.

Have fun!
Esmé

Subtraction of Scattered-Beam Induced Background

Beam scattered from slits and the beam-tube are not correctly focussed by the lens system and may hit the sample at some point away from the focussed beam. If sulphides are present, then this scattered beam may induce X-rays from elements of interest, such as Cu. In order to detect this happening, it is best to collect "blank" analyses in areas on the matrix quartz, but away from inclusions and solid phases. Ideally, try to avoid such occurrences by masking areas of sulphide with pieces of thick (100 μm) Al foil.

The procedure is basically the same as above, with a couple of additional steps. Before proceeding at step 24 above, you will need to process the spectrum from this blank area. So, continuing from step 23:

1. Import the IMG total spectrum file for the background area, into the "Spectrum Display" window. To do this, use the "File->Import->New->…" menu.
2. In the "PIXE Fit" window, enter the full integrated-charge for the background analysis into the "Q" text box. Note that this will not normally be the same as that extracted from the ellipse over the fluid inclusion.
3. Click on "Fit: One" to fit the spectrum. The result is appended to the "Fit Results" window, under the fit to the inclusion spectrum. If you like, you can save the spectrum (including the charge data) back to a SPEC file, using the "File->Save SPEC" menu on the "Spectrum Display" window.
4. Now, before step 24 above, drag select both rows in the "Fit Results" table by clicking on the first row label and, while holding the left mouse-button, drag down to the second row label.
5. While editing parameters (step 24 above), make sure you also re-enter the "Beam Shape" "X size" and "Y size" entries. This will force the same parameters to be applied to the background data. It is best to enter values for all parameters, AND hit <return> after each text entry.
6. Now click "Apply", as at step 25 above.
7. Consider the data (step 26 above) and save it to a PFR file (step 27).

8. Now, at step 28 above, in the Export options requester, select “Conc less Scatter” and “Uncertainty”, below it. These will give the inclusion composition with the scattered component subtracted, and the final uncertainty.
9. When selecting data rows from the CSV file to paste into any summary spreadsheets, use these subtracted data rows instead of the usual “Conc” rows.