

## GeoPIXE Worked Examples

*Further details and worked examples can be found in the **GeoPIXE manual** (see “Help” menu of GeoPIXE Image window) in the GeoPIXE Analysis Scenarios and Data Flow chapter and local Maia-384 and Maia-96 worked examples are provided in the **Maia-384--user-help** document. These examples are a little out of date, given new extensions to GeoPIXE and new Maia operating modes, so this workshop guide provides the most up to date worked examples at the moment.*

GeoPIXE Worked Examples .....	1
GeoPIXE Setup .....	2
Edit your ‘geopixe.conf’ file .....	2
Path Bookmarks .....	2
Path Translation.....	2
A. Spectrum Analysis and Fitting .....	4
Example A1: Spectrum display .....	4
Example A2: Fitting a PIXE spectrum .....	5
Example A3: Fitting a SXRF spectrum, Yield modelling .....	7
Example A4: Importing an old XFM Vortex SXRF spectrum .....	10
Example A5: Fitting a XFM Vortex SXRF spectrum, Pileup .....	11
Example A6: Fitting a XFM Vortex SXRF spectrum, Flux Calibration .....	12
B. Dynamic Analysis Imaging – the GeoPIXE Work Flow.....	15
Example B1: SXRF DA Imaging (a simple detector array) .....	15
Example B2: Verifying image features .....	21
Example B3: Exploring and exporting image data .....	26
C. The Maia Detector – Maia Specific Data and Processing .....	36
Example C1: Maia-384 – Probing raw data records (synchrotron) .....	36
Example C2: Maia-384 – Probing raw data records (Maia Mapper).....	37
Example C3: Maia-384 Detector – Rock3 image data .....	39
Example C4: Verifying Maia-384 detector image data .....	53
Example C5: Maia Mapper – MM 925 image data .....	54
D. XFM Ion Chamber Calibration .....	65
Example D1: Maia-384 – Mn calibration foil .....	65
Example D2: Automated Standards Analysis for Maia at XFM - the Standards Wizard.....	67
Example D3: Maia Mapper – NIST 1243 calibration standard .....	68
E. Image Corrections.....	70
Example E1: Correction for changing flux.....	70
Example E2: Correction for element interference .....	74
Example E3: Various Image Corrections .....	76
F. Correction of Yields for Spatial Composition Variation.....	85
Example F1: Correction of Images for Composition Variation.....	86
Example F2: MPDA Imaging - SXRF (the detailed way).....	89
Example F3: MPDA Imaging - PIXE (the detailed way).....	94
Example F4: Spectrum fitting using end-member analysis - SXRF.....	104
G. Depth Mapping using the Maia Detector .....	105
Example G1: Depth mapping and analysis – the Depth Wizard .....	105
H. XANES Imaging .....	113
Example H1: XANES Stacks - Correction for Image Shifts using a Hot-Spot .....	113
Example H2: XANES Stacks – Correction for Shear and Drift using Cross-Correlation .....	122
J. Exporting Data.....	126
Example J1: Exporting image pixel data .....	126

### Important Note

*The workshop and demo data sets use examples drawn from various user applications. These remain the property of these users and the data and derived images and results must not be used without permission from these users. More information can be obtained from the CSIRO Maia Mapper, CSIRO NMP and Australian Synchrotron XFM beamline scientists.*

*These examples cover both SXRF (Synchrotron X-ray fluorescence) and PIXE (Proton induced X-ray emission) applications. These techniques have much in common (ppm sensitivity, deep penetration) and produce similar spectra. Contrasting spectral features are discussed in the examples.*

## GeoPIXE Setup

### *Get Demo data*

Download a copy of the Demo data from the CSIRO FTP site: [ftp.csiro.au](ftp://ftp.csiro.au), using the “geopixe\_update.sav” program and your username/password provided. The data is located on path **/Demo**. The directories “MM” and “Maia” are very large (total 53 GB), so only get these as needed.

### *Get Workshop notes*

The Help directory of GeoPIXE has copies of the Workshop slides, notes and Worked Examples, all as PDF. If you need the PowerPoint slides, these are in the /Workshop path on the FTP site.

## Edit your ‘geopixe.conf’ file

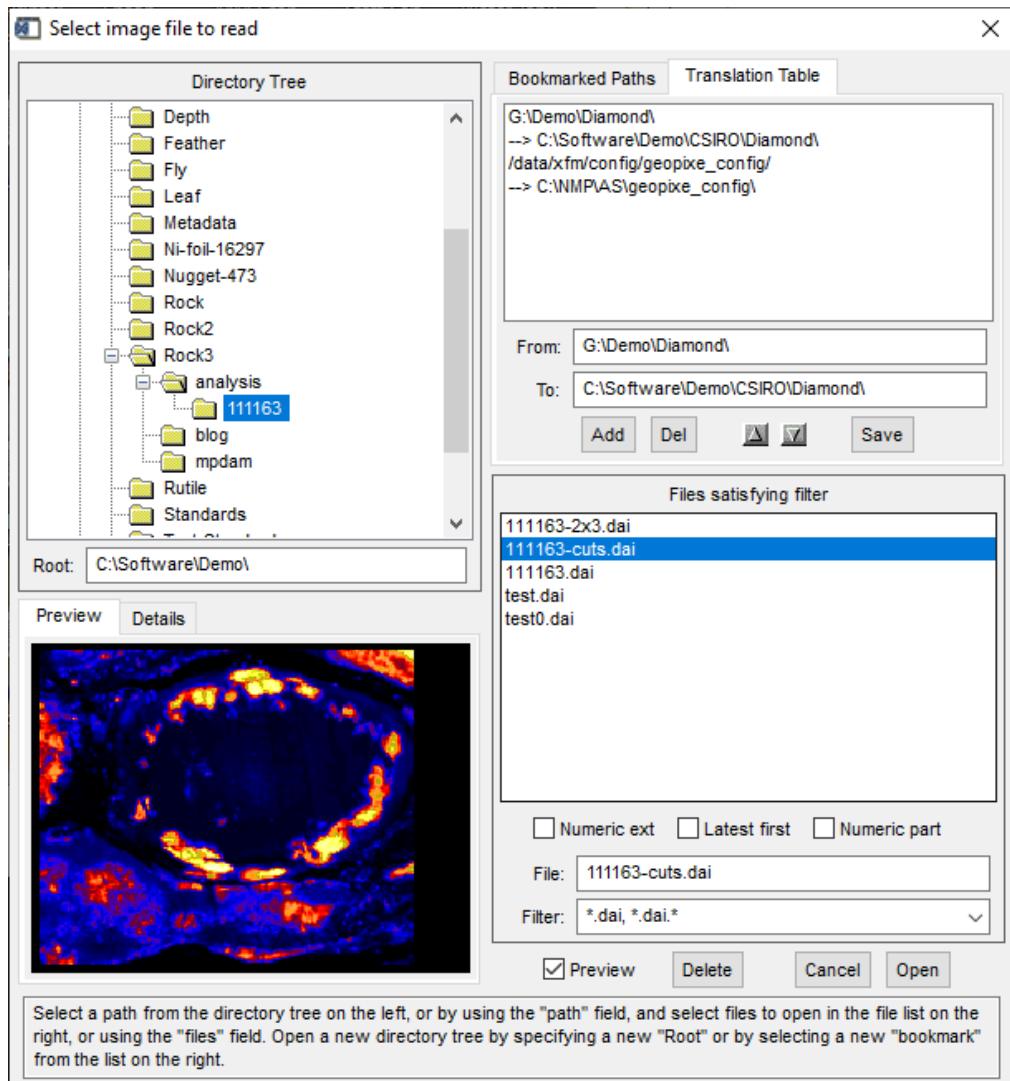
Using the *gedit* command, open your GeoPIXE configuration file ‘geopixe.conf’ located in your home “*~/.geopixe/*” directory.

## Path Bookmarks

After you have navigated to your Demo directory for data and/or analysis, **Add** this path to your Bookmarks in the GeoPIXE **File-Requester**, and **Save** these for later use.

## Path Translation

GeoPIXE can translate paths to a new location using the Path Translation tables, which are set-up in the **File-Requester**. You can define the base of some path and a new value for this base (for example, after you have copied it from the beamline computers, you might wish to translate the beamline path base to your local system path base).



Then, if a selected file path is not found, GeoPIXE will search for a translation of the old path in these Translation Tables. If the file is then found on one of these new translated paths, it is used from the new path.

## A. Spectrum Analysis and Fitting

The examples assume that the demo data has been loaded into a directory “**Demo\**”. You will need to navigate to this new path, and save it as a Bookmark in the file requester for future reference.

### Example A1: Spectrum display

- Loading spectra and display of spectra
- Energy calibration and X-ray line identification
- Escape peaks and pileup

#### Steps to try

1. Load the PIXE spectrum file “Donut2x-2-whole.spec” from directory “**Demo/CSIRO/Donut2x**”.
  - a. Find the “Demo” directory on your system in the File Requester (see tips provided by host). Click on “Add” under “Bookmarked Paths” to add it to your bookmarks list and hit “Save”. Next time you try these examples you can double-click on this bookmark to access the Demo directory directly.
  - b. Similarly, you can set-up a path translation “Translation Table” to automatically translate from old paths to the paths as you see them from your Training account.
2. Set the View markers and expand the view using “Widen”.
  - a. To select the View markers, select “View 0,1” in the marker droplist in the bottom-left corner.
  - b. Set the right marker first, at the high-energy end (left mouse click), and then the left one (set View 2-42 keV).
  - c. Perhaps drag the window larger to see more detail.
  - d. Click on “Widen” to expand View range.
3. View maximum detail in the vertical scale using “Auto” vertical scale.
  - a. Hover mouse pointer over buttons and controls to see help on them.
4. Experiment with re-sizing the *Spectrum Display* window, scrolling left and right through the spectrum using the horizontal scroll bar and changing the X scale (using “Expand”, “Shrink”, “Widen”, “Full”) and Y scale (“Up”, “Down”, “Log/Lin”).

#### X-ray Peak Identification

5. Open the *X-ray Identification* window, if it’s not open, by clicking on the “?” button.
  - a. If the *X-ray Identification* window is in “Mark Element” mode, select “Identify” mode.
  - b. Click in the *Identification window* list to select the “Identify” marker on the spectrum, or select the “Identify” marker on the spectrum droplist.
  - c. Click and drag on the spectrum to move the “Identify” marker.
  - d. The nearest X-ray energy match is highlighted in the Identification list.
  - e. Click on a line in the list to mark that energy in the spectrum (other lines for the selected element will be highlighted in the list).
6. Change the relative intensity threshold using the slider in the *Identification* window (e.g. click to the right of the slider knob to increment the threshold by ~0.05). This hides weak lines.
7. Select “Mark Element” in the *Identification* window and click on elements to mark their lines on the spectrum (e.g. Fe, Sr). Alternatively, click on the arrow buttons to increment/decrement atomic number.
8. Step across atomic numbers Z from 25 to 60 to see elements present as K lines and Z from 73 to 83 for L lines. Beware peaks that are due to “Ge escapes” or “pileup”.

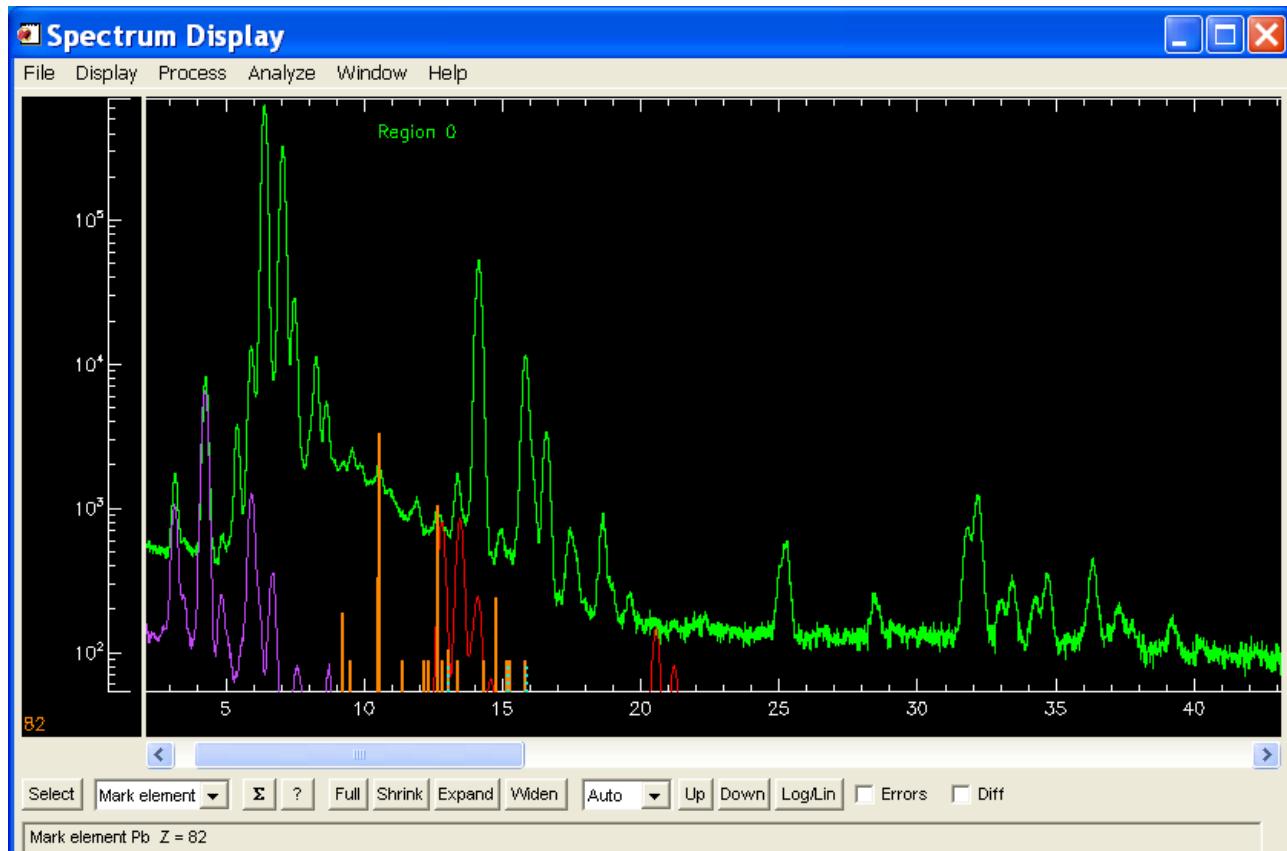
#### Escape and Pileup Peaks

9. Data were collected using a Ge detector. Calculate and display Ge escape peaks using the menu “Display->Ge Escapes”.
  - a. Escapes shown in violet are dominated in this case by the escapes of the Sr K lines. Note the presence of escapes due to both Ge K $\alpha$  (9.88 keV) and Ge K $\beta$  (10.98 keV) energy deficits.
10. Calculate pile-up using the menu “Display->Pileup->from single spectrum”.
  - b. Note the presence of Fe K $\alpha$  pile-up at 12.8 and 13.4 keV. This doesn’t seem to explain the spectrum peak near to 12.6 keV.
  - c. However, there is also Pb present in the sample, which gives a 12.6 keV contribution due to the L $\beta$ 1 line. Mark the Pb lines to see this.

#### Tips:

- To set markers, select the type using the droplist (bottom left of Spectrum window), then click to the **right** of the displayed area to position the high marker, and then to the **left** to set the low marker. Click, hold and drag the markers to position them.
- Many windows provide context-sensitive help tips in the text widget at the bottom of the window (e.g. *Spectrum Display*, *GeoPIXE Image* and *X-ray Spectrum Fit*).

- Move the cursor over a widget to see help on it.
- After resizing a window, click “Widen” again to view the View range expanded to the new window size.
- To switch to the “Identify” marker again, just click in the element list in the *X-ray Line Identification* window, or use the Dropdown on the left of the *Spectrum Display* window.
- To make the lines marked for an element show the effects of absorption (which changes the relative intensities of an element’s lines) select the appropriate filter (200 µm Al) and detector (CSIRO #34).
- This is most noticeable at low energy (e.g. mark Fe lines and change the “Filter” between 200 µm Al and 250 µm Be).



PIXE spectrum (green) showing Ge escapes (violet) and calculated pileup spectrum (red). The L lines of Pb are marked in orange.

## Example A2: Fitting a PIXE spectrum

- Spectrum fitting for quantitative analysis
- The *X-ray Identification*, *X-ray Spectrum Fit* and *Fit Results* windows.
- This example assumes an average matrix composition. See the later section “*Spectrum fitting using end-member analysis*” for methods to iterate on sample composition for more quantitative fitting results.

### Steps to try

1. Continue with previous PIXE spectrum (“Donut2x-2-whole.spec” from directory “**Demo/CSIRO/Donut2x**”).
2. Open the *X-ray Spectrum Fit* window using the menu “Window-> X-ray Spectrum Fit”.
3. Load the fit set-up file “null.pcm”, which has no elements selected.
  - a. An estimate of background will be drawn in violet on the spectrum.

### X-ray Peak Identification

4. Click on elements in the periodic table to select elements seen in spectrum (e.g. Fe, Sr, Sn and Ba).
  - a. Click once for K, twice for L shell, etc. – the colour of the button will change to reflect the of K lines (green), L lines (yellow), M lines (red), both K and L lines (light blue), etc.
  - b. Note how selecting Sr K lines shows the Ge escape peaks as well.
5. Use the *X-ray Identification* window to identify lines, or to mark lines for elements to help identify them.
  - c. Step through atomic number using the scroll arrows.

6. Select elements in the fit by either:
  - d. Left clicking on the element in the periodic table in the *X-ray Spectrum Fit* window, or
  - e. Right clicking on an element in the *X-ray Identification window*. (Note: this assumes K lines up to Ba, and L lines for La and heavier.)
7. Work through identifying all green peaks.
  - f. No, there is not Kr in this sample (collected in vacuum). So what is the 12.6 keV peak?

#### **X-ray Spectrum Fitting**

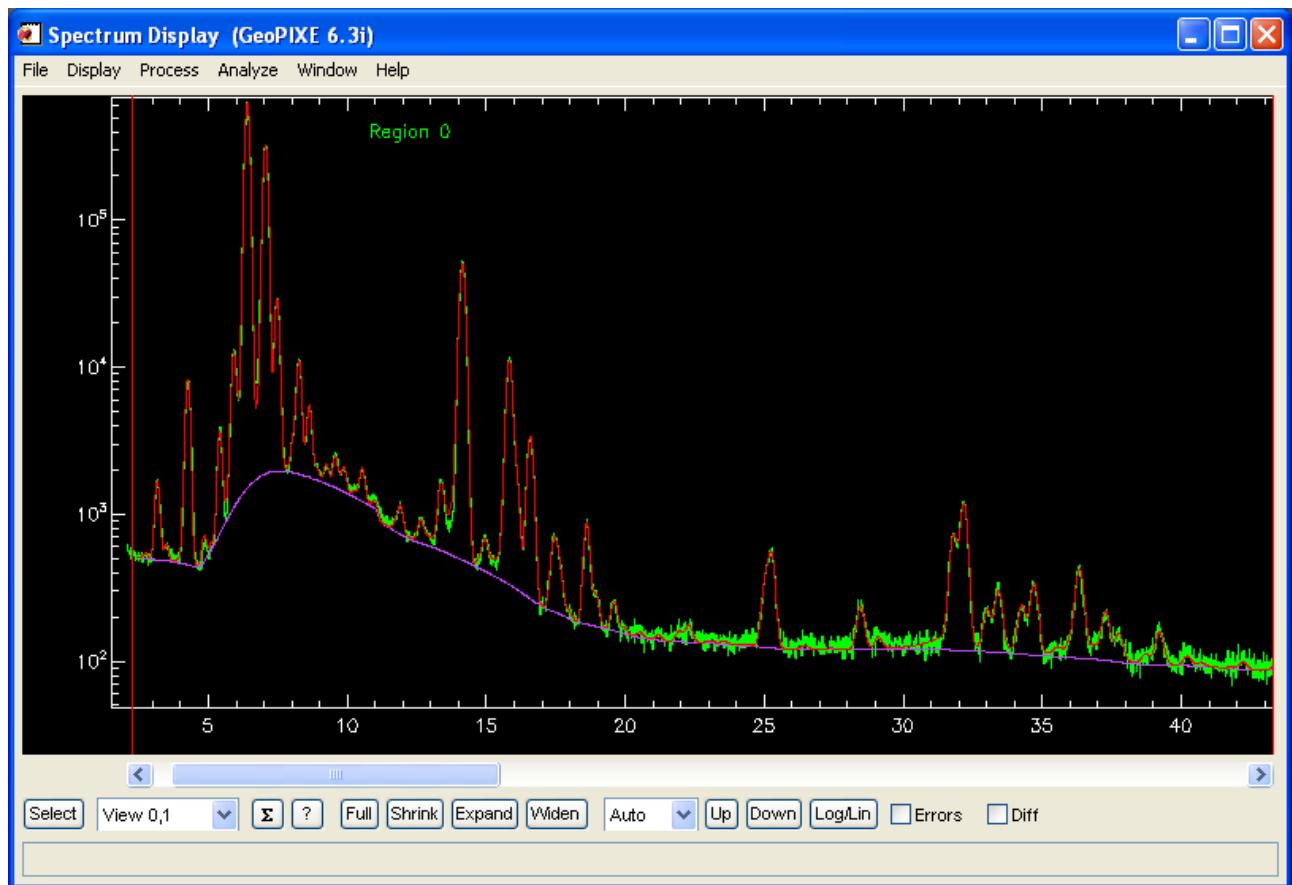
8. Once you have identified most peaks, fit the spectrum using the “Fit: One” button in the *X-ray Spectrum Fit* window.
9. Check out any green peaks that don’t seem to be explained by the elements you have selected.
  - a. Did you see Ga and Ge?
  - b. How’s the fit to the 12.6 keV peak?
    - i. Fe pileup at 12.8 keV helps, but is not quite right.
    - ii. What about Pb L lines? Click on Pb twice to select Pb L lines (now shown in yellow on the periodic table).
    - iii. Click on the “Fit: One” button again.
  - c. To see any elements you might have missed, load the set-up file “Donut2x.pcm”.

#### **Fit results**

10. Open the *Fit Results* window using the *Image Display* menu “Window->Fit Results”.
  - a. Results of the fit are shown in ppm (weight) or labelled as % (for weight %).
  - b. You might want to drag the window larger to see more columns.
11. View detection limits (ppm) by changing the drop-list from “Conc” to “MDL”.
  - a. e.g. Fe is 4.5 wt% and Br is 3.5 ppm with a MDL of 0.3 ppm.

#### **Tips:**

- Enable “Diff” on the *Spectrum Display* window to see the difference between the data and the background.
  - This can make weak lines more visible.
- To disable an element in the fit (i.e. do not fit it, but still calculate MDL), “right-click” on it with the mouse in the periodic table in the *X-ray Spectrum Fit* window (element colour darkens). To re-enable it, “left-click”.
- To clear an element entirely, “shift-left-click” on it, or keep left-clicking until it cycles back to grey (off).



*Full melt inclusion PIXE spectrum (green) showing fit (red) and background (violet)*

## Example A3: Fitting a SXRF spectrum, Yield modelling

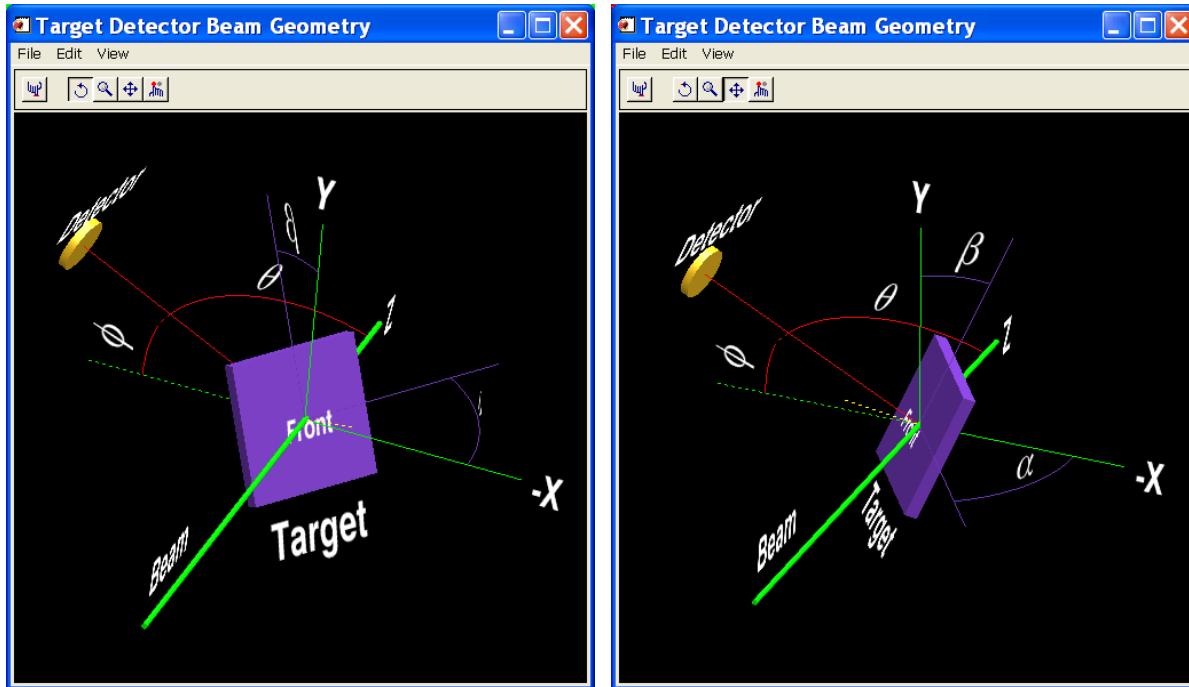
- Spectrum fitting for quantitative analysis
- SXRF yield modelling (simple detector)
- This example assumes an average matrix composition. See the later section “**Spectrum fitting using end-member analysis**” for methods to iterate on sample composition for more quantitative fitting results.

### Steps to try

1. Load the spectrum file “**Demo/APS/Pottery/sample24\_2D.spec**”.
2. Set the view around the peaks and “Widen” with “Auto” vertical scale.
3. Load the fit set-up file “**null.pcm**” from the Pottery directory into the *X-ray Spectrum Fit* window.
4. Identify peaks and select them for fitting.
  - a. Data used no additional filter, just a small “air” path, so select “none” or “Air 20 mm” filter in the *X-ray Line Identification* window to show correct relative intensities (makes a big difference for Ti and lighter elements).
  - b. Note the peaks on the right are due to Compton (inelastic) scattering (18.4 keV) and elastic scattering (19 keV).
  - c. This spectrum was collected with a Silicon-drift detector. Hence, it will only have (weak) Si-escape lines.
  - d. Did you notice the hidden peak at 13.0 keV? What’s this due to?
  - e. Load the file “**pottery.pcm**” to select all lines.

### Yield Calculation

5. Open the *Yield Calculation* window, by clicking on the “New” button adjacent to “Yields” on the *X-ray Spectrum Fit* window.
6. Load the yield set-up file “**pottery.lcm**”. Note the selection of “Photons (mono)” as “beam Particle”, an energy of 19 keV, the angles to the detector ( $90^\circ$ ) and the rotation of the target towards the detector ( $-45^\circ$ ).
  - a. To get help on widgets, move the cursor over each and look at the help in the bottom pane.
  - b. Note that the “Array” check-box is NOT set indicating that this is using a simple (small-solid-angle) detector model.
  - c. To view this beam-target-detector geometry, click on the “?” button next to the Phi detector angle widget. Left-click and drag to rotate the displayed 3D view of the sample detector geometry.
7. Experiment with other detector angles. Note that positive Theta is anti-clockwise as viewed from above. Use negative Theta for a detector on the other side. Use positive Alpha target tilt with a negative Theta (and negative Alpha with positive Theta), unless you want a transmission geometry.
  - d. Close the geometry window and click on “?” with each change to view the geometry (it does not automatically update).
  - e. Use the Rotation, Zoom, Translate modes on the geometry window to rotate and zoom the 3D view. Click and drag in the window to change the view for each mode.
8. Note that Theta is the detector angle in the plane, while Phi is the out-of-plane angle, measured as an azimuthal rotation about the beam (Z) axis. Alpha is the target rotation, about the vertical axis, while Beta is a tilt about an axis across the face of the target.
  - f. Use the context-sensitive help in the *PIXE/SXRF Yield Calculation* window for help on these angles, or experiment and use “?”
9. Try Theta=80, Phi=20, Alpha=55, Beta=25, for a more extreme example.
  - g. Close the geometry window and click “?” again to open it anew.
    - i. The meaning of the various angles is now clearer.
    - ii. Click and drag in the geometry window to rotate the 3D view.
  - h. Is there anything “wrong” with this geometry?
    - i. Now you have the detector looking at the BACK of the target.
    - ii. If transmission mode is not desired, set Alpha to -55 and check the geometry again.

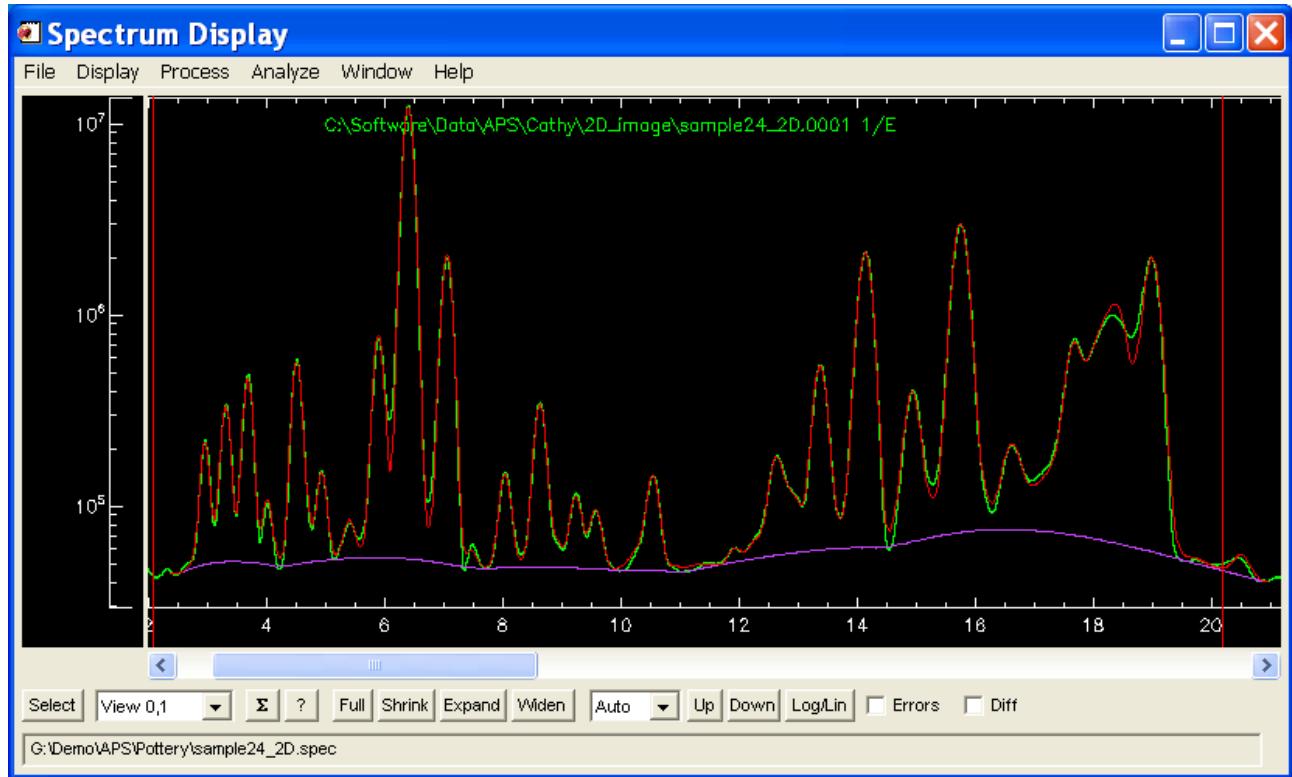


Beam-target-detector geometry for  $\alpha = 55^\circ$  (left; detector is ‘behind’ target) and  $\alpha = -55^\circ$  (right; probably the way it should be).

10. Re-load the yield set-up file “pottery.lcm” to restore geometry. Close any geometry windows.
11. To calculate yields (as well as relative intensities), including secondary fluorescence modelling, click “Calculate Yields”, and then “Save” when the requester appears (to “pottery.yield” in the same directory).
  - i. The yield results are transferred to the *X-ray Spectrum Fit* window.
12. Close the *Yield Calculation* window.

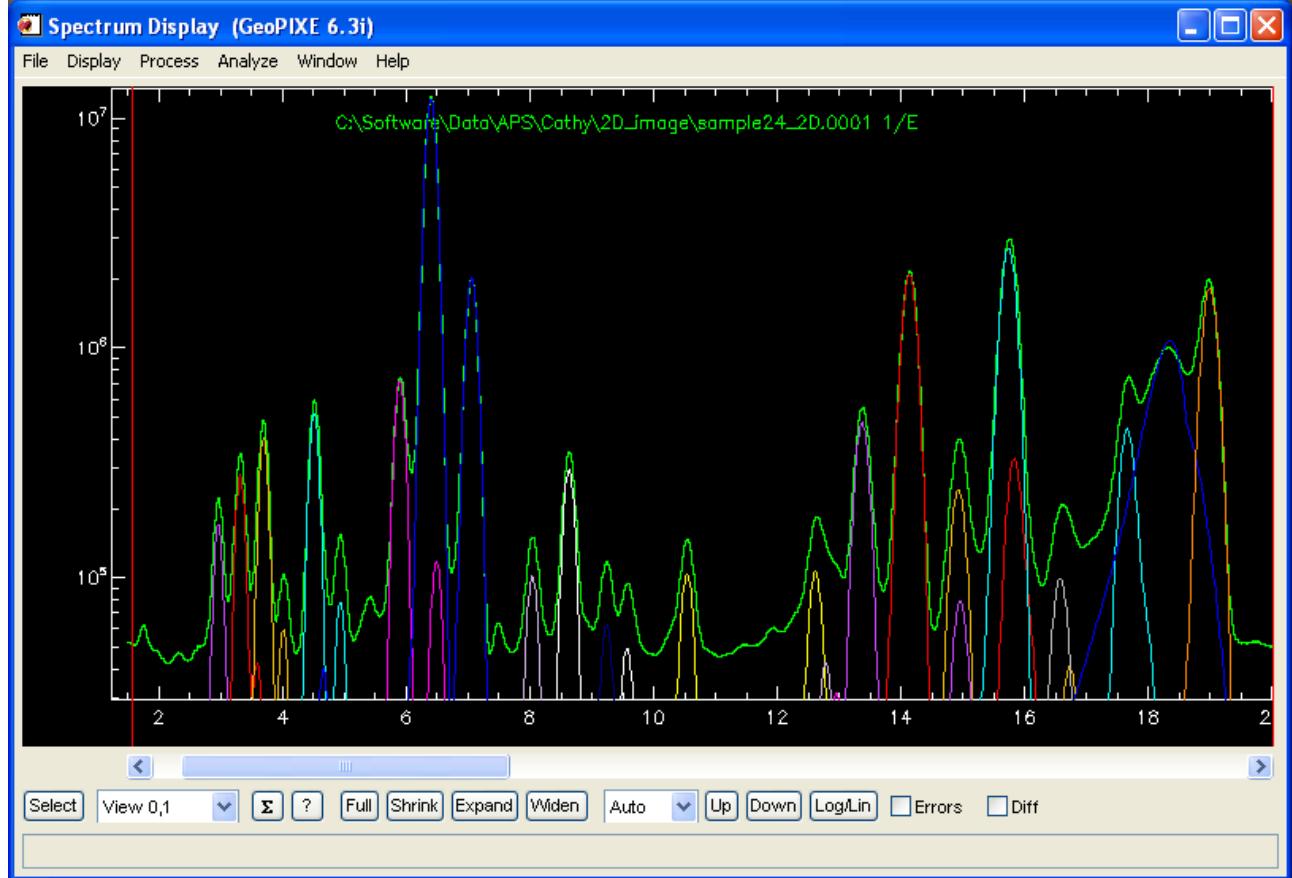
#### **X-ray Spectrum Fit**

13. The shape and position of the scatter peaks can vary and upset energy calibration fitting – let’s exclude them ...
14. Drag View markers to cover the range 1.4 to 17 keV (i.e. excluding the strong scatter peaks). Click on “Use View” in the *X-ray Spectrum Fit* window to make this range the fit range.
15. Enable “Cal On” in the *X-ray Spectrum Fit* window. Fit this by clicking on “Fit: One”.
  - a. This varies the energy cal parameters in the fit, but ignores the strong scatter peaks, which may upset the fit of energy calibration parameters due to Compton energy variation or if the beam energy is slightly in error.
16. Now extend the View range out to 20 keV, and disable “Cal On” and “FWHM”.
  - a. We now have included the scatter peaks, but with Cal and peak width parameters fixed in the fit.
17. Fit this by clicking on “Fit: One”.



Final fitted Pottery shard SXRF spectrum

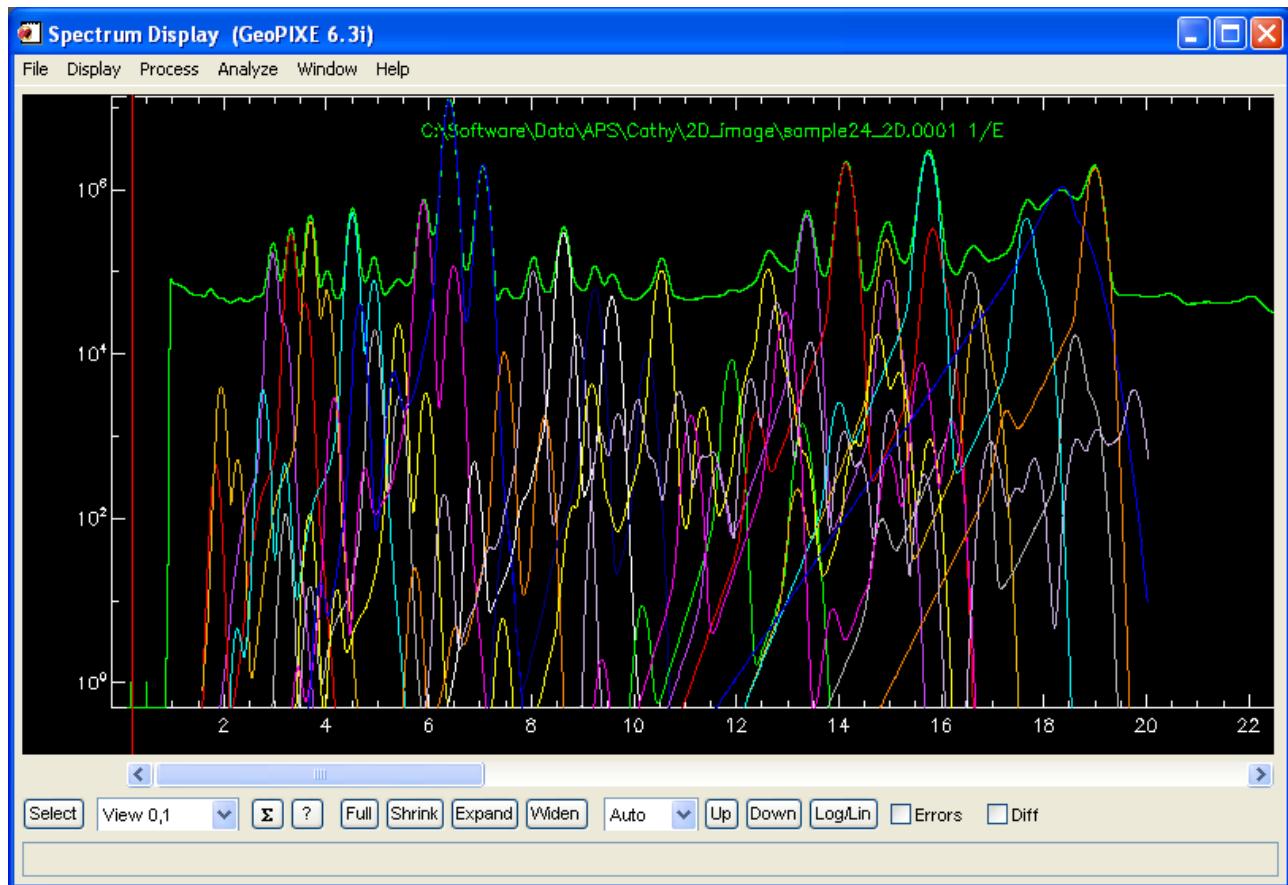
18. Note the new results in the *Fit Results* window (use the “Spectrum→Windows→Fit Results” menu”). View concentrations or detection limits (ppm) by changing the drop-list to “Conc” or “MDL”.



Pottery spectrum showing fitted elemental (and scattering) spectral components

**Tips:**

- To use the View markers, change to them using the Marker dropdown (bottom left on spectrum window).
- To overlay the elemental components of the fit, click on “Generate DA matrix”, use “Normal single energy DA matrix” mode and save to a temporary file (e.g. “temp.damx”), and then click on the “P” button (next to “Export”).
  - Scroll the X axis or “Shrink” the view to see more of the spectrum and the large number of spectral components.



Expanded view to see spectral components in all their glory!

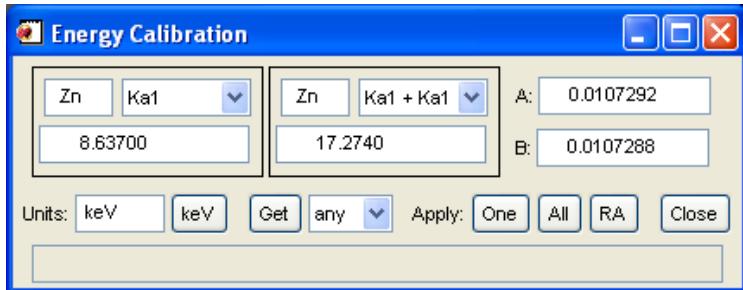
## Example A4: Importing an old XFM Vortex SXRF spectrum

- Converting an old Vortex MDA file to LST format for GeoPIXE use.
- Importing Vortex spectral image data collected at XFM

**Steps to try**

1. Convert the APS-style MDA data file to a list-mode format that GeoPIXE can use, using the Spectrum Display menu “File→Convert→APS MDA to LST”, and select file “Demo\AS\Pileup\SR05ID01IOC51\_0059.mda”, which is for a sphalerite sample (ZnS) collected at 10 keV.
2. After some pre-processing it will prompt for Ion Chamber details. Enter the following (which establish the Epics PV used for the upstream ion chamber (IC), its preamp sensitivity and Live-time counting mode):
  - a. Scaler IC channel = SR05ID01IOC51:scaler1.S4
  - b. Preamp sensitivity = 10
  - c. Preamp units = nA/V
  - d. Live time = SR05ID01IOC52:mca1.ELTM
3. After the LST file is built, import it into GeoPIXE using the menu “File→Import→Spectra” and select:
  - a. “Synchrotron SXRF”
  - b. “APS Data Cube” source device
  - c. “Extract from APS/PC LST” operation.
4. Select the file “SR05ID01IOC51\_0059.lst” created above and direct output to directory “Demo\AS\Pileup\”.
5. In the *Flux PV Select* pop-up, select “Indirect using Ion Chamber (no EPICS PV)” mode, and enter a conversion factor of 7.2e-12.

6. Open the *Spectrum Select* window (“Select” button) and notice that we have 3 spectra (E, X, Y). Click on “Delete” with “all XY and T selected” to delete the X,Y spectra.
7. Calibrate the E spectrum by opening the *Calibration* window (menu “Display→Calibrate Energy”) and setting the markers on the largest peak on the right, which is Zn pileup, and the Zn Ka, which is the largest peak in the spectrum.
8. In the Calibration window, select “Zn Ka1” and “Zn Ka1 + Ka1” for the two markers and Apply: “One”.



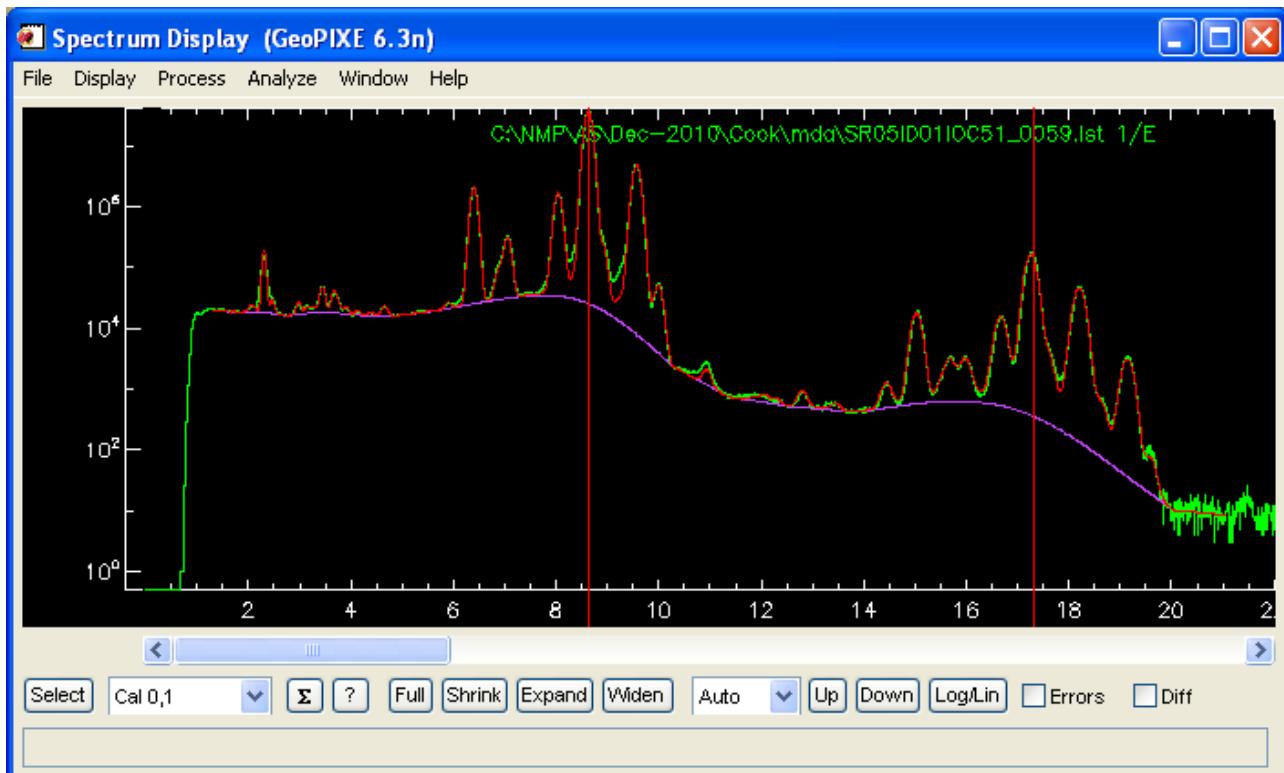
9. Save the energy calibration to file “SR05ID01IOC51\_0059-cal.spec”.

## Example A5: Fitting a XFM Vortex SXRF spectrum, Pileup

- Spectrum fitting for quantitative analysis
- Large pile-up contributions in the spectrum

### Steps to try

1. Load the spectrum file “Demo/AS/pileup/SR05ID01IOC51\_0059-cal.spec” from above example.
2. Open the *X-ray Spectrum Fit* window and select these details:
  - a. Load PCM file “Demo/AS/pileup/null-10keV.pcm”.
  - b. View range (set this using the View markers) 1.3 to 21 keV.
  - c. Detector = “AS Vortex EM SDD”.
  - d. Filter = “Air 10 mm”.
  - e. Yields = “sphalerite-10keV.yield”.
  - f. Select elements you observe (do you expect element peaks at energies above 10 keV?).
    - i. Start with just Fe, Cu and Zn.
3. Fit the spectrum.
  - a. Click “Fit One” to fit the spectrum.
  - b. Where did all those peaks above 10 keV come from?
    - i. Display calculated pileup spectrum using menu “Display→Pileup→from single spectrum”.
    - ii. (See set-up file “sphalerite-In3-2-Sn-10keV.pcm” for the full set of elements)



Fit to sphalerite spectrum (courtesy Nigel Cook, U. Adelaide, collect at 10 keV. Note strong pile-up peaks from 11-21.5 keV formed by numerous pair-wise random additions of the energies of the strong peaks between 2 and 10 keV.

## Example A6: Fitting a XFM Vortex SXRF spectrum, Flux Calibration

- Importing Vortex spectral image data collected at XFM and converted to LST.
- Yield calculation for the NIST 1832 glass film.
- Spectrum fitting to determine the “conversion” factor for quantitative analysis.

### Steps to try

- Import the LST file into GeoPIXE using the menu “File→Import→Spectra” and select:
  - “Synchrotron SXRF”
  - “APS Data Cube” source device
  - “Extract from APS/PC LST” operation.
- Select the file “SR05ID01IOC51\_0014.lst” created above and direct output to directory “Demo\AS\Pileup\”.
- Enter the old conversion factor for now: 7.2e-12.
  - The old one was determined at 10 keV. We now need to determine a new factor for this run at 12.8 keV.
- Delete X,Y spectra in *Spectrum Display* window, as above.
- Calibrate the E spectrum by opening the *Calibration* window or use the menu “Display→Get All Energy Cals” and use the energy calibration of the spectrum from the previous example.
- Open the *X-ray Spectrum Fit* window and select these details:
  - (Start with set-up file “null-12-8.pcm”)
  - View range (set this using the View markers) 1.3 to 14 keV.
  - Detector = “AS Vortex EM SDD”.
  - Filter = “Air 10 mm”.
  - Select elements you observe.
- Open the *X-ray Yield Calculation* window, and enter these details for the **NIST 1832** glass film:
  - Energy = 12.8 keV
  - Detector geometry for Vortex on the XFM nanoprobe = Theta = 90, alpha = -45
  - Layer 1 setup (see NIST data sheet next page):
    - 0.55 microns, with density = 2.742, or
    - Use mg/cm<sup>2</sup> thickness units and a thickness of 0.1508 mg/cm<sup>2</sup> (**1.517 mg / 10.06 cm<sup>2</sup>**).

- iii. Formula = Na6.8Al9.1Si21.9Ca12.3V2.8Mn2.8Co0.6Cu1.5Fe0.4O41.8
    - 1. These are “Weight %” proportions.
  - d. Calculate the yields and save them in file: “NIST-1832-12-8keV.yield”.
8. Fit the spectrum.
- a. See *Fit results* window table.
  - b. It shows Mn = 1.10%. It should be 2.80%, so change the “conversion” factor calibration
    - i. To access this calibration click on the “?” next to the Q (charge) entry field on the *X-ray Spectrum Fit* window.
  - c. Change the “Conv” value (conversion from flux count to “Charge”) and re-fit, until you get 2.80% for Mn.
    - i. Or alternatively change the Q (charge) value and re-fit until Mn is correct.
    - ii. Then click on “?” to see the new “Conv” factor.
    - iii. The final “Conv” factor is around 2.79e-12.

*“Conv” value is a conversion from Ion Chamber “flux count” to “Charge”. Charge is a representation of the number of photons. Charge is in units of  $\mu\text{C}$ , which represents  $6.242 \cdot 10^{12}$  photons.*

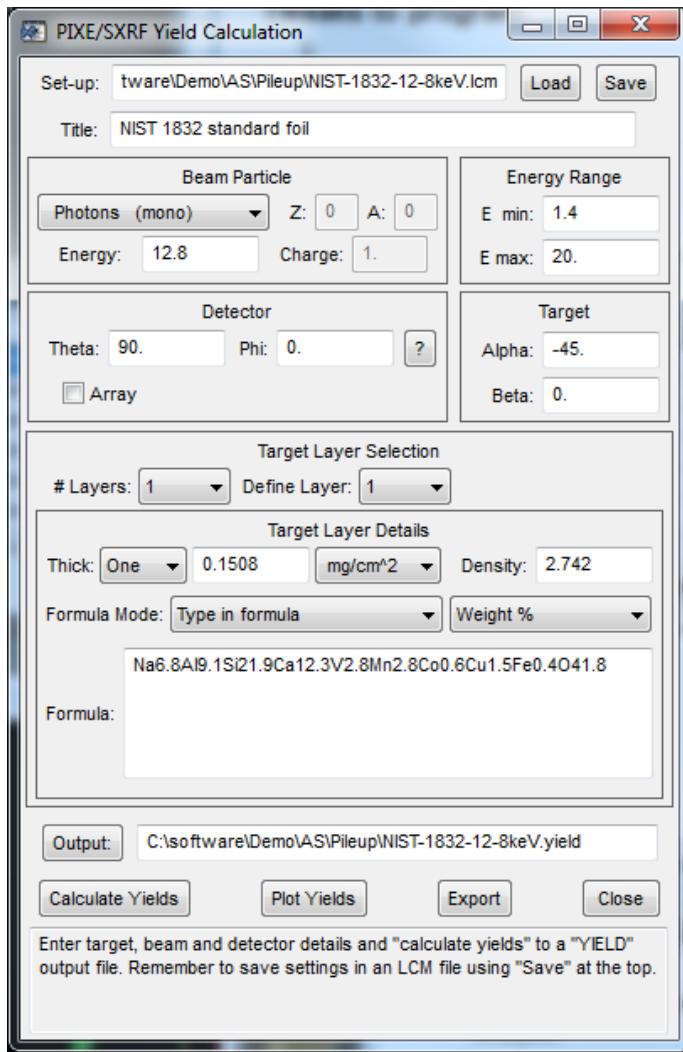
Element	Certified <sup>1</sup> Value, (% by wt.)	Estimated Uncertainty <sup>2</sup> (% by wt.)
Sodium	( 6.8 %) <sup>3</sup>	
Aluminum	9.11	± 0.6
Silicon	21.89	± 0.7
Calcium	12.28	± 0.8
Vanadium	2.78	± 0.3
Manganese	2.80	± 0.3
Cobalt	0.62	± 0.04
Copper	1.49	± 0.1
Argon	( 1.0 %) <sup>3</sup>	
Iron	( 0.4 %) <sup>3</sup>	

<sup>1</sup>The certified value listed for an element is based on the results of NBS-CAC and cooperative laboratory analyses. The values from cooperating laboratories were averaged for each element. The results were then averaged with the respective mean values from the NBS-CAC laboratories to obtain the certified values.

<sup>2</sup>The estimated uncertainty listed for an element is based on an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability.

<sup>3</sup>Values in parentheses are not certified, but are given for information purposes only.

**NOTE:** To convert the certified values from % by weight to micrograms per square centimeter, use the following expression:  $(\%) \text{ by wt.} \times 10^{-2} \times \text{film wt. } (\mu\text{g}) / 10.06 \text{ cm}^2$ . The uncertainties in the film weight and area are small compared to the estimated uncertainty in the certified values and are, therefore, not included in this expression.



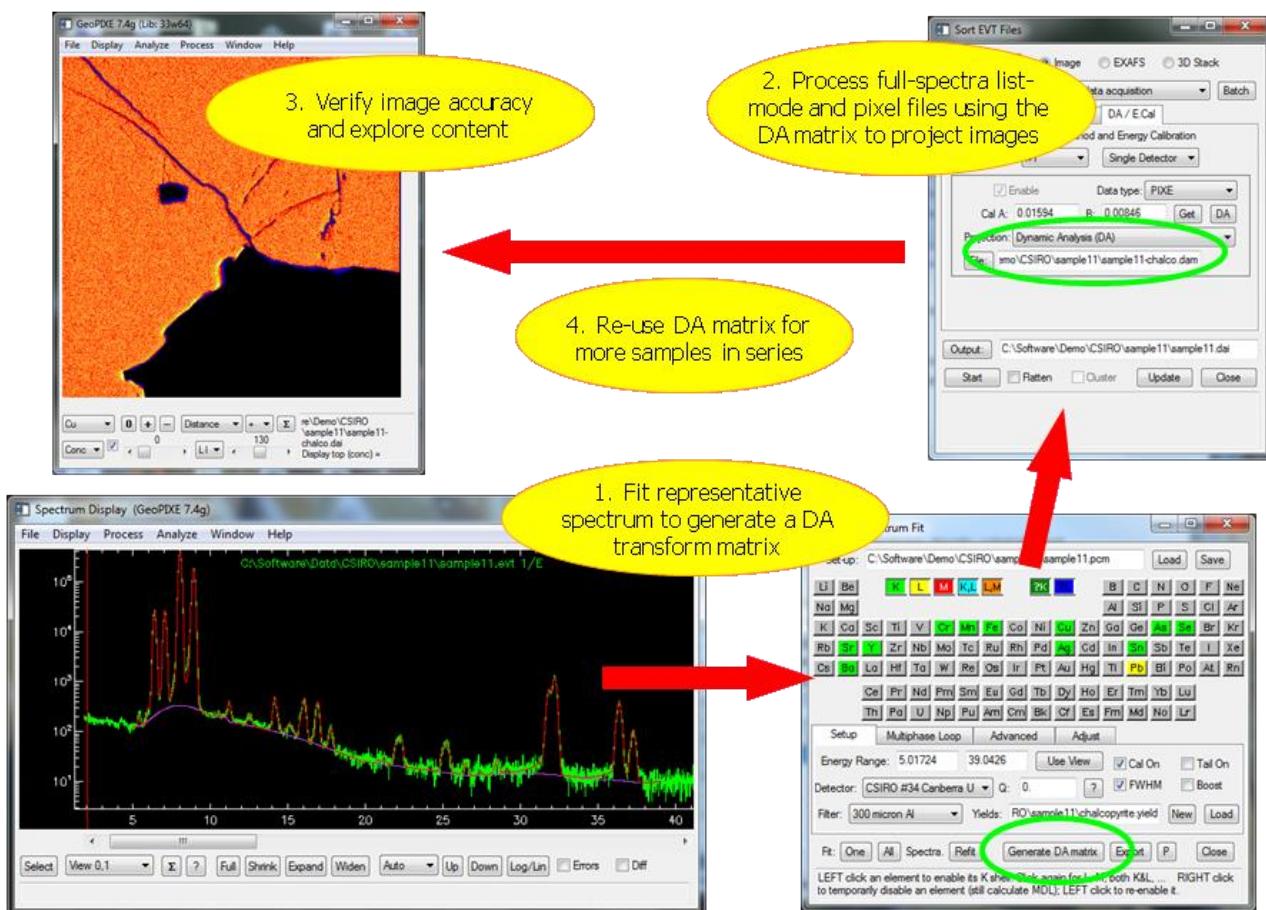
9. The results versus those expected:
  - a. NIST 1832 certified values are:
    - i. **Si = 21.9 wt%**
    - ii. Ca = 12.3 wt%
    - iii. V = 2.78 wt%
    - iv. Mn = 2.80 wt%**
    - v. Co = 0.62 wt%
    - vi. Cu = 1.49 wt%
  - b. The results for the heavier elements look good (V, Mn, Co, Cu), but Si and Ca are a little low.
    - i. What is not right with our assumptions or estimates of the detector and experimental set-up?
    - ii. Is 10 mm the correct distance for the detector (and hence absorption air path)?
  - c. Try Air 15 mm:
    - i. How do the results look now?
  - d. The final charge should be 0.00174  $\mu$ C and Conv = 2.81e-12.
    - i. This conversion is good for an energy of 12.8 keV
      1. With the present experimental set-up
      2. Assuming a 15 mm Air absorber.
      3. It differs quite a lot from the 10 keV “conversion” factor.

## B. Dynamic Analysis Imaging – the GeoPIXE Work Flow

### Example B1: SXRF DA Imaging (a simple detector array)

- Spectrum fitting for quantitative analysis and imaging
- SXRF specific issues in fitting
- SXRF yield modelling for a detector array
- Building the Dynamic Analysis (DA) transform
- Processing list-mode data using DA to project images
- Viewing elemental images

See also examples discussed in detail in the section “Raw SXRF Data to Elemental Images” in the “GeoPIXE Analysis Scenarios and Data Flow” chapter of the GeoPIXE manual (and see Help menu).



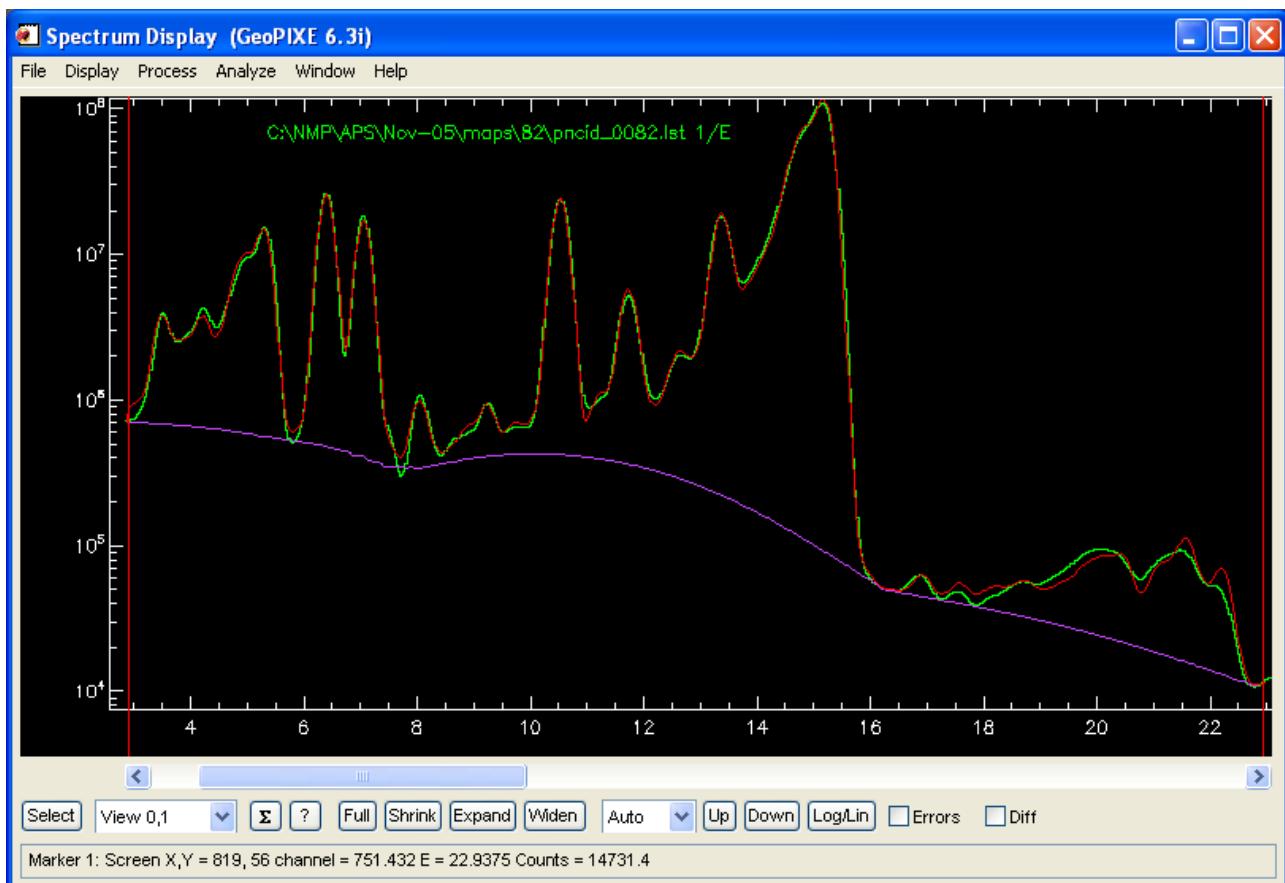
Typical work-flow in GeoPIXE to (i) fit a representative spectrum, (ii) generate a DA matrix, (iii) sort raw data into images, and (iv) explore images, and consider quality control, such as identifying missing elements.

#### Steps to try

1. Load the spectrum file “pncid\_0082.spec” from directory “**Demo/APS/Regolith-82**”.
  - a. This file contains 7 detector spectra from a small array.
  - b. To step through these, open the *Spectrum Select* window by clicking on “Select” in *Spectrum Display*.
  - c. Click on “Prev” and “Next” to step through the spectra. Or, click on “One” to display just a selected one.
2. Add the spectra from the 7 detectors in this simple Ge array by first displaying all spectra (click “All” on *Spectrum Select* window) and then using the *Spectrum Display* menu “Process→Add (re-map cal)”, which adds the spectra after mapping them to a common energy calibration.
3. Set the View range to ~3-23 keV and click on “Widen”.
  - a. The spectrum has only fair resolution (~250 eV) and pileup seen to the right of the elastic peak at 15.2 keV.
  - b. Note the large Ge escape peaks at 3.5-5.5 keV.

### Adjusting SXRF specific parameters in the fit

4. Load the fit set-up file “hematite.pem” into *X-ray Spectrum Fit*, and fit the spectrum.
  - a. Set View to 3-14 keV (to exclude the scatter peaks).
  - b. Enable “Cal On” and “FWHM” in the fit and fit the spectrum (“Fit One”).
  - c. Now disable “Cal On” and “FWHM” and set View back to 3-23 keV.
5. The fit looks a bit rough. The Compton tail still needs some adjustment (exponential tail needs to be longer).
6. Try adjusting these fitting parameters (select the “Advanced/SXRF “tab):
  - d. “Shift” adjusts the position of the Compton peak (simulating changes in mean electron momentum) and “Spread” changes its width. Click on the arrows in the SXRF panel to make small changes.
  - e. “Shift corrects the Compton energy loss, and so has a sense opposite of what seems natural on the GUI (i.e. clicking the left arrow move the peak to the right, and *vice versa* for the other arrow button).
  - f. Adjust the parameters “Compton Tail” → 1.0 and “Tail length” → 1.0. Fit again; that’s better?
    - i. Better still better is “Compton Tail” → 0.4 and “Tail length” → 1.4.
  - g. Adjust the width and position of the Compton peak using the parameters “Spread” → 1.2 and “Shift”, which effectively tune the electron momentum distribution. Fit again; that’s better?
    - i. Better still is “Spread” → 1.5 and “Shift” → -0.005.
7. Fit the sum spectrum at each step to see how the fit is improving.
8. Save the fit in file “pncid\_0082-fit.spec”.



Fitted spectrum for pncid\_0082, courtesy Rob Hough, CSIRO.

### Tips:

- On Linux, click to the left/right beside the slider knob to make small changes to the value.
- Include the pileup peaks (seen above the elastic peak) in the fit View range so that the fit will tie down the pile-up ratio in the spectrum, which is needed for the correction of concentrations for pileup losses.

### Yield Calculation using detector arrays

9. Open the *PIXE/SXRF Yield Calculation* window again, using the “New” button next to Yields”, and open the set-up file “hematite.lcm” from directory “**Demo/APS/Regolith-82**”.
10. View the 3D experiment geometry by clicking on “?” next to the “Phi text box. It shows a single ‘generic’ detector at the detector position (at angles Theta, Phi) representing a simple (small solid-angle) detector. Close this pop-up window.
11. Now enable array mode by checking the “Array” check-box.

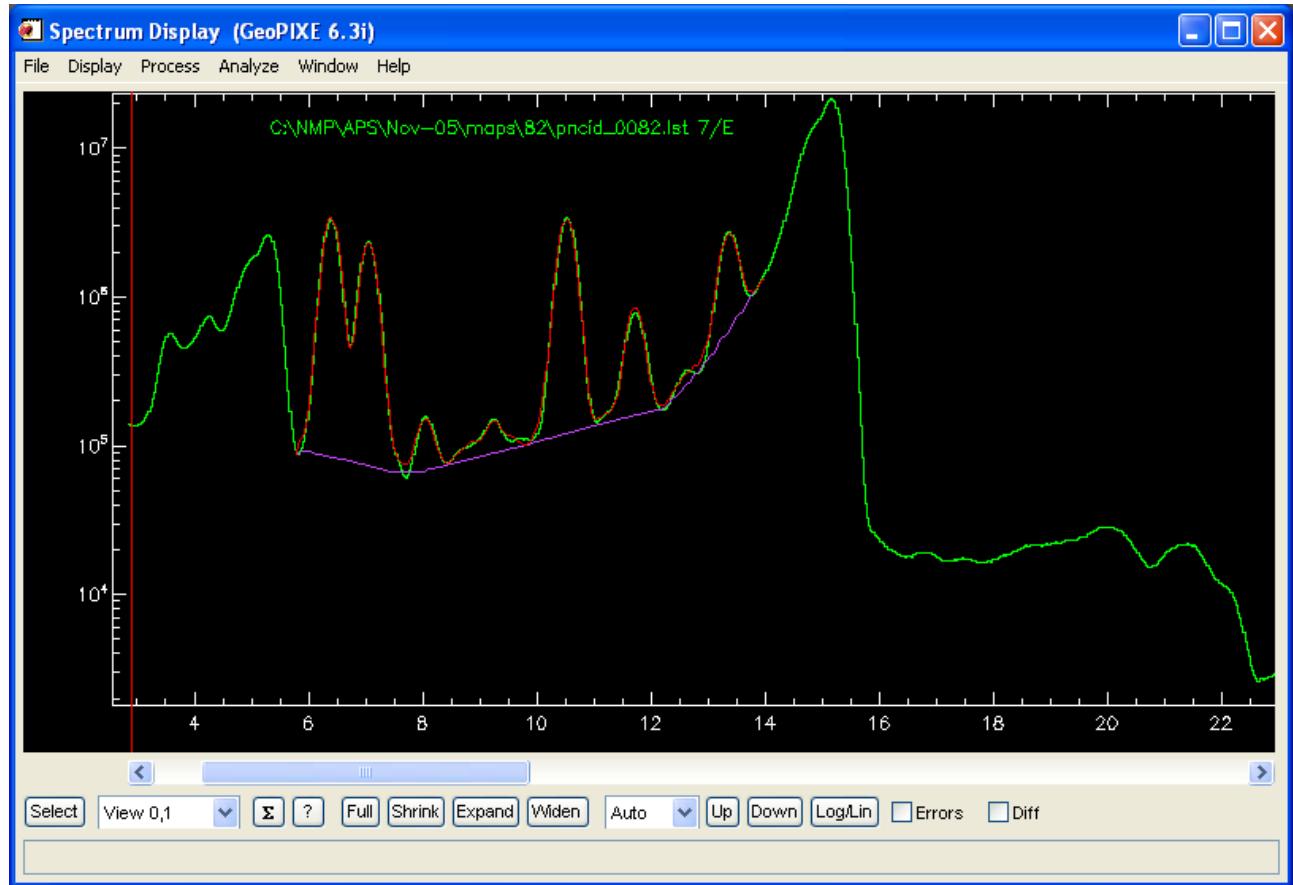
- a. This brings up a new dropdown to select array detectors.
  - b. Only full array detectors are listed in the dropdown (as opposed to simple arrays where all detectors are assumed to be identical).
  - c. Select “PNC-CAT array 7-element”.
12. Now click on “?” again. Now it shows a 7-element array at the correct distance, as set-up in this detector file. Rotate and zoom the view using modes and controls in the pop-up window.
- d. Zoom in to see the numbers on each detector (use the zoom and pan controls).
13. Click on “Calculate Yields”:
- e. This calculates yields for all elements (with lines in the E min, E max range) and relative sensitivity factors across all detectors in the array.
  - f. The calculation now has two passes:
    - i. Normal yield calculation of ‘generic’ yields, including secondary fluorescence, and
    - ii. Calculation of yields for each detector element relative to the central detector.
  - g. Save the calculation to “hematite-array.yield”, save the window set-up to “hematite-array.lcm” and close the *PIXE/SXRF Yield Calculation* window.
14. Notice that the detector specification back in the *X-ray Spectrum Fit* window is ghosted out now. Since this is now using an array detector, the details of the detector are embedded in the yield calculation and cannot be changed independently, as is the case for a simple generic detector.

#### ***Spectrum fitting and generating a Dynamic Analysis (DA) matrix file***

15. When the fit looks good ...
  - a. You might need to re-fit with “Cal On” and “FWHM” using the narrow View range again.
16. Save the fit set-up as “hematite-array.pcm”.
17. Click on “Generate DA matrix”, select “Normal single energy DA matrix” to build a DA matrix file.
  - b. Save it as “hematite-array.damx”.
  - c. This will be used later for generating images from this data-set.

#### ***Detector energy calibrations across an array***

18. Reload the spectrum file “pncid\_0082.spec” containing all 7 detector spectra.
19. Clear the Fit Results window.
20. Load the fit set-up file “hematite-array-cal.pcm”,
  - a. Which limits the fit to just the central peaks (not the scatter or its escape peaks) and enables the energy Cal parameters in the fit.
21. Now fit all spectra (one for each detector) by clicking on “Fit: All”. After 7 spectra fits, check the results by stepping through the spectra using “Next” in *Spectrum Select*.
  - b. If any detector fits look bad, you may need to re-fit that one.
  - c. Save the resulting spectra (with fit overlays, FWHM measures and revised Cal parameters) to file “pncid\_0082-cal.spec”.
  - d. Later you’ll use this file to provide an accurate energy calibration for each detector channel during imaging.

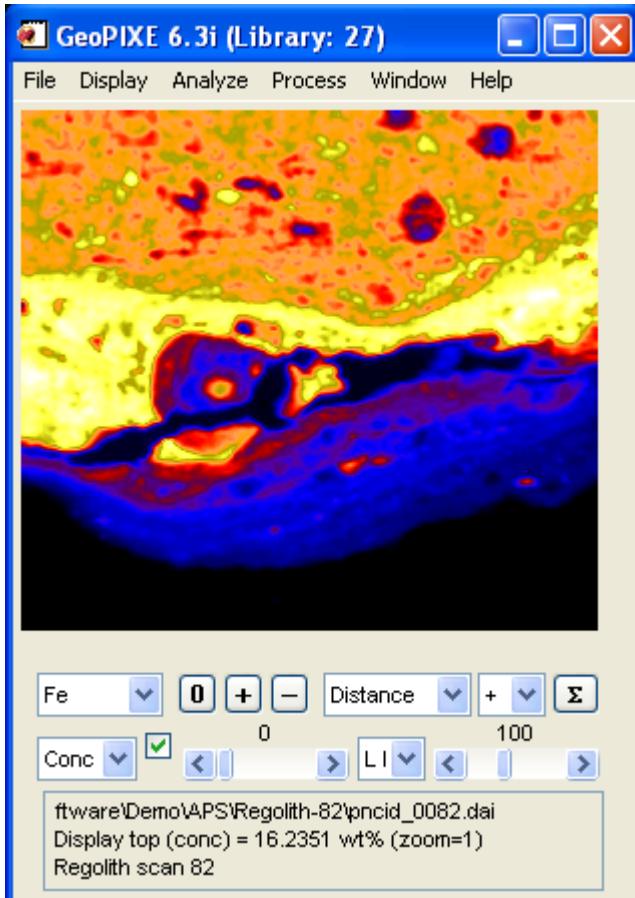


Detector channel #7 and its fit used to pin down the energy calibration coefficients for imaging

#### **Processing the data from all detectors in the array to project images**

22. Go to the *Sort EVT* window (if need be, open it from the *Image Display Windows* menu) and set the following settings:
  - a. Select “APS Data Cube - LST” device data file format.
  - b. “Files” tab
    - i. Select “LST file” to load the raw list-mode data from file: “Demo/APS/Regolith-82/raw/pncid\_0082.lst”.
    1. Answer “No” to the energy calibration pop-up question.
  - c. “Flux” tab
    - i. Select mode “Indirect using ion chamber (no EPICS PV)”.
    - ii. Enter a calibration “conversion factor” of 4.51e-8 (conversion from flux count to “Charge” – charge in  $\mu\text{C}$ ; 1  $\mu\text{C}$  represents  $6.242 \times 10^{12}$  photons).
    - iii. Leave the Charge box zero for now (it will be calculated during processing).
  - d. “DA/ E.Cal” tab
    - i. Set the detector droplist to “Detector Array”.
    - ii. Set the Data Type to “SXRF”.
    - iii. Get all energy calibrations from file “pncid\_0082-cal.spec” using the “Get” button.
      1. This also enables only the detector channels found in the spec file.
      2. Note that detector #1 is not enabled (missing from the cal spectra). Select the “channel” droplist and step through detectors to see their Cal parameters and which are enabled.
    - iv. Load the DA matrix file “hematite-array.damx” using the “File” button under the “Projection droplist” (which should be set to “Dynamic Analysis (DA)”).
  - e. Change the “Output” to “pncid\_0082.dai” in the “Demo/APS/Regolith-82/” directory, NOT the raw data dir.
23. Start the sort, by clicking on “Start” (this will take about 30 seconds to process the 1.6 GB of data)
  - f. Once finished the images will appear in the *Image Display* window.
    - i. The “Charge” field (Flux panel) should now show 1464.
    - ii. There is one image per element selected in the fit, plus “Back” to indicate underlying background intensity variation and “Sum” for basic pileup. Use the droplist on the left to select element images.
    - iii. The final images will be corrected for the relative sensitivity of the enabled detectors so that we get meaningful image concentrations regardless of which, and how many, detectors are enabled.

- iv. The variance images are also corrected for detector number. However, more detectors will result in lower variance.

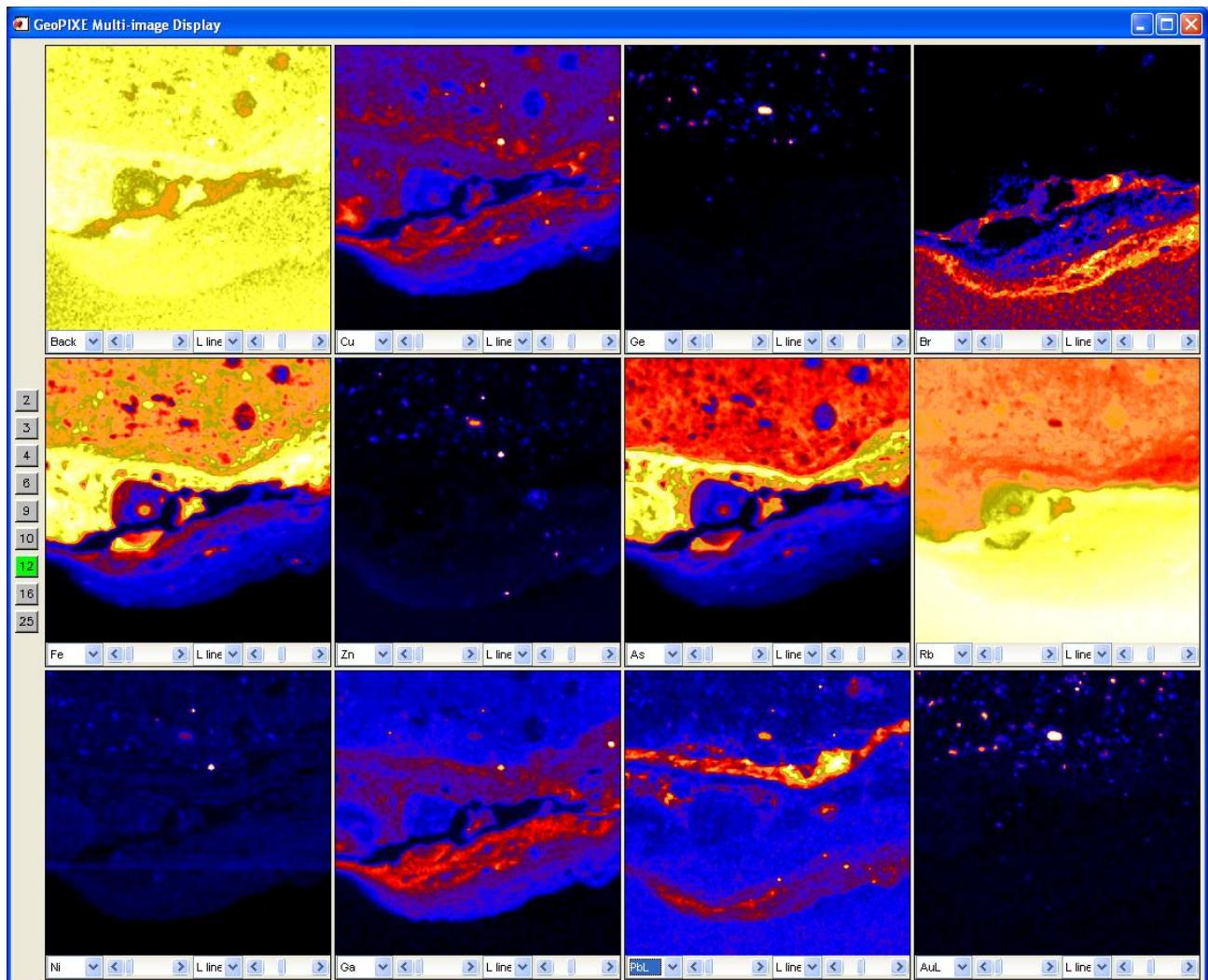


*Fe image zoomed out once (use “+” button and drag resize the window)*

### **Viewing Images**

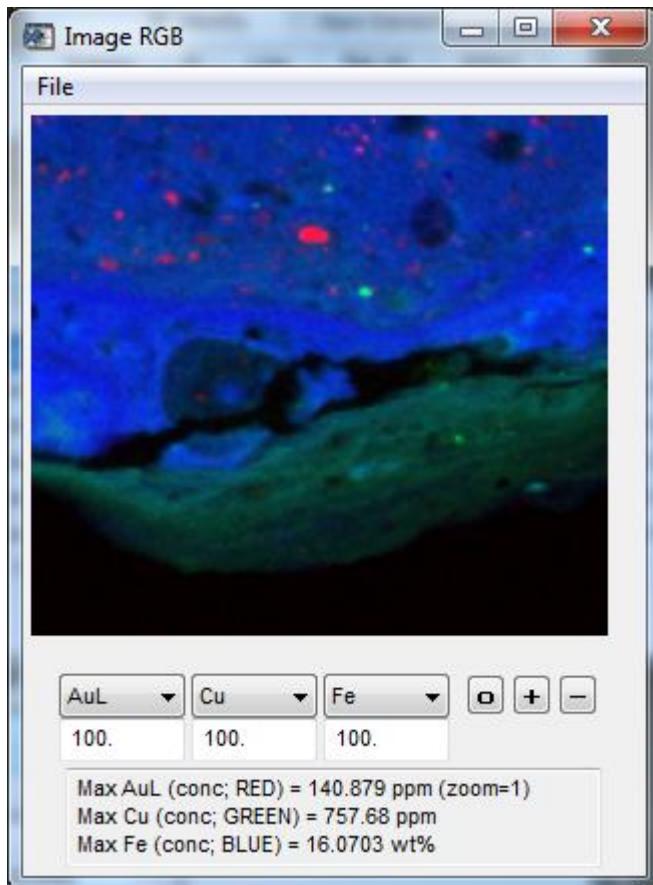
24. Zoom out the image (1 click on “+”, and resize the *GeoPIXE Image* window).
25. Step through the images using the dropdown on the left.
26. Use the bottom sliders to adjust the minimum (left slider) and maximum (right slider) concentrations mapped into the colour map.
  - a. Use the middle dropdown on the bottom row to change the concentration scale from “Linear” to “SQRT” or “Log”.
    - i. “SQRT” provides an easy way to compress the dynamic range of an image to see more features at very different concentrations.
  - b. Experiment with these and changes to the minimum and maximum sliders.
  - c. The check-box on the left enables interpolation between pixels when the image is zoomed in (zoom>0).
27. Select the Au image. It shows a strong hot-spot (and many minor ones).
  - d. Is this really gold? This is explored below.
28. Open a second *Image* window using the menu “Display→ Clone→1 clone”. Use this to compare images.
  - e. Select region shapes using the dropdown on the top row; drag out the shape, and adjust using the handles.
    - i. For example, select “Ellipse” and click and drag to set initial shape, then click and drag control points.
  - f. The shape will appear on all clone images. Use this to associate features on multiple images.

29. Open the *Multi Image* window using the menu “Display→Multi” on the *Image* window.
- These have duplicates of the low and high sliders and the Z-scale mode dropdown, as on the *Image* windows; these work in tandem with those on the *Image* window.
  - The left side has a series of buttons to select how many image frames to show in the *Multi Image* display.



*Multi Image* window display for Regolith sample 82 (courtesy Rob Hough, CSIRO).

30. Open an RGB display window (“Window→3 element RGB images” menu) and select the elements “AuL”, “Cu” and “Fe” on the Red, Green, Blue dropdown lists.
- This combines these elements into a 24-bit colour RGB composite image.
  - Adjust the intensity of each element using the controls back on the *Image* window.
31. Export the RGB image using the “File→Export” menu.
- Note that this exports the image as displayed (at the **current zoom setting**).



*RGB composite of Au (red), Cu (green), Fe (blue); the numerical field duplicates the max display intensity control from the image window.*

**Tips:**

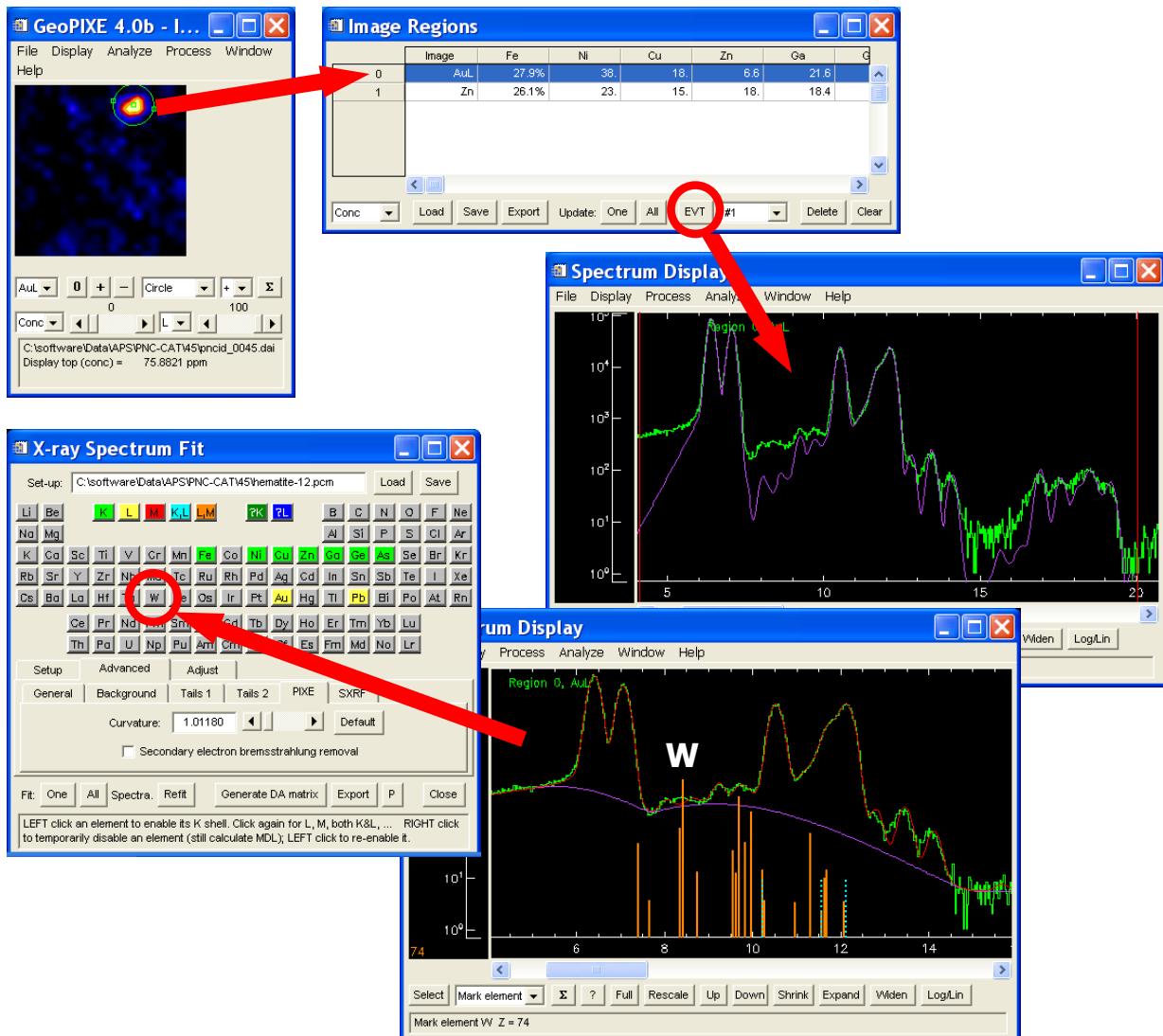
- Adjust the Max slider on one of the Image windows for each of these elements. This is linked to the RGB window and affects the intensity of the colours. You can also type in new max values in the numerical fields on the *RGB Window*.
- Next time you use Sort EVT you can re-load the saved image file (.DAI) as a template using the “From DAI” button. This template can be used for new samples or sample regions by selecting new input data file-names, and then changing any varied parameters.
- For arrays, we need to set a different Cal for each detector. This is done by fitting all spectra, deleting any bad detectors (delete their spectra), and saving the file (usually named with an “-cal.spec” appended). Then use “Get” in *Sort EVT* to
  - Load the Cal for each detector in this cal spectrum file,
  - Enable ONLY those good detectors found in the file.

## Example B2: Verifying image features

*Extracting spectra from regions of an image – validation and quality control*

- Defining regions on images and extracting quantitative estimates directly from images
- Sorting the raw data to extract spectra from these regions
- Looking for errors and missing elements
- Adding missing elements and regeneration of images

*There is another example in this theme (using PIXE data) in section “Verification of Images using Regions” or the “GeoPIXE Analysis Scenarios and Data Flow” chapter of the GeoPIXE manual.*



Illustrates the process of verifying image data: (i) Position shapes over strong features in image, (ii) extract spectra from these regions, (iii) identify any missing elements in these spectra, (iv) add any missing elements to fit and build new DA matrix, and (v) re-image using the new DA matrix.

#### Steps to try

1. Reload the image projected above “pncid\_0082.dai” and zoom it out.
2. Select the “Ellipse” shape on the “select area/analyze type” dropdown on the *GeoPIXE Image* window, and drag out a small ellipse around the Au hot-spot on the Au image projected above.
  - a. Hover cursor over widgets to get help on what each one does.
3. Click on “Σ” (may appear as “S”), which adds an extracted analysis row to the *Image Regions* window for the selected region.
  - a. Open the *Image Regions* window using the menu “Windows→Image Regions”.
  - b. This suggests an average Au in this region (depending on size) of ~100 ppm or more.
4. Perhaps do the same for any other hotspots you see for other elements
  - a. e.g. “Back”, Au and Ni, Cu, Zn have a few hot-spots too to investigate.
  - b. Try a “Circle” shape for these; use the central handle to move the circle and the handles on the diameter to resize it.
5. Save these regions with the name “pncid\_0082-q1.region”, which is the default.

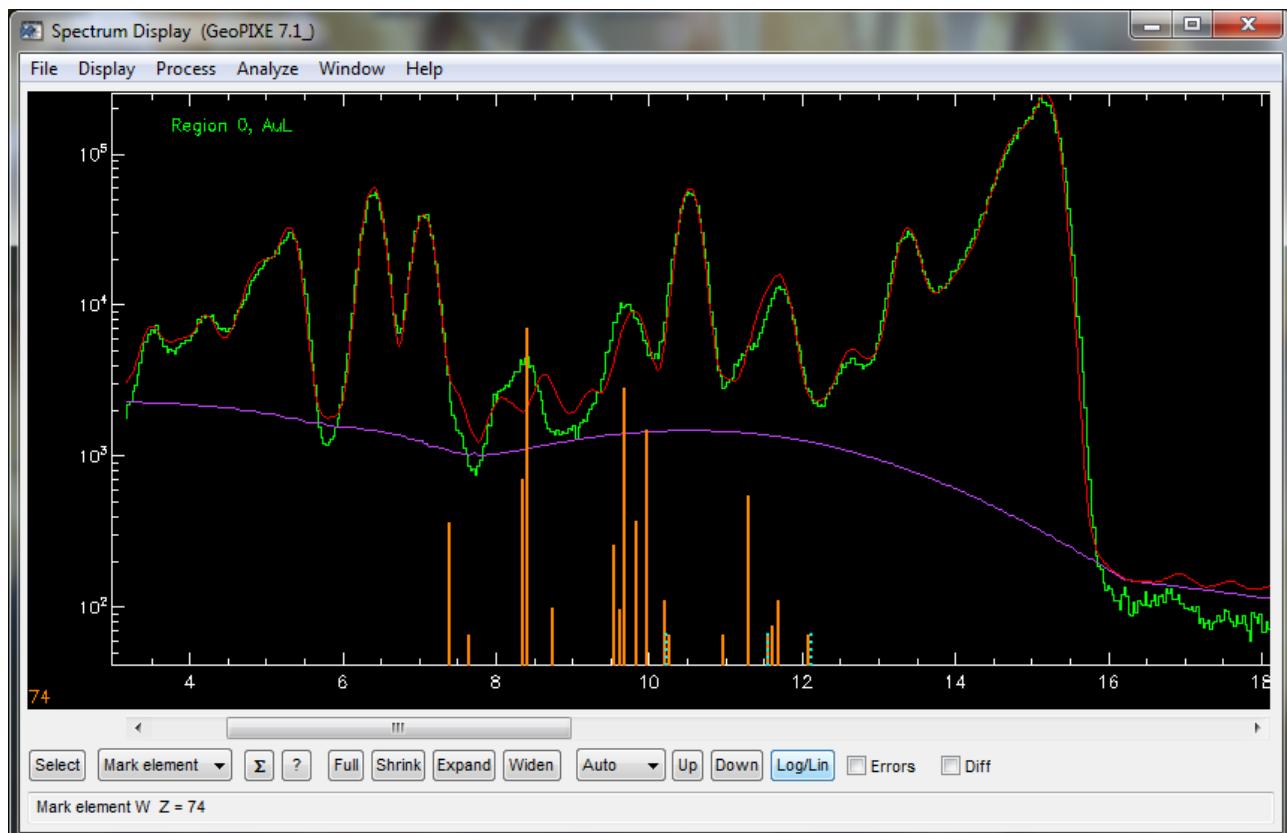
#### Extract spectra from regions

6. Extract spectra for these regions using the “Extract spectra” button on the “Extract” tab on the *Image Regions* window (the default file names are probably correct if you remembered to save the regions).

- a. First select “array” in the dropdown next to the “EVT” button, which will combine the spectrum data from all 7 detectors.
- b. Accept the default file-name “pncid\_0082-q1.spec”, which will associate these spectra with the region file of the same name.
7. Click on row 0 (for the large Au hotspot) in the *Image Regions* table, and see the extracted spectrum in the *Spectrum Display* window, with a red overlay corresponding to the DA approximation to the spectrum and a violet background.
  - a. Click “Full” or “Widen” [if your View is still set] or “Expand” the spectrum.

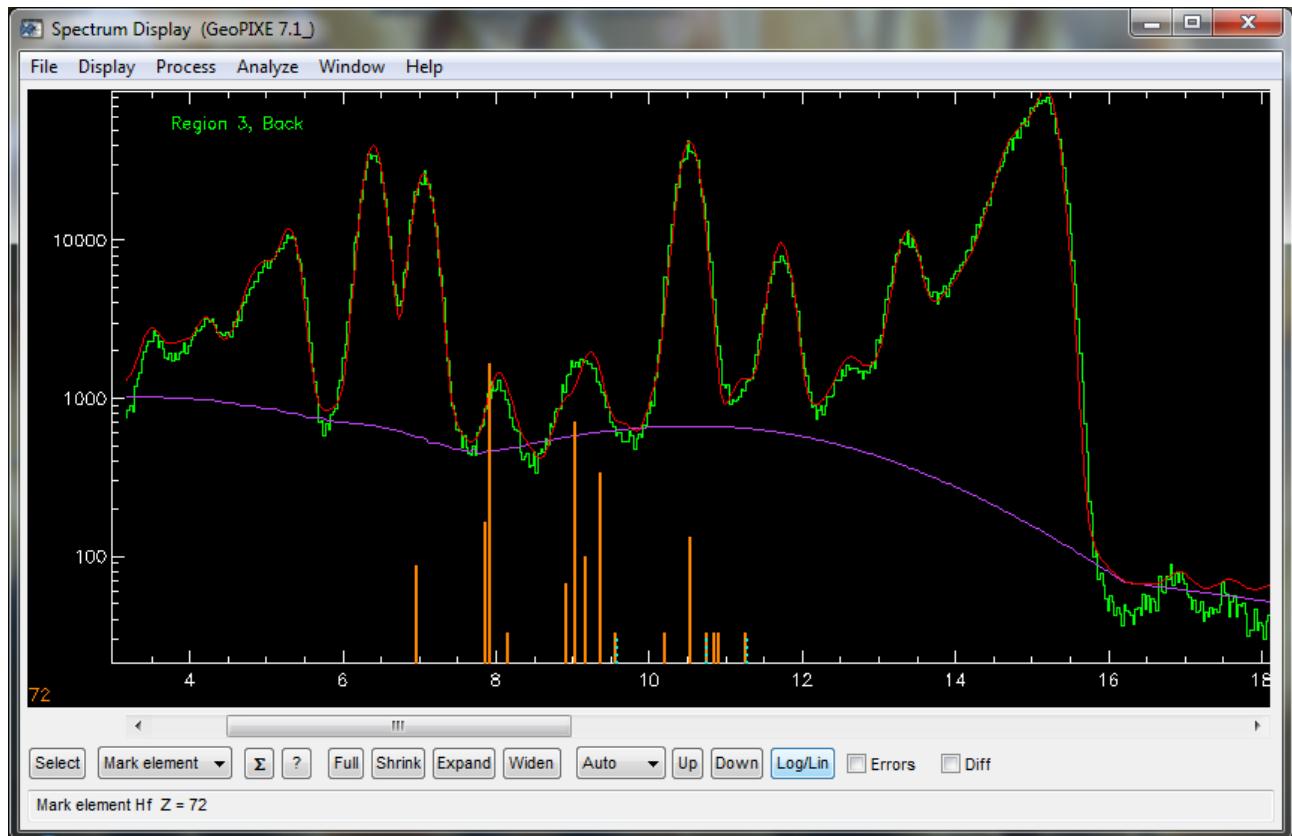
#### **Missing elements?**

8. Notice that some green data peaks do not seem to have a good matching red overlay, such as at 8.4 keV.
  - a. What is this due to?
9. Open the *X-ray Identification* window (set relative intensity threshold to 0.05) and investigate this new peak.
  - a. What element is missing?
  - b. Candidates appear to be Yb and W.
10. Switch the Identification to “Mark Element” and mark the lines for Yb and W.
  - a. Which element is it do you think?
  - b. The lines for W seem to match well, as well as the small discrepancy at 9.7 keV (this is significant as this is also the Au L $\alpha$  energy).



Region #0 (Au hot spot) spectrum (green) with DA overlay (red) and W L lines marked (orange)

11. Similarly, investigate the hot-spot seen high on the far right of the “Cu” image.
  - c. What element is that due to?

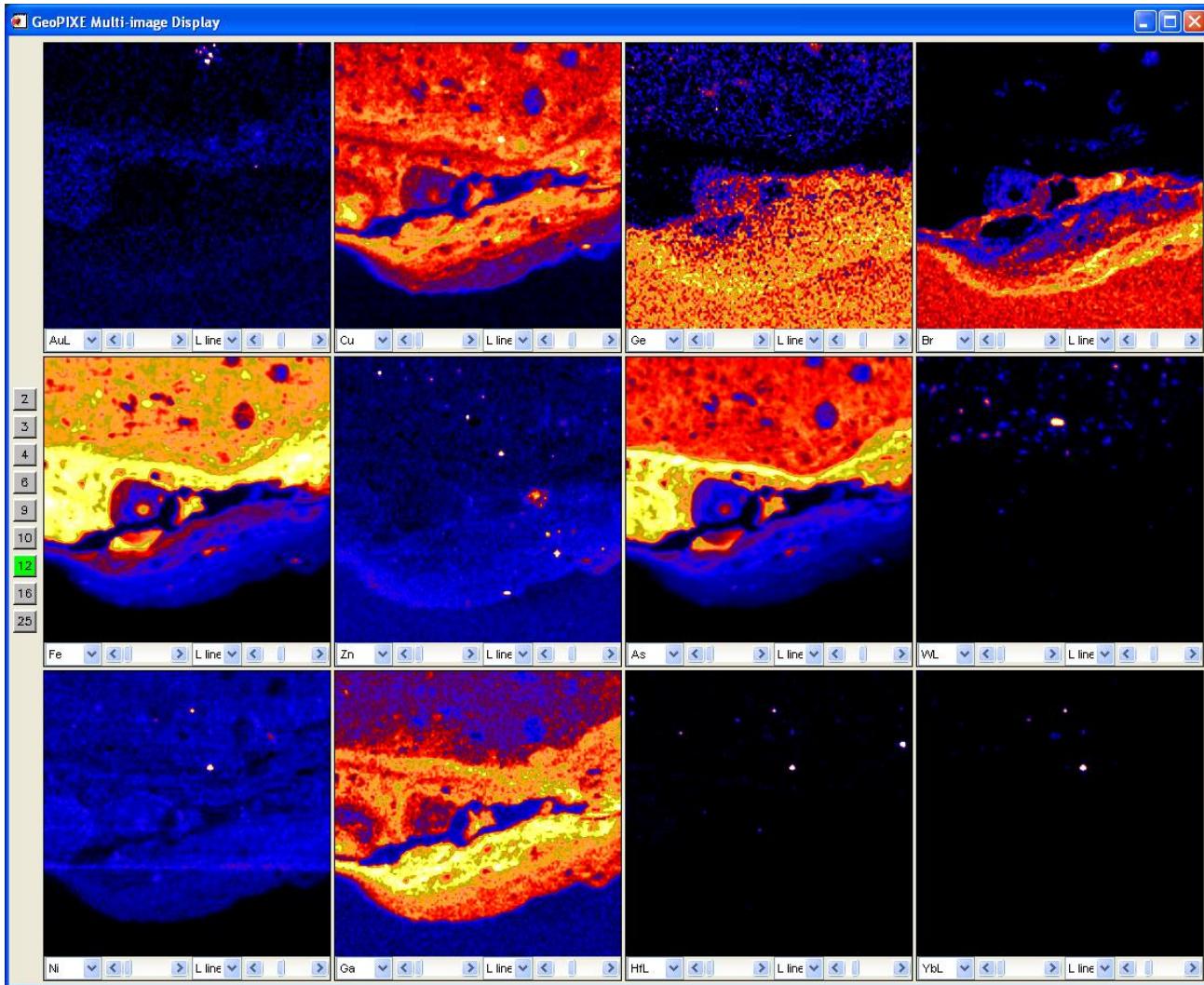


Region #2 ("Back" hot-spot high on far right side) with Hf lines marked. Remember there is Cu there too.

#### Add new elements to the fit

12. Open the X-ray Spectrum fit window again, and the set-up file "hematite-array.pcm".
13. Fit the Au hot-spot region #0 spectrum (click "Fit: One").
  - a. Notice the poor fit around energies of ~8 and ~9 keV.
14. W is a missing element, not included in our original assessment of the total spectrum.
  - b. Significantly, the L $\beta$  lines of W overlap with the Au L $\alpha$  lines (9.71 keV) and will disrupt our Au image projection.
15. Add "W" to the fit as L lines (click twice to change it to yellow), and fit the spectrum again. It now provides a much better description of the data.
16. Also add "Yb L" and "Hf L" lines. Fit again.
17. Save the fit-setup parameters in "hematite-array-with-W-Hf-Yb.pcm".
18. Generate a new DA matrix, and save it as "hematite-array-with-W-Hf-Yb.damx".
19. In Sort EVT, select this new DAMX file as the DA matrix file and start the sort again.

The *Multi Image* display (if it does not update, close it and re-open it) now shows quite contrasting images for Au, W, Yb and Hf. Try sliding the Max display slider for Au to the right to see the few hot-spots remaining. Evidently, our original "Au" hot-spot was mostly due to W, while the hot-spot on the right has a lot of Hf in it. Interestingly, there are still a few real Au hot-spots remaining.



Run #82 with W, Hf, Yb added to element list. Note the contrasting Au image to that obtained previously and the new images for W, Yb and Hf.

#### Update Region concentrations

20. Reload the regions file again, and click on “Update: All” to re-apply these region shapes to the new images (and new element list).
  - a. Now the region #0 hot-spot shows mainly a strong W concentration, while the one on the right show high Hf.
21. You might want to add a new region around the remaining real gold hot-sots at the top.
22. Save the regions to disk in file “pncid\_0082-with-W-Yb-Hf-q1.region”.
23. Repeat spectra extract for these regions and check out the improved DA overlay to each spectrum.

#### Tips:

- After generating new images always check out any hotspots in this way, especially for the “Back” image.
  - Back, which is the intensity of the underlying background term, often collects any new elements that appear somewhere in the image data but are not included in the fit or overlap with elements that are.
  - Check these, and add any new elements to the fit, build a new DA matrix and repeat generation of images.
- It would be a good idea to extract the spectra from these updated regions again.
  - Do the new DA overlays explain the green data peaks in the original hot-spot better?

#### Questions:

1. Why didn't the W signal appear in “Back” in this example?

## Example B3: Exploring and exporting image data

- Image navigation, zooming in
- More on simple regions and shapes
- Extracting linear (and curvilinear) traverses from image data
- Using two-element Associations to select pixels in images
- RGB composite Image display
- Export of images as images and plot files (also see the section on “*Exporting data*”)
- Extracting a sub-set of an image as a “Windowed sort” file at full resolution

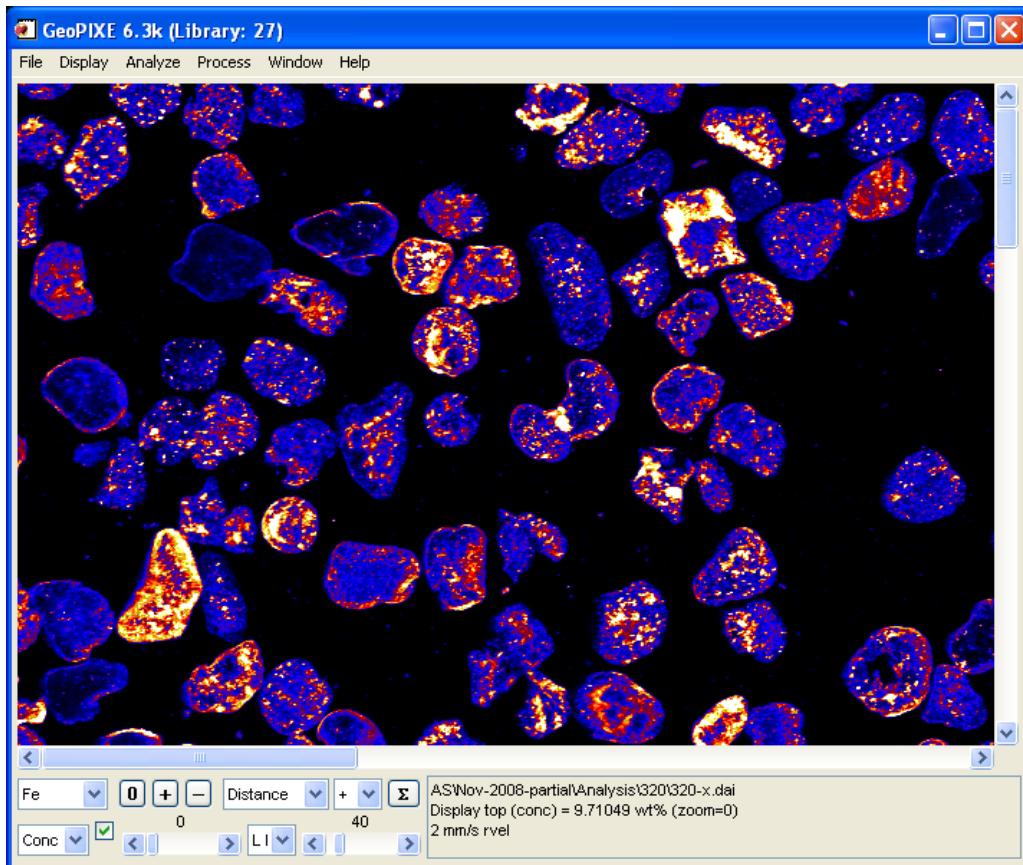
### Element images – zooming in

1. Load the image file “**Demo\Maia\Rutile\320-x.dai**”, which contains the image of a rutile mineral concentrate sample (2000 x 2000 pixels, compressed from original data of 8000 x 8000 pixels collected using Maia-96).
2. Open the *Image Properties and History* window. This shows details of the image, such as original size (in pixels and in mm), compression and current pixel size, origin, files used for processing (linearization, pileup, throttle) and the detectors used.

*This file is a piece of history, being one of the first Maia-96 prototype images acquired at XFM. The file pre-dates some features and contains black stripes due to beam drop-outs as it pre-dates Martin’s magic run scripts that would now pause data collection.*

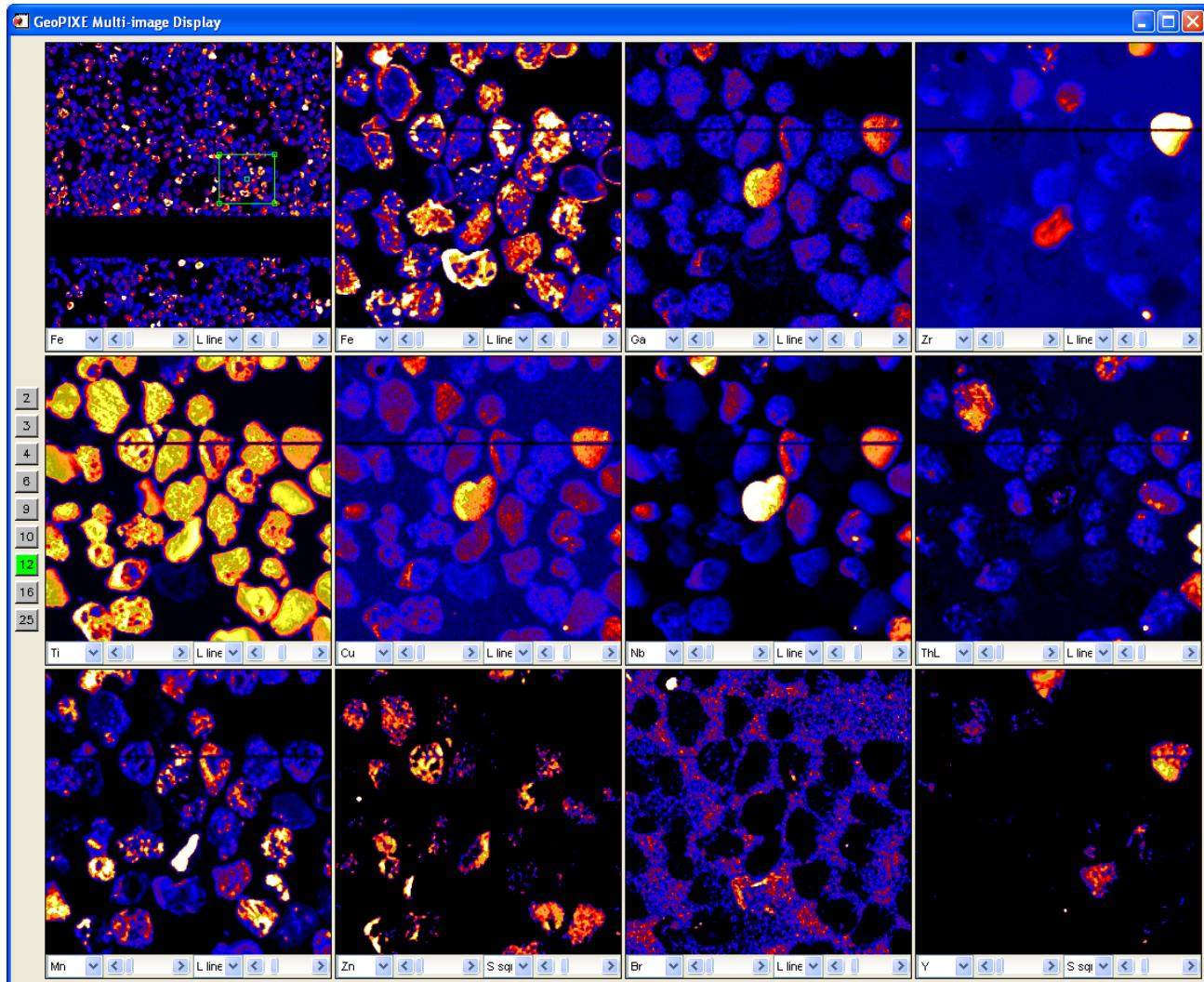
**Take Care:** *This is a large image (2000 x 2000 pixels). If you zoom in (make image larger) more than 2 clicks, you may run out of display memory, and the zoom will fail. At best, probably, you can click on the “0” button to see the image without any reduction in size.*

3. Click on the “0” button to see the image actual size. You can select elements and pan around using the scroll bars.
  - a. Scan around and see the great variety of grain types and internal structures apparent. Ti, Fe, Nb and Th are interesting.



Rutile Fe image (courtesy Peter Kappen, Latrobe U.) at natural resolution (2000 x 2000 pixels; after “0” button) – note “zoom=0” in the legend.

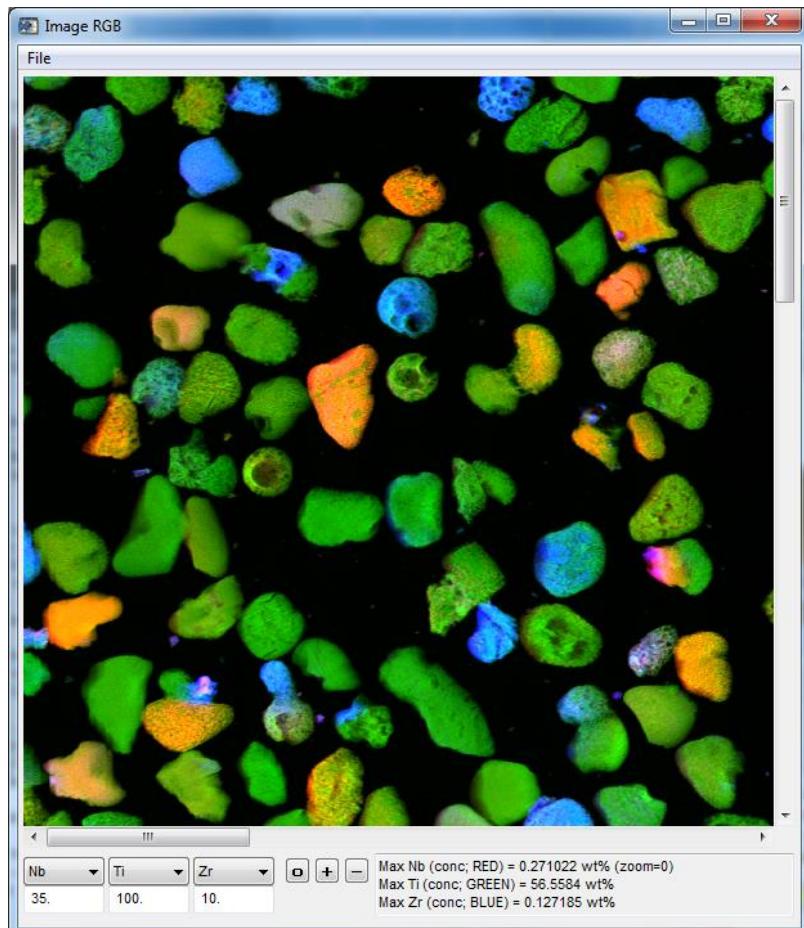
4. Open a *Multi Image* window (“Display->Multi Image” menu), which can display a large number of elements.
5. Elements can be selected on each element frame, and the display Z scale can be changed as for the Image window
  - a. Indeed they work in tandem – changes made to one are seen in the other.
6. Zoom in on a small portion of the top-left image: ‘left-click-drag’ to drag out a box on the first (top left) element image frame. This region is displayed expanded in the other frames. Select and move the handles to change the view.
  - a. There is a lot of detail in these Maia image (even with the 4x compression on both axes). Try a small box and move it around using the central handle, to zoom in on various small areas. Select a different element for the top-left image to use as a guide.



Rutile concentrate image file 320-x.dai (10 x 10 mm; 8000 x 8000 compressed to 2000 x 2000 pixels) displayed in Multi Image window. A small zoom region is selected in the first frame (top-left), which is shown expanded in the other element frames.

#### **RGB Image display**

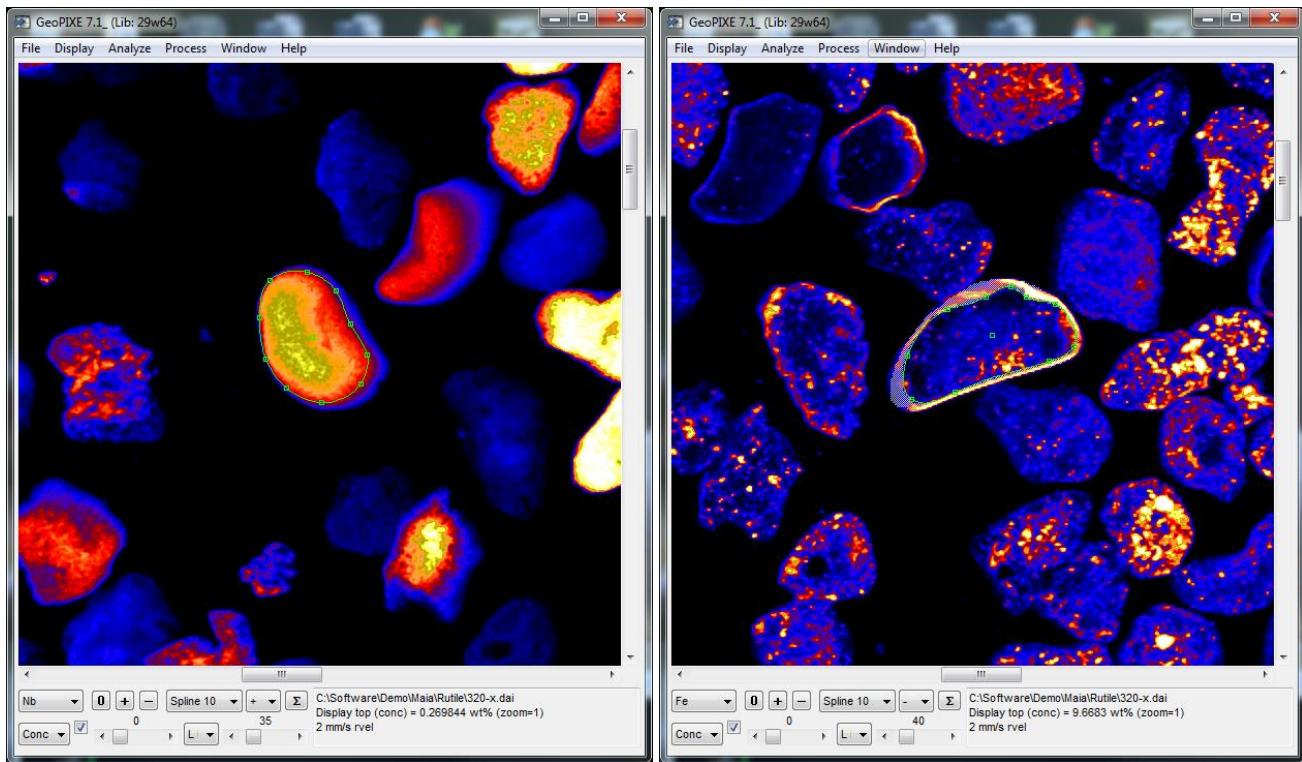
7. Open the RGB Image window (“window->3 Element RGB Images” menu).
  - a. Zoom it out too using “0“.
8. Select the following elements for Red, Green and Blue: Nb, Ti, Zr.
9. Select each of these elements in turn in the image window and reduce their display maximum to produce a well-balanced RGB image.
  - b. As the scale maxima are changes, the RGB image changes to reflect the new scales.
  - c. You can also type in new display Maxima in the text fields below the three element dropdowns.
10. Click once on “+” to zoom the RGB window in again, and move around the image using the scroll bars. You might also try making the window full screen.
11. Export the image using the “File->Simple Image->Save as PNG” menu for a lossless image, and “File->Simple Image->Save as JPEG” for a smaller file. See later for an export mode with axes and other features.



*RGB image for the Rutile image showing Nb (red), Ti (green) and Zr (blue).*

#### **Shapes and regions used to probe average concentrations**

12. Select shapes and extract concentration averages from various regions of the above images (“**Demo\Maia\Rutile\320-x.dai**”).
13. Shapes can be selected on the middle dropdown below the image display. Try each.
14. Select circle, and drag out a circle. Click on “Σ” to integrate within this shape; the average concentrations appear in the *Image Regions* window. try also an “Ellipse”.
15. Select Box and drag out a box. Try clicking and moving the control points. The small internal one connected to one corner allows the box to be rotated. The central one allows the box to be moved as a whole. Click “Σ” to integrate the shape.
16. Select a 10-point spline curve, which may be more appropriate to these grains, and drag it out. Then move the control points to shape it to encompass some feature. Click “Σ” to integrate the average concentrations.
17. After applying “Σ” to a region, switch to exclude mode, using the dropdown beside the shape dropdown to select “-“ (exclude mode).
18. Then select a new shape and drag it out. When you apply “Σ” it subtracts or “excludes” it from the previous selection.



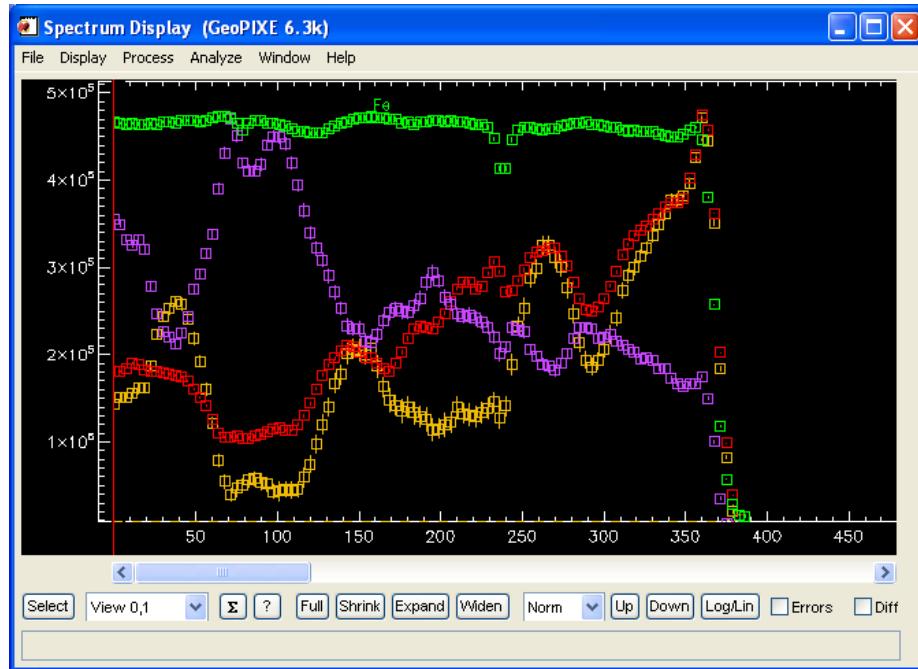
Region selected using (i) a 10-point spline shape (left) and (ii) a 10-point spline (“+” include mode) followed by a 10-point spline (“-“ exclude mode) (right).

#### Tips:

- Between exclude mode (“+”) and exclude mode (“-“), remember to integrate the region using “ $\Sigma$ ”.
- Exclusions can be applied multiple times to successively remove pieces of a selection (shown shaded). You can even use an exclude shape like an eraser to successively chip away at a prior included region.
- To clear all shapes and start again, use the menu “Display→Clear ALL Shapes”.

#### Linear and Curvilinear Traverses

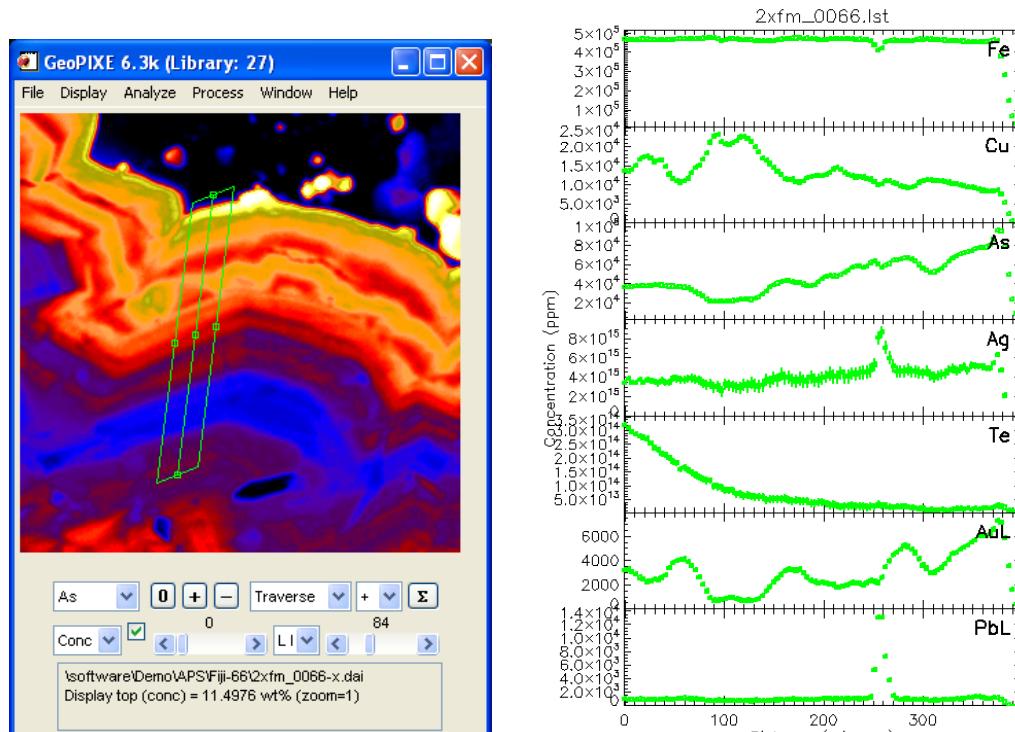
19. Try the “Traverse” shape (a good example is “Demo\APS\Fiji\2xfm\_0066-x.dai”).
20. Drag out a profile region across the zonation.
  - a. Left-click and drag the side handles to change the width;
  - b. Right-click and drag them to change the shear (data are projected and integrated along lines parallel to the end faces).
  - c. Try to get the end faces parallel to the zonation (see figure below).
21. Click on “ $\Sigma$ ” to form the line profile, which is displayed in the connected *Spectrum Display* window (the *Region* window just shows the average concentrations across the whole traverse region).
  - d. Click on “Full” to show the full profile spectrum range.
22. Click on “Prev” and “Next” in the *Spectrum Select* window to step through the element profiles.
  - e. The error bars come from the Variance images that are acquired in parallel with the concentration images.
23. Change the spectrum display to “Linear” rather than log scale.
  - f. Change the vertical display mode to “Norm” to normalize each to its maximum to show relative variation.
24. Use the *Spectrum Select* window to select elements to view:
  - g. Use “Prev”, Next” to step through the element profiles.
  - h. Toggle “Display” on selected elements to display a few overlaid together.



Traverse (see image view below) displayed (“Norm” mode) for Fe (green), Cu (violet), As (red) and Au (gold). The symbol colour legend is shown as coloured boxes in the Spectrum Select window list “Sample” column.

25. Try exporting the traverse plots as a CGM graphic (e.g. as below).
  - i. Use the menu “File→Export→CGM Plots” to pop-up export panel.
  - j. Set the background colour to “white” and preview it using “Preview”.
  - k. Un-check “Landscape” to produce a more Portrait shaped plot.
  - l. Select elements to display using the “Select Spectra” button.
  - m. Click “OK” and give the CGM file a name. CGM files can be inserted into PowerPoint\* as an image.

\*NOTE: A recent security update of Windows has stopped CGM file import into PowerPoint and Word. See the following doc in the GeoPIXE Help dir for a solution: “Error importing CGM files into Windows.pdf”.

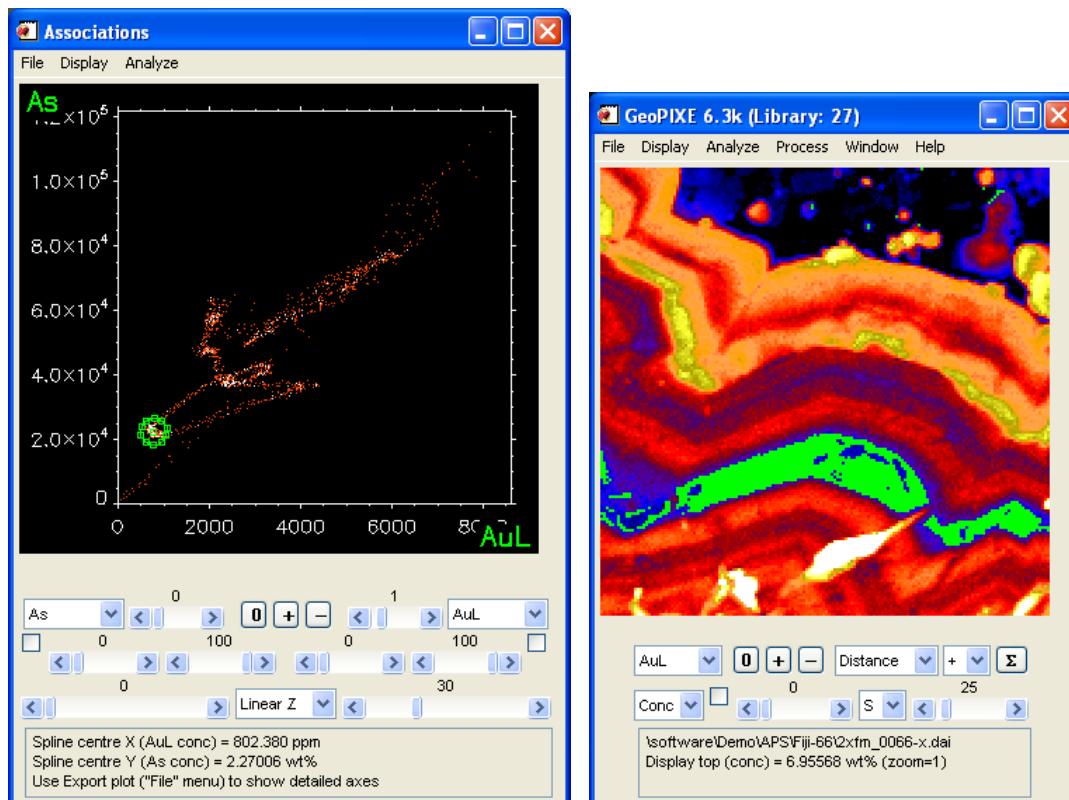


Traverse shape position and export of traverse profiles for a few elements as a CGM file (white background). (Data courtesy Chris Ryan, CSIRO).

26. Also try the “Curve 8” shape, which also defines a traverse, but with the ability to move spline handles (along centre-line) to make the traverse follow a non-linear curve.
  - n. Move the centre handle (or right-click move sides) to change to a ‘piece of pie’ shape, for example.
27. Click on “Σ” to form the line profile, which appears in the *Spectrum Display Window*.

#### **Element Associations (an alternative selection method)**

28. Continue with the example “Demo\APS\Fiji\2xfm\_0066-x.dai”.
29. Clear the traverse shape selection using the “Display→Clear ALL Shapes” menu in the *GeoPIXE Image* window.
30. Open the *Associations* window (“Element Associations” on the windows menu).
31. Select “As” and “AuL” as axes. Disable Log axes (uncheck the boxes on the sides under the element dropdowns).
  - a. See the context sensitive help info in the bottom window area as you move the cursor over the many controls in this window.
32. Now select a Traverse shape in the Region Table.
  - b. The easy way to select the traverse again is to click on that row in the *Image Regions* window list from the previous exercise.
  - c. Now the *Association* window only plots pixel data that is within this selected Region area.
  - d. Make sure that this region does NOT include the gold minerals grains towards the bottom of the image.
33. Bump up the intensity display (slide right slider at bottom of *Associations* to lower values, e.g. 30).
  - e. You might adjust the pre-smooth applied to each axis image prior to plotting on the scatter histogram using the top sliders on the *Associations* window (As = 0, Au = 1 works well).
34. Now click and drag on the plot to form a small spline curve circle within the *Associations* window.
  - f. Right click in the *Associations* window if you need to clear a prior shape.
  - g. Grab the central handle and drag this over a feature of the Au-As relation and select “within spline” on the “Analyze” menu.
  - h. Note that the corresponding pixels are selected on the image, and a new row is added to the *Regions* window.
  - i. Right click in the *Image* window to temporarily hide these highlighted pixels.
    - i. This defaults *Association* back to all pixels; you’ll need to click on the region traverse row to select these pixels again.



Left: Association between As and Au along the traverse through the growth zonation, which shows a complex time series of evolution of the two elements as a trail on the As-Au plot. Right: pixels selected using the spline field on the As-Au plot.

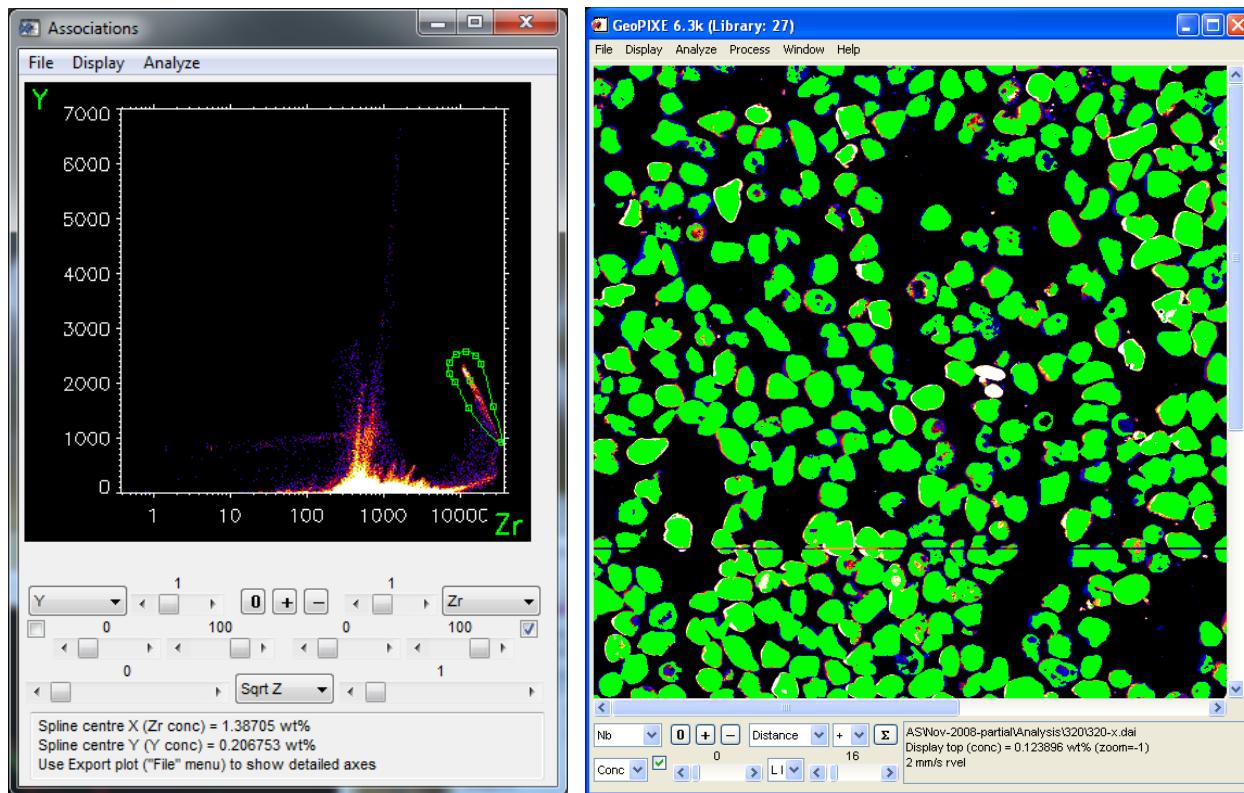
35. Right-click in the *Association* window to clear the spline curve, and try another one.
  - j. Click and drag individual control points to shape the spine curve.
36. Each of these association highlighted regions is saved in the *Image Regions* window, and the average concentration for this region is integrated and shown.
  - k. These regions and their concentration averages, both for simple shapes and two-element Association selections, can be saved to a file for later use.
  - l. You can extract spectra from these selected pixels in the same way as region shapes.

#### **Element Associations (contrasting chemical features)**

37. Continue with the case “**Demo\Maia\Rutile\320-x.dai**”.
38. Select Y versus Zr on the Associations window plot.
  - a. Linear on Y axis, and reduce top slider (bottom) row to zero to show all points.
  - b. Use minimal pre-smooth (=1 on both axes; top sliders)
  - c. Drag out a selection spline curve and drag control points around features.
  - d. Select menu “Analyze→Within Spline” to select all pixels with the selected Y-Zr relation.
    - i. This is a good way to select contrasting phases in a material.

#### **Use threshold to select all grains**

39. Select Ti on both axes, linear scales, and select SQRT Z scale with maximum equal to zero (display all).
  - a. Set different pre-smooths to make it less of a boring straight line (e.g. Y=1, X=4).
40. Drag out a circle, and move it to fill the top-right  $\frac{3}{4}$  of the displayed trend.
  - b. Make sure you include the top-right of the plot.
41. Use menu “Analyze→Within Spline” now colours all rutile grains green, and the region window gives an average composition of the grains.
  - c. A valid question to ask would be: Is the layered yield model appropriate for a rutile grain such as this? In this case, they were ground down and polished into uniform thickness ‘slabs’. So the answer is probably ‘yes’, apart from edge effects.



Left: Association of Y and Zr in the rutile grains of run 320 showing numerous distinct phases characterized by contrasting correlation (or anti-correlation, as shown, perhaps indicating coupled substitution) between Y and Zr.  
 Right: Ti versus Ti Association used as a threshold to select all rutile grains.

#### **Image Export**

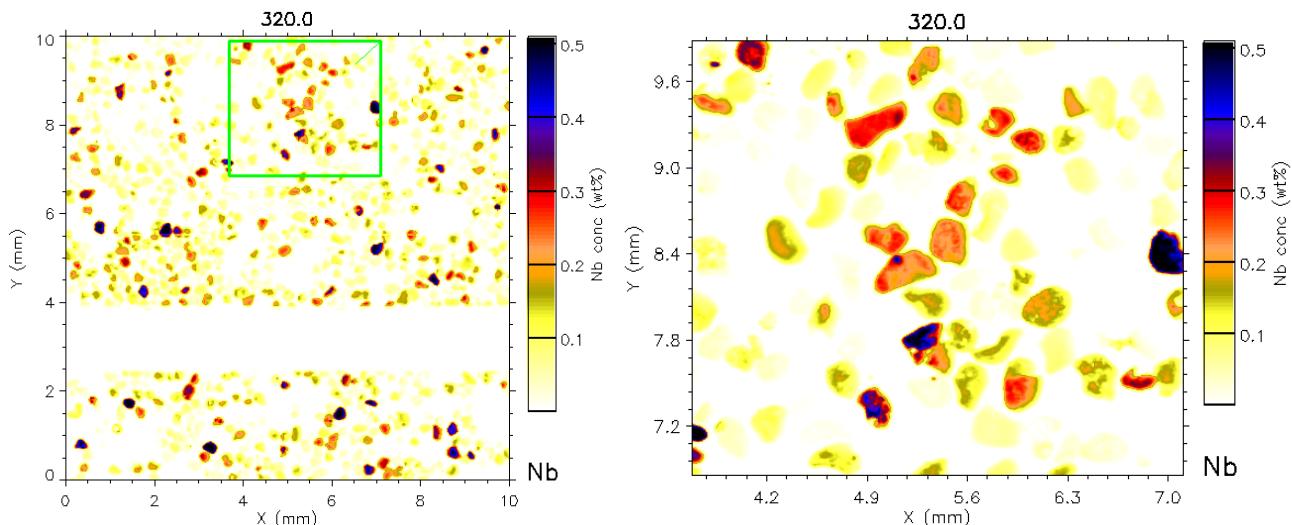
Also refer to the section below on “Exporting Data” ...

Continue with the example “Demo\Maia\Rutile\320-x.dai”.

42. Save all images as a web-page (HTML) format file using the menu ”File->Save All as HTML (PNG)”.
  - a. This creates a web-page file with small thumbnail images of each element. These are linked to the full images.
  - b. View this in a browser (e.g. double-click on the HTML file just created).
  - c. Click on the images to see the full image for an element.\
43. Select the Nb image.
  - d. Open the CGM export window (“File→Export→Image Plot→Current image as JPEG” menu).
  - e. Click on Preview to see the current output figure.
  - f. Click “OK” to create a CGM output file. This file can be inserted in a PowerPoint display or Word doc.

*Note: Windows systems now block the CGM standard in favour of their own WMF format. However, WMF is not supported on Linux systems. To avoid this impasse, use the option to plot to JPEG, which creates a very large JPEG bitmap to enable good quality axes and axis labels despite using a bitmap.*

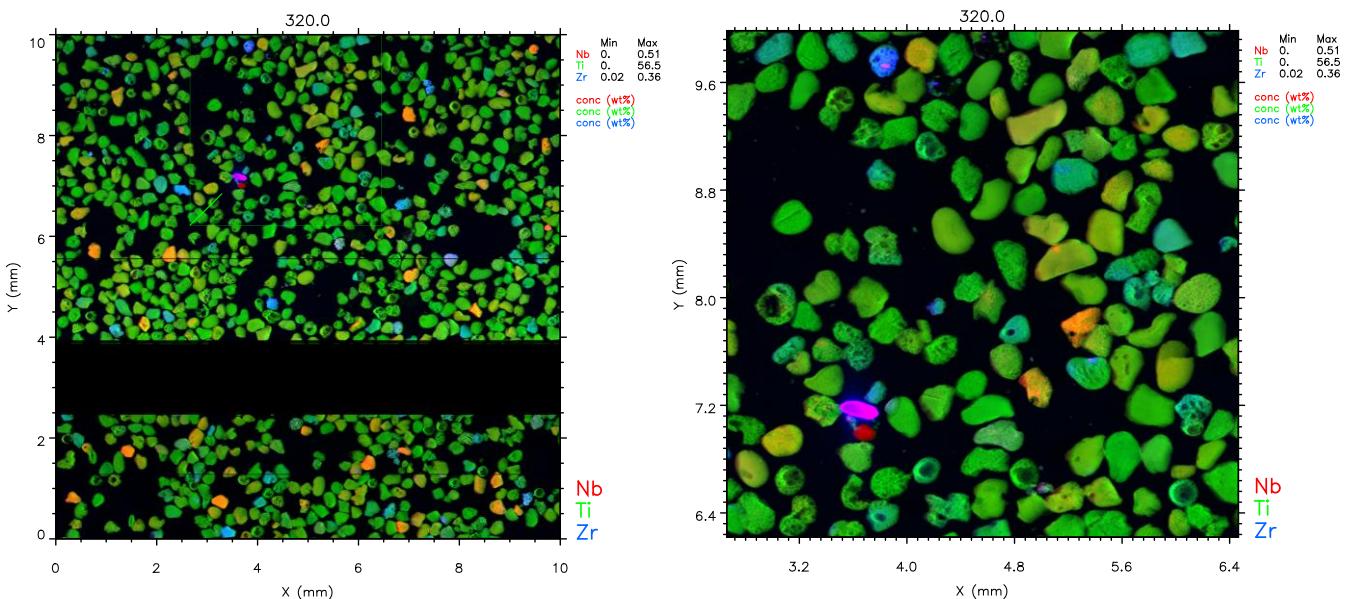
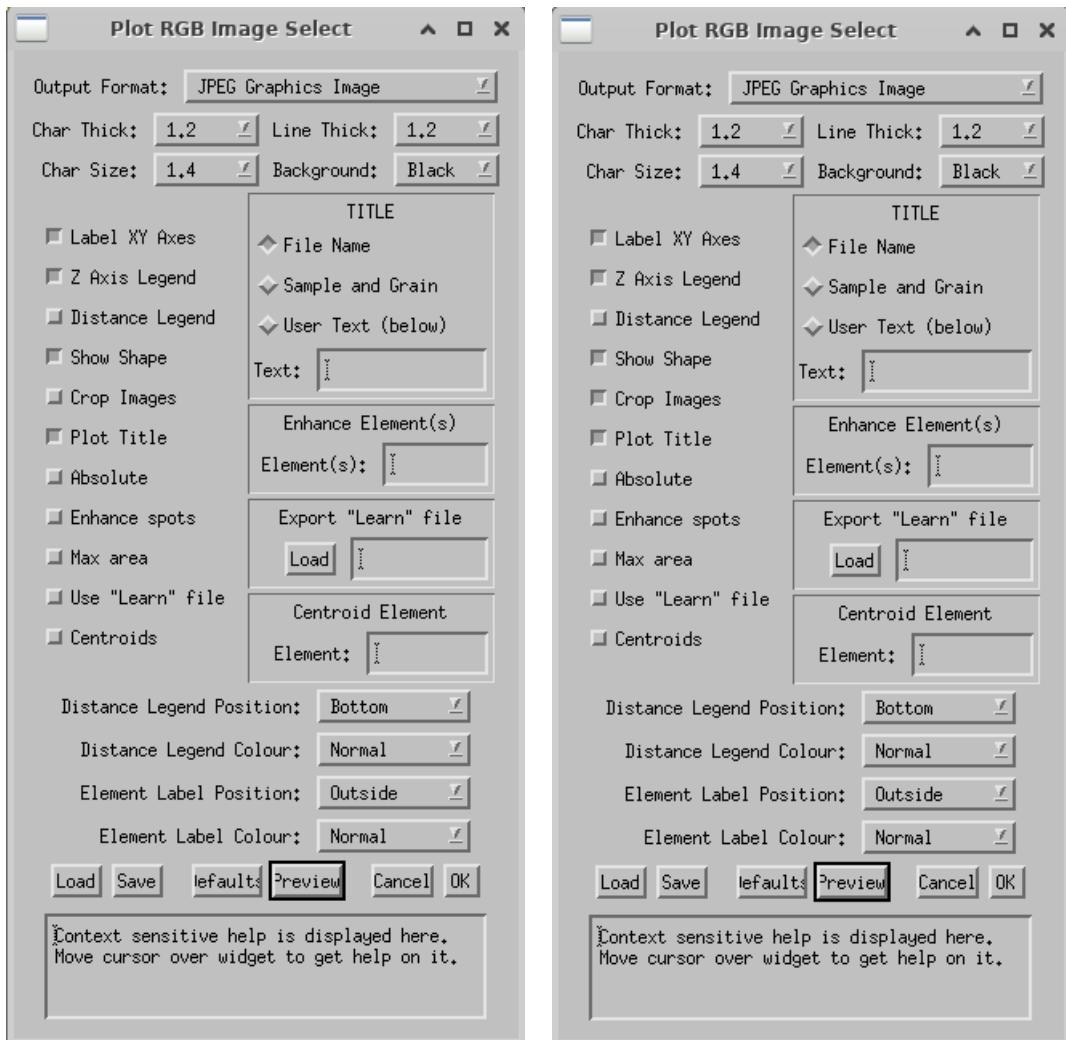
44. Experiment with various export parameters in the panel:
  - g. Black or white background (for white it is best to first select a different colour map from the “Display→Colours→Colour Table” or “Display→Colours→Invert Colour Table” menus). This will enable an image with white background to be produced, which works well for printing.
  - h. Select a “Box” region on the image, and enable “Crop images” on the export pop-up panel. This will restrict the export plot to just the selected region.
45. Finally, click “OK” to save the plot file to disk.
  - i. Note that the filename you give is just a ‘stub’ – the element name for the image will be appended to it.



Left: export of Nb image (after inverting colour map) with “Show Shape” enabled; Right: export of Nb for the selected region using “Crop Images” enabled.

#### Image RGB Export

1. Open the RGB Image window and display Nb, Ti and ThL in Red, Green and Blue.
2. Select “Export→Image plot→RGB Image as JPEG” from the File menu.
  - a. A number of options can be selected from the Export pop-up window, such as:
    - i. axes with tick marks (Label XY axes),
    - ii. a distance legend and bar (Distance Legend),
    - iii. a Z axis intensity legend (Z Axis Legend) and
    - iv. either relative coordinates from the image origin or absolute coordinates (Absolute) making use of the stage XY origin stored for each image.
    - v. The position of the element labels and the distance bar can be selected.
    - vi. You can also crop to the bounds of a region shape (Crop Images) or overlay the shape on the plot (Show Shape).



#### Extract a sub-set of an image at full resolution

1. A sub-set of a scan area can be extracted or sorted from the data-set at full resolution using the “Windowed sort” option. This is illustrated in the worked example for “**Maia Mapper – MM 925 image data**”.

#### Tips:

- Open the GeoPIXE manual from the Help menu.

- Worked examples are in the “GeoPIXE Analysis Scenarios and Data Flow” section.
- User guide detail of menus and controls can be found in the “GeoPIXE User's Guide” section.
- Some shapes are for concentration averages (box, circle, spline curves, ellipse), and some are for traverse extraction (traverse, curve 8).
- The Export of RGB images uses the image actually displayed (at the current Zoom level). To save the full resolution image, press “o” zoom button first.
- Use WMF (Windows Metafile) format on Windows machines, as Microsoft tends to not allow CGM by default (anti-social behaviour), and use CGM (Computer Graphics Metadata) under Linux. Or better still, just use the JPEG option, which works everywhere (it creates a very large JPEG to preserve character resolution).

*Further examples can be found in the GeoPIXE manual, in the **GeoPIXE Analysis Scenarios and Data Flow** chapter and local Maia worked examples are provided in the **Maia-384-user-help** document.*

## C. The Maia Detector – Maia Specific Data and Processing

### Example C1: Maia-384 – Probing raw data records (synchrotron)

A useful diagnostic tool is the **Blog\_browser** routine, which enables you to examine exactly what is stored in your Maia data files, various metadata information, what Epics PVs were logged, etc. This example explores ...

- Probing Maia data files using **Blog\_Browser**
- Checking parameters set during data acquisition
- DA imaging details and per pixel values, Detector channel activity
- Examining Maia ET data in detail.
- Examining Epics PV values, such as IC counter and Maia FC0 flux counter, beam energy

In normal usage you may never need to dig deep into the data files like this. But if you need to check something, this is the way.

1. Run **Blog\_Browser** using the “Windows->Blog Browser” menu on either the *Image Display* or *Spectrum Display* windows or run the file “blog\_browser.sav” directly (using **Linux command “blogbrowse”**).
2. Click on “Run” and select the raw data file (e.g. 112686.0) in “**Demo/Maia/Metadata/blog/112686/**”.
3. Select a “Buffer size” of 10 Mbytes.
4. Now, click on the file 112686.0 in the Segment list on the left to show all records (in the first 10 Mbytes).
  - a. Each record has a “tag” identifier, a sequence number and a “tag sequence” number, which is the order of records appearing for each tag, and a time stamp.
  - b. Scroll to the top (first record) and click on a record to see details of it appear in the lower panel.

*Maia blog record types (note that older data may not have some of these records, such as ‘metadata’)*

5. Scroll back to the top and find the following record types (tag number):
  - a. **Event\_1** (34): ET event records (find one that has data in it rather than “no E(T) found”):
    - i. Pixel address (0, 1, 2) for the three encoder axes interfaced to Maia
    - ii. BT value – time acquiring this record (we sum all BT for a pixel to get Dwell)
    - iii. FC0 – flux counter input #0 count (we sum all FC0 for a pixel to get Flux #0 count)
    - iv. FC1 = flux counter #1
    - v. Event list, grouped as **Energy, Time (detector channel)** for each event
    - vi. Click on an **event\_1** record to show a list of events in the lower panel and a plot of E versus T for this record and a histogram of detector activity.
  - b. **DA\_accum** (35): DA accumulator for each pixel, giving RT element contributions to each pixel.
    - i. XY pixel address
    - ii. Dwell time “duration” (ms)
    - iii. NN – total events from all detectors
    - iv. NNPU – pileup rejected events (fall off normal E-T curve)
    - v. TT – total of T for all events in pixel (proportional to dead-time)
6. Now click on “Select Record Tags” and de-select the more common tags (“event\_1”, “DA\_accum”, “client”) to see the rare ones:
  - c. **Scaninfo\_1** (47): Scan information
    - i. image size and origin (in XYZ stage coordinates)
    - ii. image pixel size and pixel pitch in microns
    - iii. “requested” stage velocity and nominal dwell per pixel
    - iv. Some beamlines show beam energy and flux sensitivity here too
  - d. **Monitor** (26): monitor records that show Epics PVs as they are sampled
    - i. Click on an Epics PV in the bottom window to see a histogram of this PV for the whole buffer.
  - e. **Metadata** (55): Metadata information (note there are two or more of these)
    - i. Run information
    - ii. flux\_chan0\_coeff = Flux #0 preamp gain multiplier (e.g. “500”)
    - iii. flux\_chan0\_unit = Flux #0 preamp gain units (e.g. “pA/V”)
    - iv. throttle\_enable = “1” indicates throttle was active
    - v. throttle\_info = throttle file-name
    - vi. deadtime\_coeff0 = DTcalB deadtime calibration offset
    - vii. deadtime\_coeff1 = DTcalA deadtime calibration slope
    - viii. sample\_type = type of sample (“user” or “standard”)
    - ix. sample\_name = sample name string from beamline control

- x. beam\_energy = beam energy (eV)
- xi. scan\_dim0\_origin = axis 0 origin
- xii. scan\_dim0\_unit = axis 0 units
- xiii. scan\_dim0\_pitch = axis 0 pixel size
- xiv. scan\_dim0\_extent = number of pixels in scan on axis 0
- f. **Var\_Val** (46): Lists parameters used in Maia during data acquisition (several of these records)
  - i. cal.det[].coeff[] = energy calibration for each detector channel
  - ii. deadtime.time.coeff[] = DTcalA, DTcalB deadtime calibration
  - iii. flux.chan[].coeff = Flux #0,1 preamp gain multipliers
  - iv. flux.chan[].unit = Flux #0,1 preamp gain units
  - v. pileup.enable = “1” indicates pileup active
  - vi. pileup.info = pileup correction field file-name
  - vii. throttle.enable = “1” indicates throttle mode active
  - viii. throttle.info = throttle vector file-name
  - ix. throttle.energy[].factor = throttle vector
  - x. pixel.dim[].origin = origin in axes 0,1,2
  - xi. pixel.dim[].pitch = pixel size for axes 0,1,2
  - xii. pixel.dim[].coord.extent = number of pixels in scan for axes 0,1,2
  - xiii. scan.info[] = text labels from beamline control
  - xiv. scan.size[] = number of pixels in axes 0,1,2
  - xv. status.detector.bias.voltage = detector bias (V)
  - xvi. status.detector.bias.current = detector leakage ( $\mu$ A)
  - xvii. status.flux.chan[].rate = Flux #0,1 count-rates (/s)
  - xviii. metadata.datum[]... = repeats some beamline metadata
- g. **DA\_info** (44): DA real-time imaging set-up (if running)
  - i. elements selected for RT imaging
  - ii. Dead-time calibration (DTcal A and B coefficients)
- h. **DT\_accum** (37): Dead-time accumulator
  - i. NN, NNPU, TT per detector, accumulated over a longer duration (e.g. 2 s)
  - ii. Pile-up loss fraction per detector (scroll down)
  - iii. Dead-time fraction per detector (requires DTcalA to be set correctly in Maia Control)
  - iv. Shows plots of deadtime and pileup losses (%) for this record
- i. **Activity** (39): Activity as a count for each detector.
  - i. Count in each detector per “Duration” (0.5 seconds), plus a histogram plot.
- j. **Id2** (28): Identity
  - i. Target file path on blog server, run number.
- k. **Summary2** (32): Summary info
  - i. Total blocks, bytes, data rate to disk.

**Tips:**

- Click on “Select Record Tags” and de-select the more common tags to see the rare ones.
- Start with a small “buffer size”, such as 10 Mbytes, for better speed.
- You might need to increase the buffer size to the full 100 Mb to see rare records that come at the end.

## Example C2: Maia-384 – Probing raw data records (Maia Mapper)

A useful diagnostic tool is the **Blog\_browser** routine, which enables you to examine exactly what is stored in your Maia data files, various metadata information, what Epics PVs were logged, etc. This example explores ...

- Probing Maia data files using **Blog\_Browser**
- Checking parameters set during data acquisition
- DA imaging details and per pixel values, Detector channel activity
- Examining Maia ET data in detail.
- Examining metadata values, such as detector identity, scan details, nominal dwell time or following the track of the raster scan as it goes.

In normal usage you may never need to dig deep into the data files like this. But if you need to check something, this is the way.

1. Run **Blog\_Browser** using the “Windows->Blog Browser” menu on either the *Image Display* or *Spectrum Display* windows or run the file “blog\_browser.sav” directly (using **Linux command “blogbrowse”**).
2. Click on “Run” and select the raw data file (e.g. 925.0) in “**Demo/MM/blog/925/**”.
3. Select a “Buffer size” of 10 Mbytes.
4. Now, click on the file 925.0 in the Segment list on the left to show all records (in the first 10 Mbytes).
  - a. Each record has a “tag” identifier, a sequence number and a “tag sequence” number, which is the order of records appearing for each tag, and a time stamp.
  - b. Scroll to the top (first record) and click on a record to see details of it appear in the lower panel.

**Maia blog record types** (note that older data may not have some of these records, such as ‘metadata’)

5. Scroll back to the top and find the following record types (tag number):
  1. **Event\_1** (34): ET event records (find one that has data in it rather than “no E(T) found”):
    - i. Pixel address (0, 1, 2) for the three encoder axes interfaced to Maia
    - ii. BT value – time acquiring this record (we sum all BT for a pixel to get Dwell, which is used on Maia Mapper as a proxy for flux)
    - iii. Event list, grouped as **Energy, Time (detector channel)** for each event
    - iv. Click on an **event\_1** record to show a list of events in the lower panel and a plot of E versus T for this record and a histogram of detector activity.
  - m. **DA\_accum** (35): DA accumulator for each pixel, giving RT element contributions to each pixel.
    - i. XY pixel address
    - ii. Dwell time “duration” (ms)
    - iii. NN – total events from all detectors
    - iv. NNPU – pileup rejected events (fall off normal E-T curve)
    - v. TT – total of T for all events in pixel (proportional to dead-time)
6. Now click on “Select Record Tags” and de-select the more common tags (“event\_1”, “DA\_accum”, “client”) to see the rare ones:
  - n. **Scaninfo\_1** (47): Scan information
    - i. image size and origin (in XYZ stage coordinates)
    - ii. image pixel size and pixel pitch in microns
    - iii. “requested” stage velocity and nominal dwell per pixel
  - o. **Metadata** (55): Metadata information (note there are two or more of these)
    - i. Run information
    - ii. flux\_chan0\_name = “dwell.time” (for Maia Mapper)
    - iii. flux\_chan0\_coeff, flux\_chan0\_unit (1,1 for Maia Mapper)
    - iv. throttle\_enable = “1” indicates throttle was active
    - v. throttle\_info = throttle file-name
    - vi. deadtime\_coeff0 = DTcalB deadtime calibration offset
    - vii. deadtime\_coeff1 = DTcalA deadtime calibration slope
    - viii. sample\_type = type of sample (“user” or “standard”)
    - ix. sample\_name = sample name string from beamline control
    - x. beam\_energy = beam energy (eV) (fixed for now, at 24100 eV for Maia Mapper)
    - xi. scan\_dim0\_origin = axis 0 origin
    - xii. scan\_dim0\_unit = axis 0 units
    - xiii. scan\_dim0\_pitch = axis 0 pixel size
    - xiv. scan\_dim0\_extent = number of pixels in scan on axis 0
  - p. **Var\_Val** (46): Lists parameters used in Maia during data acquisition (several of these records)
    - i. cal.det[].coeff[] = energy calibration for each detector channel
    - ii. deadtime.time.coeff[] = DTcalA, DTcalB deadtime calibration
    - iii. pileup.enable = “1” indicates pileup active
    - iv. pileup.info = pileup correction field file-name
    - v. throttle.enable = “1” indicates throttle mode active
    - vi. throttle.info = throttle vector file-name
    - vii. throttle.energy[].factor = throttle vector
    - viii. pixel.dim[].origin = origin in axes 0,1,2
    - ix. pixel.dim[].pitch = pixel size for axes 0,1,2
    - x. pixel.dim[].coord.extent = number of pixels in scan for axes 0,1,2
    - xi. scan.info[] = text labels from beamline control
    - xii. scan.size[] = number of pixels in axes 0,1,2
    - xiii. status.detector.bias.voltage = detector bias (V)

- xiv. status.detector.bias.current = detector leakage ( $\mu$ A)
- xv. status.flux.chan[].rate = Flux #0,1 count-rates (/s)
- xvi. metadata.datum[]... = repeats some MM metadata and also shows the MM installation/site and end-station ID (e.g. MM.Mel, 1).
- q. **DA\_info** (44): DA real-time imaging set-up (if running)
  - i. elements selected for RT imaging
  - ii. Dead-time calibration (DTcal A and B coefficients)
- r. **DT\_accum** (37): Dead-time accumulator
  - i. NN, NNPU, TT per detector, accumulated over a longer duration (e.g. 2 s)
  - ii. Pile-up loss fraction per detector (scroll down)
  - iii. Dead-time fraction per detector (requires DTcalA to be set correctly in Maia Control)
  - iv. Shows plots of deadtime and pileup losses (%) for this record
- s. **Activity** (39): Activity as a count for each detector.
  - i. Count in each detector per "Duration" (0.5 seconds), plus a histogram plot.
- t. **Id2** (28): Identity
  - i. Facility ID
  - ii. Target file path on blog server, run number.
- u. **Summary4** (56): Summary info
  - i. Total blocks, bytes, data rate to disk.

**Tips:**

- Click on "Select Record Tags" and de-select the more common tags to see the rare ones.
- Start with a small "buffer size", such as 10 Mbytes, for better speed.
- You might need to increase the buffer size to the full 100 Mb to see rare records that come at the end.

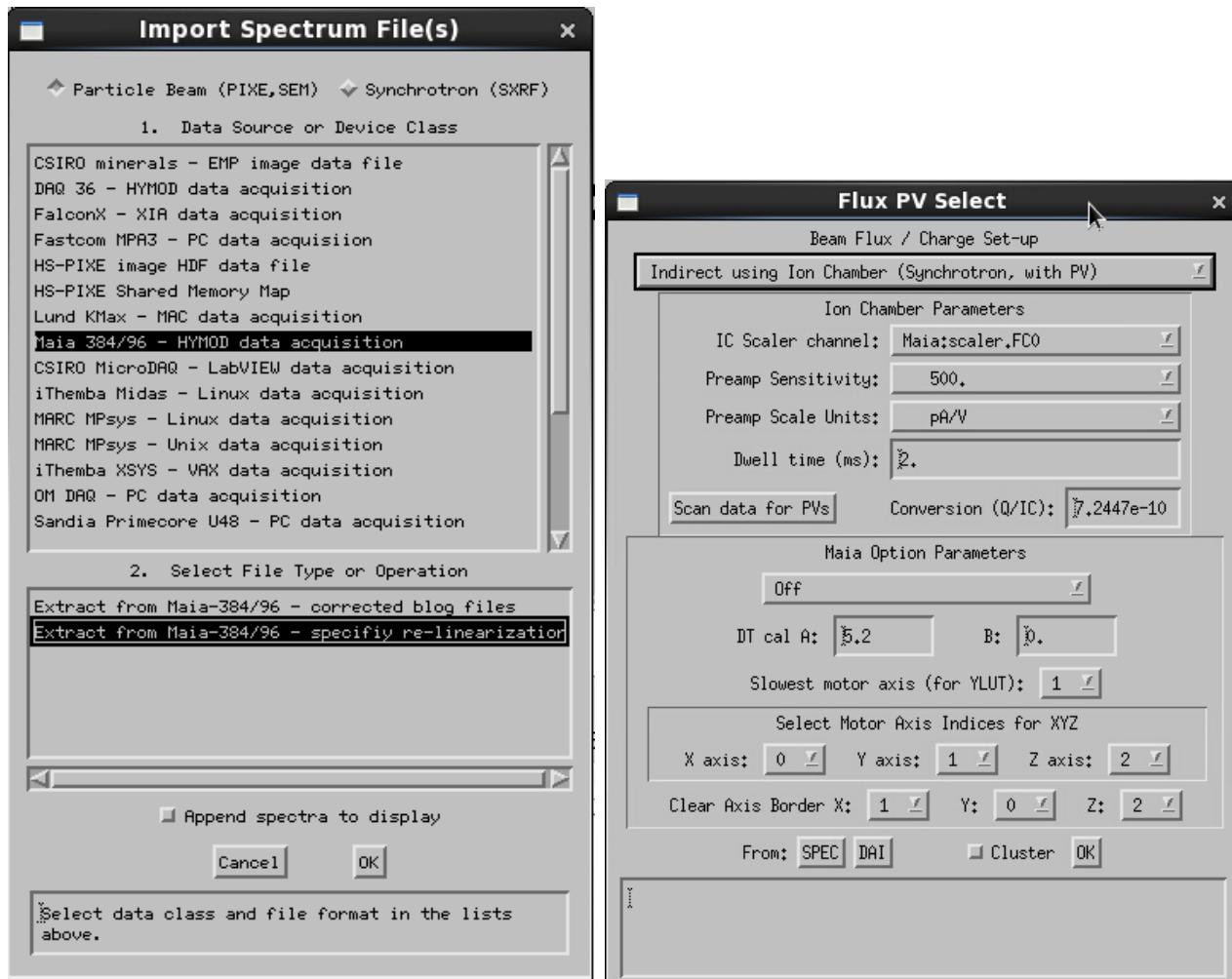
## Example C3: Maia-384 Detector – Rock3 image data

- Importing spectra for all 384 detectors from the raw data files.
- Using a 're-linearization' tweak to spectrum linearity.
- Checking the 'pileup' field used for pileup rejection.
- Fitting a representative spectrum and building a DA matrix
- Calculation of X-ray yields
- Imaging using the DA method

### Import Spectra and Calibrate Energy

1. Import spectra from the raw Maia data files for run #111163 (directory "**Demo/Maia/Rock3/**", data courtesy of Anais Pages, CSIRO) using the File→Import menu on *Spectrum Display*.
  - a. Select "Synchrotron (SXRF)" data from the "Maia 384/96 – HYMOD data acquisition".
  - b. Select file type: "**Extract from Maia-384/96 – specify re-linearization**".
    - i. NOTE: Normal Maia data would use the "**Extract from Maia-384/96 – corrected blog files**" option.
  - c. Drag-select about 10 files in directory "**Demo/Maia/Rock3/blog/111163/**".
    - i. Select 111163.0 (always select the .0 file to get header information)
    - ii. Scroll down to about the middle of the file list.
    - iii. Control-click and drag-select another ~10 files (e.g. 111163.36 – 111163.44).
    - iv. (This approach gives a reasonable sampling of the middle of the image area.)
  - d. Direct output to directory "**Demo/Maia/Rock3/analysis/111163/**".
  - e. Select flux scaler and sensitivity:
    - i. The header in "111163.0" does not contain flux sensitivity metadata (as described in example 9).
    - ii. So we could enter it directly: set the "IC Scaler channel", "Preamp Sensitivity", "Preamp Scale Units" and "Conversion" factor manually.
    - iii. But this time to save time, use the "From DAI" button to read previously used settings from the file "111163.dai", which selects using the Maia hardware scaler "Maia:scaler.FC0" and the calibration "Conversion" factor of 7.24e-10.
    - iv. Click "OK" to close the Flux selection pop-up.
  - f. Select the "linearization" file: "111163i-relinear-Hg-fit.linear" in the "analysis" directory.
    - i. Although Maia data is corrected for linearity when acquired in the real-time processor, this particular data-set benefitted from a further refinement of the linearization.
  - g. Selecting Pileup and Throttle:

- i. (If your “Translation table” is functioning well, you may not be prompted for pileup and throttle files as the Pileup and Throttle file-paths found in the file header will be translated into your local paths.)
- ii. If not, select Pileup=”109714-Mn\_MM33086\_384C14\_12900eV.pileup.var” and Throttle=”pearce\_18\_5keV\_scatter.throttle.var” in the “analysis” directory.



Import options and Flux PV settings, including the Maia hardware scaler “Maia:scaler.FC0”, sensitivity and the “conversion” factor.

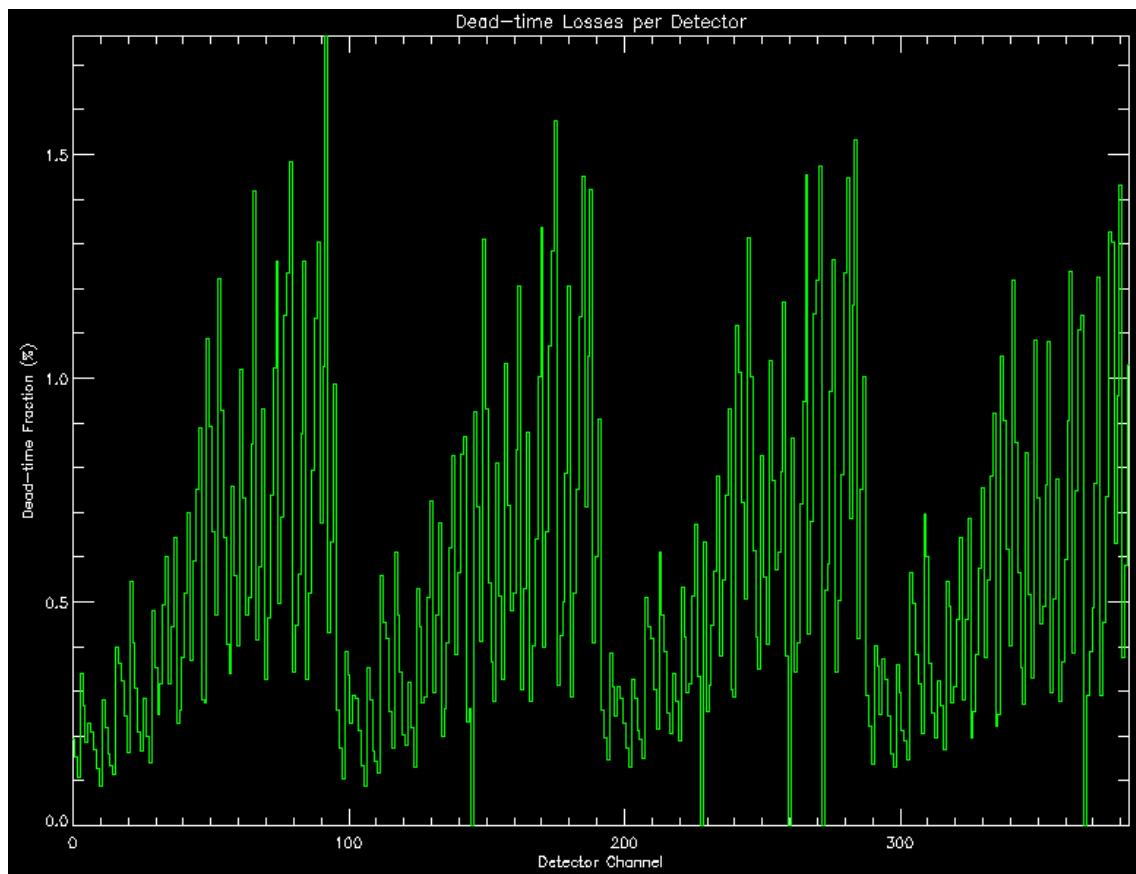
2. Open the *Spectrum Select* window and delete all XY and T spectra:
  - a. Select “all X,Y and T” on dropdown at right; click “Delete:” to delete all X,T and T spectra.
  - a. You might want to drag the window much bigger to see a range of detectors and more columns, including the CalA and CalB coefficients.
3. In this case the energy calibrations for all detectors were found in the header and set for you.
  - a. However, a better refined set of energy calibrations was constructed later.
  - b. To use the refined cal, use the *Spectrum Display* menu “Display->Get All Energy Cals” and select the file “111173\_refined\_fit.spec”, located in the “analysis” directory.
  - a. This will use the energy calibrations for each detector channel in the file to set the energy Cals of the current spectra; any missing detector channels will be disabled.
4. Sum all spectra to for a single sum spectrum:
  - a. Click on “All” in Spectrum select to display all detectors.
  - b. Sum all spectra, mapped onto a single energy calibration (for the first detector) using the menu “Process→Add (re-map cal)”.
  - c. Save this sum spectrum for later (e.g. “111163-E-sum.spec”).

### Using the plugins to examine various diagnostic plots for these spectra

Plugins are small IDL routines written (some by users) to perform special spectrum processing operations, or in this case, examine spectrum parameters and histograms that are stored with the data.

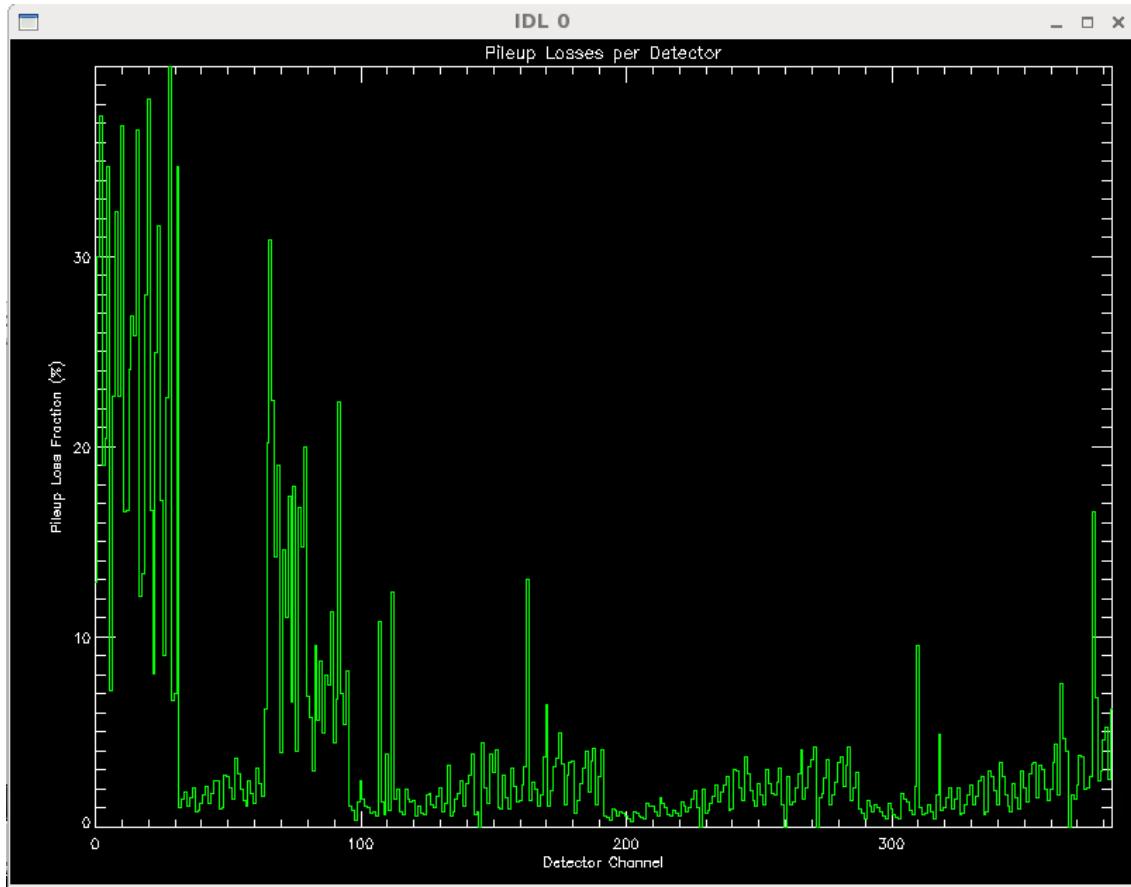
Try the “Process→User Plugins→ ...” menu on *Spectrum Display* window to run this plugins:

5. **Deadtime by detector:** Detector by detector dead-time fraction histogram – shows DT per detector channel for this spectrum. This data-set had a count-rate of about 1 M/s, which is low for Maia, so the dead-time averages only ~1%.



Dead-time per detector channel (max ~3%).

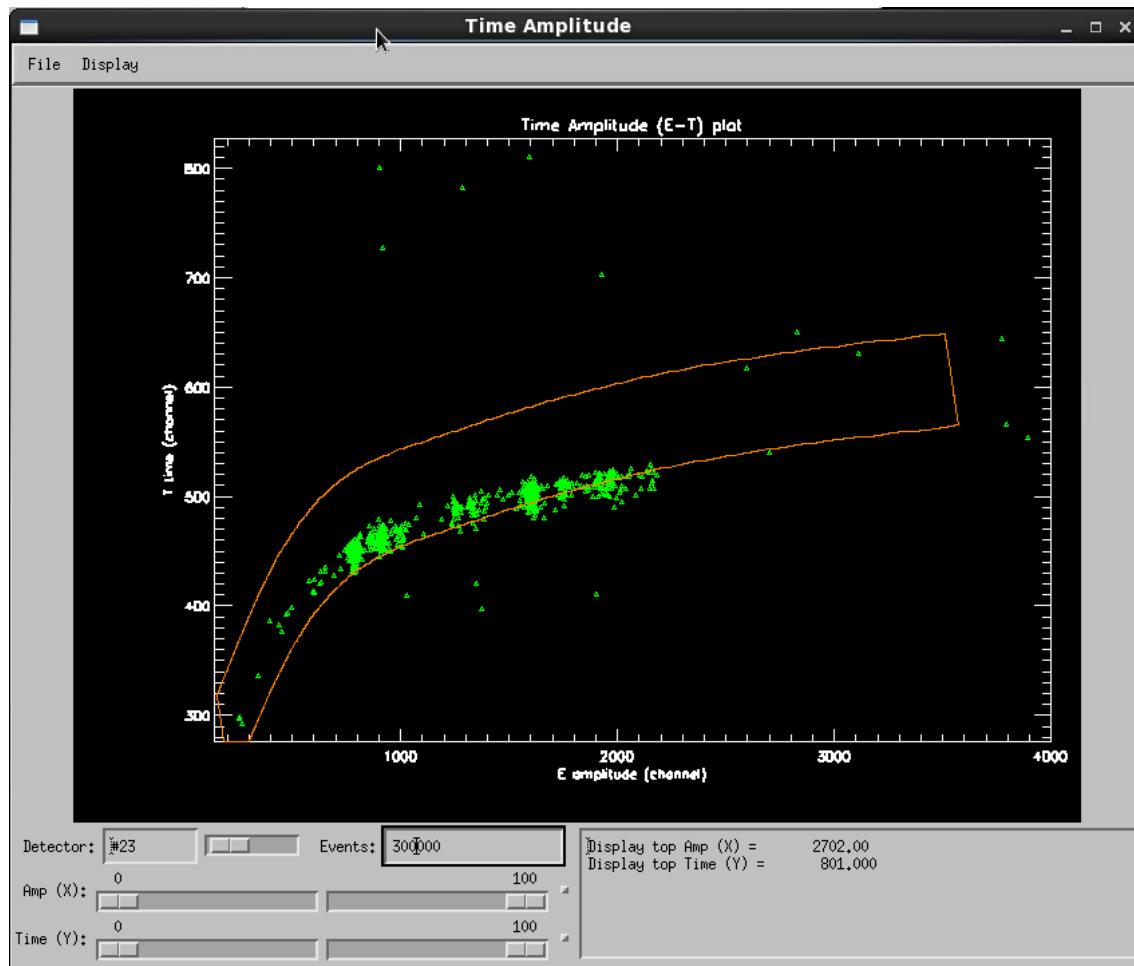
6. **Pileup by detector:** Detector by detector pileup loss fraction histogram – shows PU per detector channel for this spectrum. This shows high loss fraction for channels 0-32. Why is that?



*Pileup losses per detector channel.*

#### ***Let's investigate these Pileup losses:***

7. Open the *Time Amplitude* window and select 300,000 events and the blog files 111163.0 and 111163.1
  - a. Use “File->Extract Time Amp Maia” menu.
8. Overlay the pileup field from file “109714-Mn\_MM33086\_384C14\_12900eV.pileup.var”
  - a. Use “Overlap Maia pileup field” menu.
9. Now step through by detector (click slider knob and use arrow keys):
  - a. Notice that the pileup field defined here is rather generous (perhaps too much so). However, it rejects some few *bona fide* events. It appears that the trimming between Maia channels was overdue for calibration.
  - b. Notice that the channels between 0-31 show E-T points that fall outside the pileup field.
    - i. This is why we get a high PU rejection % for these channels.
  - c. Evidently, the T for these channels does not match the others (channels 32-383 seem OK, except for a few between 65 and 92).
    - i. The differences between channels 0-31 (and perhaps 64-91) suggest the XFM beamline crew need to redo the gain-trimming in Maia (actually this was done a week later).
    - ii. We can probably define a new pileup field that does a better job ...



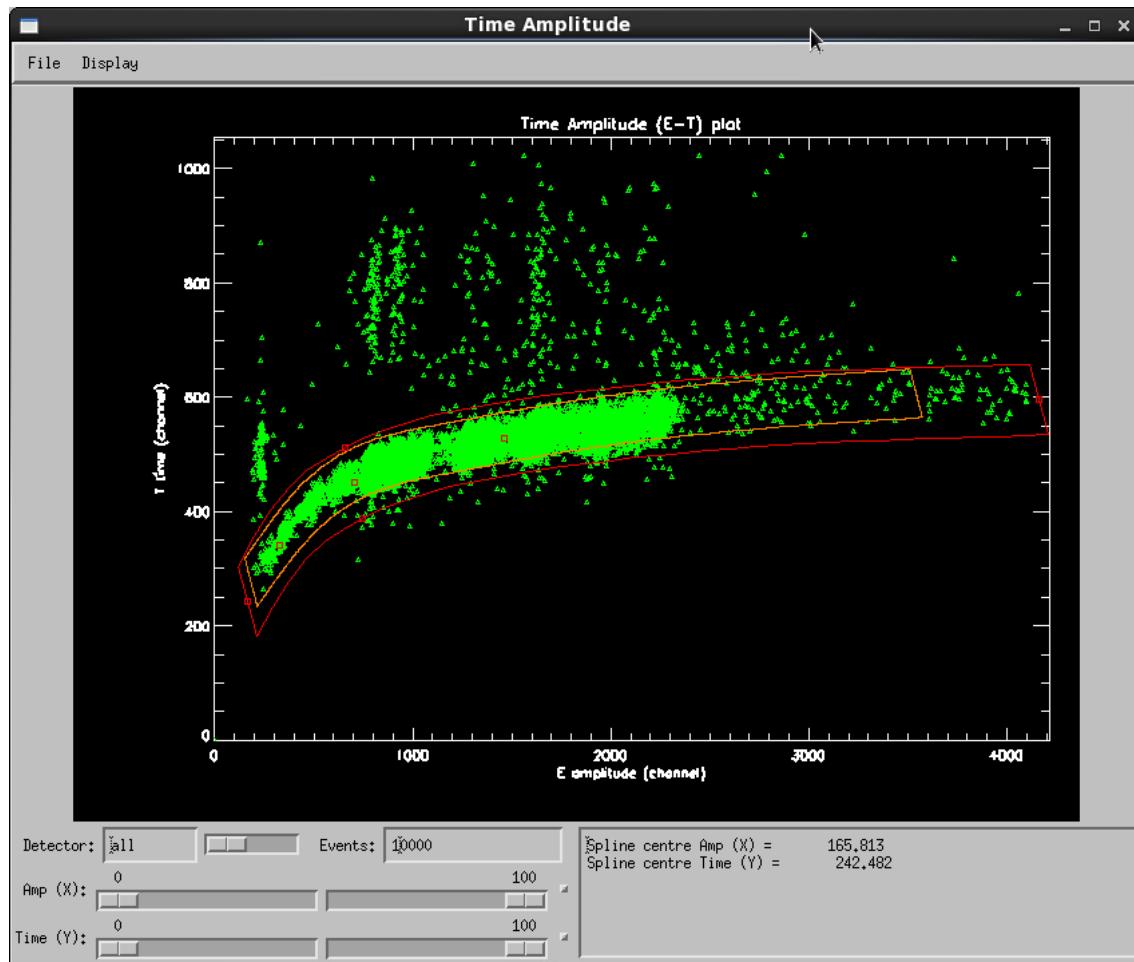
*Time amplitude plot for detector #241 showing “odd channel” glitches, which appear as lost events at low-E and above the pileup field.*

#### Defining a new pileup field

10. Open the *Time Amplitude* window again and select 10,000 events and the blog files 111163.0 and 111163.1.
11. Overlay the pileup field from file “109714-Mn\_MM33086\_384C14\_12900eV.pileup.var”.
  - a. This is shown as an orange selection field on the plot.
12. Now select “All” detectors (left text box and slider).
13. Click and drag out a new pileup selection field:
  - b. Click at the low energy end of the main trend, below the green trend, and while holding the left mouse button, drag the field out to the top of the spectrum.
  - c. Now select and drag each spline control point (handles on centre line of RED spline curve) in turn and move them to shape the curve.
  - d. Set the width of the field using the handles on the side.
  - e. You can adjust the overall placement of the whole field by using the handles in the centre at the ends.
  - f. Shape the field to better catch the missing green points below the orange field.
14. Save the field to a new pileup file (e.g. “111163\_refined.pileup.var”).
  - g. You can select this later when processing this data-set (and others in the same experiment).

#### Tip:

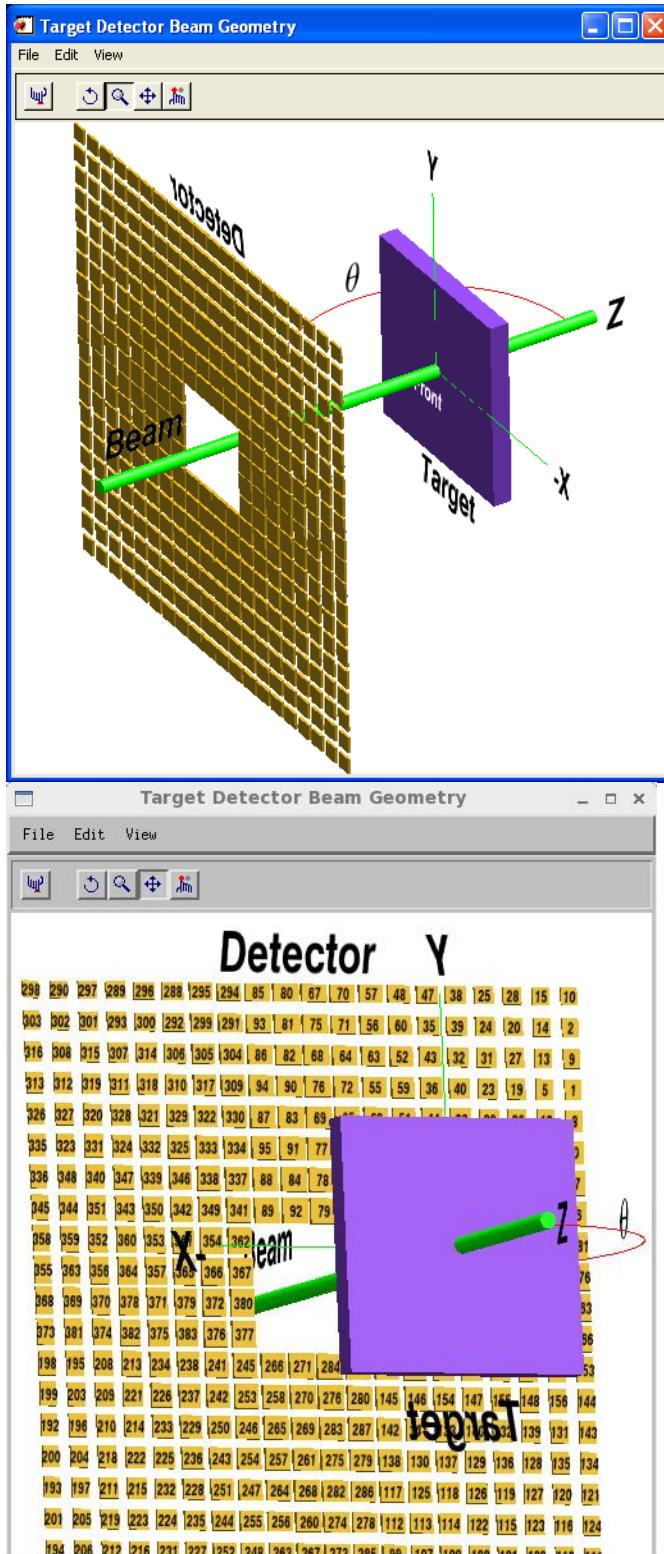
- Moving the control points more to the left (where more curvature is needed, as shown) gives the desired curve control.



E-T plot of the first 10,000 events, showing the old (orange) and new (red) pileup fields.

#### **Calculation of SXRF yields for the sample and detector array**

15. Load the sum spectrum saved previously (e.g. “111163-E-sum.spec”).
16. Open the X-ray Spectrum Fit window and load the fit parameters from file “Shale\_refined3\_18500eV.pcm”.
  - a. Notice that the “Detector” field in X-ray Spectrum Fit is greyed out and cannot be changed. This is because the Yield calculation has explicitly specified an array detector. Let’s do that now ...
17. Open the PIXE/SXRF Yield Calculation window by clicking on “New” next to “Yields” in X-ray Spectrum Fit.
18. Open the file “Shale\_100um\_SiO\_1mm\_384C14\_18500eV.lcm”. This specifies a two layered sample:
  - a. a thin section (100  $\mu\text{m}$ ) matrix of a mineral mix (layer #1), on quartz (layer #2),
  - b. a beam energy of 18.5 keV photons and
  - c. a target-detector geometry with the detector at 180° and no target rotation ( $\alpha$ ,  $\beta$  = 0°).
  - d. an array detector “AS Maia 384C 10 mm v6”, which is the new Maia 384C at AS/XFM.
19. Click on the “?” button under Detector to view the geometry in 3D.
  - a. Click in the window and drag, to rotate the 3D model.
  - b. This verifies that the detector is looking at the front side of the sample and in the case of Maia-384, the beam comes through the detector.
  - c. Rotate the view and zoom in on the face of the detector. In this way we see that detector numbers 10, 298, ... are in the outer corners, while detectors 245, 341, 284, 79, ... are around the central opening.
    - i. This detector layout can also be viewed using the “Array ...” button on the Spectrum Select window.
20. To calculate yields, click on “Calculate Yields”. This has 2 passes.
  - a. The first does a complete yield model; calculation for all elements throughout the sample structure including secondary fluorescence enhancement for a generic “central” detector element.
  - b. The second pass then performs the modelling for all other detectors to calculate relative sensitivity factors relative to the central detector, but without secondary fluorescence; the secondary fluorescence enhancement for each is taken from the first pass.

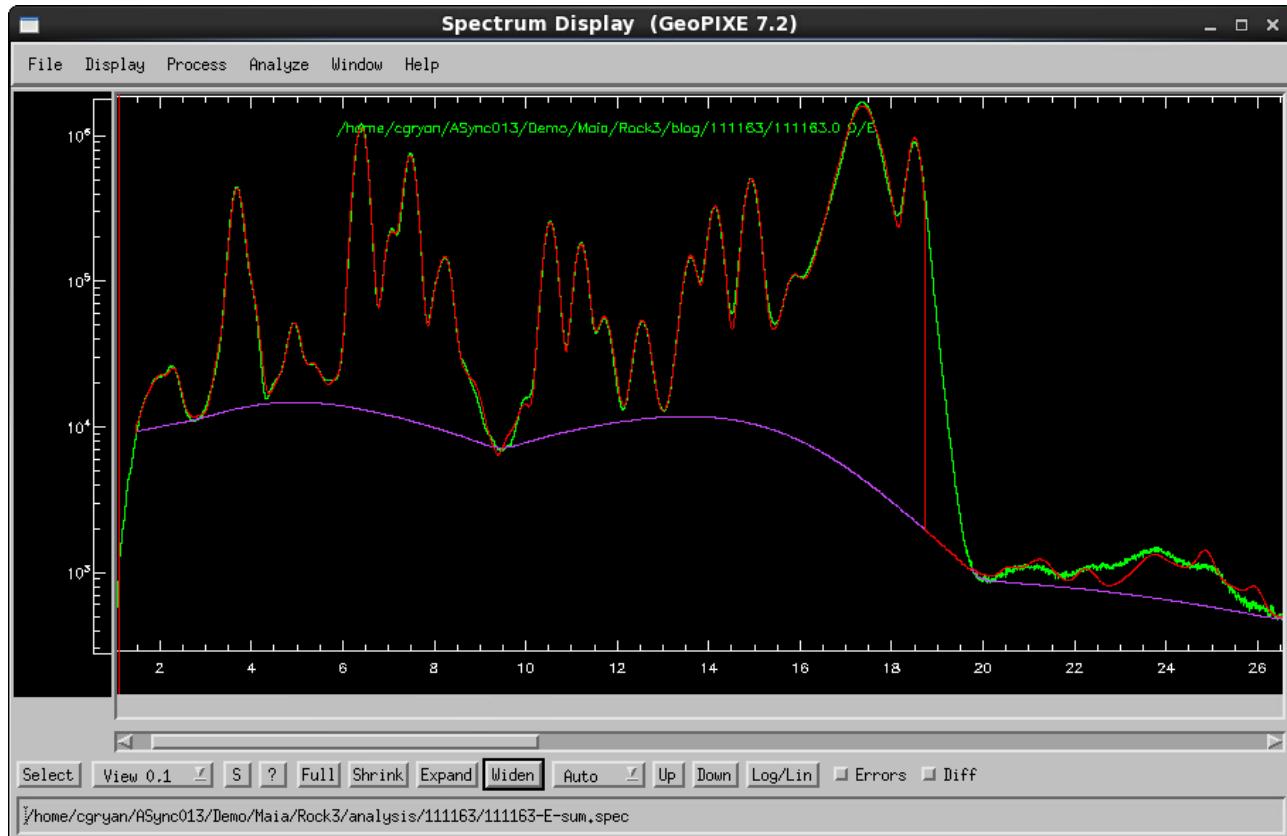


21. To finish, select the filename for the yields file (e.g. "Shale\_100um\_SiO\_1mm\_384C14\_18500eV.yield" in the local "analysis" directory).
  - a. The set-up details for this panel need to be saved separately using the Save button at the top (e.g. to file "Shale\_100um\_SiO\_1mm\_384C14\_18500eV.lcm" in the local "analysis" directory).

#### **Fit the sum spectrum and build the DA matrix**

22. Continuing in the X-ray Spectrum Fit window, with set-up "Shale\_refined3\_18500eV.pcm" loaded ...
23. Set the View range to 1.5-15.5 keV (click on "Use View") and enable variation of the peak widths and energy calibration in the fit by checking "FWHM" and "Cal On" in the main ("Setup") fit panel in X-ray Spectrum Fit.

24. Click on “Fit: One” in *X-ray Spectrum Fit* to fit the spectrum and refine the energy calibration.
  - a. This is a complex data-set with 33 elements, U includes both L and M lines.
25. Disable “Cal On” and “FWHM” and open the View range out to 1.5-27 keV (to include the pileup peaks).
  - b. Fit again, which will also include the scatter and an approximation to pileup contributions.
26. If a good fit is obtained, then click on the “Generate DA matrix” button to build and save a DA matrix file “Shale\_refined3\_18500eV.damx”.



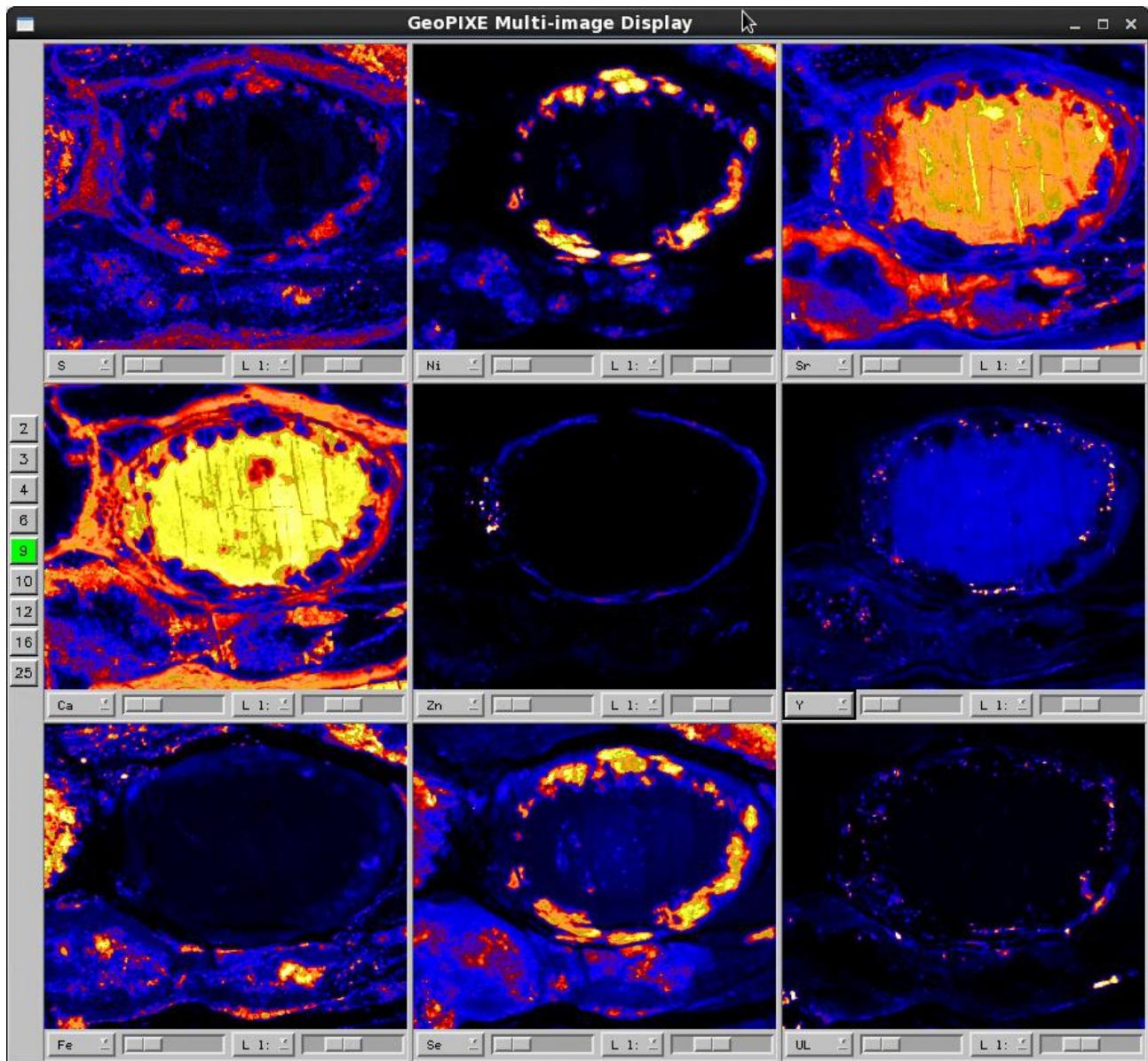
*Sum spectrum for #111163 with overlays for fit (red), background (violet). Data courtesy of Anais Pages, CSIRO. The right side of the elastic peak has been removed from the fit using a CUT.*

#### Using this DA matrix to generate images

Often an existing image can be loaded into *Sort EVT* to provide a template for a new scan (using “From DAI” button), which minimizes the parameters that need to be set. In this example we’ll set them all by hand.

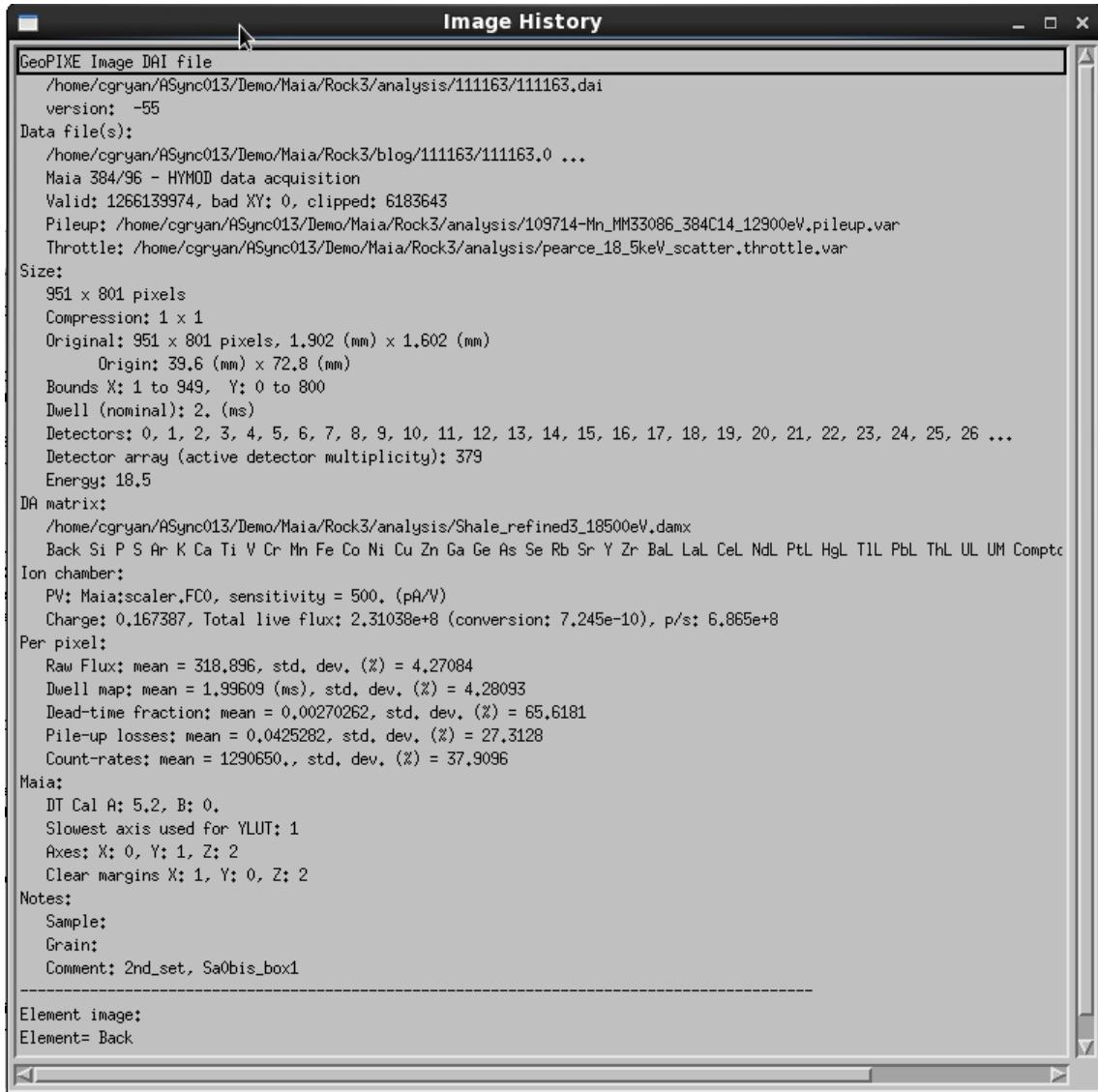
27. First on the *Sort EVT* window select “Image” mode, and the **“Maia 384/96 – HYMOD data acquisition”** device.
28. Files tab:
  - a. Select the first (111163.0.0) event file to process in dir **“Demo/Maia/Rock3/blog/111163”**.
    - i. Answer “yes” to using energy cal, pileup and throttle as found in the file header.
  - b. Select the last (111163.63) event file.
  - c. Select the pileup file:
    - i. The file-name comes from the header **“109714-Mn\_MM33086\_384C14\_12900.pileup.var”**.
    - ii. Navigate to where it is in your data **“Demo/Maia/Rock3/analysis”**.
  - d. Select the throttle file:
    - i. The file-name comes from the header **“pearce\_18\_5keV\_scatter.throttle.var”**.
    - ii. Navigate to where it is in your data **“Demo/Maia/Rock3/analysis”**.
  - e. Select the Linearize file (for the linearity adjustment):
    - i. The file-name comes from the header **“111156i-relinear-Hg-fit.linear”**.
    - ii. Navigate to where it is in your data **“Demo/Maia/Rock3/analysis”**.
  - f. Change the output file to the directory **“Demo/Maia/Rock3/analysis/111163/”**.
  - g. Enable “Flatten” to normalize out flux variation after processing.
29. Device tab:
  - h. The DTcalA dead-time calibration should be set from the header to 5.2 ns.
  - i. We use the default assignment of encoder axes: X=0, Y=1, Z=2, with 1 as the ‘slow axis’.
  - j. Clear just 1 border pixel in X and none in Y.

30. Scan tab:
  - k. The scan size (951 x 801 pixels; 1.902 x 1.602 mm) has been read from the first blog file header.
  - l. Leave 'X,Y compress' at 1, so we do not compress pixels.
31. Flux tab:
  - m. Select mode: "Indirect using Ion Chamber (synchrotron, with PV)".
  - n. Select the Flux IC details:
    - i. In future, these will come from the Metadata. For now see details in the XFM log file:
    - ii. "Demo/Maia/Rock3/logs/second-set\_15\_1111162.txt"
    - iii. Set "Sensitivity", "Units" = 500 pA/V.
    - iv. Enter the calibration "conversion" factor = 7.245e-10.
  - o. Ignore the zero "Charge" as this will be determined from the flux count as the data is processed.
32. DA/ E.Cal tab:
  - p. Select "Detector array" mode.
  - q. Select "SXRF" data type.
  - r. "Get" the energy calibration for all detector channels from file "111173\_refined\_fit.spec".
  - s. Select "Dynamic Analysis (DA)" projection mode.
  - t. Load DA matrix file "Shale\_refined3\_18500eV.damx".
33. Enable "Cluster" mode (if you using a full IDL license).
34. Click "Start" to commence processing. The file following file is written: "111163.dai"
  - u. Processing should take about 80 seconds to process the 6.3 Gbytes ( $1.3 \times 10^9$  events) in cluster mode.
  - v. The images appear in the main *GeoPIXE Image* window.
  - w. You can also view them in the *Multi-Image* window (menu "Display→Multi Image").



DA Multi-Images of run #111163 projected using the DA matrix "Shale\_refined3\_18500eV.damx".

35. Open the *Image History* window using menu "Window→Image Properties and History".
  - x. It shows parameters used to generate the images, as well as:
    - i. Detector number used.
      1. This should read a multiplicity of 379 in this case.
      2. If it shows multiplicity of 1, then you forgot "Detector Array" mode in *Sort EVT*.
    - ii. Dwell time encountered (this is the nominal dwell time per pixel).
      1. Note: Images have been corrected for actual dwell variation via pixel-by-pixel flux accounting using "Maia:Scaler.FC0".
    - iii. Total flux count and "charge" for the image (excluding any cleared margins/borders) and the estimated beam photons per second ("p/s").
    - iv. Ion chamber PV and sensitivity used.
    - v. Beam energy
    - vi. DA matrix file name used and element list.
    - vii. Maia device parameters selected: DTcal A, Axes, Slow Axis, Margins.
  - y. An alternative menu "Image Properties and Pixel Statistics" also includes pixel statistics (but this takes time for large images).



```

GeoPIXE Image DAI file
/home/cgryan/ASync013/Demo/Maia/Rock3/analysis/111163/111163.dai
version: -55
Data file(s):
/home/cgryan/ASync013/Demo/Maia/Rock3/blog/111163/111163.0 ...
Maia 384/96 - HYMOD data acquisition
Valid: 1266139974, bad XY: 0, clipped: 6183643
Pileup: /home/cgryan/ASync013/Demo/Maia/Rock3/analysis/109714-Mn_MM33086_384C14_12900eV.pileup.var
Throttle: /home/cgryan/ASync013/Demo/Maia/Rock3/analysis/pearce_18_5keV_scatter.throttle.var
Size:
951 x 801 pixels
Compression: 1 x 1
Original: 951 x 801 pixels, 1.902 (mm) x 1.602 (mm)
Origin: 39.6 (mm) x 72.8 (mm)
Bounds X: 1 to 949, Y: 0 to 800
Dwell (nominal): 2. (ms)
Detectors: 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26 ...
Detector array (active detector multiplicity): 379
Energy: 18.5
DA matrix:
/home/cgryan/ASync013/Demo/Maia/Rock3/analysis/Shale_refined3_18500eV.damx
Back Si P S Ar K Ca Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Rb Sr Y Zr BaL LaL CeL NdL PtL HgL TlL PbL ThL UL UM Comptc
Ion chamber:
PV: Maia:scaler.FCO, sensitivity = 500. (pA/V)
Charge: 0,167387, Total live flux: 2.31038e+8 (conversion: 7.245e-10), p/s: 6.865e+8
Per pixel:
Raw Flux: mean = 318,896, std. dev. (%) = 4,27084
Dwell map: mean = 1,99609 (ms), std. dev. (%) = 4,28093
Dead-time fraction: mean = 0,00270262, std. dev. (%) = 65,6181
Pile-up losses: mean = 0,0425282, std. dev. (%) = 27,3128
Count-rates: mean = 1290650., std. dev. (%) = 37,9096
Maia:
DT Cal A: 5,2, B: 0.
Slowest axis used for YLUT: 1
Axes: X: 0, Y: 1, Z: 2
Clear margins X: 1, Y: 0, Z: 2
Notes:
Sample:
Grain:
Comment: 2nd_set, Sa0bis_box1
Element image:
Element= Back

```

*Image properties, including Pixel statistics*

#### Tips:

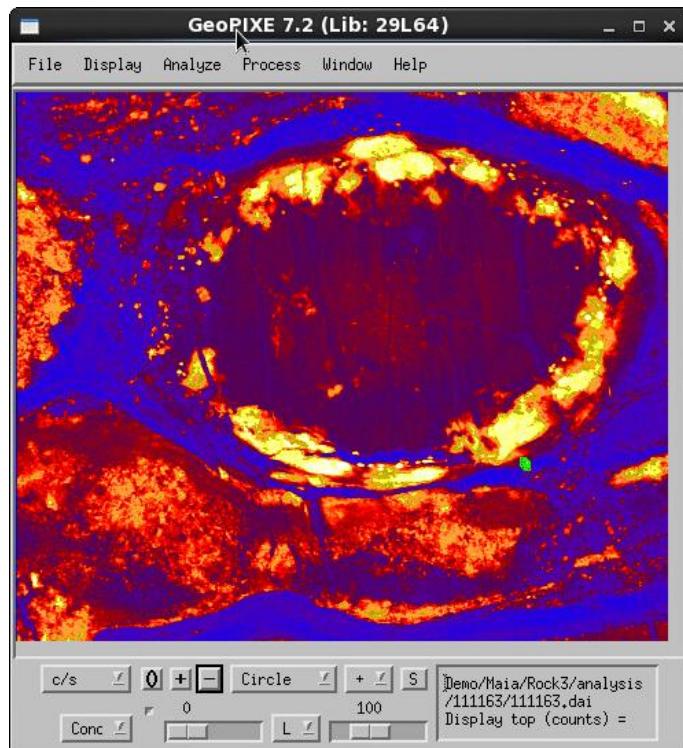
- The following parameters for a run can be found in the relevant “log” file for this run (“Demo/Maia/Rock3/logs/second-set\_15\_1111162.txt”):
  - Beam energy
  - Scan size, pixel step, nominal dwell time
  - Upstream IC scaler sensitivity (e.g. A1: KB Mon = scaler1.cts1.C).
    - Note: “Maia:Scaler.FCO” is the same as “scaler1.cts1.C”.
  - Note any warning about improper preamp ranges, etc.
- The increasing use of Metadata in blog file headers will obviate the need for viewing these log files.

#### Using the plugins to examine various diagnostic maps for these images

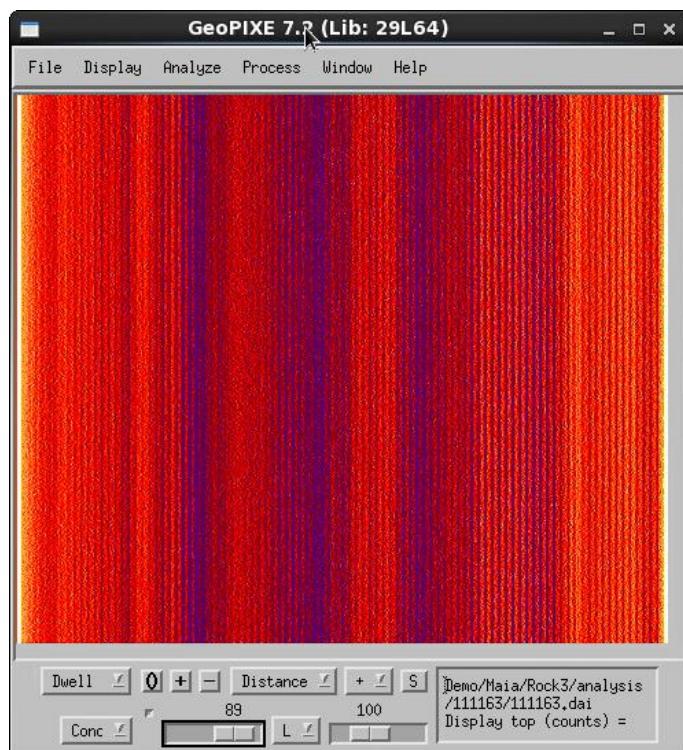
Plugins are small IDL routines written (some by users) to perform special image processing operations, or in this case, examine image parameters and maps.

Try the “Process→Plugins→ ...” menu on the main *Image* window to run these plugins:

36. Count-rate map (max display scale = 3.6 M/s)

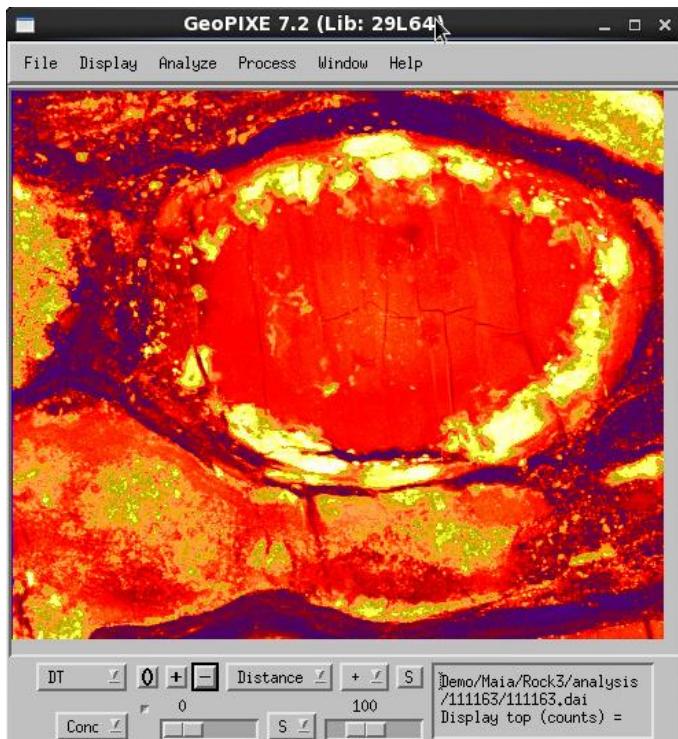


37. Dwell time map – shows the map of dwell times recorded in the BT values in ET records, summed over each pixel.



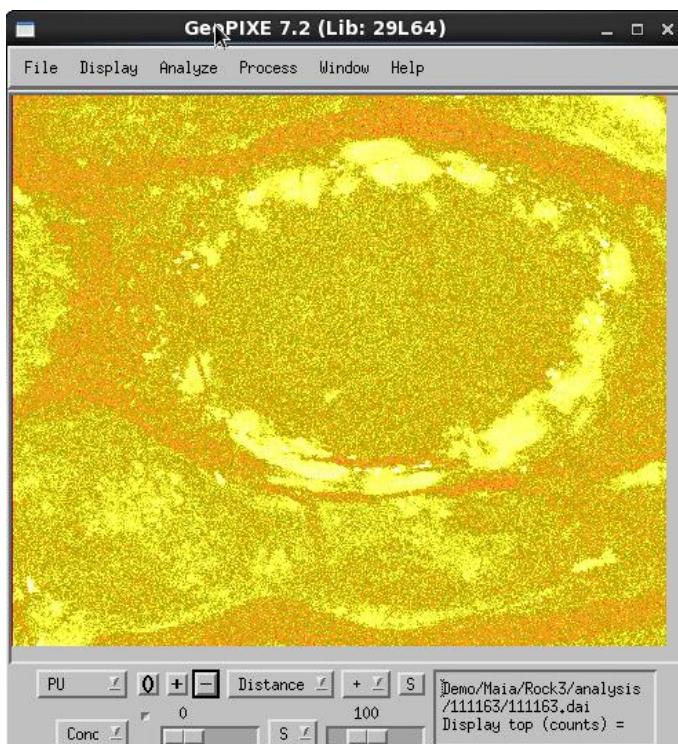
*Dwell time map, with exaggerated scale (89-100%) to show small ~1% variation in stage velocity (hence dwell) and longer delays at the end of X lines.*

38. Dead-time fraction map –



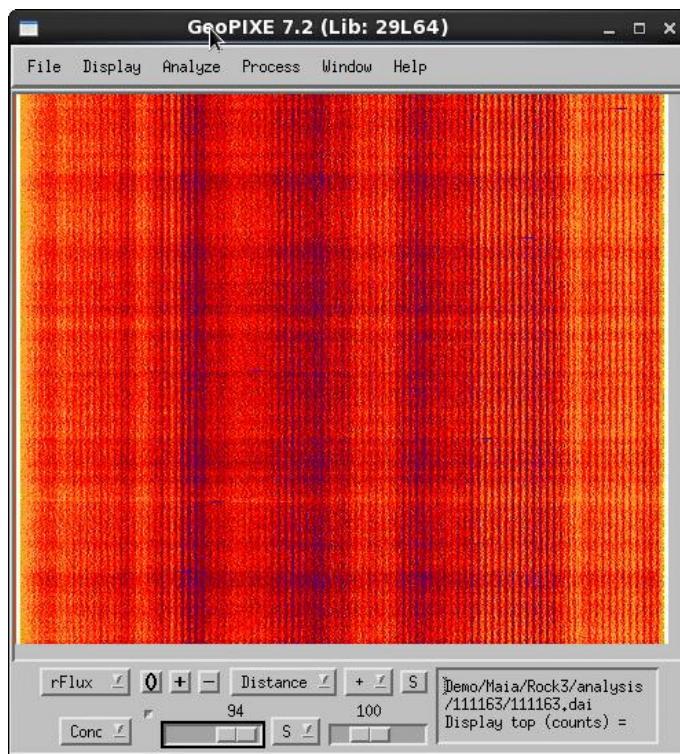
Dead-time fraction map (display max set to 1.1%, DT max ~3% in this case). The map follows count rate generally.

39. Pileup losses map –



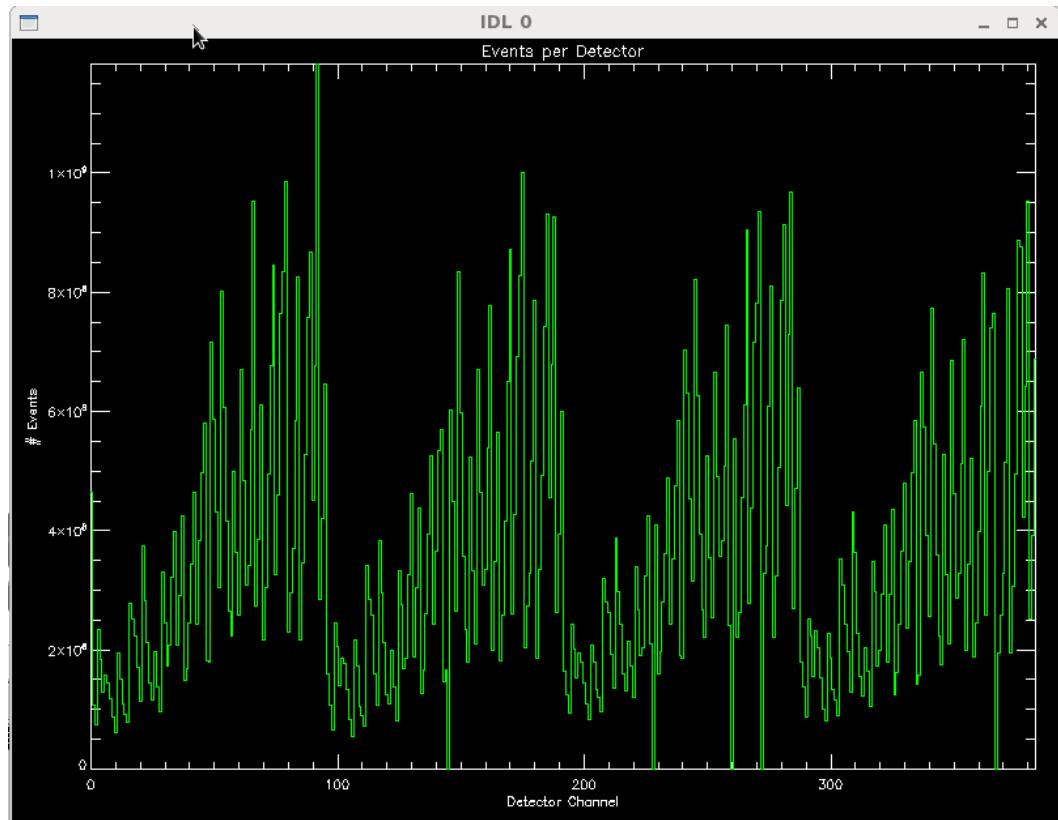
Pileup loss fraction map (display max 8%).

40. Raw flux map – Flux map before “flatten” applied to normalize it and element maps.



Raw flux map before “flatten” normalization, showing fine-scale variation in stage velocity at % level.

41. Detector counts histogram – Histogram of total counts accepted for each detector channel, after imaging, border clearing and pile-up rejection (if enabled).

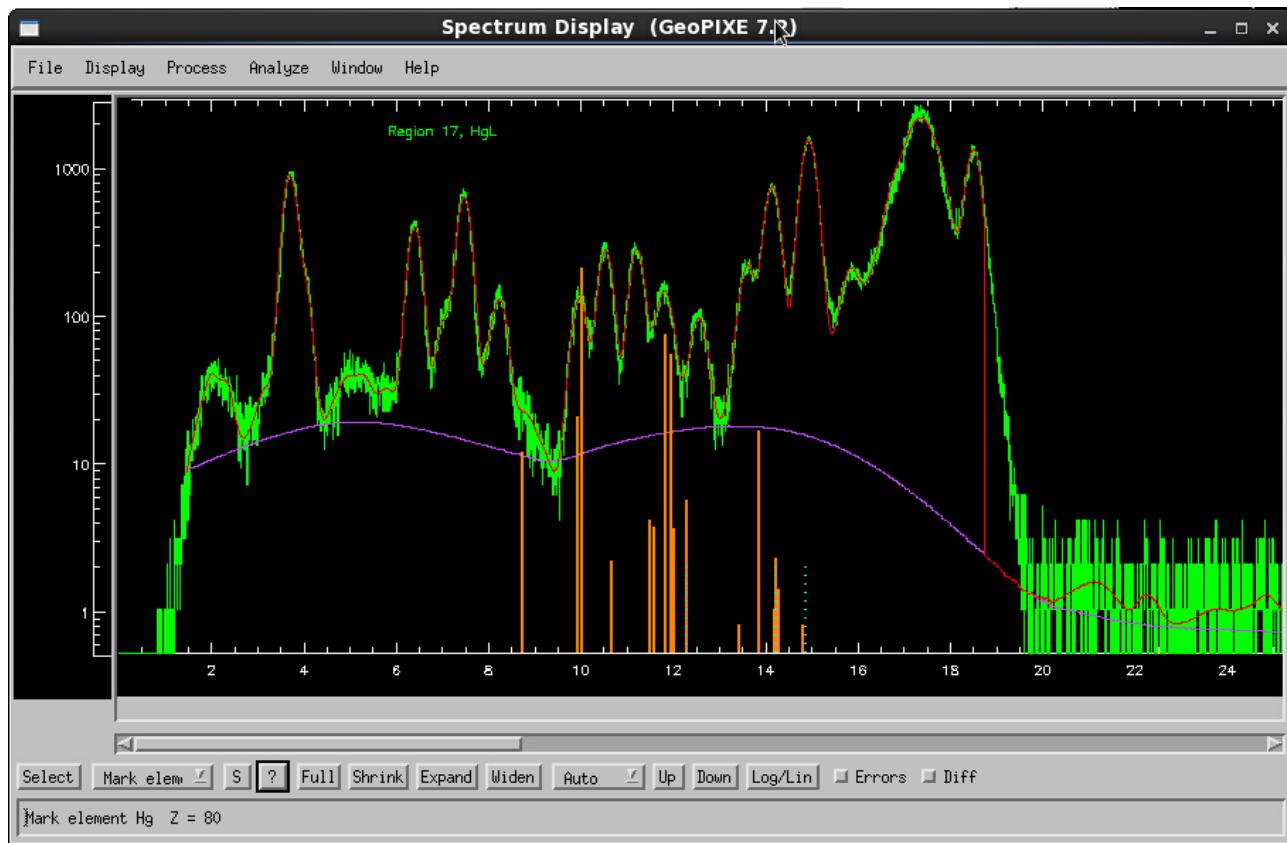


Detector histogram for this image, counting all events by detector accepted after PU and border reject.

## Example C4: Verifying Maia-384 detector image data

Once images have been created, they can be explored in the same ways as before (e.g. Examples 7, 8). This is illustrated here.

- Extract spectra from regions.
  - Close examination of spectra from these regions
  - Checking for correct or missing elements.
1. Continue with the last data-set, and image for Rock3 #111163.
  2. Open the *Image Regions* window.
  3. Load the regions file “111163-q1.region”.
    - a. If you are prompted for the DAMX file, navigate to “Shale\_refined3\_18500eV.damx”.
  4. On the *Image regions* window, click on “Update: All”.
    - a. This will re-apply these region pixels to the images and determine average concentrations in each.
    - b. Save the regions to file “111163-q1.region”.
  5. Select “array” spectrum extraction mode from the droplist.
    - a. Enable “Cluster” mode if you can use it.
    - b. Click on “EVT”.
    - c. Accept the default SPEC output file name, which matches the REGION file.
  6. When the extracted spectra appear in the *Spectrum Display* window, click on each row in the *Image Regions* table in turn to display the spectrum for each:
    - a. Examine each spectrum.
      - i. Does the red DA overlay account for the peaks observed?
      - ii. See if you do better with a fresh “Fit: One” applied to this spectrum with your present element list.
    - b. Examine Region #17, on a hot-spot in the “HgL” map:
      - i. Try fitting this spectrum with or without Hg L enabled.
      - ii. To temporarily disable HgL, simply right-click it; to re-enable it use left-click.
      - iii. Does any other element fit better than Hg ?
        1. e.g. Ge (already on), Br, WL, ReL?
        2. Look at the peaks at 10 and 11.7 keV.



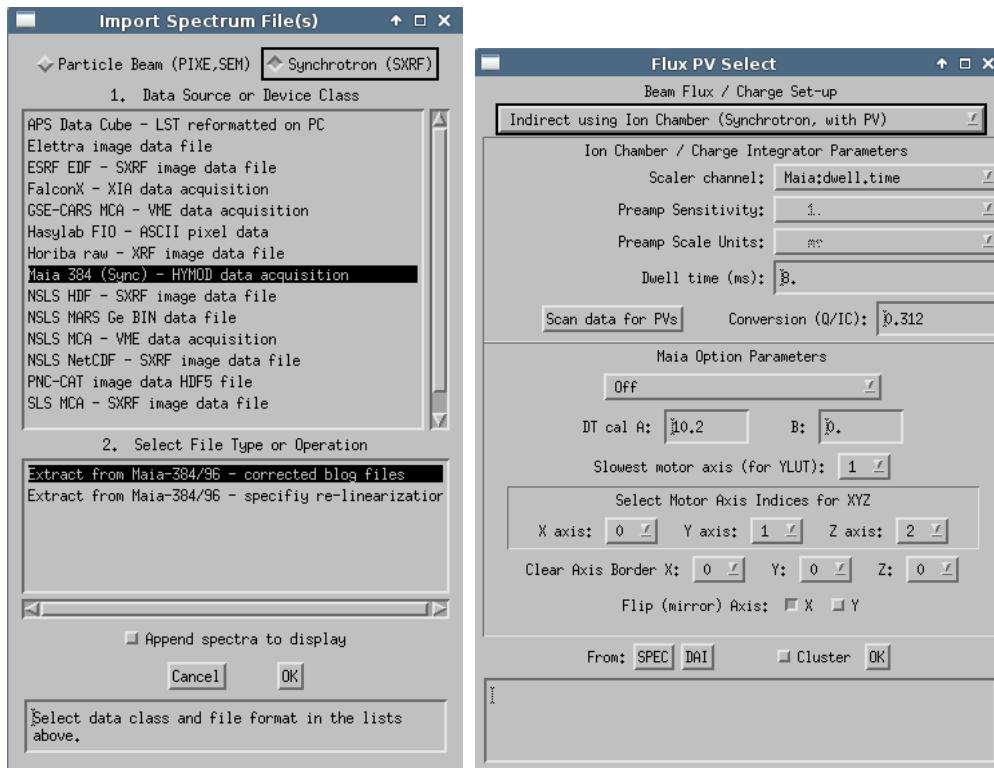
Region #17 selected on a hot spot in the “HgL” image, with Hg L lines marked.

## Example C5: Maia Mapper – MM 925 image data

- Importing spectra for all 384 detectors from the raw data files.
- Checking the ‘pileup’ field used for pileup rejection.
- Fitting a representative spectrum and building a DA matrix
- Calculation of X-ray yields
- Imaging using the DA method
- Extracting a sub-set of an image as a “Windowed sort” DAI file at full resolution

### Import Spectra and Calibrate Energy

- Import spectra from the raw Maia data files for run #925 (directory “**Demo/MM/analysis/**”, data courtesy of *Margaux Le Vaillant*, CSIRO) using the File→Import” menu on *Spectrum Display*.
  - Select “Synchrotron (SXRF)” data from the “Maia 384/96 – HYMOD data acquisition”.
  - Select file type: “**Extract from Maia-384/96 – corrected blog files**”.
  - Drag-select about 10 files in directory “Demo/MM/blog/925/”:
    - Click on “Numeric Ext” to order files numerically.
    - Select 925.0 (always select the .0 file to get header information)
    - Scroll down to about the middle of the file list.
    - Control-click and drag-select another ~10 files (e.g. 925.160 – 925.169).
    - (This approach gives a reasonable sampling of the middle of the image area.)
  - Direct output to directory “**Demo/MM/analysis/925/**”.
  - Select flux scaler and sensitivity:
    - The header metadata includes “dwell.time” as proxy flux and “ms” as the units of flux, which automatically selects a “scaler channel” of “Maia:dwell.time” and “Preamp Sensitivity” = 1, “Preamp Scale Units” = “ms”, which are now fixed.
    - Note also that the “Flip (mirror) axis X” is set ON automatically, because the Left side stage runs backwards.
    - Set the calibration “Conversion” factor to 0.478.
    - Click “OK” to close the Flux selection pop-up.
  - Selecting Pileup and Throttle:
    - (If your “Translation table” is functioning well, you may not be prompted for a pileup file as the Pileup file-path found in the file header will be translated into your local paths.)
    - If not, select Pileup=“900-wide.pileup.var” in the “config/384D19/pileup” directory.



*Import options and Flux PV settings, including the Maia Mapper effective flux scaler based on dwell (“Maia:dwell.time”) and the “conversion” factor. Note: Mirror X axis (for Left stage data)*

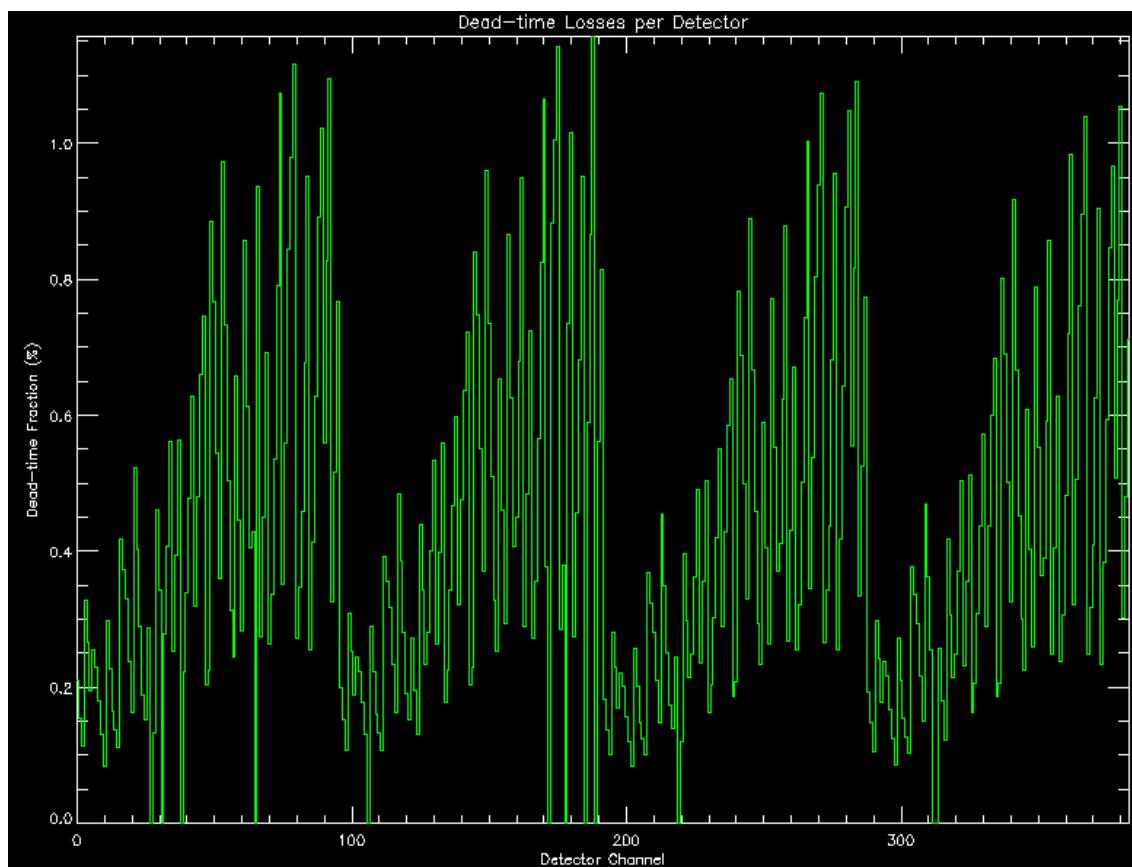
2. Open the *Spectrum Select* window and delete all XY and T spectra:
  - a. Select “all X,Y and T” on dropdown at right; click “Delete:” to delete all X,T and T spectra.
  - b. You may wish to drag the window much bigger to see a range of detectors and more columns, including the CalA and CalB coefficients.
  - c. Set the View markers on about 2 – 30 keV and click “Widen” (set high marker first, then low).
3. In this case the energy calibrations for all detectors were found in the header and set for you.
  - a. However, a better refined set of energy calibrations was constructed later.
  - b. To use the refined cal, use the *Spectrum Display* menu “Display->Get All Energy Cals” and select the file “926-E-cal.spec”, located in the “config/384D19/energy” directory.
  - b. This will use the energy calibrations for each detector channel in the file to set the energy Cals of the current spectra; any missing detector channels will be disabled.
4. Sum all spectra to form a single sum spectrum:
  - a. Click on “All” in Spectrum select to display all detectors.
  - b. Sum all spectra, mapped onto a single energy calibration using the menu “Process→Add (re-map cal)”.
  - c. Save this sum spectrum for later (e.g. “925-E-sum.spec”).

#### Using the plugins to examine various diagnostic plots for these spectra

*Plugins are small IDL routines written (some by users) to perform special spectrum processing operations, or in this case, examine spectrum parameters and histograms that are stored with the data.*

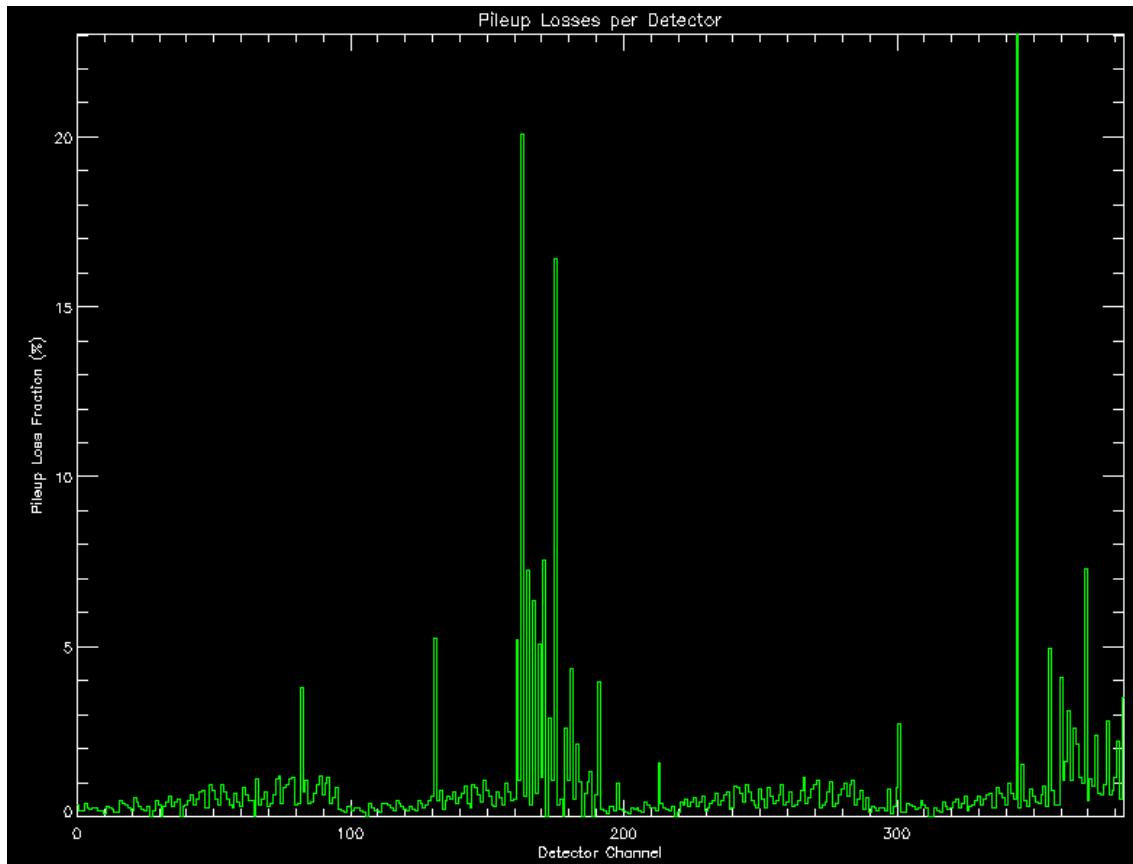
Try the “Process→User Plugins→ ...” menu on *Spectrum Display* window to run this plugins:

5. **Deadtime by detector:** Detector dead-time fraction histogram – shows DT per detector channel for this spectrum. This data-set had a count-rate less than 1 M/s, which is very low for Maia, so the dead-time averages only ~0.6%.



Dead-time per detector channel (max ~1.1%).

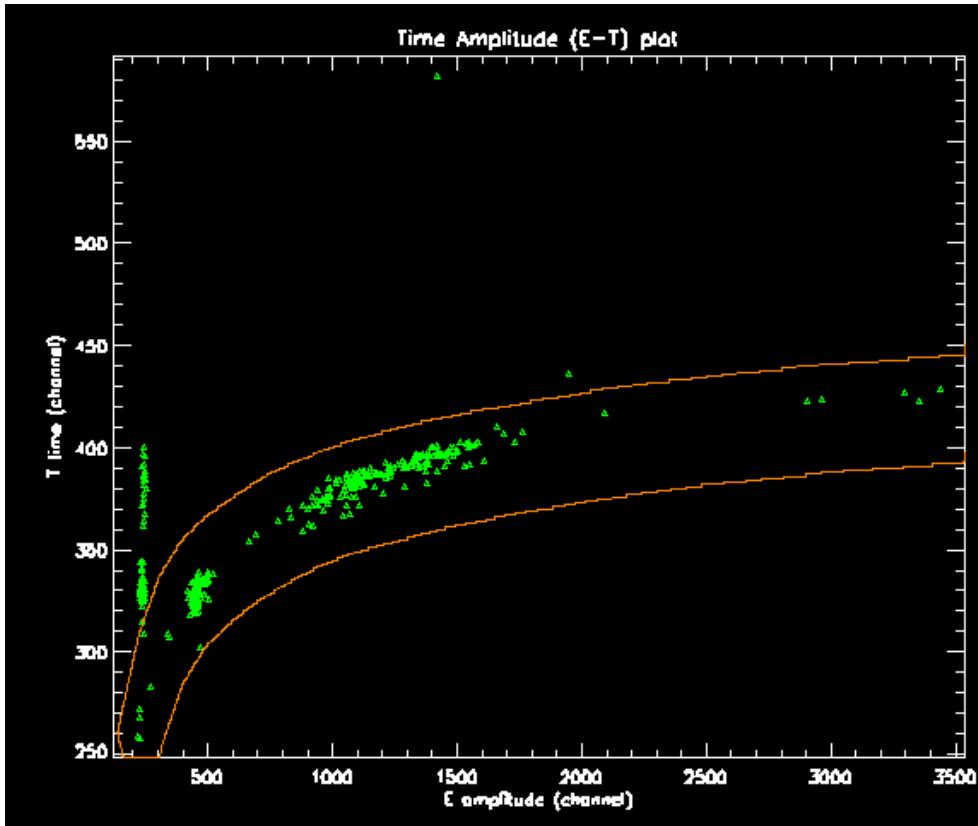
6. **Pileup by detector:** Detector by detector pileup loss fraction histogram – shows PU per detector channel for this spectrum. This shows high loss fraction for channels 0-32. Why is that?



*Pileup losses per detector channel.*

***Let's investigate these Pileup losses:***

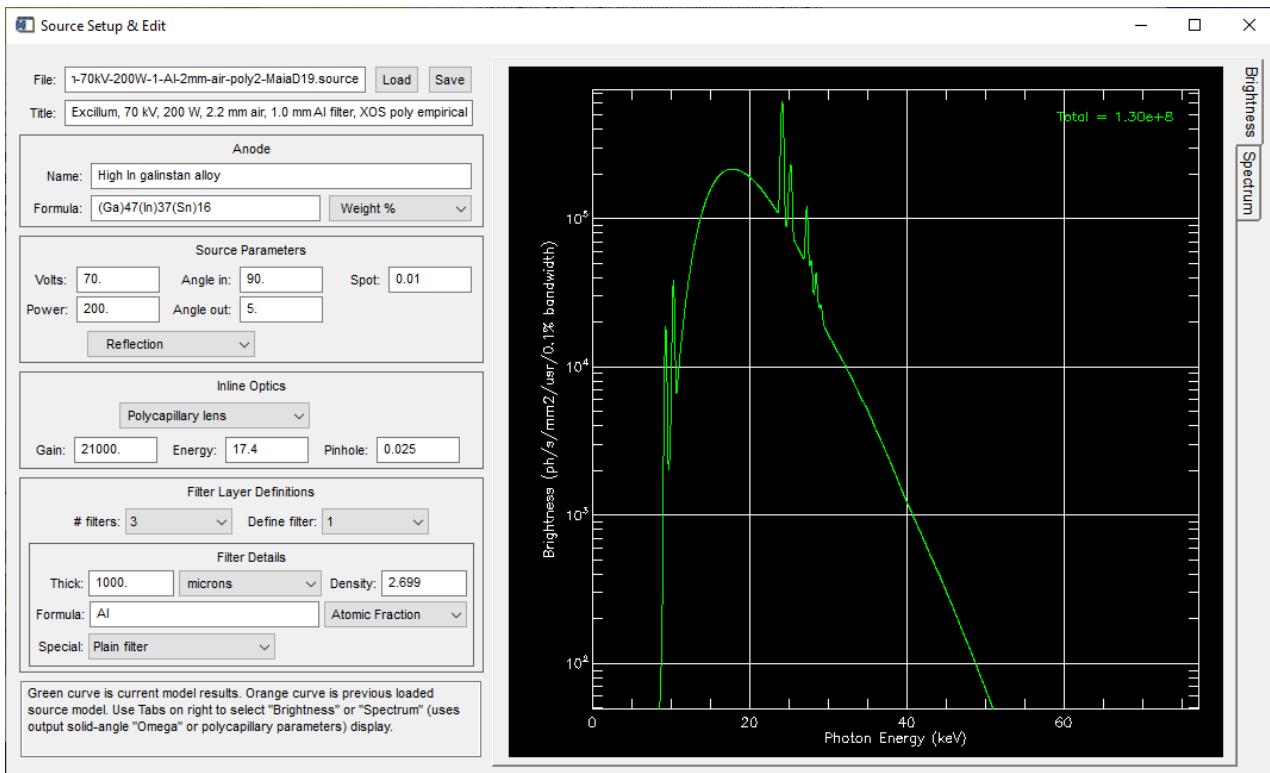
7. Open the *Time Amplitude* window and select 300,000 events and the blog files 925.0 and 925.1
  - a. Use “File->Extract Time Amp Maia” menu.
8. Overlay the pileup field from file “900-wide.pileup.var”
  - a. Use “Overlap Maia pileup field” menu.
9. Now step through by detector (click slider knob and use arrow keys):
  - a. Notice that the pileup field defined here is rather generous (perhaps too much so). However, it rejects some few *bona fide* events. It appears that the trimming between Maia channels was overdue for calibration.
  - b. Notice that the channels between 160-200 show E-T points that fall outside the pileup field mostly around channel ~220.
    - iii. This is why we get a high PU rejection % for these channels.
    - iv. This is a glitch in Maia that appears for some channels. They are rejected (so don't interfere with light elements) and this is why the PU rejection score is high for this channel (about 20% for channel 163).



Time amplitude plot for detector #163 showing “odd channel” glitches, which appear as lost events at low- $E$  and above the pileup field.

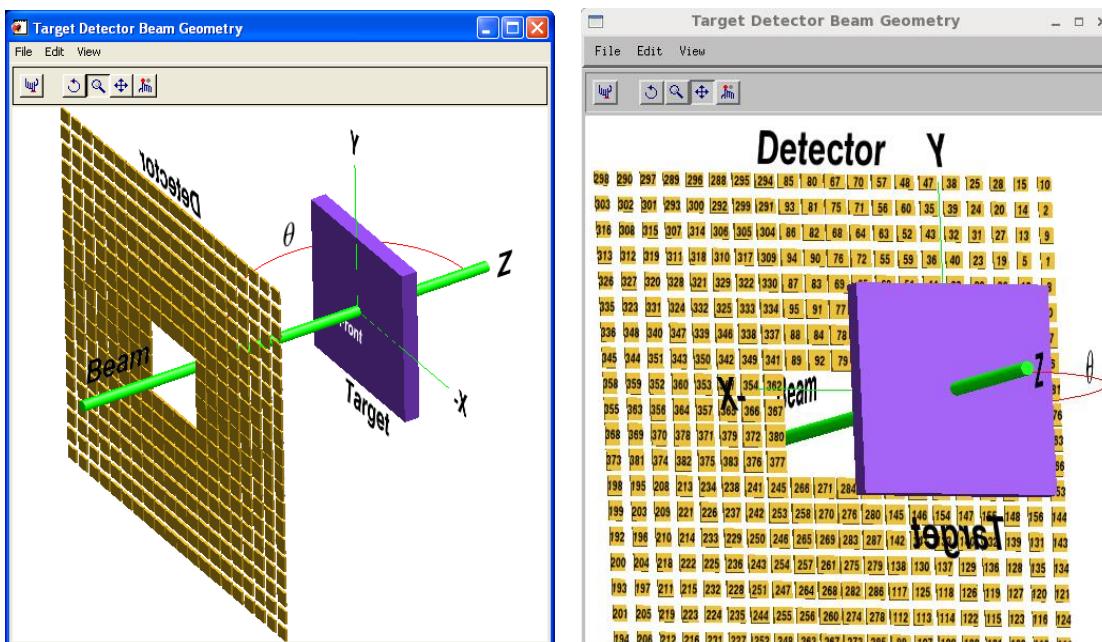
#### **Calculation of SXRF yields for the sample and detector array**

10. Load the sum spectrum saved previously (e.g. “925-E-sum.spec”).
11. Open the *X-ray Spectrum Fit* window and load the fit parameters from file “fifield-Pd-poly2.pcm”.
  - b. Notice that the “Detector” field in X-ray Spectrum Fit is greyed out and cannot be changed. This is because the Yield calculation has explicitly specified an array detector. Let’s do that now ...
12. Open the *PIXE/SXRF Yield Calculation* window by clicking on “New” next to “Yields” in *X-ray Spectrum Fit*.
13. Open the file “rock-slab4-poly2.lcm”. This specifies a one layered sample with:
  - a. a “Photon (continuum)” beam, loaded from file “Excillum-70kV-200W-1-Al-2mm-air-poly2-MaiaD19.source”,
  - b. a thick section (8 mm) of a nominal rock matrix (20% Fe<sub>2</sub>O<sub>3</sub>, remaining SiO<sub>2</sub>),
  - c. a target-detector geometry with the detector at 180° and no target rotation (alpha, beta = 0°).
  - d. an array detector “Maia Mapper 384D 10 mm v1”, which is the new Maia 384D19.
14. Click on “New” under “Beam particle” and load file “Excillum-70kV-200W-1-Al-2mm-air-poly2-MaiaD19.source”.
  - a. This shows the source model, which includes:
    - i. bremsstrahlung and characteristics X-rays for 70 kV and 200W power
    - ii. generated from the Ga (47%), In (37%), Sn (16%) liquid alloy anode
    - iii. transmitted through the XOS polycapillary (gain = 21,000 at 17.4 keV)
    - iv. hardened passing through 100 μm Be, 2.2 mm air and a 1.0 mm Al filter.
15. To calculate yields, click on “Calculate Yields”. This has 2 passes.
  - a. The first does a complete yield model; calculation for all elements throughout the sample structure including secondary fluorescence enhancement for a generic “central” detector element.
    - i. The second pass then performs the modelling for all other detectors to calculate relative sensitivity factors relative to the central detector, but without secondary fluorescence; the secondary fluorescence enhancement for each is taken from the first pass



Excillum source spectrum model with polycapillary transmission and hardening filter

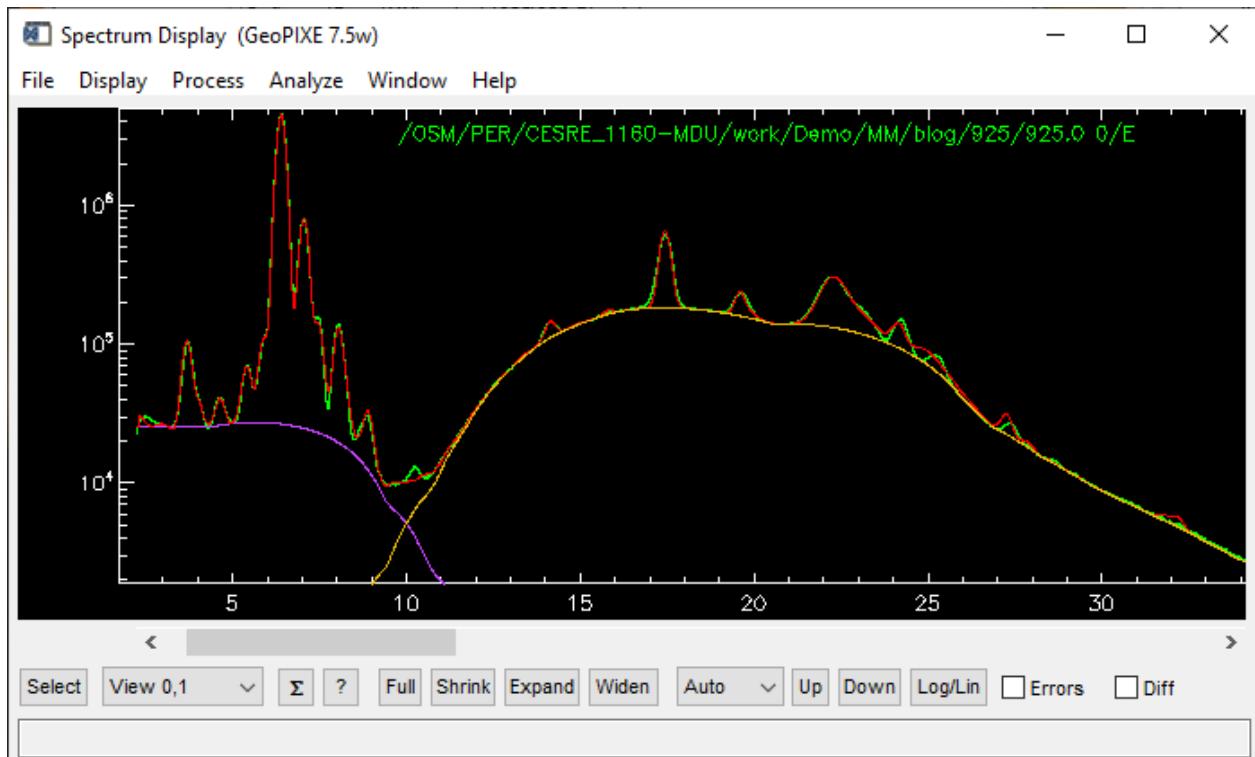
16. Click on the “?” button under Detector to view the geometry in 3D.
  - a. Click in the window and drag, to rotate the 3D model.
  - b. This verifies that the detector is looking at the front side of the sample and in the case of Maia-384, the beam comes through the detector.
  - c. Rotate the view and zoom in on the face of the detector. In this way we see that detector numbers 10, 298, ... are in the outer corners, while detectors 245, 341, 284, 79, ... are around the central opening.
    - i. This detector layout can also be viewed using the “Array ...” button on the *Spectrum Select* window.



17. To finish, select the filename for the yields file (e.g. “rock-slab4-poly2.yield” in the local “analysis” directory).
  - a. The set-up details for this panel need to be saved separately using the Save button at the top (e.g. to file “rock-slab4-poly2.lcm” in the local “analysis” directory).

### **Fit the sum spectrum and build the DA matrix**

18. Continuing in the X-ray Spectrum Fit window, with set-up “fifield-Pd-split-back-poly2.pcm” loaded ...
  - a. Notice that the background is split into two components, with “Split Back” enabled, to cater for separate fitting of the low energy background and the source bremsstrahlung hump between 10-40 keV.
19. Set the View range to 2.3-37 keV (click on “Use View”) and enable variation of the peak widths and energy calibration in the fit by checking “FWHM” and “Cal On” in the main (“Setup”) fit panel in X-ray Spectrum Fit.
20. Click on “Fit: One” in X-ray Spectrum Fit to fit the spectrum and refine the energy calibration.
  - a. This is a complex data-set with 33 elements, U includes both L and M lines.
21. Disable “Cal On” and “FWHM” and open the View range out to 1.5-27 keV (to include the pileup peaks).
  - b. Fit again, which will also include the scatter and an approximation to pileup contributions.
22. If a good fit is obtained, then click on the “Generate DA matrix” button to build and save a DA matrix file “fifield-Pd-split-back-poly2.damx”.



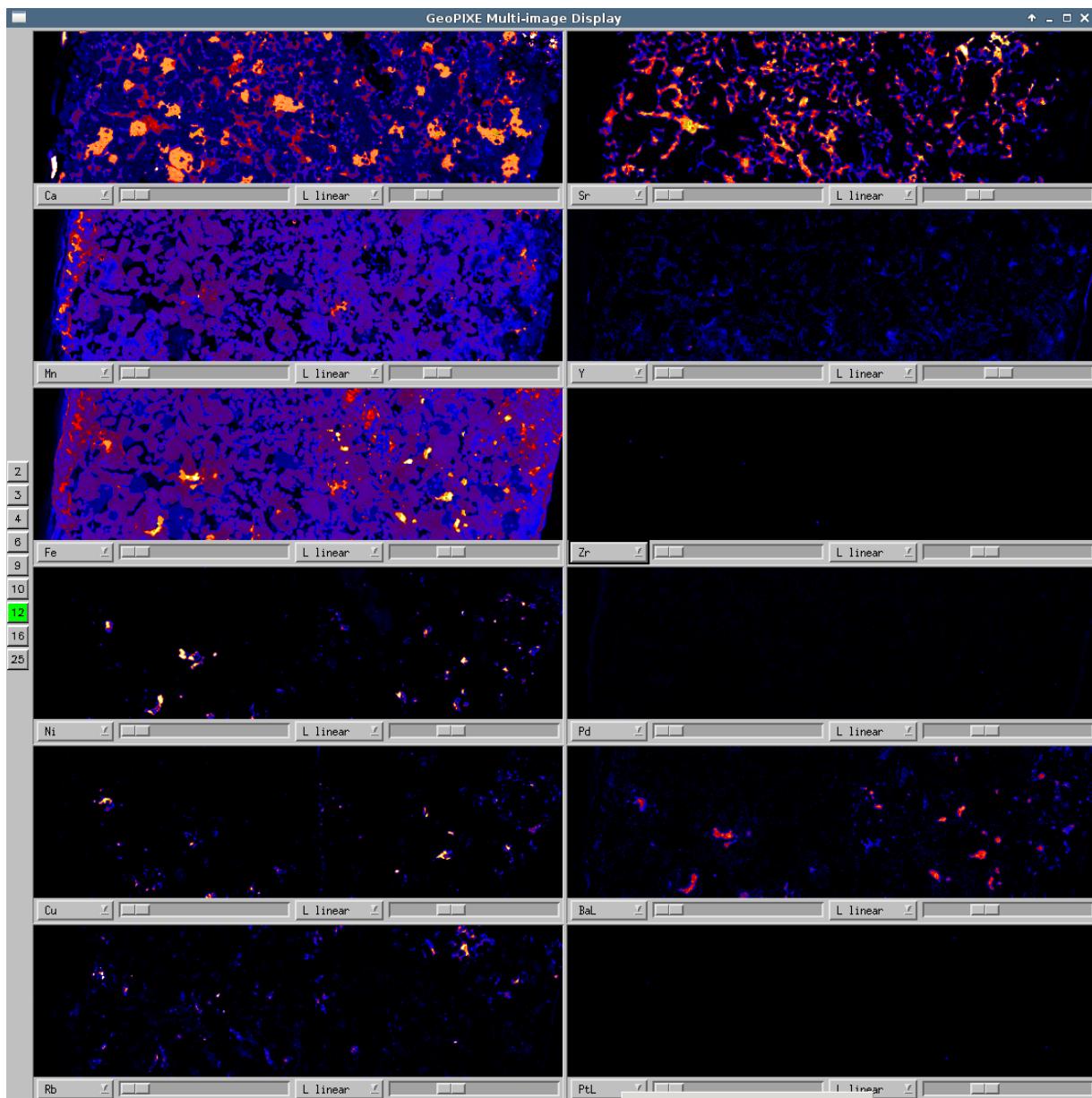
Sum spectrum for #925 with overlays for fit (red), background (violet, orange). Data courtesy of Margaux Le Vaillant, CSIRO.

### **Using this DA matrix to generate images**

Often an existing image can be loaded into Sort EVT to provide a template for a new scan (using “From DAI” button), which minimizes the parameters that need to be set. In this example we’ll set them all by hand.

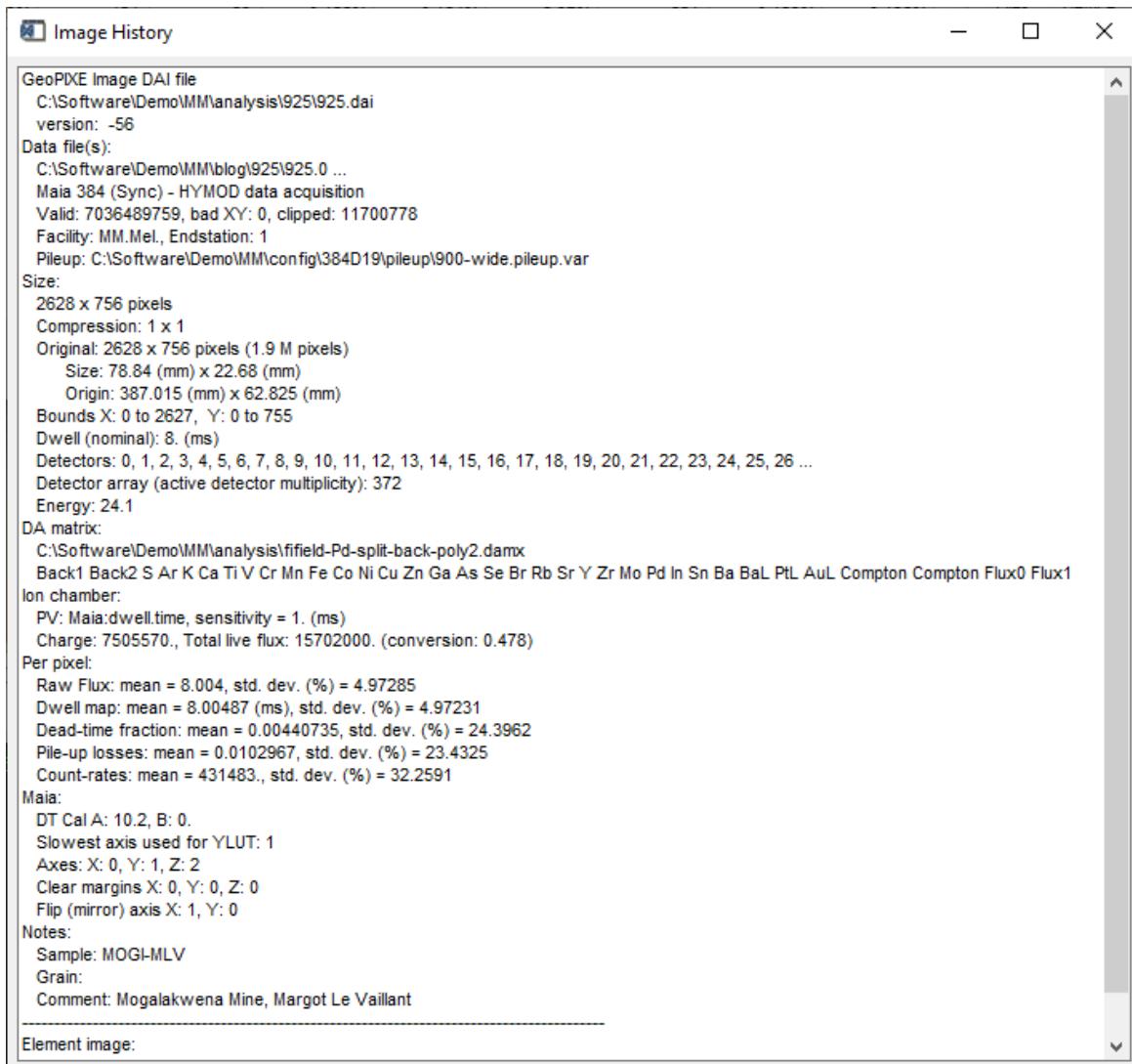
23. First on the Sort EVT window select “Image” mode, and the “**Maia 384/96 – HYMOD data acquisition**” device.
24. Files tab:
  - a. Select the first (925.0.0) event file to process in dir “**Demo/MM/blog/925**”.
    - ii. Answer “yes” to using energy cal, pileup as found in the file header.
  - b. Select the last (925.291) event file or leave this field blank to use all files.
  - c. Select the pileup file:
    - i. The file-name comes from the header “900-wide.pileup.var”.
    - ii. Navigate to where it is in your data “**Demo/MM/config/384D19/pileup**”.
  - d. Change the output file to the directory “**Demo/MM/analysis/925/**”.
  - e. Enable “Flatten” to normalize out flux variation and “Cluster” for parallel processing.
25. Device tab:
  - f. The DTcalA dead-time calibration should be set from the header to 10.2 ns.
  - g. We use the default assignment of encoder axes: X=0, Y=1, Z=2, with 1 as the ‘slow axis’.
  - h. Note that “Flip (mirror) X axis” has been enabled (MM Left stage has reversed X motion).
26. Scan tab:
  - i. The scan size (2638 x 756 pixels; 78.8 x 22.7 mm) has been read from the first blog file header.

- j. Leave ‘X,Y compress’ at 1, so we do not compress pixels.
- 27. Flux tab:
  - k. For Maia Mapper this is set to: “Indirect using Ion Chamber (synchrotron, with PV)”.
  - l. With “maia:dwell.time” scaler channel, and 1 ms sensitivity/ range.
  - z. Enter the calibration “conversion” factor = 0.478.
  - m. Ignore the “Charge” as this will be determined from the flux count as the data is processed.
- 28. DA/ E.Cal tab:
  - n. Select “Detector array” mode.
  - o. Select “SXRF” data type.
  - p. “Get” the energy calibration for all detector channels from file “config/384D19/energy/926-E-cal.spec”.
  - q. Select “Dynamic Analysis (DA)” projection mode.
  - r. Load DA matrix file “fifield-Pd-poly2.damx”.
- 29. Enable “Cluster” mode (if you are using a full/RT IDL license).
- 30. Click “Start” to commence processing. The file following file is written: “925.dai”
  - s. Processing should take about 380 seconds to process the 29 Gbytes ( $6 \times 10^9$  events) in cluster mode.
  - t. The images appear in the main *GeoPIXE Image* window.
  - u. You can also view them in the *Multi-Image* window (menu “Display→Multi Image”).



DA Multi-Images of run #925 projected using the DA matrix “fifield-Pd-poly2.damx”.

31. Open the *Image History* window using menu “Window→Image Properties and History”.
- v. It shows parameters used to generate the images, as well as:
    - i. Detector number used.
      - 1. This should read a multiplicity of 372 in this case.
      - 2. If it shows multiplicity of 1, then you forgot “Detector Array” mode in *Sort EVT*.
    - ii. Dwell time encountered (this is the nominal dwell time per pixel) = 8 ms..
      - 1. Note: Images have been corrected for actual dwell variation using “Maia:dwell.time”.
    - iii. Total flux count and “charge” for the image (excluding any cleared margins/borders).
    - iv. Beam energy (nominal In K $\alpha$  for Maia Mapper).
    - v. DA matrix file name used and element list.
    - vi. Maia device parameters selected: DTcal A, Axes, Slow Axis, Margins.
  - w. An alternative menu “Image Properties and Pixel Statistics” also includes pixel statistics (this takes time for large images).
    - a. Remember to close this window, as it will continue to get triggered for updates as you select elements to display in the *Image Display* window.



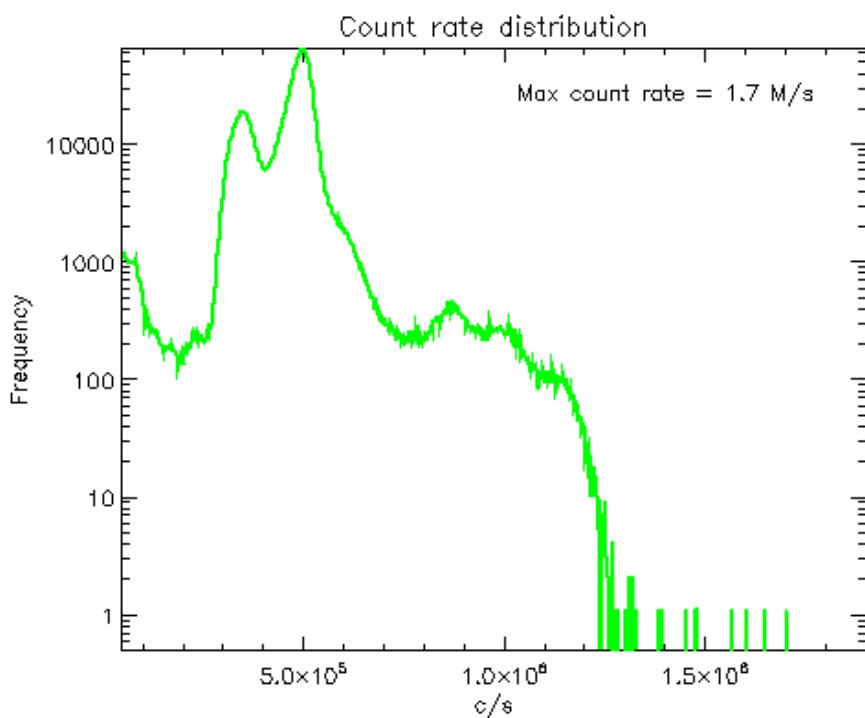
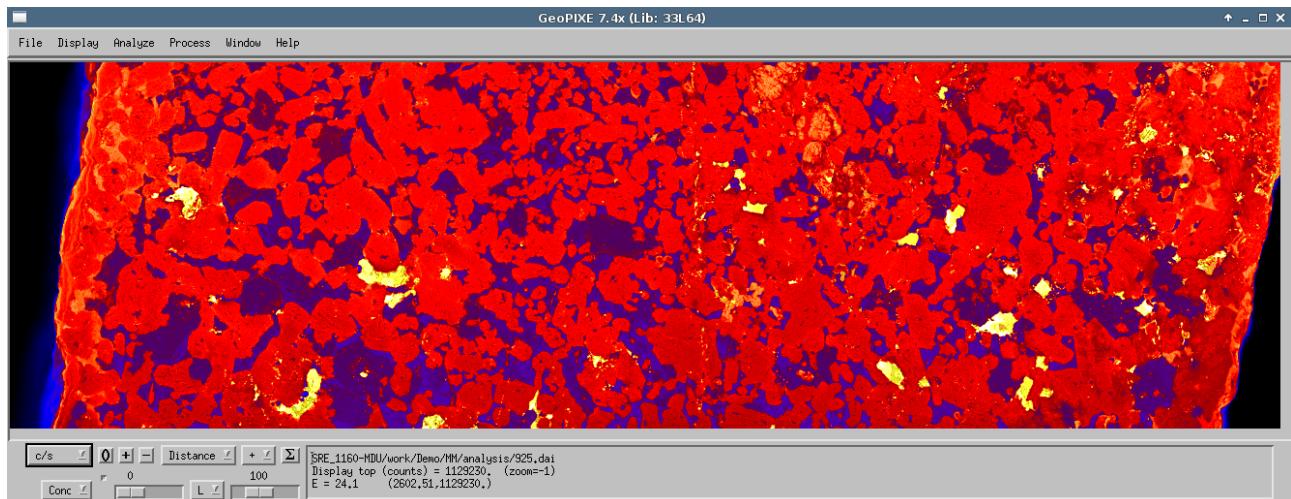
*Image properties, including Pixel statistics*

#### **Using the plugins to examine various diagnostic maps for these images**

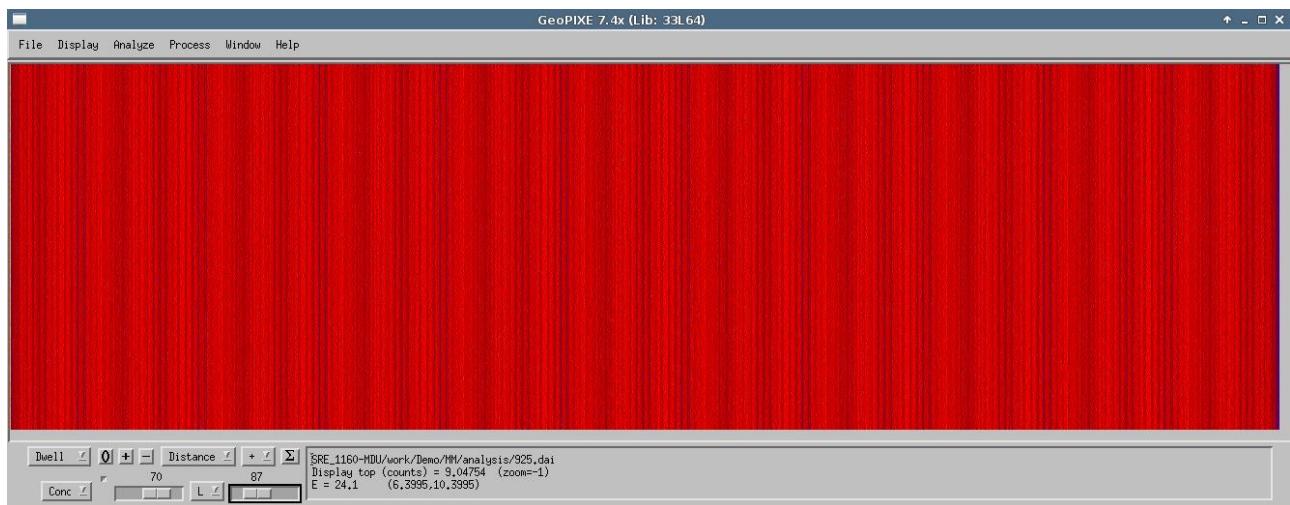
Plugins are small IDL routines written (some by users) to perform special image processing operations, or in this case, examine image parameters and maps.

Try the “Process→Plugins→ ...” menu on the main *Image* window to run these plugins:

32. Count-rate map (max display scale = 1.13 M/s)



33. Dwell time map – shows the map of dwell times recorded in the BT values in ET records, summed over each pixel.

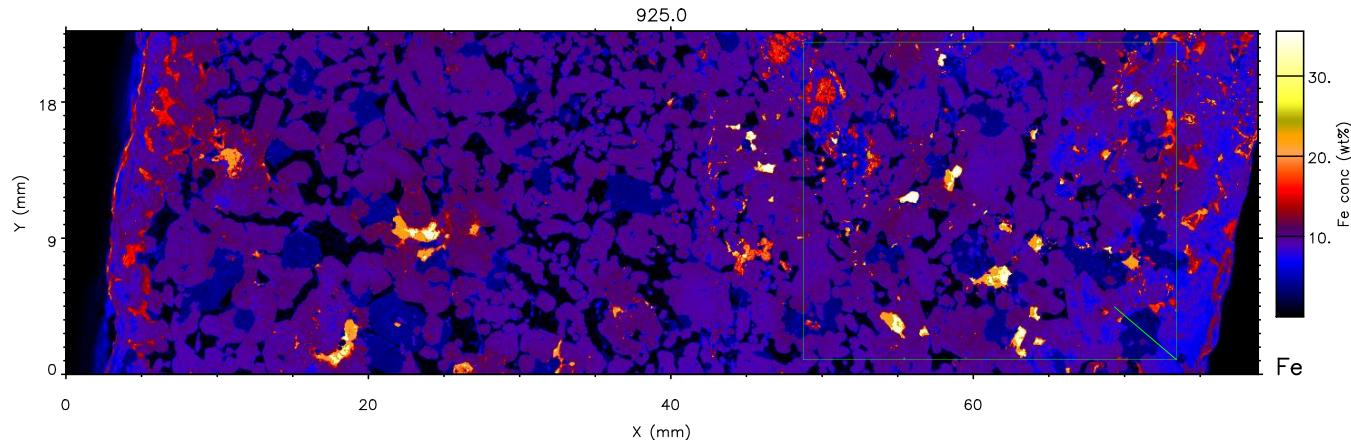


Dwell time map, with exaggerated scale (70-87%) to show small ~1% variation in stage velocity (hence dwell) and longer delays at the end of X lines.

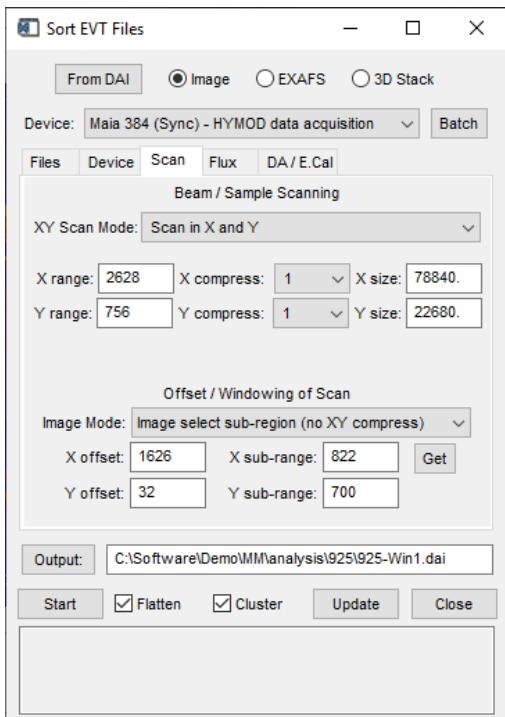
#### **Extract a sub-set of an image at full resolution**

A sub-set of a scan area can be extracted or sorted from the data-set at full resolution using the “Windowed sort” option.

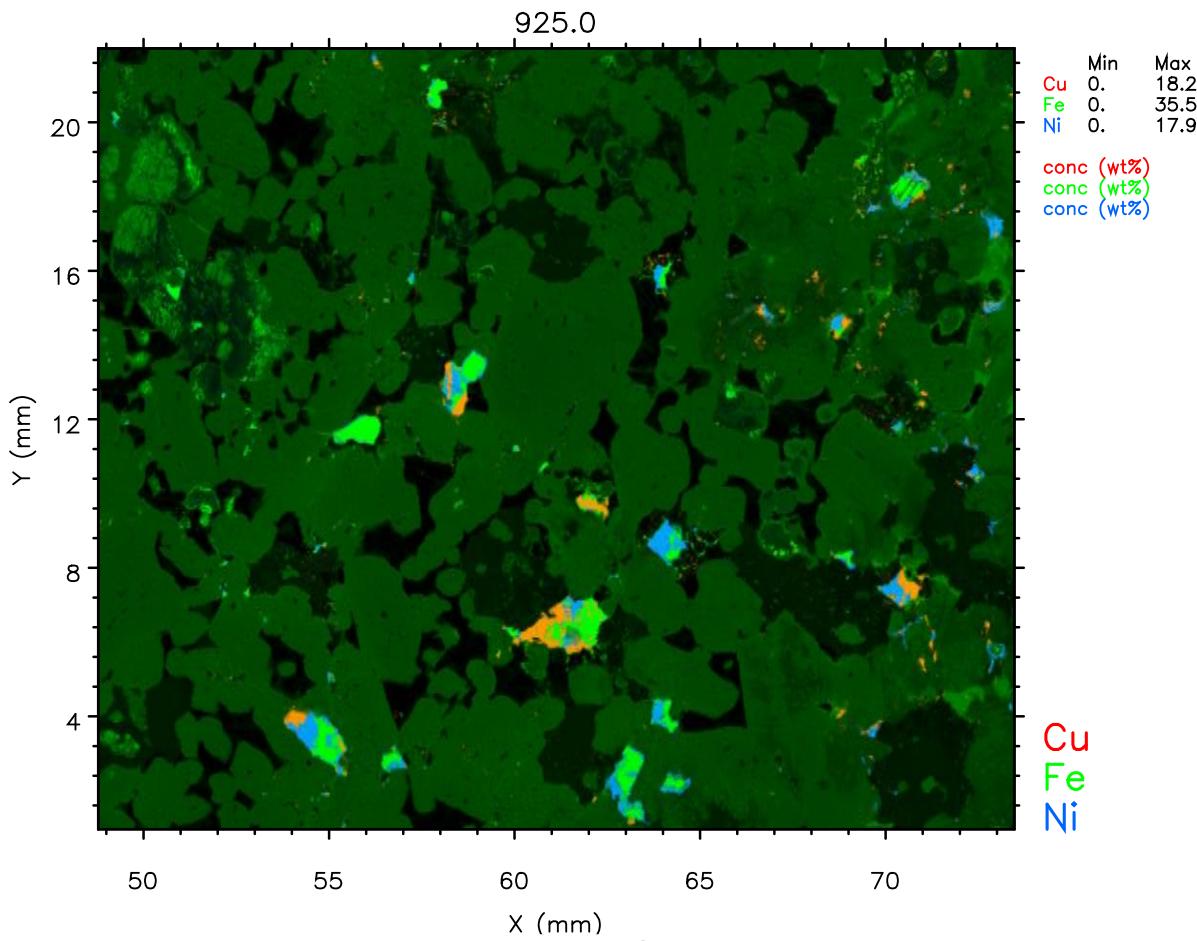
1. Load the image DAI file for this data-set (“MM/analysis/925/925-Bi-x.dai”) back into the *Sort EVT* window.
2. Make sure all file paths are correct (blob files, pileup, damx).
3. Open the *Image Regions* window and load the region from file “925-Win1.region”). The region looks like this …



4. On the Scan tab of the *Sort EVT* window, select “Image select sub-region” in the “Offset/WINDOWING of Scan” drop-list.
5. Click on the “Get” button, which will retrieve the current box shape region selection shown on the Image window.



- Change the output file name to “925-Win1.dai” on the Sort EVT window, and click “Start”, which will sort this data again, but only within this region. It will ignore any XY compress settings and make a full-resolution DAI file, only for this windowed region.



## D. XFM Ion Chamber Calibration

### Example D1: Maia-384 – Mn calibration foil

- Importing spectra for all 384 detectors from the raw data files.
- Applying energy calibrations to all detectors.
- Fitting a representative spectrum and determining the calibration “conversion factor”
- Calculation of X-ray yields
- Building a DA matrix and imaging (not an exciting case, perhaps)
- Verifying image values using a Region.

#### Import Spectra and Calibrate Energy

1. Import spectra from the raw Maia data files for run #112658 (directory “**Demo\Maia\Standard\blog\112658\**”) using the “File->Import” menu on *Spectrum Display*.
  - a. Select “Synchrotron (SXRF)” data from the “Maia 384/96 – HYMOD data acquisition” data source and the “Extract from Maia-384 - corrected blog files” operation.
  - b. Select all blog files 112658.\*
  - c. Select the directory “**Demo\Maia\Standard\analysis\112658\**” as the output path.
2. On the “Flux PV select” pop-up:
  - a. This data-set has metadata built-in, so the following are set automatically:
    - i. IC scaler channel = Maia:scaler.FC0
    - ii. Preamp sensitivity/units = 2 nA/V
    - iii. DT cal parameters A = 5.2, B = 0 (ns)
  - b. For now, entry a “Conversion” factor of 1.0
3. Pileup file:
  - a. If you have your “Translation Tables” in order, this should not prompt at all.
  - b. If it does, select the pileup file = “109714-Mn\_MM33086\_384C14\_12900eV.pileup.var”.
4. Throttle file:
  - a. If you have your “Translation Tables” in order, this should not prompt at all.
  - b. If it does, select the throttle file = “pearce\_18\_5\_scatter.throttle.var”.
5. The *Spectrum Display* shows an overlay of E, X, Y and T spectra for all 384 detector channels.
  - a. E – energy spectrum for each detector
  - b. X, Y – projections for each detector on X and Y axes
  - c. T – time-over-threshold spectrum for each detector
6. Open the *Spectrum Select* window to remove XY and T spectra (so we just have the E spectra)
  - a. Select “all X,Y and T” in the dropdown and click on “Delete:”.
  - b. Load energy calibrations for all channels using *Spectrum Display* menu ”Display->Get All energy channels”
    - i. Select the file “111173\_refined\_fit.spec”.
  - c. Click on “Full” to see the full spectra.
  - d. See prominent Mn K lines and the Compton (17 keV) and elastic (18.5 keV) scatter peaks.
7. Sum all detectors together using the menu “Process→Add (remap cal)”.
8. Save the spectrum for now as “112658 -E-sum.spec” in directory “**Demo\Maia\Standard\analysis\112658\**”.

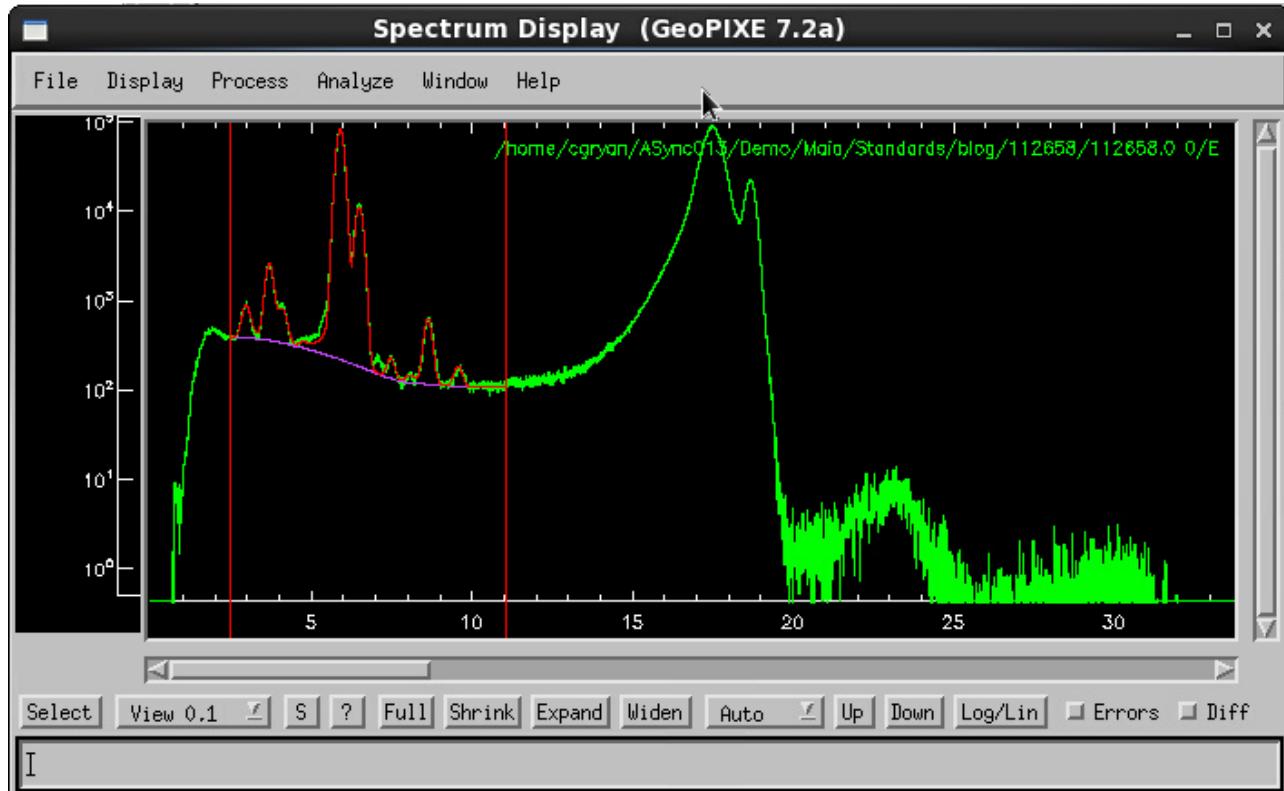
#### Question

- Repeat the above Import for #112663 and Get All Energy Cals.
- Why is the overlay of all detector channel spectra look so bad in this case? The Cals don’t seem to have worked ...
- Hint: Use **blogbrowse** to check if **gaintrim** was enabled (Var\_Val records).

#### Fit the spectrum, determine flux “conversion” factor and build the DA matrix

9. Open the X-ray *Spectrum Fit* window.
  - a. Load file “**Mn\_MM33087\_384C14\_18500eV.pcm**” from dir “**Demo\Maia\Standard\config\384C14\standards\18500eV\**”.
10. Open the *Yield Calculation* window:
  - a. Load file “**Mn\_MM33087\_384C14\_18500eV.lcm**” from the same dir .
11. This shows the settings for our Mn foil:
  - a. 18.5 keV photons
  - b. Theta = 180, Phi = 0, alpha = 0, beta = 0
  - c. Detector “Array” enabled and “AS Maia 384C 10 mm v6” detector array selected.
  - d. Set-up for one layer of  $52.2 \mu\text{g}/\text{cm}^2$  thick Mn.
12. Fit the spectrum using these tips:

- a. Fitting the whole spectrum should work, but using the range 2.5 – 11 keV may be more reliable.
- b. Avoid enabling the elements next to Mn (Cr, Fe) as they aren't there and their inclusion may make Mn less reliable if the energy calibration changes a little.



Fit to the 112663 -E-sum spectrum for the Mn foil including pileup peaks.

13. Open the *Fit Results* window (menu “Window→X-ray spectrum fit” on *Spectrum Display*) and clear the list.
14. Fit the spectrum again and note the Mn concentration show in *Fit Results*.
  - a. We want this to read 100% Mn in the foil by changing the “charge” value (“Q” entry box) in the fit window.
  - b. **Tip:** Enter 1.0 for charge in the fit window, hit <return> and re-fit. If the Mn conc shows as 1.86%, then enter 0.01861 for charge and fit again. It should now show 100% Mn.
15. Open the “Flux to Charge” pop-up by clicking on “?” next to the charge entry box.
  - a. Note the new “Conv” factor of **1.968e-10**.
  - b. This is your new calibration factor for the current experiment and conditions (e.g. energy, slits, etc.).
16. Generate a DA matrix and save it for later:
  - a. To file “**Mn\_MM33087\_384C14\_18500eV.damx**” in dir “**Demo\Maia\Standard\analysis\112658\**”.

#### **Generate the DA images and probe concentrations in regions of the image**

17. Open the *Sort EVT* window and select Device = “**Maia 384/96 – HYMOD data acquisition**”.
18. Select the following entries by panel (many are set automatically from the header from 1-Aug-2015):
  - a. Files:
    - i. First file = “Demo\Maia\Standard\blog\112658\112658.0”.
      1. Answer “yes” to inherit Ecal, Pileup and Throttle files from blog header.
    - ii. Last file = < leave it blank, which means do all >
    - iii. Pileup file:
      1. Set from header to file = “109714-Mn\_MM33086\_384C14\_12900eV.pileup.var”.
      2. If you have your “Translation Tables” in order, this should not prompt later.
    - iv. Throttle file:
      1. Set from header to file = “pearce\_18\_5\_scatter.throttle.var”.
      2. If you have your “Translation Tables” in order, this should not prompt later.
  - b. Scan:
    - i. Scan dimensions should be set automatically from header.
  - c. Flux:
    - i. As before, this should be automatically from header now:
      1. Maia:scaler.FC0

2. 2 nA/V
  - ii. Conv 1.968e-10.
  - d. DA/E.Cal:
    - i. Detector array mode.
    - ii. SXRF data type
    - iii. "Get" all calibrations (and enable good channels) from file "111173\_refined\_fit.spec".
    - iv. "Dynamic Analysis" projection mode.
    - v. DA file = "Mn\_MM33087\_384C14\_18500eV.damx".
  - e. Make sure the output "112658.dai" is back in the same dir "Demo\Maia\Standard\analysis\112658".
19. "Start" the sort, which should take a few seconds.
20. The Mn image should look quite uniform. Open the *Image Regions* window (if it's not there).
21. Drag out a large Box shape on the Image and press Σ. The image sum should show close to 100% in the *Image Regions* Window consistent with the fit to the full sum spectrum.

## Example D2: Automated Standards Analysis for Maia at XFM - the Standards Wizard

- The Wizard is designed to automate the standard foil analysis process. It makes use of metadata variables set in the Maia control socket and passed into the Maia raw blog data files. The steps for semi-automated analysis are:
  - Scan selected dir for all blog files.
  - Applying energy calibrations to all detectors.
  - Building a DA image for each.
  - Use a large Box Region to sample foil concentration.
  - Set the "Conv" factor to reproduce the desired foil composition (e.g. Mn = 100% in this example).
- It reads a "standards.csv" file from "**Demo/Maia/Standard/config/**", which contains the details of each foil at each energy (on Linux, use "**more ~/Demo/Maia/Standard/config/standards.csv**" to view this file).

### Set-up the Wizard

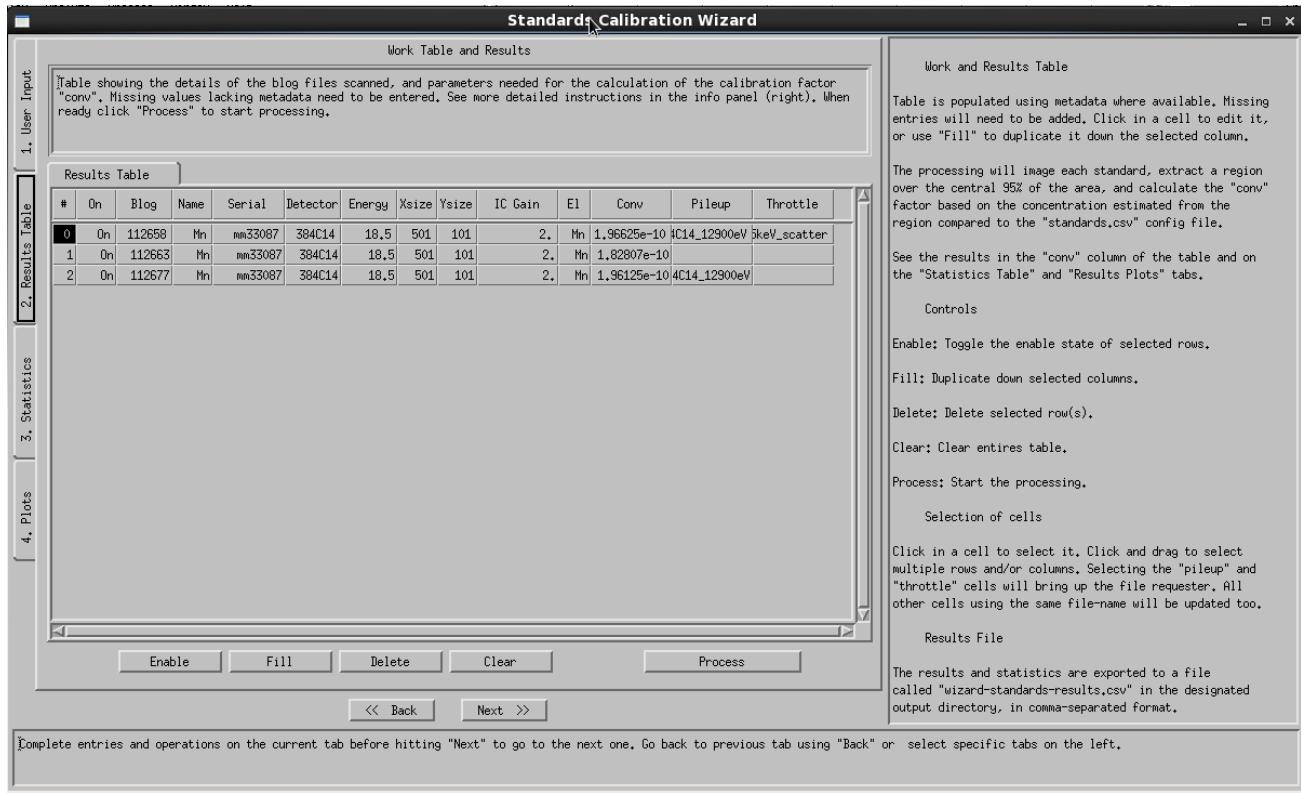
1. Run the Wizard using menu "**Process→Wizards→Standards Wizard**".
  - a. See the Info panel on the right for detailed information and view context sensitive help at the bottom of the window as you move the mouse cursor over widgets.
2. Select dir and energy cal:
  - a. Select the blog dir for the standard foil raw Maia data: "**Demo/Maia/Standard/blog**".
  - b. Select the energy calibration file: "**Demo/Maia/Standard/analysis/111173\_refined\_fit.spec**"
  - c. Select the output path (defaults to neighbouring "analysis" dir tree).
  - d. Select the Resources path: "**Demo/Maia/Standard/config**"
    - i. Defaults to the "path config" in your ".geopixe/geopixe.conf" file.
3. Scan the blog dir tree.
  - a. Click on "Scan Blog Dir" to scan the selected blog dir tree for all raw Maia data.
  - b. Use the option to accept "All event/run files".
  - c. This operation takes a couple of minutes and populates the Results Table.
4. Check settings:
  - a. For raw data with metadata (created after 1-Aug-2015), most columns will be populated for you.
  - b. Missing entries can be entered. Use "Fill" to duplicate down from a selected cell.
  - c. The only thing missing now, is the empty (zero) "Conv" column.

### Process the data

5. Click on "Process" to process data, which does the following:
  - a. Forms DA image for each in Sort EVT.
  - b. Draws a large Box region on image for selected element (e.g. "Mn").
    - i. Avoids null areas.
  - c. Integrates the Region and calculates "Conv"
  - d. Updates Results Table (and Plots on later page) with "Conv".
6. Results:
  - a. See "Conv" column in Results Table.
  - b. The "statistics" table shows a column "SD/Error". If all is well, this should be close to 1, which means the variation between pixels is just Poisson statistics.
    - i. Much larger than 1 suggests a non-uniform image; should not be the case for a standard foil.
    - ii. In this case, open the image DAI file and check it out. There is a problem.
  - c. Plots of "Conv" versus run number on "Plots" page.

## 7. Notes:

- a. If the pileup or throttle file is not found, you will be prompted (just for first occurrence of each file).
- b. The lack of gaintrim for run #112663 in this case appears to have biased the "conv" factor low.



## Example D3: Maia Mapper – NIST 1243 calibration standard

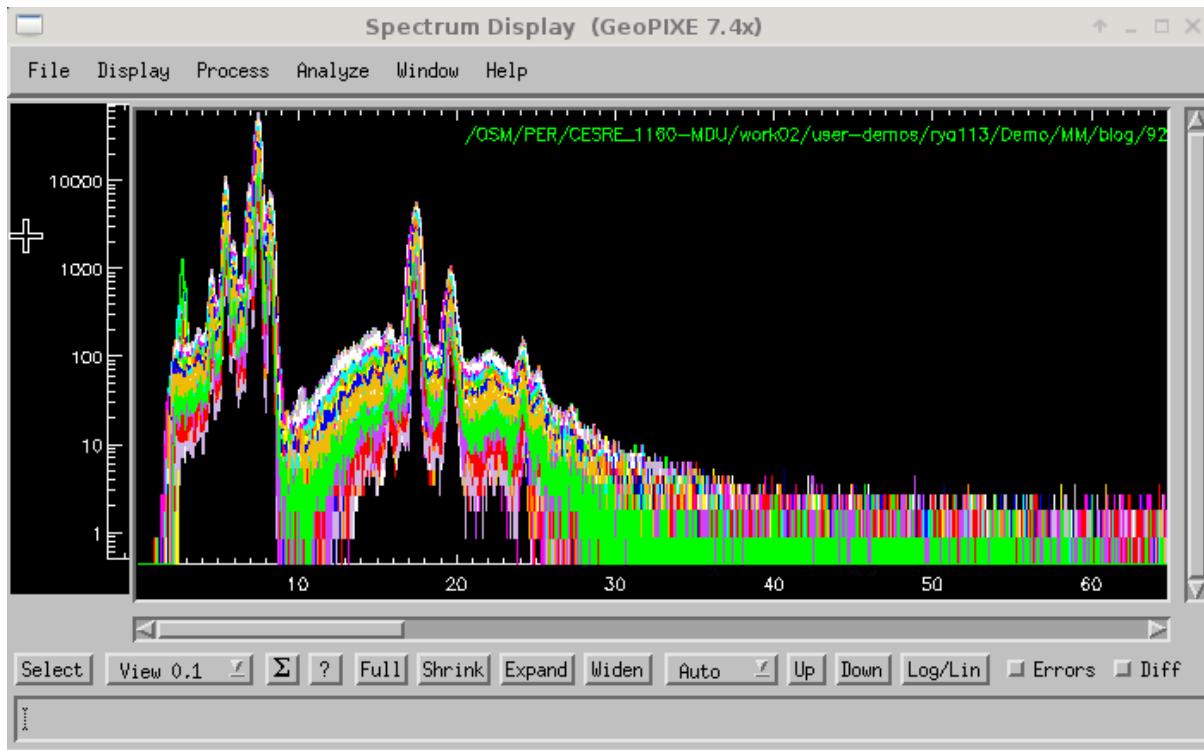
- Importing spectra for all 384 detectors from the raw data files.
- Applying energy calibrations to all detectors.
- Fitting a representative spectrum and determining the calibration “conversion factor”
- Calculation of X-ray yields
- Building a DA matrix and imaging (not an exciting case, perhaps)
- Verifying image values using a Region.

*NOTE: Typically, this standards evaluation would be done using the **Standards Wizard**. But here we look at the steps involved.*

### Import Spectra and Calibrate Energy

1. Import spectra from the raw Maia data files for run #112658 (directory “**Demo/MM/blog/924/**”) using the “File->Import” menu on *Spectrum Display*.
  - a. Select “Synchrotron (SXRF)” data from the “Maia 384/96 – HYMOD data acquisition” data source and the “Extract from Maia-384 - corrected blog files” operation.
  - b. Select all blog files 924.\*
  - c. Select the directory “**Demo/MM/analysis/924/**” as the output path.
2. On the “Flux PV select” pop-up:
  - a. This data-set has metadata built-in, so the following are set automatically:
    - i. IC scaler channel = Maia:dwell.time
    - ii. Preamp sensitivity/units = 1 ms
    - iii. DT cal parameters A = 5.2, B = 0 (ns)
  - b. For now, entry a “Conversion” factor of 1.0
3. Pileup file:
  - a. If you have your “Translation Tables” in order, this should not prompt at all.
  - b. If it does, select the pileup file = “900-wide.pileup.var”.
4. The *Spectrum Display* shows an overlay of E, X, Y and T spectra for all 384 detector channels.
  - a. E – energy spectrum for each detector

- b. X, Y – projections for each detector on X and Y axes
  - c. T – time-over-threshold spectrum for each detector
5. Open the *Spectrum Select* window to remove XY and T spectra (so we just have the E spectra)
- a. Select “all X,Y and T” in the dropdown and click on “Delete:”.



6. Fit the spectra to refine energy calibration:
- a. Open the *X-ray Spectrum Fit* window and load the fit-setup file “NIST\_1243\_384D19\_24100eV.pcm” from directory “Demo/MM/config/384D19/standards/24100eV/”.
  - b. Set the View range to 3.5 to 40 keV and click on “Use View” on the *X-ray Spectrum Fit* window.
  - c. Click on “Fit: All” to fit all individual detector channels.
  - d. Check that all spectra now appear well aligned, with the Ni and Mo K lines well aligned in energy.
  - e. Save the spectrum file as the energy calibration: “924-E-cal.spec” in the “MM/analysis/924” directory.
7. Sum all detectors together using the menu “Process→Add (remap cal)”.
- a. Fit the spectrum with “Fit: One”.
8. Open the Fit Results window
- a. Observe that the Ni concentration appears to be 30%, assuming “conv” = 1.0.
  - b. If we change the conv factor to 0.5, we should get Ni close to the CRM value of 58.78%.
    - i. Click on “?” next to “Q” on the *X-ray Spectrum Fit* window.
    - ii. In the pop-up enter Conv: Change to: to 0.5 and hit OK.
    - iii. Fit the spectrum again, and the Ni value should be close to the CRM value.
    - iv. Conv = 0.5 is our new calibration (to convert dwell time in ms to effective flux).

### **Question**

- The other approach (like for the *Standards Wizard*) is to form the image of the NIST-1243 sample, evaluate a Box region of uniform Ni and observed the Ni value, for the conv value assumed for the imaging.
  - Try this to form “conv”.
    - Tip: use the *Image* window “Image Properties and History” to see current conv value.
    - You can also double click on this row in the properties to set a new conv value.
    - If you do, “Update: All” on the *Image Regions* window to update the evaluated concentrations.

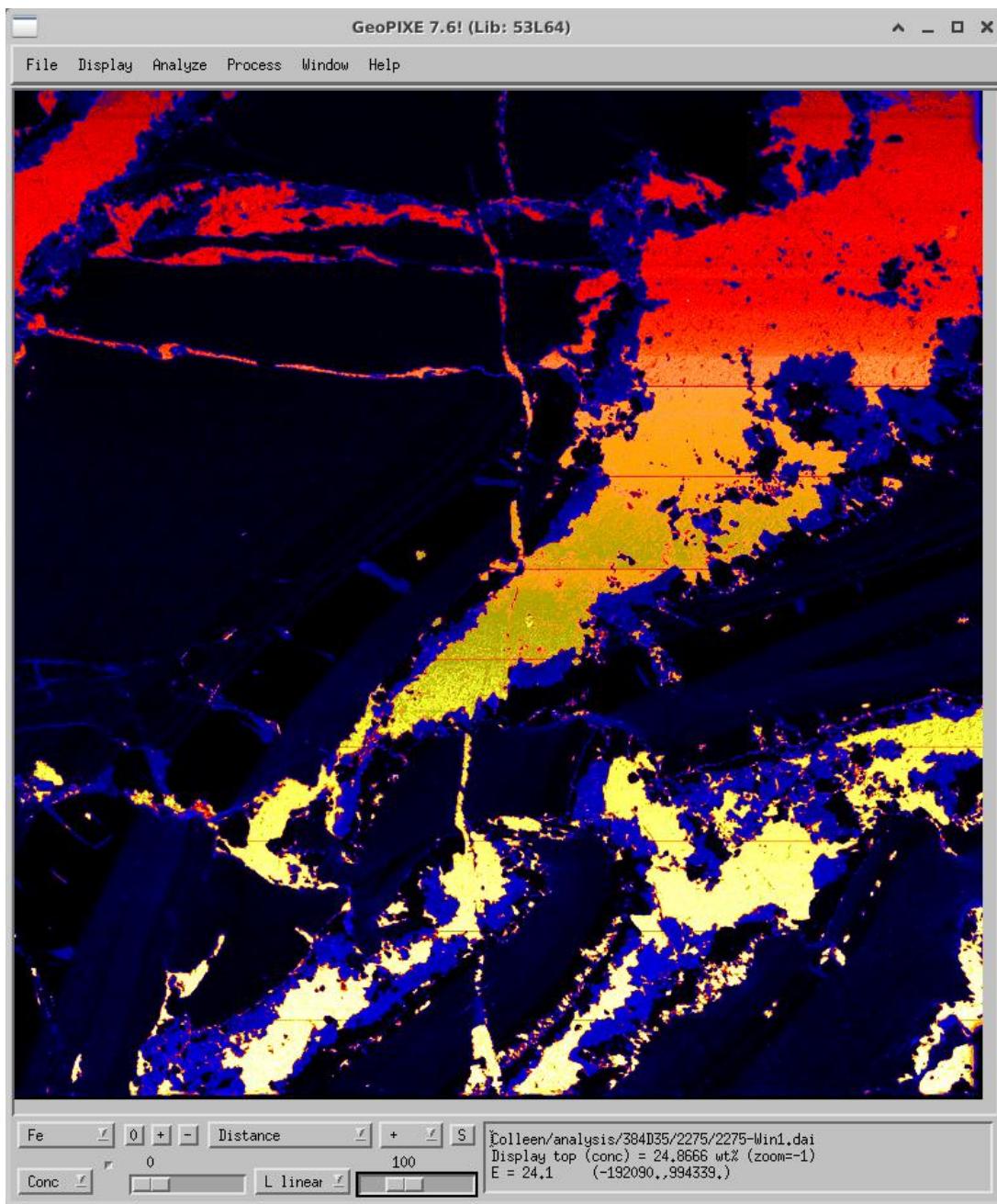
## E. Image Corrections

### Example E1: Correction for changing flux

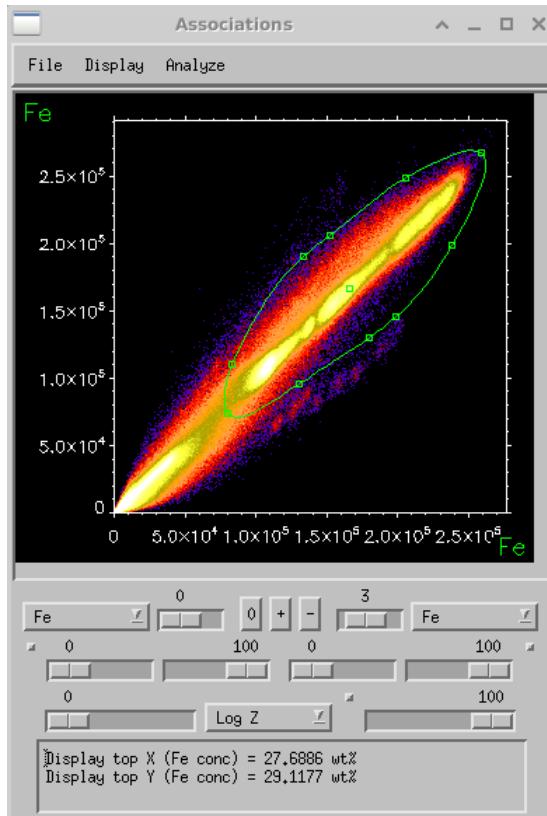
Changes in beam/flux with time (and pixel) can be generally corrected if pixel-by-pixel beam/flux data are acquired. However, for some systems this is not possible. In the case of Maia Mapper, flux cannot be measured. However, the source is typically very steady, varying only within a range of  $\pm 0.1\%$  so this is not a problem. Nevertheless, some instrument issues can make it vary much more, which produces an apparent change in element concentration as a function of Y scan line. If we have a common phase, with a consistent concentration of some major element, occurring somewhere on each scan line, then we can correct for this effect. This is illustrated here.

#### Correct for Y variation

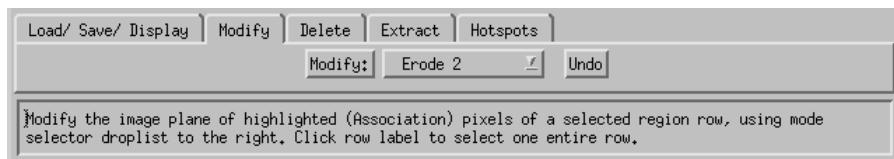
1. Load the image file “**demo\Maia\MM\2275-Win1.dai**”. Use the element droplist to view the Fe image.
  - a. The image shows a general decline in apparent Fe as the scan proceeds from bottom ( $Y=0$ ) to top. There are also a few scan lines with some missing value. Evidently, an additional problem caused lost beam briefly too.

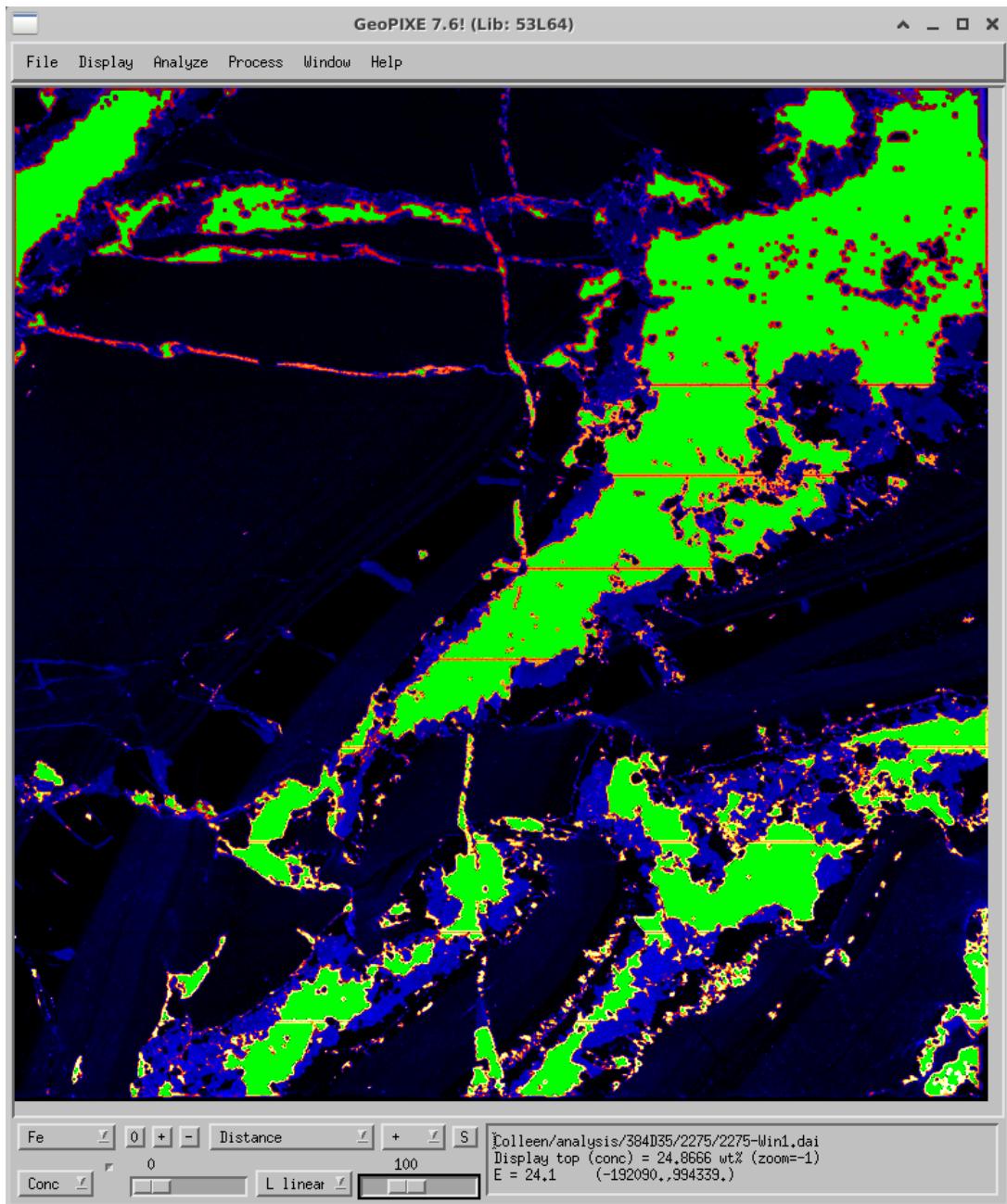


- b. Now open the *Association* window and plot Fe versus Fe. Make the pre-smooth of data zero on one axis to make the trend clearer (e.g. set smooth for Y to zero, as shown above).
  - i. *Tip: hover mouse over widgets to see what each does.*
- c. Drag out a spline curve to enclose the diagonal trend.

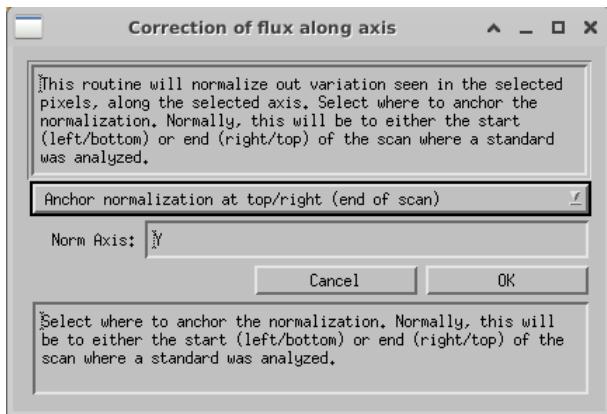


- d. Select the *Association* menu: “Analyze->Include points within Spline”, which will highlight these pixels on the image. These may also include some grain boundary mixed pixels. Hence, also apply an **Erosion** operation on the selection using the options on the **Modify** tab of the *Image Regions* window.

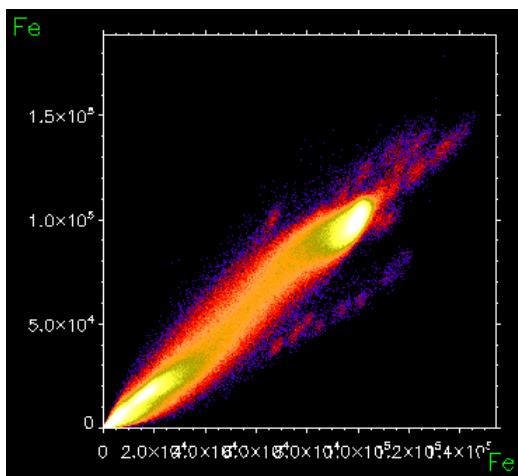




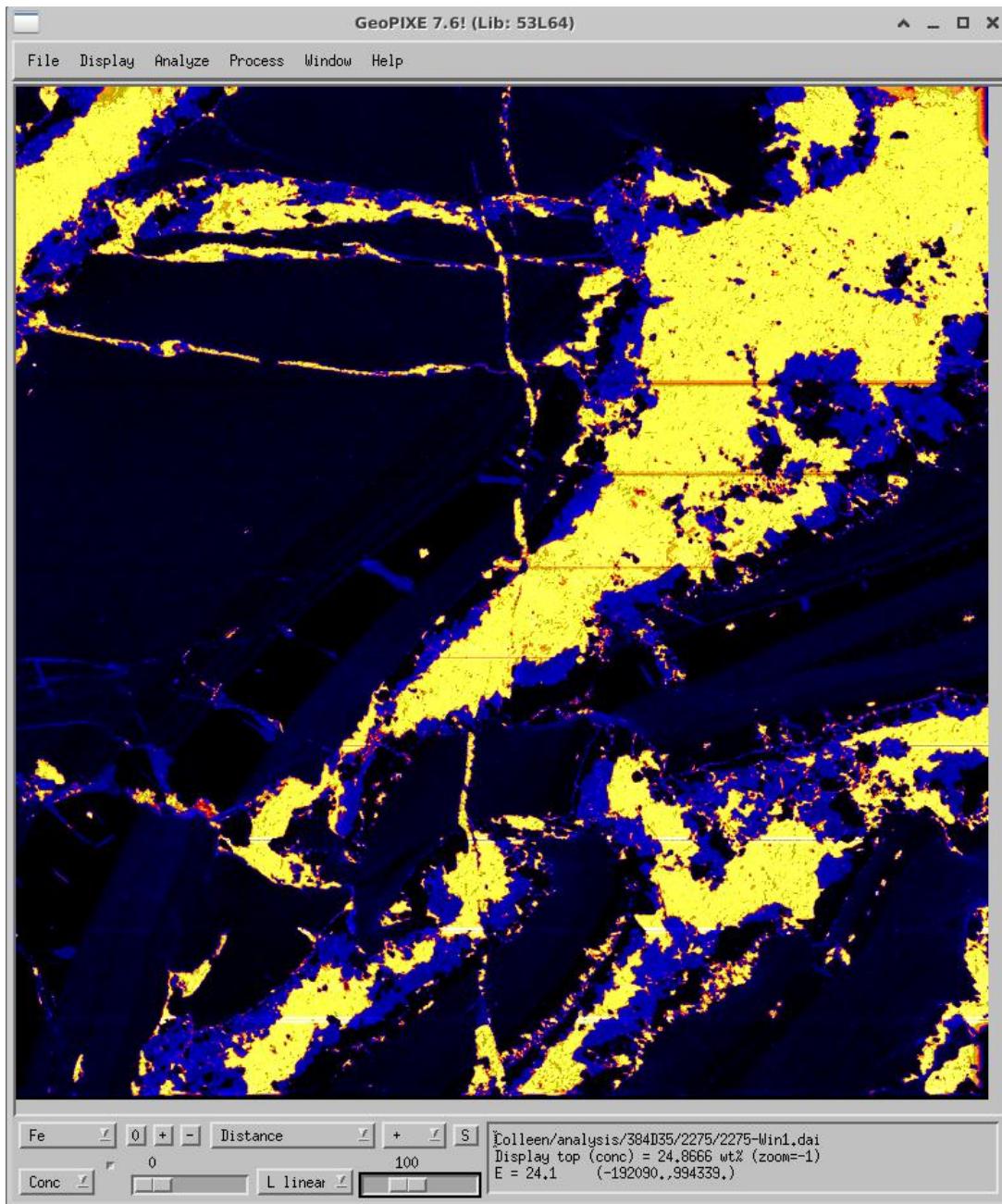
- e. Now, use these selected pixels to correct for Y variation using the *Image* window menu: "Process->Correct->\* Ystep Current" (the "\*" indicates that all image planes will be corrected).
- f. A pop-up window provides some options for anchoring the normalization. Our scan was followed by a scan of a standard (this will be used to set the flux calibration factor for this scan). Hence, we choose to anchor our normalization at the end of the scan (top).



- g. A warning may indicate that some rows are not sampled. This may be due to the top and bottom and missing rows, especially as they were widened by the Erode. These are filled in using a linear interpolation across any gaps. The resulting *Association* plot now shows far less variation ...



- h. Another pop-up will inform you of the range of correction (0.469 to 1.0 in this case, anchored at the end) and the name of a text file, which contains the normalization factors per Y line.
  - i. *Tip: You can use this file to repeat the same correction (e.g. if data is reprocessed from raw data) using the menu: "Process->Correct->CorrectY from file".*



- i. The missing rows may be tackled using another of the correction methods outlined below.

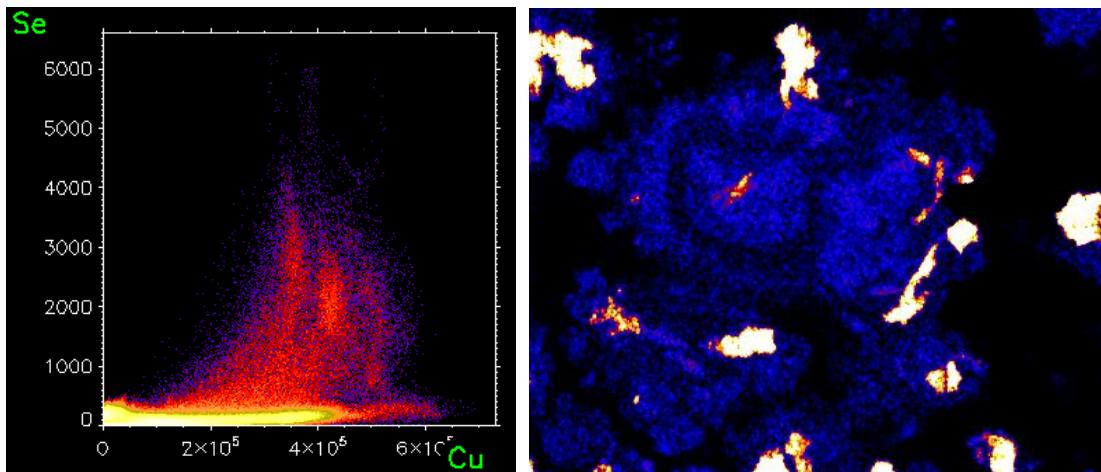
*Tip: For severe changes in flux, you may need to do the correction twice. The first one provides a coarse correction, and then a second pass is done more carefully (e.g. including an erode) to converge on a better correction across the image.*

## Example E2: Correction for element interference

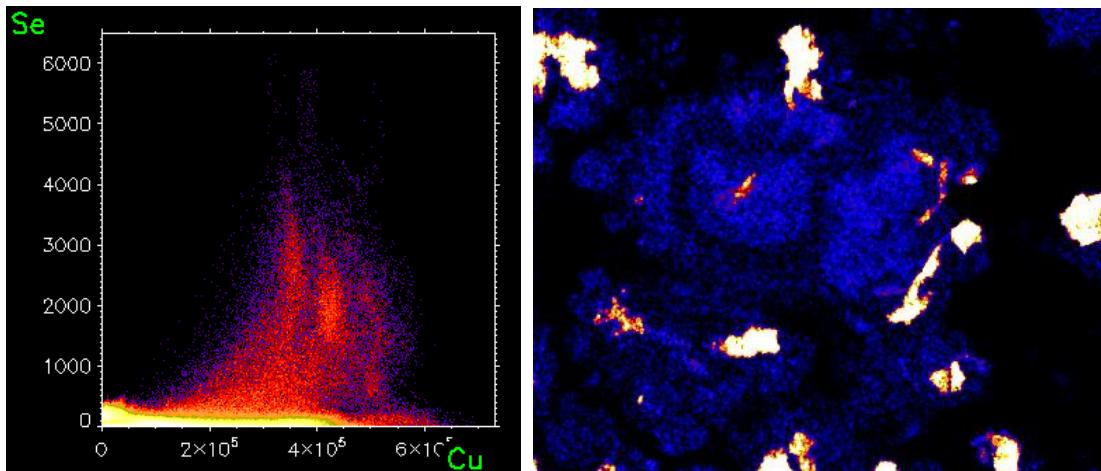
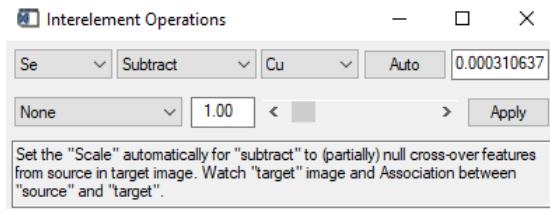
While the DA method does target good spectral decomposition and the removal of the effects of spectral overlap in images, some contamination of a strong element can still be present in another element image. The **Interelement Operations** tool allows some pragmatic correction for these residual effects. This is illustrated here.

### Correct for element crosstalk

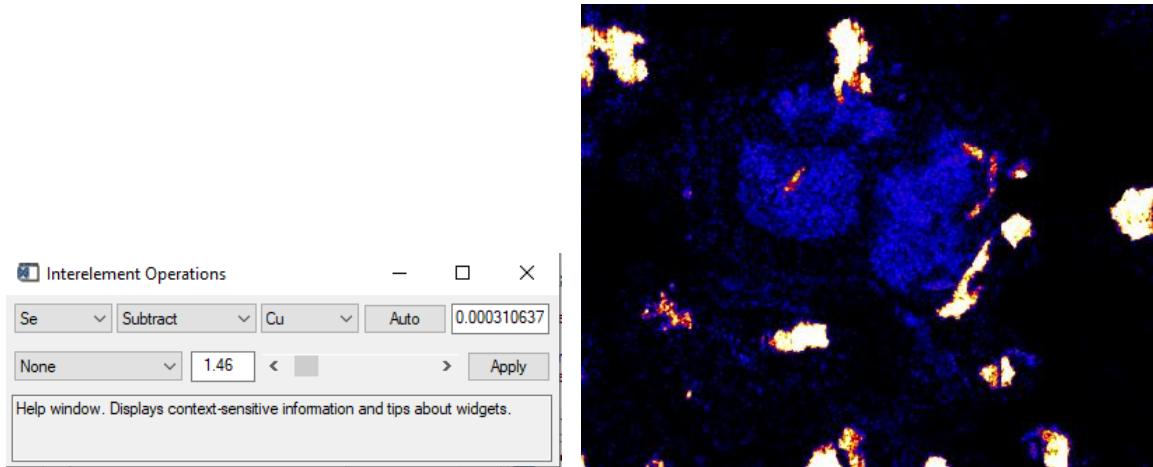
1. Load the image file “**demo\Maia\CSIRO\c4-x\c4-x-3.dai**”. Use the element dropdown to view the Se image.
  - a. Open the Association window and plot Se versus Cu. The plot shows a sub-parallel bright trend at very low Se, correlated with Cu, which is suspected to be a residual artefact of Cu on Se image pixels (Se shown below).



2. Open the *Interelement Operations* window and set the left dropdown to Se. This is the “target” element, which is to be corrected (mouse over it to see the context sensitive help).
  - a. Select “Subtract” as the *operation*, on the second dropdown, and Cu as the “source” on the third.
  - b. Click on “Auto”, which will attempt to conservatively estimate the amount of Cu image to subtract from the Se image. The Se-Cu trend is now lower.



3. On the *Interelement Operations* window, adjust the slider to fine adjust how much Cu is subtracted and watch the Se window. As the control is dragged, the image changes and you can now see the spatial area where the artefact resides and adjust the amount of subtraction to remove it.



- a. Note that the Se image has not been altered yet, so you can always remove the correction (e.g. set operation to “off” or close the window)
- b. Click on “Apply” modify the Se image pixel values to confirm the change. A History note is added to Se.

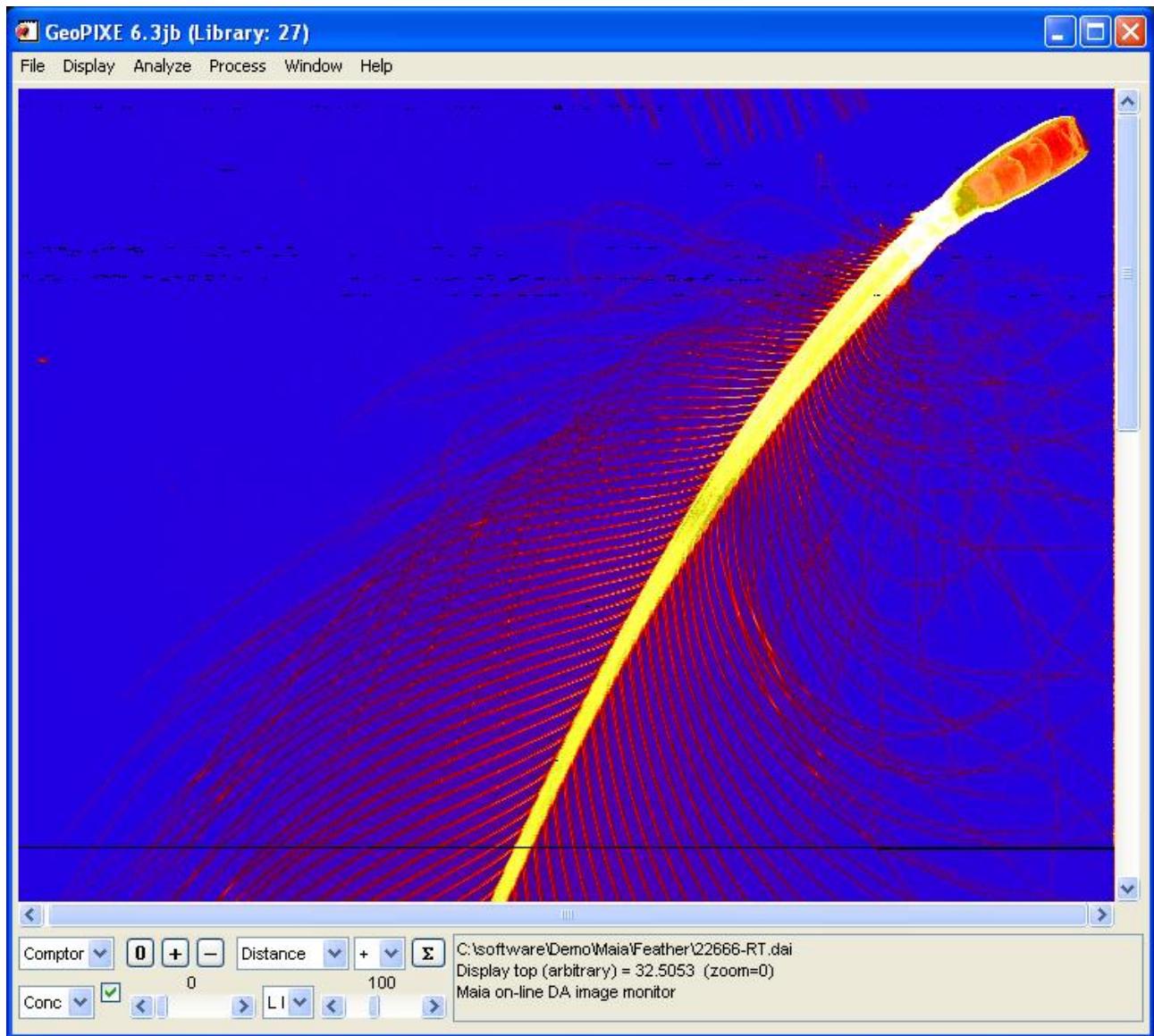
### Example E3: Various Image Corrections

Various errors or artefacts in images can be corrected. These are available on the “Process”, “Process →Correct” and “Process →User Plugins” menus. This is illustrated here.

- Removing zero pixels (e.g. Maia RT missing pixels).
- Removing missing lines (e.g. beam fill blank partial lines).
- Correcting for stage speed fluctuations (dwell time variation)
  - Normalizing to elastic/ Compton

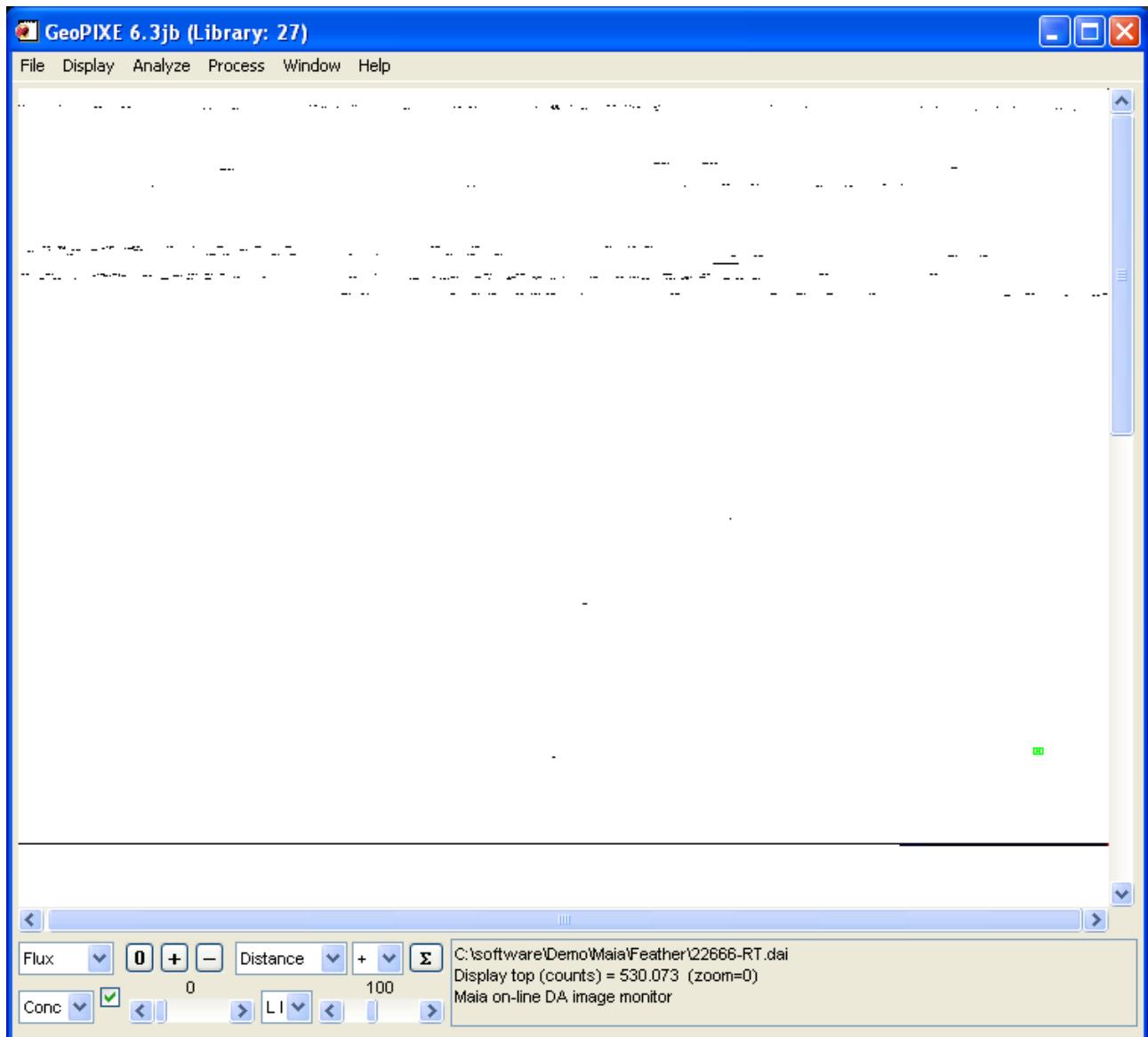
#### Zero Pixel Correction

1. Load the image file “**demo\Maia\Feather\22666-RT.dai**”. Use the element dropdown to view the Compton image.
  - a. The image shows a number of missing pixels and a whole line missing. The missing pixels are DA image packets missed by the on-line display. The full image (without these zero pixels) can be obtained from the full Maia data on disk, or we can correct for them using image processing.



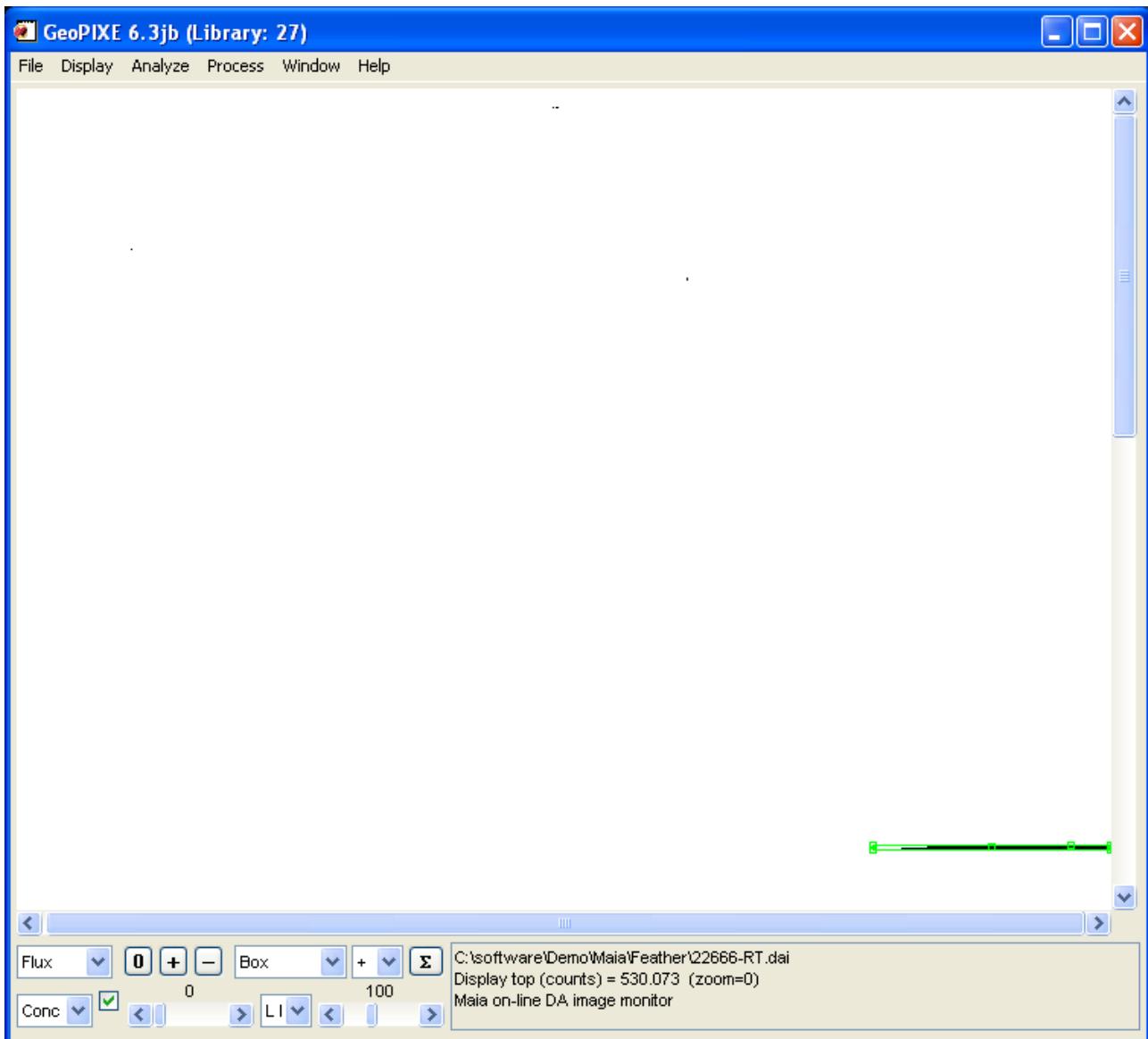
Real-time DA image collected on-line during the analysis of a feather (R. Banati, ANSTO). Note zero pixels and a missing row.

2. A useful approach is to use the “flattened” flux map to view these missing pixels clearly and provide a good basis for correction. Load the flux map using the “Flux Image plugin” (see “Process->Plugins” menu).
  - a. At the end of the run, variations in flux are used to normalize all element images to uniform flux, and the flux map itself. This is referred to as “Flattening” in GeoPIXE and the Maia Control on-line system.
  - b. In the on-line system, missing pixels remain “black” or zero.
  - c. This flux map is the result AFTER flattening. The raw flux map can also be viewed using the plugin: “Raw flux image”.



"Flattened" flux map for the feather run.

3. Now apply the zero pixels removal procedure using the menu "Process→Correct→Zero pixels".
  - a. This procedure uses 3% of the top intensity as displayed (set with the top slider) as a threshold; anything less than this is deemed "zero".
  - b. It then replaces any pixel surrounded by at least 5 good pixels with the average of these good pixels.
  - c. It does this in all image planes. Return to Compton to see.
  - d. Zero pixels surrounded by more than 3 zero pixels do not get corrected (such as the double row seen here). The isolated zero pixels could benefit from a second application of the Zero Pixel correction.

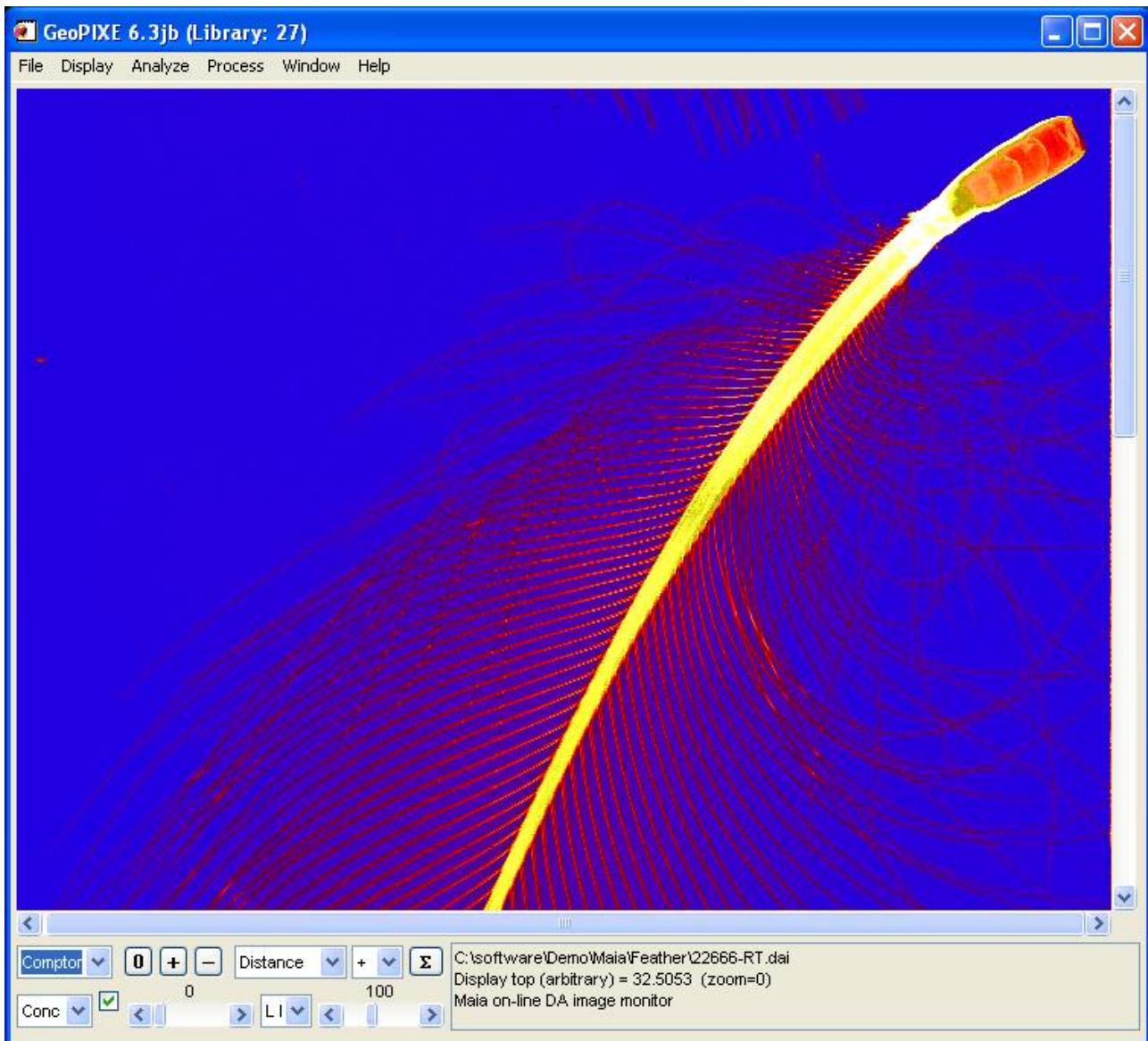


*Flux map after correction for zero pixels. A portion of the missed row is evidently a double row and has not been corrected. The green shape is a Box used to correct the “Missing row” (see below).*

4. The result has repaired most zero pixels. A few that were surrounded by more than 3 zero pixels remain. A second iteration could catch the isolated ones.

#### ***Missing Row correction***

5. Continuing with the Feather example, the missing row can be corrected. Start by dragging a long narrow “Box” that just encloses the missing pixels (as illustrated above).
  - a. This box is also region #1 in the file “22666-RT-missing-rows.region”.
6. Now use the menu “process->Correct->Missing rows”, which replaces the missing pixels with the average of pixels in the neighbouring image rows. Clear the box to see the results more clearly (menu “Display->Clear ALL shapes”).
  - b. After 2 iterations of the zero pixel routine and this missing row correction, all zero pixels should be repaired and zero pixels in all element images replaced by the local averages as well.

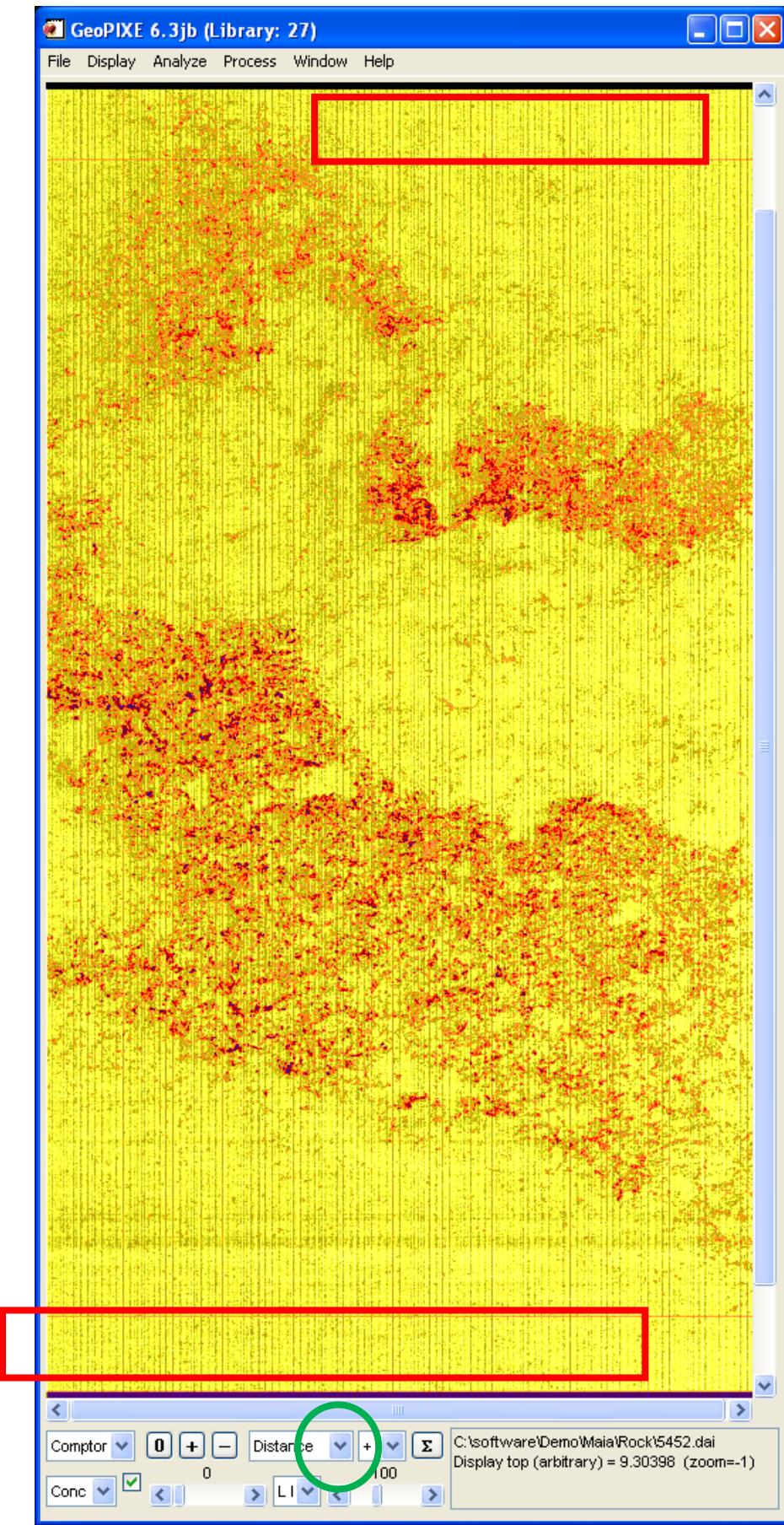


*Feather after zero pixel and missing rows corrections.*

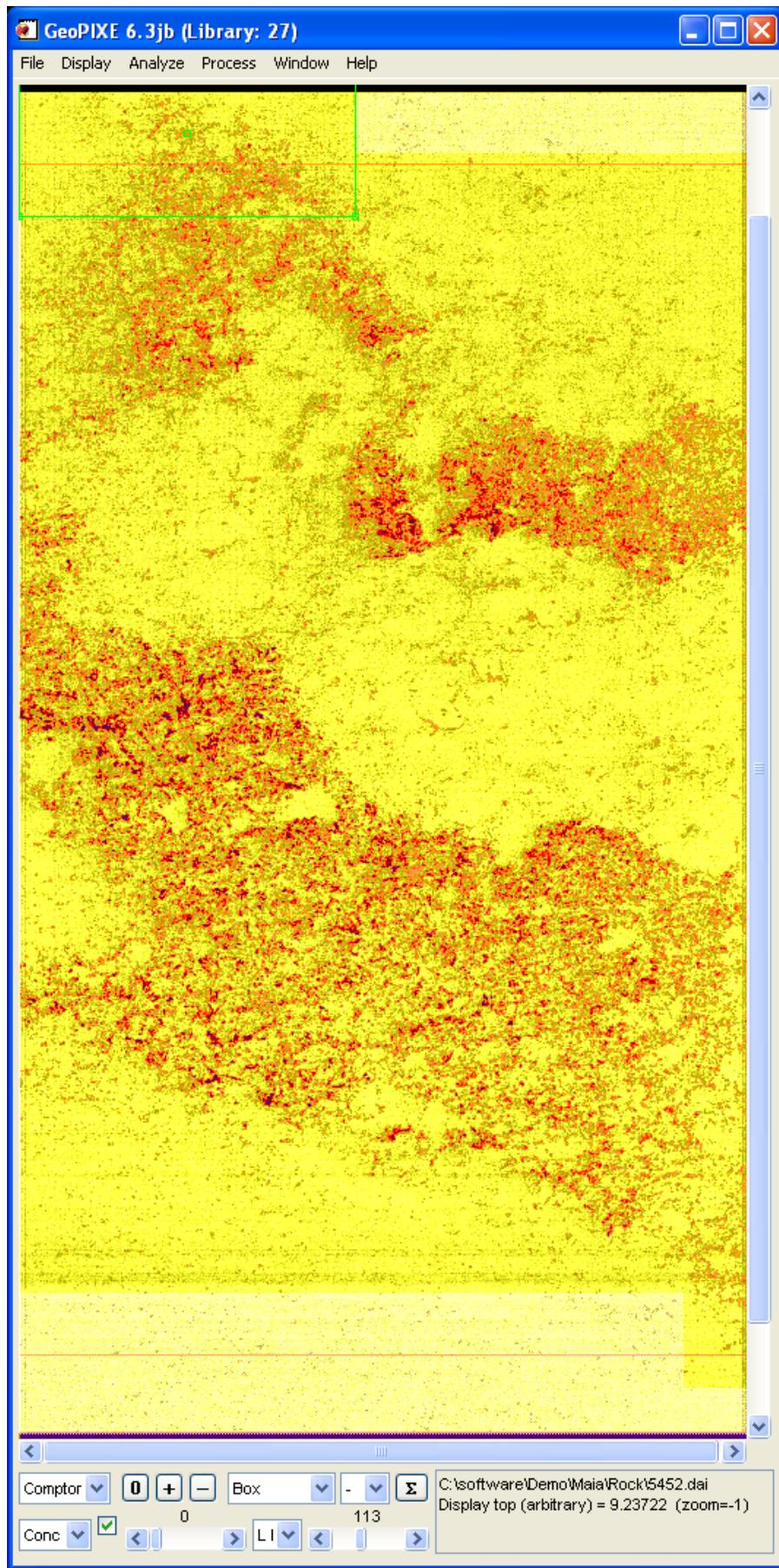
### *Correcting for stage speed fluctuations (dwell time variation)*

#### **X variation only**

7. Load a new image example: “**Demo\Rock\5452.dai**”.
  - a. The image shows an image of a rock section (courtesy, Rob Hough, CSIRO) that suffers from stage speed variation. The dominant effect appears as vertical stripes or a “brown paper” effect.
8. Now, see if you can select only the portion of the image enclosed in red boxes on the figure below.
  - b. These appear to be relatively flat, apart from the “brown paper” effect. Collectively, taken together, these regions span the full X width and might work well to provide a normalization to correct for dwell variation in X.
9. Hint: Try the following:
  - c. Drag a “Box” shape that spans the full image (except for the top and bottom stripes when the beam was off).
  - d. Change the “area analyze mode” to exclude (“-“ on the dropdown; see figure below), which means remove pixels from the current selection.
  - e. Now, use various size boxes successively to cut away from the current selection (shaded area) to leave just the fields within the red boxes.



*Compton scattering image for rock section (courtesy, Rob Hough, CSIRO) showing “brown paper” artefact. The red boxes are suggested region areas for “CorrectX” correction and the green circle shows the area analyze mode dropdown (see below).*

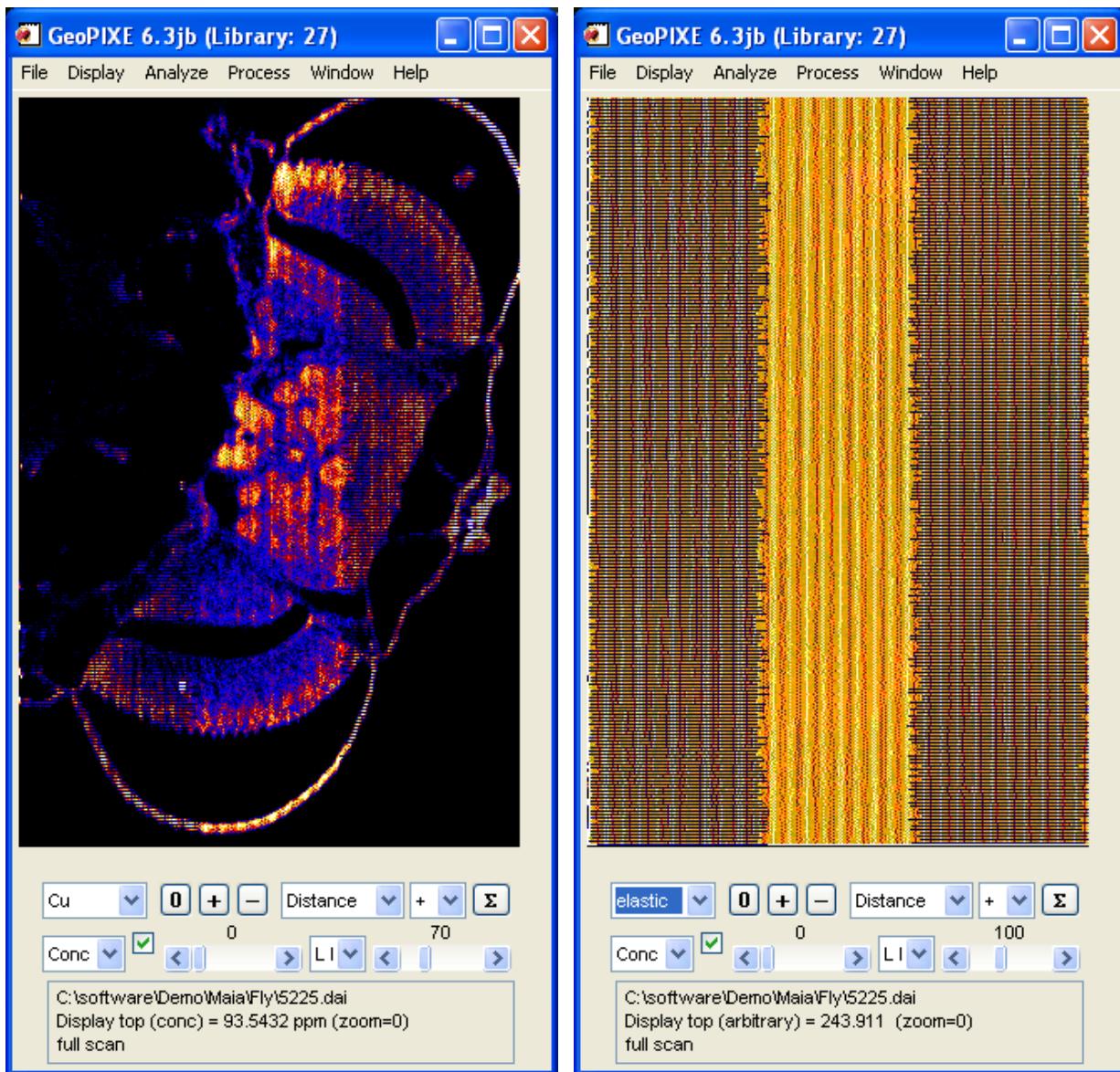


Compton image after CorrectX correction. All other element images were corrected in the same operation. The lighter shaded areas are our final selection after the “Exclude” cutting away procedure.

10. Now, use the menu “Process->Correct->Xstep current” which provides a normalization as a function of X for all Y, and then corrects all images.
  - f. It also saves a file “5452-CorrectX.txt” that contains the variation in X that might be used again at a later time to do the same correction using the menu “Process->Correct->CorrectX from file”.

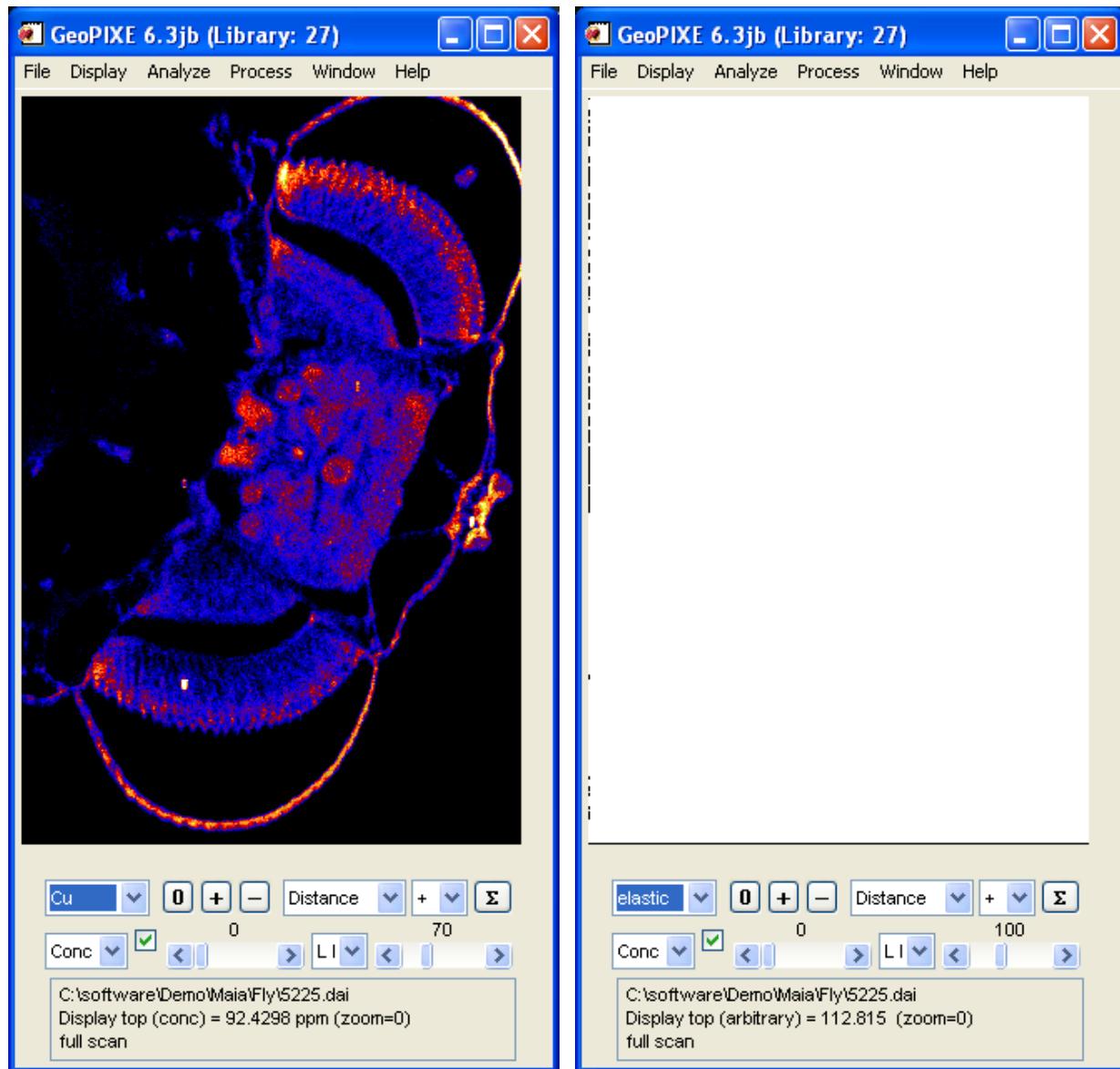
### Pixel by pixel

11. Load a new image example: “Demo\Fly\5225.dai”.
  - a. The image shows a fly’s head (courtesy of ?. Burke, Monash U.), which suffers from wild stage speed variations that result in large variations in the dwell time in each pixel. Fortunately, the elastic image is dominated by the kapton support and shows now structure due to the fly. This can form the basis for normalization.



Fly’s head image (courtesy of R. Burke, Monash U.) showing Cu (left) and elastic (right) images, which suffer from stage speed variations.

12. Apply the plugin “\* Flatten to elastic image”. The “\*” prefix means that all images will be affected.
  - b. This plugin does a simple normalization, pixel by pixel, to the elastic image.
  - c. The source for this plugin is available in the GeoPIXE distribution.
13. Note that this process tends to increase the noise in the element images by the noise found in the elastic image, and so only really works well for a high statistics elastic image.



Fly's head Cu (left) and elastic (right) images after normalization to the elastic image.

## F. Correction of Yields for Spatial Composition Variation

Sample composition varies generally across an image area. This means that the yield model assumed in projecting the images using the DA method will only be an approximation to any given pixel. This section describes a method to correct model yields on a pixel-by-pixel basis based on an end-member analysis of the image using a set of end-members that adequately describe the range of variation in composition (at least for the heavy elements that strongly affect self-absorption and secondary fluorescence) (see C.G. Ryan, "Quantitative Trace Element Imaging using PIXE and the Nuclear Microprobe", International Journal of Imaging Systems and Technology [Special issue on Quantitative Imaging] 11 (2000) 219-230 and C.G. Ryan, E. van Achterbergh, C.J. Yeats, Tin Tin Win and G. Cripps, "Quantitative PIXE trace element imaging of minerals using the new CSIRO-GEMOC Nuclear Microprobe", Nucl. Instr. Meth. B189 (2002) 400-407).

A similar issue relates to fitting the spectra of regions with some unknown average composition. An end-member analysis can also be done, as part of the fit, to determine the proportion of end-members (in this region associated with the spectrum under fit) in order to correct yields for composition and iterate towards an improved fit to composition in the region. See the fitting example below.

**Note:** The phase decomposition method here aims to span composition space sufficiently to produce better estimates of absorption, which dominates the modelling of X-ray yields, in order to correct images towards quantitative results. It may not provide a complete mineralogical breakdown of the phases present in a sample, which is quite a different goal.

## Example F1: Correction of Images for Composition Variation

*Correct images for matrix (composition) effects using an end-member decomposition approach. This should be done before trying any MPDA examples.*

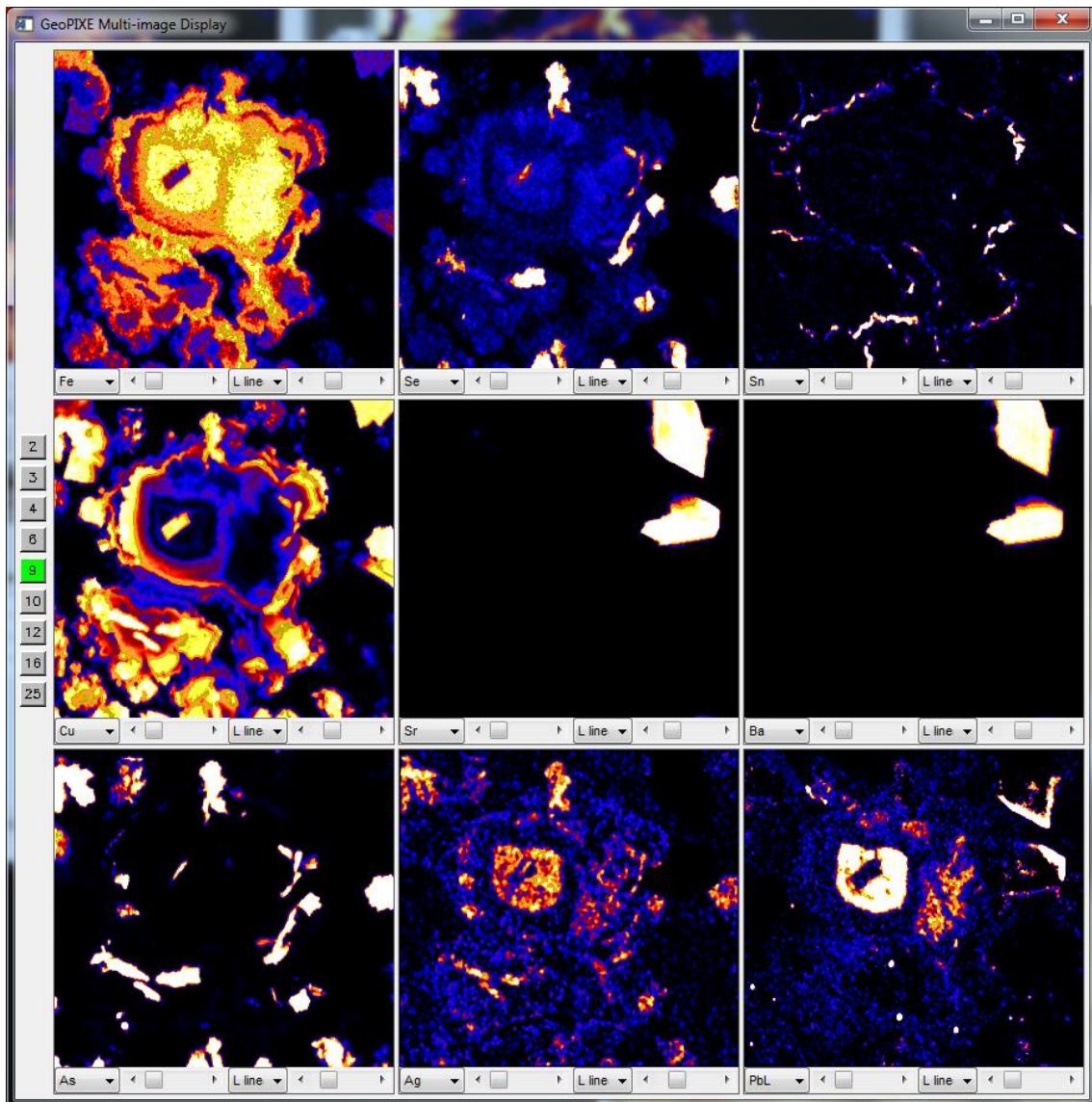
- Background on composition effects in DA and the composition correction.
- Selection of end-members.
- Construction of the transformation matrix.
- Correction for composition spatial variation.
- Transformation onto images of end-member phase proportion.

### ***Background on Matrix Correction of images***

1. The calculation of SXRF or PIXE yields (counts per ppm per unit of beam flux, etc.) is sensitive to the composition of the sample. A high average atomic number sample matrix will generally produce lower yields. But it depends on the energy of the fluoresced X-rays versus absorption edges caused by component elements in the sample matrix.
2. The calculation of yields tends to follow the rule that the yields are dominated by the effects of self-absorption and vary roughly inversely with self-absorption. Hence, we can use “the Bragg Rule” and combine inverse yields ( $1/Y$ ) for the end-members in proportion to their mass fraction.
3. DA imaging assumes a uniform sample/matrix composition. Hence, in some phases there may be significant errors in concentration of many elements.
4. After producing DA images assuming some uniform matrix composition, the images provide a first approximation to the composition variation, at least of the major elements that are visible via SXRF or PIXE. This allows us to estimate the proportion of the main end-member components in each pixel in order to improve the calculation of yields in each pixel. These improved yields can be used to converge on the true concentration images.

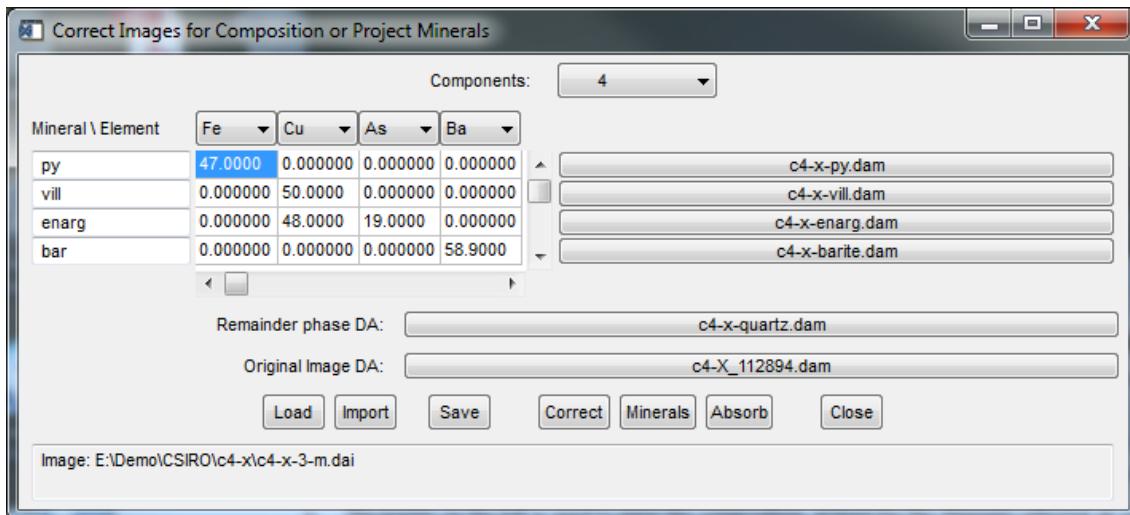
### ***Example data – selection of end-members***

5. Load the PIXE example image data-set “**Demo/CSIRO/c4-x/c4-x-3-b.dai**”. Look at the various element images and note the major elements observed (Fe, Cu, As, Ba). In this case with a filter in front of the detector to enhance the detection of heavier trave element, light elements such as O, Si and S are not detected.
6. The sample is a geological one with known phases present: pyrite ( $\text{FeS}_2$ ), villmannite ( $\text{CuS}_2$ ), enargite ( $\text{Cu}_3\text{AsS}_4$ ), barite ( $\text{BaSO}_4$ ) and gangue silicates that we might represent as a quartz end-member ( $\text{SiO}_2$ ). The pyrite and villmannite form a solid-solution series in practice and so any composition on the mixing line between these is likely to be present. Note that some elements appear in multiple end-members (i.e. Cu).



#### **End-member correction matrix**

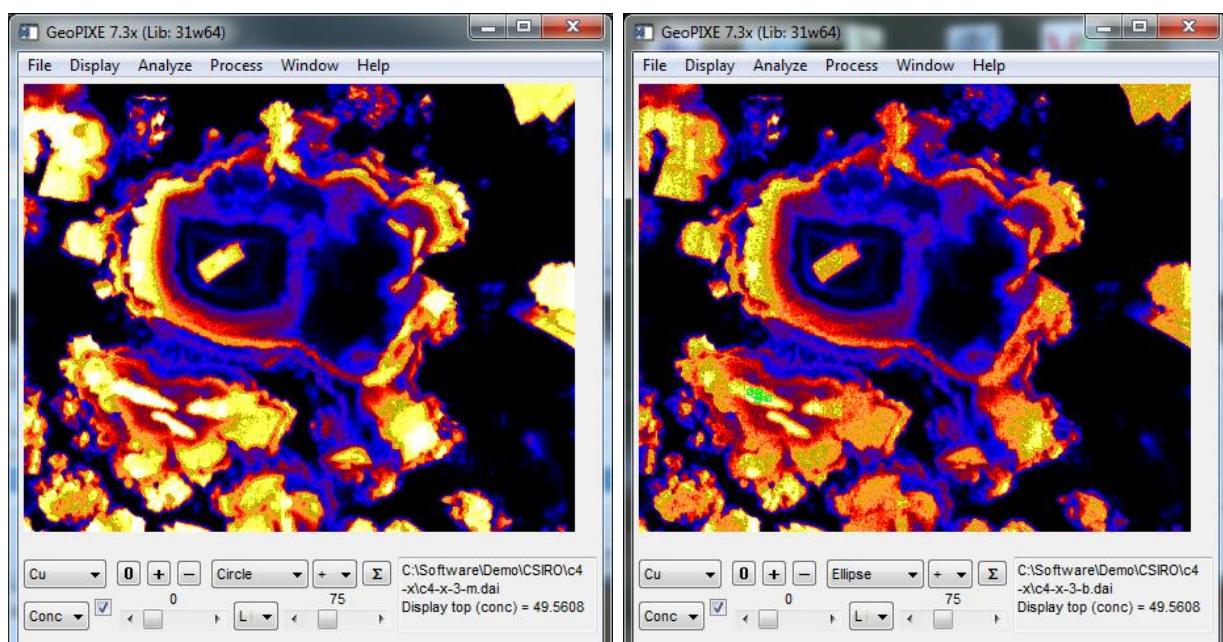
7. Open the *Correct Images* window using the *Image* window menu “Window→Correct Yields (N=6)”, which allows up to 6 component end-members, and load the correction file “py-vill-enarg-bar.correct” appropriate to this sample.
  - a. The element columns are the main elements that we can observe, one for each end-member, which we have named as row headings (“py”, “vill”, “enarg”, “bar”). These are the elements that help distinguish between the end-member components.
  - b. The buttons on the right allow us to load a DA matrix for each end-member phase.
    - i. Each DA matrix is generated using a fit to a region of the image that closely approaches the end-member term.
    - ii. This will be discussed more in the MPDA example.
  - c. The “remainder phase” button selects what end-member we associate with concentration not accounted for by the other end-members, the so-called “remainder”.
    - i. The “remainder” DA matrix is generated using a fit to a region of the image outside the main end-member areas, like the silicate in the present case.
  - d. The “original image DA” button selects the DA matrix used to process the original DA images.
    - i. GeoPIXE will generally set this automatically from history when an image is loaded.
  - e. The matrix on the left is used to enter the composition, at least for the elements selected, in each end-member phase (e.g. villamannite = 50 wt% Cu and enargite = 48 wt% Cu + 19 wt% As, etc.).



### Correct images using the correction matrix

8. Now click on the “Correct” button. This will:

- Invert the end-member composition matrix to make a transformation from element concentration map to maps of end-member proportion.
- Use these to form a better estimate of X-ray yields for all elements in each pixel.
- Based on the ratio of these new yields to the original yields, as in “original DA”, it will then correct all element images.
- Usually you will need 2-3 iterations, so press “Correct” again, and observe how the Cu image changes with each iteration (very little change is seen at iteration 3).



Original Cu (left) and after 2 iterations of Correct Yields (right). Note how the dynamic range has been reduced (ratio of highest to lowest Cu)

**Tip:** To make this comparison, open a separate Image window using the menu “Window → Image Display (unlinked)” and open the original DA images for this example to view them side-by-side.

9. Open the *Image Regions* window using menu “Window → Image Regions” and load regions “c4-x-3-q1.region”.
- Click on “Update All” to apply these region pixel selections to the current corrected images. Note how the concentrations change.
  - Compare these to the region sums for the original images. Use the second image window for this.

Region	Fe	Cu	As	Ba
--------	----	----	----	----

Region	Fe	Cu	As	Ba
--------	----	----	----	----

0	14.8%	35.9%		
1				45.7%
2	11.4%	46.3%	14.4%	
3	43.7%	0.65%		
4	15.9%	31.9%	0.40%	

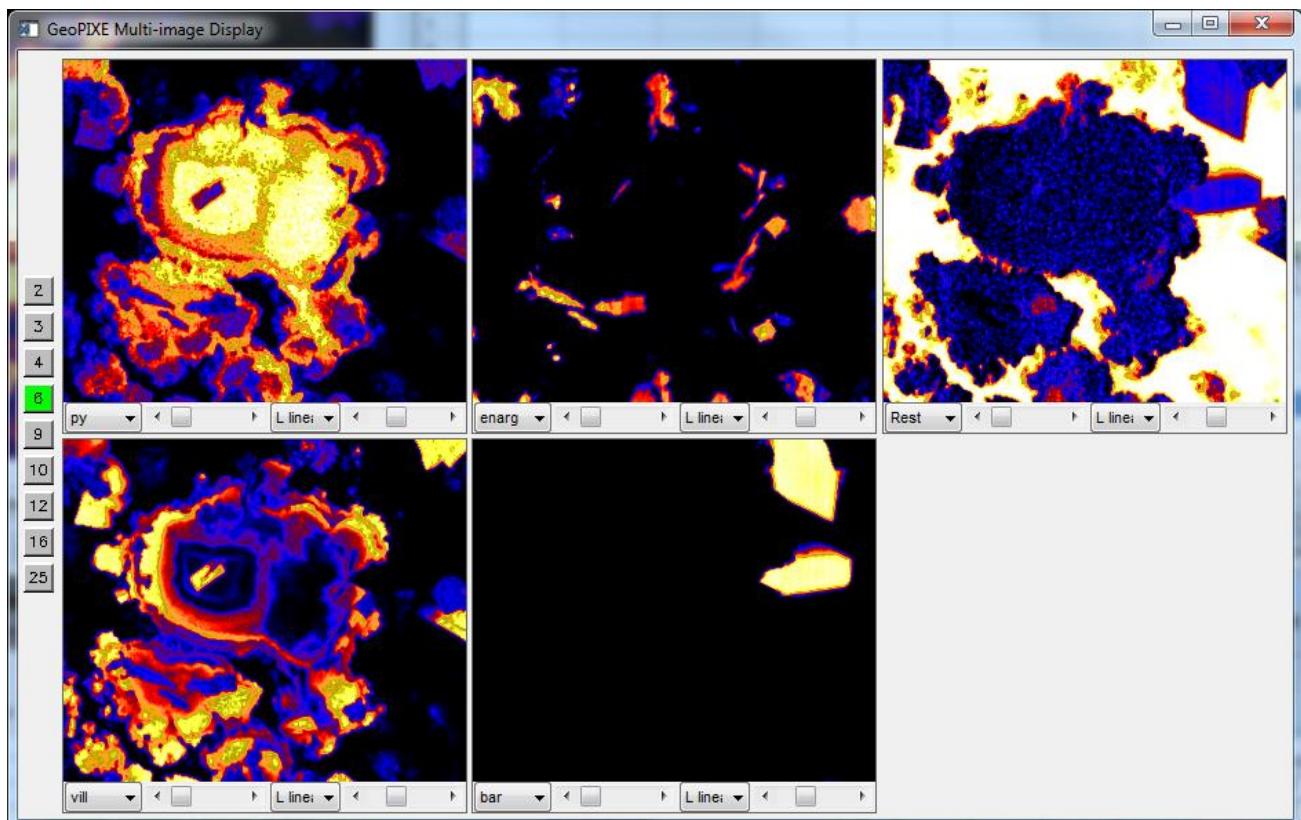
0	15.2%	45.1%		
1				46.1%
2	12.1%	65.1%	16.1%	
3	41.8%	0.57%		
4	16.3%	39.4%	0.46%	

Region sums for corrected images (left table) and the original images (right table)

- c. Note how the concentrations in the Cu-rich phases (e.g. region #0,2,4) and As-rich phase (e.g. region #2) change dramatically after correction due to the change in end-member phase mix compared to the original DA assumption.
- d. Electron microprobe analysis of region #4 gave: Fe = 15.4%, Cu = 32.8%. The corrected PIXE image region values are now within about 3%.

#### Transform onto maps of end-member proportions

10. Now click on the “Minerals” button in the *Correction Images for Composition* window, which will transform the element images into images of end-member phase proportion. “Rest” shows the image of the remainder, what’s left over (1 - sum of all end-members).



End-member phase proportion maps. “Rest” in the remainder fraction attributed to quartz in this case. Note the parts that were high Cu but became enargite (low “vill” and high “enarg”).

## Example F2: MPDA Imaging - SXRF (the detailed way)

Construct fully matrix (composition) corrected SXRF images using the new Multiphase DA (MPDA) method. This example works through it in detail. In future we may have an MPDA Wizard to guide/automate the process.

- Background and the goals.
- Identifying end-members and fitting a representative spectrum for each to make a DA matrix.
- Constructing a correction matrix scheme, similar to the **Correction of Yields** example.
- Using this CORRECT matrix to form “Phase Maps”, maps of end-member proportion.
- Repeating the *Sort EVT* step, now with MPDA enabled, using the Phase Maps and CORRECT matrix.
- Examining region spectra – are we better off now?

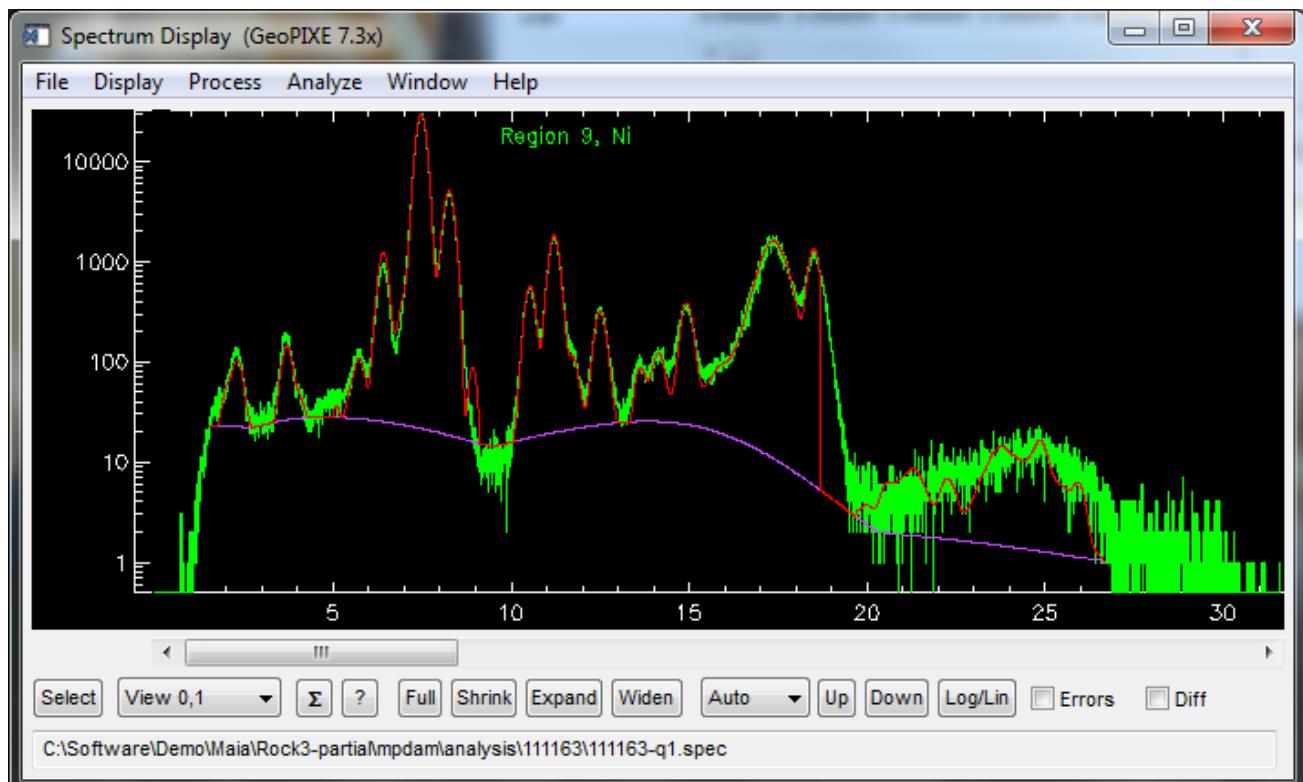
**Refer to section “Multiphase DA imaging (MPDA)” in the GeoPIXE Users Guide and read the background notes above under “Correction of Images for Composition Variation”.**

#### Background on MPDA

1. The **Correction of Yields** example above used a decomposition of images into maps of end-member proportion to refine the yields per pixel to correct the images for better concentrations per pixel. However, this ignores the effects of composition on X-ray relative intensities (which affects spectral overlaps and deconvolution into element terms) and background shape.
2. The MPDA method aims to extend the Correction of Yields approach to also track changes in X-ray relative intensities and background shape as composition changes spatially. It does this by re-processing the event data armed with the Phase Maps and the CORRECT matrix. It uses the end-member components in each pixel to blend both the yields as well as intensities and background shape to better accumulate each event’s contribution to the images so that overlaps and background are subtracted more accurately.
3. The ‘blend’ of terms uses the principal of averaging 1/yield terms in proportion to end-member weight fraction, to mirror the Bragg rule, as discussed in the **Correction of Yields** example (see Ryan (2000) paper in Help directory).

#### Load the DA images and normal regions and extracted spectra

4. Load the images (“111163.dai”) from run #111163 (directory “**Demo/Maia/Rock3/mpdam/analysis/111163/**”, data courtesy of Anais Pages, CSIRO), and open the *Image Regions* window and load the regions file “111163-q1.region”, from this directory (note there are more regions in this file than in the earlier example).
  - a. Browse around the element images and the regions. Note any regions that seems to have a poor overlay (violet = background, red = elements plus background). For example, see region #9.



Region #9 spectrum and overlay. Note the poor intensity ratios for Fe and Ni and the background that seems too high at 9-10 keV

#### Identifying end-members and fitting a representative spectrum for each to make a DA matrix

5. From the images you will see that there are a number of dominant elements detected that will have a significant effect on self-absorption (and hence yields and relative intensities). See the table below.
  - a. Browse around the element images and note which ones have peak concentrations of many wt%.
  - b. Some high-Z elements may have an effect at even lower concentrations (e.g. U).

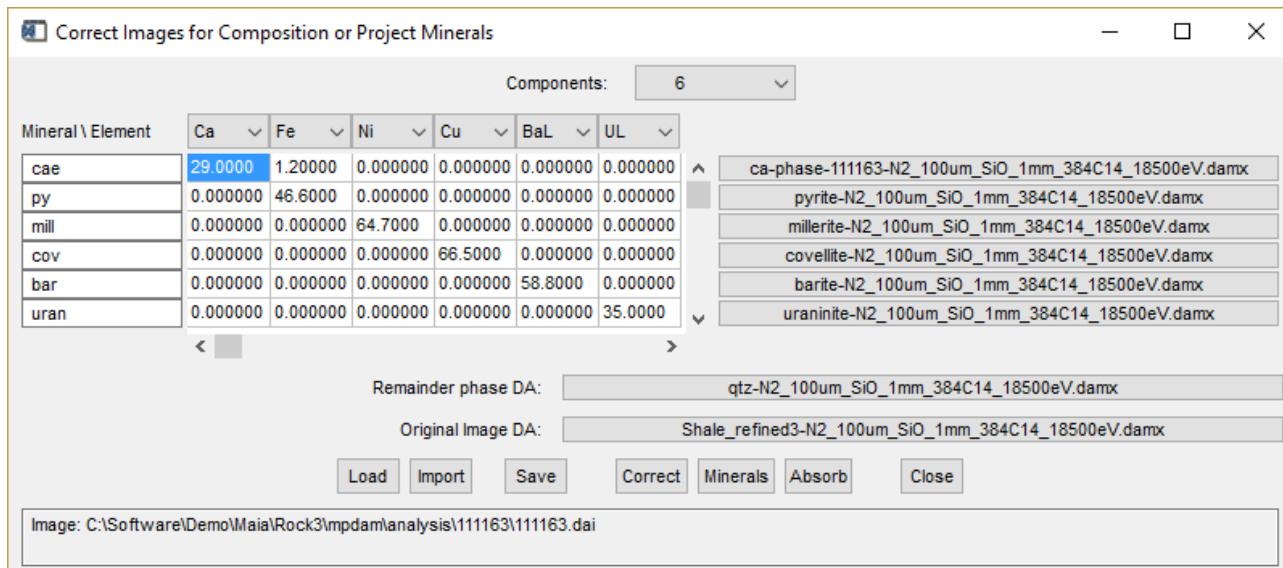
Region	name	phase	notes
3	cae	ca-phase	empirical collophane (Ca phosphate) diluted with silicate
7	py	pyrite	FeS <sub>2</sub>
9	mill	millerite	NiS
10	cov	covellite	CuS
25	bar	barite	BaSO <sub>4</sub>
35	uran	uraninite	UO <sub>2</sub> diluted with 60% wt SiO <sub>2</sub>
5	Rest	quartz	light gangue “remainder” represented as quartz SiO <sub>2</sub>

Regions that sample the main identified ‘effective’ end-member phases

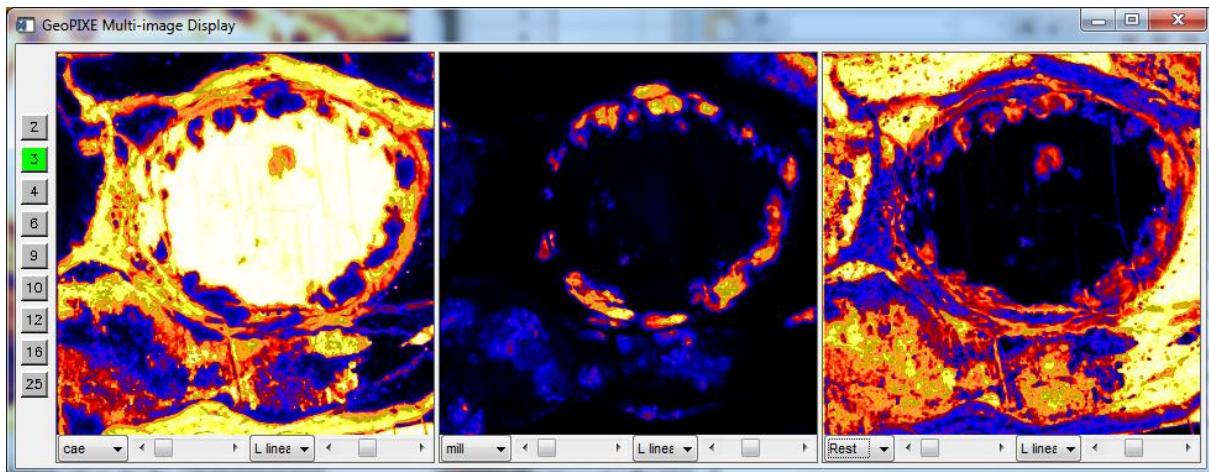
6. For each end-member, fit the region spectrum that samples it well, so that we get a reasonable fit to its background shape.
  - a. Select the region listed in the table and load the PCM file that will be used to fit it. These have names like “ca-phase-111163-N2\_100um\_SiO\_1mm\_384C14\_18500eV.pcm”.
  - b. Fit the spectrum, and generate a DA matrix for each one, using the default file names, which should follow the PCM file names.
  - c. Remember to select a region that approaches the Remainder (“Rest”) phase and fit that too.

#### Constructing a correction matrix scheme and projecting Phase Maps

7. Open the Correct Yields window using menu “Window→Correct Yields (N=6)” and load the CORRECT file “phases4.correct”.
  - a. The element dropdown column headings show the main major elements that help distinguish between phases.
  - b. The row headings show our end-member phase names (“cae”, “py”, etc.).
  - c. The matrix table contains the concentration of each of these elements in the end-members in wt%.
  - d. The buttons on the right select the DA matrix for each end-member phase. Click on each and load the DA matrices you have just generated.
  - e. The “Remainder DA” button is used to select your remainder DA matrix (“Rest” in the table).
  - f. The “Original DA” shows the DA matrix used originally to make the first DA images. Leave that be.



8. Correct Yields and project Phase Maps.
  - a. Click on “Correct” 3 times to correct images for composition in 3 iterations. Note how the image for an element such as Ni becomes reduced in dynamic range. The third click shows no significant change.
  - b. Save the corrected images as “111163-corrected4.dai”.
  - c. Click on “Minerals” to project these element maps onto maps of phase proportion, labelled by the end-member name.
  - d. Save these as “111163-phases.dai”. These are your new Phase Maps.



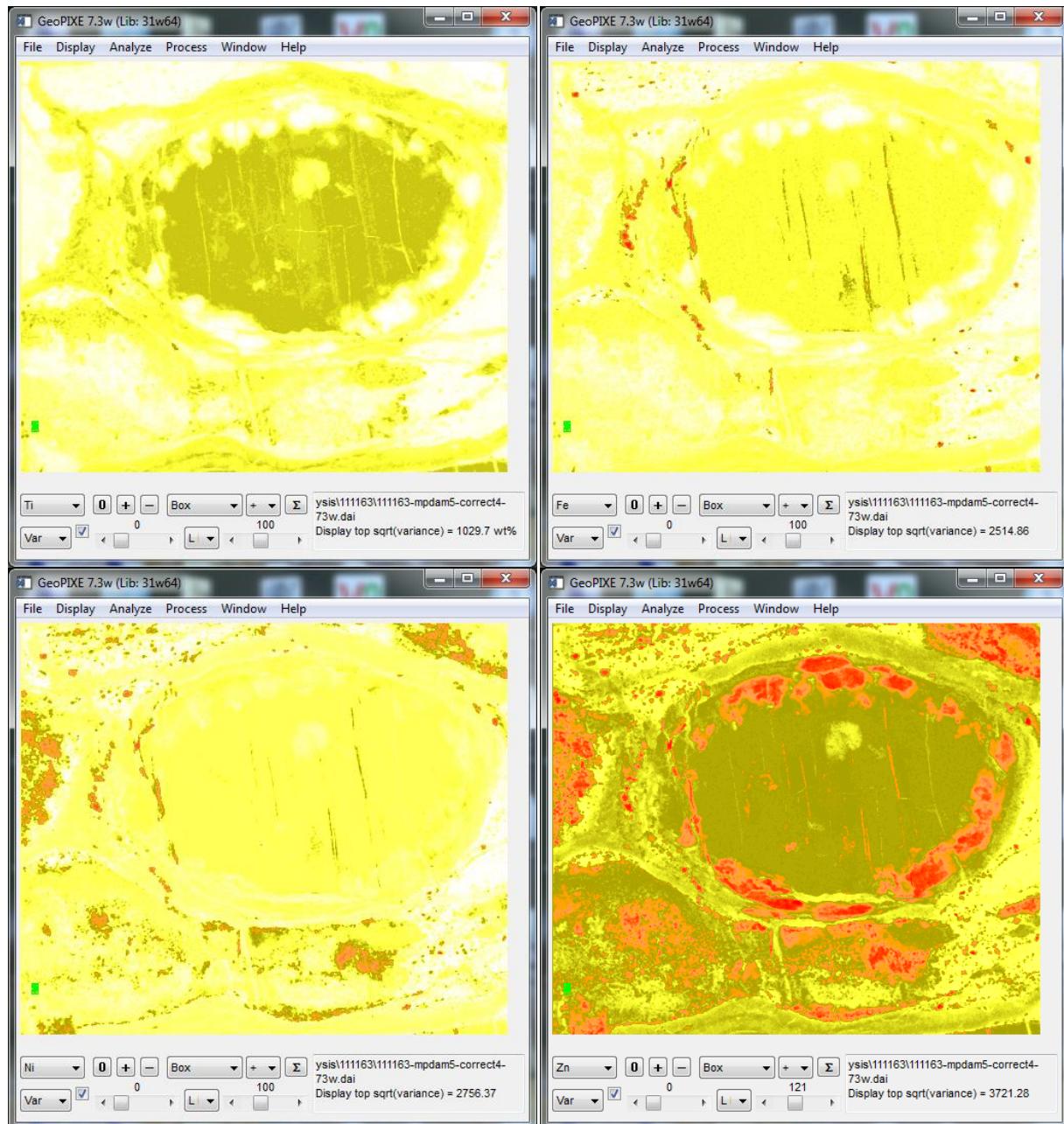
Three of the 7 Phase Maps. The last image is “Rest” for the light silicate remainder

#### Using MPDA to re-process your event data

9. Load the original DAI file “111163.dai” into the *Sort EVT* window using the “From DAI” button.
  - a. Go to the “DA/E.Cal” tab and change the “Projection” mode to “Multiphase (MPDA)”.
  - b. Click on “new” to open the MPDA set-up pop-up and select:
    - a. “Phase Maps”: your new phase map images.
    - b. “Correct yields”: the CORRECT file “phases4.correct” (in parent dir).
    - c. “New MPDAM file”: an output file-name, such as “111164-correct4.mpdam” in your 111163 dir (note that this file is specific to this run #111163 as it contains the phase maps).
  - c. Change the output file-name to something like: “111163-MPDA.dai”.
  - d. Select “cluster” mode to enable parallel processing (MPDA is slower than normal DA).
  - e. Click on “Start” to start processing.

#### What's new in MPDA images?

10. Explore the following additional plugin results (menu “Process→User Plugins”):
  - a. **“Yield maps (var)”**: Loads the final per pixel yields into the ‘var’ maps that parallel the element ‘conc’ maps.
    - a. Use the dropdown below the element dropdown to select “var”. The ‘var’ maps are used as these and the yields have half the spatial resolution compared to the conc maps.
    - b. Contrast the Fe and Ni yield maps (var). The Ni yields drop substantially in areas of high Fe due to the strong absorption of Ni K $\alpha$  above the Fe K edge.
  - b. **“Phase maps”**: Loads the phase maps, which are stored in the MPDA image DAI file.
    - a. Note that the element names do not change, so the “ca-phase” appears as “Back”, “py” as “Si”, “mill” as “P”, “Rest” as “Ca”, etc. To look at these phases, it is better to load the phase map image file. (Note: A better way to view the phases is to open a new *Image* window (unlinked) and load the phase maps.)



*Yield maps: Note lower yields for Ti in Ca phase, Ni in Fe phase compared to Fe and Zn in Ni phase. The lows left of centre (in Fe, Ni, Zn) are high in Ba, Cr, which absorbs Fe, Ni, Zn, etc.*

#### Update region sums and extract spectra from regions

11. Load the MPDA images again ("111163-mpda.dai") and then the original regions file "111163-q1.region" into the *Image Regions* window.
  - a. Click on "Update All" to apply the region pixel selections to the new MPDA images.
  - b. Save these new region sums in file "111163-mpda-q1.region".
12. Select "array" in the dropdown next to "EVT" in the *Image Regions* window.
  - a. Click on "EVT" to extract spectra from all regions; confirm the output SPEC file-name (should be the same as your region file).

#### Compare MPDA regions to normal DA

13. Open an independent *Image* window using menu "Window→Image Display (unlinked)" and load the original image file "111163.dai". Open an *Image Regions* window and a *Spectrum Display* window from there and load the original regions file "111163-q1.dai"; their spectra should appear in the matching *Spectrum Display* window.
  - a. First compare the images for DA versus MPDA.
    - a. Try the Ni image. Note how much the display maximum has changed with a better matrix correction (52.6 wt% now becomes 38.1 wt%).

- b. Click on a row in the *Image Regions* window attached to your MPDA images and click a matching row in the normal DA regions table.
- c. Compare the spectra in the attached *Spectrum Display* windows. Do you see improvements in terms of X-ray line relative intensives and/or background?

#### **How to apply MPDA to further data/images in the same sample series?**

14. To apply the same end-member decomposition to other samples, or areas on the same sample, where similar minerals and elements are involved, follow these steps:
  - c. **Normal DA image**
    - a. First sort the new sample/area using the normal DA approach.
  - d. **Build phase maps**
    - a. Use the *Correct Yields* window, and the same CORRECT file, to “Correct” the images for sample composition variation. Do this for 3 iterations.
    - b. Map onto “Mineral” proportions. These are the phase maps we need.
    - c. Save these to a phase DAI file (e.g. append “-phases.dai” to the name).
  - e. **Create MPDAM file**
    - a. Select “Multiphase DA (MPDA)” mode on the DA/E.Cal tab.
    - b. Click on “New” next to “File” and select the new phase maps DAI file and the CORRECT file to create the MPDAM file.
  - f. **Change output filename?**
    - a. Typically, you would change the filename (e.g. append “-mpda.dai” to the name).
  - g. **Process using MPDA**
    - a. Click “Start” (with “cluster” enabled usually).
15. **NEW:** Automated MPDA workflow feature
  - a. Starting with GeoPIXE version 7.6u, the *Sort EVT* window automates these steps above.
    - a. Select the new source raw data files “xxxx”. The output file-name will get a “-MPDA.dai” appended.
    - b. Click on “Start”.
    - c. If the phase maps “xxxx-phases.dai” do not exist, they will be generated from the Normal DA maps (“xxxx.dai”) using image yield correction (3 passes) based on the CORRECT file referenced in the MPDAM file.
    - d. A new MPDAM file (“xxxx.mpdam”) will be generated as well.
    - e. If Normal DA maps do not exist, a normal DA sort will be done first into file “xxxx.dai”.
    - f. Once Normal DA maps are done, yield correction and phase project are done and there is a new MPDAM file, then the MPDA sort will proceed automatically as a further pass.

### **Example F3: MPDA Imaging - PIXE (the detailed way)**

*Construct fully matrix (composition) corrected PIXE images using the new Multiphase DA (MPDA) method. This example works through it in detail, while a later example will use the new MPDA Wizard.*

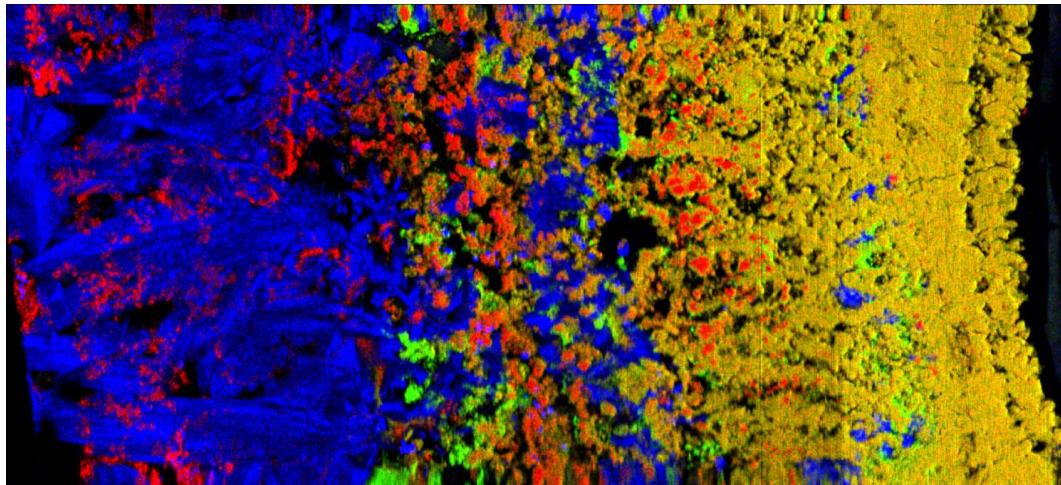
- Background and the goals.
- Identifying end-members and fitting a representative spectrum for each to make the DA matrices.
- Constructing a correction matrix scheme, similar to the Correction of Yields example.
- Using this CORRECT matrix to form “Phase Maps”, maps of end-member proportion.
- Repeating the Sort EVT step, now with MPDA enabled, using the Phase Maps and CORRECT matrix.
- Examining region spectra – are we better off now?

#### **Background on MPDA**

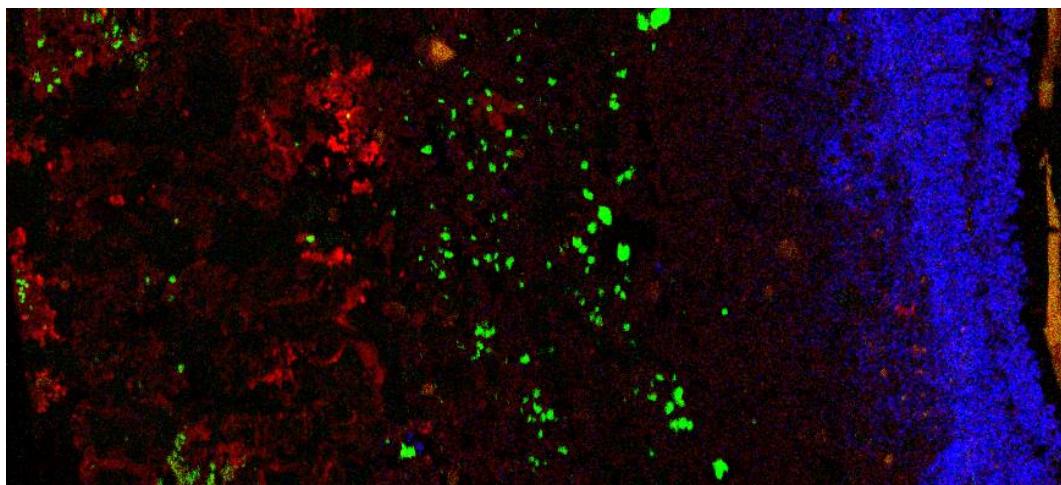
1. The Correction of Yields example used a decomposition of images into maps of end-member proportion to refine the yields per pixel to correct images for better concentrations per pixel. However, this ignores the effects of composition on X-ray relative intensities (which affected spectral deconvolution into element terms) and background shape.
2. The MPDA method aims to extend the Correction of Yields approach to also track changes in X-ray relative intensities and background shape as composition changes spatially. It does this by re-processing the event data armed with the Phase Maps and the CORRECT matrix. It uses the end-member components in each pixel to ‘blend’ both the yields as well as intensities and background shape to better accumulate each event’s contribution to the images so that overlaps and background are subtracted more accurately.
3. The ‘blend’ of terms uses the principal of averaging 1/yield terms in proportion to end-member weight fraction, to mirror the Bragg rule, as discussed in Ryan (2000).

#### **Load the DA images and normal regions and extracted spectra**

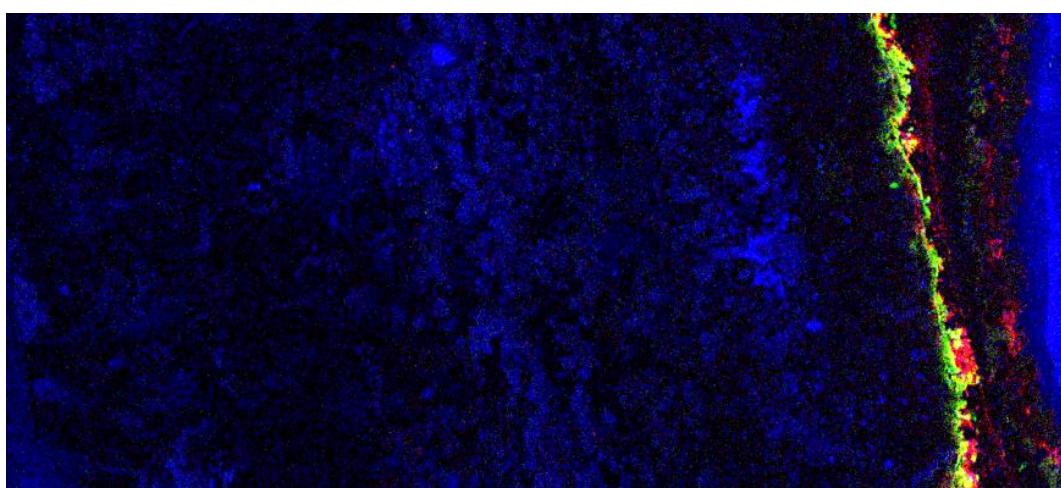
4. Load the images “216-16Hz-m.dai” (directory “**Demo\CSIRO\Smoker\**”, data courtesy of Chris Yeats, CSIRO), and open the *Image Regions* window and load the regions file “216-16Hz -q1.region”.



Fe Cu Sr RGB

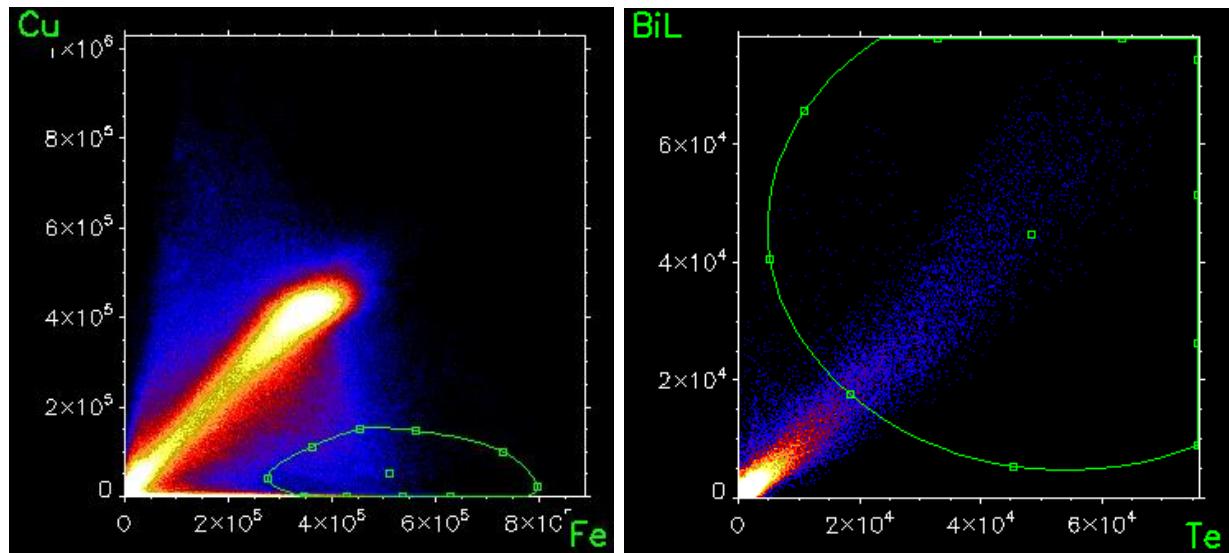


Zn Ba Se



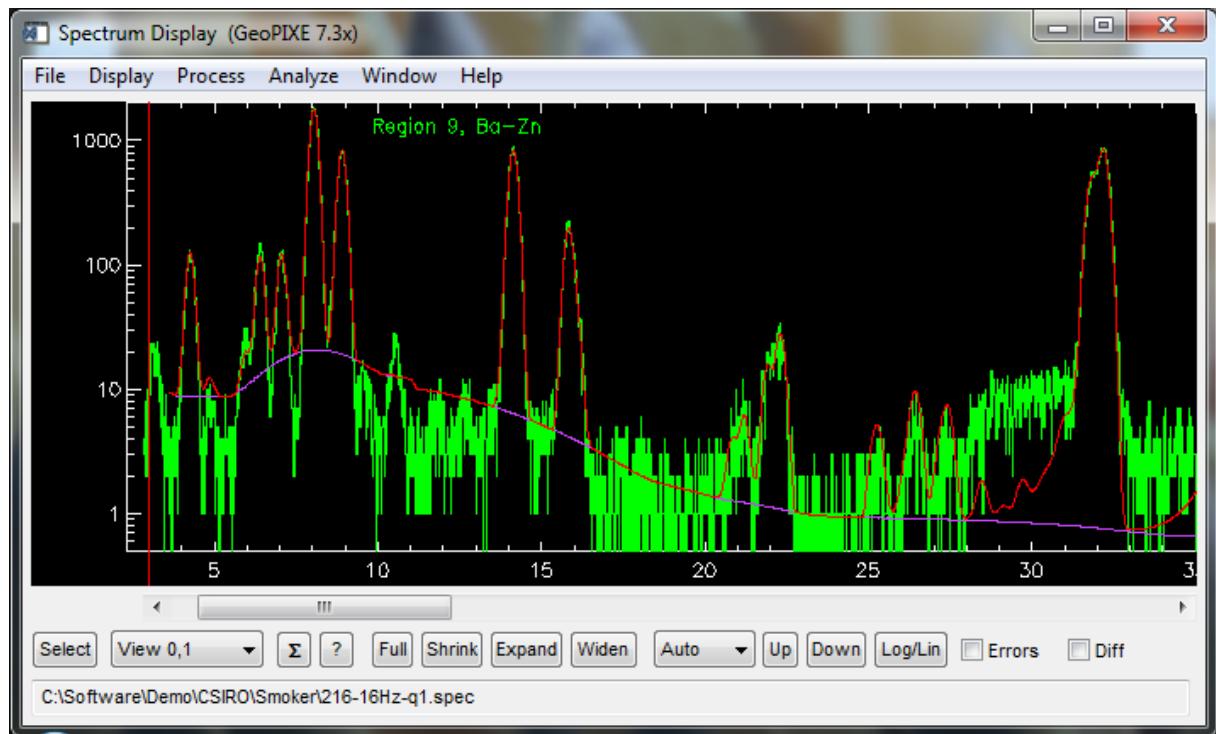
Bi Mo As

5. Note that many of these regions have been set using the element *Associations* window, such as for region #2, 8:

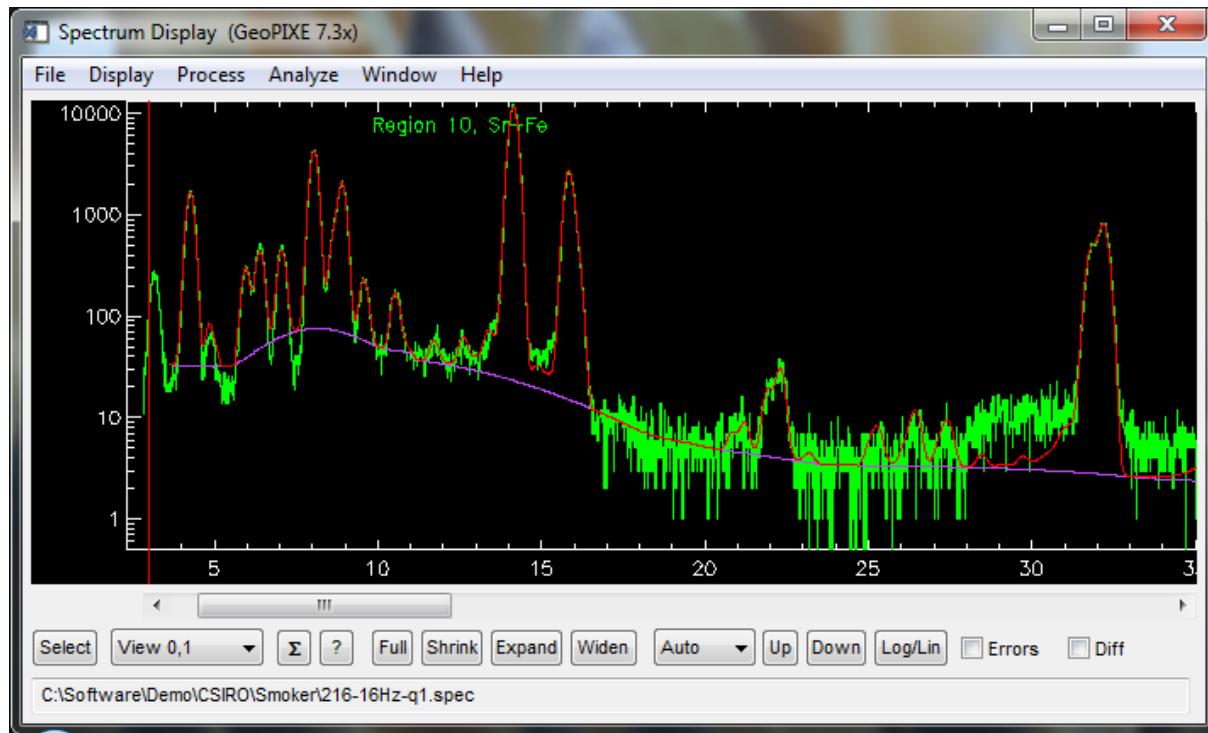


Element associations showing the selections for regions #2 and 8.

- Browse around the element images and the regions. Note any regions that seem to have a poor overlay (violet = background, red = elements plus background). For example see regions #9, 10.



Region #9, Background shape not right



Region #10, Background shape not right, and poor line intensities on the Mn, Fe peaks (5.9, 6.4, 7.0 keV)

- Also note any regions with strange compositions reported, such as region #3 which shows 64.9% Cu, which is more than any known Cu phase in this sample. This is because these initial DA images assumed a composition of pure pyrite everywhere, which is only a first approximation and a poor approximation for a Cu-rich phase.

#### **Identifying end-members and fitting a representative spectrum for each to make a DA matrix**

- From the images you will see that there are a number of dominant elements detected that will have a significant effect on self-absorption (and hence yields and relative intensities). See the table below.
  - Browse around the element images and note which ones have peak concentrations of many wt%.
  - Some high-Z elements may have an effect at even lower concentrations (e.g. Ba, Te).

Mineral	Name	Element #	Region *	Charge
pyrite	Py	Fe	2 (0)	0.48
covellite	Cov	Cu	3 (1)	0.208
sphalerite	Sph	Zn	0 (2)	0.157
arsenopyrite	Aspy	As	5 (3)	0.127
carbonate w/ Sr	Carb	Sr	10 (4)	2.99
barite	Bar	Ba	9 (5)	0.157
complex sulphide	Sul	Bi	8 (6)	0.135
silicate gangue	Qtz	rest	11 (7)	0.218

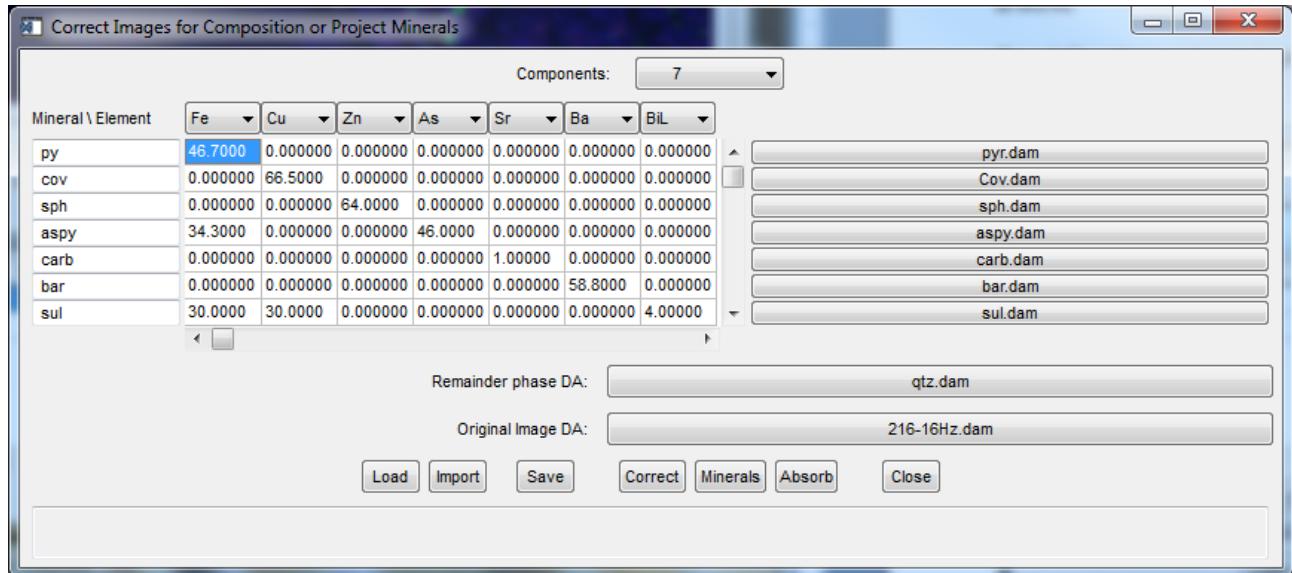
Regions that sample the main identified end-member phases (\*first number is index into "216-16Hz-q1.region" and the number in brackets is the index into "216-Hz-phases-q1.region") (# Main element that identifies the end-member)

- For each end-member, fit the region spectrum that samples it well, so that we get a reasonable fit to its background shape.
  - Select the region listed in the table and load the PCM file "216-16.pcm" into X-ray Spectrum Fit. Then load the yield file appropriate to that end-member with names like "covelite.yield".
  - Fit the spectrum, and generate a DA matrix for each one, selecting a file name that matches the end-member (e.g. "Cov.dam").

#### **Constructing a correction matrix scheme and projecting Phase Maps**

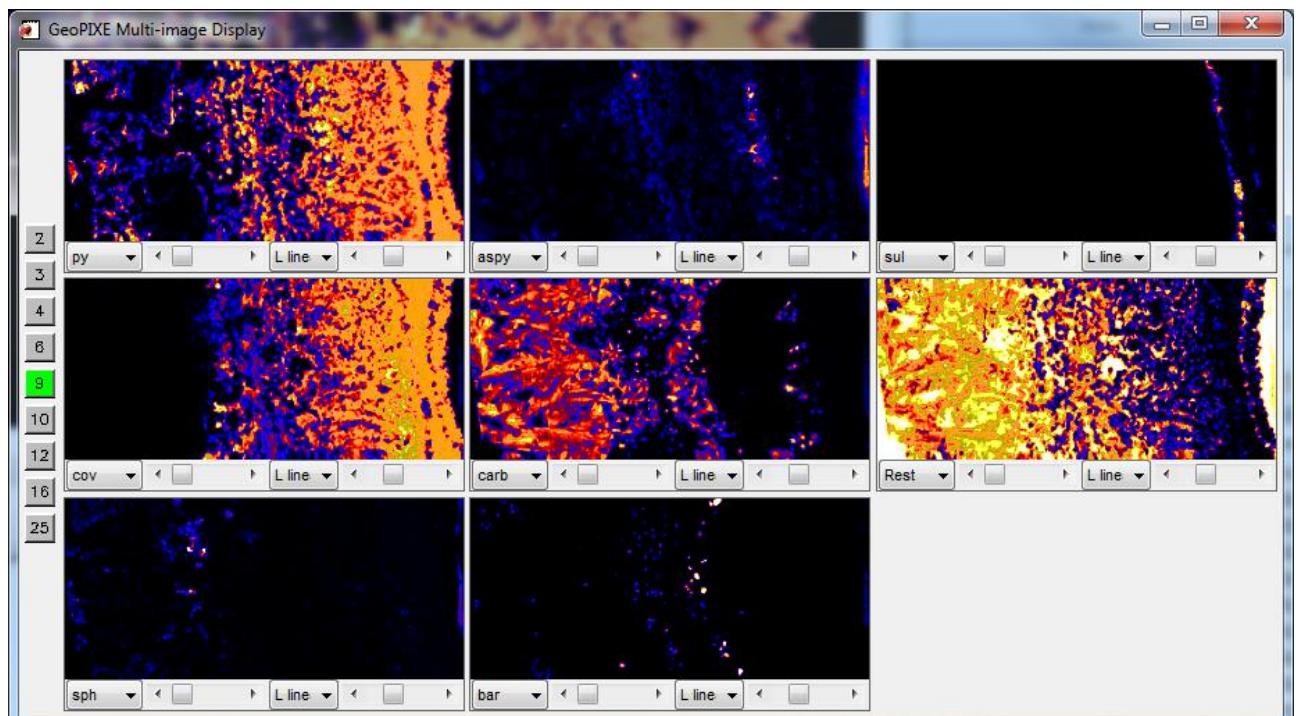
- Open the Correct Yields window using menu "Window→Correct Yields (N=16)" and load the CORRECT file "phase.correct".
  - The element dropdown column headings show the main major elements.

- h. The row headings show our end-member phase names ("py", "cov", etc.).
- i. The matrix table contains the concentration of each of these elements in the end-members in wt%.
- j. The buttons on the right select the DA matrix for each end-member phase. Click on each and load the DA matrices you have just generated.
- k. The "Remainder DA" button is used to select your remainder DA matrix (qtz in this case).
- l. The "Original DA" shows the DA matrix used originally to make the first DA images. Leave that be.

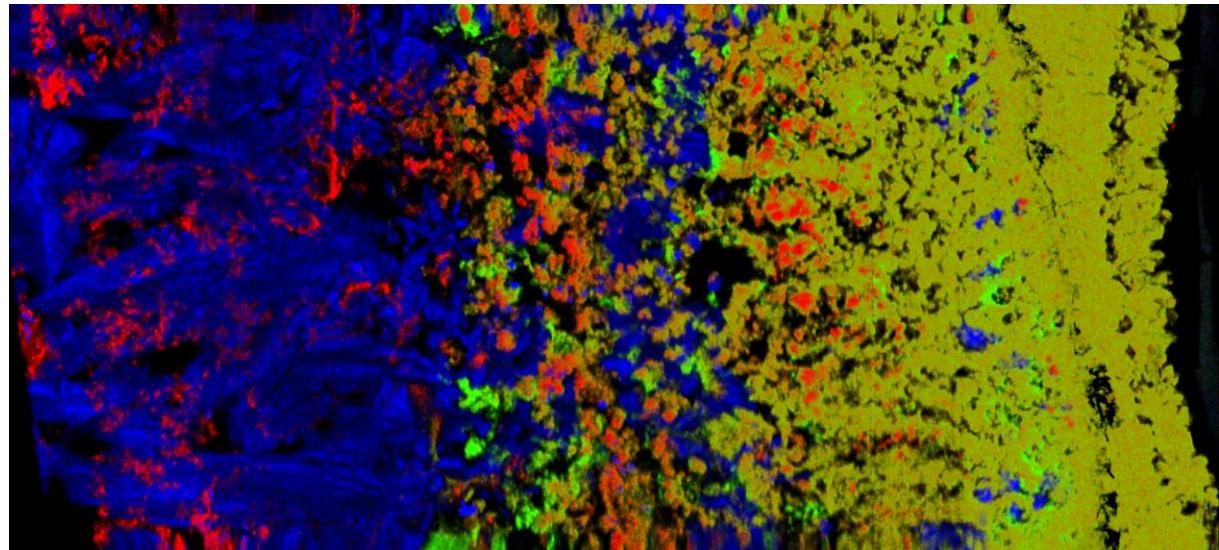


11. Correct Yields and project Phase Maps.

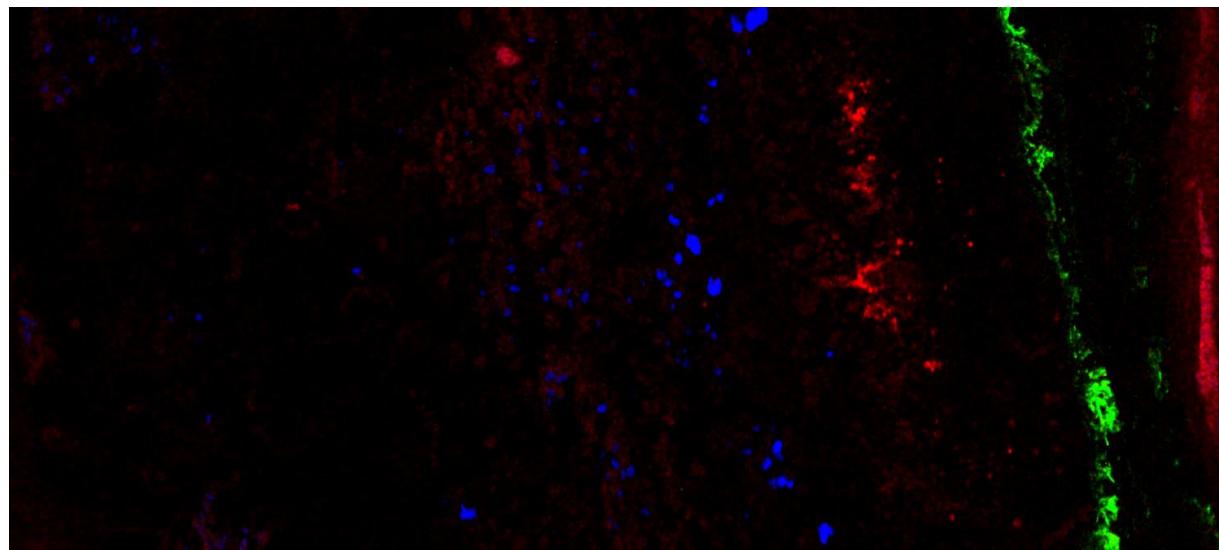
- e. Click on "Correct" 3 times to correct images for composition in 3 iterations. Note how the image for an element such as Cu becomes reduced in dynamic range.
- f. Save the corrected images as "216-16Hz-correct.dai".
- g. Click on "Minerals" to project these element maps onto maps of phase proportion, labelled by the end-member name.
- h. Save these as "216-16Hz-correct-minerals.dai". These are your new Phase Maps.



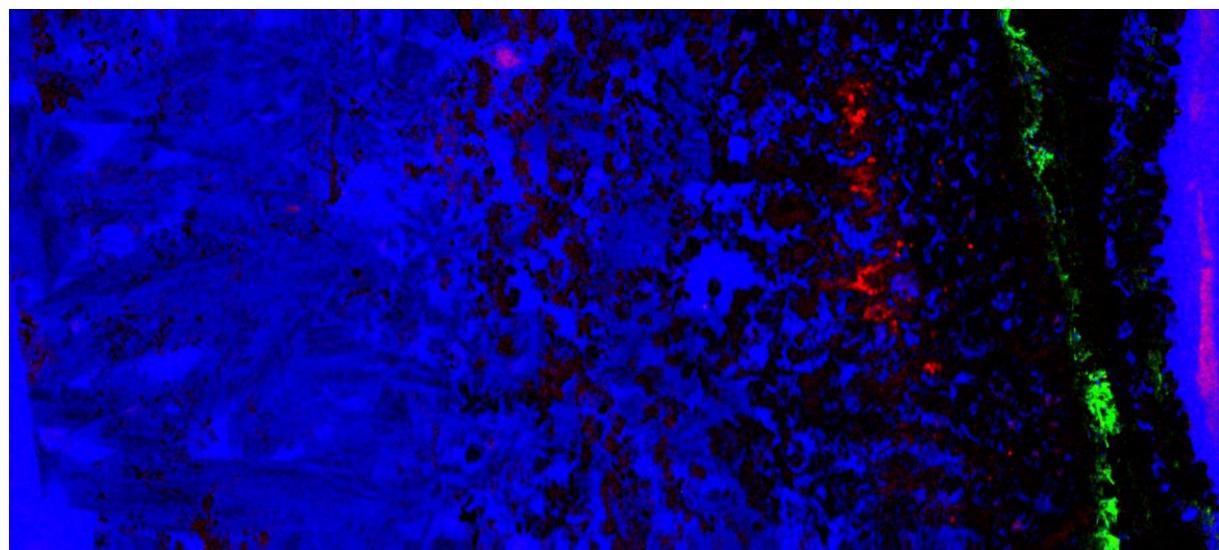
Phase maps



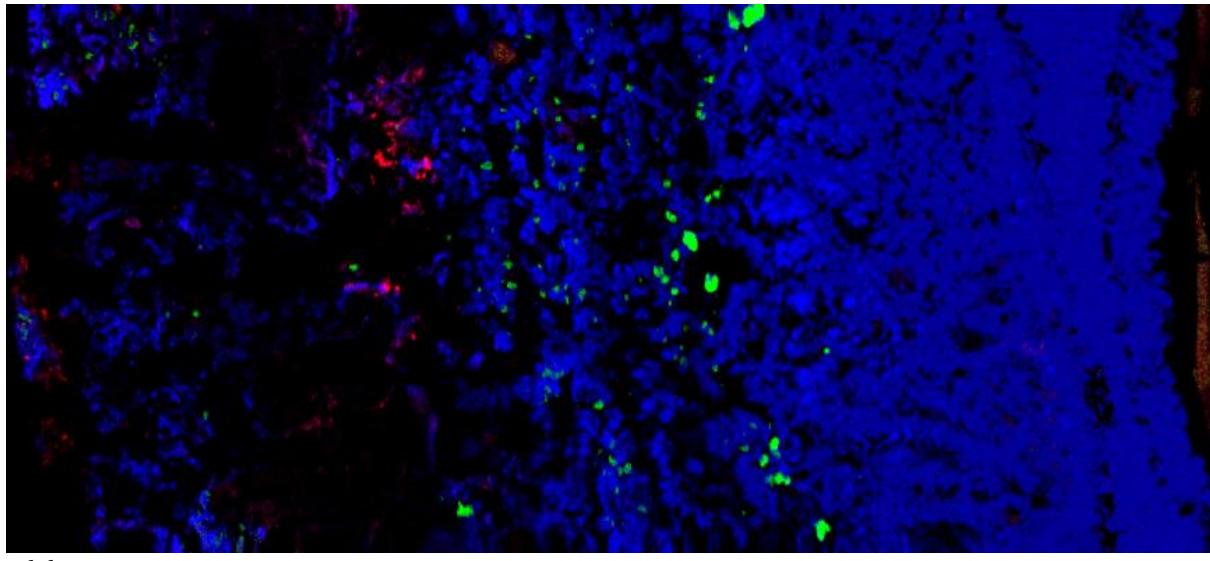
*py cov carb RGB*



*asp y sul bar*



*asp y sul Rest*



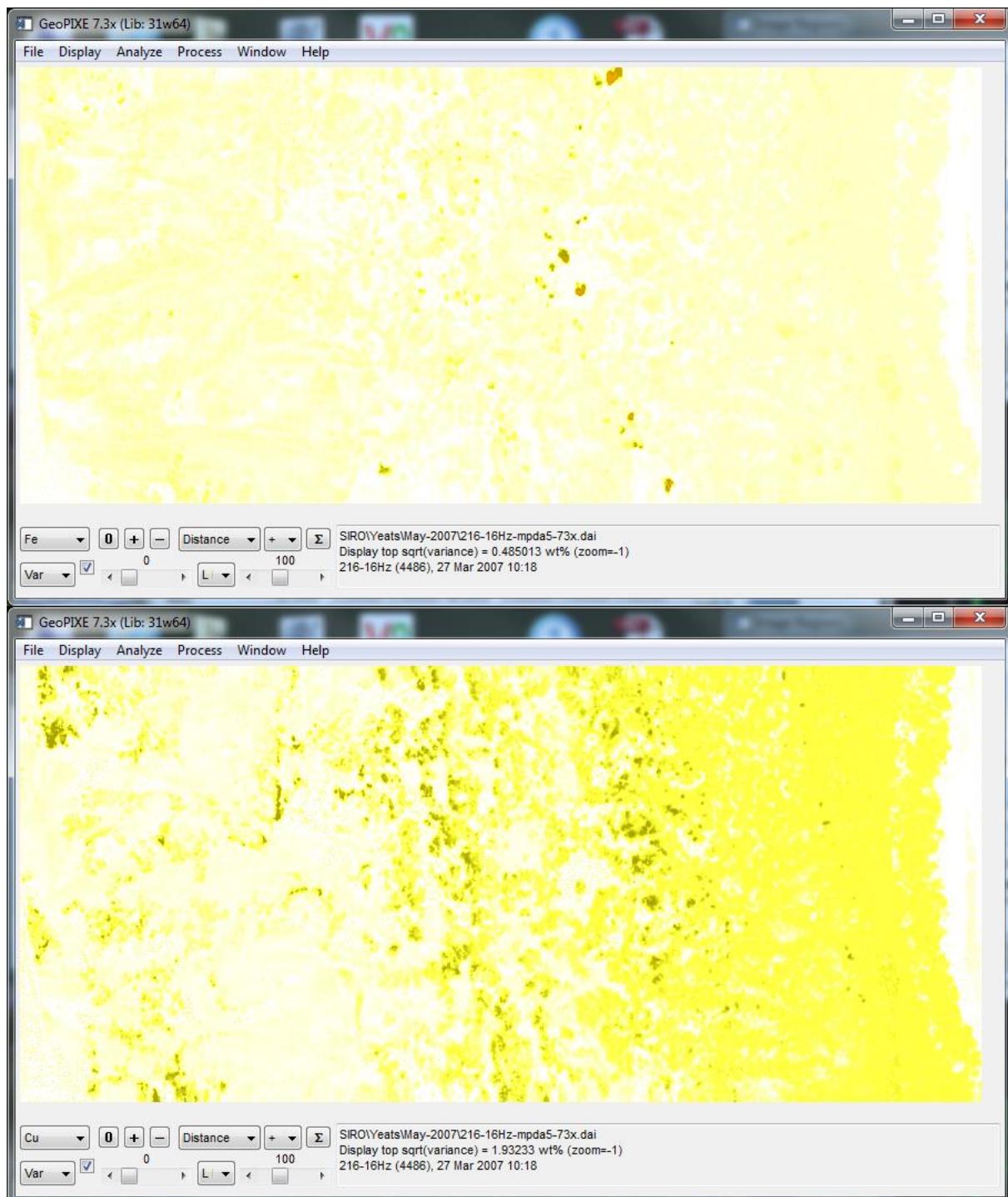
sph bar py

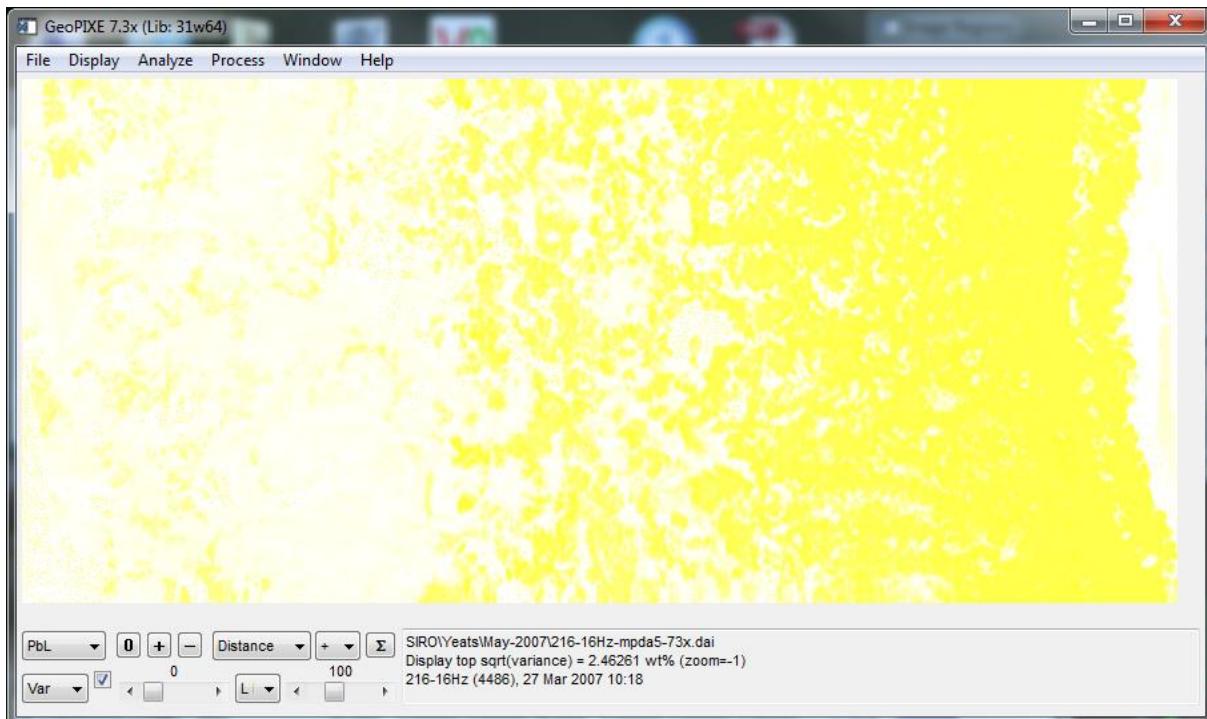
#### Using MPDA to re-process your event data

12. Load the original DAI file “216-16Hz.dai” into the *Sort EVT* window using the “From DAI” button.
  - f. Locate the raw data file in the directory: “Demo/CSIRO/Smoker/raw”.
  - g. Go to the “DA/E.Cal” tab and change the “Projection” mode to “Multiphase (MPDA)”.
  - h. Click on “new” to open the MPDA set-up pop-up and select:
    - a. “Phase Maps”: your new phase map images.
    - b. “Correct yields”: the CORRECT file “phase.correct” (in parent dir).
    - c. “New MPDAM file”: an output file-name, such as “216-16Hz.mpdam” in your Smoker dir.
  - i. Change the output file-name to something like: “216-16Hz -mpda.dai”.
  - j. Click on “Start” to start processing.

#### What's new in MPDA images?

13. Explore the following plugin results (“Process→User Plugins”):
  - b. “Yield maps (var)”: Loads the final per pixel yields into the ‘var’ maps that parallel the element ‘conc’ maps.
    - a. Use the dropdown below the element dropdown to select “var”.
    - b. Contrast the Fe and Cu yield maps (var). The Cu yields drop substantially in areas of high Fe due to the strong absorption of Cu K $\alpha$  above the Fe K edge.
  - c. “Phase maps”: Loads the phase maps, which are stored in the MPDA image DAI file.





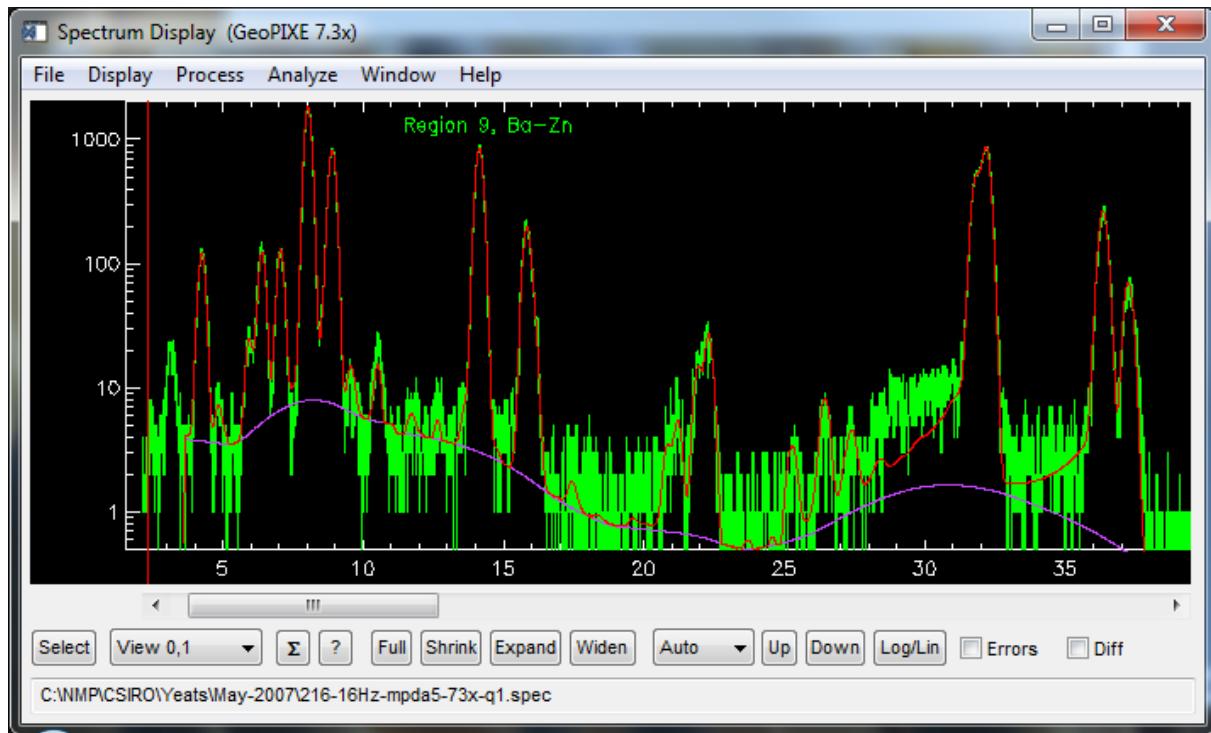
*Yield maps: Note lower yields for Fe in Ba phase, Cu in Fe phase and PbL in Cu phase.*

#### **Update region sums and extract spectra from regions**

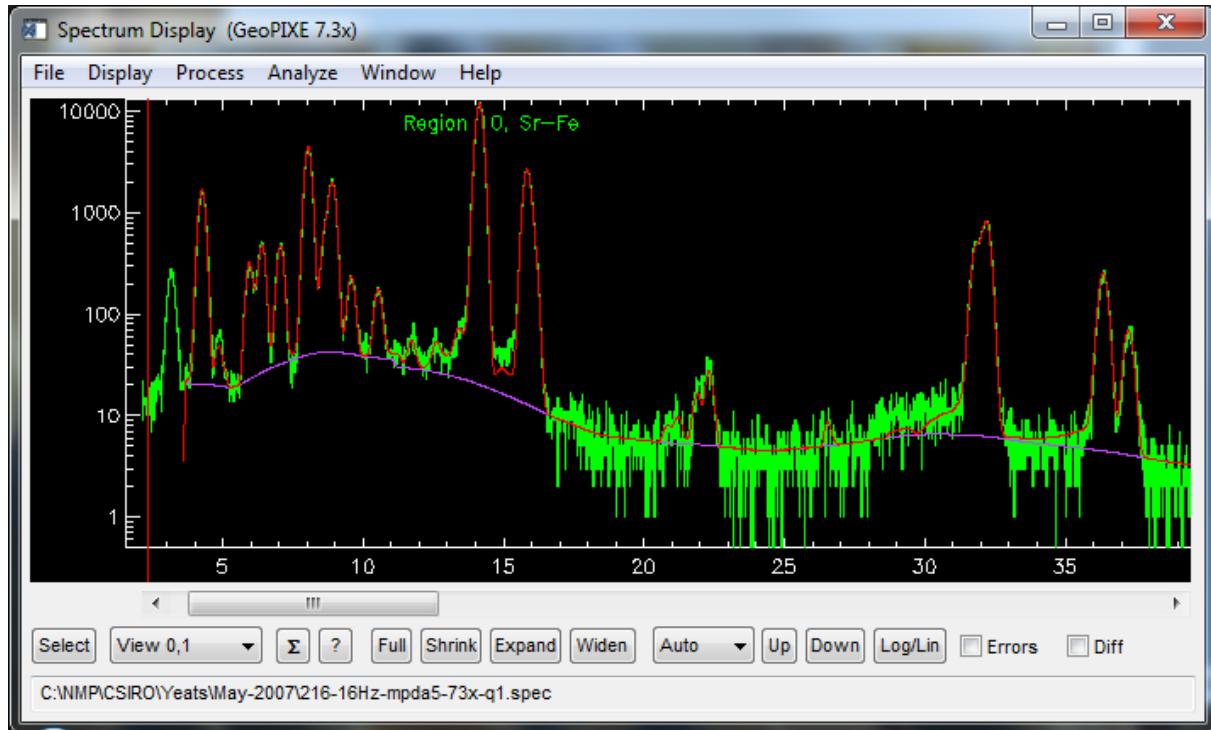
14. Load the original regions file “216-16Hz -q1.region” into the *Image Regions* window.
  - c. Click on “Update All” to apply the region pixel selections to the new MPDA images.
  - d. Save these new region sums in file “216-16Hz -mpda-q1.region”.
15. Select “1” in the dropdown next to “EVT” in the *Image Regions* window to select detector 1 used for PIXE
  - b. Click on “EVT” to extract spectra from all regions; confirm the output SPEC file-name (should be the same as your region file).

#### **Compare MPDA regions to normal DA**

16. Open an independent *Image* window using menu “Window→Image Display (unlinked)” and load the original image file “216-16Hz.dai”. Open an *Image Regions* window and a *Spectrum Display* window from there and load the original regions file “216-16Hz -q1.region”; their spectra should appear in the matching *Spectrum Display* window.
  - d. Click on a row in the *Image Regions* window attached to your MPDA images and click a matching row in the normal regions table.
  - e. Compare the spectra in the attached *Spectrum Display* windows. Do you see improvements in terms of X-ray line relative intensives or background?



Region #9 spectrum and overlay – compare it to the previous normal DA – note improved background



Region #10 spectrum and overlay – compare it to the previous normal DA – note improved background and Fe lines

#### NEW: Automated MPDA workflow feature

17. Starting with GeoPIXE version 7.6u, the *Sort EVT* window automates the application of an MPDAM specification to new raw data file..
  - d. Select the new source raw data files “xxxx”. The output file-name will get a “-MPDA.dai” appended.
  - e. Click on “Start”.
  - f. If the phase maps “xxxx-phases.dai” do not exist, they will be generated from the Normal DA maps (“xxxx.dai”) using image yield correction (3 passes) based on the CORRECT file referenced in the MPDAM file.
  - g. A new MPDAM file (“xxxx.mpdam”) will be generated as well.

- h. If Normal DA maps do not exist, a normal DA sort will be done first into file “xxxx.dai”.
- i. Once Normal DA maps are done, yield correction and phase project are done and there is a new MPDAM file, then the MPDA sort will proceed automatically.

## Example F4: Spectrum fitting using end-member analysis - SXRF

*Fitting the spectra of regions with some unknown average composition uses an end-member analysis to determine the proportion of end-members in a region in order to correct yields for composition and iterate towards an improved fit to composition in the region.*

*Refer to section “Multiphase Loop” in the GeoPIXE Users Guide.*

### Background

1. The **Correction of Yields** example above used a decomposition of images into maps of end-member proportion to refine the yields per pixel to correct the images for better concentrations per pixel. This same approach can be used to determine the proportion of end-members in a region in order to correct yields for composition and iterate towards an improved fit to composition.
2. The ‘blend’ of terms uses the principal of averaging 1/yield terms in proportion to end-member weight fraction, to mirror the Bragg rule, as discussed in the **Correction of Yields** example (see Ryan (2000) paper in Help directory).

### Load the DA images and some regions and extracted spectra

3. Load the images (“111163.dai”) from run #111163 (directory “**Demo/Maia/Rock3/mpdam/analysis/111163/**”, data courtesy of Anais Pages, CSIRO), and open the *Image Regions* window and load the regions file “111163-q1.region”, from this directory.

### Identifying end-members and model yields for each

4. This example uses the end-members from the previous example “**MPDA Imaging - SXRF (the detailed way)**”.

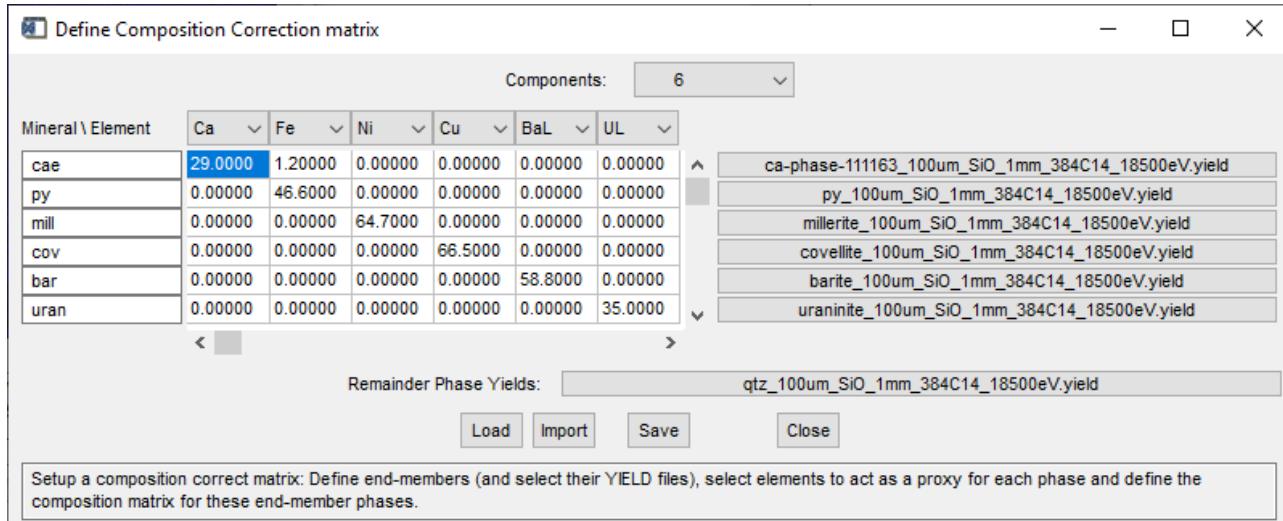
name	phase	notes
cae	ca-phase	empirical collophane (Ca phosphate) diluted with silicate
py	pyrite	FeS <sub>2</sub>
mill	millerite	NiS
cov	covellite	CuS
bar	barite	BaSO <sub>4</sub>
uran	uraninite	UO <sub>2</sub> diluted with 60% wt SiO <sub>2</sub>
Rest	quartz	light gangue “remainder” represented as quartz SiO <sub>2</sub>

5. For each end-member, create a yield model and generate yields (YIELD file) and also save the yield setup LCM file.
  - a. These YIELD files were already created for this previous example.

### Constructing a correction matrix scheme for fitting

6. Load the following PCM file into the *X-ray Spectrum Fit* window: “Shale\_refined3-N2\_100um\_384C14\_18500eV.pcm”.
7. Go to the “Multiphase Loop” tab and select “Multiphase fit, loop N=3” from the dropdown.
8. Use the “New” button on the “Multiphase” row to open the “*Define Composition Correction Matrix*” window.
  - m. The element dropdown column headings show the main major elements that help distinguish between phases.
  - n. The row headings show our end-member phase names (“cae”, “py”, etc.).
  - o. The matrix table contains the concentration of each of these elements in the end-members in wt%.
  - p. The buttons on the right select the yield model for each end-member phase. Click on each and load the YIELD files that you previously generated.
  - q. The “Remainder DA” button is used to select your remainder yield model (“Rest” in the table).

*NOTE: You can also import the matrix from the CORRECT file used for the example “**MPDA Imaging - SXRF (the detailed way)**”. Or load the file “phase4.comat”.*



#### **Fit region spectra using the Multiphase Loop**

9. Select a row of the *Image Regions* table to show its matching spectrum in the *Spectrum Display* window.
10. Click on “Fit One” in *X-ray Spectrum Fit*.
  - a. The fit will happen in 3 passes. After each pass, the proportion of end-members inferred from the fit will be used to improve the model of yields for this region. After 3 iterations, the final composition will have converged and the results will reflect the average composition of this region.
  - b. Repeat for other region spectra.
  - c. Open the *Fit Results* window to see the final fitted results.
  - d. For comparison, for the last region fitted, switch the mode in *X-ray Spectrum Fit* back to “Single phase, single fit”, and fit again. Compare the results.

## **G. Depth Mapping using the Maia Detector**

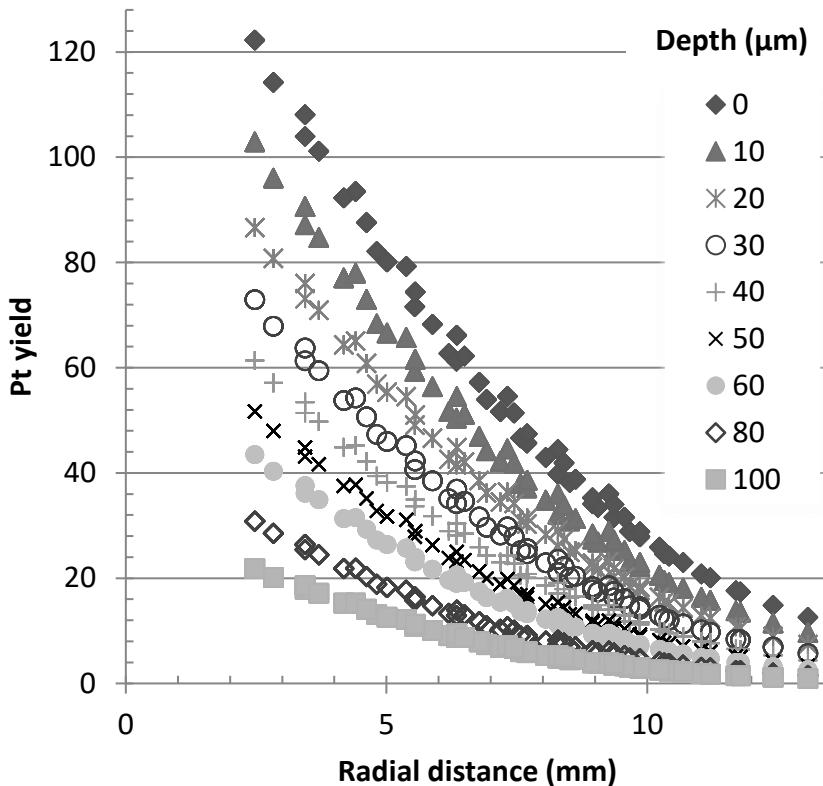
### **Example G1: Depth mapping and analysis – the Depth Wizard**

*Make use of the large range of take-off angles of detectors in the Maia array to*

- Model X-ray yield ratios with depth to provide a tool for depth measurement.
- Determine particle depths using the yield-ratio depth profile.
- Form images colour-coded for particle depth.
- Use the *Depth Wizard*, with its built-in explanation help text and figures, to guide you through the analysis steps.

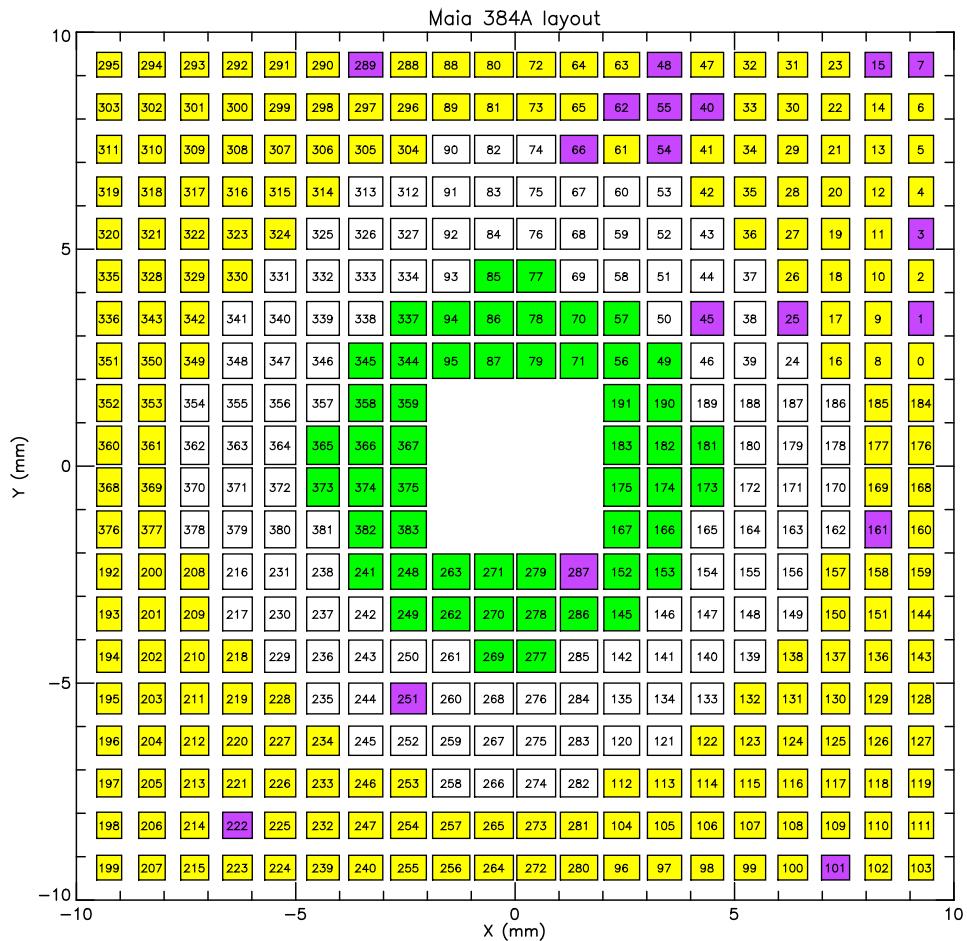
#### **Background Notes**

1. The large size of the detector array, located just 10 mm from the beam spot on the sample, provides a large range of take-off angles at the sample surface, with the angle between sample normal and rays to masked detector element centre varying between 13.9° and 52.6°. This makes the outer detector elements much more sensitive to self-absorption for X-rays from a particle at depth in a sample than the inner detectors. Such a difference can be harnessed to provide an imaging contrast based on particle depth and also a quantitative measure of depth for discrete identified particles.
2. The variation in model yields across the array is illustrated below, which shows the yield (counts/ppm/μC) of Pt La X-rays from a buried particle for individual detector elements plotted as a function of the radial distance of each detector from the centre of the array (beam axis). Each symbol type represents the yield from a 1 μm layer of Pt at various depths within an olivine sample matrix. Subtle absorption-related effects of particle shape versus a simple layer have been ignored in this discussion. The interesting apparent scatter in these points reflects the non-equivalent positions and orientations of detectors around the square array. Masking, distance and angle gradually reduces the efficiency of the more peripheral detector elements. Depth to the Pt layer has a marked effect on yields, with stronger attenuation seen in the more peripheral detectors.

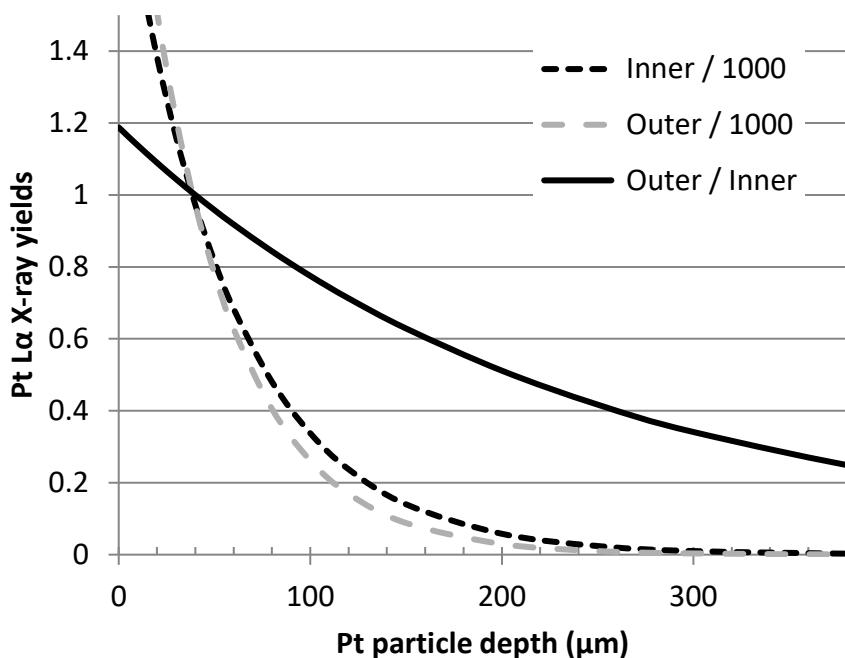


*Yield (counts/ppm/ $\mu$ C) of Pt La X-rays from a small Pt particle (modelled as a 1  $\mu$ m buried Pt layer in olivine) versus radial position of individual Maia detector elements for various depths ( $\mu$ m) to the particle.*

3. If the two-layer sample model is correct, i.e. has the correct depth to the PGM, then both inner and outer detectors will “measure” the same effective concentration of Pt in the buried or “unknown” layer. Hence, since conc = area/yield, then  $(\text{area}/\text{yield})_{\text{inner}} = (\text{area}/\text{yield})_{\text{outer}}$ . Or, we can relate  $(\text{area}_{\text{inner}}/\text{area}_{\text{outer}}) = (\text{yield}_{\text{inner}}/\text{yield}_{\text{outer}})$ . Therefore, we can relate the Ratio =  $(\text{yield}_{\text{inner}}/\text{yield}_{\text{outer}})$  to the measured ratio  $(\text{area}_{\text{inner}}/\text{area}_{\text{outer}})$ . They should be equal at the correct depth.
4. Combining yields for 51 “inner” and 199 “outer” detectors as shown in the Layout below, chosen to give a similar total yield for a surface feature, produces the inner/outer yield ratio curves below. The ratio of “outer”/“inner” yields produces a good contrast with the depth of the particle. This can be applied to estimate the depth of a small particle feature, given the assumption that the host mineral composition is uniform above it.



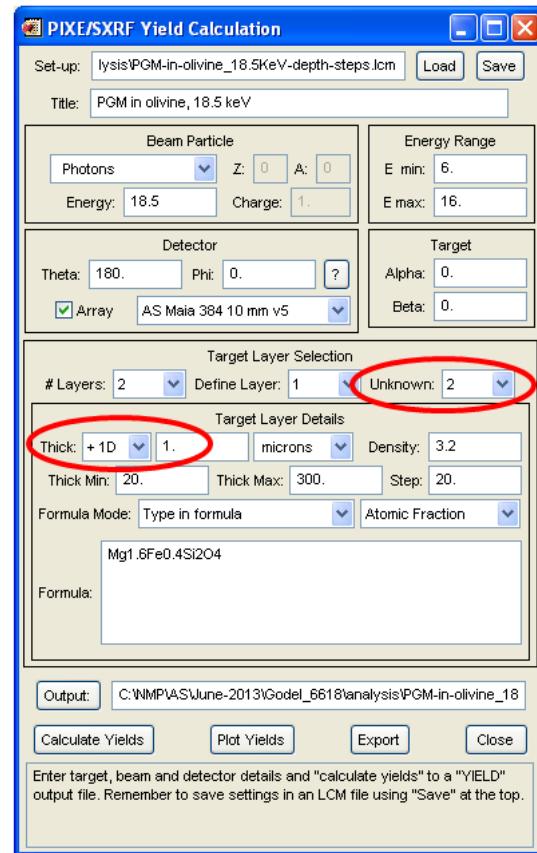
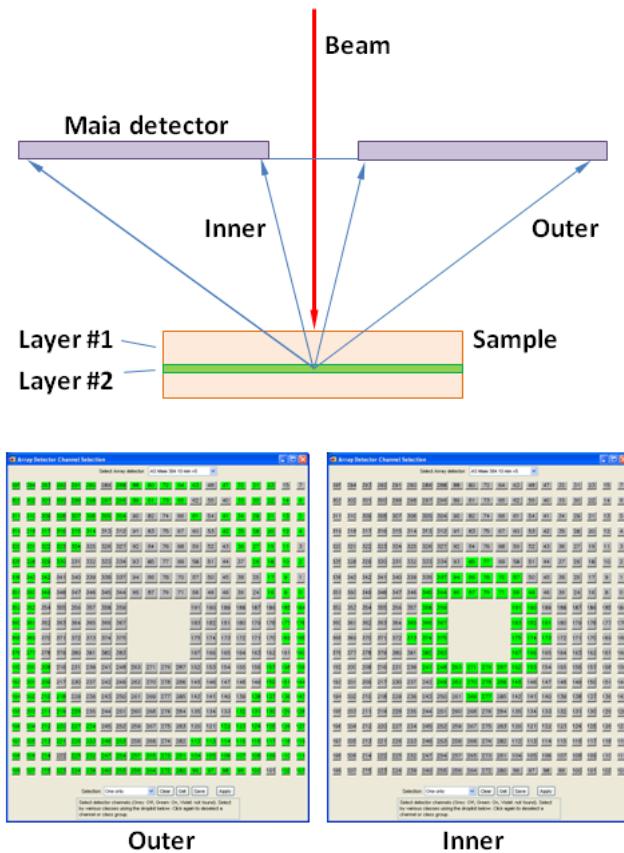
*Layout of Maia detector array (old model A) showing detector index and the masked effective size of detectors. Shaded detectors for 51 “inner” (green), 199 “outer” (yellow) and 18 disabled (violet) detector elements.*



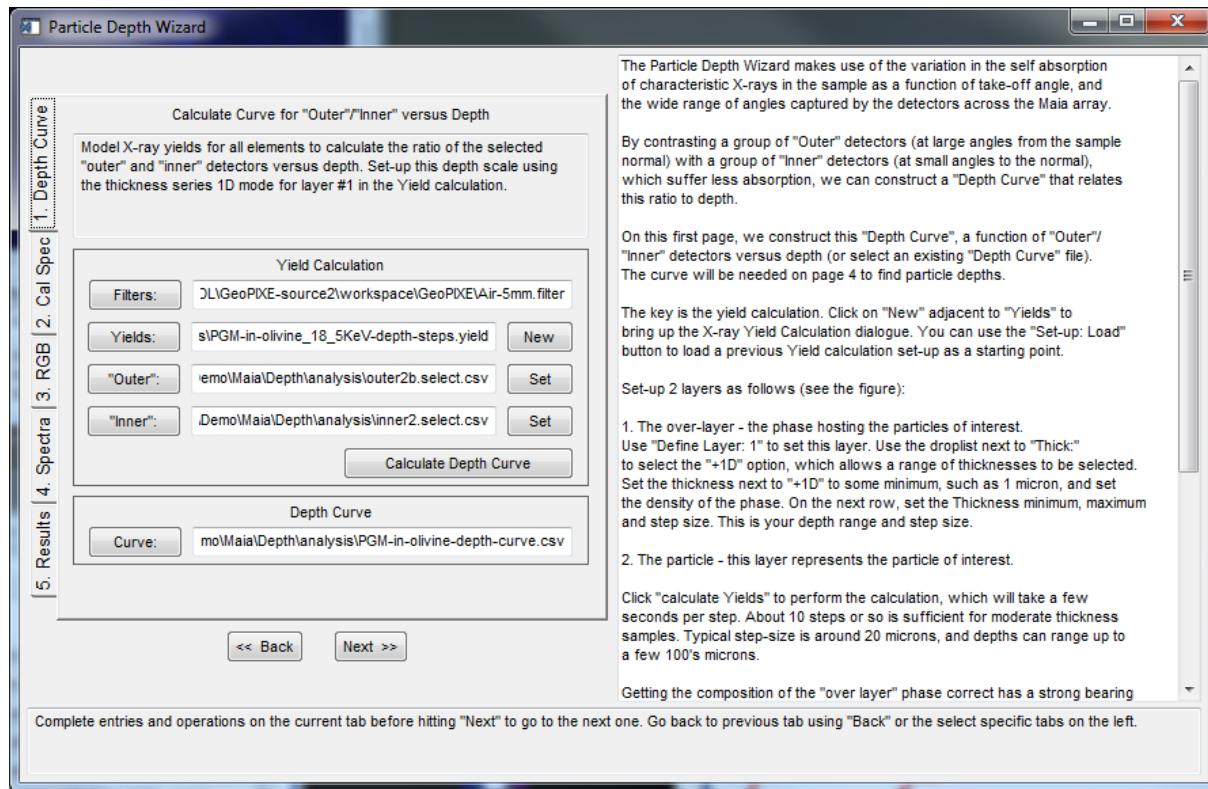
*Yields of Pt La X-rays (counts/ppm/ $\mu$ C) summed over “inner” and “outer” rings of detectors scaled by 0.001, as a function of Pt particle depth ( $\mu$ m) in olivine, and the ratio of “outer” to “inner” yields.*

### Calculate a Depth Curve

- Start the *Depth Wizard* using the *Image* window menu “Process→Wizards→Wizard\_Depth”. Read the information provided and follow the steps described in the Wizard. Also, move your mouse pointer over widgets to get more information on what each one does.



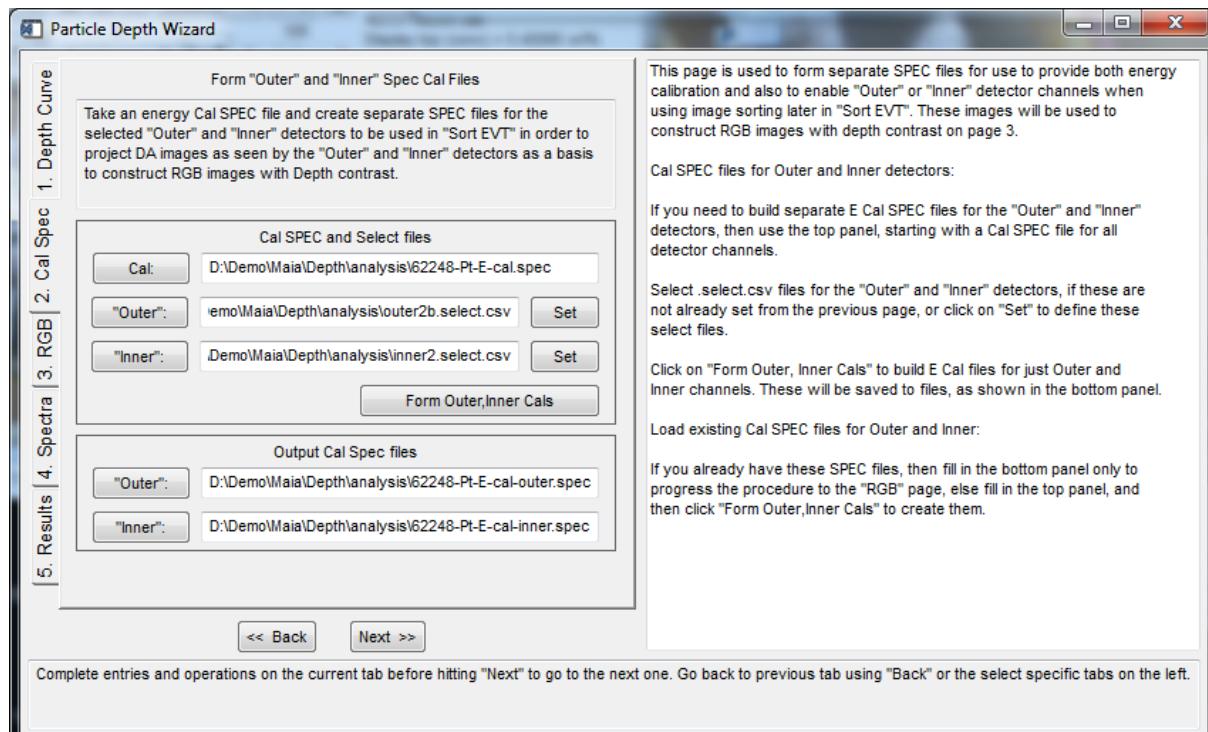
- On the first page (tab “**1. Depth Curve**”), set the Filter as relevant for the data (“Air 5 mm” in this case).
- Open the *X-ray Yield Calculation* window by clicking on “New” adjacent to “Yields:” and load the LCM file “**Demo/Maia/Depth/analysis/PGM-in-olivine\_18.5KeV.lcm**”.
- For layer #1 (the top olivine over-layer), change the “Thick:” mode to “+ 1D” to allow a range of thicknesses to be modelled, set initial Thick to 1 µm, Min to 20 µm, Max to 300 µm and Step to 20 µm, and change the output file name to something like “PGM-in-olivine\_18.5KeV-depth-steps.yield”.
- Click “Calculate Yields” to start the modelling for all layer #1 thickness steps (15 depth steps for 384 detectors, including secondary fluorescence will take some time). The output file-name will appear in the “Yields” field.
  - In future you can just load this file using the “Yields” button.
- Select the “Outer” detectors by clicking on “Set” opposite “Outer”. Select the detector layout for the old Maia 384A detector array (“AS Maia 384 10 mm v5”) from the dropdown.
- Try setting the detector pad selection mode to “Radial” and clicking on detectors in different radial position to toggle the selection on/off of detectors at this radius from the centre. Once done, click “Save” to save this selection to a “select.csv” file (e.g. “test.select.csv”).
- Back on the *Depth Wizard* panel, click “Outer” and load the file “outer2b.select.csv”, which shows our Outer detector selection. Also load the “Inner” detector selection from file “inner2.select.csv”.



13. Click on “Calculate Depth Curve” on the *Depth Wizard* panel, to form a model curve that relates the ratio of “outer/inner” to depth”. It will be prompt for a filename. This will be needed on later tabs.
  - a. In future, for this sample matrix, you may only need to load this depth curve file.

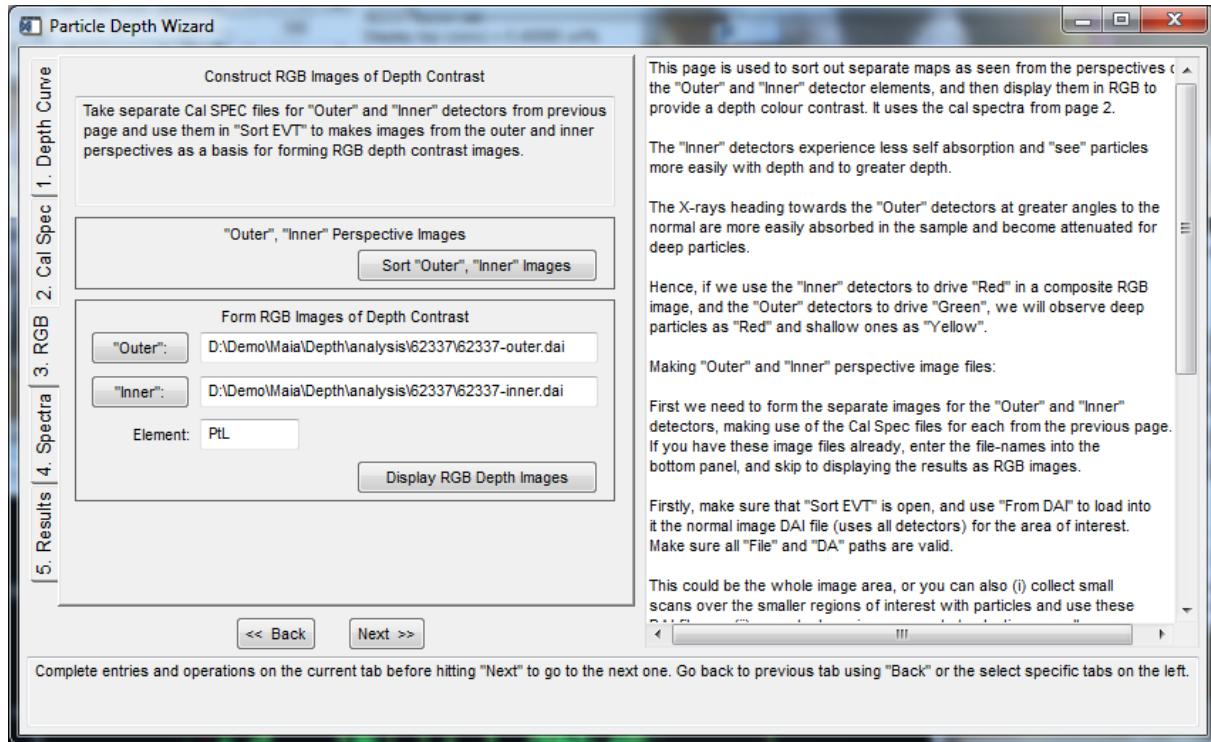
#### **Form “Inner” and “Outer” detector calibration files**

14. Advance to the next tab (“2. Cal Spec”). Here we load the per detector channel energy calibration file “**62248-Pt-E-cal.spec**” and click on “Form Outer, Inner Cals” to derive separate energy cal files for generating images for just Outer or Inner detectors (remember how the “Get” button on the DA tab of *Sort EVT* also selects the detector channels to use).
  - a. In future, just load the inner, outer cal files if you have done this before.

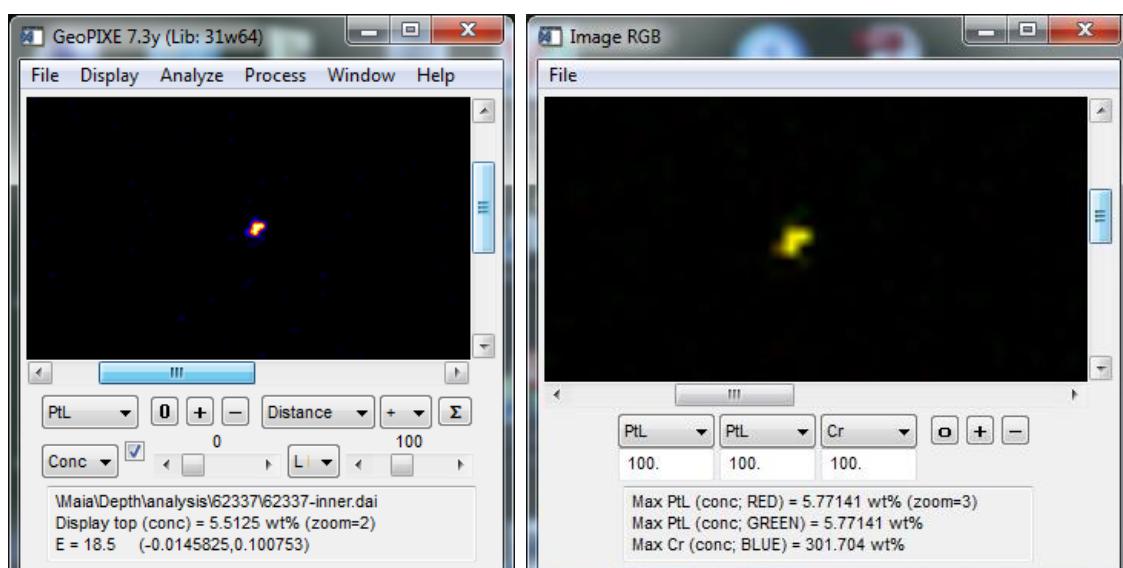


### Form “RGB” depth images

- Advance to the next tab (“3. RGB”), load the DAI file “62337.dai” into the Sort EVT window and make sure the paths to all “Files” and the DA matrix are correct.



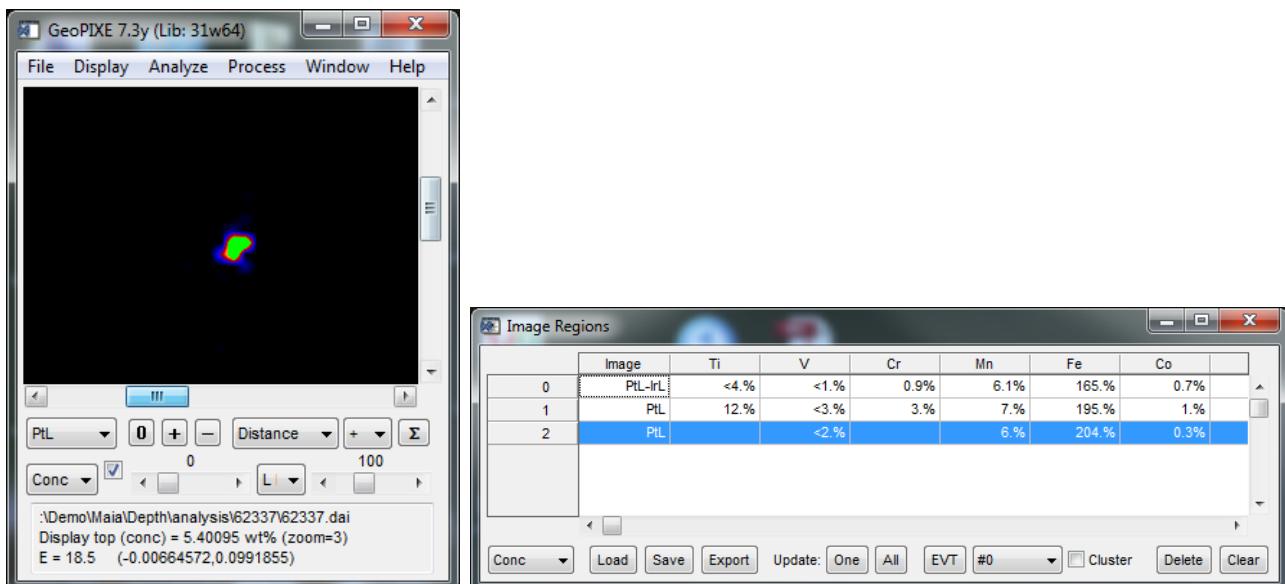
- Click on “Sort Outer, Inner images” to sort images for Inner and Outer detectors.
  - There may be warnings about windows that are needed, but not open. Open these and try again.
  - In future, you can just load the Outer and Inner images, if they have been previously sorted.
- Select the element “PtL” on tab “3. RGB”. Click on “Display RGB Depth Images” to form RGB images colour-coded for depth.
  - RED is loaded with the inner detector images, while GREEN is driven by the Outer detector images. Shallow particles will appear Yellow, while deep ones will appear Red and intermediate depth particles will appear Orange. Zoom in a couple of clicks ...



Pt image (left) and depth coded Pt image (right), which compares the Outer and Inner detector Pt images. Pt is yellow, which indicates quite a shallow inclusion depth.

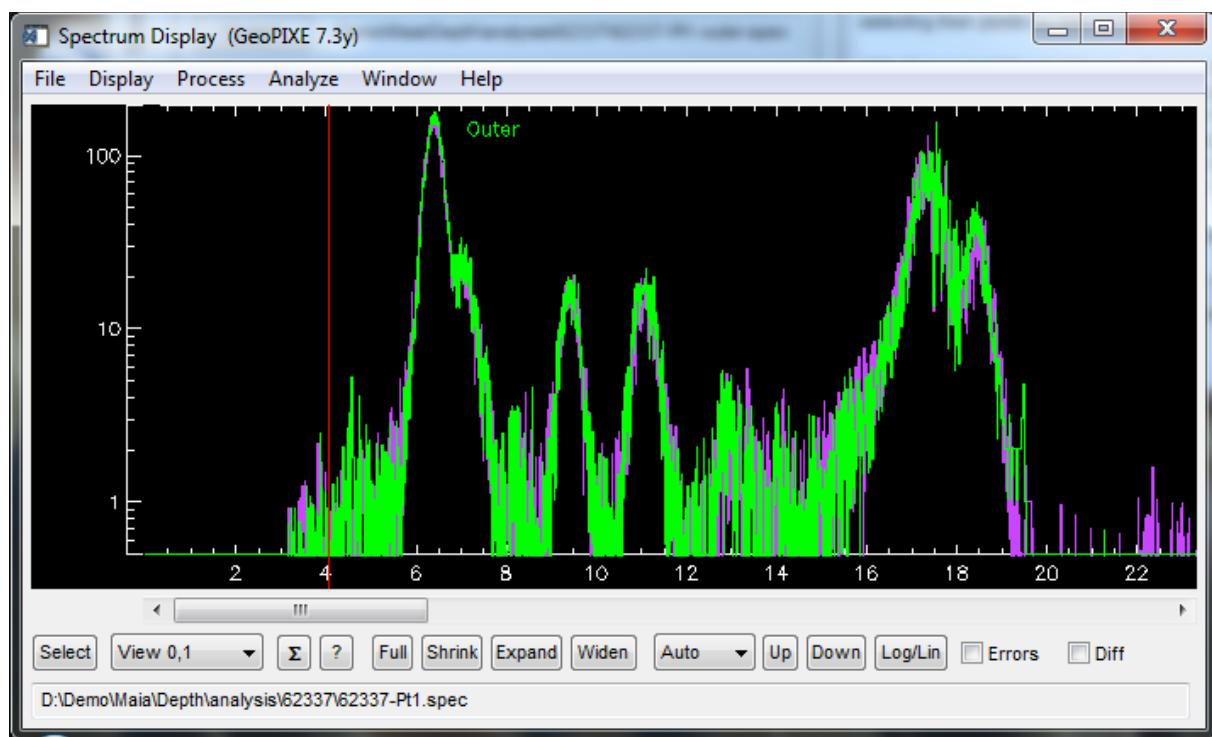
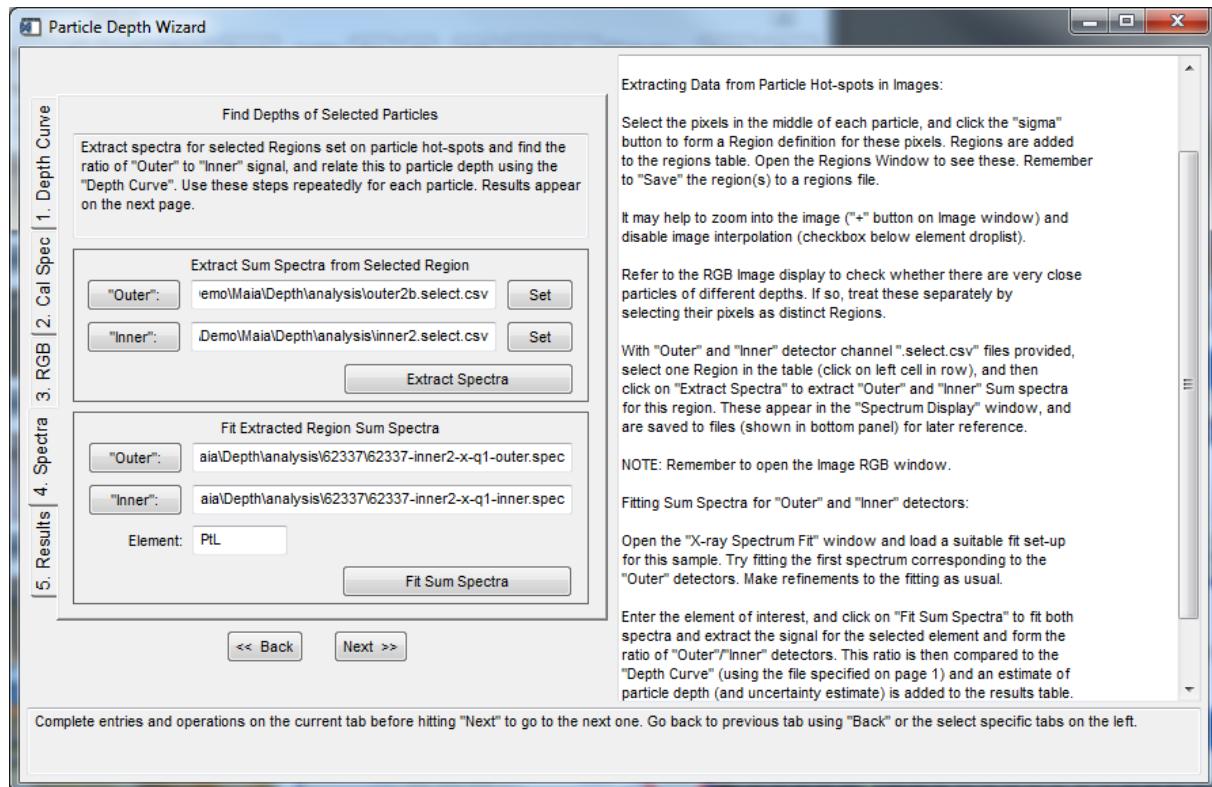
### **Fit Region spectra for particles to determine depth**

18. Advance to the next tab (“**4. Spectra**”). Load the original image DAI file (“Demo/Maia/Depth/62337.dai”, for all detector channels) into *Sort EVT* that shows your buried particle and open the *Image Regions* window.
  - a. NOTE: You must load the DAI file for **ALL detectors**, so we can extract inner/outer subset spectra.
19. As noted in the help text for this tab, select pixels on the particle and make each region.
  - a. Use simple shapes (e.g. Box or Single Pixel) and click on “ $\Sigma$ ”.
  - b. Use element correlations in the *Associations* window to click and drag a shape to select pixels. Then use the menu “Analyze/Include all pixels within the spline”.
20. Repeat this for other particles, and then “Save” the regions to a file. See example file “62337-Pt1.region”, which has 3 regions defined.



Zoom in (+3 zoom) on a PGM particle, showing just 3 pixels selected on the grain using Ir-Pt in Association window

21. Open the *X-ray Spectrum Fit* window and load a suitable PCM set-up file for fitting your particles (“PGM-in-olivine-18\_5keV.pcm”).
22. Click on the row title for a selected particle region and then “Extract Spectra”, which will apply your Outer and Inner detector channel selections and extract a sum spectrum for the selected region.
  - a. Accept the initial base file-name for individual detector spectra.
  - b. The resulting file-names are entered into the spectra fields.
23. Enter the element of interest (“PtL” in this example) and click on "Fit Sum Spectra" on the Wizard to fit both spectra and extract the signal for the selected element and form the ratio of "Outer"/"Inner" detectors.



Sum spectra for Outer (green) and Inner (violet) detector channels

24. The ratio of "Outer"/"Inner" detectors is then compared to the "Depth Curve" (using the file specified on page/tab 1) and an estimate of particle depth (and uncertainty estimate) is added to the results table.
25. Repeat steps 22-24 for regions #1,2 in the example file "62337-Pt1.region", which select just a ~single pixel each.
26. The results (e.g. 18 µm for the 3 pixels selected in region #0) will appear on the final tab. Note how different the two single pixel results are for regions 1,2; evidently we have two particles at different depths here (~0, 35 µm).
27. Use the Export button to export the results to a CSV file.

**Particle Depth Wizard 7.6s (GeoPIXE 8.6s)**

**Results for Depths of Selected Particles**

Particle depth results: Element peak areas and errors determined from "Outer" and "Inner" sum spectra on the previous page.

O Err	I Area	I Err	Ratio	eRatio	Depth	eDepth
22.6	630.0	22.7	1.0606	0.0495	18.00	11.037
16.9	269.0	16.3	1.172	0.0809	0.	15.54
15.0	234.0	15.4	0.981	0.0929	34.68	22.30

This page shows the results table, with depths determined from the page 4.

The table shows the total peak area for all detectors in "Outer" detectors as "O Area" and its simple statistical uncertainty as "O error". Similarly, "Inner" area is "I area". The ratio of "Outer/Inner" and its uncertainty are shown as "Ratio" and "eRatio".

Depth is determined from a spline interpolation on the Ratio versus Depth curve from the "Depth Curve" file on page 1.

For now, the uncertainty in depth only includes the effect of statistical error in the areas, and does not include the effect of errors in the composition of the first layer in the yield model, which may be much larger. Try making small changes to composition in the yield calculation and re-do the depth determination to see the effects of these changes on depth.

Use the "Export Results" button to save the results to a CSV file.

<> Back      Figure      Next >>

Complete entries and operations on the current tab before hitting "Next" to go to the next one. Go back to previous tab using "Back" or the select specific tabs on the left.

## H. XANES Imaging

### Example H1: XANES Stacks - Correction for Image Shifts using a Hot-Spot

Construct XANES image stacks from Maia images collected at a sequence of energies across an absorption edge

- File organization notes for XANES imaging.
- Image generation, using Batch mode.
- Assembling the XANES Image Stack.
- Correction for image shifts.
- Contrast regions using *Associations* between energies of XANES spectral features.
- Apply regions from element images (e.g. using *Associations* between elements) to XANES stacks

#### File organization for XANES imaging

1. For the Batch processing mode to work easily and find all files associated with your XANES image stack, it is best to re-arrange your directories to create a directory for the raw blog files in each stack. In the “blog” directory, create new dirs one for each XANES stack. Similarly, create output stack dirs in the “analysis” directory.

Organize the “blog” (and “analysis”) directory with this type of structure (typically, this structure is build at XFM by setting the “Group” before each XANES stack run):

1. blog/
  - a. xanes-18791-18878/
    - i. 18791/
      1. all blog data files for run 18791
    - ii. 18792/
      1. all blog data files for run 18792
    - iii. 18793/
      1. all blog data files for run 18793
    - .
    - .
    - iv. 18878/
      1. all blog data files for run 18878

2. analysis/
  - a. xanes-18791-18878/
    - i. all output .DAI and .YLUT files for this stack.

#### **Batch mode imaging method**

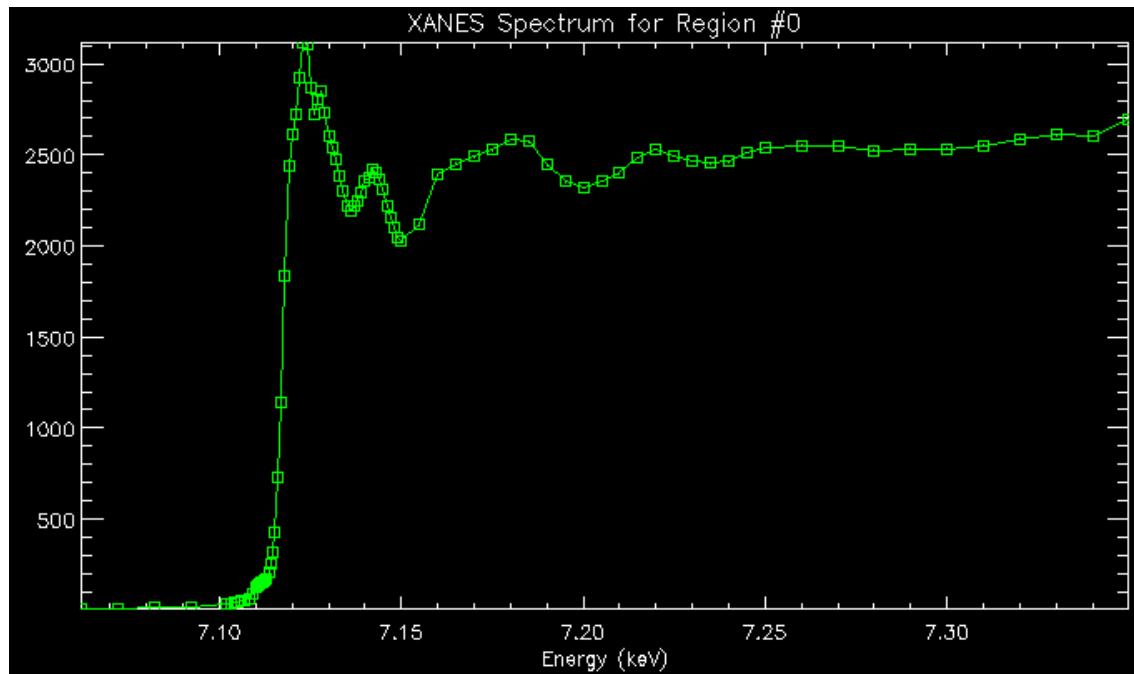
2. The raw data is not available for this example (160 Gb). However, the method would proceed as follows ...
  - a. Load the first energy image into *Sort EVT* using the “From DAI” button.
    - i. Process this first image using a “XANES series DA matrix” file.
      1. This is a DA matrix where you specify a file of XANES energies during the “Generate DA matrix” step after the spectrum fit.
      2. The XANES energies file can be imported from the raw blog data using the new “File→Import→Energies from list-mode” menu in *Spectrum Display*.
    - ii. Make sure all the paths and files are correct as for any image data-set (e.g. select “First File” from “blog/xanes-18791-18878/18791/18791.0”).
    - iii. Point output to “analysis/xanes-18791-18878”).
    - iv. Click on “Start” in the *Sort EVT* window to process this frame.
  - b. Open the *Batch Sort* window, using “batch” button on *Sort EVT*.
  - c. Click on “Dir” button and navigate to your XANES directory (“blog/xanes-18791-18878”) constructed above. Click “OK” to scan this directory for all blog files (if we had the raw data).
  - d. Check that the output paths are correct (the output path is inherited from the DAI file you created above in *Sort EVT*).
  - e. Set options (e.g. “Skip sort step if DAI already exists”).
  - f. If each image frame has more than ~10 blog files, you might want to enable “Cluster” on the *Sort EVT* window.
  - g. “Start” (if we had the raw data) to start the batch.
  3. An example batch set-up file can be loaded from “**Demo/Maia/XANES/analysis/xanes-18791-18878.sbatch**”.

#### **Assembling the XANES Image Stack**

4. Open the *3D Image Stack* window using the menu “Display→3D Stack Image”.
5. Import the XANES image frames from directory “**Demo/Maia/XANES/xanes-18791-18878**” using the *3D Image Stack* window menu “File→Import→XANES Image Stack (DAI files)”. Drag select all DAI files or click on the first, scroll down and shift-left-click on the last.
6. Select the element whose edge has been scanned for this XANES series (Fe in this case).
  - a. The result is a series of images, one for each energy. You can select energy using the dropdown on the left, and scroll through using the up/down arrow keys.
7. These images show some shearing (backlash) between even and odd (stage return) rows.
  - a. To correct this, in this case, use the menu “**Process→Shift→Odd Rows +0.5 (toggle w/ odd Y rows)**”.
8. Save the stack in file “18791-18878-Fe-x.xan”.

#### **Examine some XANES spectra for regions**

9. Open the “XANES Stack Regions” window and drag out a couple of regions on different areas of the image.
10. Click “Σ” to add these to the regions table.
  - a. See the file “18791-18878-Fe-shifted-q1.region”, for example.
11. Click on each row and observe the XANES spectrum in the pop-up plot window.



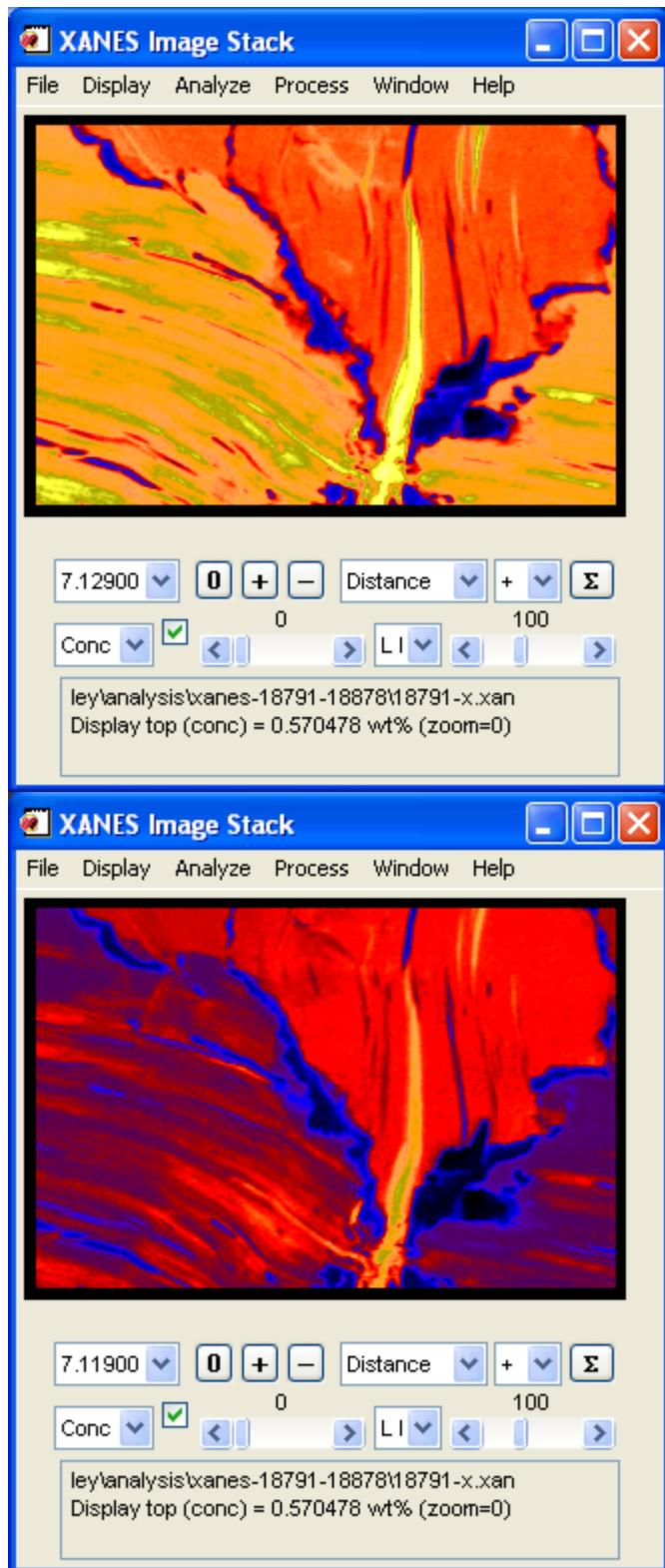
#### ***Correction for image shifts (using a hot-spot)***

(See *XANES Stack example 2 for an alternative approach using cross-correlation*)

12. Import the XANES image frames again, but select the element “Ti” to look for a hot-spot present in all images that we might use to track image shifts between image frames.
13. Set a small shape around a suitable hot-spot that is strong and present on all images, and click on “Σ” on the *3D Image Stack* window.
  - a. The region data now includes a centroid for each energy plane.
  - b. One example is given in the Region file “18791-xanes-Ti-centroids.region”.
  - c. Open the *XANES Stack Regions* window and load this regions file.
  - d. Click “Update All”, or clear the list and click on “Σ” on the *3D Image Stack* window, as you would for a new shape that you have just set.
14. Save the region file (with its centroids) to “18791-xanes-Ti-centroids.region”.
15. Open the Fe XANES stack again from file “18791-18878-Fe-x.xan”.
16. Load the region file “18791-xanes-Ti-centroids.region” again.
17. Select region #0 row (ignore the plot window XANES spectrum for Ti?) and click on “Match centroids”
  - a. This will move each image to match the position of the (Ti) centroids in each frame.
  - b. Under Linux, you will see the shifts reported in the shell window.
18. A second iteration of this method can be applied from file “18791-xanes-Ti-centroids-2nd-iter.region”, which uses a different hot-spot and a smaller region to tighten the shift correction.
19. Save the final Fe XANES stack to file “18791-18878-Fe-shift-Ti-iter2.xan”.

#### ***Contrast regions using Associations between energies of XANES spectral features***

20. Load the XANES image stack file “**demo/Maia/XANES/xanes-18791-18878/18791-18878-Fe-shift-Ti-iter2.xan**”.
  - a. Use the element/energy dropdown and the keyboard up/down arrow keys to scan through the energies.
  - b. Note the contrast between energies 7.129 and 7.119 keV near the Fe absorption edge.



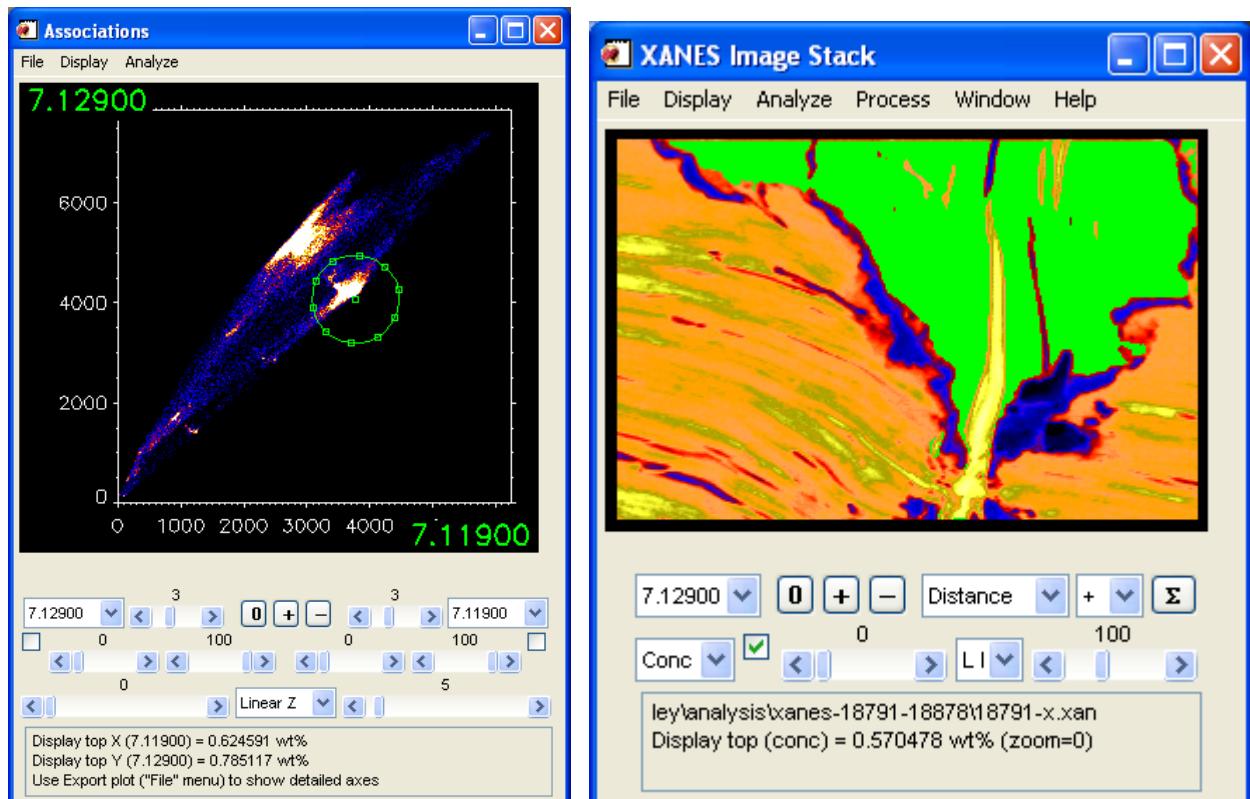
*Contrast between images of biotite acquired at 7.129 (left) and 7.119 keV (right), probably dominated by the orientation of the biotite planes to the synchrotron beam polarization in the horizontal plane. (Data courtesy of James Cleverley, CSIRO.)*

21. Open the Association window from the Window menu on the 3D Image Stack window, and also the XANES Stack Regions window.
22. Change to linear axes, with 7.129 on the Y axis and 7.119 on X.
  - a. Adjust the Z 2D histogram intensity scale (bottom right slider) to see more.
  - b. You may need to clear all region selections using the “Display→Clear All Shapes” menu on the Stack window

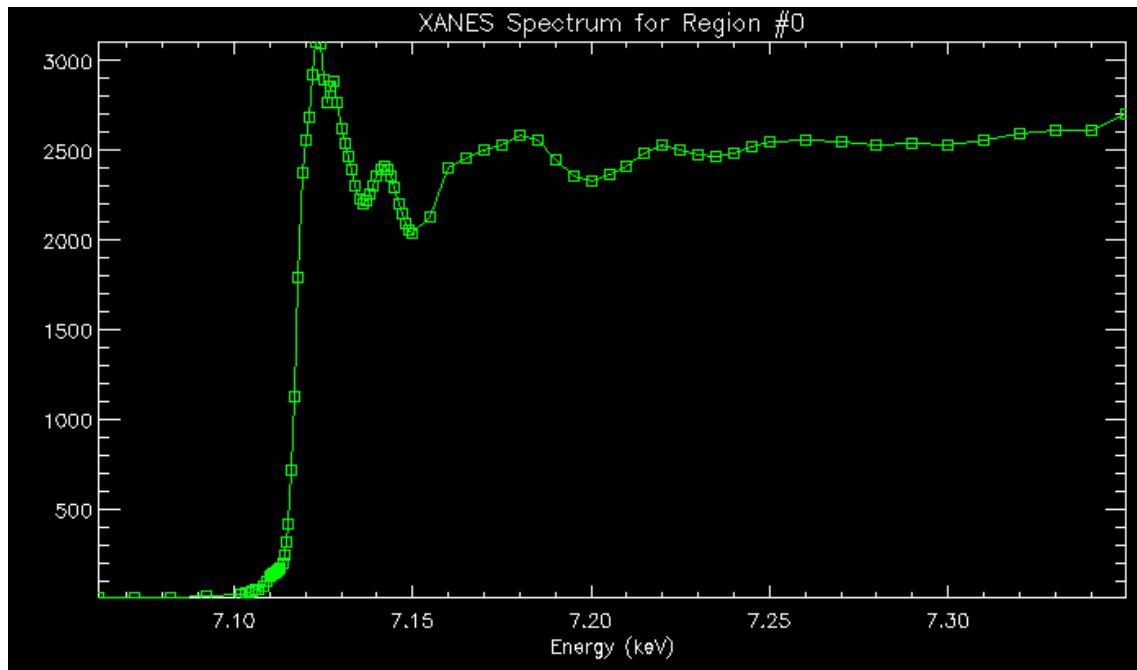
23. Drag out a spline curve to surround one of the branches seen on the Associations window. Select the menu “Analyze→within Spline” to select all pixels linked to this branch.
- The average Fe values across these pixels at each energy are shown in the regions table.
  - Click on a row label to see the XANES spectrum displayed in the plot window.



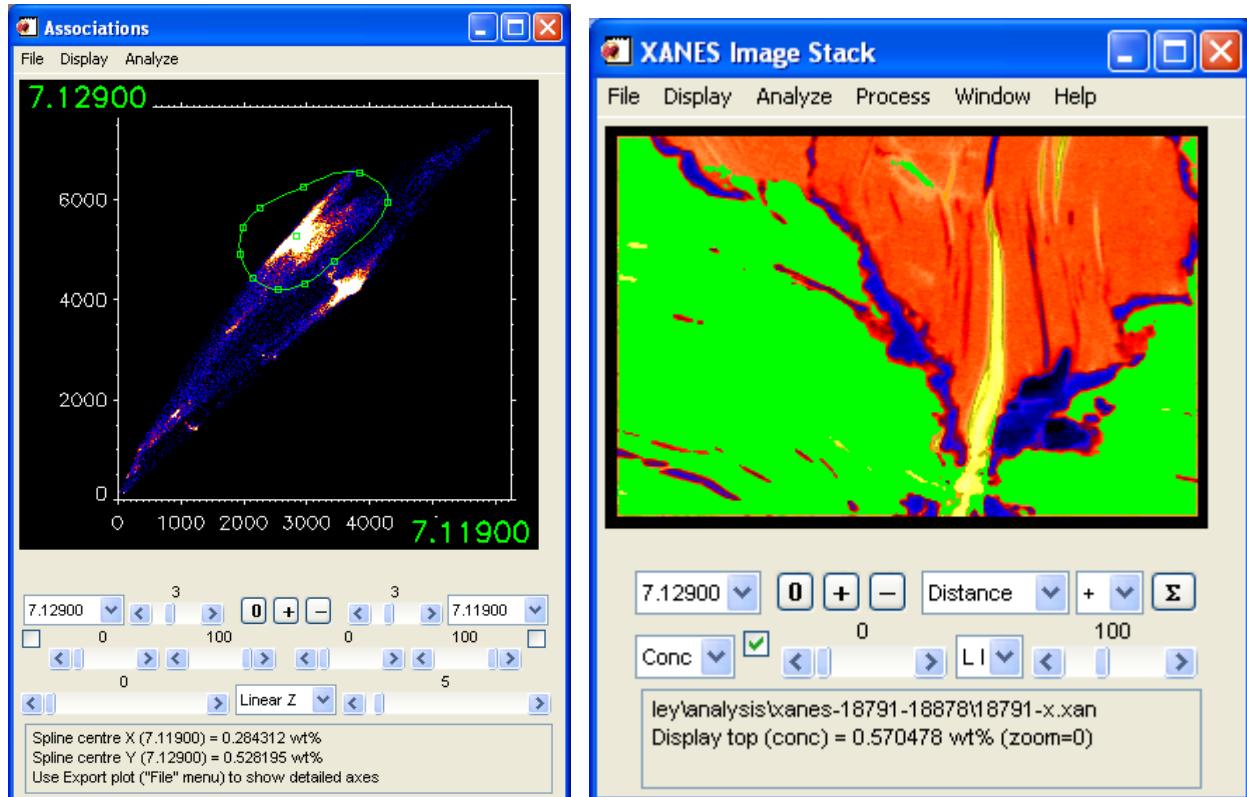
XANES Stack Region window showing the two selections in 7.129 versus 7.119 Association space. Each is the average XANES spectrum in these selected pixels.



Association plot for 7.129 versus 7.119 keV images and spline selection #0 (left) and the pixels highlighted by this selection (right)

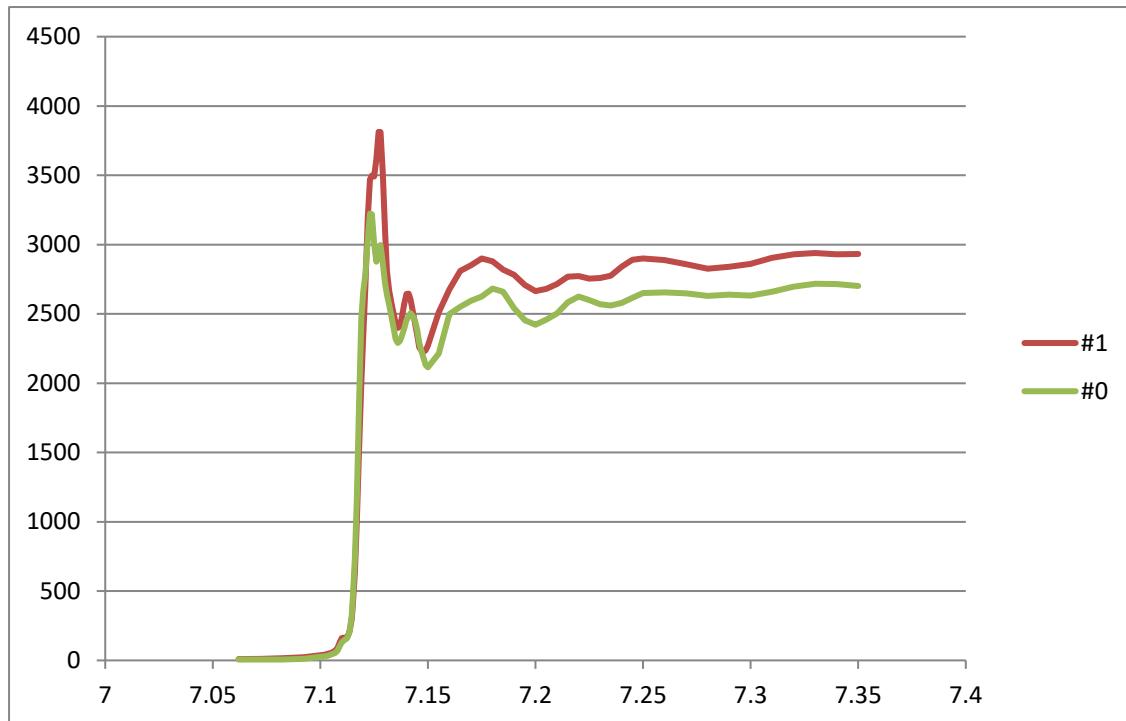


XANES spectrum for region row #0



Association plot for 7.129 versus 7.119 keV images and spline selection #1 (left) and the pixels highlighted by this selection (right)

24. Save these regions to file “18791-polarization-q1.region”.
25. Click on the “Export” button on the XANES Stack Regions window, which allows you to export this data to a CSV file (“18791-polarization-q1.csv”) for display in Excel or for analysis in XAS software packages.
  - a. You might unselect the “Flux” column in the “Select Elements” pop-up.
26. Double click on the CSV file in a file explorer window to open Excel. Select the energy and Fe signal rows (or transpose these to columns) and plot the results:



XANES spectra for pixels selected in Regions #0 and #1.

**Contrast regions using Associations between normal element images and apply these to the XANES stack**

27. Load the first element image in the stack at the top energy (18791.dai) into a normal *Image* window and define some regions based on *Associations* between two elements (e.g. Fe, Mn).

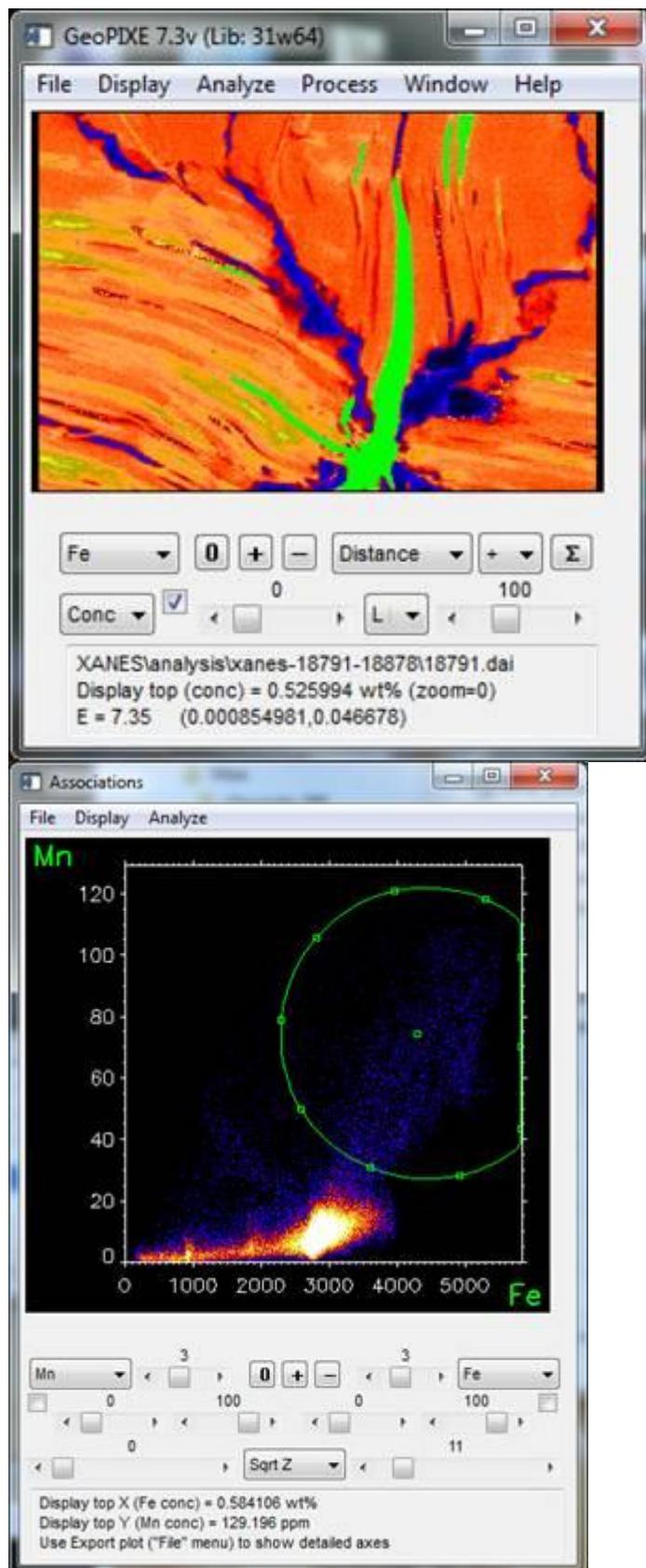


Image	Ti	Cr	Mn	Fe	Ni	Cu	Zn
0	Fe-Mn		6.6	61.9	0.411%		
1	Fe-Mn		4.68	8.06	0.279%		
2	Fe-Mn	2.0	3.4	3.7	0.201%		
3	Fe-Mn	13.6	2.16	0.6	877.		
4	Fe-Mn		<0.8	35.6	0.162%		

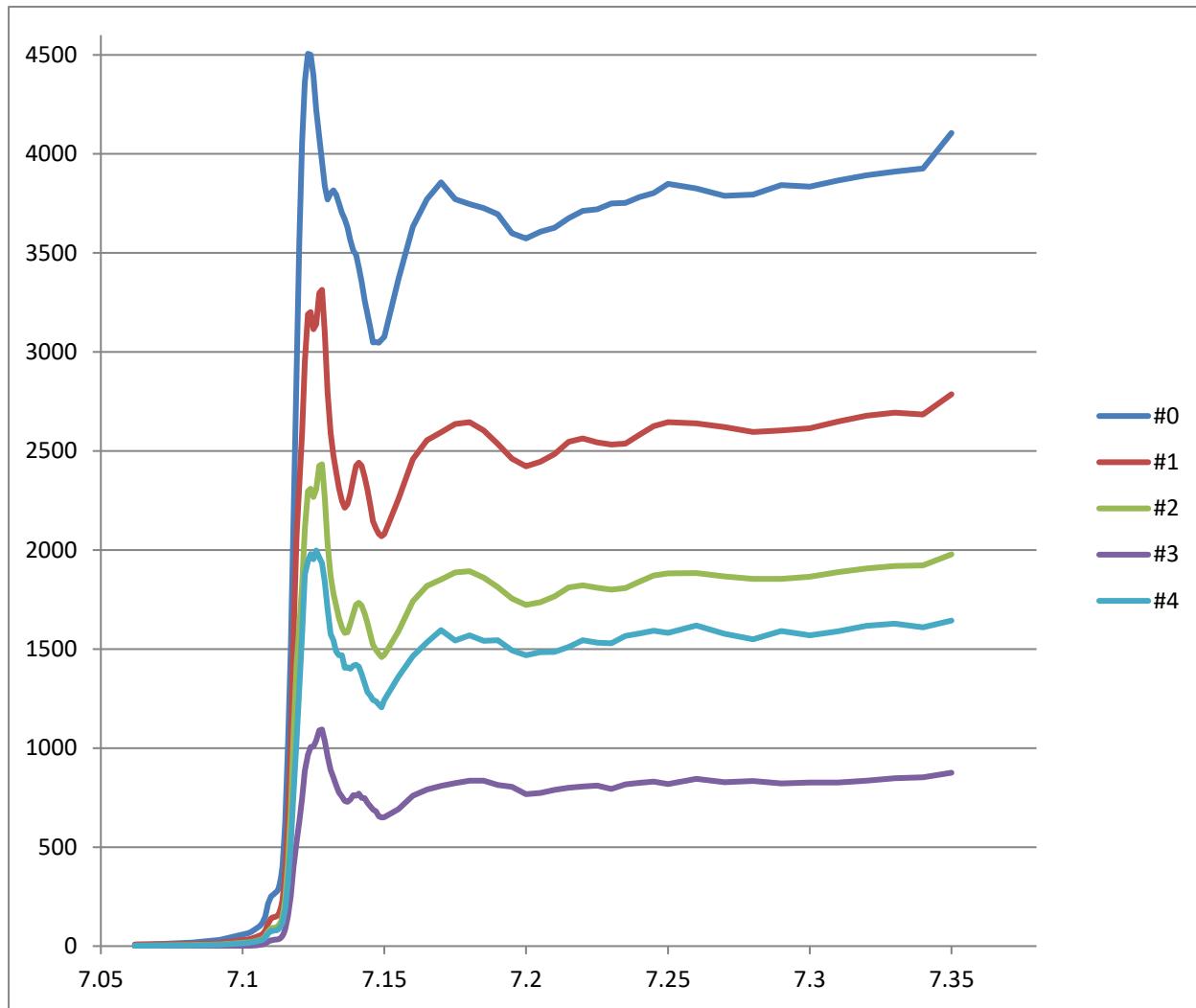
Buttons at the bottom: Conc, Load, Save, Export, Update: One, All, EVT, #0, Cluster, Delete, Clear.

28. Save the regions to a Regions file (e.g. 18791-q1.dai).
29. Load the XANES image stack file “demo/Maia/XANES/xanes-18791-18878/18791-18878-Fe-shift-Ti-iter2.xan” into the 3D Image Stack window.
30. Open the Regions window from the 3D Image Stack window and load the Regions file from step 1.
  - a. It will still show the element concentrations in the table initially, as before.
31. Now click on “Update All” to ‘apply the regions’ to the stack data.
  - a. Now the table shows the XANES spectra for these regions, the signal as a function of beam energy step.
  - b. Click on a row to view the XANES spectrum for this region.

Image	7.35	7.34	7.33	7.32	7.31	7.3	7.2
0	Fe-Mn	0.411%	0.393%	0.391%	0.389%	0.387%	0.384%
1	Fe-Mn	0.279%	0.269%	0.269%	0.268%	0.265%	0.262%
2	Fe-Mn	0.198%	0.192%	0.192%	0.191%	0.189%	0.186%
3	Fe-Mn	876.	853.	847.	836.	826.	826.
4	Fe-Mn	0.164%	0.161%	0.163%	0.162%	0.159%	0.157%

Buttons at the bottom: Conc, Load, Save, Export, Update: One, All, Match Centroids, Delete, Clear.

32. Use Export to export and display the XANES spectra for these element Association regions.



## Example H2: XANES Stacks – Correction for Shear and Drift using Cross-Correlation

Construct XANES image stacks from Maia images collected at a sequence of energies across an absorption edge

- More notes on file organization notes for XANES imaging.
- Determination of odd/even row shear.
- Determination of image position shifts between frames.
- Correction for odd/even row shear.
- Correction for image shifts.

### File organization for XANES imaging (more useful refinement)

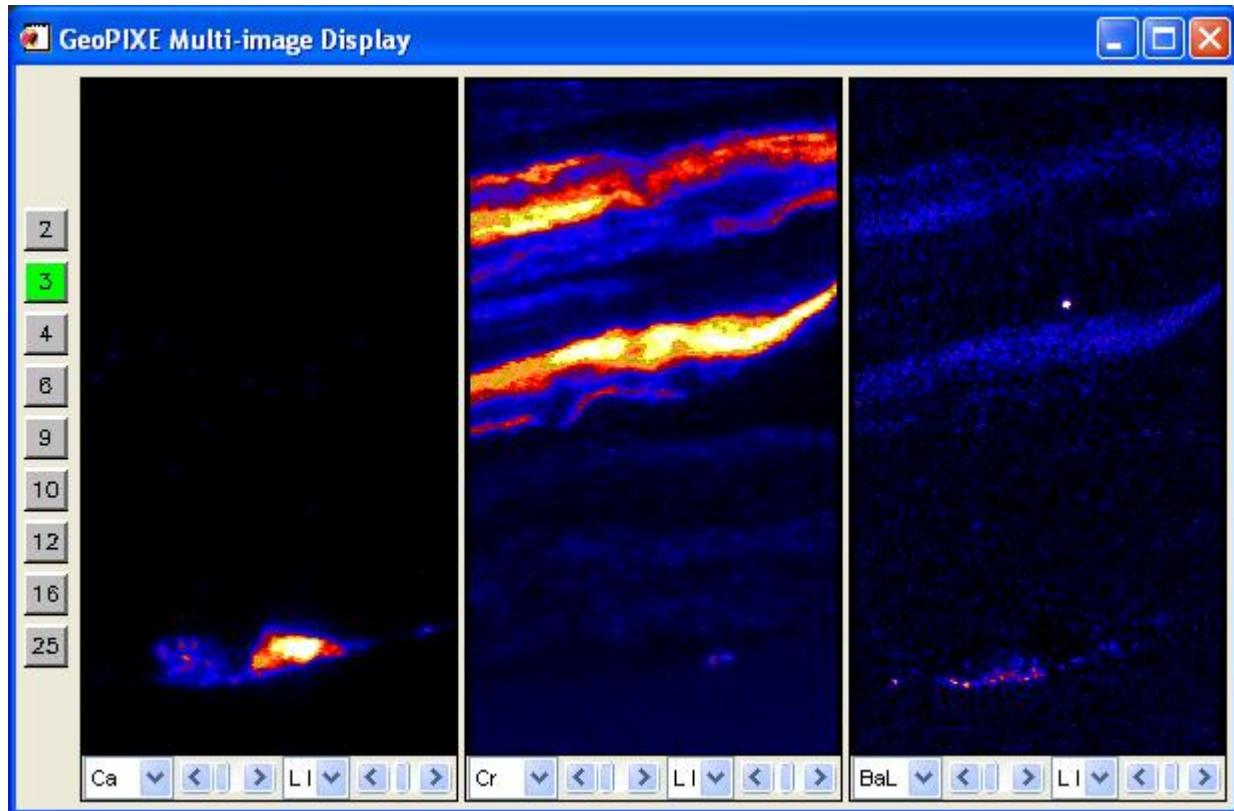
1. For the Batch processing mode to work easily and find all files associated with your XANES image stack, it is best to re-arrange your directories to create a directory for the raw blog files in each stack. In the “blog” directory, create new dirs one for each XANES stack. This can be done now using the “Group” specification in Maia and the blog server. Similarly, create output stack dirs in the “analysis” directory.

A further refinement of the file organization discussed above is to put all the output DAI files (and their YLUT files) in a sub-directory “images” or in a separate directory for each frame. This makes opening the file requester faster for non-DAI files; the “images” directory is only needed when you Import the stack:

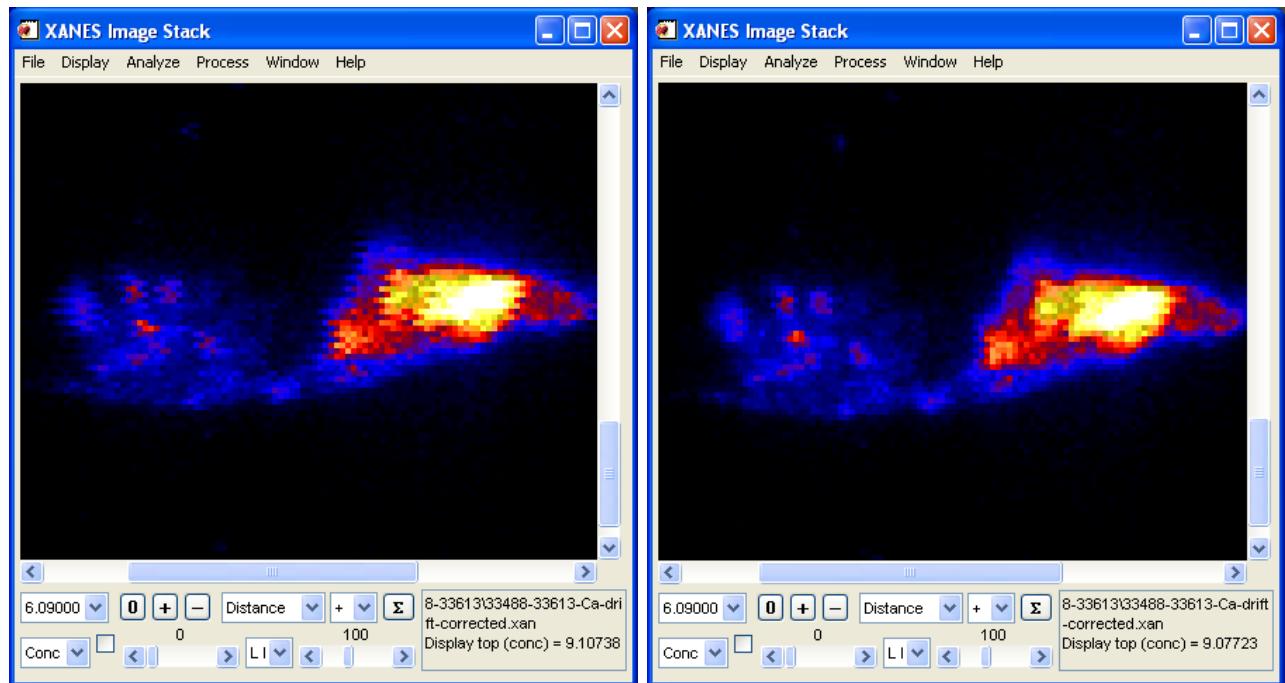
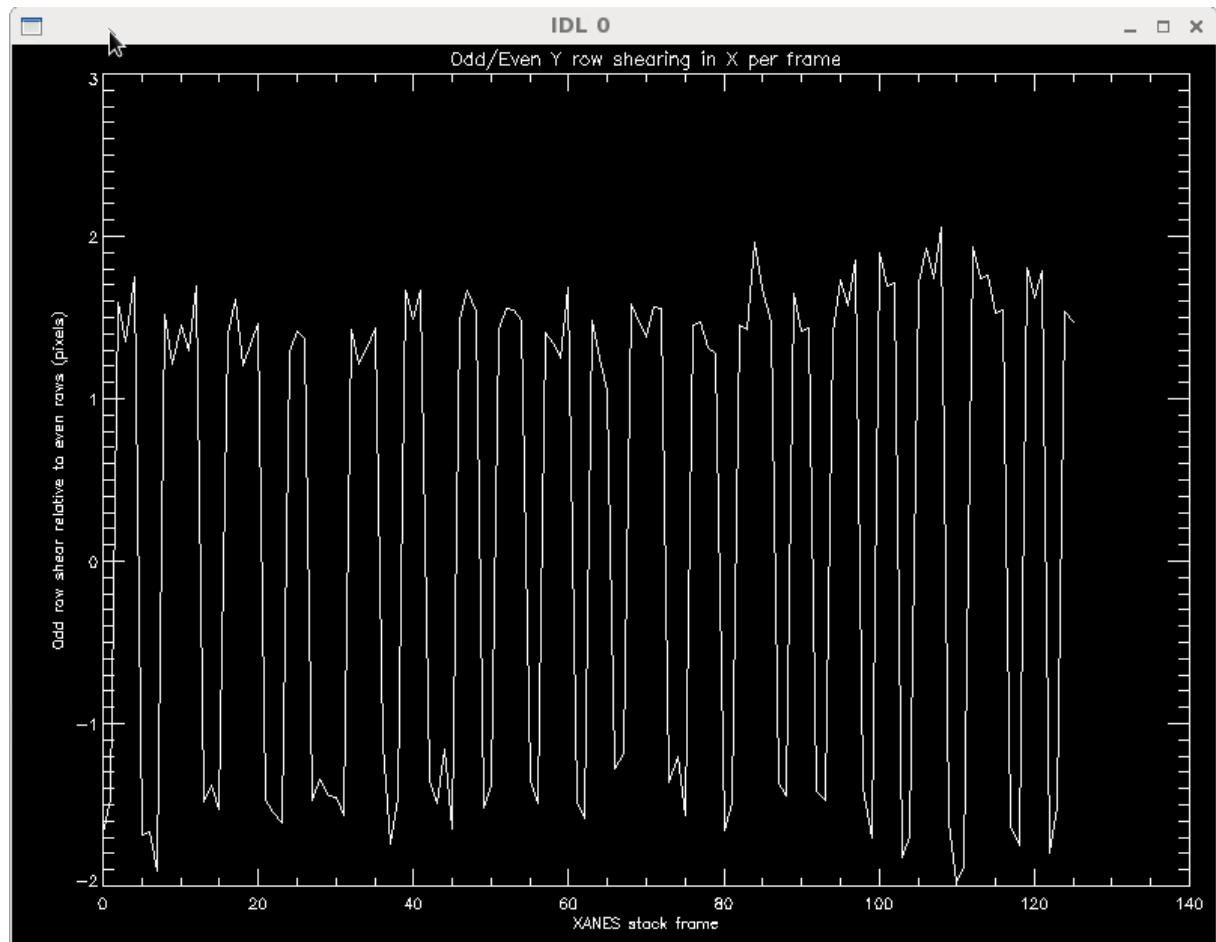
1. analysis/
  - a. xanes-33488-33613/
    - i. files related to this stack
    - ii. images/
      1. all output .DAI and .YLUT files for this stack.

### **Quantifying image shear and position jitter**

2. Open the image file “Demo/Maia/XANES2/analysis/xanes-33488-33613/images/33488.dai” (the first [highest energy, 6.090 keV], frame in a Cr XANES stack series) in the main *GeoPIXE Image* window. Note the Ca, Cr and Ba maps here, or in the *Multi Image* window:
  - a. Ca is concentrated in a large feature, isolated from the edges of the image, but shows evidence for shearing between odd and even rows (zoom in to see this; perhaps switch off the interpolation check-box to see the actually pixel data).
  - b. Cr of interest has lots of spatial structure and some hot-spots.
  - c. Ba occurs in a few hot-spots, which might be useful for shift correction (like Ti before).



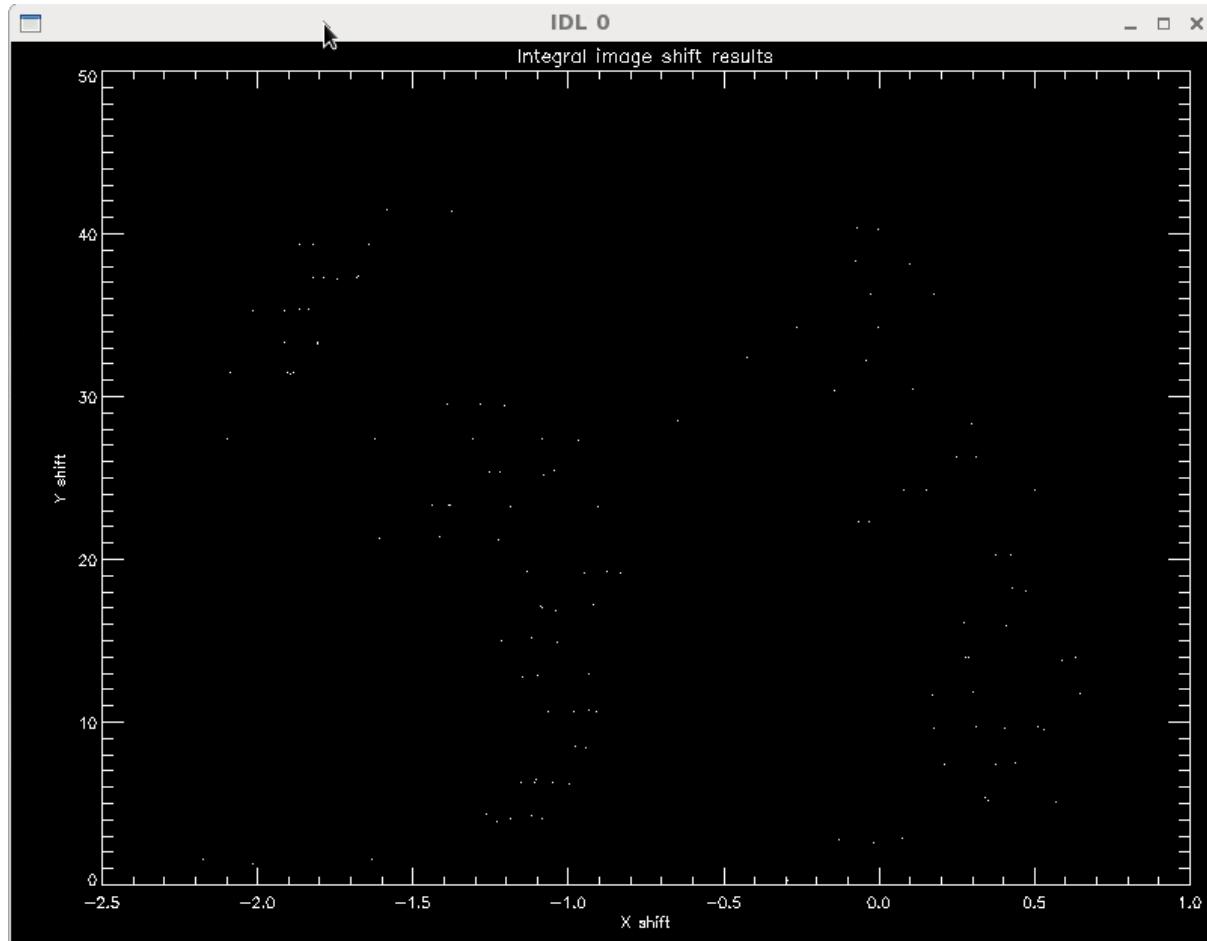
3. Open the *XANES Image* window, and import the XANES image frames for XANES stack 33488-33613 from directory “Demo/Maia/XANES2/analysis/xanes-33488-33613/images”, but select the element “Ca”.
  - a. This time we'll use the extended feature of high-Ca to sample both of the following:
    - i. Odd/even row shear: the feature spans many Y rows, which makes it sensitive to shear error between rows.
    - ii. Position shifts between frames: the well bounded, extended Ca feature is perfect to track and correct any movement between stack frames.
    - iii. Save the stack as “Demo/Maia/XANES2/analysis/xanes-33488-33613/33488-33613-Ca.xan”.
4. Zoom in on the Ca feature (click “+” twice, drag window larger and scroll) to see these artefacts:
  - a. Scroll through the stack planes using the left drop-list and the arrow keys.
  - b. Note the shearing between odd/even Y rows.
  - c. Note the random position error jumps.
5. Run the “Shear Correct Image” plugin (“process→User Plugins→Shear Correct Image plugin” menu on the XANES Image window) and select the default pop-up option “Use odd/even row cross-correlation”.
  - a. This uses Martin de Jonge’s idea to compare odd row X position with the average of neighbouring even rows.
  - b. It uses the cross-correlation image *align* routine of Chris Jacobsen (Stony Brook; see “<http://xray1.physics.sunysb.edu/data/software.php>”).
  - c. Using the default option above, it corrects the shear and outputs a file of shear values in pixels per frame to “33488-33613-Ca-shear.csv” and a plot of shear per frame.
    - iv. These can be used to correct other element stacks in this data-set.
    - v. Zoom and scroll to see the shear corrected, but the position jumps remain.



Ca stack frame 6.090 keV before (left) and after (right) shear correction

6. Run the “Align Image” plugin (“process→User Plugins→Align Image plugin” menu on the XANES Image window) and select the default pop-up option “Use inter-frame image cross-correlation”.
  - a. This uses the cross-correlation image *align* routine of Chris Jacobsen (Stony Brook; see “<http://xray1.physics.sunysb.edu/data/software.php>”) to measure the shift between subsequent frames and to align them.

- b. It also outputs a file containing the cumulative X,Y position corrections for each frame to a file “33488-33613-Ca-shift.csv”.
  - vi. These can be used to correct other element stacks in this data-set.
- c. Save the corrected stack to file “33488-33613-Ca -shear-shift-corrected.xan”.
  - vii. Scroll through the series to see how well it is corrected.

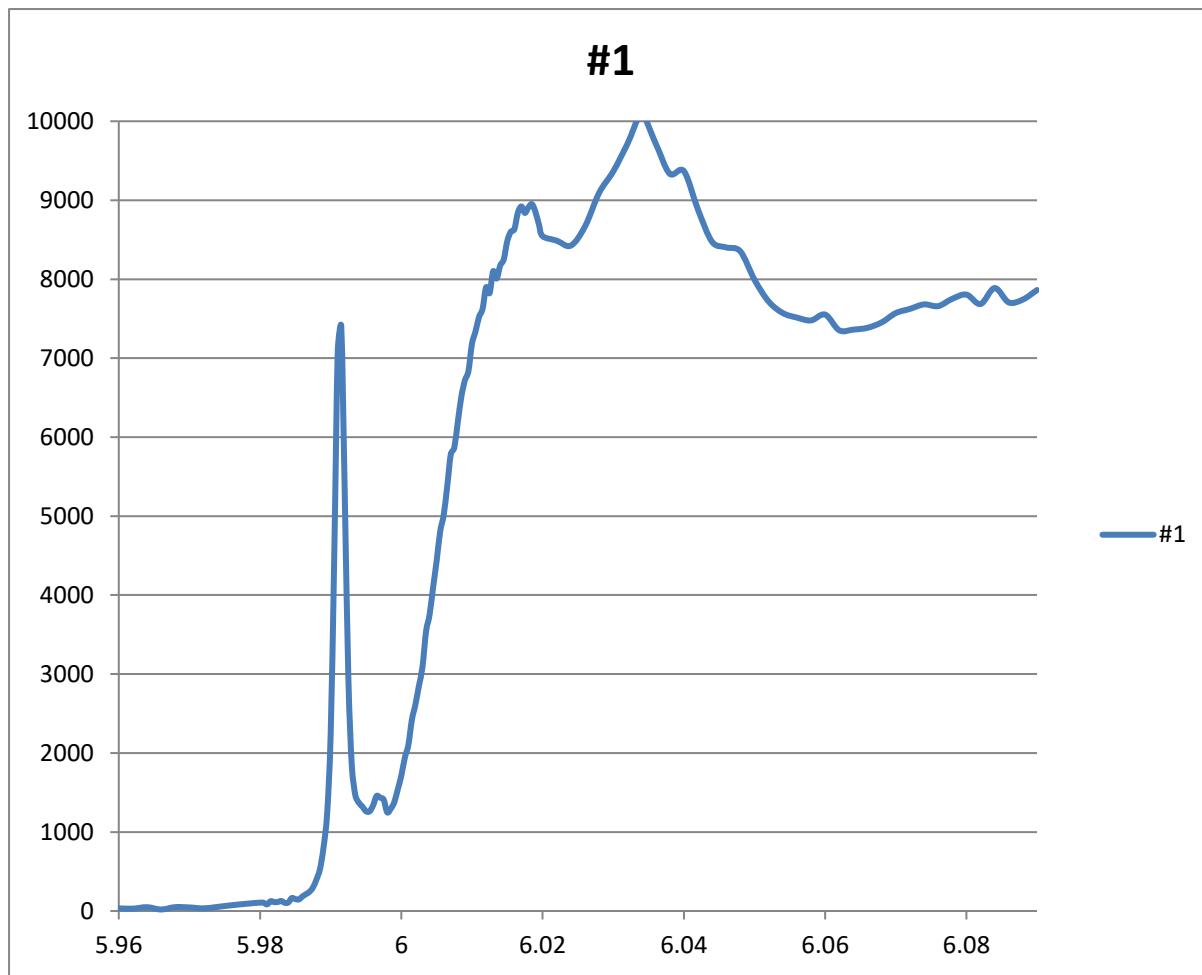


#### **Applying image shear and position jitter corrections**

7. Import the XANES image frames for XANES stack 33488-33613 from directory “Demo/Maia/XANES2/analysis/xanes-33488-33613/images”, but select the element “Cr”.
8. Run the “Shear Correct Image” plugin (“process→User Plugins→Shear Correct Image plugin” menu on the XANES Image window) and select the pop-up option “Read X shear values from CSV file” and load the shear corrections file obtained earlier using the Ca feature “33488-33613-Ca -shear.csv”.
  - a. Scroll through the stack: the shear has been corrected, but the position errors remain.
9. Run the “Align Image” plugin (“process→User Plugins→Align Image plugin” menu on the XANES Image window) and select the pop-up option “Use inter-frame image cross-correlation” and select the shift file determined above using for Ca “33488-33613-Ca-shift.csv”.
  - a. Scroll through the stack: the shear and position errors are now largely corrected.
  - b. Save the corrected stack to file “33488-33613-Cr -shear-shift-corrected.xan”.

#### **Regions and XANES spectra**

10. Select a small Circle shape, and analyze small regions on the stack image with  $\Sigma$ .
11. Open the XANES Regions window and Export then to a CSV file.
12. The file can be viewed in Excel to plot the XANES spectra, or imported into your favourite XAS software package.



Typical small region plot from the exported CSV file, showing a dominantly Cr(VI) XANES spectrum.

## J. Exporting Data

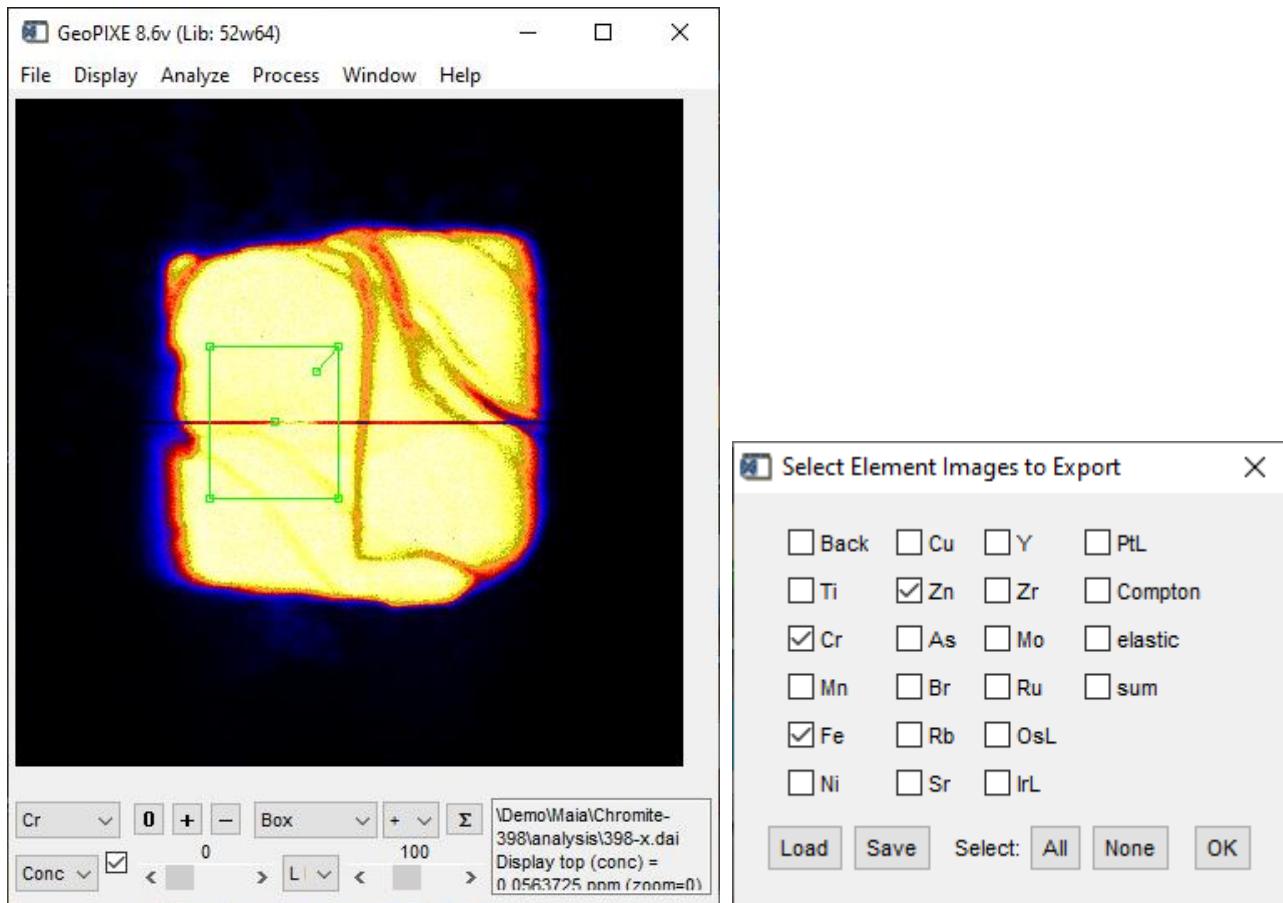
### Example J1: Exporting image pixel data

Exporting pixel concentration data for a whole image (can be huge) or for a selected region involves:

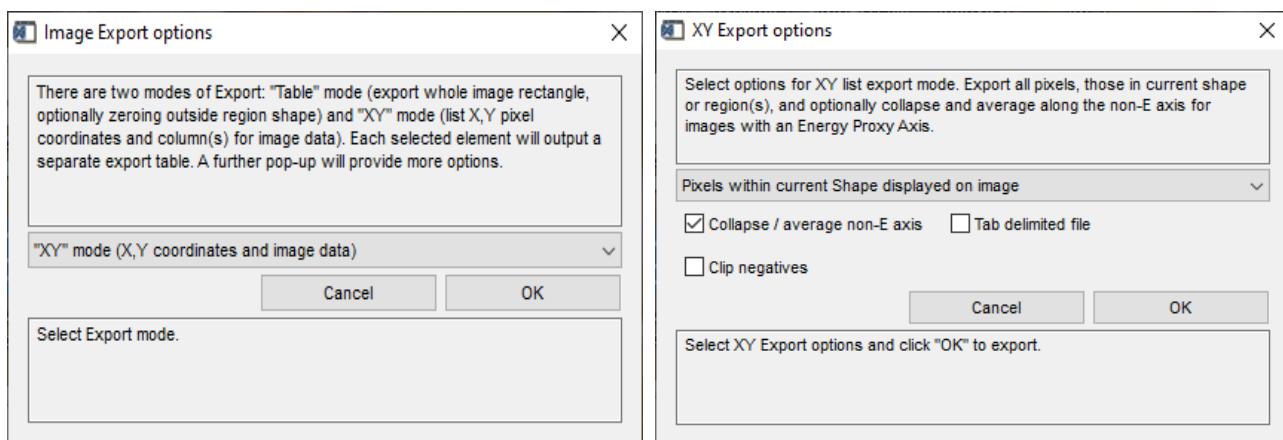
- Using a region or exporting the whole image area.
- Selection of the subset of elements (and other planes) to export.
- Selection of output table format modes.

#### Using a region

1. Load the example data-set “Demo/Maia/Chromite-398/398-x.dai”.
2. Select a small region (or load the examples in “398-x-export-q1.region”).



3. Use the *Image* window menu “Export → Image data → Selected images as ASCII tables”.
4. Select an output filename root for the export CSV file (element names will be appended later).
5. Select the element planes to export (use “Load” if you have a preset list of names that you use).
6. Select table mode between
  - a. “Table” mode
    - i. Will export the whole image, where only the pixels within the region will be non-zero.
  - b. “XY” mode
    - i. Will export each pixel on a row starting with the X,Y pixel coordinate.



7. Select further “XY Export” options:
  - a. Pixels within the current Shape displayed on the image
    - i. This is the shape as seen, without the need to use “Σ” to add it to the Image Region table.
  - b. All image pixels
    - i. The entire image.
  - c. Pixels for current image region

- i. Use the currently selected row in the regions table.
  - d. Pixels for all image regions.
    - i. Repeat the export for all regions.
  - e. Collapse / average non-E axis
    - i. Used with Line XANES, it averages across the non-energy axis so you get a single XANES spectrum output per region.
  - f. Tab delimited
    - i. Switch to a tab delimited output file format (.txt), instead of command-separate (.csv).
  - g. Clip negatives
    - i. Use this to output only positive values; all negatives are clipped at zero.
8. For now, just use the first option, and the others at their defaults and click “OK”.
- a. The result is a separate CSV file for this region “shape” (e.g. “398-x-shape-xy.csv” containing 5 columns: X, Y, Cr, Fe, Zn concentration (ppm). Sadly, this early Maia prototype datafile is not quantitative, so these numbers are strange.

X	Y	Cr	Fe	Zn
142	234	0.049218	0.041999	0.000464
143	234	0.05496	0.044956	0.000479
144	234	0.047933	0.042076	0.000479
145	234	0.05387	0.041085	0.000449
146	234	0.052577	0.043848	0.000433
147	234	0.049117	0.044627	0.000452
148	234	0.047688	0.045191	0.000469
149	234	0.051524	0.042566	0.000474
150	234	0.052079	0.041976	0.000456

9. If you have a number of regions defined in a region file, then load this and select “pixels for all regions” as the output option. This will create a separate file for each region (e.g. “398-x-region0-xy.csv”) with the same element columns.

#### ***Line XANES images and regions***

- 10. If the image data is for Line XANES, in which one axis is related to energy and one is a spatial move axis, then the data export works a little differently.
  - a. For multiple regions, you get a separate file by element with multiple columns based on the regions.
  - b. The ‘collapse’ option can be used to average across the non-energy/spatial axis onto the energy axis, so that you get a single XANES spectrum per region.
  - c. The XAMNES spectra will be plotted in a window to view when you select a row of the *Image Regions* table.
- 11. Setting Line XANES mode
  - a. Line XANES is set or identified by use of the “Energy Proxy Axis” dropdown on the “DA/E.Cal” tab of the *Sort EVT* window (and in the summary in the *Image History* window – see *Image* window “Help→Image properties an history” menu).