

PyMca instructions

To open PyMca:

- Open a terminal (under activities, or right-click mouse)
- Type: module load pymca
- Then: pymca

This brings up the main window.


To open up data:

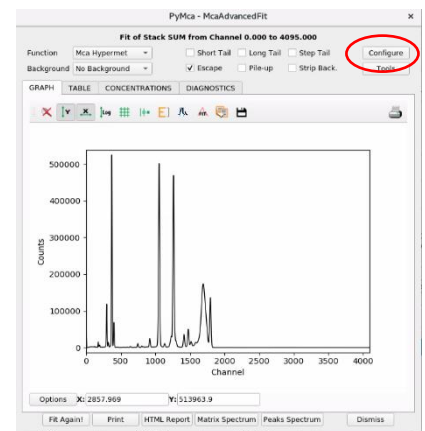
- Choose Tools>ROI imaging>
- Find your data in your folder (computer, /, dls, i14, data, 'experimental year', 'experimental directory', processed/)
 - o This should be a .nxs XRF file (you may need to change the file type to HDF5 Files to show the contents of the folder), from the 'processed' folder
- Click open, then in second drop down menu ('processed', then 'result'), open data (double click) and in the box below make sure 'Signals' is ticked. Click done.
- The data will then load in and display as maps and a spectrum

In the main window you can:

- a) Use spectra selection to edit map, or type in channel amount in new ROI
- b) Select regions in the new map, 'Replace MCA' will show summed spectra from that region

To fit the data using spectral lines:

- In main Window, Click FIT (Mountain shaped logo)  > Advanced
- The program will tell you 'No peaks defined'. Click ok, then choose Configure:
- Here you can edit aspects of the configuration, but to begin with load a configuration file with some of the parameters are pre-set
- Load I14 configuration file



In the Fit tab

You can control the background by choosing 'SNIP' instead of strip and editing the background width (in Setup) to fit your data

You can also choose channels to select a part of the spectrum you want to fit by using 'Limit the fitting region to:' and then editing First and Last channels e.g. 150 – 1100 (each channel is 10 eV). Minimum fitting range is 550 wide.

In the Beam tab tick 'Energy 0' and enter the photon energy you used in KeV (e.g. 12). The weight should be 1.

In the Peaks tab, you can choose the elements to include. Click on the element, and then select the lines (e.g. K or L). Choosing K will include both Ka and Kb as a single unit with a pre-defined ratio between them, whereas selecting them individually they will be fitted as separate peaks. Most common is to use K. You will be unable to select lines outside of your fitting range.

In the Attenuators tab you need to set up the specific experimental parameters based on the set up you used. These are needed for quantification calculations to be accurate. The configuration file has several aspects of the I14 beamline already included.

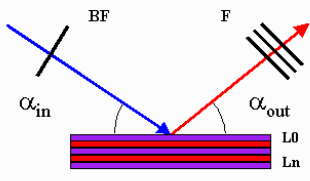
Variation may come for your sample in the support (e.g. a silicon nitride window) which will have a specific thickness, or if your sample has layers. This can be edited by choosing 'Matrix' as your material and editing in the following matrix tab.

FIT	DETECTOR	BEAM	PEAKS	PEAK SHAPE	ATTENUATORS	MATRIX	CONCENTRATIONS	XRFMC
Attenuator	Name	Material	Density (g/cm3)	Thickness (cm)	Funny Factor			
<input type="checkbox"/>	Filter 0	kapton	-	0.0	0.0	1.0		
<input checked="" type="checkbox"/>	Filter 1	atmosphere	Air	0.0012048	1.0	1.0		
<input checked="" type="checkbox"/>	Filter 2	deadlayer	Si1	2.33	0.0002	1.0		
<input type="checkbox"/>	Filter 3	absorber	-	0.0	0.0	1.0		
<input checked="" type="checkbox"/>	Filter 4	window	Be	1.848	0.00125	1.0		
<input checked="" type="checkbox"/>	Filter 5	contact	Au1	19.37	1e-06	1.0		
<input type="checkbox"/>	Filter 6	Filter 6	-	0.0	0.0	1.0		
<input type="checkbox"/>	Filter 7	Filter 7	-	0.0	0.0	1.0		
<input type="checkbox"/>	BeamFilter0	BeamFilter0	-	0.0	0.0	1.0		
<input type="checkbox"/>	BeamFilter1	BeamFilter1	-	0.0	0.0	1.0		
<input checked="" type="checkbox"/>	Detector	Detector	Si1	2.33	0.45	1.0		
<input checked="" type="checkbox"/>	Matrix	Matrix	MULTILAYER	0.0	0.1	1.0		

In the Matrix tab, the incoming and outgoing samples can be set up as 0 and 135 degrees, respectively. Here is where you can add and edit layers for multi-layered materials. For example, for the AXO standard, the sample was deposited directly on a silicon nitride window of 200 nm thickness. **Disclaimer:** Remember to change this composition if your samples are resin-embedded sections of different thicknesses.

PyMca - MCA Fit Parameters


FIT DETECTOR BEAM PEAKS PEAK SHAPE ATTENUATORS MATRIX CONCENTRATIONS XRFMC



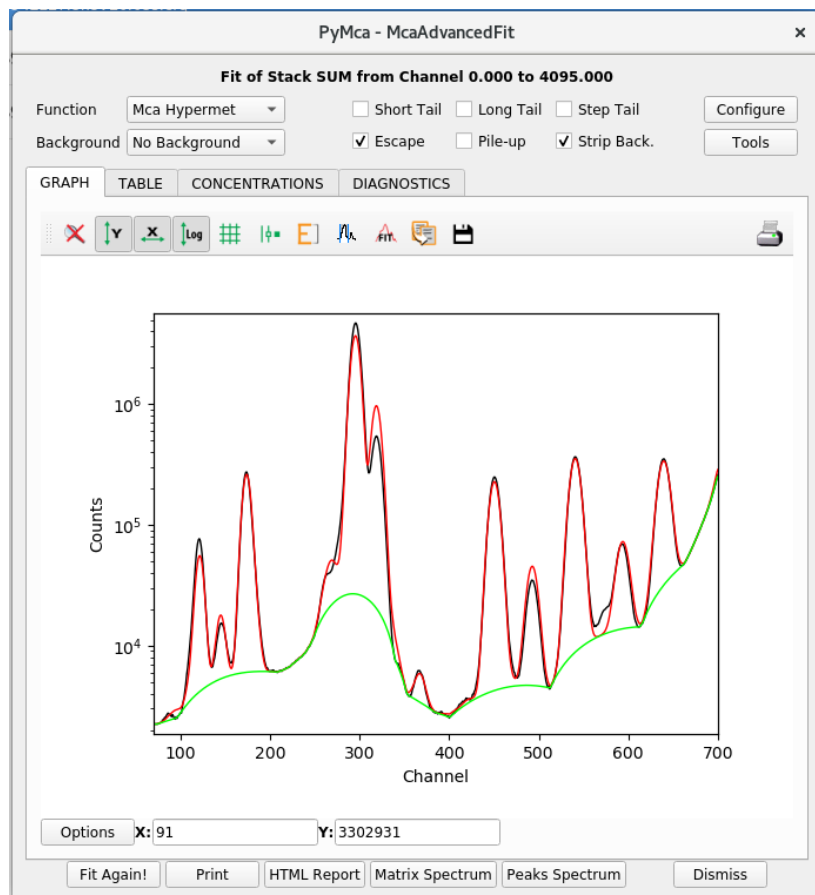
Incoming Angle (deg.): 0
 Outgoing Angle (deg.): 135
☐ Scattering Angle (deg.): 135

This matrix definition will only be considered if Matrix is selected and material is set to MULTILAYER in the ATTENUATORS tab.

Layer	Name	Material	Density (g/cm ³)	Thickness (cm)
<input checked="" type="checkbox"/> Layer0	Layer0	Si3N4	2.33	2e-05
<input type="checkbox"/> Layer1	Layer1	-	0.0	0.0
<input type="checkbox"/> Layer2	Layer2	-	0.0	0.0
<input type="checkbox"/> Layer3	Layer3	-	0.0	0.0
<input type="checkbox"/> Layer4	Layer4	-	0.0	0.0
<input type="checkbox"/> Layer5	Layer5	-	0.0	0.0
<input type="checkbox"/> Layer6	Layer6	-	0.0	0.0
<input type="checkbox"/> Layer7	Layer7	-	0.0	0.0
<input type="checkbox"/> Layer8	Layer8	-	0.0	0.0
<input type="checkbox"/> Layer9	Layer9	-	0.0	0.0


Finally click OK and then Fit Again! if no fit appears. Plotting the graph on a log scale (using this button ) can make it easier to see details in the fit. You can then iterate changes to elements to include or remove elements until you are happy with the fit. Make sure you have 'Strip back.' Ticked when fitting.


Several iterations may be required adding/removing elements from the "peaks" tab in order to get a reasonable fit for the spectrum, such as:



When you are happy, save the configuration file in your processing folder, and then go back to the main page of pymca.

Fast preliminary analysis, and stacking different elemental maps into one image:

Use the window that was opened when selecting the ROI Imaging tool. Click the gear icon , and select Fast XRF Stack Fitting. Choose the configuration file you created with the peak fitting, choose an output directory where the individual elemental maps will be saved. Make sure to select "Tiffs", so that Tiff files with the individual maps will be created and saved.

A window has now opened displaying the individual elemental maps. Choose the ones you want to stack, adding them to the stack by clicking "Add image". In the ROI Imaging window you can now give each element a different colour by ticking and unticking the boxes at the bottom left, and save the image with the save icon .

Batch fitting:

The ROI Imaging tool can give a quick and preliminary analysis of the data, but a more thorough analysis is done through batch fitting. Batch fitting is performed as follows:

Click Tools > Batch Fitting

Point it to the processed file you've been looking at (.nxs) for Input File list

Choose configuration file (the one you previously saved) in 'Fit Configuration File'.

In 'Output dir' choose where you want the output files to be stored (make a new folder for this).

Choose the file types you want by clicking, then click start.

This will take approximately 1 hour to process.

Concentrations/Quantification:

For concentrations, you will need to have an AXO standard scan run at the same parameters as your sample. You can then fit the AXO spectra and use the 'Concentrations' tab to edit the flux until the values given under 'mass fraction' match the known ones from the AXO standard documentation.

1. Load the 'processed' file of your AXO standard (tools>ROI Imaging)
2. Choose Fit>Advanced as above, then 'Configure'
3. Load config file (you can use the I14 config, or the specific AXO standard config that has elements already chosen) and correct the energy to the one you used to collect the standard
4. Make sure that in the 'Concentrations' tab, the time is changed to the dwell time you used, and the active area detector is set to **1.54** and the sample distance to **1.7**
5. Click ok
6. Edit the fit (may need adding/taking away elements as described above) until your fit matches the data well. Remember to choose 'log' to see the fit in detail. You will probably need to include W, and some other elements.
7. In the 'Concentrations tab' of the Fit window, check the amounts in 'mass fraction' for each of the AXO elements match the given numbers for specific elements in the AXO documentation:

Details for AXO (01)

	<i>ng.mm²</i>	<i>mass fraction</i>	<i>ppm</i>
Cu	2.4	0.0034	3394
Fe	3.1	0.0044	4384
Cr	5.4	0.0076	7637
Ti	6.2	0.0088	8768

To get ppms directly in the 'mass fraction column': -E06 from the flux

8. Refine flux up and down to make the elemental ppm/mass fraction values correct, or as close as can be (they may not match exactly). Ppms are more precise (more sig. figs).

Save your config file with the energy and scan number for reference. You can now use this flux as the set amount for your own scans. Keeping this number the same, as well as all other values in the 'Concentrations' tab will mean you are getting a 'close enough' quantification as well as being comparable between scans.