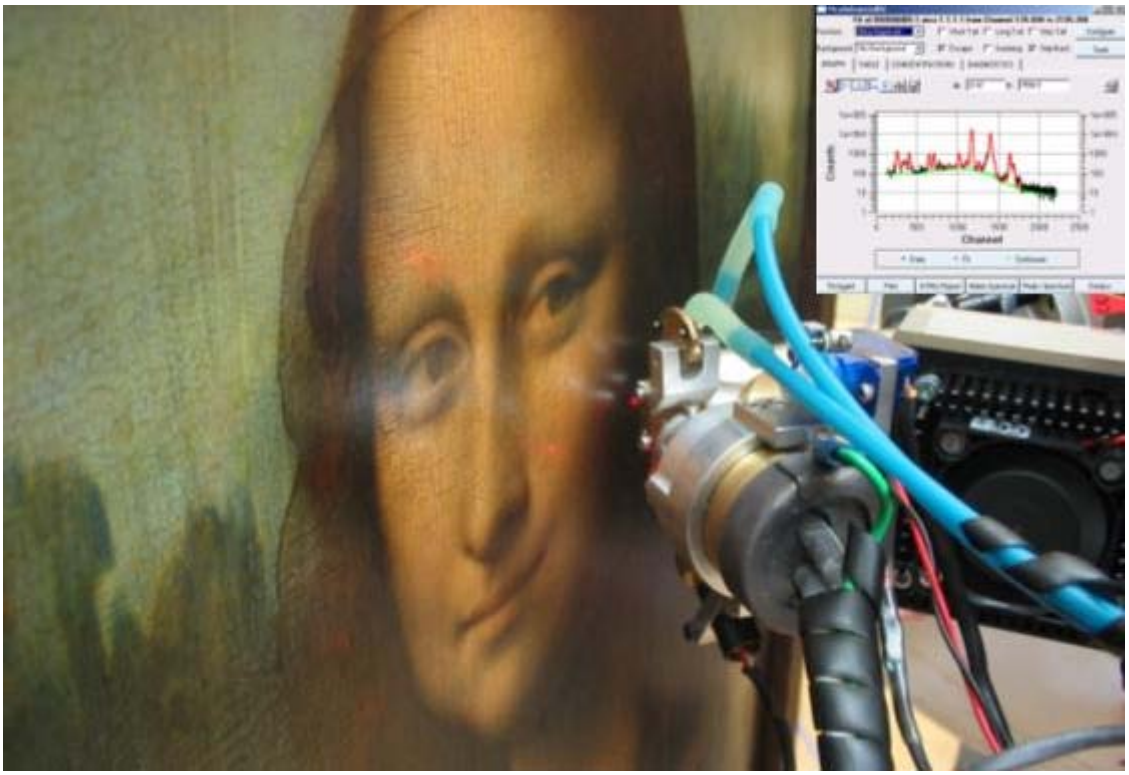
Fluorescence Spectra Analysis in Diamond

MX Village

PyMca User Guide



diamond

Using PyMca in Diamond MX Village

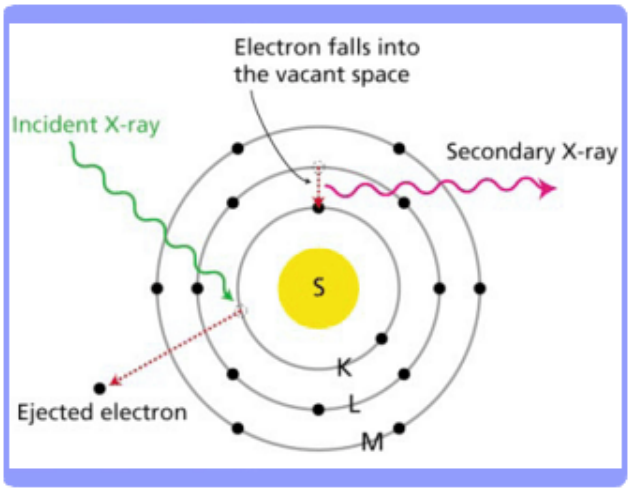
Introduction

PyMca is an X-ray Fluorescence Toolkit developed by the Software Group of the European Synchrotron Radiation Facility (ESRF)[1]. PyMca provides a powerful collection of tools for the analysis of fluorescence spectra collected using synchrotron radiation.

Within the MX Village at Diamond we anticipate the main use PyMca to identify the presence of atoms useful for phasing within soaked crystals. Rather than doing a specific edge scan taking around minutes to complete and only allowing you to check for one component at a time, it may be better to take an mca spectrum at a high energy (eg. 16000eV) which will enable you to search for the presence of a number of components with a 1s exposure.

The PyMca program is open source software with a number of options for use. This manual will guide you through the use of PyMca as it is installed in the MX Village at Diamond to identify any peaks in the mca specta you collect.

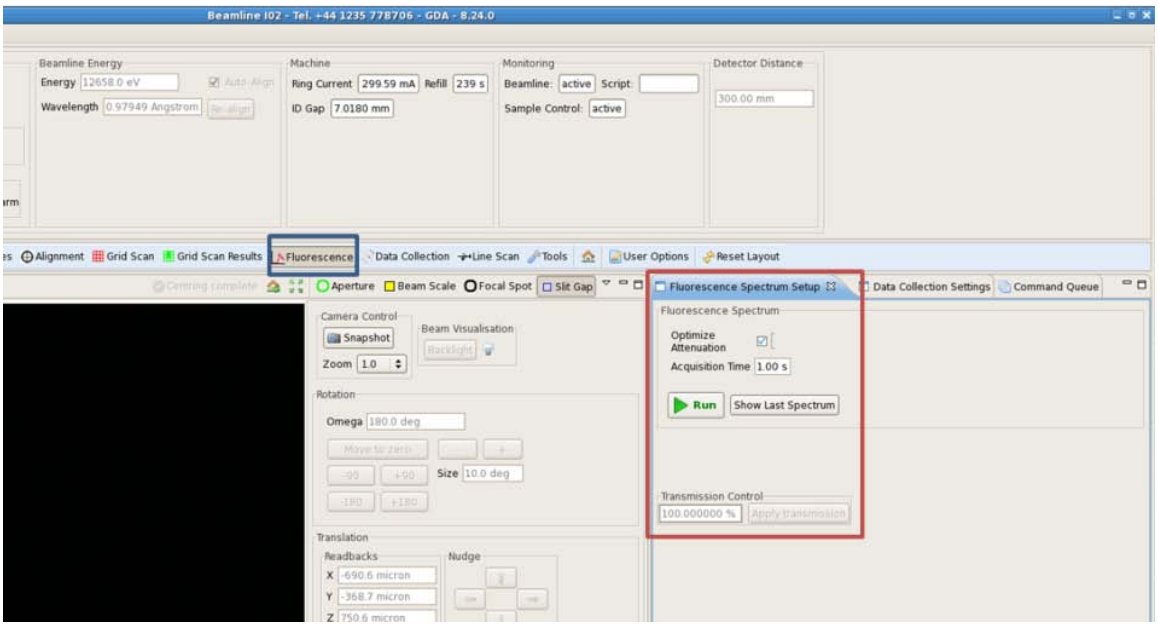
The fluorescence detectors in the MX village at Diamond can typically see fluorescence peaks (secondary X-rays) from between 1000eV and 15 000eV. The incident beam energy can be changed between 5000eV and 20 000eV. Please see the tables at the back of this manual or the orange X-ray Data Booklet to determine whether your sample fluorescence can be excited and/or detected.



[1] V.A. Solé, E. Papillon, M. Cotte, Ph. Walter, J. Susini, A multiplatform code for the analysis of energy-dispersive X-ray fluorescence spectra, Spectrochim. Acta Part B 62 (2007) 63-68.

Collecting an MCA Spectrum

Before PyMca is of any use you need to collect an MCA spectrum. This is done in GDA from the fluorescence perspective, highlighted with a blue box below. Simply go to the Fluorescence Spectrum Setup (in the red box in the figure) and click 'Run'. With 'optimise attenuation' selected you should obtain a scan with peaks in the spectrum for the elements present in the sample. If you are unable to see the peaks you expect, or if they appear with very low counts, you could deselect 'optimise attenuation' and instead change the transmission to 20% and collect data for 1s. This will expose your crystal to more radiation.



This spectrum can be collected at any energy, but you do need to consider how fluorescence works. **Elements will only fluoresce if the incident beam energy is higher than the excitation energy of the element**. A peak will also be observed due to the inelastic scatter of the incident beam.

Consequently, you are best collecting a spectrum from a high energy (say 20 000eV) which will give an inelastic scatter peak well away from the elements you might be interested in, and will excite a large range of fluorescent elements.

Collecting your MCA spectrum at 12 658eV (the standard beamline energy) will give an inelastic scatter peak that can easily be confused with selenium or bromide fluorescence, giving an ambiguous result (see example on page 10).

Opening PyMca

There is a launcher for PyMca in the DLS-Launchers folder on your Desktop. This will launch PyMca in a directory where you can save results and will put a typical configuration file in your processing directory.

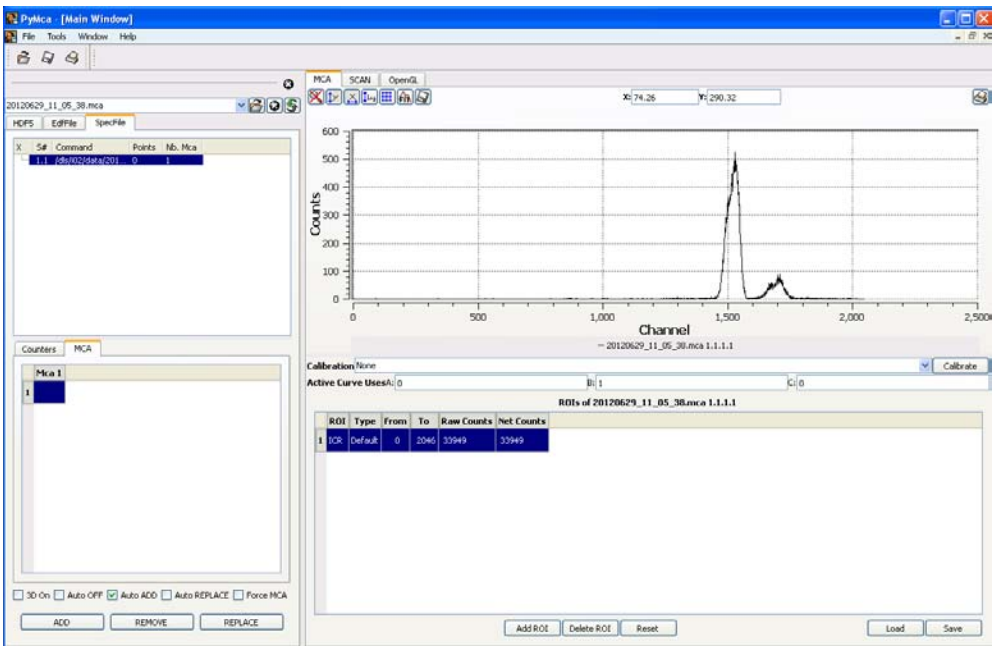
To launch PyMca double click on this icon. This will open the main PyMca window. Open your MCA spectrum:

File→Open→Data Source→Select file location

Your MCA file will be saved in the top directory for your visit with a name in the format:

/dls/i0X/data/2012/mxXXXX-X/20121009_08_51_35.mca where the filename is the date and time of the data collection.

Click 'Open' and the spectrum will appear in the plot window.

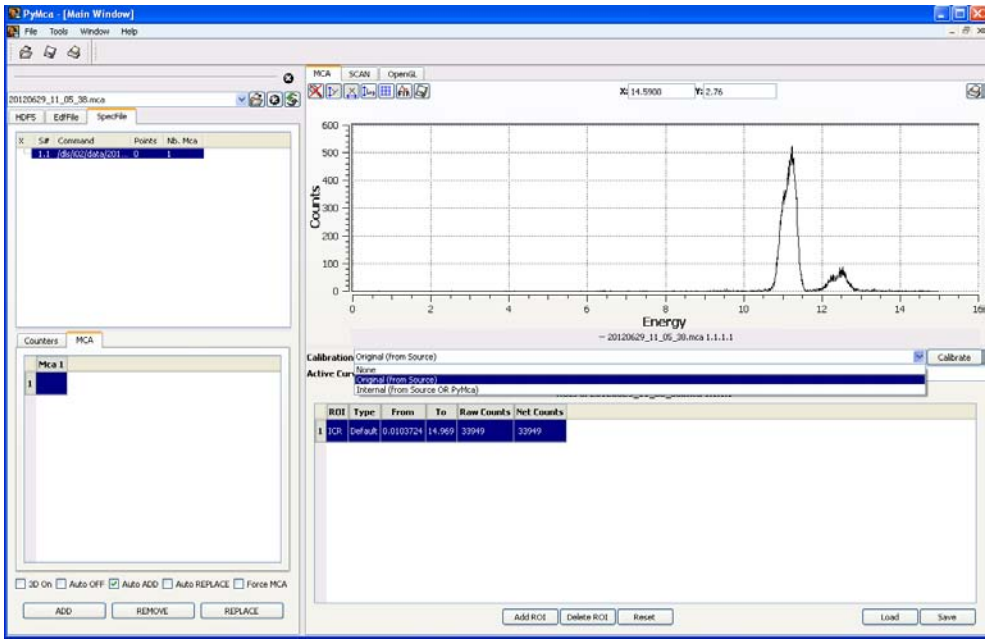


Callibrate your MCA Plot

Below the plot window there is a drop down menu of calibration options. Each beamline produces calibrated MCA spectra so the internal calibration will be correct.

Select calibration from source

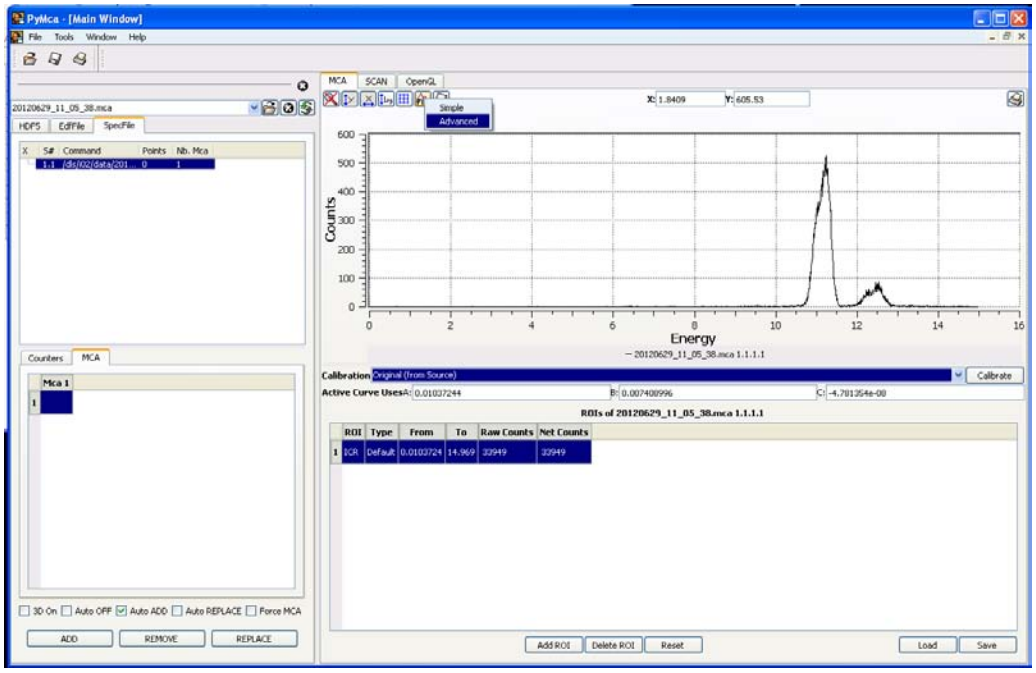
The plot window will now have an x-axis reading of Energy rather than channel as it had been previously.



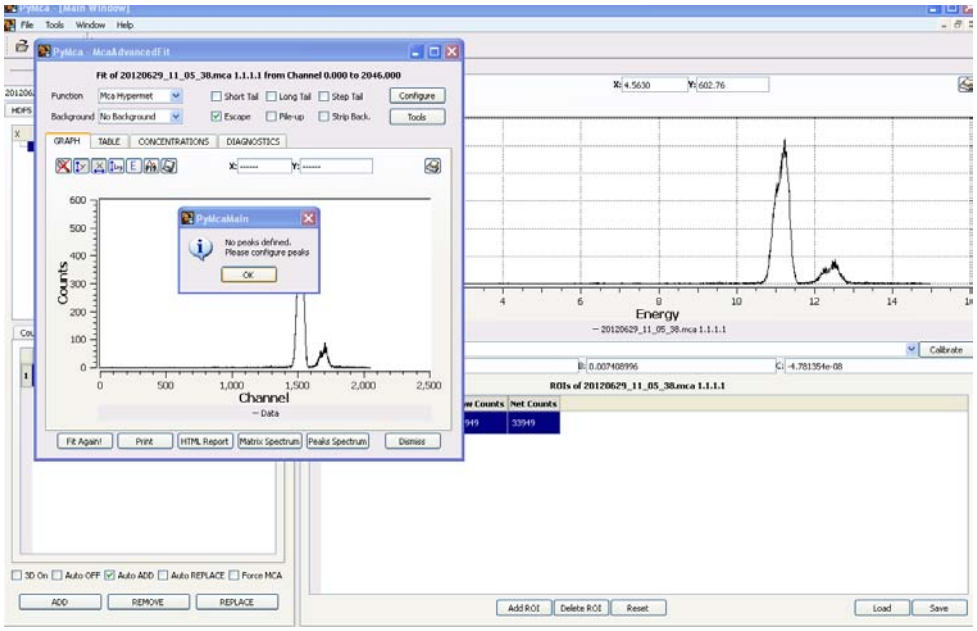
Fit your MCA Spectrum

This is the process by which PyMca is able to offer suggestions as to the composition of the fluorescence in your sample.

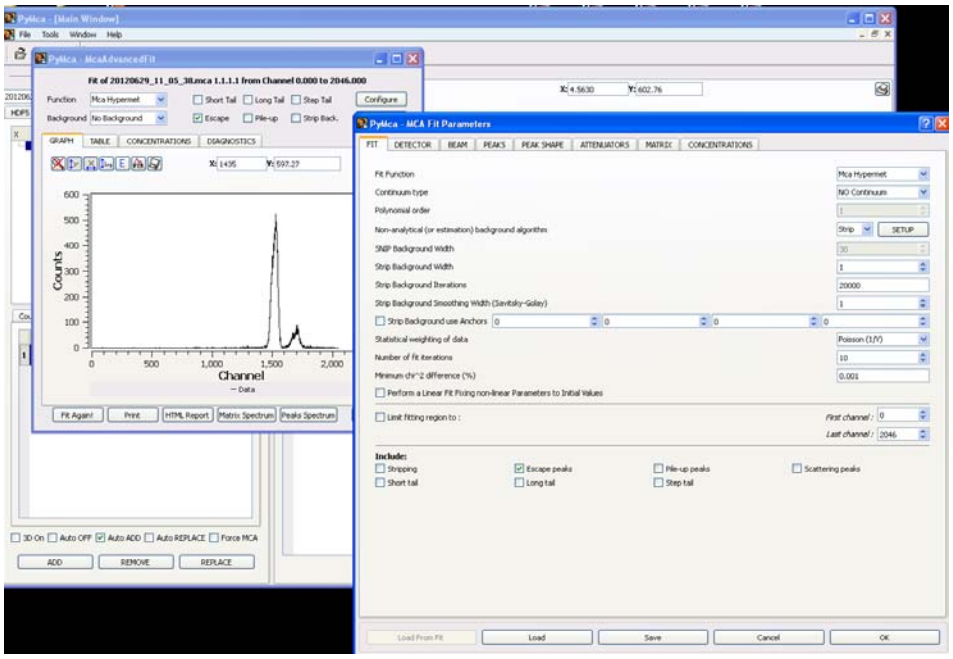
1. Press the FIT button above the plot window and select Advanced to open the MCA Advanced Fit Window.



2. The fit window will open with an error 'No peaks defined'. Click OK on the message box.



3. Click on configure to load a configuration file. Select load and then navigate to your visit processing directory where you will find a file called 'Pymca.cfg' which you should load.
   If this file is not available, try reloading PyMca using the icon in the DLS-Launcher. If this is unsuccessful please speak with your local contact.



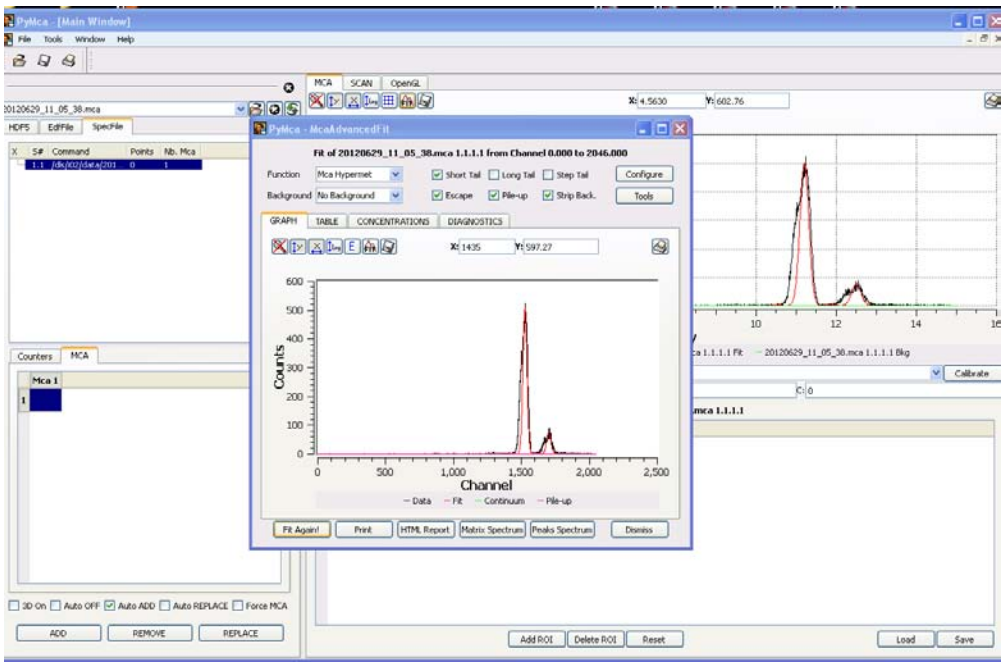
The prepared configuration file will search for suitable edges of:

a. Sodium, Magnesium, Phosphorous, Sulphur, Chlorine, Potassium, Calcium, Manganese, Iron, Cobalt, Nickel, Copper, Zinc, Arsenic, Selenium, Bromine, Molybdenum, Silver, Cadmium, Iodine, Tungsten, Platinum, Mercury
b. It will search assuming an incident wavelength of 20 000 eV

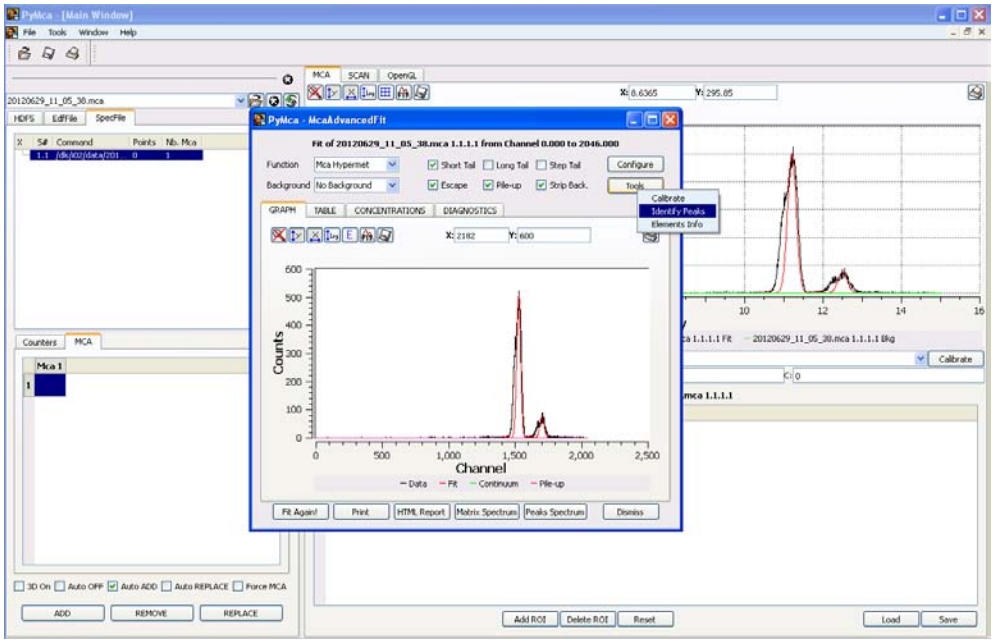
After loading the configuration file you can navigate the tabs to alter the incident energy to that which is correct for your sample and to add or remove elements you are searching for.

When you are ready, click on 'OK'.

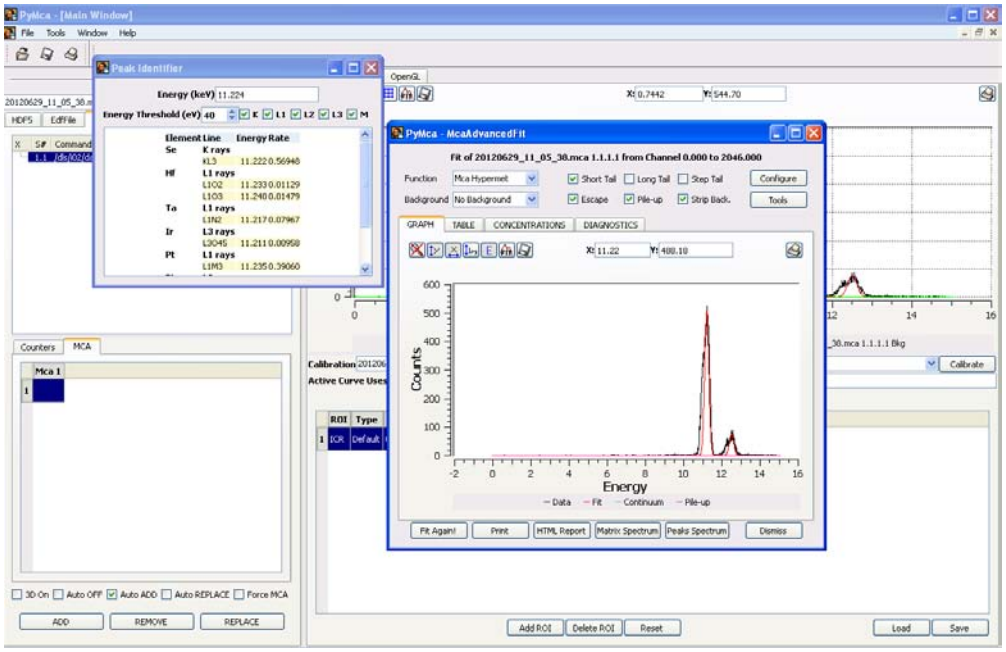
4. Click on ‘Fit Again!’ to get a fit to your spectrum



5. Click on Tools and select ‘Identify peaks’



6. Left click on the peaks you have and decide which element is present from the 'Peak Identifier' window



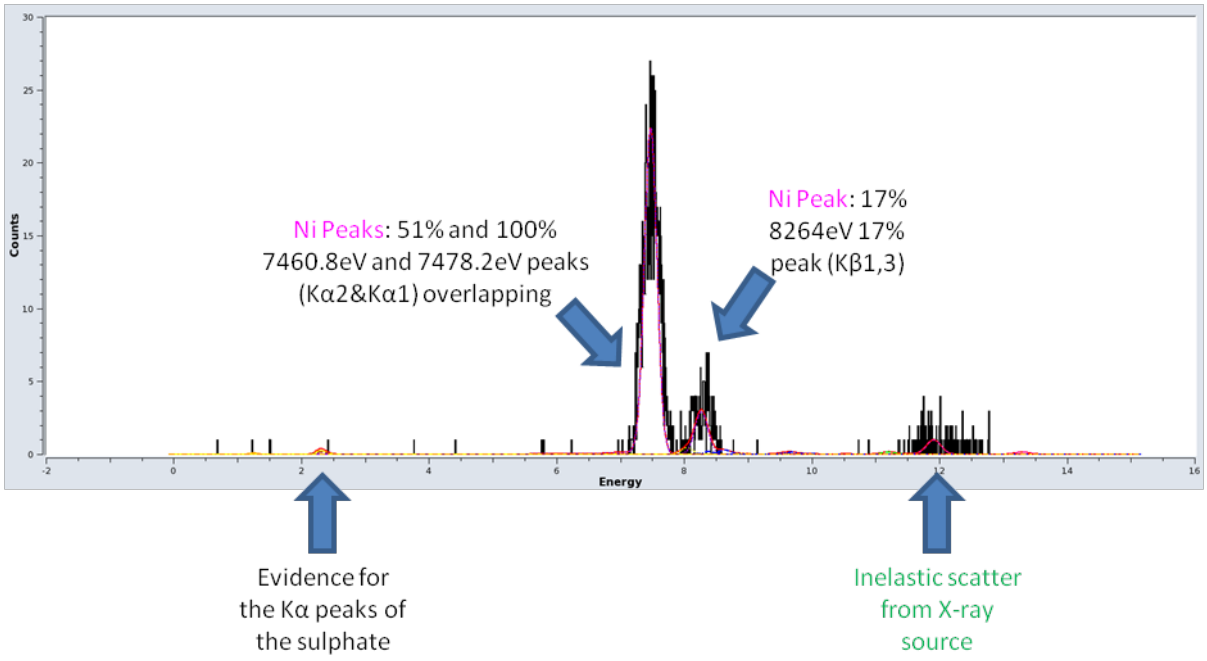
7. Choose the element that makes sense from the peaks.

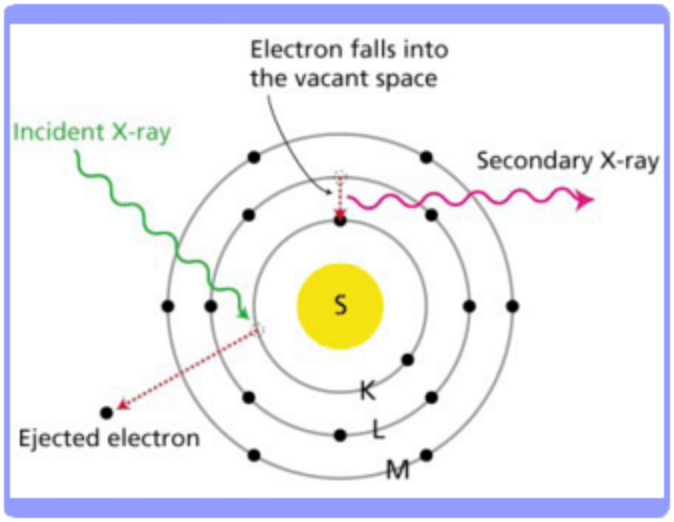
8. An alternative method to identify your peaks is to click on the 'Peaks Spectrum' tab beneath the plot. This will graphically illustrate the peaks that have been fitted with a table legend to explain which curve is for which element.

9. Finally, if you click on HTML Report and select a location that this can be saved in, you will have a report of the 'goodness-of-fit' for each element. Those that are likely to be in your sample will have a high chi-squared value and a high fitted area value.

Example MCA Spectrum

Below is an MCA spectrum collected on I02 at Diamond. The incident X-ray beam was at 12400eV. The sample was a loop containing nickel sulphate. The image is that of the data with the fit shown as a red curve.

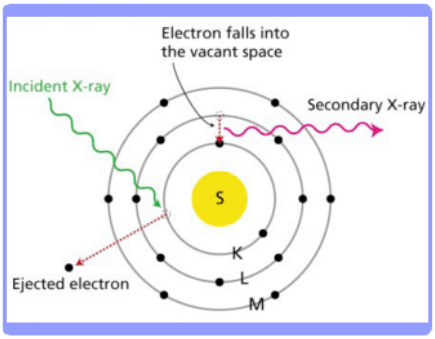




Excitation Energies for Common Elements

To excite fluorescence from an element it is recommended that the incident X-ray beam have energy of 1000eV higher than the excitation energy[2] in the table below[3]. To see peaks from a number of elements ensure they incident beam has a higher energy than the highest component in the mixture. The table is in order of atomic number, which approximately correlates to excitation energy.

	Element	Excitation	
		Edge	Energy (eV)
Na	Sodium	K	1072.1
P	Phosphorous	K	2145.5
S	Sulphur	K	2472.0
Cl	Chlorine	K	2822.4
K	Potassium	K	3607.4
Ca	Calcium	K	4038.1
Ti	Titanium	K	4966.4
V	Vanadium	K	5465.1
Mn	Manganese	K	6539.0
Fe	Iron	K	7112.0
Co	Cobalt	K	7708.9
Ni	Nickel	K	8332.8
Cu	Copper	K	8978.9
Zn	Zinc	K	9658.6
As	Arsenic	K	11866.7
Se	Selenium	K	12657.8
Br	Bromine	K	13473.0
Mo	Molybdenum	L1	2865.5
Ag	Silver	L1	3805.8
Cd	Cadmium	L1	4018.0
I	Iodine	L1	5188.1
La	Lanthanum	L1	6266.3
Sm	Samarium	L1	7736.8
Eu	Europium	L1	8052.0
Gd	Gadolinium	L1	8375.6
Dy	Dysprosium	L1	9045.8
Ho	Holmiun	L1	9394.2
Yb	Ytterbium	L1	10486.4
Lu	Lutetium	L1	10870.4
W	Tungsten	L1	12099.8
Re	Rhenium	L1	12526.7
Os	Osmium	L1	12968.0
Ir	Iridium	L1	13418.5
Pt	Platinum	L1	13880.5
Au	Gold	L1	14352.8
Hg	Mercury	L1	14839.3
Pb	Lead	L1	15860.8



[2] Where a number of electrons can be excited, only the highest accessible energy is given.
[3] Data taken from the PyMCA element analysis details

Excitation Energies for Common Elements

The table below gives the main emission energies for a number of commonly used elements. Where a number of emission peaks exist the highest peaks have been given, along with the percentage height of the peak[4]. The table is in order of atomic number, which approximately correlates to emission energy.

	Element	Emission Kα2		Emission Kα1		Emission Kβ3		Emission Lα1		Emission Lβ1		Emission Lβ2	
		Energy (eV)	Percent	Energy (eV)	Percent	Energy (eV)	Percent	Energy (eV)	Percent	Energy (eV)	Percent	Energy (eV)	Percent
Na	Sodium	1041.0	150										
P	Phosphorous	2012.7	50	2013.7	100								
S	Sulphur	2306.6	50	2307.8	100								
Cl	Chlorine	2620.8	50	2622.4	100								
K	Potassium	3311.1	50	3313.8	100								
Ca	Calcium	3688.1	50	3691.7	100								
Ti	Titanium	4504.9	50	4510.8	100								
V	Vanadium	4944.6	50	4952.2	100								
Mn	Manganese	5887.6	50	5898.8	100								
Fe	Iron	6390.8	50	6403.8	100	7058.0	17						
Co	Cobalt	6915.3	51	6930.3	100	7649.4	17						
Ni	Nickel	7460.9	51	7478.2	100	8264.7	17						
Cu	Copper	8027.8	51	8047.8	100	8905.3	17						
Zn	Zinc	8615.8	51	8638.9	100	9572.0	17						
As	Arsenic	10508.0	51	10543.7	100	11726.2	13						
Se	Selenium	11181.0	52	11222.0	100	12495.9	13						
Br	Bromine	11877.6	52	11924.2	100	13291.4	14						
Mo	Molybdenum	17374.3	52	17479.3	100	19608.3	15	2293.2	100				
Ag	Silver							2984.3	100	3150.9	56		
Cd	Cadmium							3733.7	100	3316.6	58		
I	Iodine							3937.6	100	4220.7	61		
La	Lanthanum							4651.0	100	5042.1	60	5383.5	21
Sm	Samarium							5636.1	100	6205.1	61	6587.0	21
Eu	Europium							5845.7	100	6456.5	62	6843.2	21
Gd	Gadolinium							6057.2	100	6713.2	62	7102.6	21
Dy	Dysprosium							6495.2	100	7247.7	62	7635.7	20
Ho	Holmiun							6719.8	100	7525.3	64	7911.0	20
Yb	Ytterbium							7415.6	100	8401.8	65	8758.8	20
Lu	Lutetium							7655.5	100	8709.0	66	9048.9	19
W	Tungsten							8397.6	100	9672.4	67	9961.5	21
Re	Rhenium							8652.5	100	10010.0	66	10275.2	22
Os	Osmium							8911.7	100	10355.3	67	10598.5	22
Ir	Iridium							9175.1	100	10708.3	66	10920.3	22
Pt	Platinum							9442.3	100	11070.7	67	11250.5	25
Au	Gold							9713.3	100	11442.3	67	11584.7	23
Hg	Mercury							9988.8	100	11822.6	67	11924.2	24
Pb	Lead							10551.5	100	12613.7	66	12622.6	25

[4] Data taken from the orange X-ray Data Booklet published in 2009 by Lightsorces.org from the Lawrence Berkely National Laboratory