XANES data treatment by Dawn, Mantis & Athena

I14 Beamline
Diamond Light Source

DISCLAIMER: This document does not aim to be a comprehensive guide describing all the capabilities of DAWN, MANTiS or ATHENA, but rather a descriptive document on how to use these software for I14 data visualisation

Required software – Linux resources

We recommend the use of NoMachine software to connect to a 'New Virtual Desktop' through ssh.diamond.ac.uk or nx-user.diamond.ac.uk (see "how_to_connect_to_i14-workstations" guide already distributed), for being able to access these resources, by typing the following command-lines on a Linux terminal

- Data Analysis WorkbeNch -- DAWN

```
module load dawn
dawn &
```

- Multivariate Analysis Tool for Spectromicroscopy -- MANTiS

```
cd /dls_sw/i14/scripts/
./launch_mantis_py3.sh
```

- Demeter software – Athena

```
cd /dls_sw/i14/scripts/
./launch_athena.sh
```

Alternatively, these software can be downloaded for using them on Windows computers (see next slide), although they may present some operational issues.

Required software – Free downloads

- Data Analysis WorkbeNch -- DAWN

Eclipsed based application for scientific data analysis fully supported by Diamond Light Source
Download at: https://dawnsci.org/ (accessed on 26/11/2019)

- Multivariate Analysis Tool for Spectromicroscopy -- MANTiS

Cross-platform tool developed in Python for spectromicroscopy data analysis
Download at: http://spectromicroscopy.com/ (accessed on 26/11/2019)

- Demeter software – Athena

Comprehensive system for processing and analysing X-ray Absorption Spectroscopy data
Download at: https://github.com/bruceravel/demeter (accessed on 26/11/2019)

* Note that each individual software has their own references to be cited (included in their webpages)

Data Analysis WorkbeNch -- DAWN

Dawn is the recommended software for a quick visualisation of your data, either using the ‘DataVis’ perspective or the ‘Mapping’ (for a view resembling the GDA acquisition software)

To load Dawn, just type in the Linux terminal the next two lines consecutively, hitting ‘Enter’ afterwards:

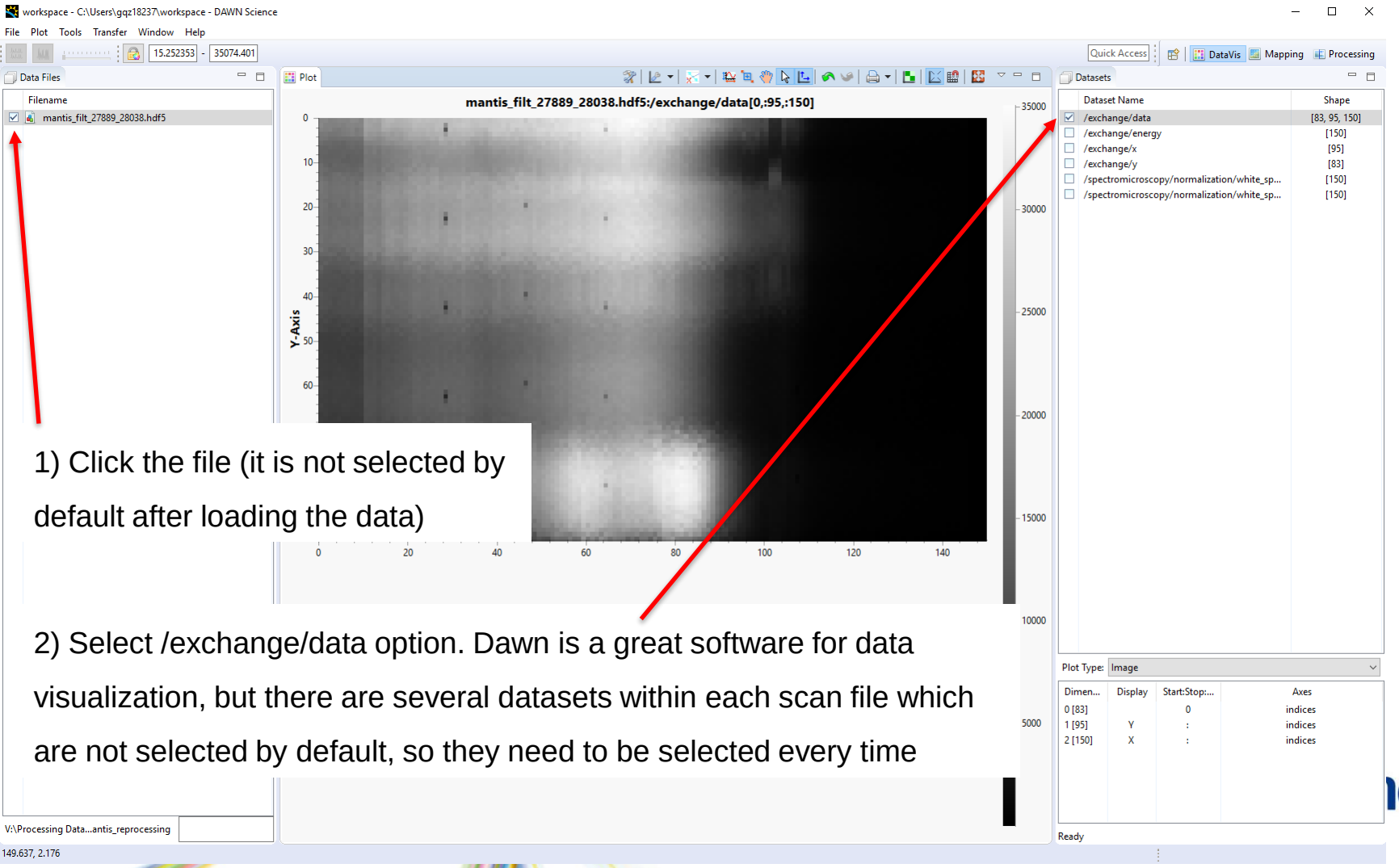
```
module load dawn
dawn &
```

Finally, a video-tutorial on “loading DAWN for inspecting metadata from the ‘i14-xxxxx.nxs’ files” can be found at: https://www.diamond.ac.uk/Instruments/Imaging-and-Microscopy/I14/Access-to-I14/Beamtime-preparation/Manuals-and-tutorials.html

DAWN -- Getting started -- Opening .hdf5 files

All files are located in: /dls/staging/dls/i14/data/2019/sp20627-1/processing/

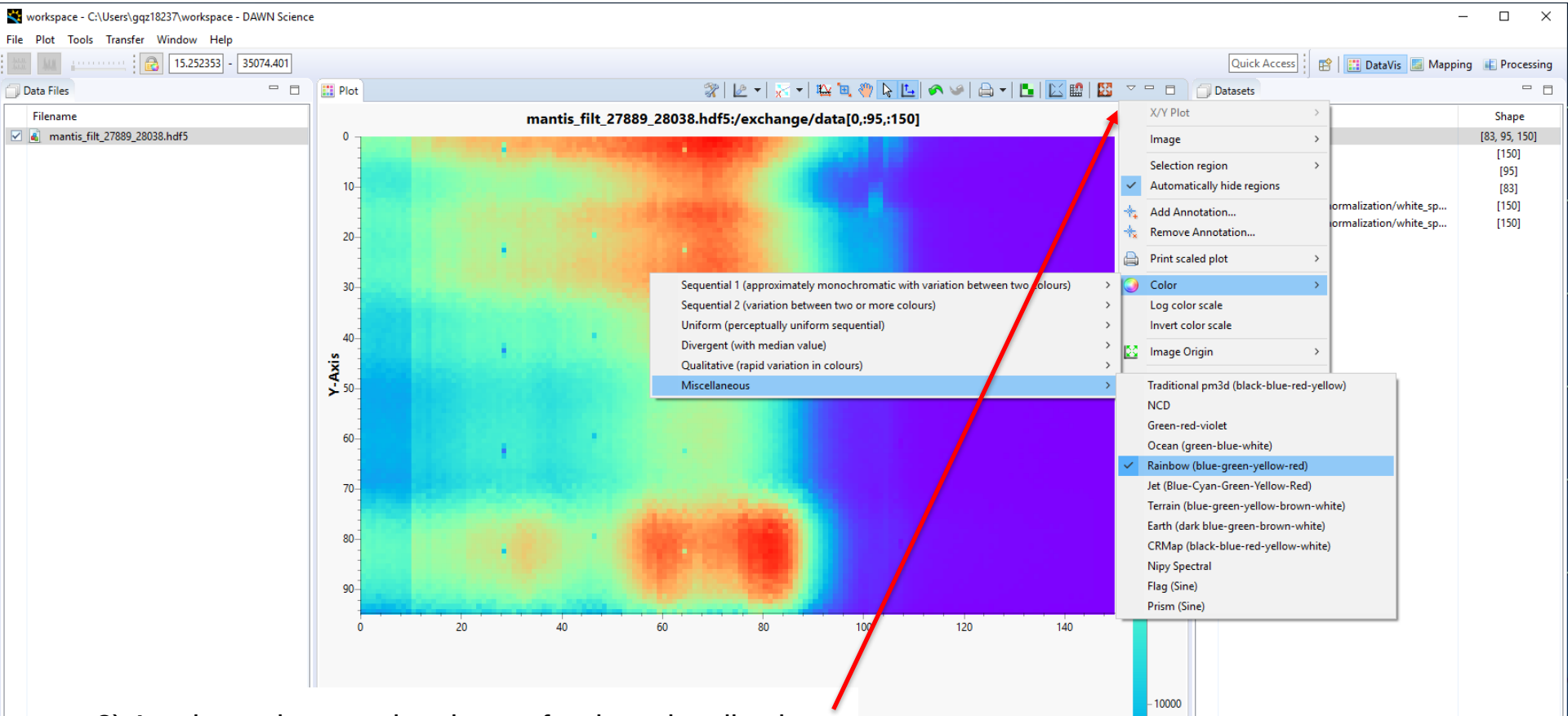
example: mantis_filt_27889_28038.hdf5



1) Click the file (it is not selected by default after loading the data)

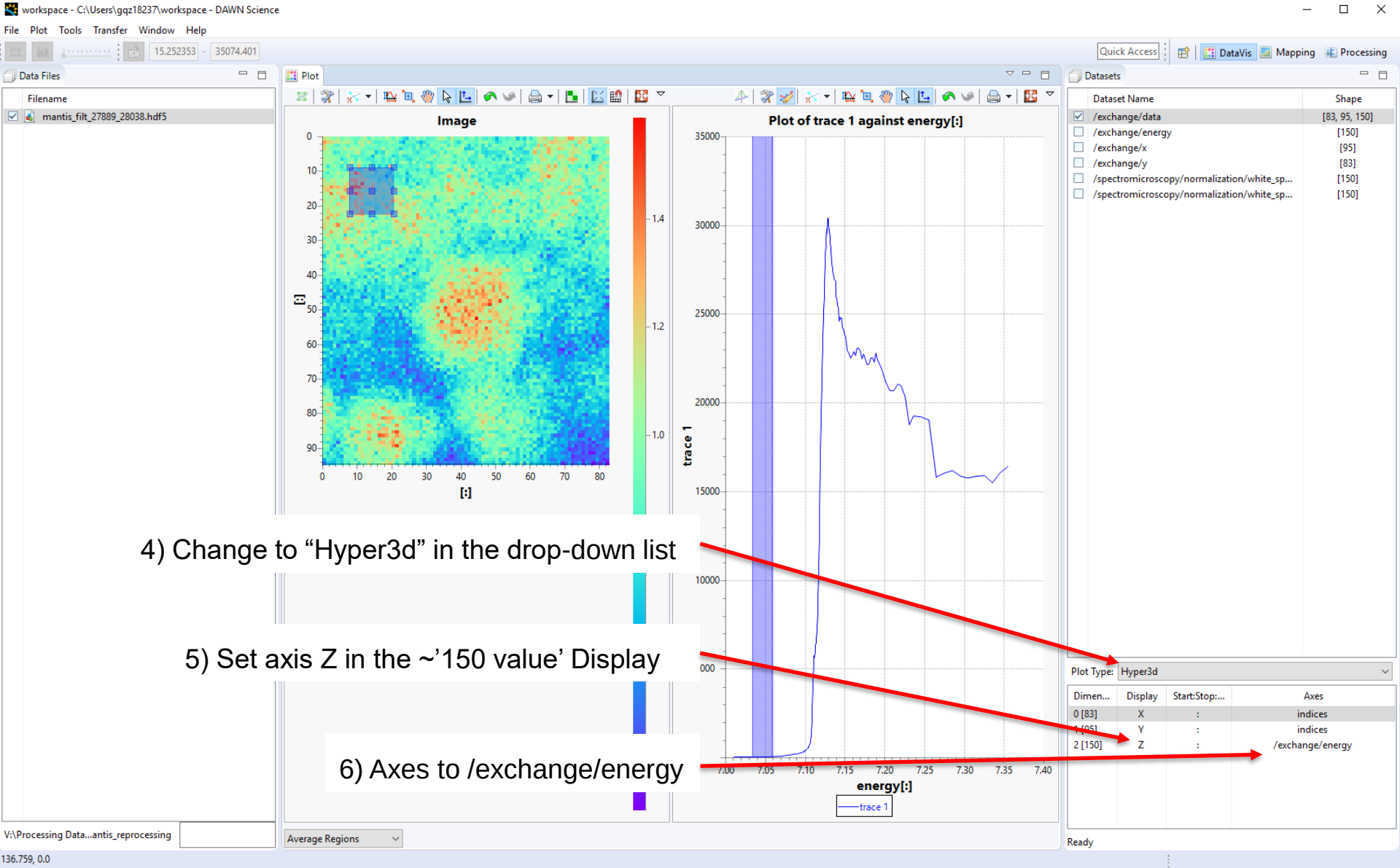
2) Select /exchange/data option. Dawn is a great software for data visualization, but there are several datasets within each scan file which are not selected by default, so they need to be selected every time

Visualisation of the data by DAWN

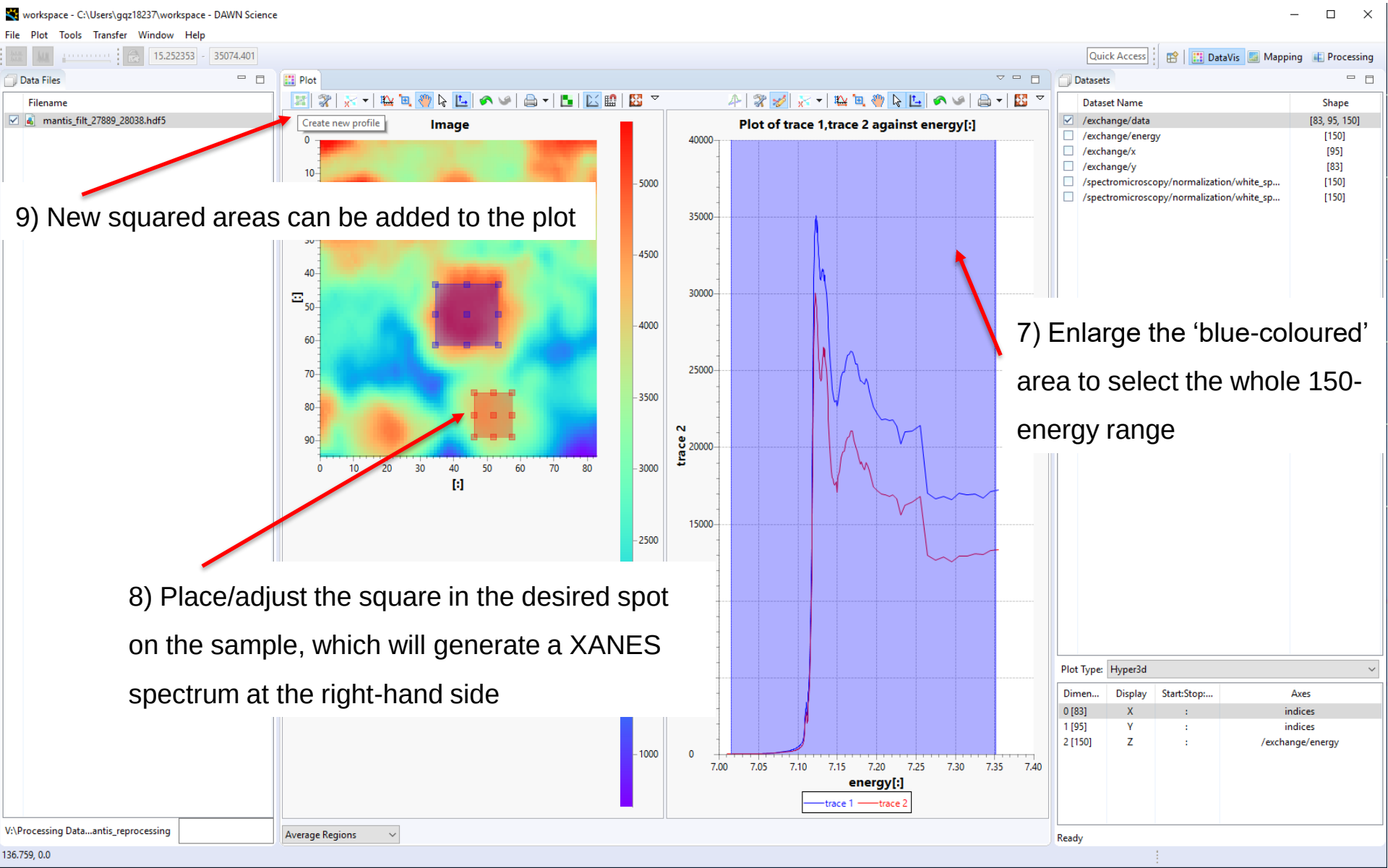


3) Another colour can be chosen for data visualisation

Visualisation of the data by DAWN



Visualisation of the data by DAWN

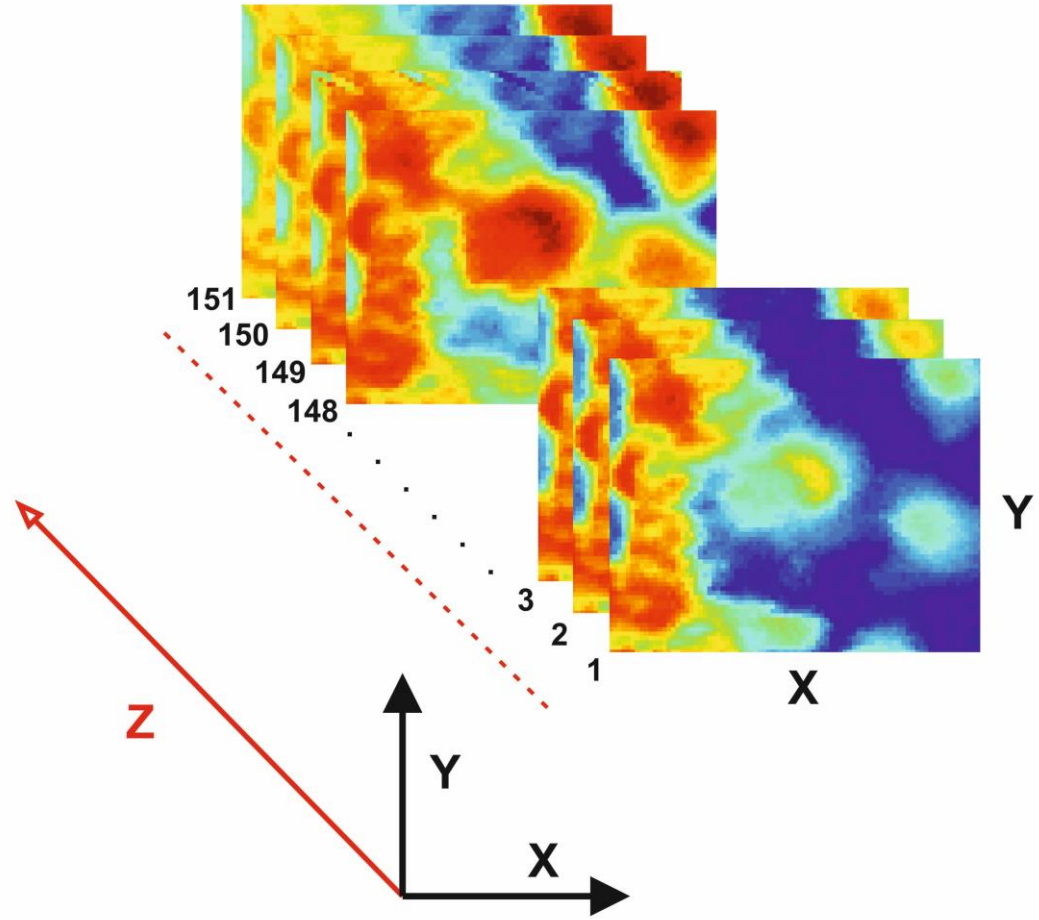


9) New squared areas can be added to the plot

7) Enlarge the ‘blue-coloured’ area to select the whole 150-energy range

8) Place/adjust the square in the desired spot on the sample, which will generate a XANES spectrum at the right-hand side

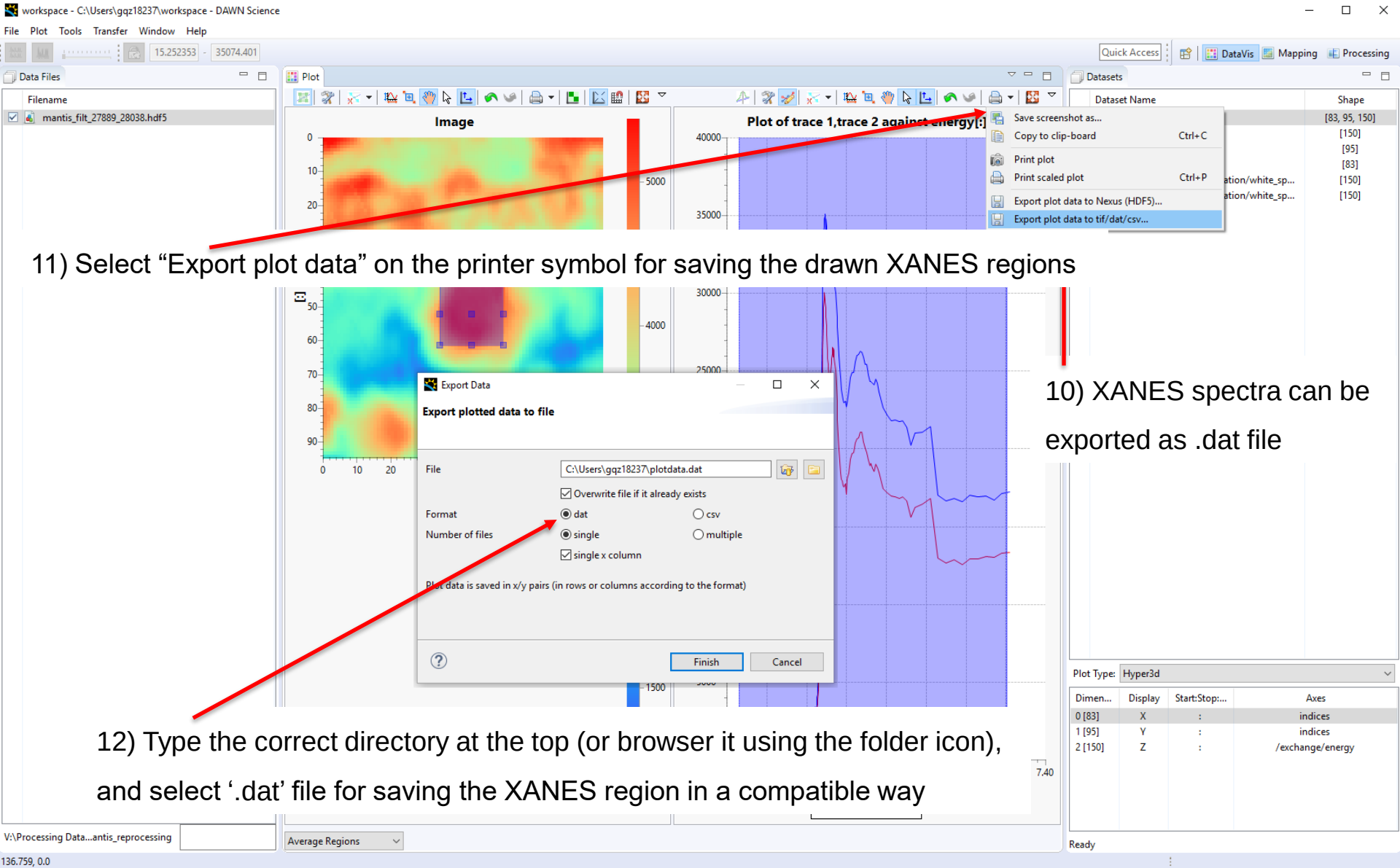
Visualisation of the data by DAWN



~150 energies are being visualised

These 'mantis' files contains the sum of all the 150 energies in the Z-axis, offering an "average" signal

Visualisation of the data by DAWN



11) Select “Export plot data” on the printer symbol for saving the drawn XANES regions

10) XANES spectra can be exported as .dat file

12) Type the correct directory at the top (or browser it using the folder icon), and select ‘.dat’ file for saving the XANES region in a compatible way

MANTiS software – Multivariate analysis

Cross-platform tool developed in Python for spectromicroscopy data analysis.

There is an extensive guide on how to analyse the data in the Github author's webpage (github.com/mlerotic):
https://docs.spectromicroscopy.com/

MantiS

DISCLAIMER: The following slides only describes how to do load and perform a basic data analysis using the files generated at I14, but please, read thoroughly the guide from the above link for a complete understanding on how to get the most of your data.

* Note that this software has its own references to be cited (included in the indicated Github account)

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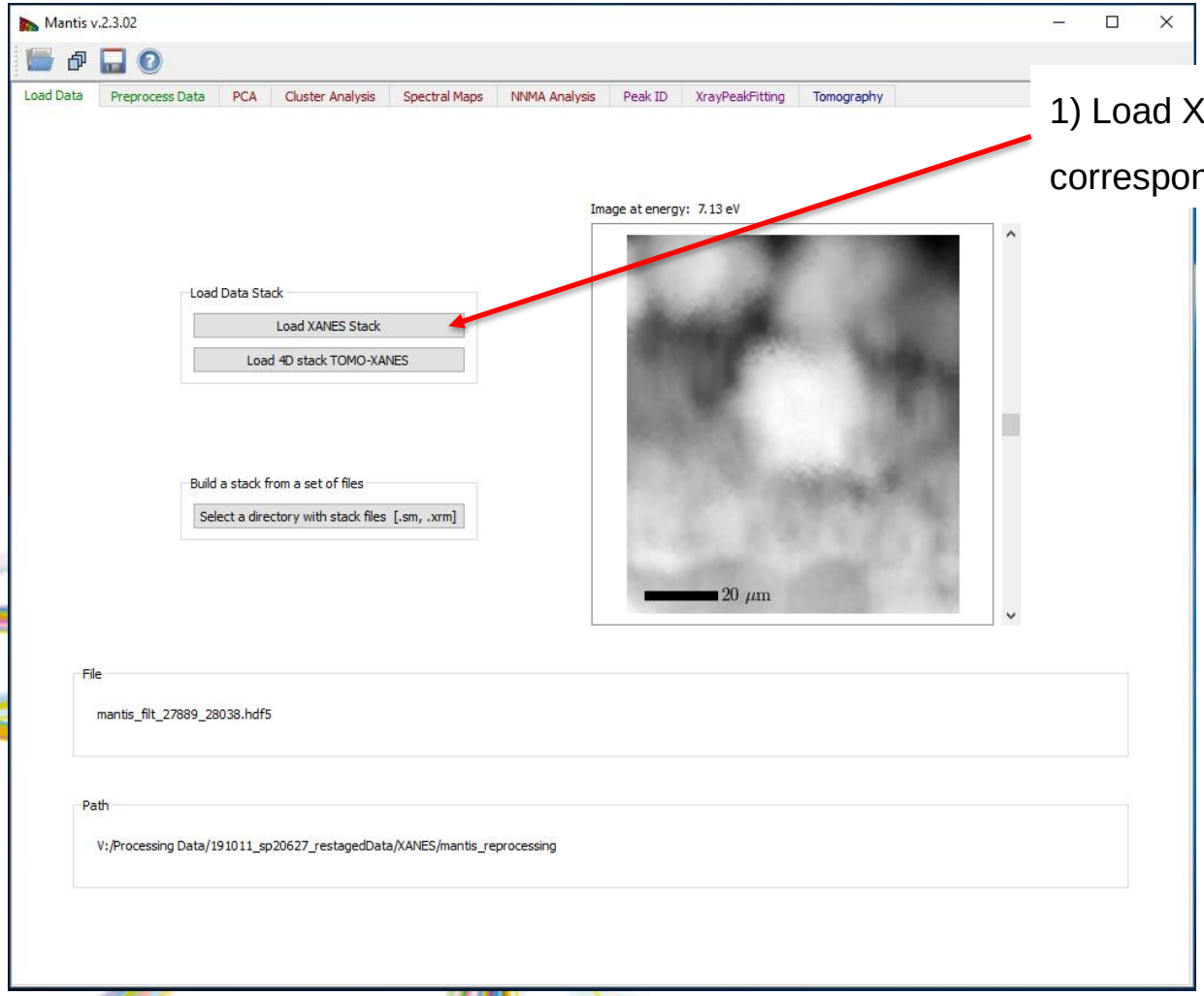
MANTIS – I14 data -- Opening .hdf5 files

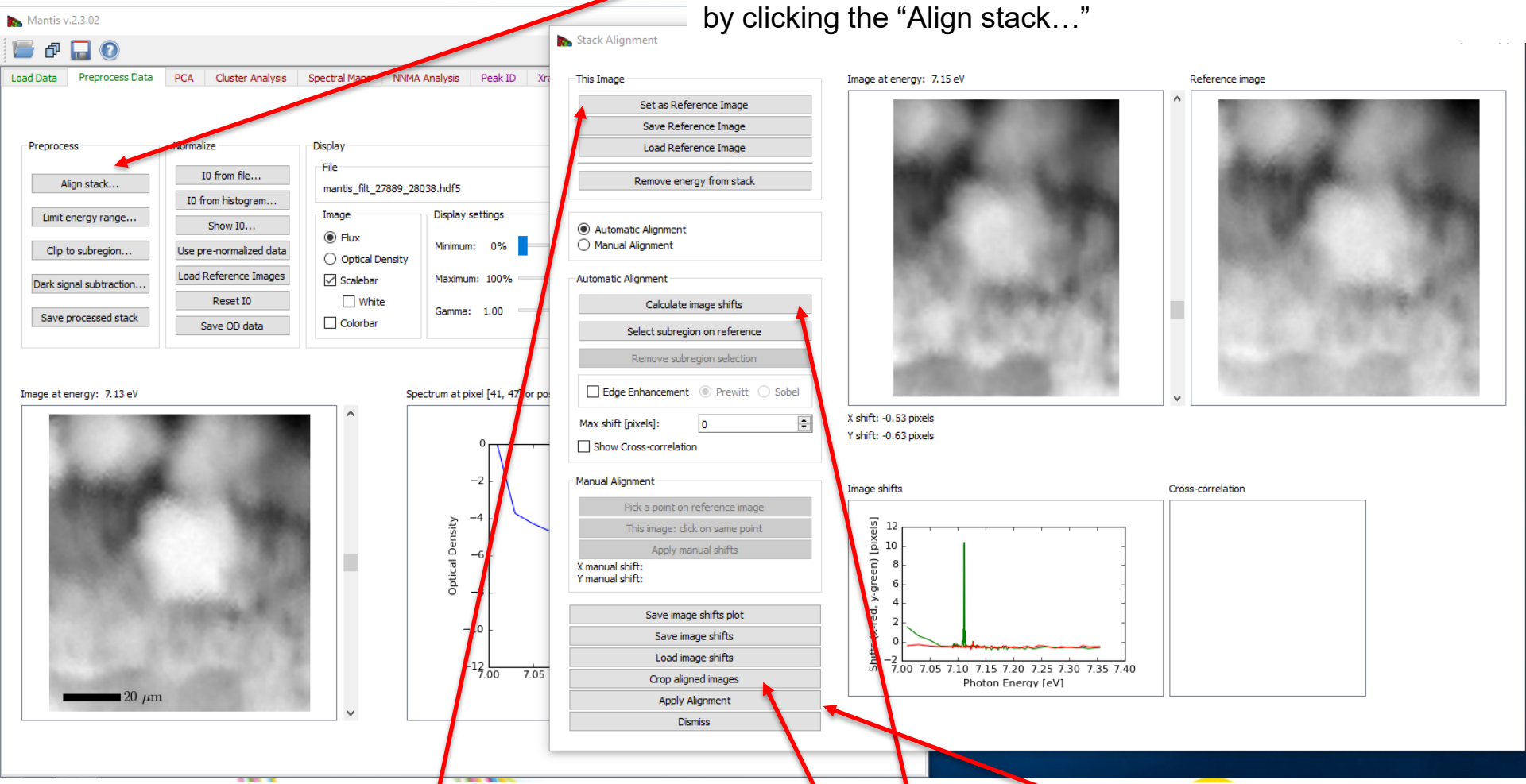
All files are located in: /dls/i14/data/2019/sp20627-1/processing/
example: mantis_filt_27889_28038.hdf5



1) Load XANES Stack – Select the corresponding .hdf5 file

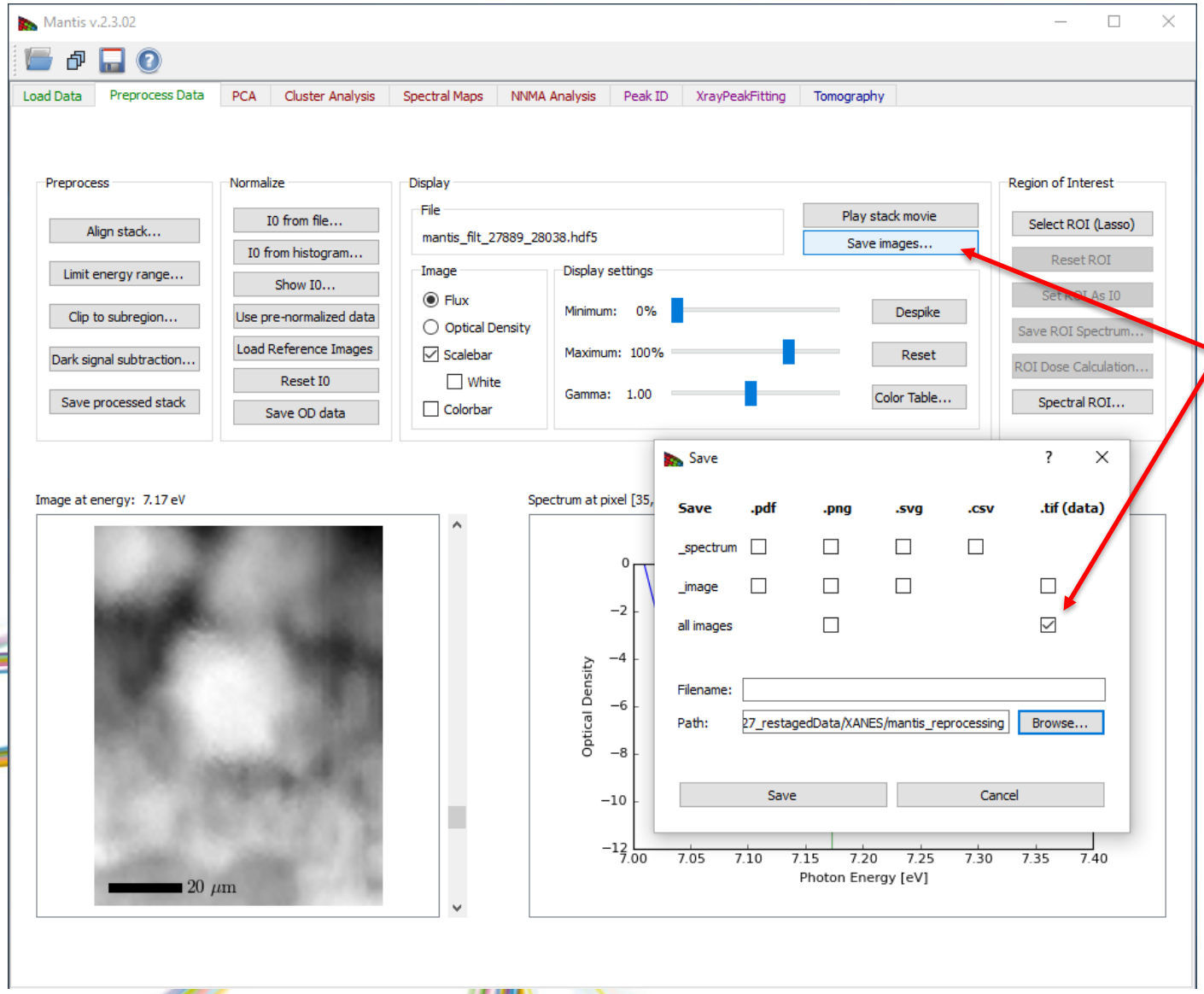
Data processing by MANTIS

2) In the Reprocess tab, data could be re-aligned if required by clicking the “Align stack…”



Later: i) set a nice image as “Reference Image”, ii) click “Calculate image shifts”, and if happy with the result (scroll the vertical bar at the right of the images to visualise): iii) “Crop aligned images” & iv) “Apply Alignment”

Data processing by MANTIS

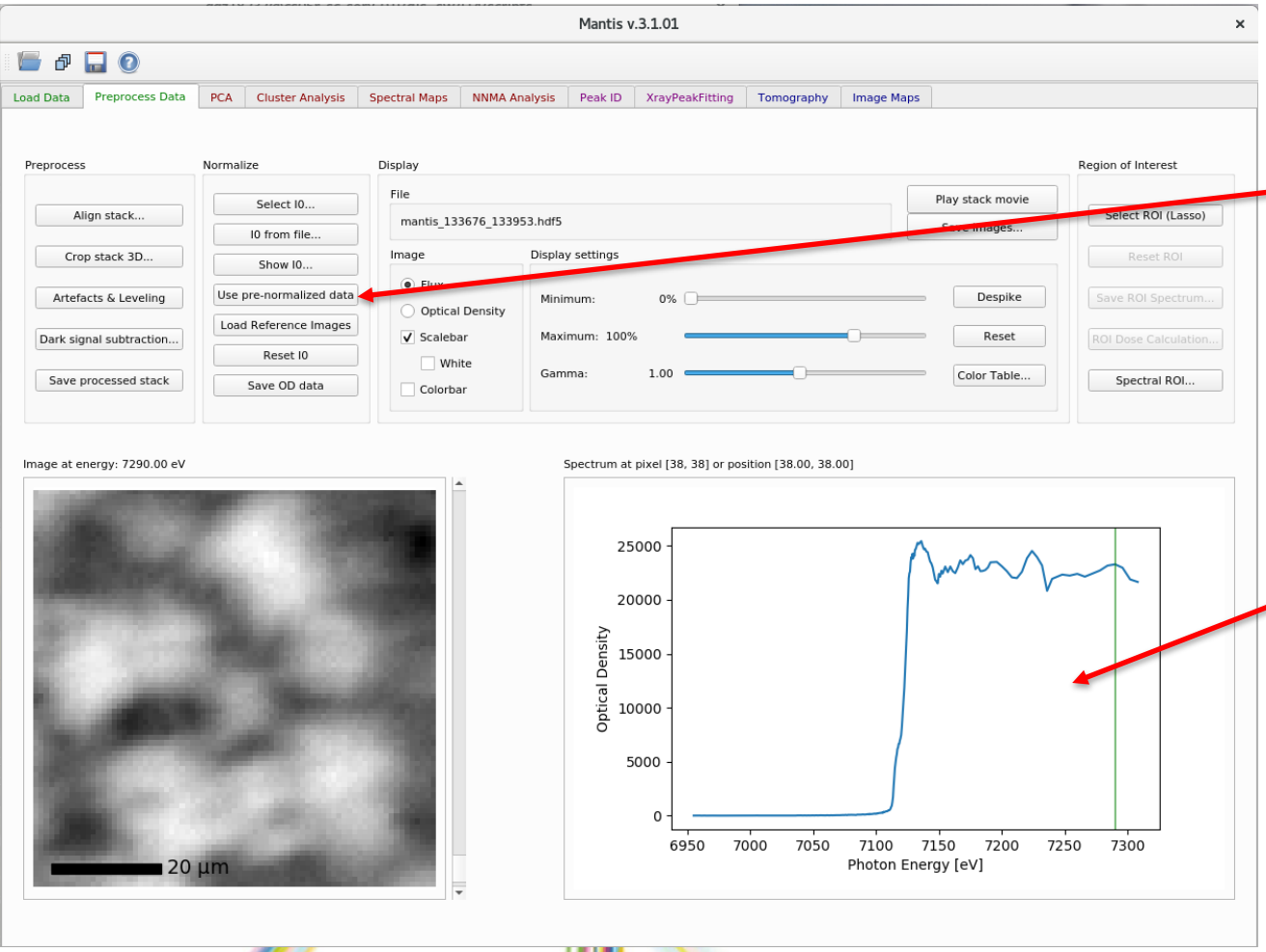


3) The whole aligned image-stack can be saved as individual .tif files (if required)

This allows further “reprocessing” of the individual images by other software's such as Matlab

Data processing by MANTIS

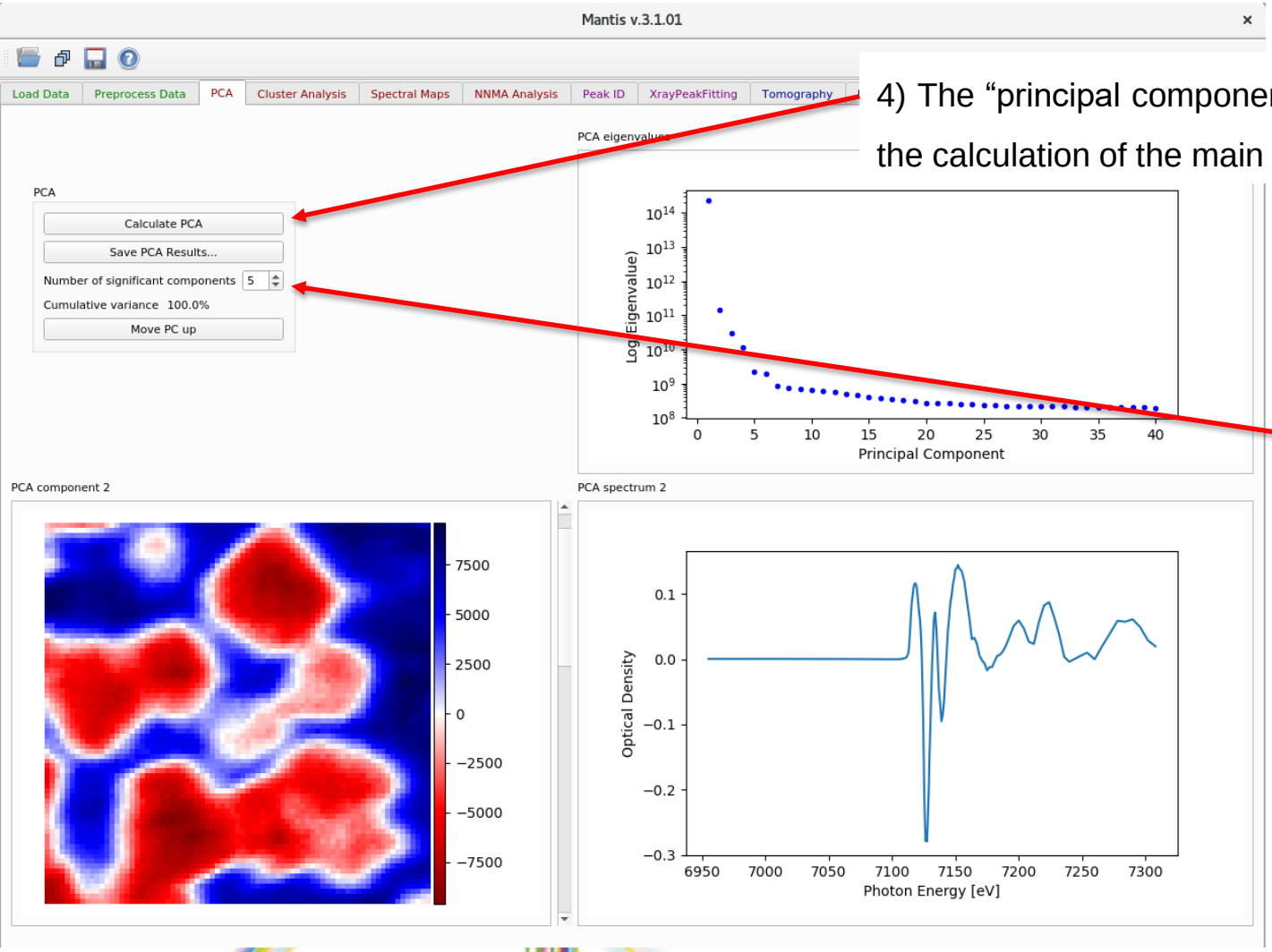
Update for mantis_python-3 version
(available at /dls_sw/i14/scripts/)



If the file to load has been pre-normalised (and aligned) by i14 jupyter notebooks, please, remember to select: “Use pre-normalized data”

A non good-looking XANES spectrum in the pre-visualisation at the bottom-right, is also an indicative of not having selected this option.

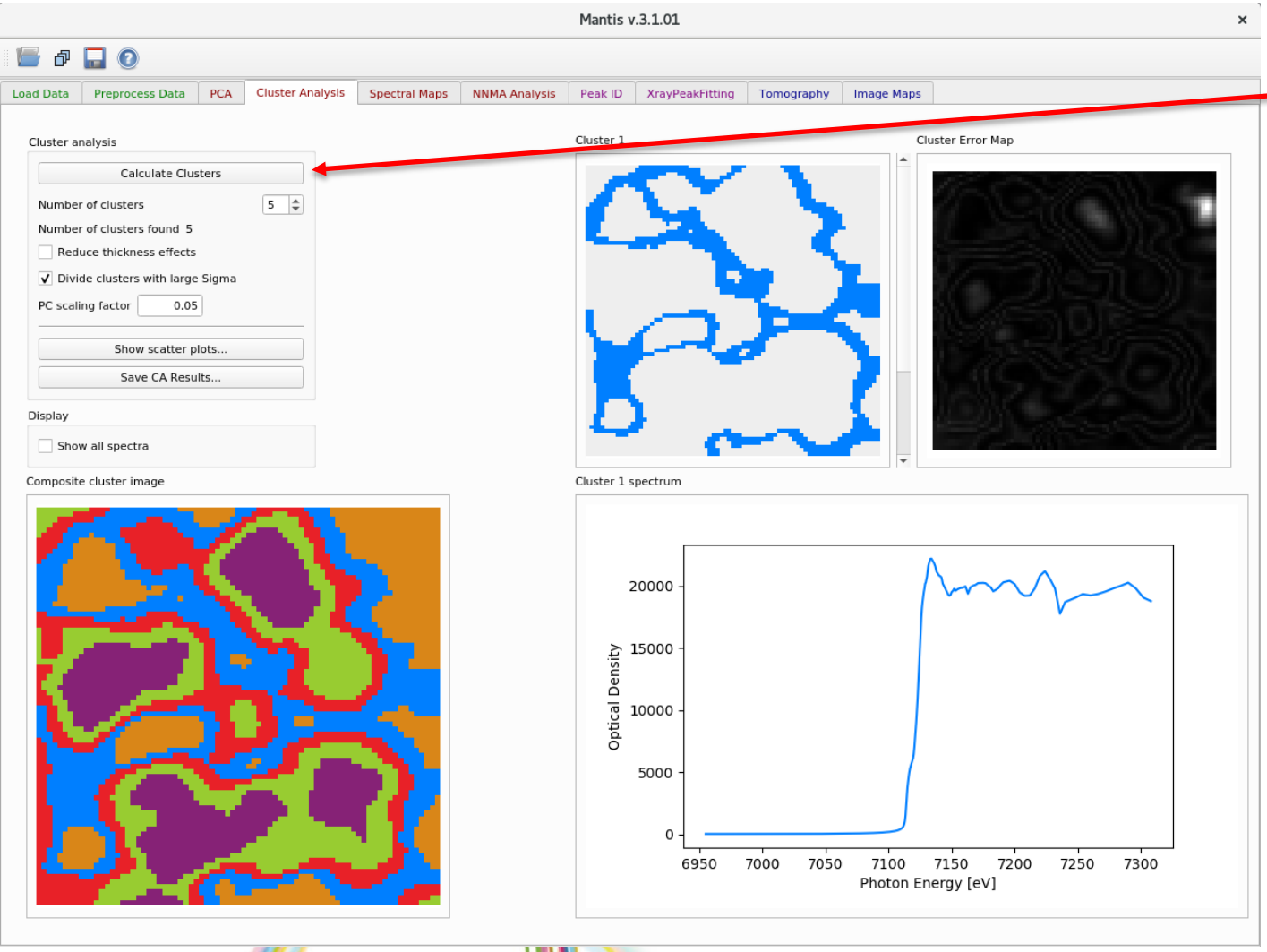
Data processing by MANTIS



4) The “principal component analysis” (PCA) tab allows the calculation of the main components of the sample

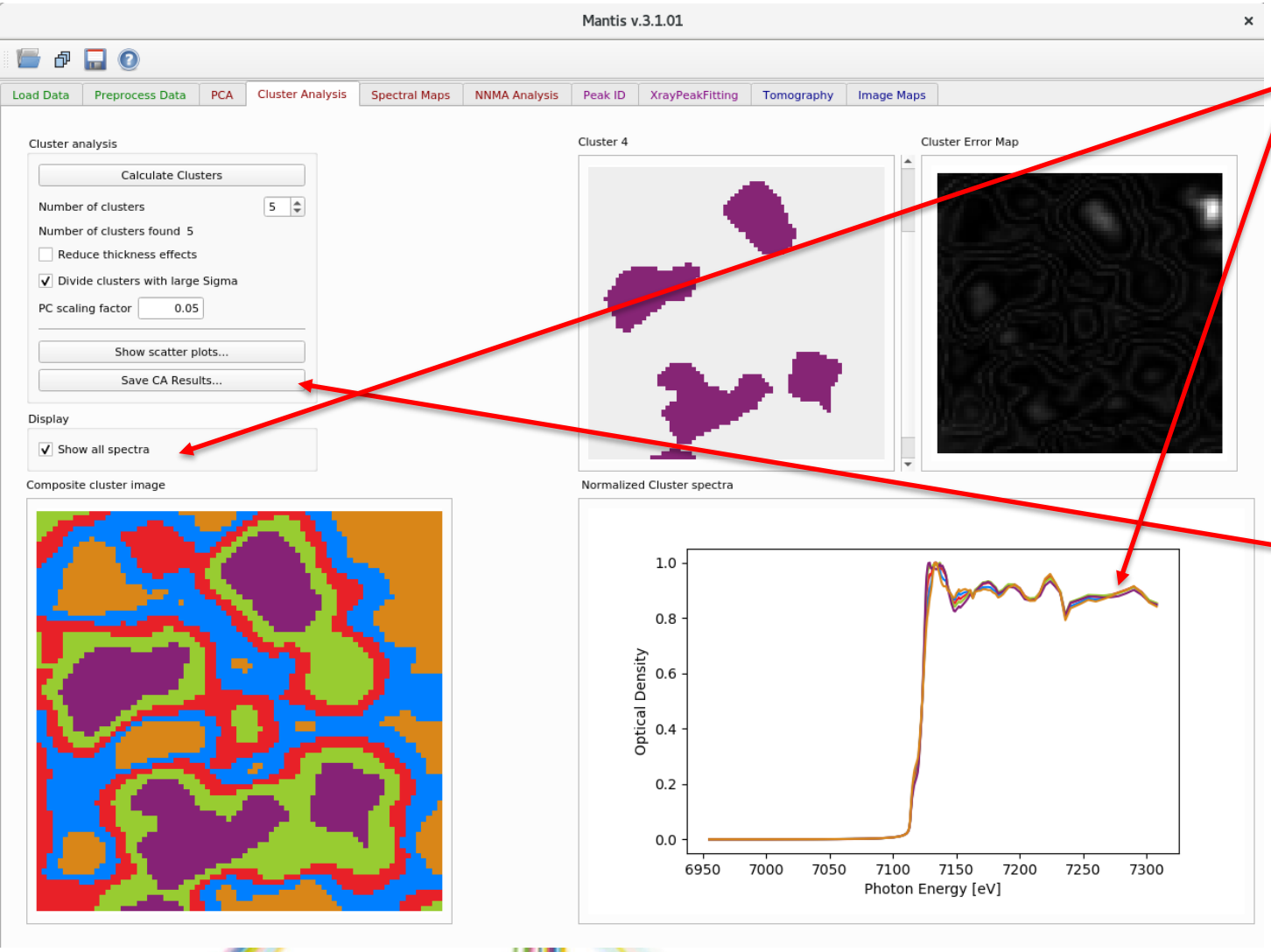
5) The number of significant components may be manually modified, and the PCA recalculated again (see Github guide for more information)

Data processing by MANTIS



6) The cluster analysis calculation (feeding the corresponding number) combines the regions with similar X-ray absorption spectra (XAS) features

Data processing by MANTIS



7) All clusters' spectra can be simultaneously displayed by selecting this option in the right-side of the window

8) The spectra arising from cluster analysis calculations can be saved as .dat/.csv file for further XAS reprocessing

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Athena – From Demeter software

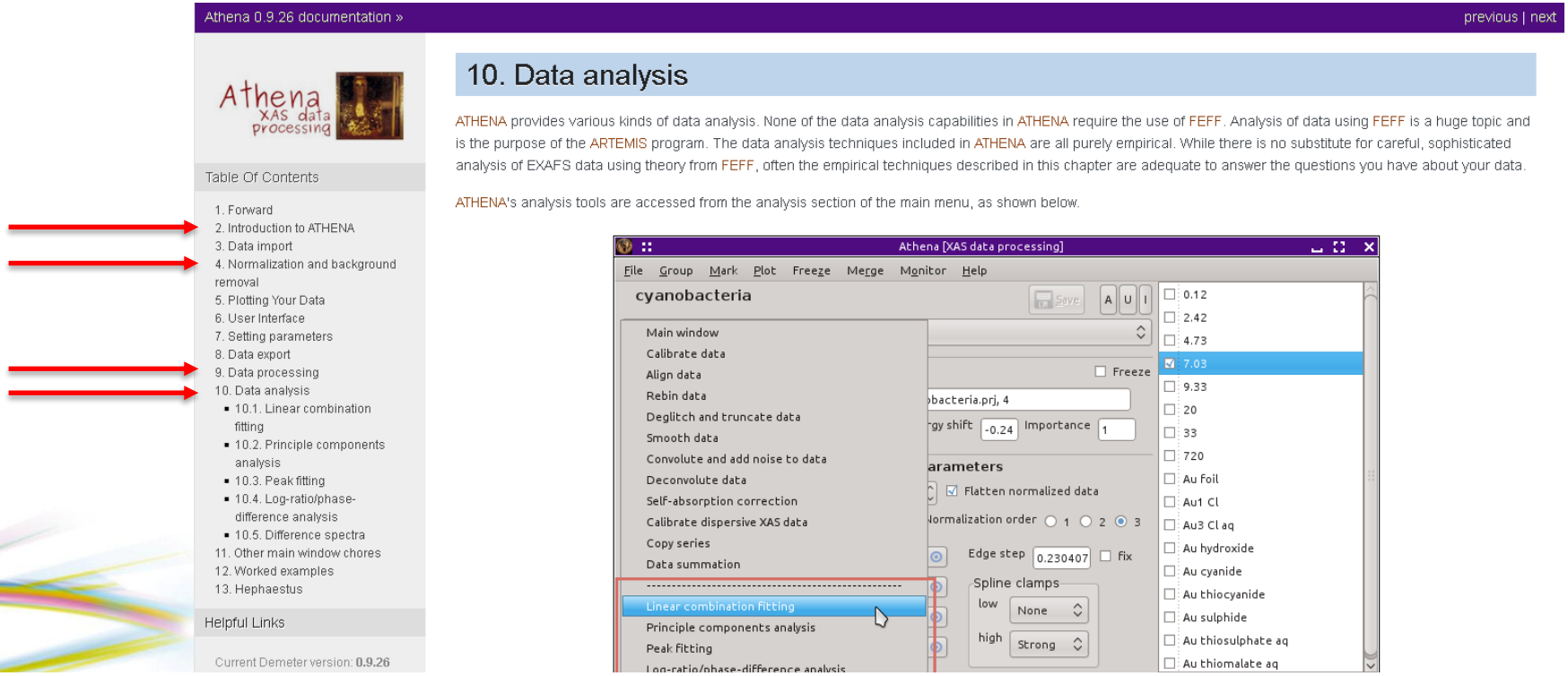
Athena is a great software to perform linear combination fitting analysis using the ".dat" or ".txt" files already generated by DAWN/MANTiS from your regions of interest (check pages 10 and 18 from this guide).

There are videos from Bruce Ravel (software owner and dveeloper) on how to analyse XAS data from a workshop organised at Diamond:
https://vimeo.com/340202552

Alternatively, there are some more tutorial and examples on the website:
https://xafs.xrayabsorption.org/tutorials.html

Athena -- Getting started

- *A complete step-by-step guideline can be found on-line:*
  http://bruceravel.github.io/demeter/documents/Athena/index.html (accessed on 26/11/2019)

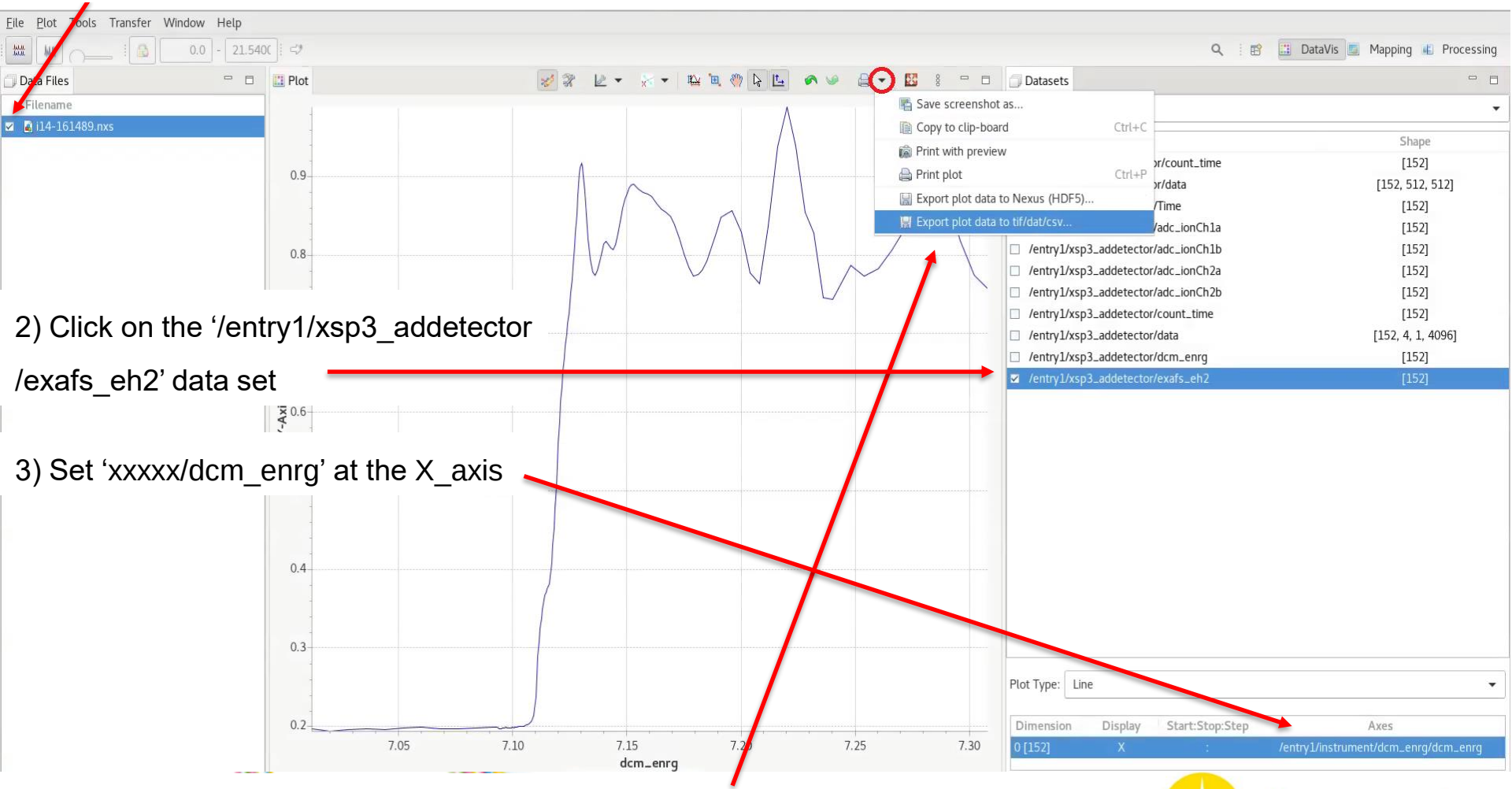


- *For understanding the theory behind the XAS calculations, there are many papers and books available, but I recommend:*

Kelly, S. D.; Hesterberg, D.; Ravel, B. Analysis of Soils and Minerals Using X-Ray Absorption Spectroscopy. Ulery AL, Dress R, Eds. Methods soil Anal. Part 5. Mineral. methods. Madison, WI Soil Sci Soc Am 2008, 387–464

Using DAWN to export the calibration foil for Athena

1) Select the nexus file (it is not selected by default after loading the data)



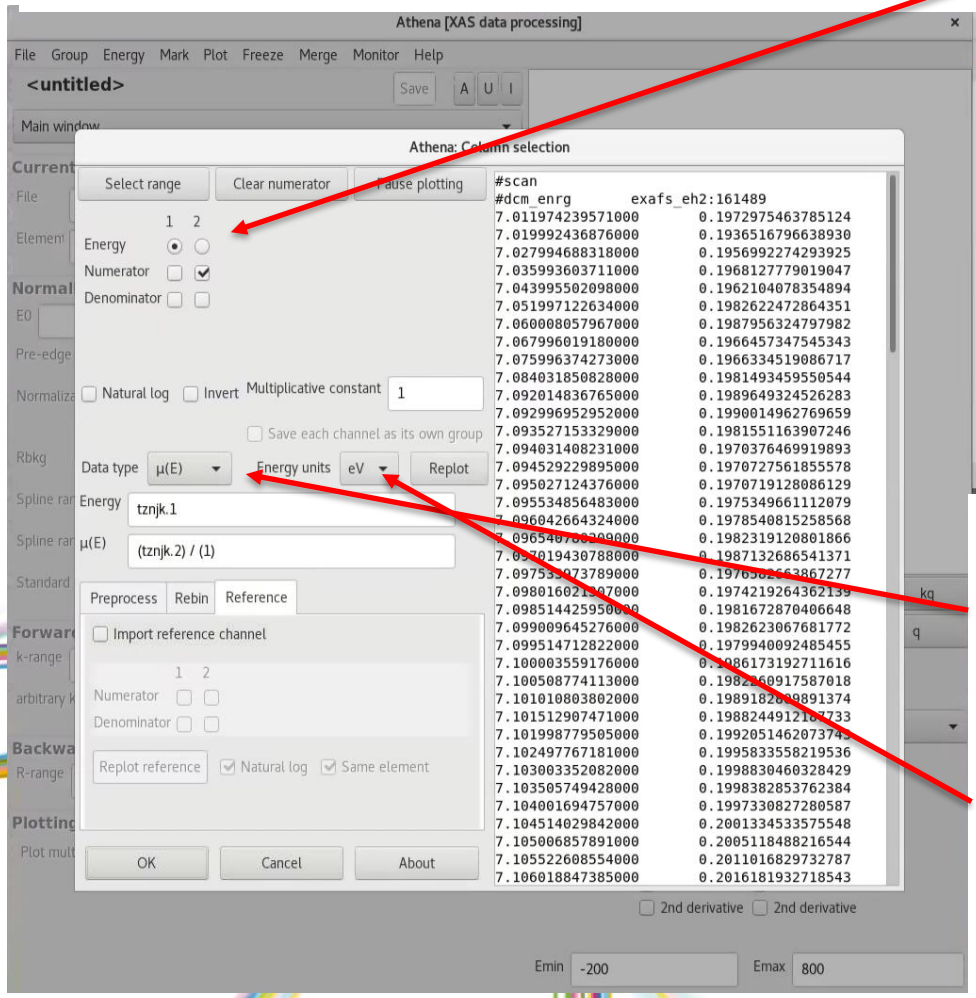
2) Click on the '/entry1/xsp3_addetector /exafs_eh2' data set

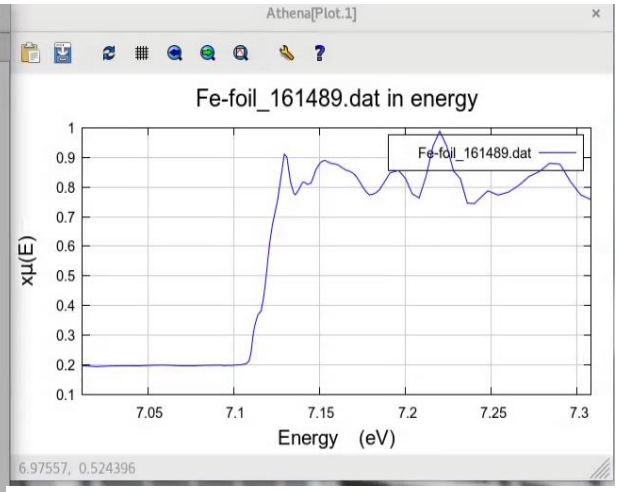
3) Set 'xxxxx/dcm_enrg' at the X_axis

4) On the arrow icon, select 'Export plot data to tif/dat/csv' and save the xanes spectrum as .dat

Load .dat files in Athena

5) The .dat file of the metal foil has to be loaded. Then select the “Energy” as column 1 and the “Numerator” as column 2





6) Select data type as ‘μ(E)’ for the EXAFS/XANES scans, or ‘xanes’ for visualising the near-edge only

7) The energy units should be changed accordingly, either as eV or keV

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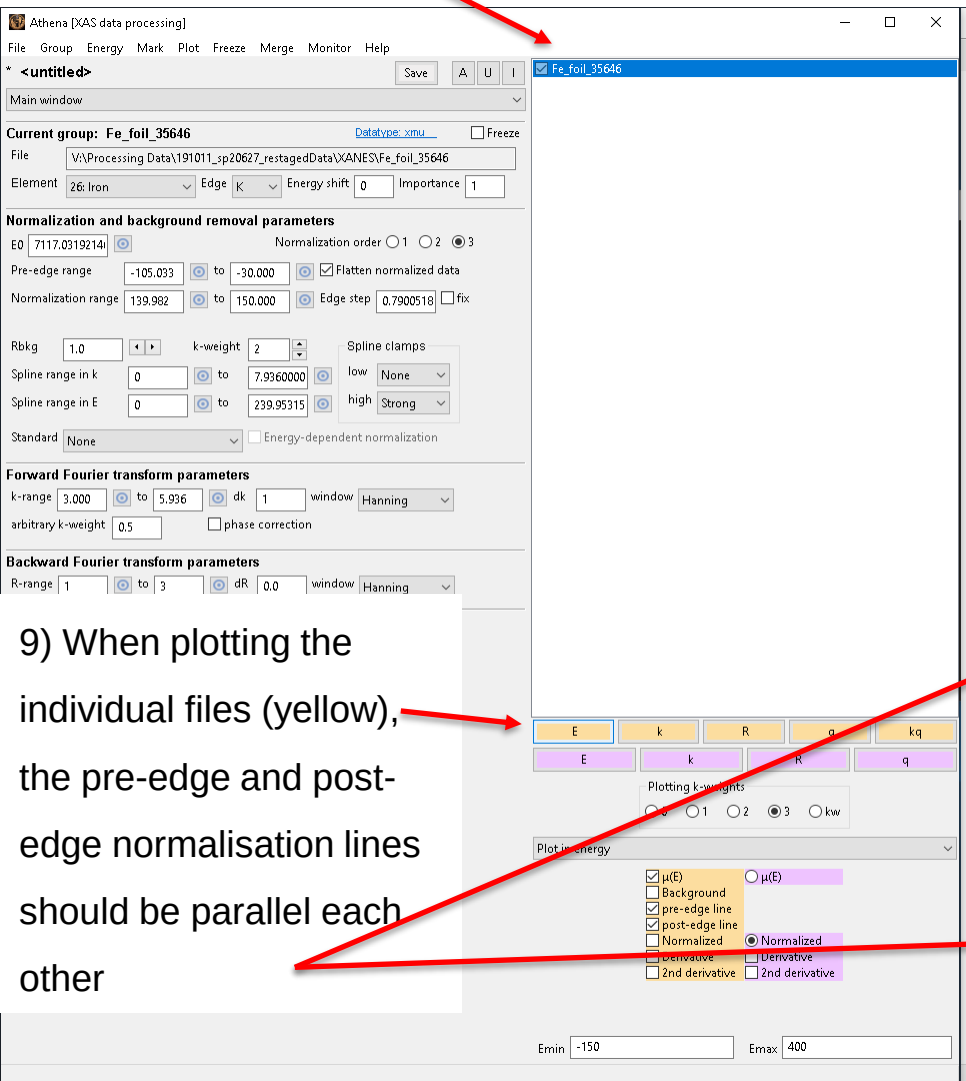
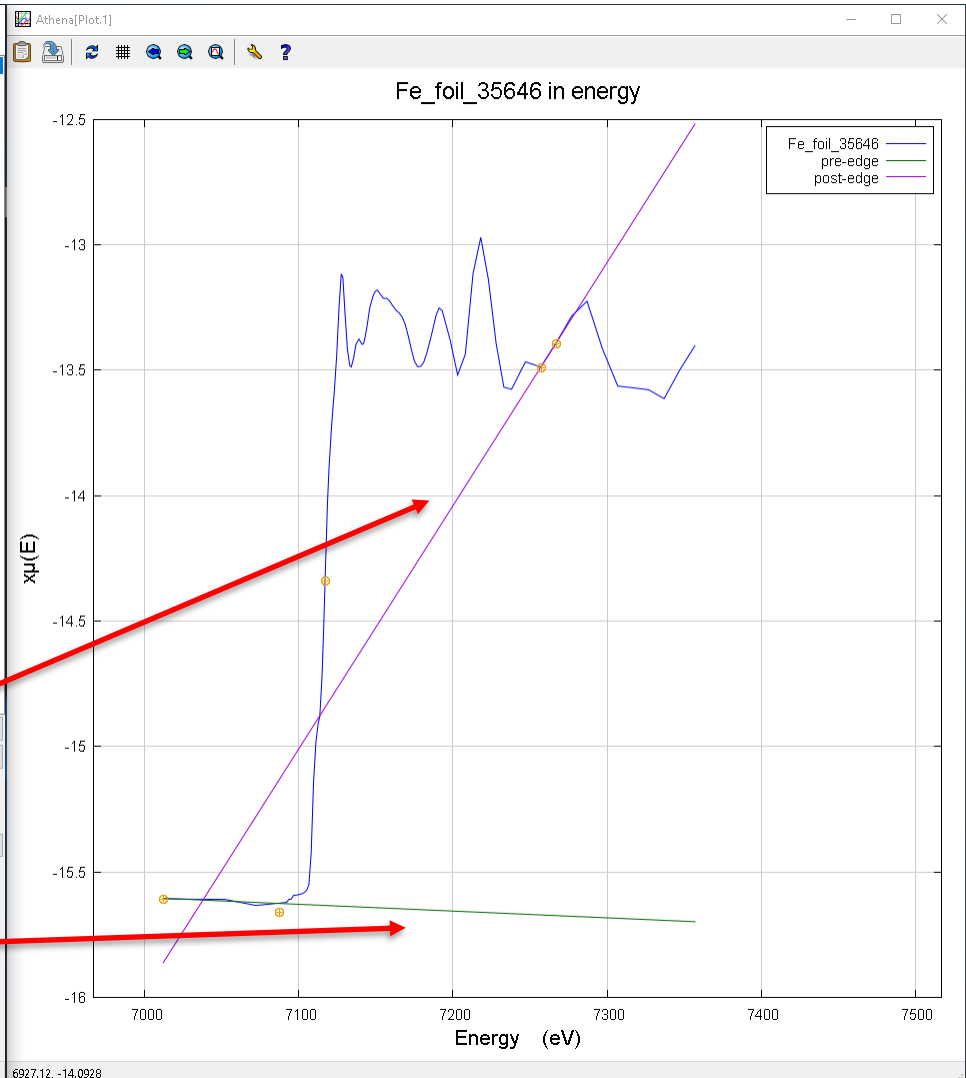
Data analysis by Athena – Calculating the E shift

8) A calibration foil (or standard) is required to calculate the Energy shift for every experiment

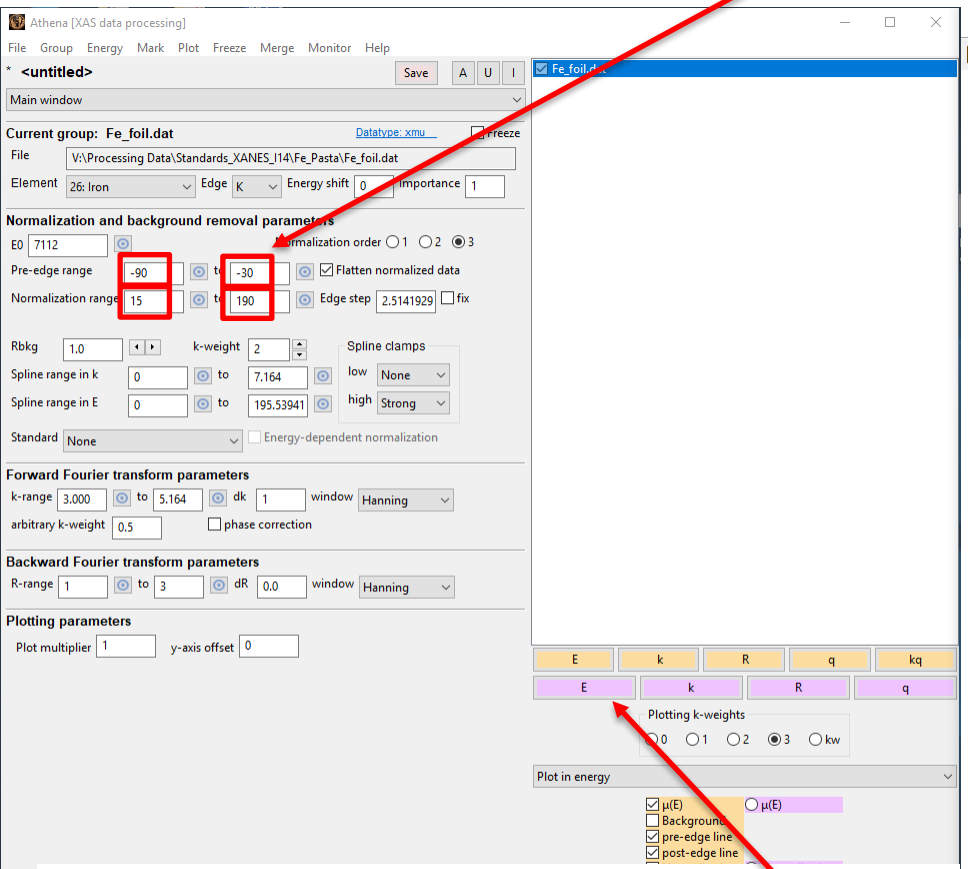


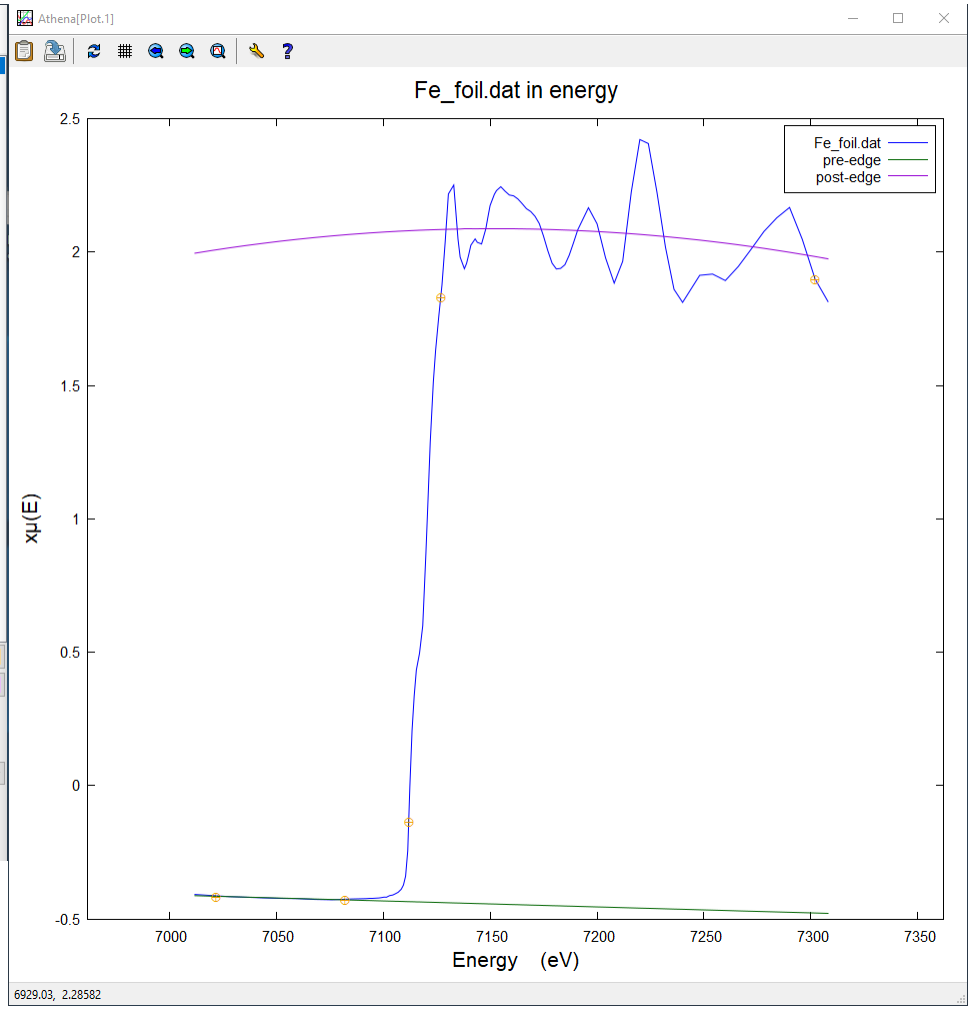
9) When plotting the individual files (yellow), the pre-edge and post-edge normalisation lines should be parallel each other



Data analysis by Athena

10) “Pre-edge” and “Normalisation” ranges can be modified manually, for getting the green and violet lines parallel

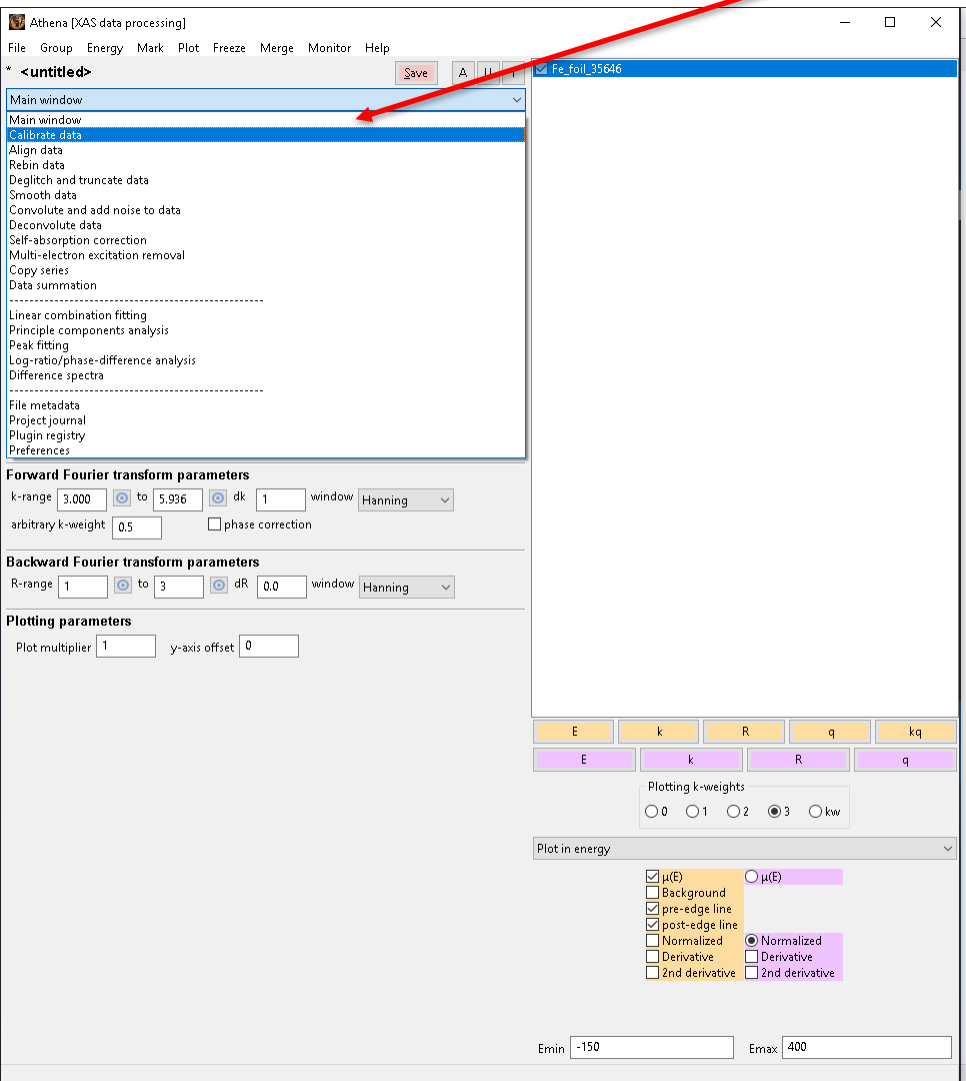


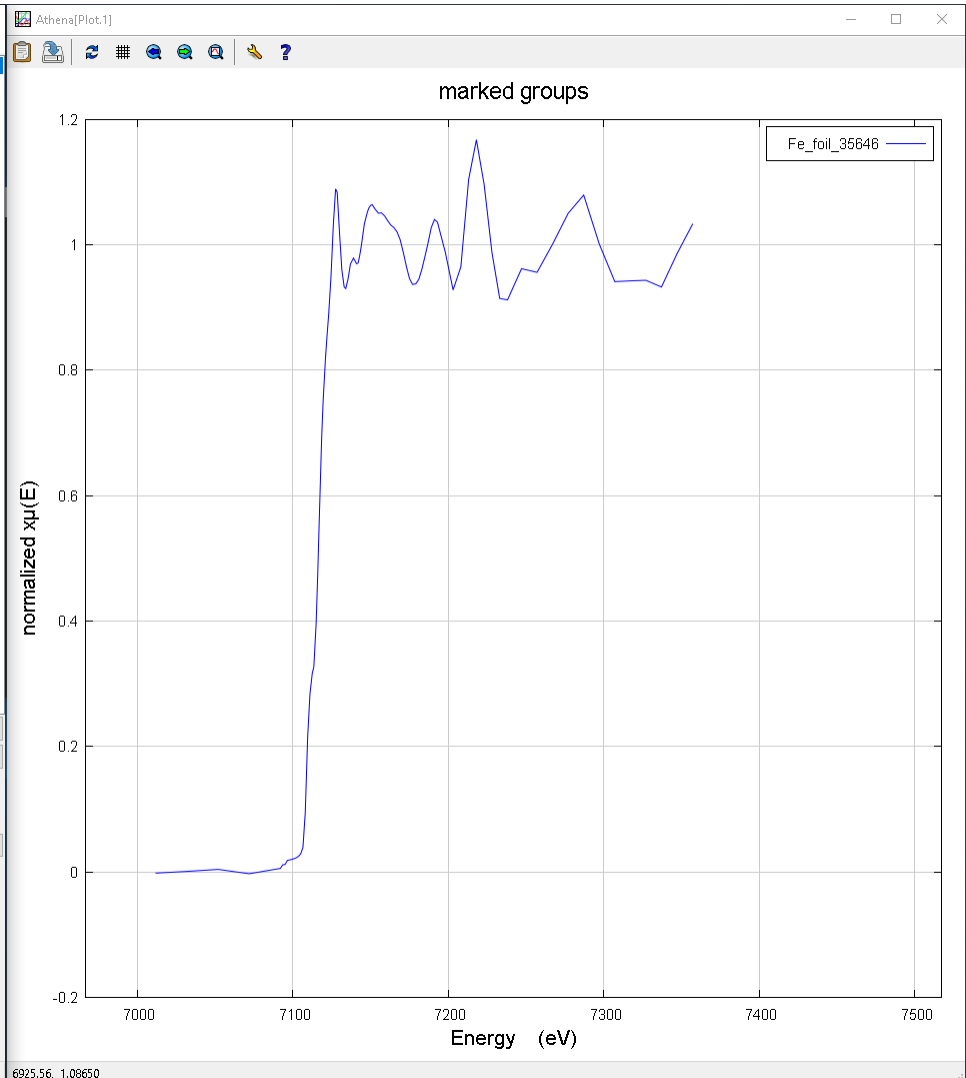


11) Using subsequently with the norm E button (violet) will display the normalised data at the right

Data analysis by Athena

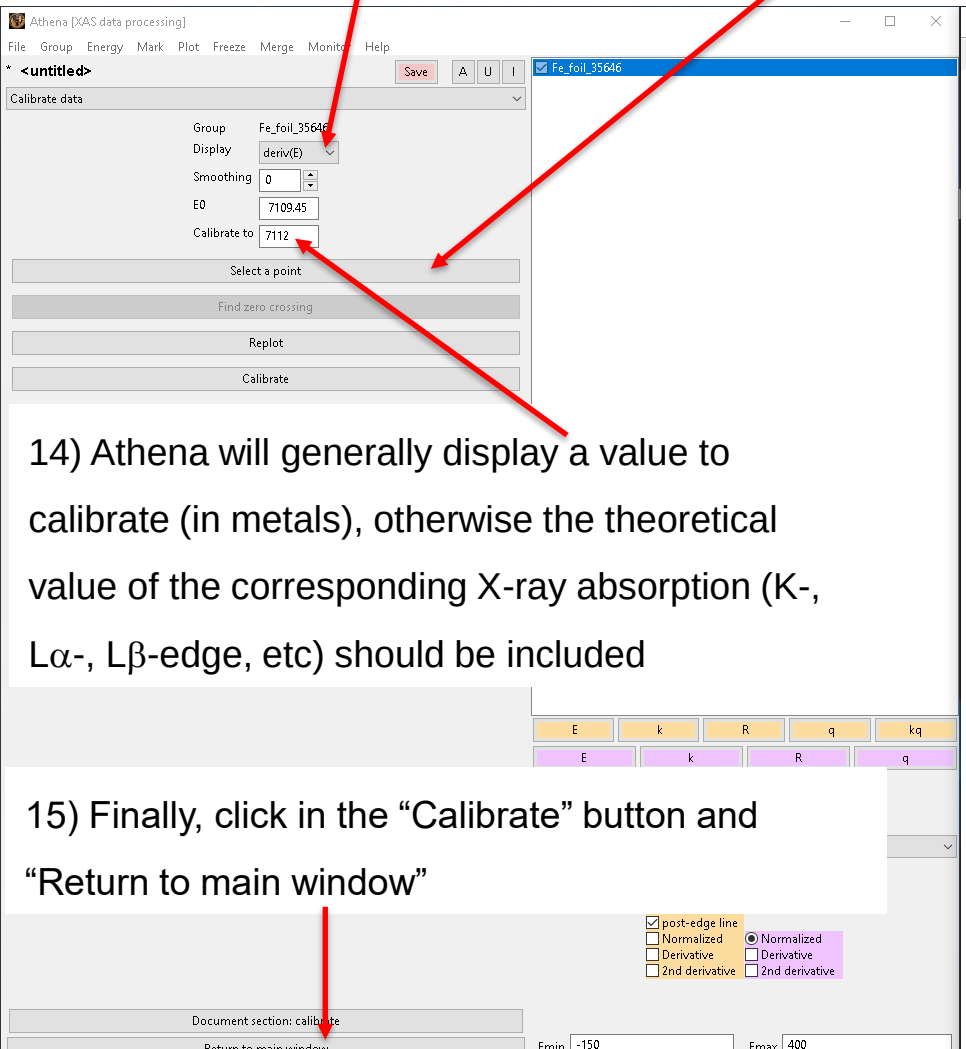
12) Using the scroll-down main window menu, different calculations can be made, including the data calibration





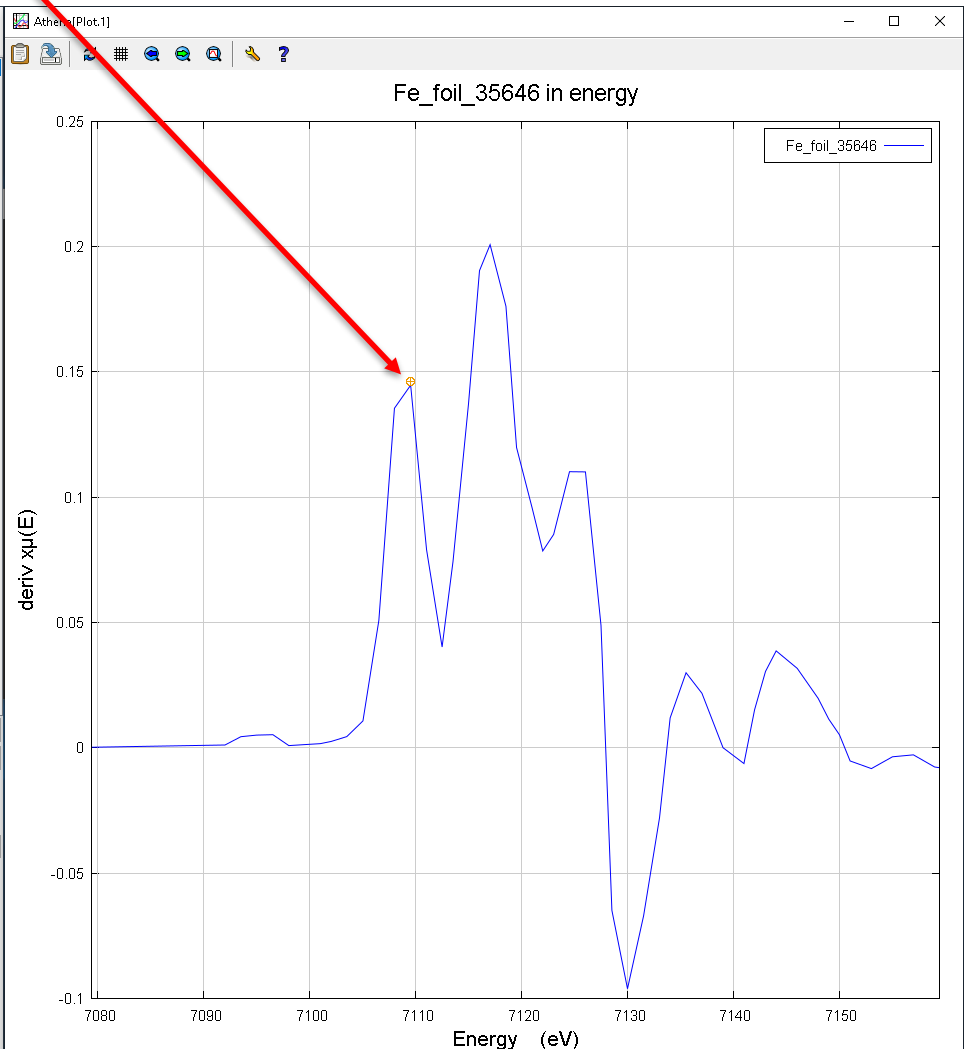
Data analysis by Athena

13) Displaying the deriv(E) representation, click “Select a point” and actually double-click the first-derivate position in the plot at the right, which will remain marked as orange



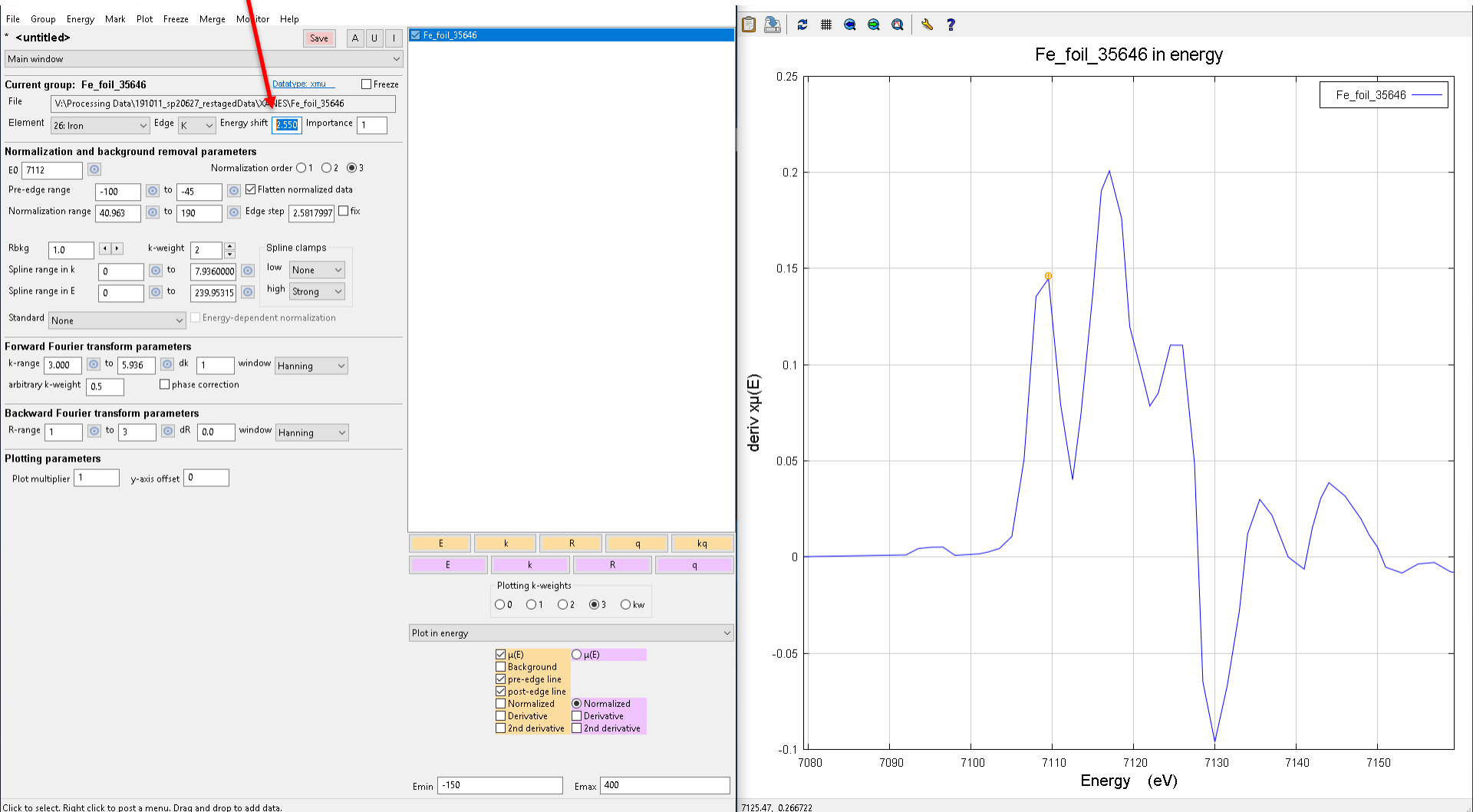
14) Athena will generally display a value to calibrate (in metals), otherwise the theoretical value of the corresponding X-ray absorption (K-, Lα-, Lβ-edge, etc) should be included

15) Finally, click in the “Calibrate” button and “Return to main window”



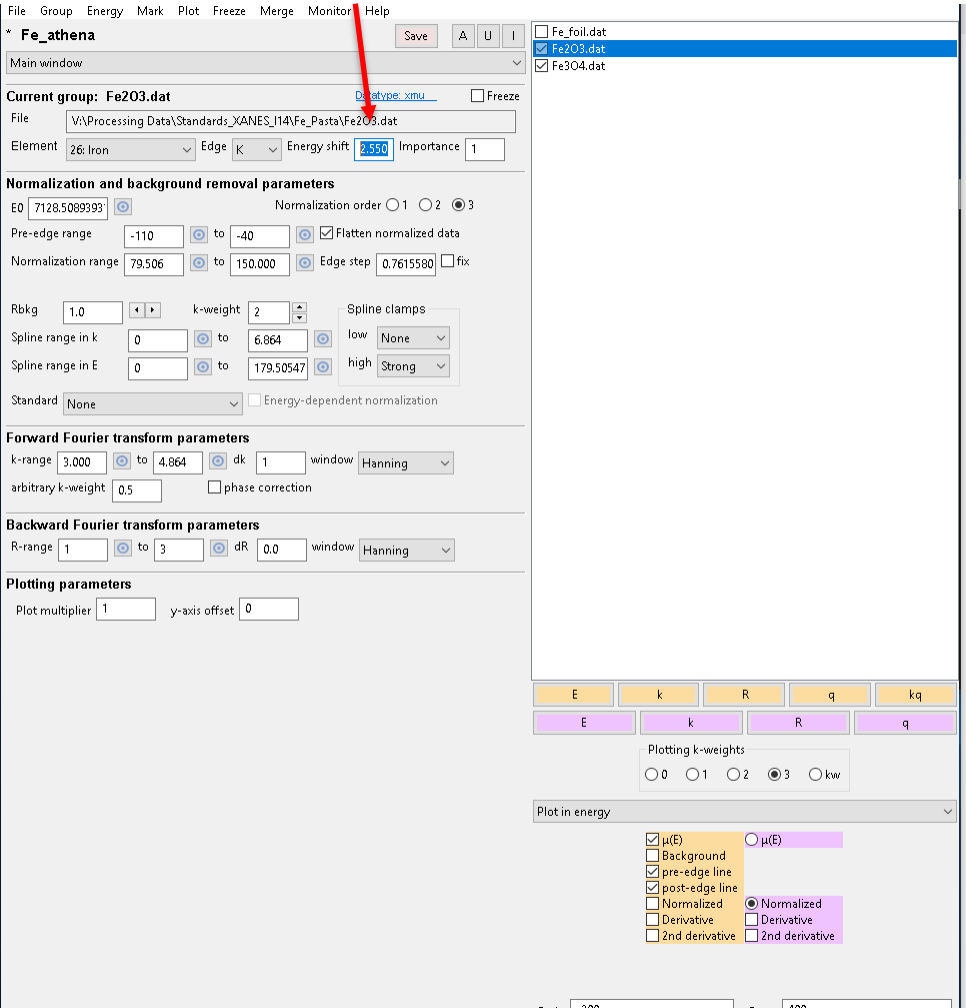
Data analysis by Athena

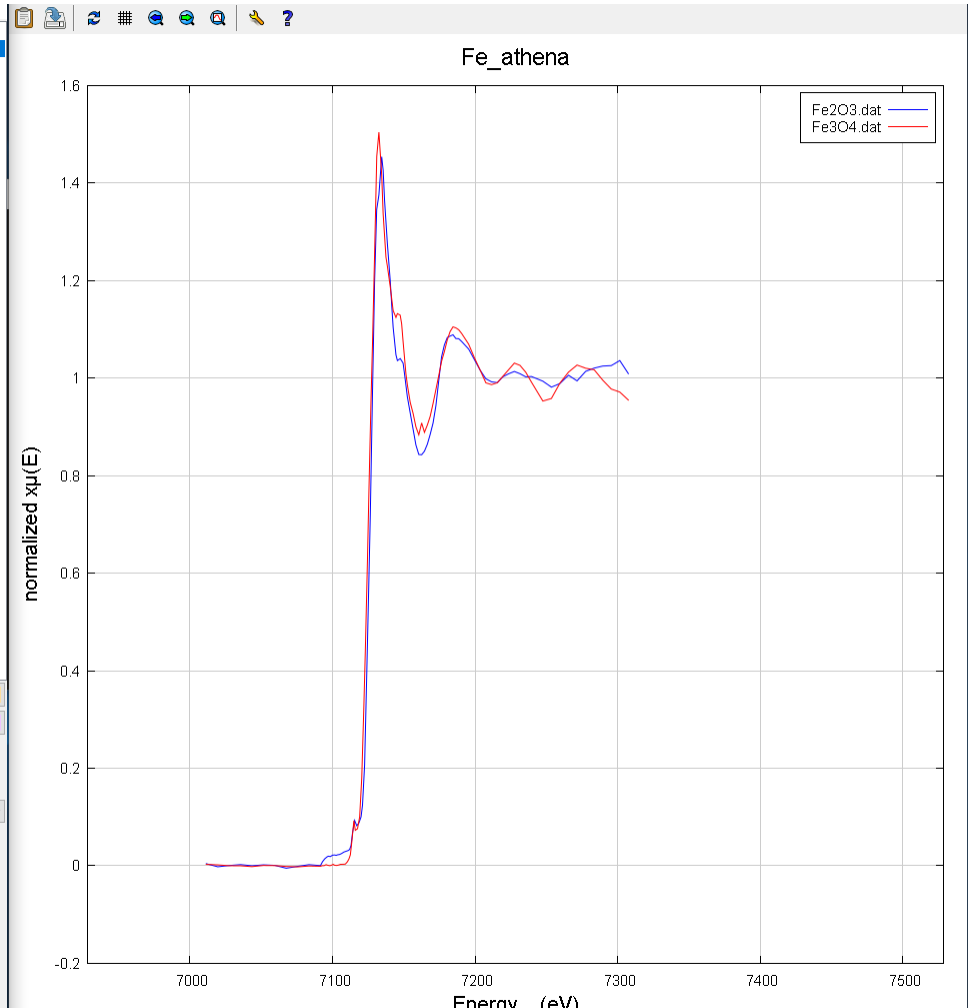
16) Copy the energy shift calculated by this methodology. This is the instrumental “off-set” to be applied to all the acquired datasets



Data analysis by Athena

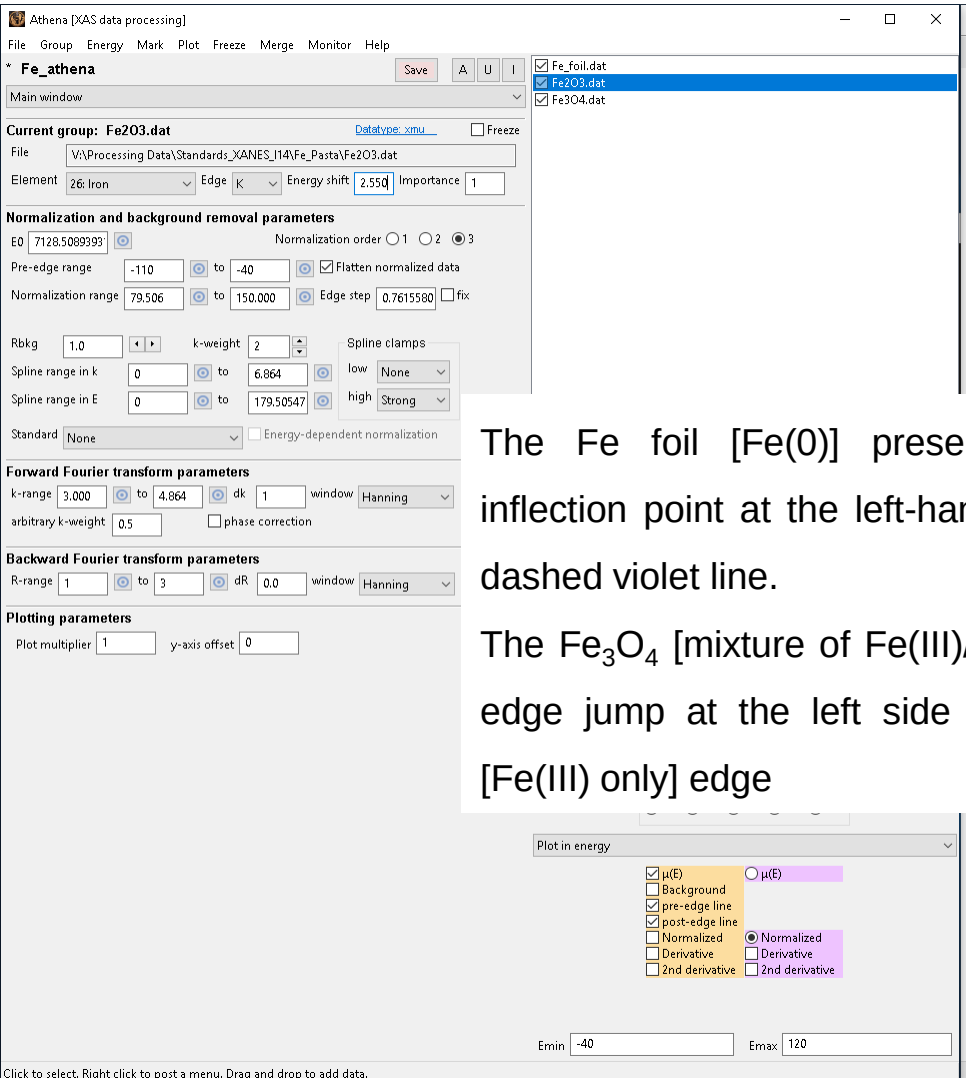
17) If you want to compare standards and samples acquired in different beamtimes, this off-set value has to be updated to ALL standards and samples analysed at the same beamtime. If you are only using data from one experimental session, all your samples and standards will already present the same energy off-set

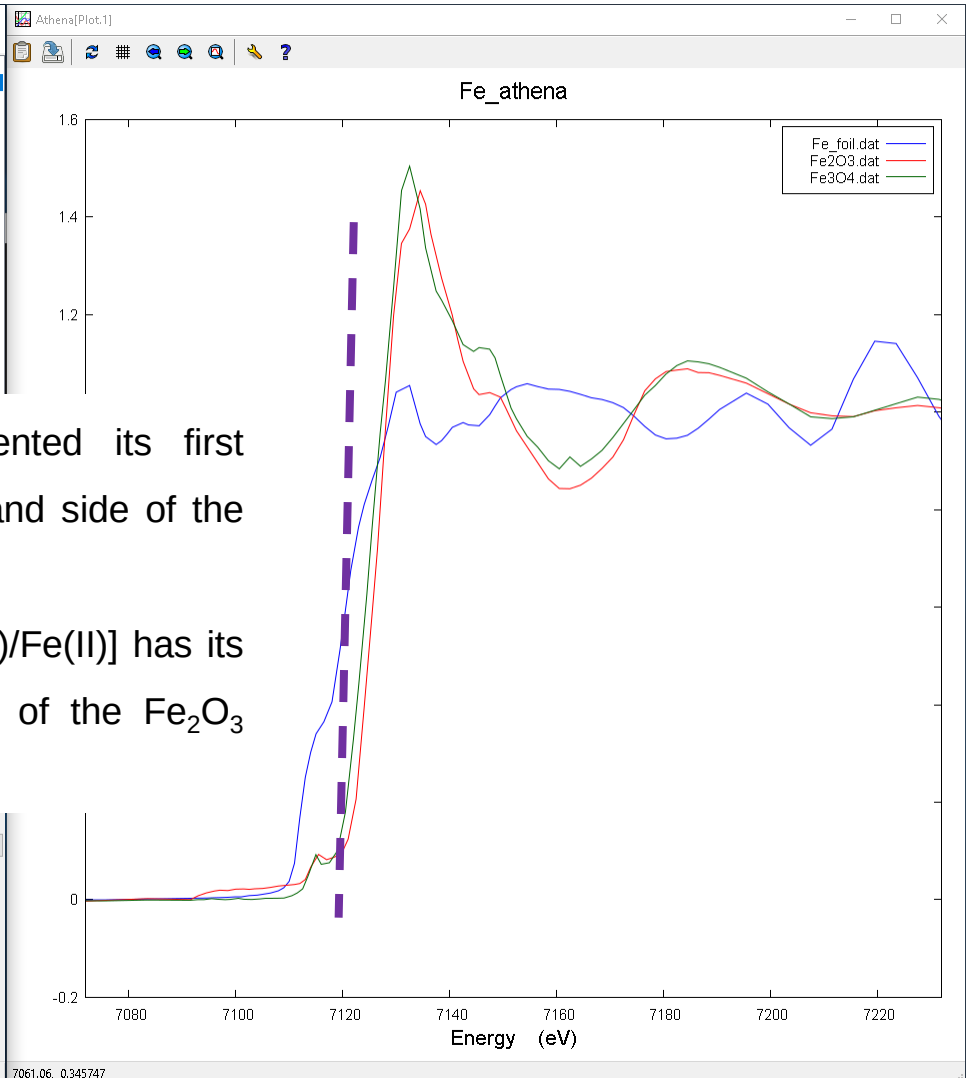




Data analysis by Athena

18) Once the calibration is done, you can evaluate the oxidation state of the different samples



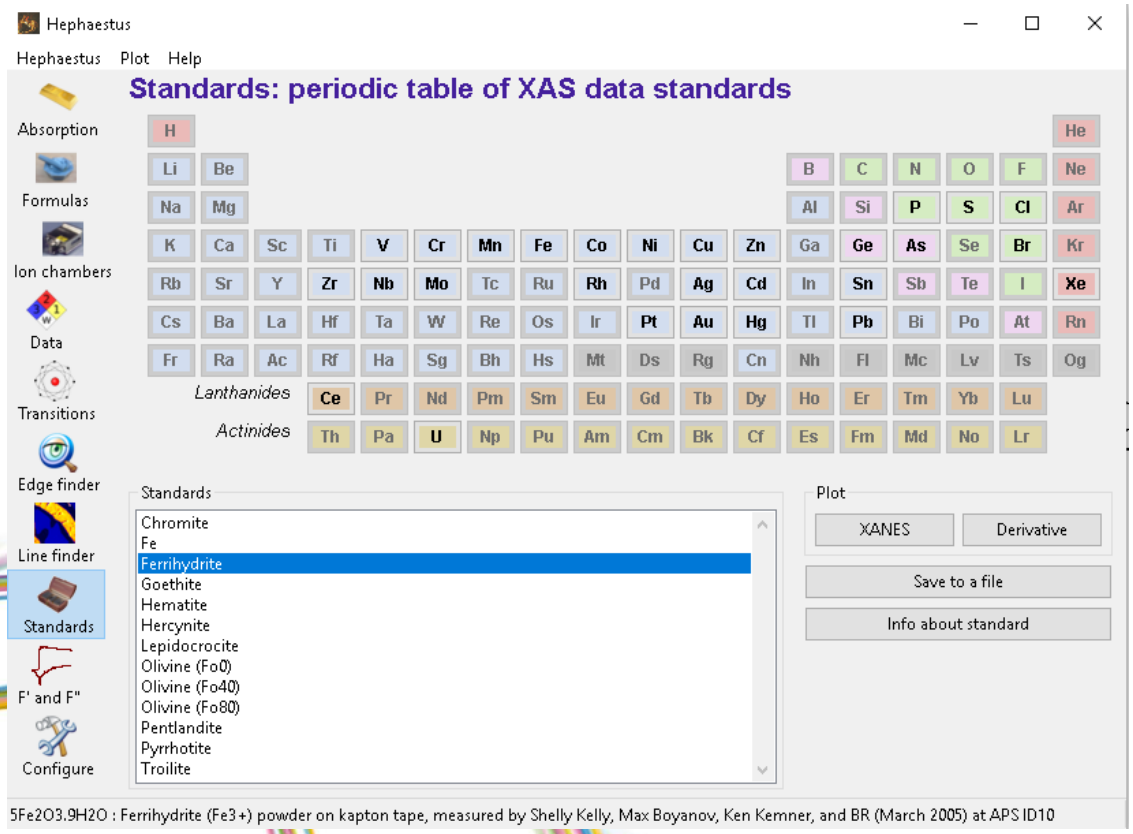


The Fe foil [Fe(0)] presented its first inflection point at the left-hand side of the dashed violet line.

The Fe_3O_4 [mixture of Fe(III)/Fe(II)] has its edge jump at the left side of the Fe_2O_3 [Fe(III) only] edge

Data analysis by Athena

19) Ideally, several standards will be acquired within the beamtime, providing the XAS features of each sample nature

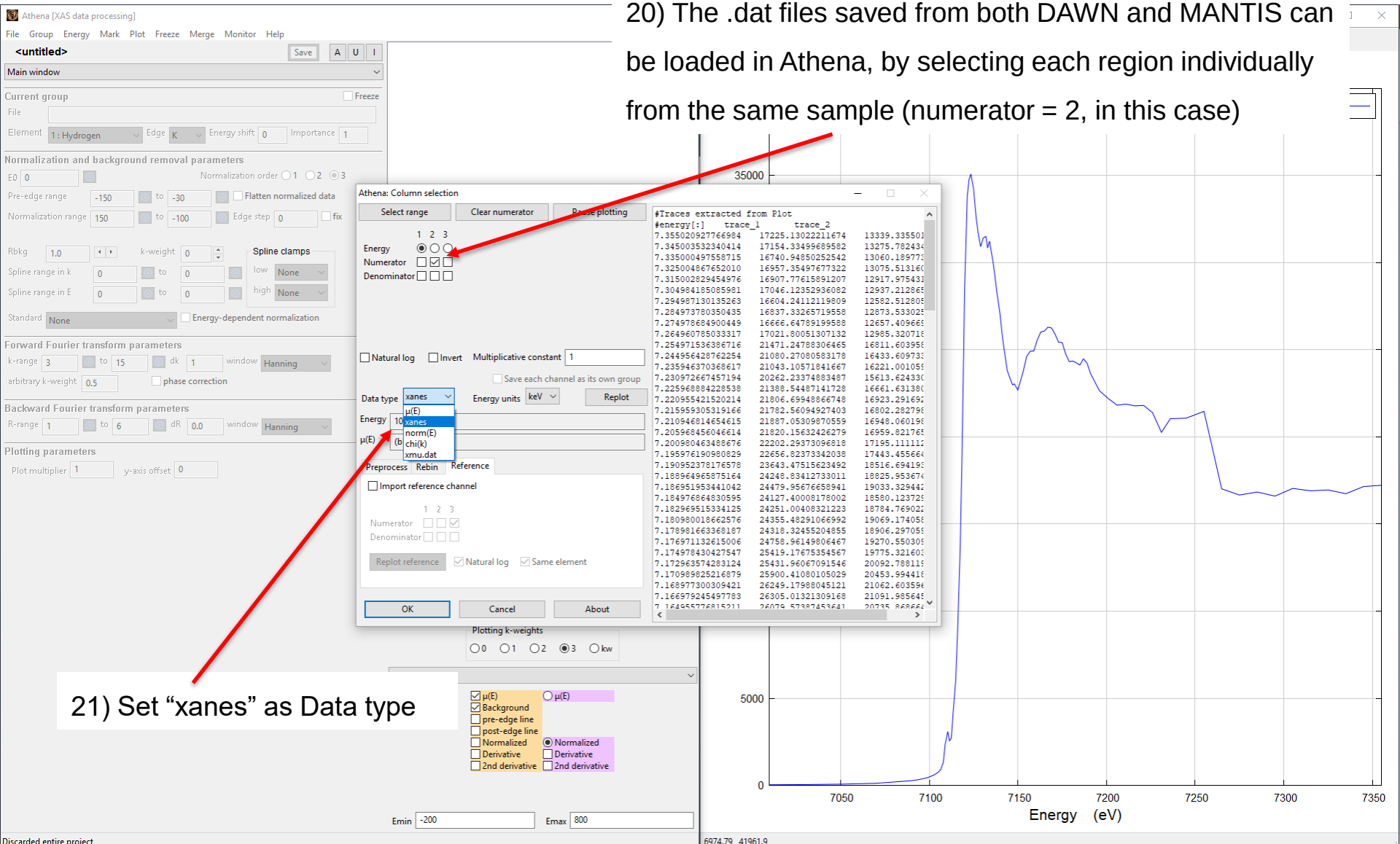


Hephaestus (downloaded automatically within Demeter software) also have some elemental standards, which can be saved to a file and used in Athena.

These XAS spectra should be calibrated (off-set) with their corresponding beam acquisition energy shift

Athena -- Opening .dat/.csv files

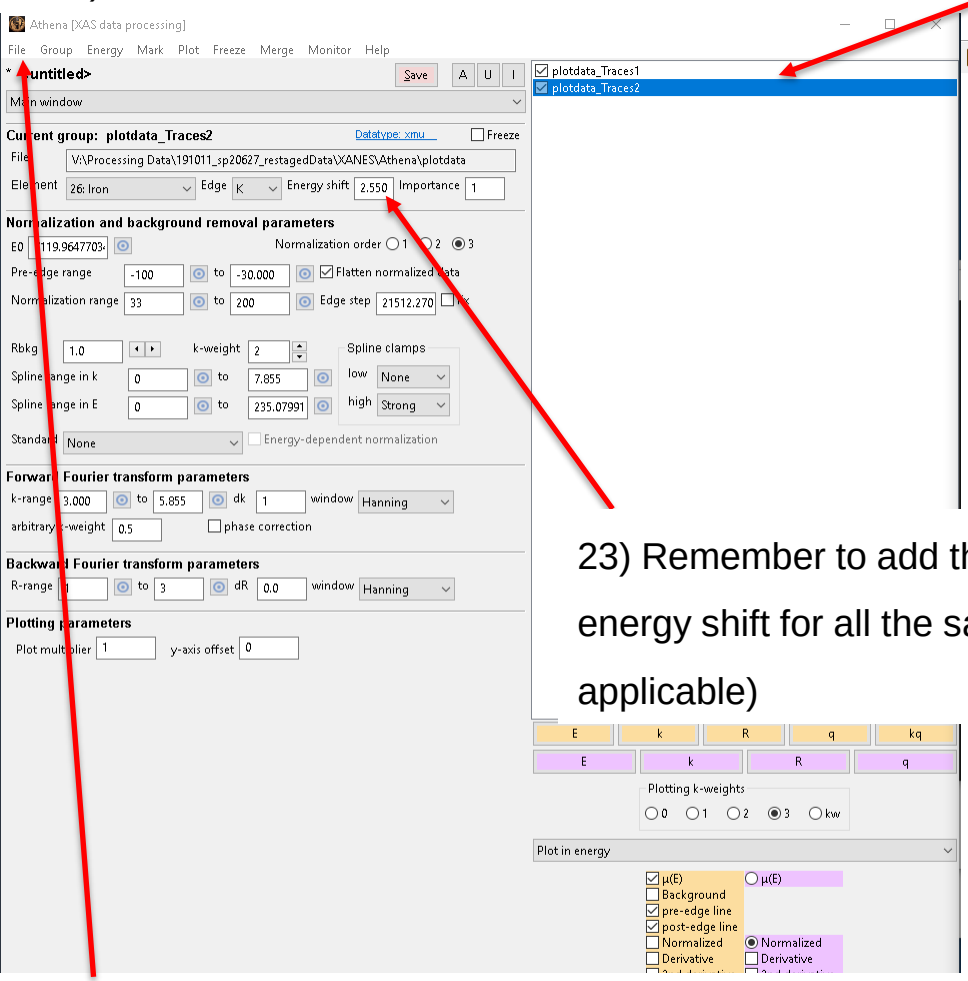
20) The .dat files saved from both DAWN and MANTIS can be loaded in Athena, by selecting each region individually from the same sample (numerator = 2, in this case)

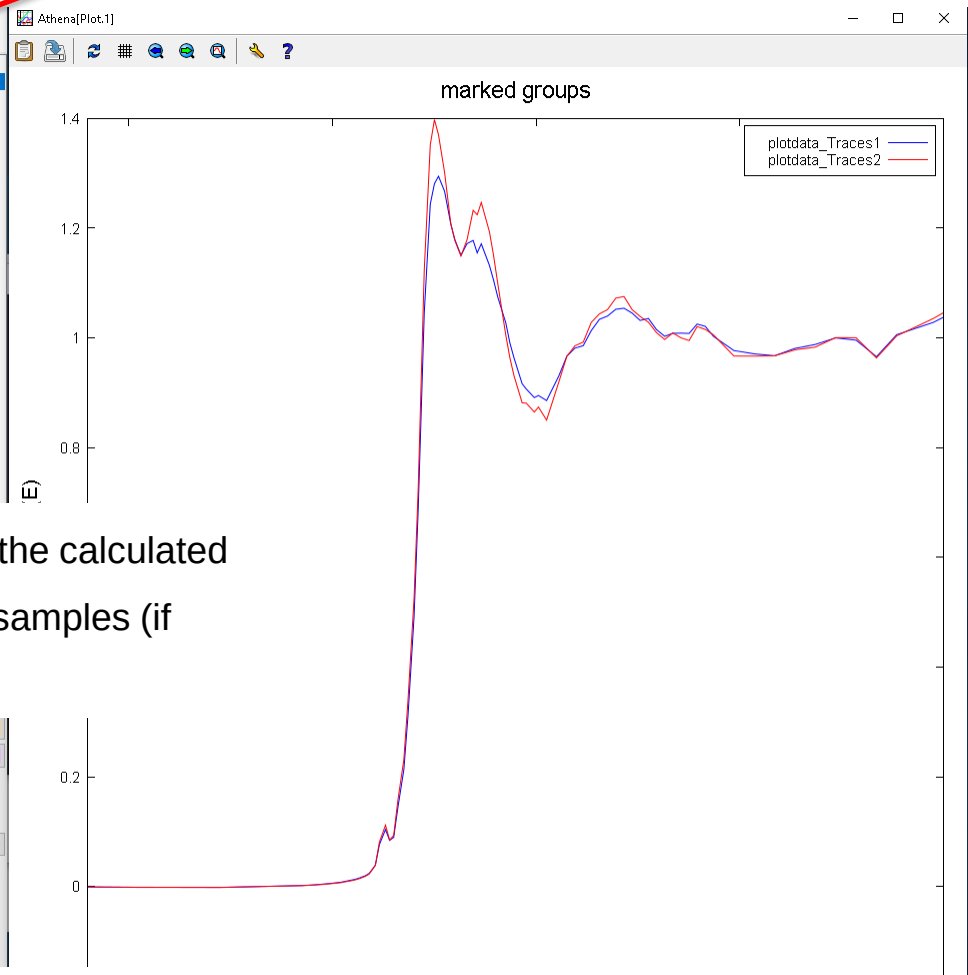


21) Set “xanes” as Data type

Athena -- Opening .dat/.csv files

22) Different regions from the same .dat file should be loaded consecutively (they can not be opened at the same time)



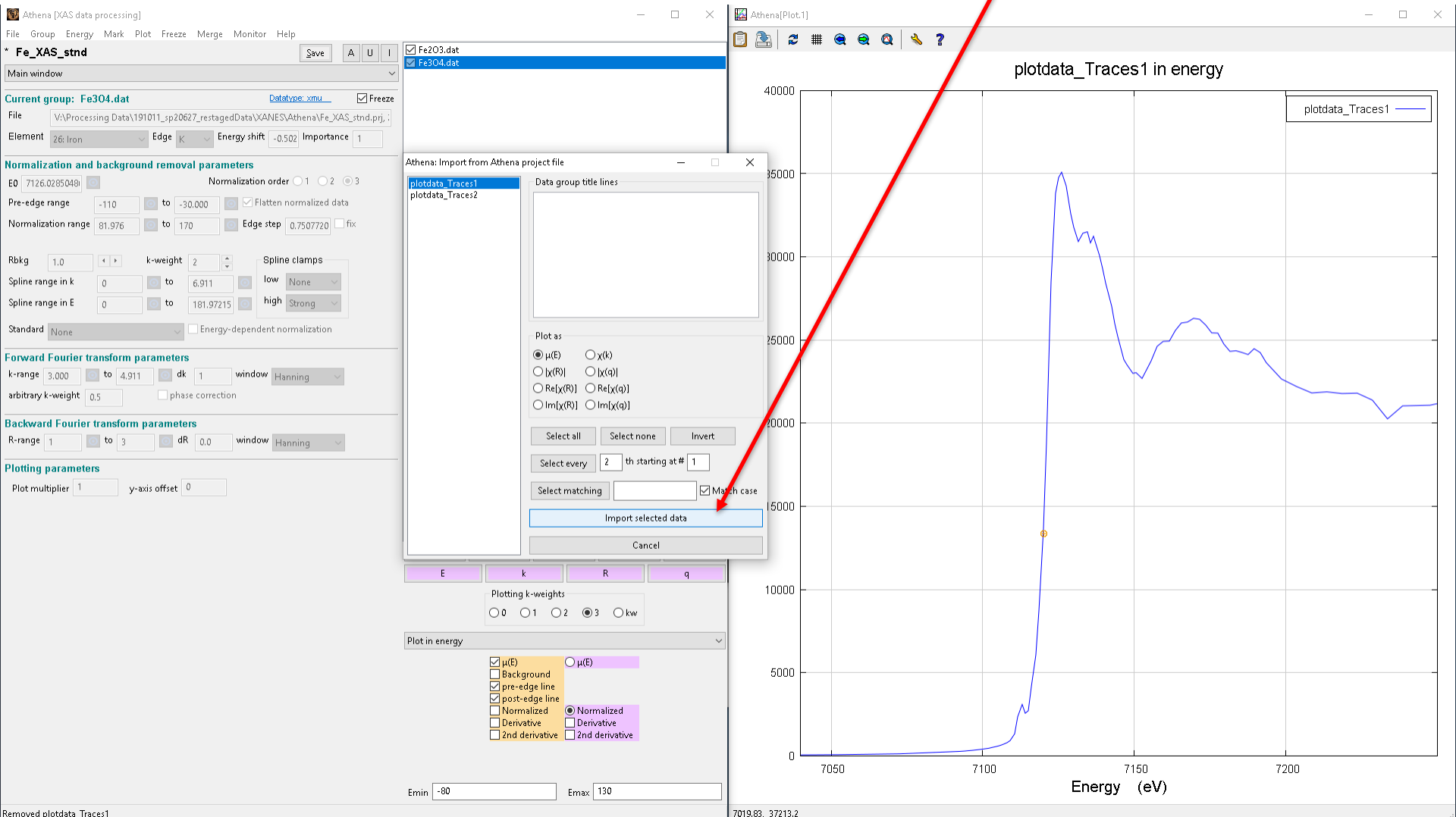


23) Remember to add the calculated energy shift for all the samples (if applicable)

24) Later, it is advisable to save the XAS scans into a “sample”.prj (Athena project)

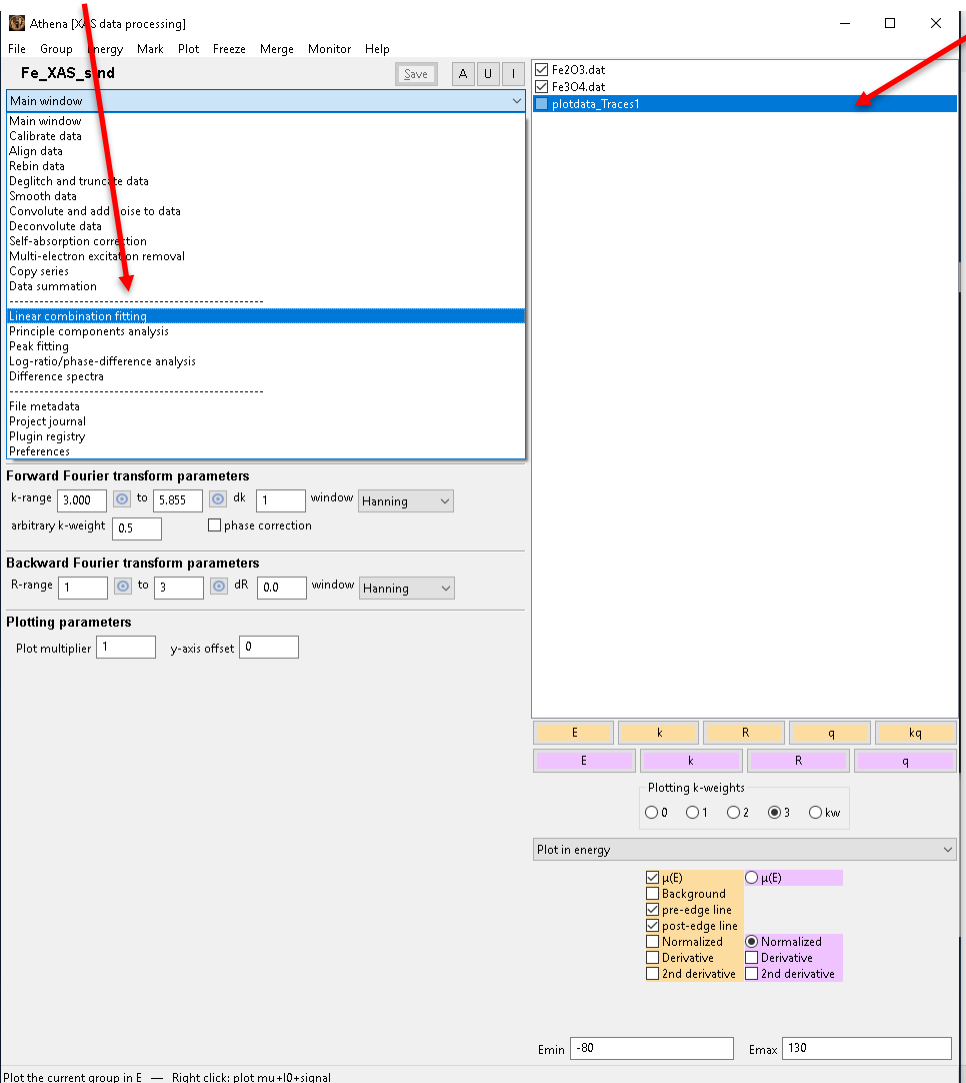
Data analysis by Athena

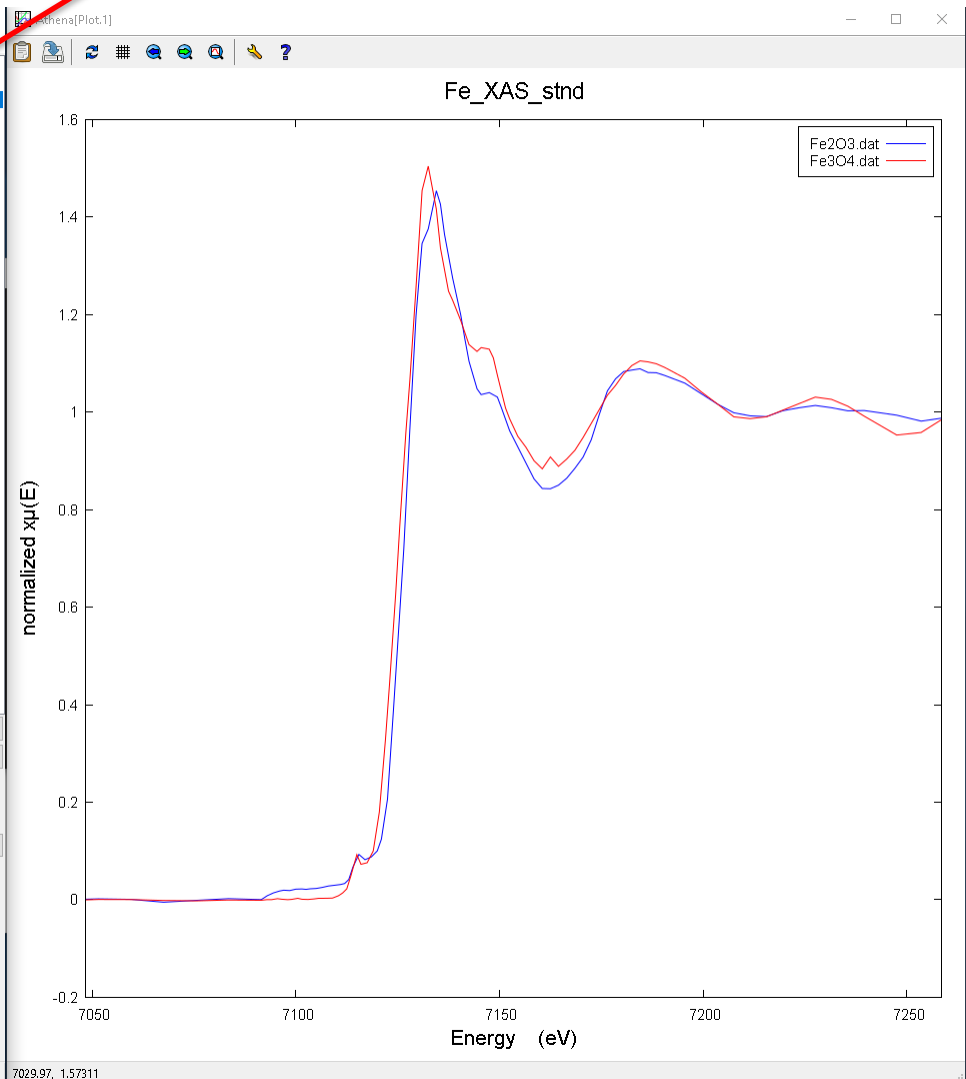
25) Open the Athena project with the standards (Fe_XAS_stnd (*example*), and load each sample to fit individually (File → Open → sample_athena.prj)



Data analysis by Athena

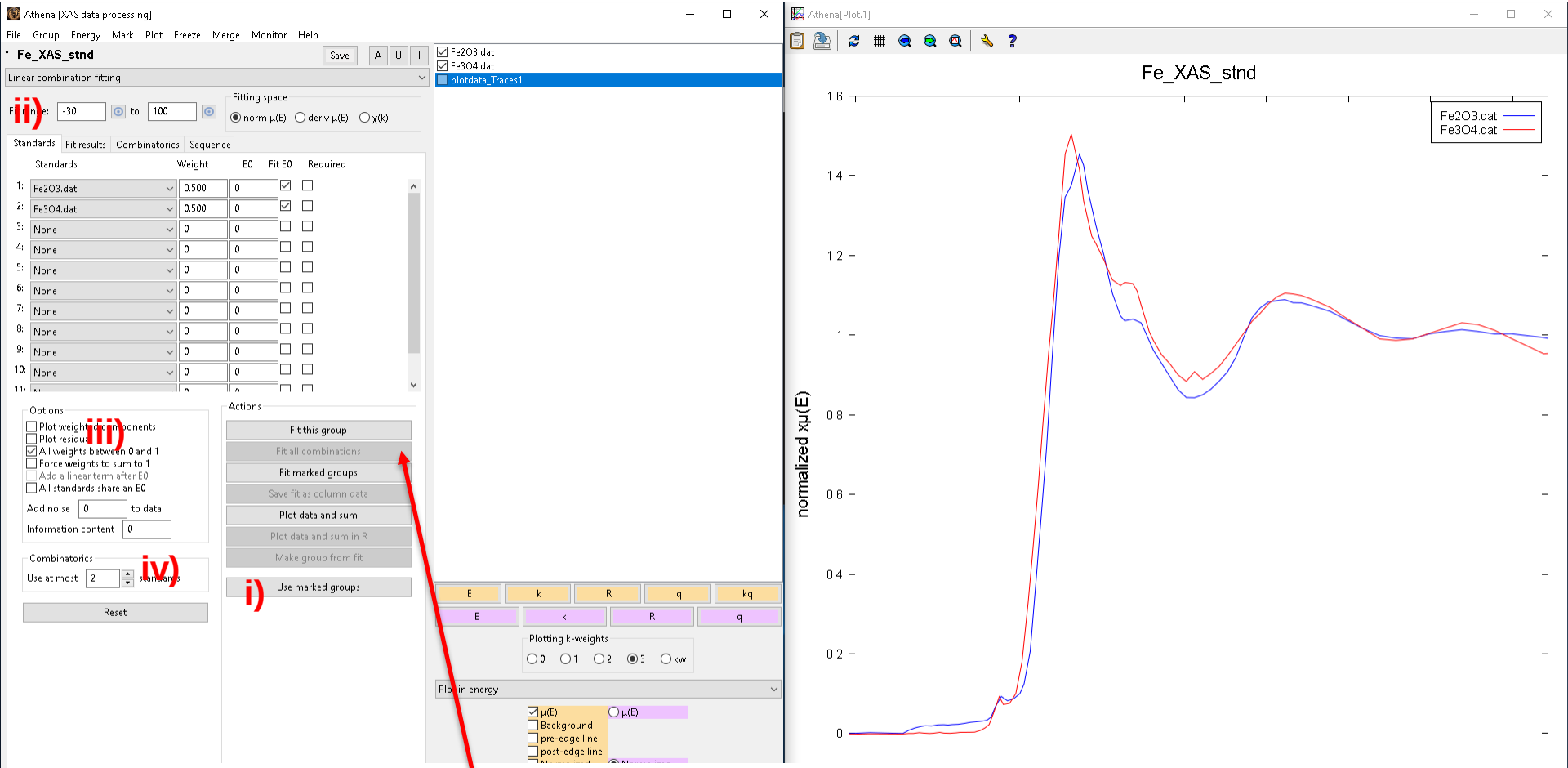
26) Mark all the standards and select the sample to analyse (highlighted in blue). Then choose Linear combination fitting in the scroll-down “Main window” menu





Data analysis by Athena

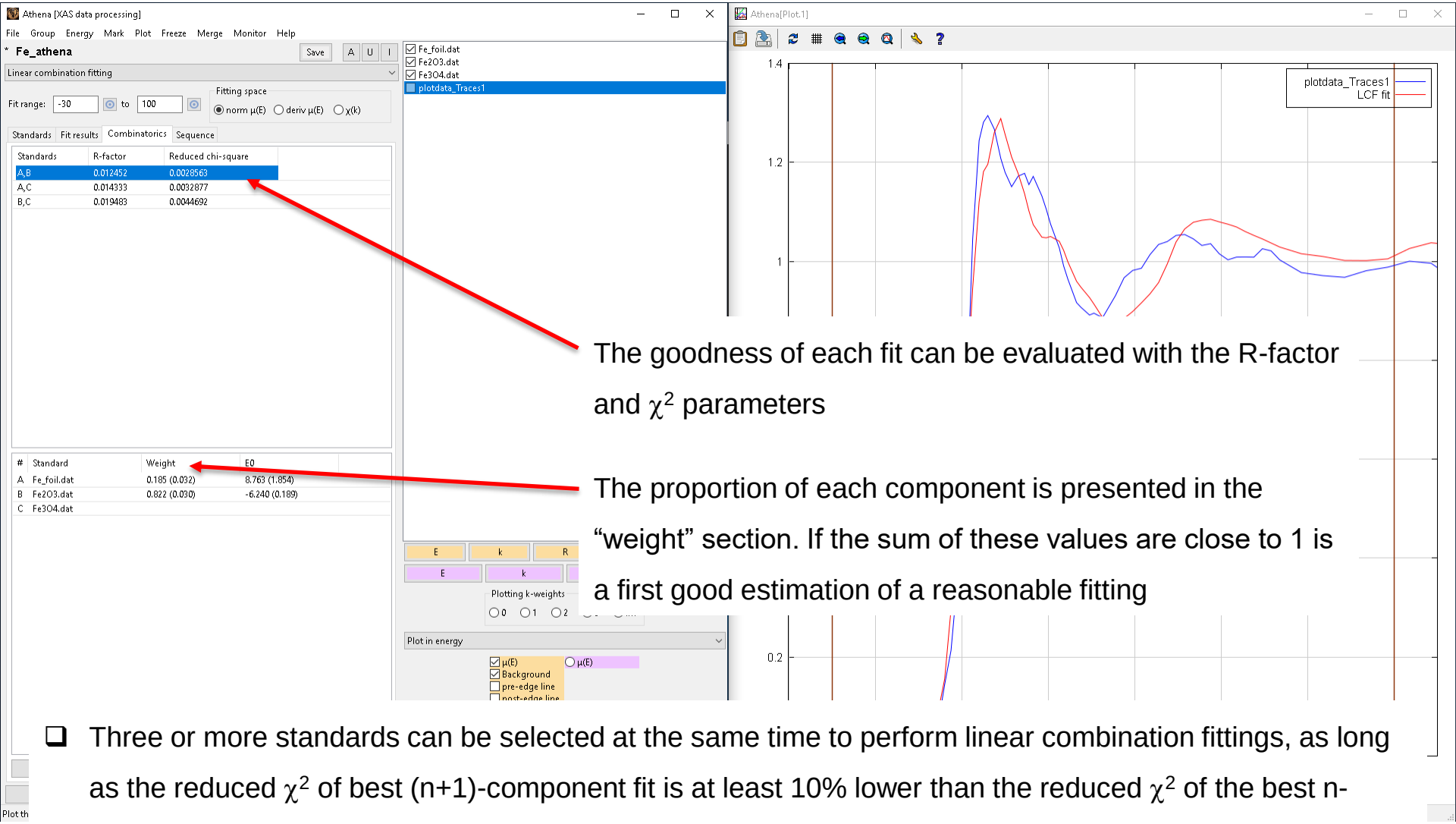
27) Follow this list of indications: i) Click “Use marked groups”, ii) select the Fit range (XANES region or larger), iii) unmark “Force weights to sum to 1” and iv) set “2” as most standards to use (to begin with)



20) Select “Fit this group” to compare against two standards. If 3 or more standards are loaded, select the “Fit all combinations” option for performing the LCF analysis

Data analysis by Athena

28) Here an example of the LCF results when using the Fe_foil as standard



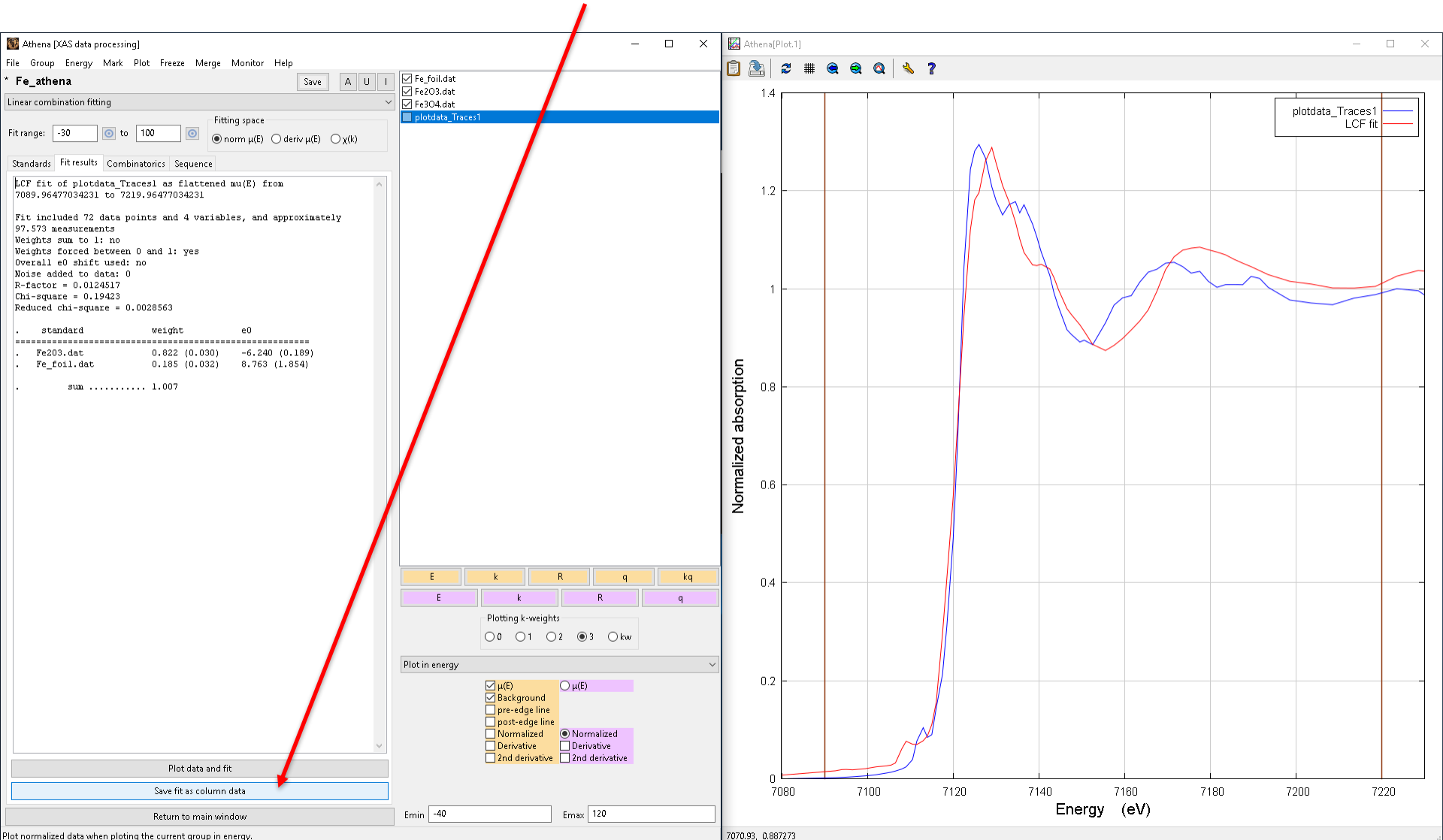
The goodness of each fit can be evaluated with the R-factor and χ^2 parameters

The proportion of each component is presented in the "weight" section. If the sum of these values are close to 1 is a first good estimation of a reasonable fitting

- Three or more standards can be selected at the same time to perform linear combination fittings, as long as the reduced χ^2 of best (n+1)-component fit is at least 10% lower than the reduced χ^2 of the best n-component fit; and if none of these individual components accounted for less than 5% of total.

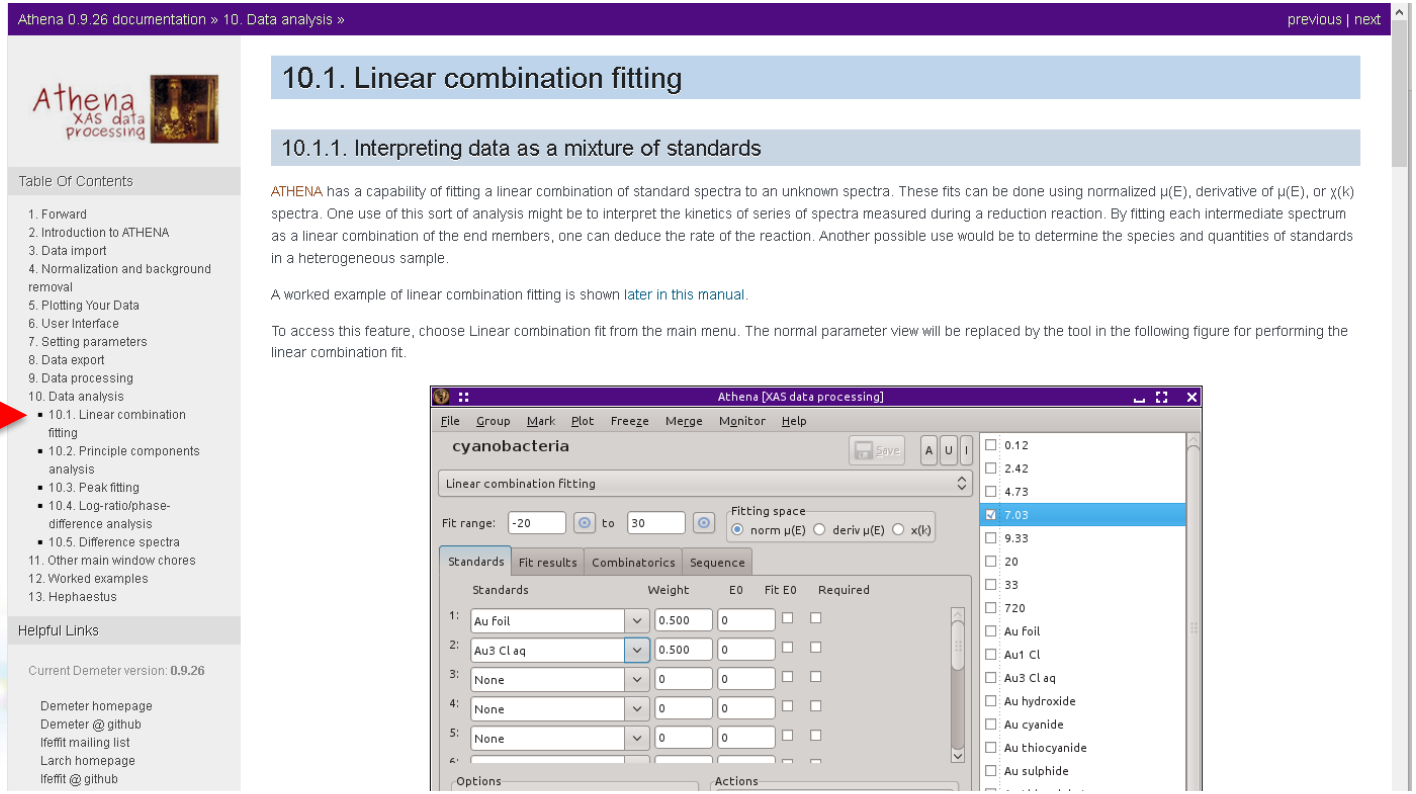
Data analysis by Athena

29) Individual fittings can be saved in the “Fit Results” tab for further poltting, by selecting ‘Save fit as column data’



Athena – Further information

- *A complete description of LCF analysis (including examples) can be found on-line: http://bruceravel.github.io/demeter/documents/Athena/index.html (accessed on 26/11/2019)*



- *There are several tutorials and videos from the author (Bruce Ravel) on how to analyse XAS data in the following links: These are really recommendable resources for PhD students or researchers wanted to extend their knowledge on XAS data treatment):*

/https://xafs.xrayabsorption.org/tutorials.html
https://vimeo.com/340202552