

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)



MantiS

❖ 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 &
```

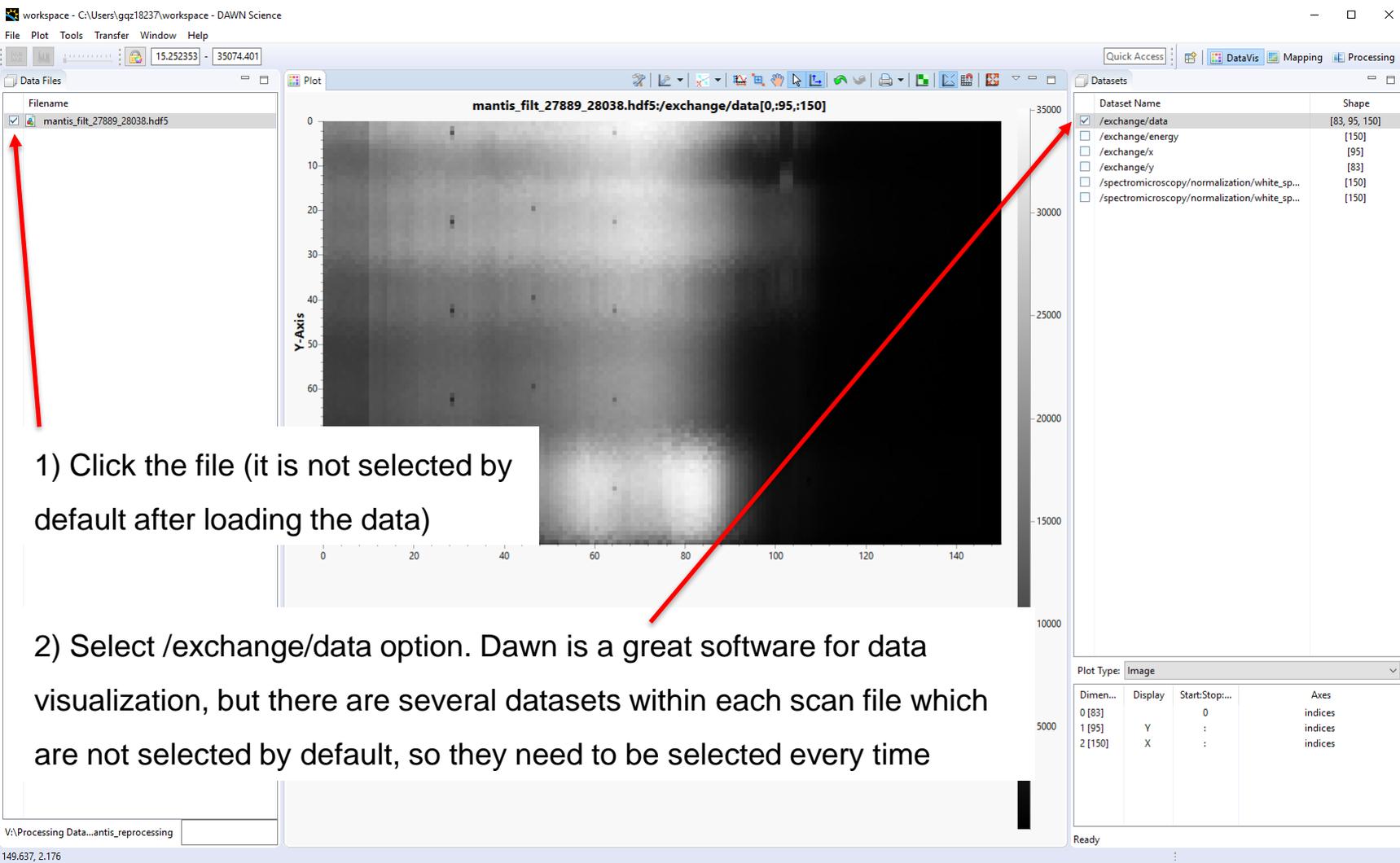
Finally, a video-tutorial on "loading DAWN for inspecting metadata from the 'i14-xxxx.nxs' files" can be found at:

<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`



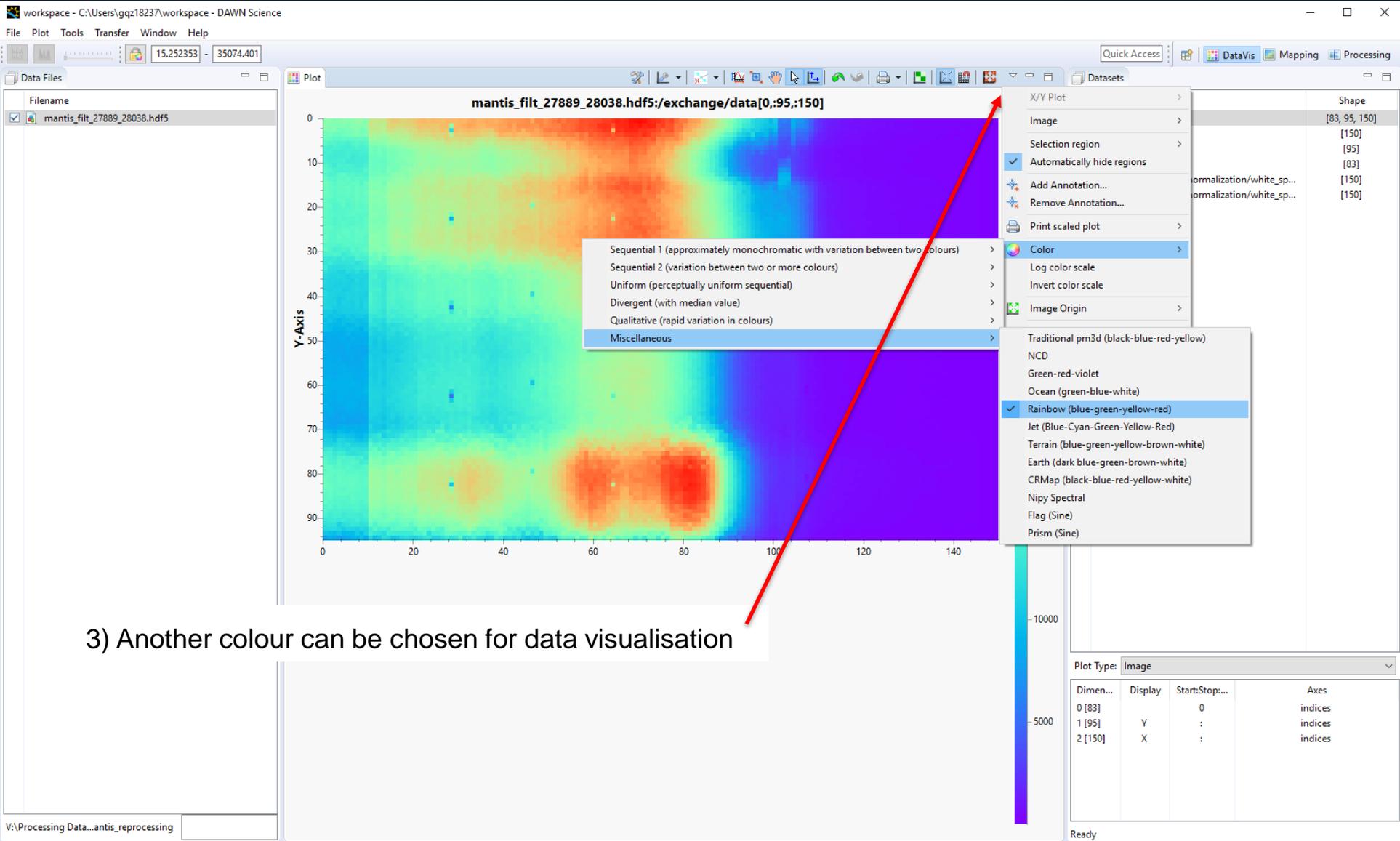
The screenshot shows the DAWN software interface. On the left, the 'Data Files' panel lists the file `mantis_filt_27889_28038.hdf5`. A red arrow points to this file. In the center, a plot titled `mantis_filt_27889_28038.hdf5:/exchange/data[0.:95.:150]` displays a grayscale image. A red arrow points to the plot. On the right, the 'Datasets' panel lists several datasets, with `/exchange/data` selected. A red arrow points to this dataset. Below the datasets panel, the 'Plot Type' is set to 'Image', and a table shows the dimensions and display settings for the plot.

Dimen...	Display	Start:Stop:...	Axes
0 [83]		0	indices
1 [95]	Y	:	indices
2 [150]	X	:	indices

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

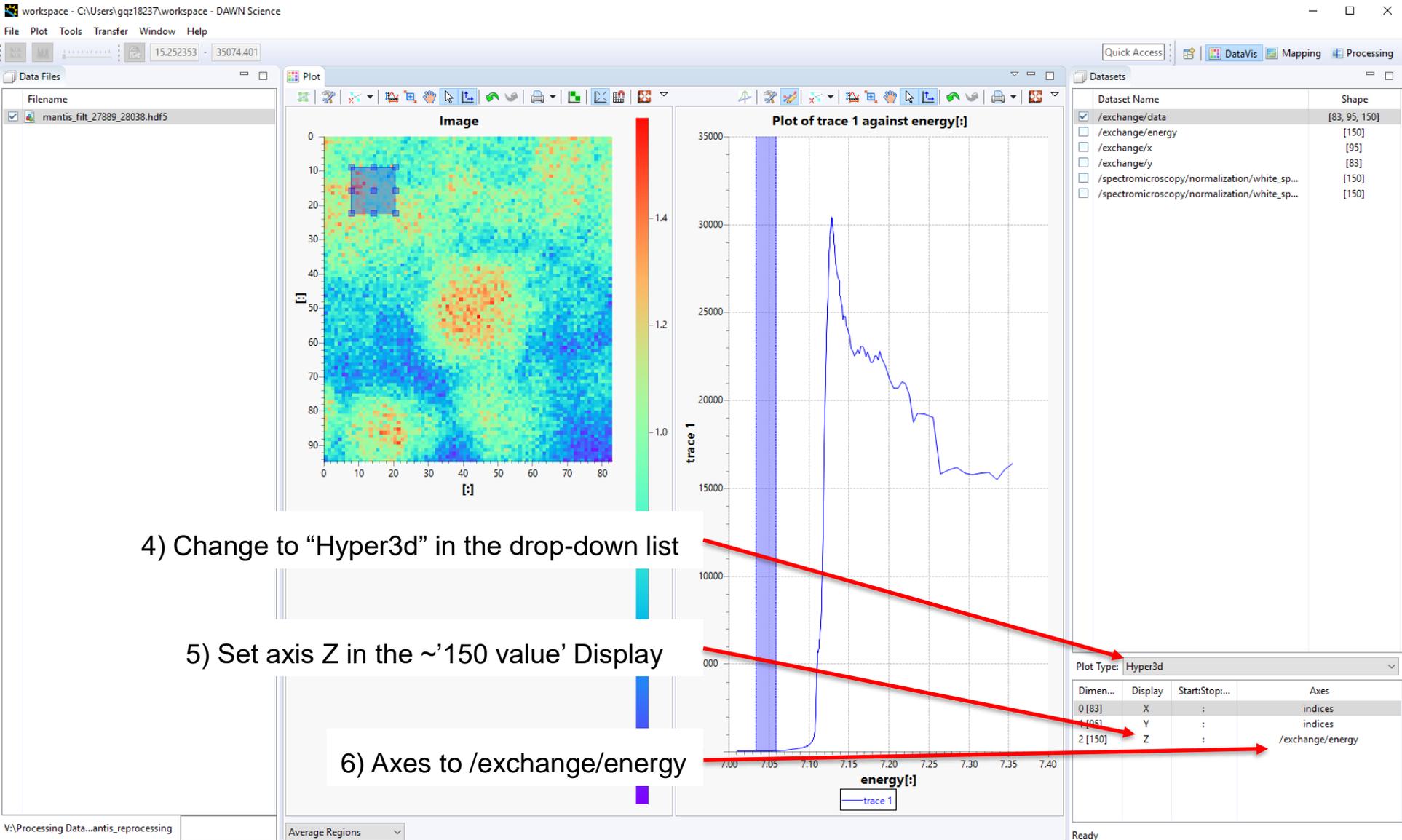
2) Select `/exchange/data` option. Dawn is a great software for data visualization, but there are several datasets within each scan file which are not selected by default, so they need to be selected every time

Visualisation of the data by DAWN



3) Another colour can be chosen for data visualisation

Visualisation of the data by DAWN

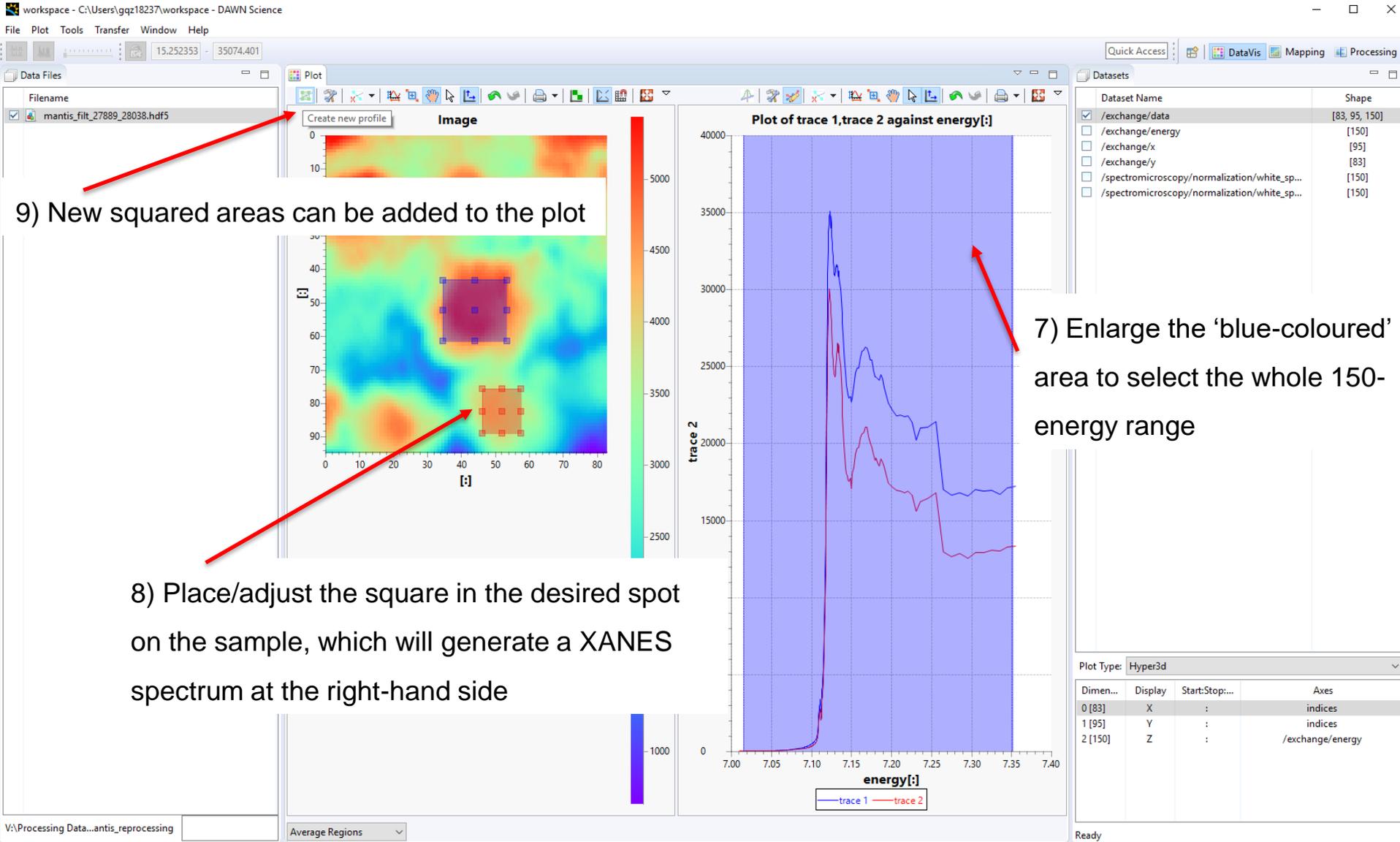


4) Change to "Hyper3d" in the drop-down list

5) Set axis Z in the '~150 value' Display

6) Axes to /exchange/energy

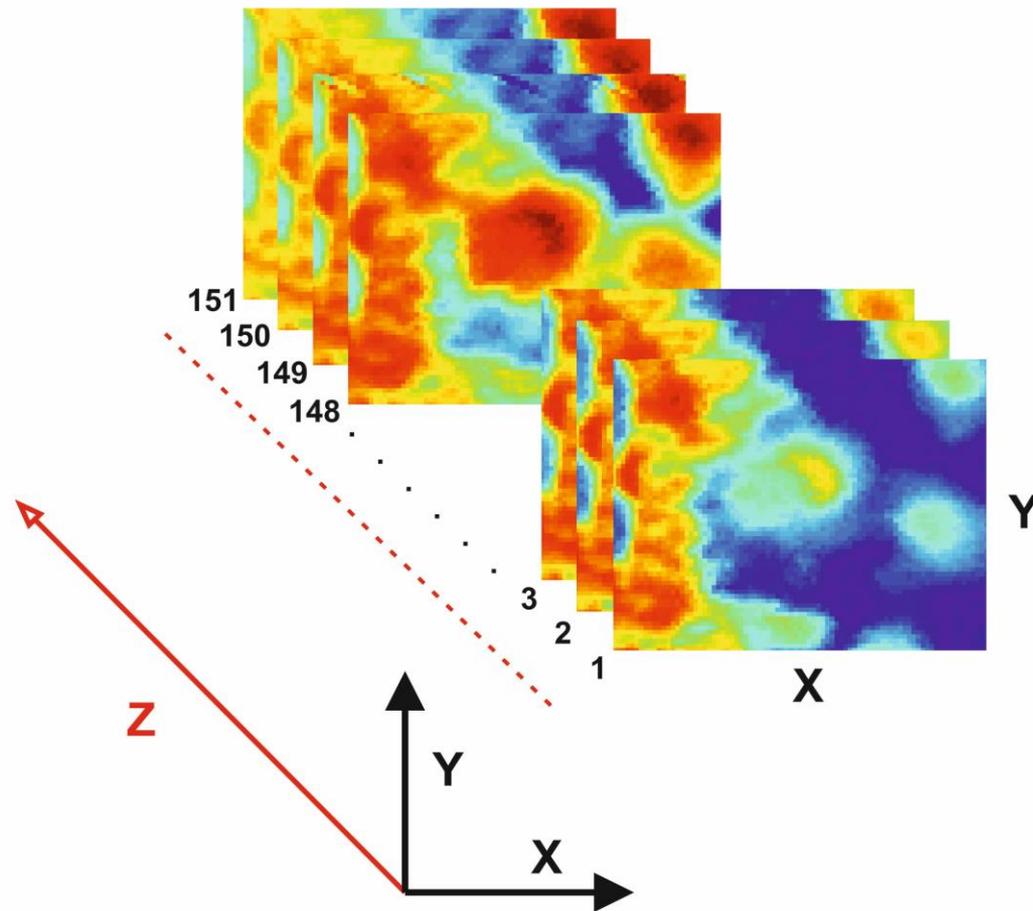
Visualisation of the data by DAWN



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

The screenshot displays the DAWN Science software interface. On the left, a 'Data Files' panel shows 'mantis_filt_27889_28038.hdf5'. The main plot area contains two subplots: 'Image' (a heatmap) and 'Plot of trace 1, trace 2 against energy'. A context menu is open over the plot, with 'Export plot data to tif/dat/csv...' selected. A red arrow points from this menu item to the text '11) Select "Export plot data" on the printer symbol for saving the drawn XANES regions'. Below this, an 'Export Data' dialog box is shown, with 'File' set to 'C:\Users\gqz18237\plotdata.dat', 'Format' set to 'dat', and 'Number of files' set to 'single'. A red arrow points from the 'single' radio button to the text '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'. To the right, a 'Dataset Name' table is visible, and a 'Plot Type' dropdown is set to 'Hyper3d'. A table below it shows dimensions and axes.

Dimen...	Display	Start:Stop:...	Axes
0 [83]	X	:	indices
1 [95]	Y	:	indices
2 [150]	Z	:	/exchange/energy

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/>



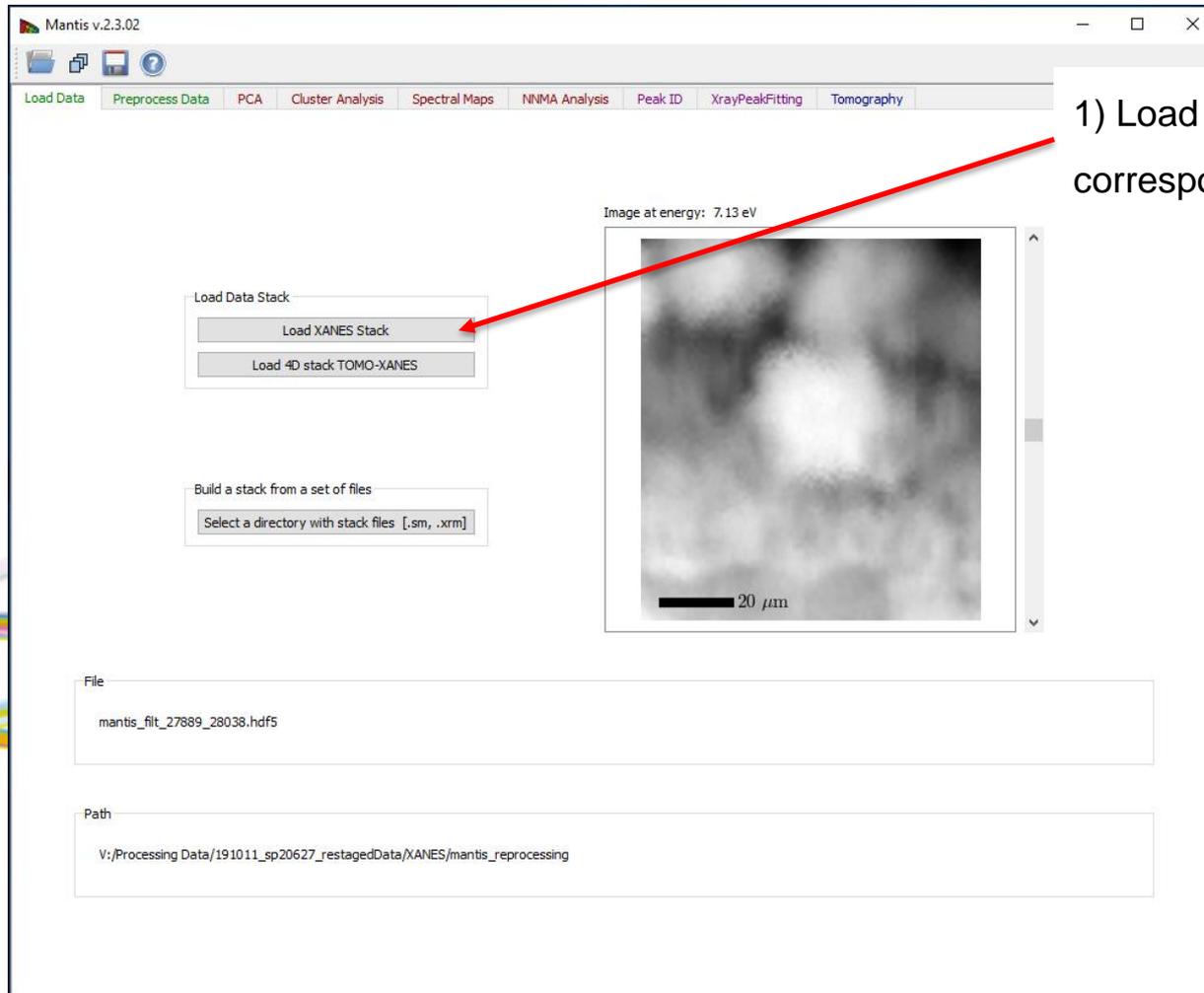
MantiS

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)

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...”

The screenshot displays the MANTIS v.2.3.02 software interface. The main window is divided into several panels. On the left, the 'Preprocess' panel contains buttons for 'Align stack...', 'Limit energy range...', 'Clip to subregion...', 'Dark signal subtraction...', and 'Save processed stack'. The 'Normalize' panel includes 'IO from file...', 'IO from histogram...', 'Show IO...', 'Use pre-normalized data', 'Load Reference Images', 'Reset IO', and 'Save OD data'. The 'Display' panel shows 'File' (mantis_fit_27889_28038.hdf5), 'Image' settings (Flux selected, Scalebar checked), and 'Display settings' (Minimum: 0%, Maximum: 100%, Gamma: 1.00). The main view shows an 'Image at energy: 7.13 eV' with a 20 μm scale bar and a 'Spectrum at pixel [41, 47] or po' plot. A 'Stack Alignment' dialog box is open, showing options for 'This Image' (Set as Reference Image, Save Reference Image, Load Reference Image, Remove energy from stack), alignment methods (Automatic Alignment selected, Manual Alignment), and automatic alignment options (Calculate image shifts, Select subregion on reference, Remove subregion selection, Edge Enhancement, Prewitt, Sobel, Max shift [pixels]: 0, Show Cross-correlation). The dialog also shows 'Manual Alignment' options (Pick a point on reference image, This image: click on same point, Apply manual shifts, X manual shift, Y manual shift) and actions (Save image shifts plot, Save image shifts, Load image shifts, Crop aligned images, Apply Alignment, Dismiss). The dialog displays 'Image at energy: 7.15 eV' and 'Reference image' side-by-side, with 'X shift: -0.53 pixels' and 'Y shift: -0.63 pixels'. Below the images are plots for 'Image shifts' (Shifts in x, y-green [pixels] vs Photon Energy [eV]) and 'Cross-correlation'.

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

The screenshot displays the MANTIS v.2.3.02 software interface. The main window has a menu bar with options: Load Data, Preprocess Data, PCA, Cluster Analysis, Spectral Maps, NNMA Analysis, Peak ID, XrayPeakFitting, and Tomography. Below the menu bar are several panels:

- Preprocess:** Includes buttons for 'Align stack...', 'Limit energy range...', 'Clip to subregion...', 'Dark signal subtraction...', and 'Save processed stack'.
- Normalize:** Includes buttons for 'IO from file...', 'IO from histogram...', 'Show IO...', 'Use pre-normalized data', 'Load Reference Images', 'Reset IO', and 'Save OD data'.
- Display:** Shows the file 'mantis_fit_27889_28038.hdf5'. It has radio buttons for 'Flux' (selected) and 'Optical Density', and checkboxes for 'Scalebar', 'White', and 'Colorbar'. It also features sliders for 'Minimum' (0%), 'Maximum' (100%), and 'Gamma' (1.00), along with buttons for 'Play stack movie', 'Save images...', 'Despike', 'Reset', and 'Color Table...'.
- Region of Interest:** Includes buttons for 'Select ROI (Lasso)', 'Reset ROI', 'Set ROI As IO', 'Save ROI Spectrum...', 'ROI Dose Calculation...', and 'Spectral ROI...'.

In the bottom left, there is a panel titled 'Image at energy: 7.17 eV' showing a grayscale image with a 20 μm scale bar. In the bottom right, there is a panel titled 'Spectrum at pixel [35, ...]' showing a plot of Optical Density versus Photon Energy [eV].

A 'Save' dialog box is open in the foreground, showing options to save files in various formats: .pdf, .png, .svg, .csv, and .tif (data). The 'all images' checkbox under the .tif (data) format is checked. The 'Filename' field is empty, and the 'Path' field is set to '27_restagedData/XANES/mantis_reprocessing'. The 'Save' button is highlighted.

A red arrow points from the 'Save images...' button in the 'Display' panel to the 'Save' dialog box.

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/)*

Mantis v.3.1.01

Load Data Preprocess Data PCA Cluster Analysis Spectral Maps NNMA Analysis Peak ID XrayPeakFitting Tomography Image Maps

Preprocess
Align stack...
Crop stack 3D...
Artefacts & Leveling
Dark signal subtraction...
Save processed stack

Normalize
Select IO...
IO from file...
Show IO...
Use pre-normalized data
Load Reference Images
Reset IO
Save OD data

Display
File: mantis_133676_133953.hdf5
Image: Fluor
 Optical Density
 Scalebar
 White
 Colorbar
Display settings
Minimum: 0%
Maximum: 100%
Gamma: 1.00
Buttons: Play stack movie, Save images..., Despike, Reset, Color Table...

Region of Interest
Select ROI (Lasso)
Reset ROI
Save ROI Spectrum...
ROI Dose Calculation...
Spectral ROI...

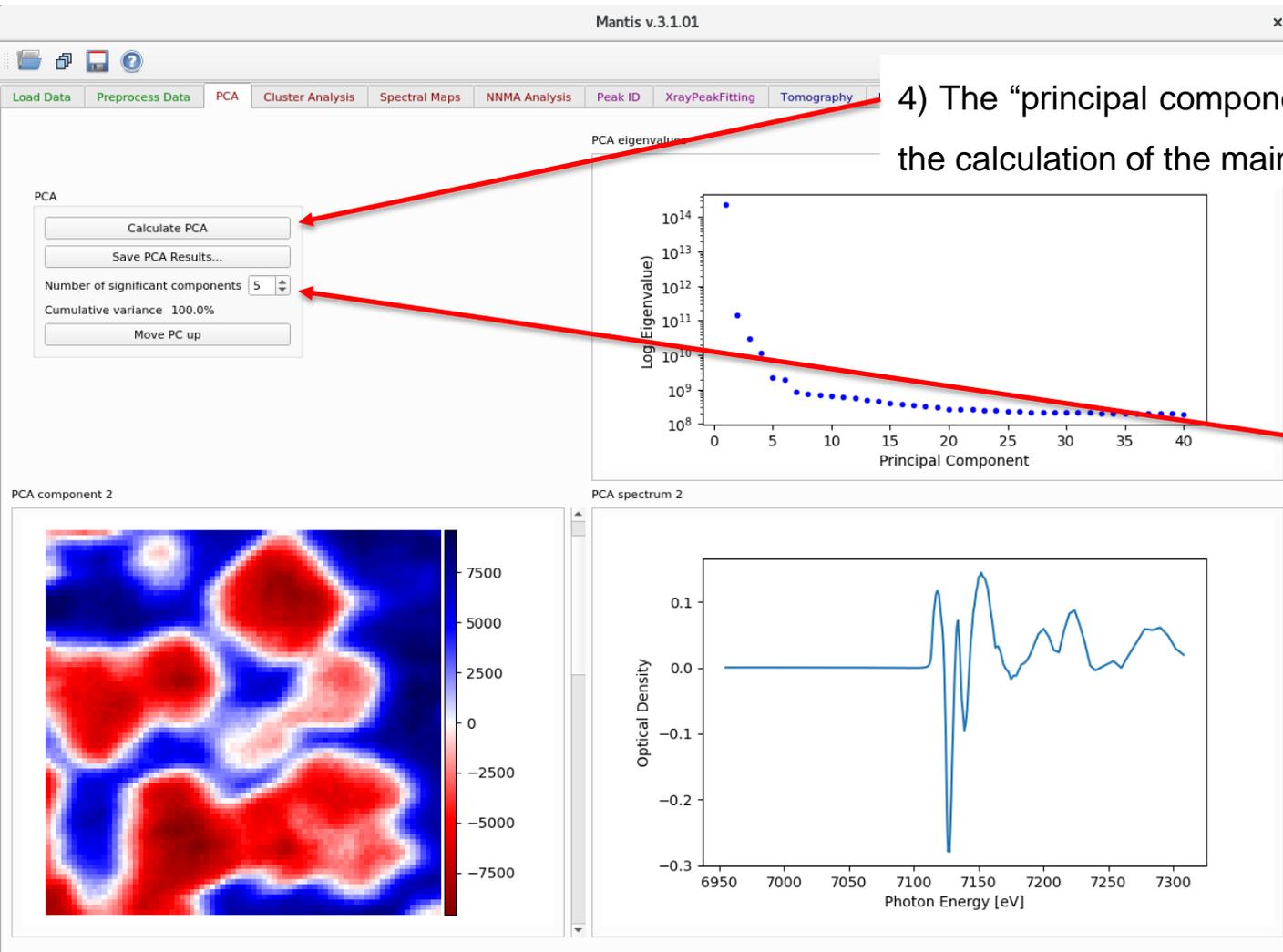
Image at energy: 7290.00 eV
Spectrum at pixel [38, 38] or position [38.00, 38.00]

Optical Density vs Photon Energy [eV]

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.

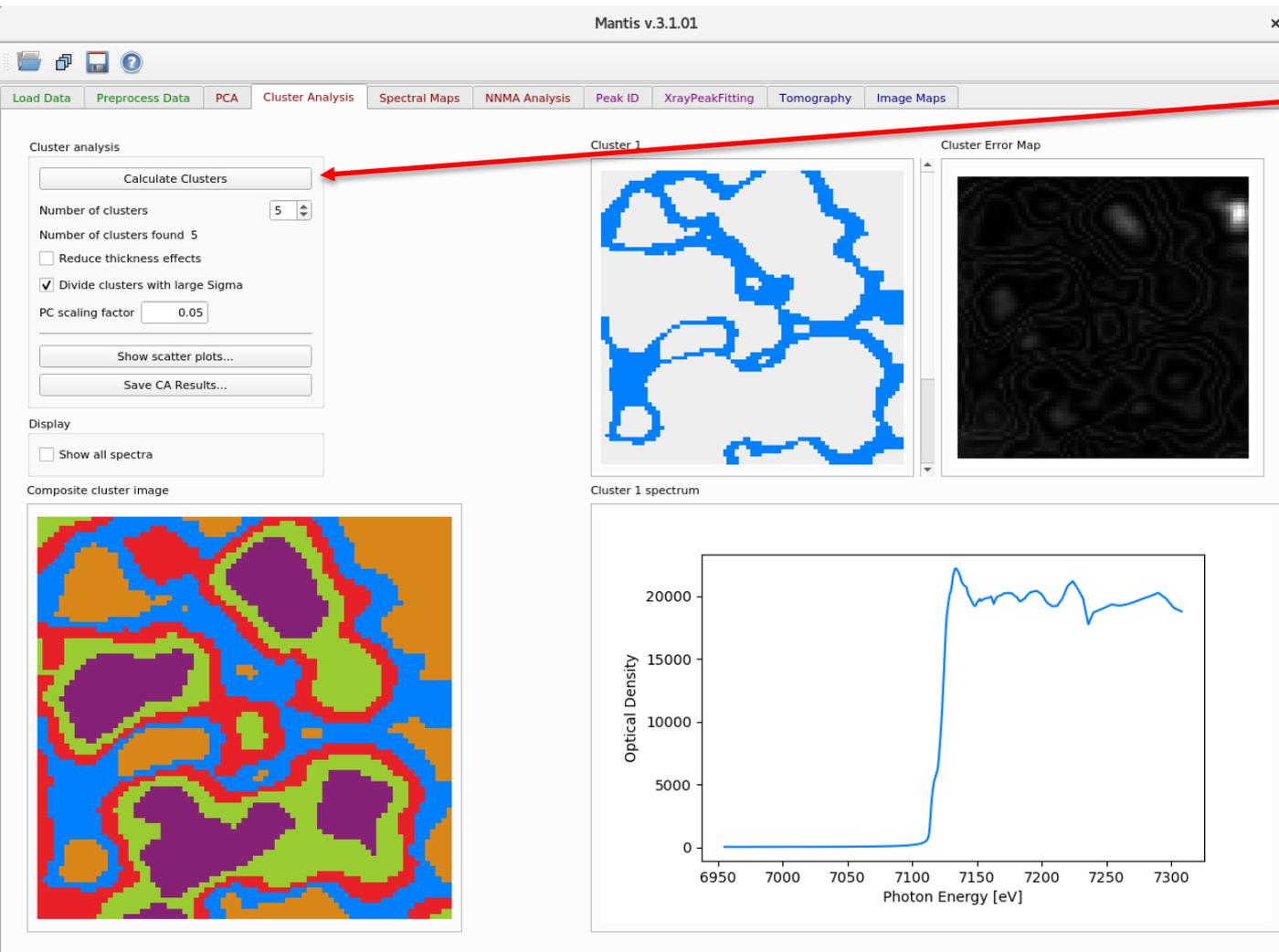
Data processing by MANTIS



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

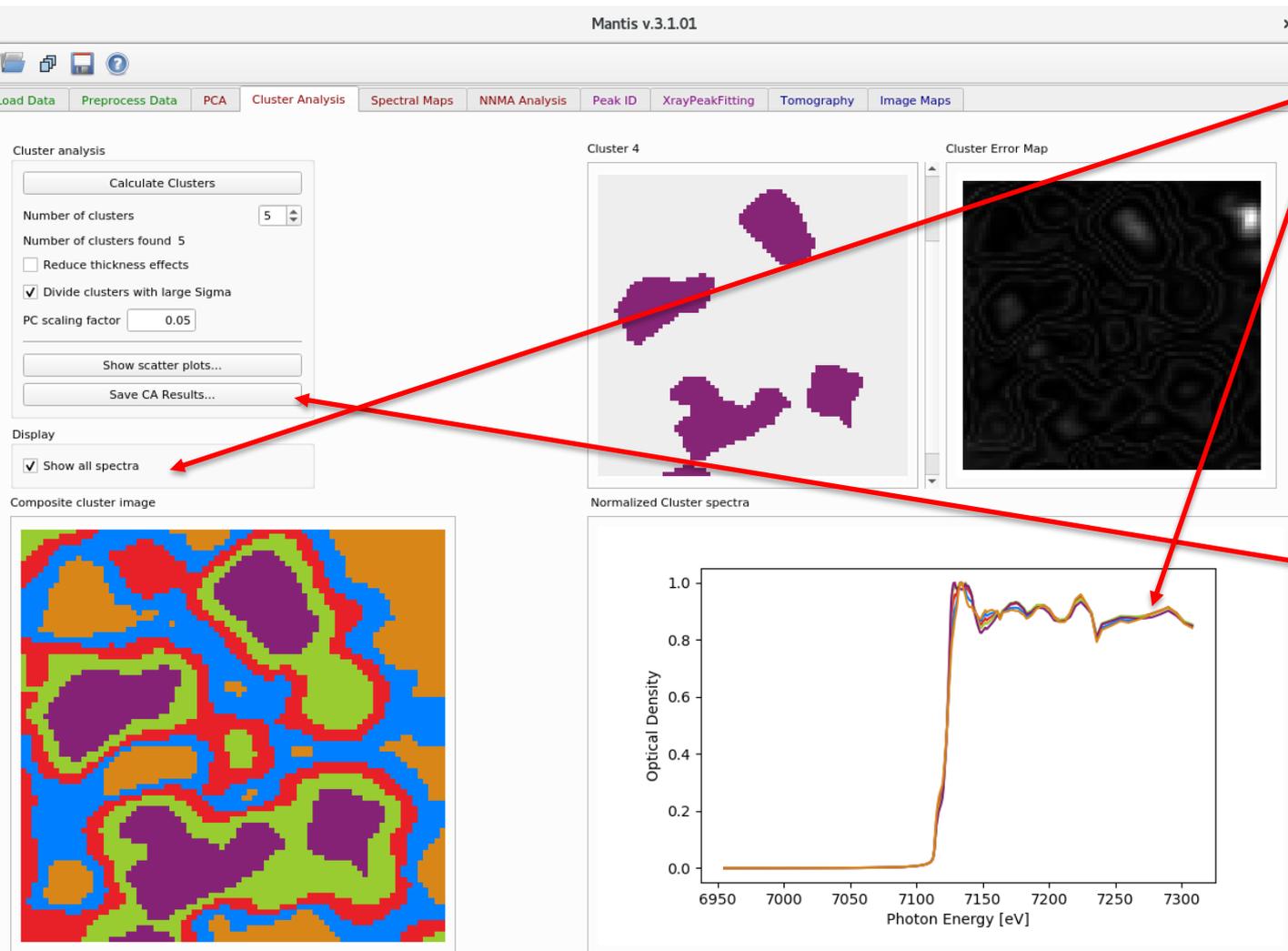
5) The number of significant components may be manually modified, and the PCA recalculated again (see Github guide for more information)

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

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 developer) 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)

Athena 0.9.26 documentation » previous | next

10. Data analysis

ATHENA provides various kinds of data analysis. None of the data analysis capabilities in ATHENA require the use of FEFF. Analysis of data using FEFF is a huge topic and is the purpose of the ARTEMIS program. The data analysis techniques included in ATHENA are all purely empirical. While there is no substitute for careful, sophisticated analysis of EXAFS data using theory from FEFF, often the empirical techniques described in this chapter are adequate to answer the questions you have about your data.

ATHENA's analysis tools are accessed from the analysis section of the main menu, as shown below.

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1. Forward
2. Introduction to ATHENA
3. Data import
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5. Plotting Your Data
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9. Data processing
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 - 10.1. Linear combination fitting
 - 10.2. Principle components analysis
 - 10.3. Peak fitting
 - 10.4. Log-ratio/phase-difference analysis
 - 10.5. Difference spectra
11. Other main window chores
12. Worked examples
13. Hephaestus

Helpful Links

Current Demeter version: 0.9.26

Linear combination Fitting

Principle components analysis

Peak fitting

Log-ratio/phase-difference analysis

Athena [XAS data processing]

File Group Mark Plot Freeze Merge Monitor Help

cyanobacteria

Main window

Calibrate data

Align data

Rebin data

Deglitch and truncate data

Smooth data

Convolute and add noise to data

Deconvolute data

Self-absorption correction

Calibrate dispersive XAS data

Copy series

Data summation

Save A U I

Freeze

bacteria.prj, 4

rgy shift: -0.24 Importance 1

Parameters

Flatten normalized data

Normalization order 1 2 3

Edge step 0.230407 fix

Spline clamps

low None

high Strong

0.12

2.42

4.73

7.03

9.33

20

33

720

Au foil

Au Cl

Au3 Cl aq

Au hydroxide

Au cyanide

Au thiocyanide

Au sulphide

Au thiosulphate aq

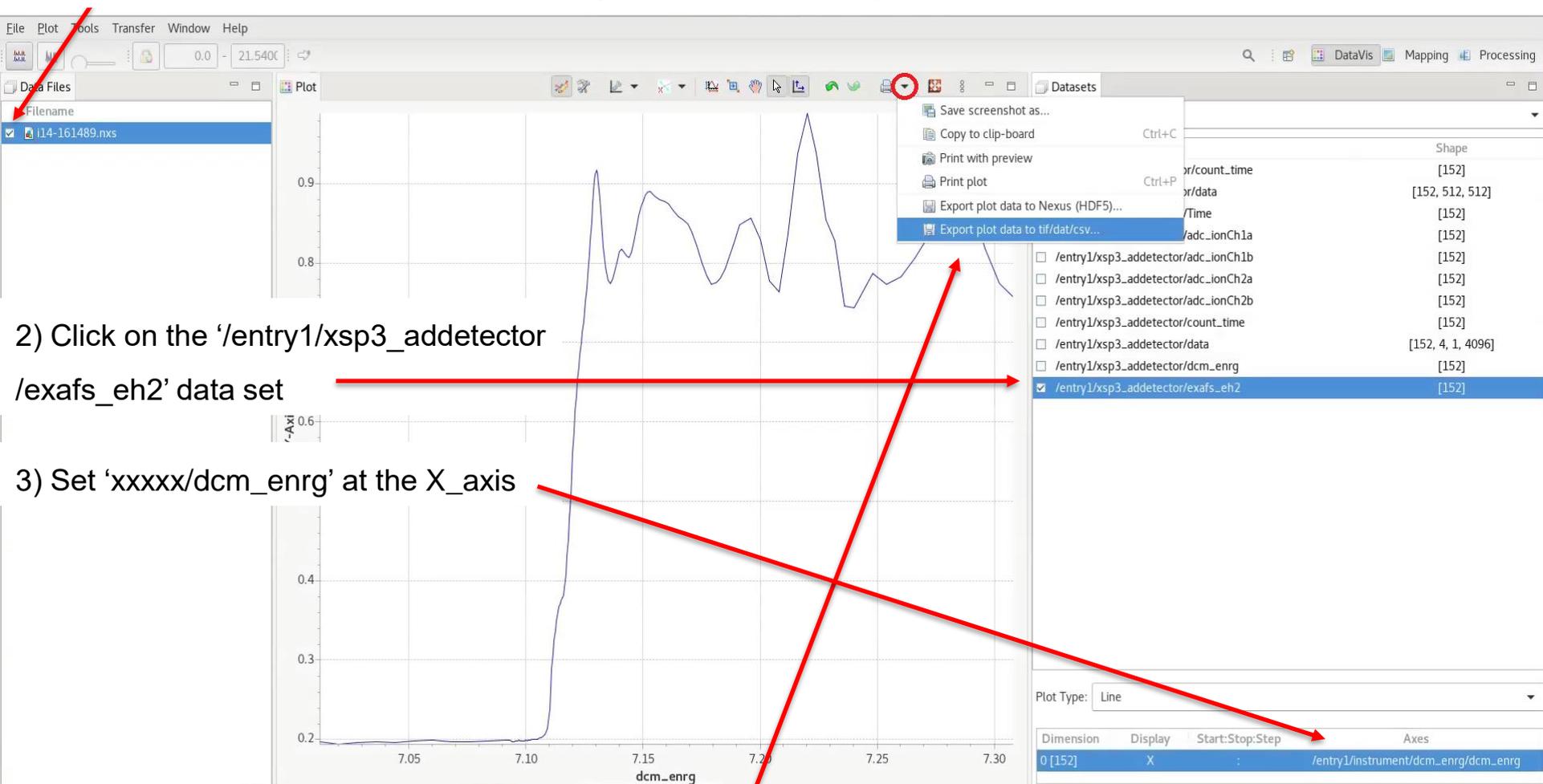
Au thiomalate aq

❑ 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_eng' 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

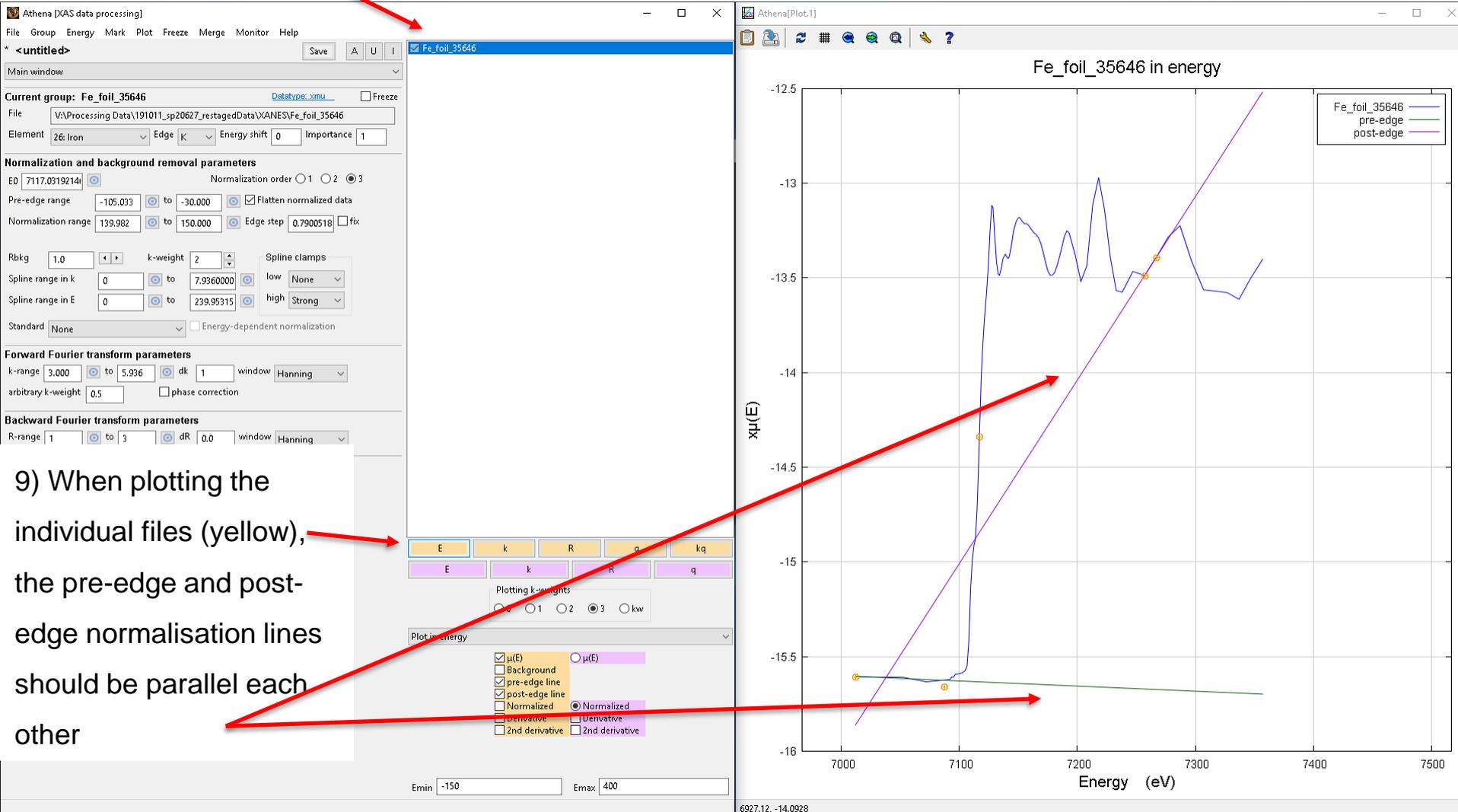
The screenshot shows the Athena software interface. The main window is titled 'Athena [XAS data processing]' and contains a menu bar (File, Group, Energy, Mark, Plot, Freeze, Merge, Monitor, Help) and a toolbar. A dialog box titled 'Athena: Column selection' is open, showing a table of columns and their corresponding data. The table has columns for '#scan', '#dcm_engr', and 'exafs_eh2:161489'. The 'Energy' column is selected as column 1, and the 'Numerator' column is selected as column 2. The 'Data type' is set to $\mu(E)$ and the 'Energy units' are set to eV. The plot window, titled 'Athena[Plot.1]', shows a plot of $\chi(E)$ versus Energy (eV) for the file 'Fe-foil_161489.dat'. The plot shows a sharp peak at approximately 7.1 eV, followed by a series of smaller peaks and troughs. The x-axis ranges from 7.05 to 7.3 eV, and the y-axis ranges from 0.1 to 1.0. A red arrow points from the text above to the 'Energy' column in the dialog box. Another red arrow points from the text above to the 'Numerator' column. A third red arrow points from the text above to the 'Data type' dropdown menu. A fourth red arrow points from the text above to the 'Energy units' dropdown menu.

6) Select data type as ' $\mu(E)$ ' 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

Data analysis by Athena – Calculating the E shift

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



Data analysis by Athena

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

Athena [XAS data processing]

File Group Energy Mark Plot Freeze Merge Monitor Help

* <untitled> Save A U 1 Fe_foil.dat

Main window

Current group: Fe_foil.dat Datatype: xmu

File V:\Processing Data\Standards_XANES_J14\Fe_Pasta\Fe_foil.dat

Element 26: Iron Edge κ Energy shift 0 Importance 1

Normalization and background removal parameters

E0 7112 Normalization order 1 2 3

Pre-edge range -90 to -30 Flatten normalized data

Normalization range 15 to 190 Edge step 2.5141929 fix

Rbkg 1.0 k-weight 2 Spline clamps

Spline range in k 0 to 7.164 low None

Spline range in E 0 to 195.53941 high Strong

Standard None Energy-dependent normalization

Forward Fourier transform parameters

k-range 3.000 to 5.164 dk 1 window Hanning

arbitrary k-weight 0.5 phase correction

Backward Fourier transform parameters

R-range 1 to 3 dR 0.0 window Hanning

Plotting parameters

Plot multiplier 1 y-axis offset 0

E k R q kq

E k R q

Plotting k-weights

0 1 2 3 kw

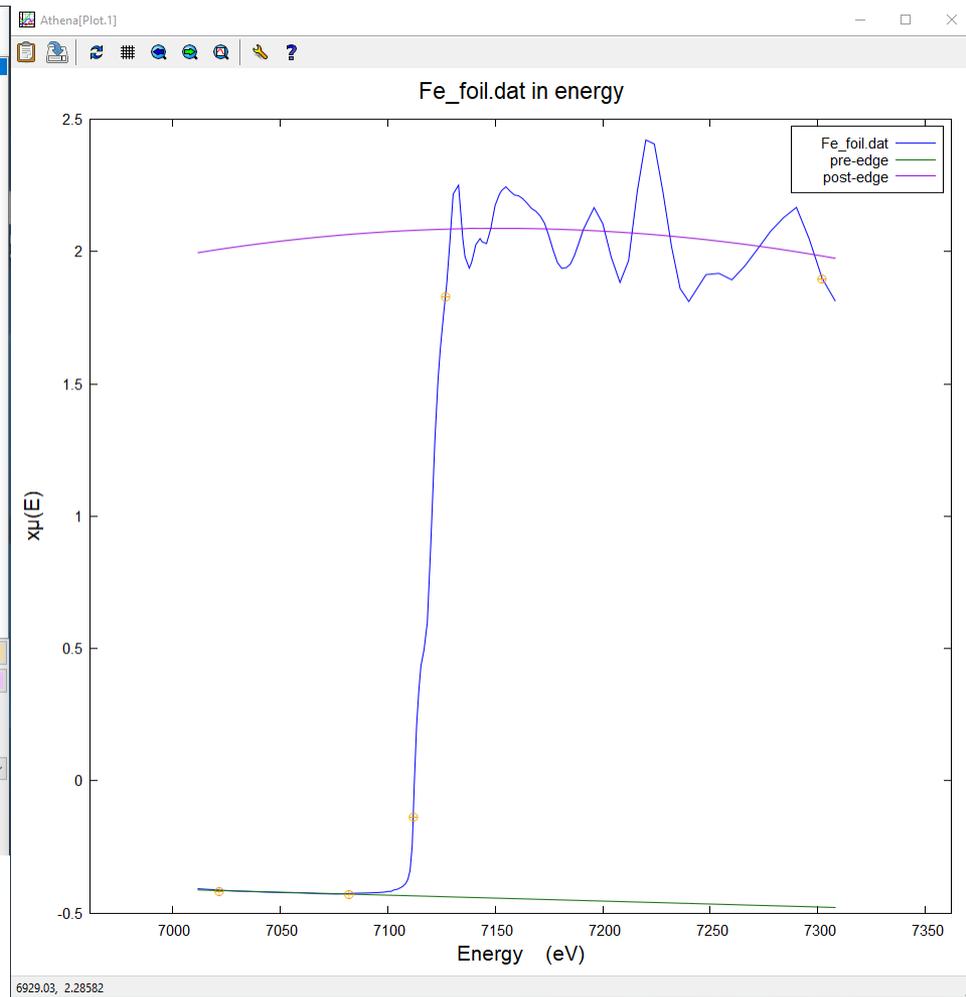
Plot in energy

$\mu(E)$ $\mu(E)$

Background

pre-edge line

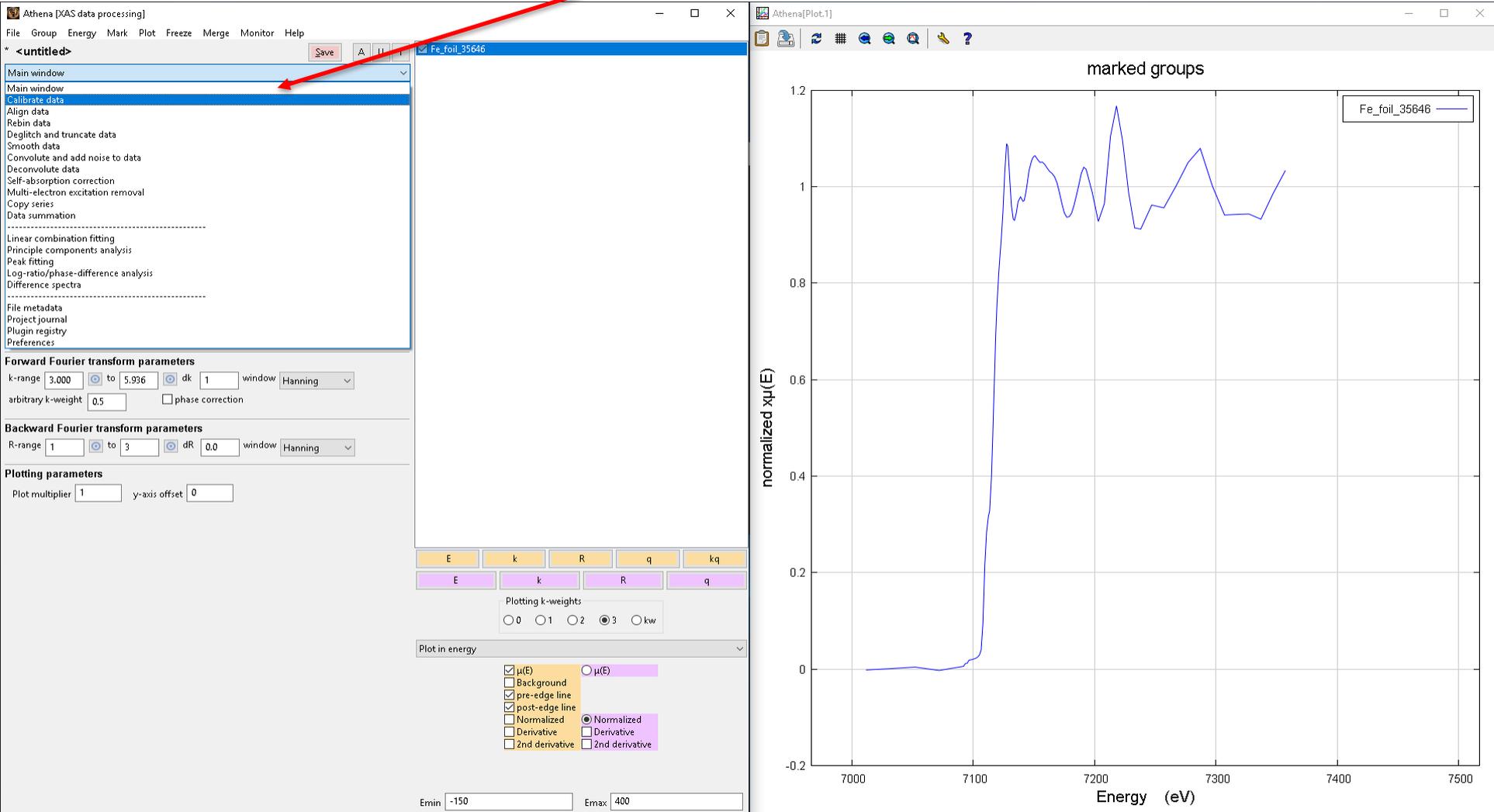
post-edge line



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

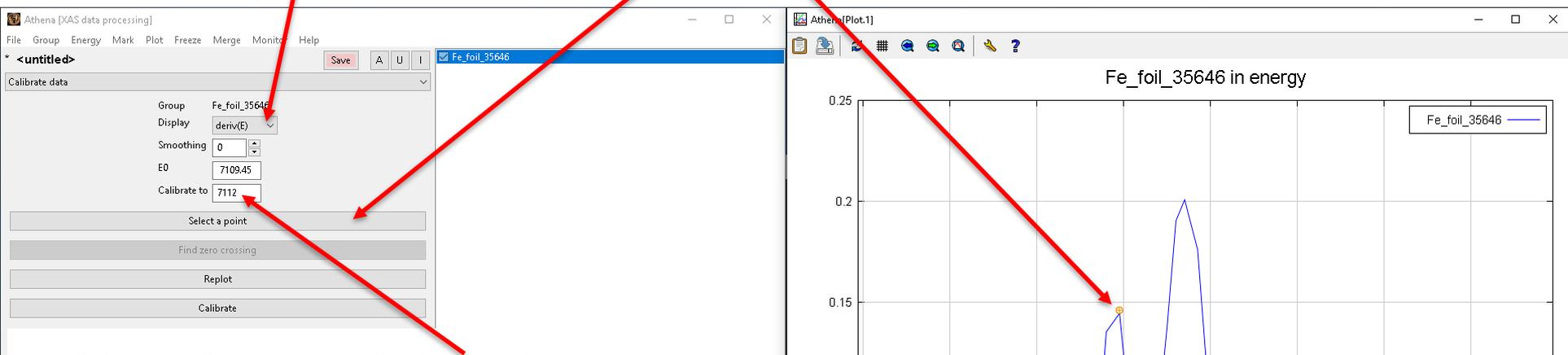
Data analysis by Athena

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



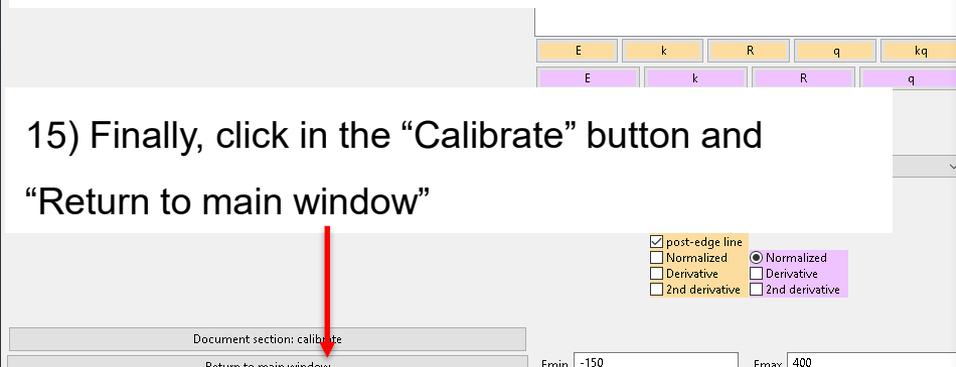
Data analysis by Athena

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-, L α -, L β -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

File Group Energy Mark Plot Freeze Merge Monitor Help

* <untitled> Save A U I

Main window Fe_foil_35646 Datatype: xmu Freeze

Current group: Fe_foil_35646

File: \\Processing Data\191011_sp20627_restagedData\NES\Fe_foil_35646

Element: 26: Iron Edge: k Energy shift: 2.550 Importance: 1

Normalization and background removal parameters

E0: 7112 Normalization order: 1 2 3

Pre-edge range: -100 to -45 Flatten normalized data

Normalization range: 40,963 to 190 Edge step: 2,581,7997 fix

Rbkg: 1.0 k-weight: 2 Spline clamps: low None high Strong

Spline range in k: 0 to 7,996,0000

Spline range in E: 0 to 239,95315

Standard: None Energy-dependent normalization

Forward Fourier transform parameters

k-range: 3,000 to 5,936 dk: 1 window: Hanning

arbitrary k-weight: 0,5 phase correction

Backward Fourier transform parameters

R-range: 1 to 3 dR: 0,0 window: Hanning

Plotting parameters

Plot multiplier: 1 y-axis offset: 0

Plot in energy

$\mu(E)$ $\mu(E)$

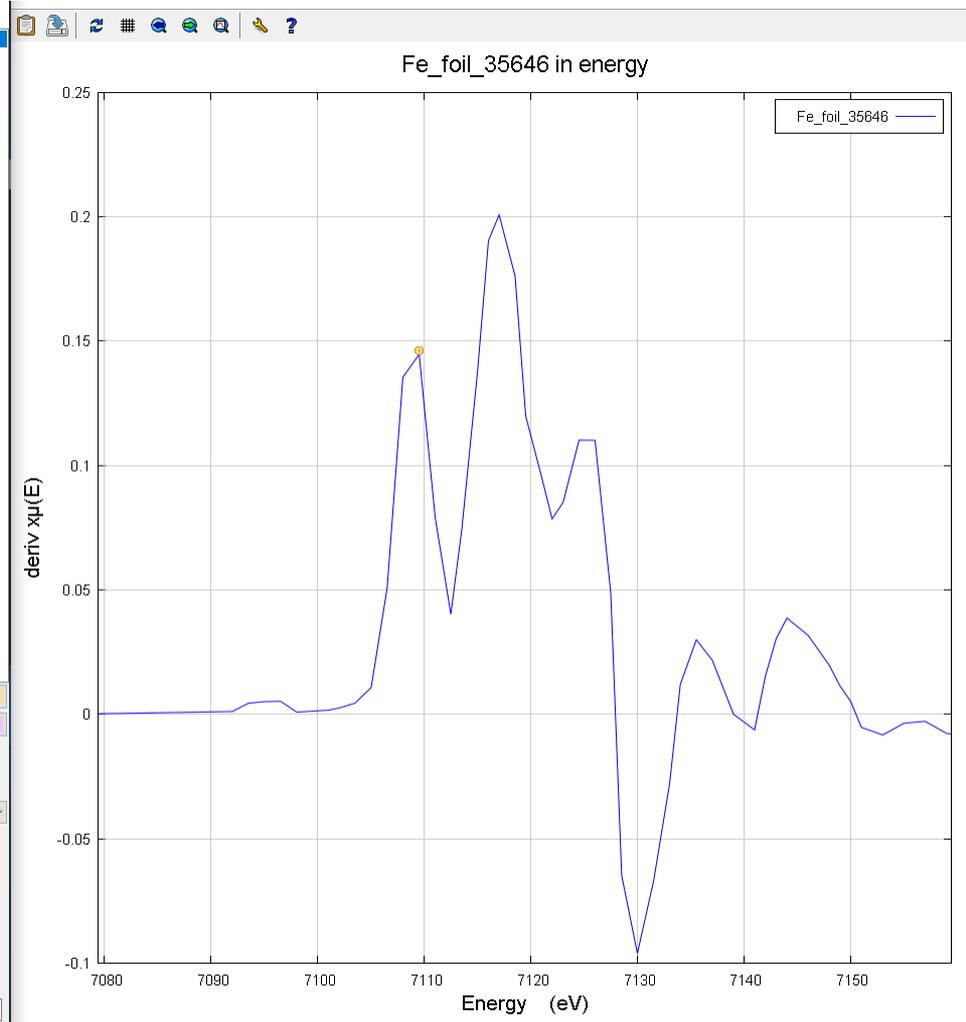
Background pre-edge line post-edge line

Normalized Normalized

Derivative Derivative

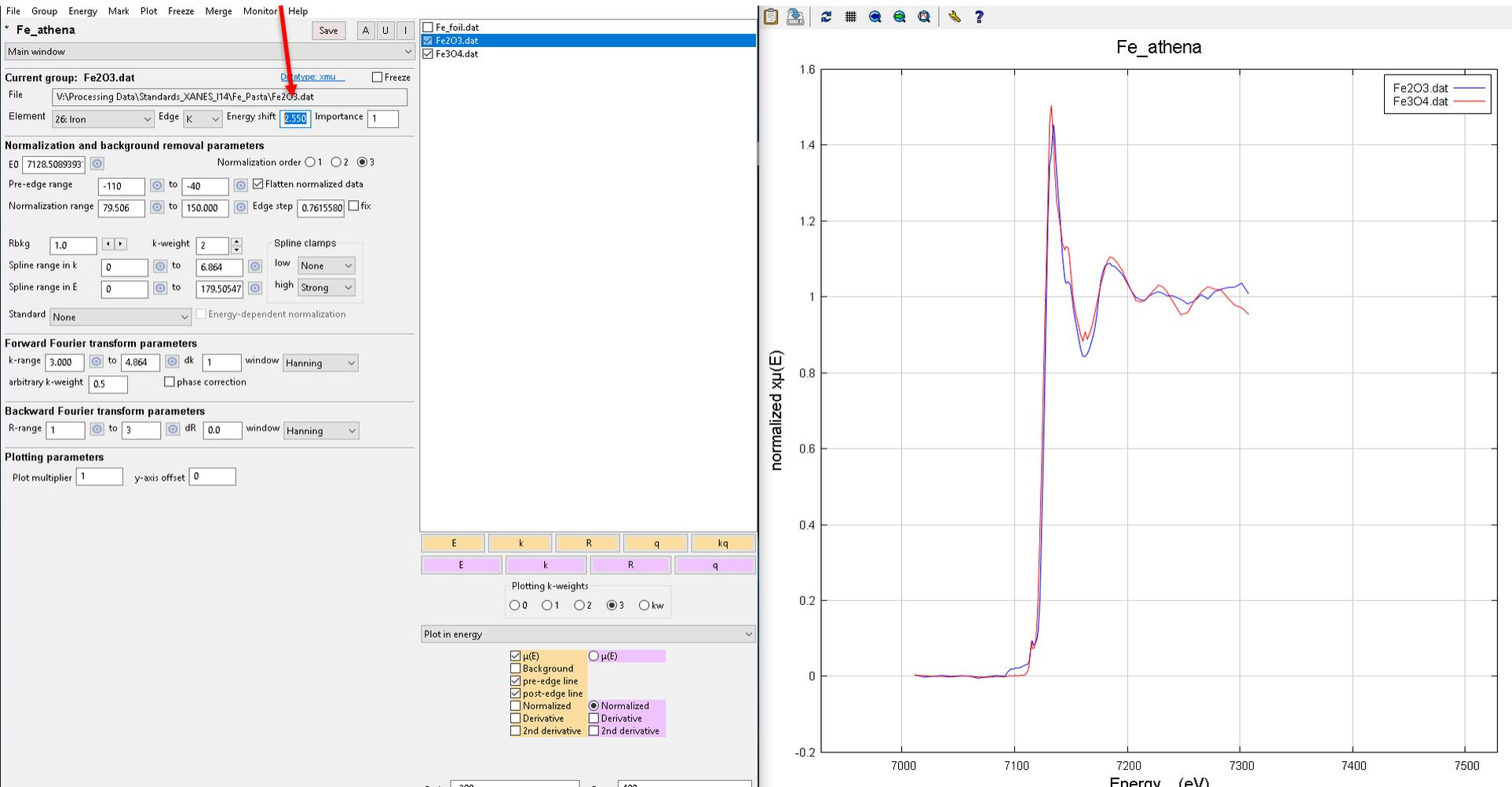
2nd derivative 2nd derivative

Emin: -150 Emax: 400



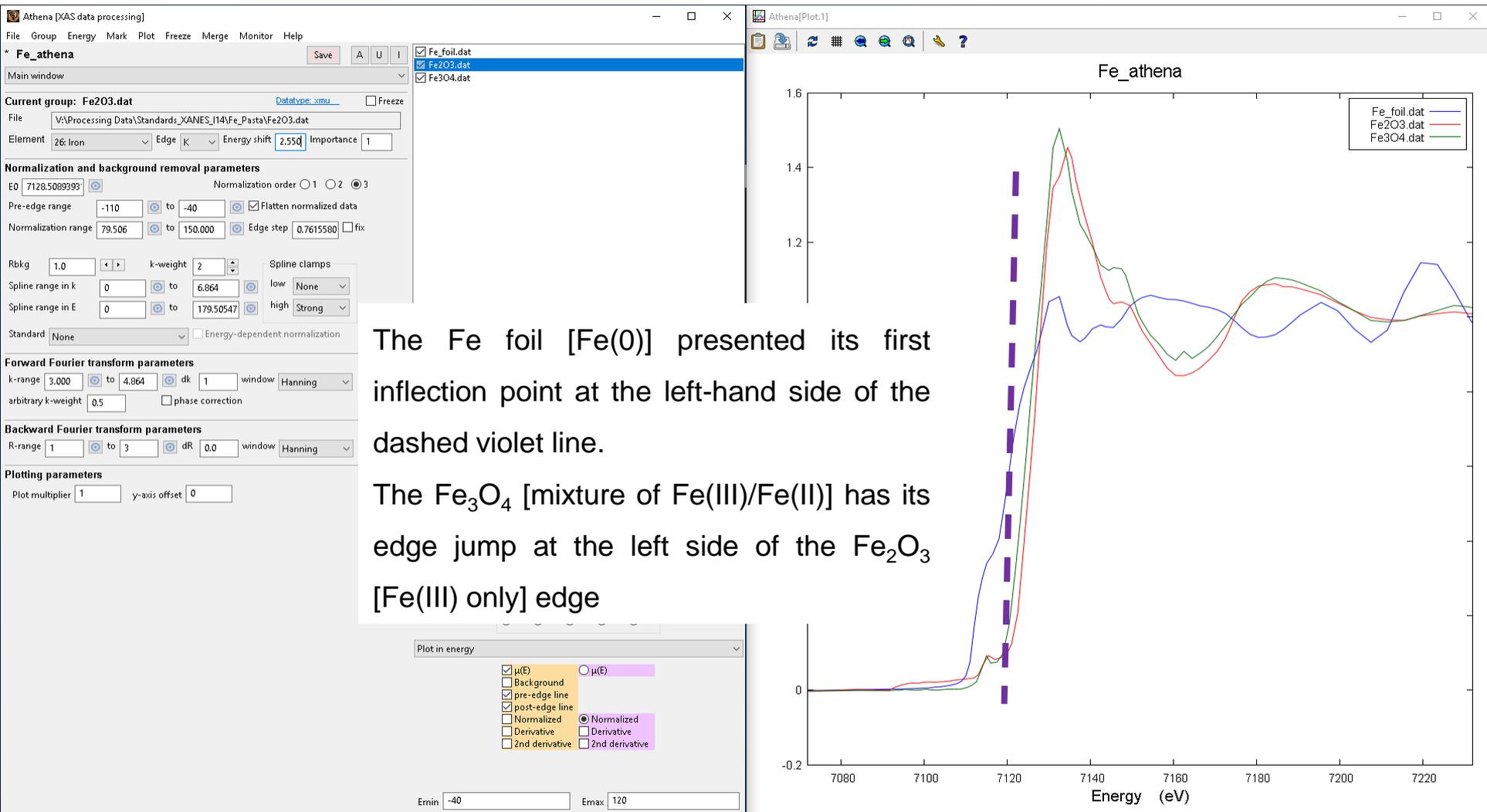
Data analysis by Athena

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



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)

The screenshot displays the Athena [XAS data processing] window. The main window shows various processing parameters such as 'Current group', 'Element' (Hydrogen), 'Edge' (K), and 'Normalization and background removal parameters'. A 'Column selection' dialog box is open, showing a list of traces extracted from the plot. The 'Data type' dropdown is set to 'xanes', and the 'Energy' dropdown is set to 'μ(E)'. The 'Plotting k-weights' section shows 'Normalized' selected. A red arrow points from the 'xanes' option in the 'Data type' dropdown to a text box containing '21) Set "xanes" as Data type'. Another red arrow points from the 'μ(E)' option in the 'Energy' dropdown to a text box containing '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)'. The plot shows a blue line representing the XAS data, with a sharp peak at approximately 7100 eV. The x-axis is labeled 'Energy (eV)' and ranges from 7050 to 7350. The y-axis ranges from 0 to 5000.

21) Set "xanes" as Data type

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)

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)

The screenshot displays the Athena software interface. On the left, the 'Athena [XAS data processing]' window shows various configuration panels. The 'Current group' is 'plotdata_Traces2'. The 'Element' is set to '26: Iron' and 'Energy shift' is '2,550'. The 'Normalization and background removal parameters' section includes 'E0' at 119.9647709, 'Pre-edge range' from -100 to -30.000, and 'Normalization range' from 33 to 200. The 'Forward Fourier transform parameters' section shows 'k-range' from 3.000 to 5.855. The 'Plotting parameters' section has 'Plot multiplier' at 1 and 'y-axis offset' at 0. The 'Athena[Plot.1]' window on the right shows a plot titled 'marked groups' with two traces: 'plotdata_Traces1' (blue) and 'plotdata_Traces2' (red). The y-axis is labeled 'E' and ranges from 0.8 to 1.4. A red arrow points from the text '22) Different regions...' to the 'Energy shift' field in the Athena window. Another red arrow points from the text '23) Remember to add the calculated energy shift...' to the 'Energy shift' field. A third red arrow points from the text '24) Later, it is advisable to save the XAS scans...' to the 'Save' button in the Athena window.

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_std (example), and load each sample to fit individually (File → Open → sample_athena.prj)

The screenshot displays the Athena software interface for XAS data processing. The main window is titled "Athena [XAS data processing]" and shows the "Fe_XAS_std" project. The "Current group" is set to "Fe3O4.dat". The "Element" is "26: Iron" and the "Edge" is "K". The "Energy shift" is "-0.502" and the "Importance" is "1".

The "Normalization and background removal parameters" section includes:

- EO: 7126.02850491
- Normalization order: 1
- Pre-edge range: -110 to -30.000
- Normalization range: 81.976 to 170
- Edge step: 0.7507720
- Rbkg: 1.0
- k-weight: 2
- Spline range in k: 0 to 6.911
- Spline range in E: 0 to 181.97215
- Standard: None

The "Forward Fourier transform parameters" section includes:

- k-range: 3.000 to 4.911
- arbitrary k-weight: 0.5

The "Backward Fourier transform parameters" section includes:

- R-range: 1 to 3

The "Plotting parameters" section includes:

- Plot multiplier: 1
- y-axis offset: 0

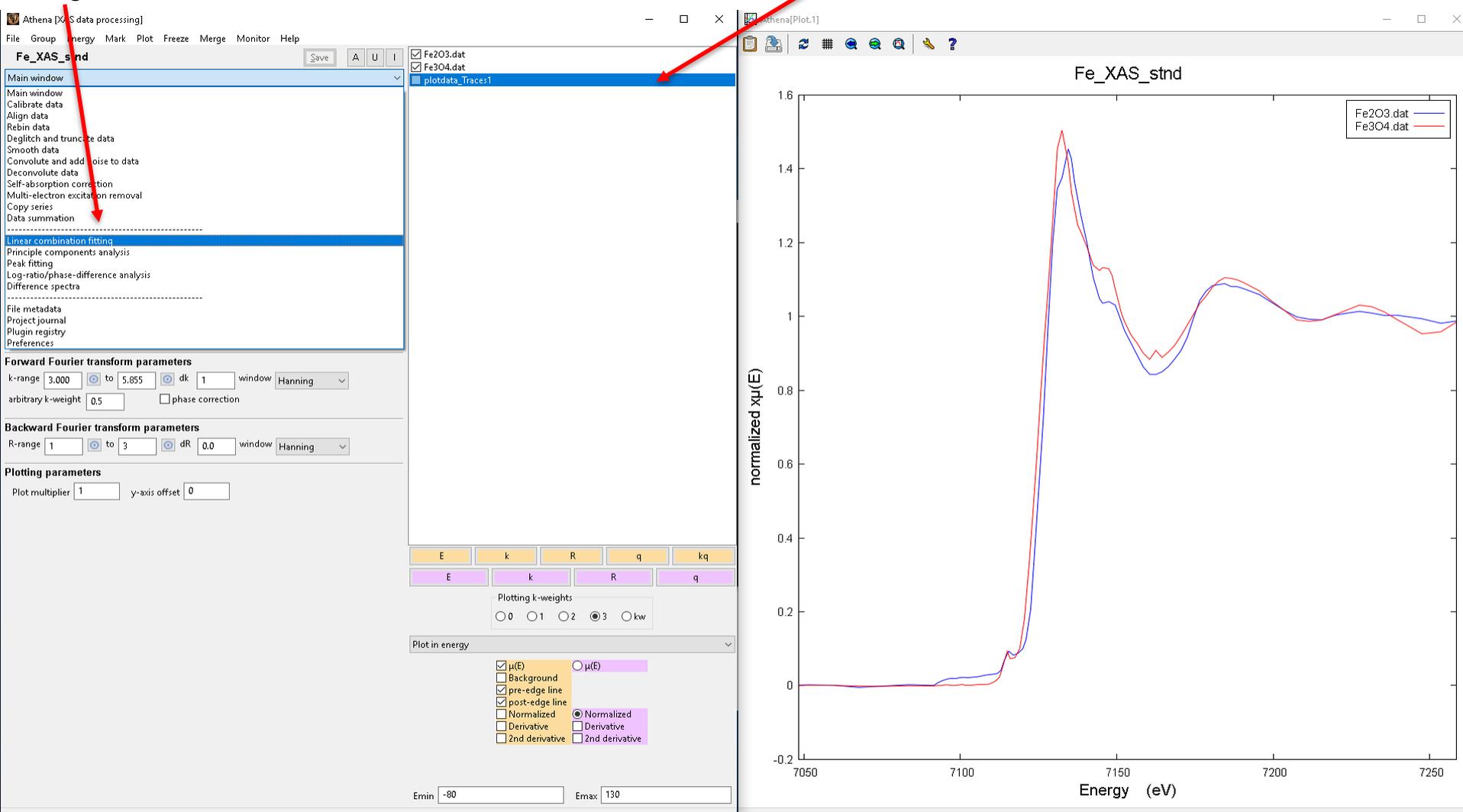
An "Athena: Import from Athena project file" dialog box is open, showing the "Data group title lines" and "Plot as" options. The "Plot as" options include $\mu(E)$, $\chi(k)$, $\chi(R)$, $\chi(q)$, $\text{Re}\chi(R)$, $\text{Re}\chi(q)$, $\text{Im}\chi(R)$, and $\text{Im}\chi(q)$. The "Import selected data" button is highlighted with a red arrow.

The main plot, titled "plotdata_Traces1 in energy", shows the XAS data for "Fe3O4.dat". The x-axis is "Energy (eV)" ranging from 7050 to 7200. The y-axis ranges from 0 to 40000. The plot shows a sharp peak at approximately 7135 eV. The plot is titled "plotdata_Traces1" and the legend indicates "plotdata_Traces1".

At the bottom of the plot, there are tabs for "E", "k", "R", and "q". The "Plotting k-weights" section includes options for 0, 1, 2, 3, and kw. The "Plot in energy" section includes checkboxes for $\mu(E)$, Background, pre-edge line, post-edge line, Normalized, Derivative, and 2nd derivative. The "Emin" is -80 and the "Emax" is 130.

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)

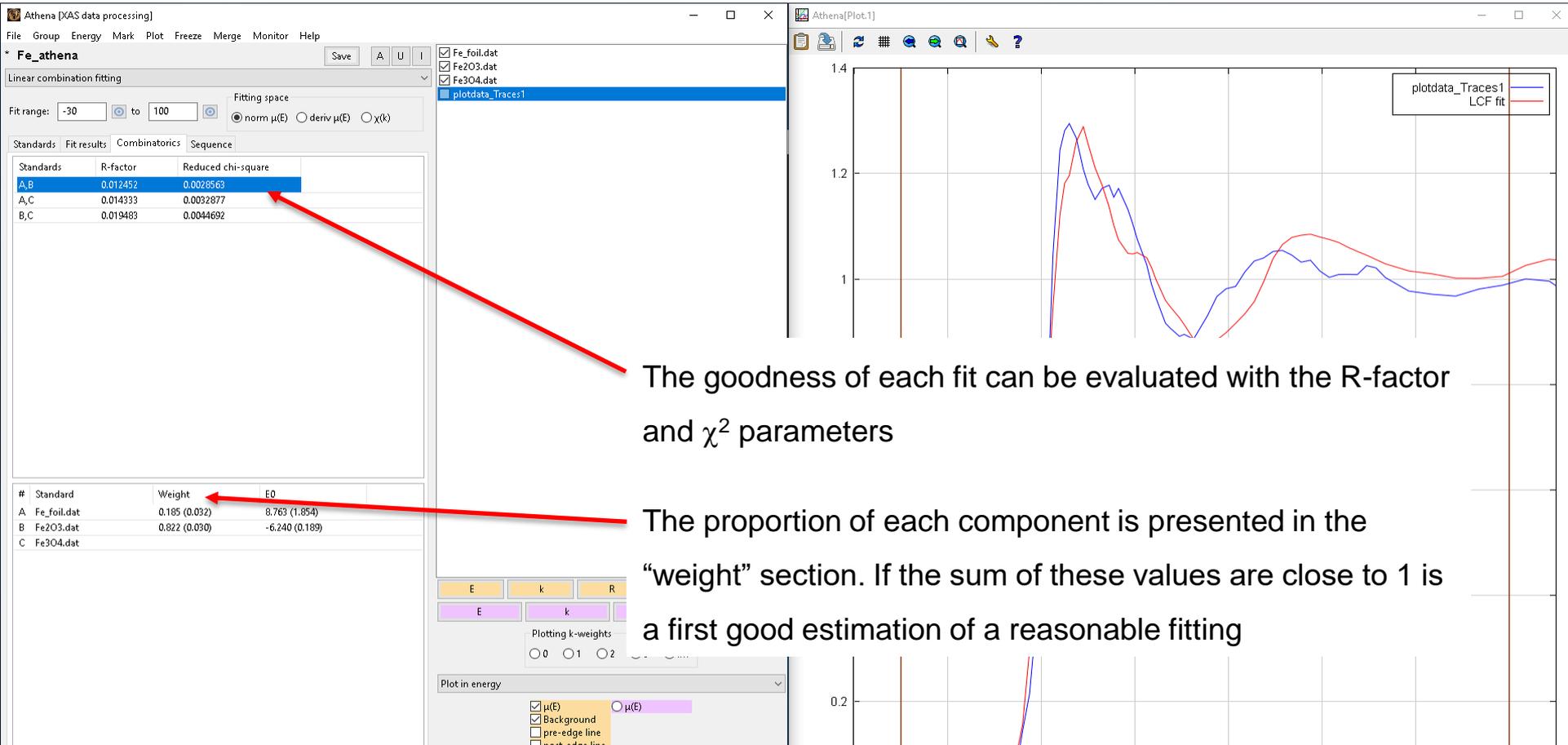
The screenshot displays the Athena software interface for XAS data processing. The main window is titled "Fe_XAS_stnd" and shows the "Linear combination fitting" (LCF) settings. The "Fit range" is set from -30 to 100. The "Standards" table lists Fe2O3.dat and Fe3O4.dat with weights of 0.500 each. The "Options" section has "Force weights to sum to 1" unselected. The "Combinatorics" section has "Use at most" set to 2. The "Actions" section has "Use marked groups" selected. The "Plotting k-weights" section has "3" selected. The "Plot in energy" section has "μ(E)" selected. The "Athena[Plot.1]" window shows the resulting plot of normalized xμ(E) vs energy, with two traces: Fe2O3.dat (blue) and Fe3O4.dat (red). A red arrow points to the "Fit this group" button in the "Actions" section.

Standards	Weight	E0	Fit E0	Required
1: Fe2O3.dat	0.500	0	<input checked="" type="checkbox"/>	<input type="checkbox"/>
2: Fe3O4.dat	0.500	0	<input checked="" type="checkbox"/>	<input type="checkbox"/>
3: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
4: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
5: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
6: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
7: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
8: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
9: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
10: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
11: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>

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 plotting, by selecting ‘Save fit as column data’

Athena [XAS data processing]

File Group Energy Mark Plot Freeze Merge Monitor Help

* Fe_athena

Linear combination fitting

Fit range: -30 to 100

Fitting space

norm $\mu(E)$ deriv $\mu(E)$ $\chi(k)$

Standards Fit results Combinatorics Sequence

LCF fit of plotdata_Traces1 as flattened $\mu(E)$ from 7089.96477034231 to 7219.96477034231

Fit included 72 data points and 4 variables, and approximately 97,573 measurements

Weights sum to 1: no

Weights forced between 0 and 1: yes

Overall e0 shift used: no

Noise added to data: 0

R-factor = 0.0124517

Chi-square = 0.19423

Reduced chi-square = 0.0028563

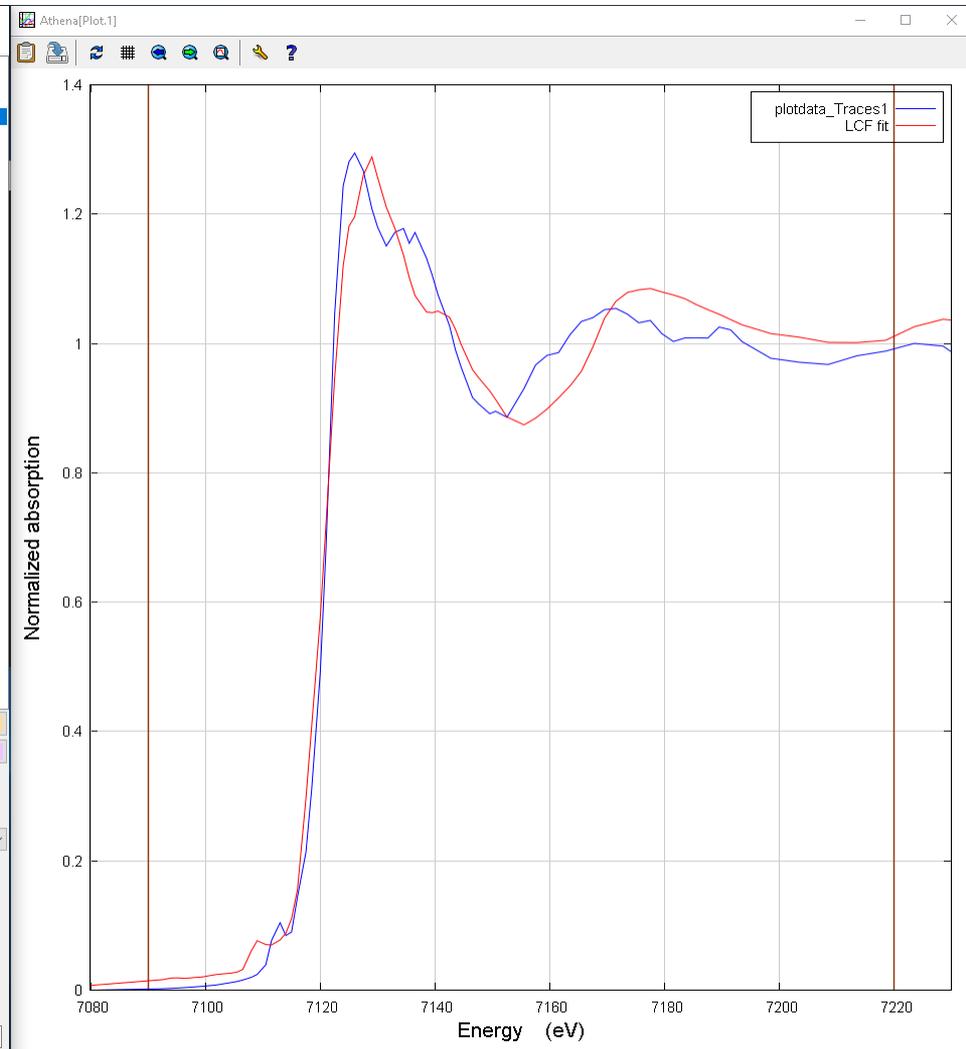
standard	weight	e0
Fe203.dat	0.822 (0.030)	-6.240 (0.189)
Fe_foil.dat	0.185 (0.032)	8.763 (1.854)
sum	1.007	

Plot data and fit

Save fit as column data

Return to main window

Emin -40 Emax 120



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)

The image shows two screenshots. On the left is the Athena 0.9.26 documentation website. The top navigation bar includes 'Athena 0.9.26 documentation » 10. Data analysis »' and 'previous | next'. The main content area is titled '10.1. Linear combination fitting' and '10.1.1. Interpreting data as a mixture of standards'. A red arrow points to the '10.1. Linear combination fitting' section in the Table of Contents on the left. The right screenshot shows the Athena software interface. The title bar reads 'Athena [XAS data processing]'. The window title is 'cyanobacteria'. The 'Linear combination fitting' panel is active, showing a 'Fit range' of -20 to 30 and 'Fitting space' options: 'norm $\mu(E)$ ' (selected), 'deriv $\mu(E)$ ', and ' $x(k)$ '. Below this is a table for standards:

Standards	Weight	E0	Fit E0	Required
1: Au Foil	0.500	0	<input type="checkbox"/>	<input type="checkbox"/>
2: Au3 Cl aq	0.500	0	<input type="checkbox"/>	<input type="checkbox"/>
3: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
4: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>
5: None	0	0	<input type="checkbox"/>	<input type="checkbox"/>

On the right side of the software window, there is a list of energy values and standard names, with '7.03' highlighted in blue. The list includes: 0.12, 2.42, 4.73, 7.03, 9.33, 20, 33, 720, Au foil, Au1 Cl, Au3 Cl aq, Au hydroxide, Au cyanide, Au thiocyanide, Au sulphide.

- ❑ 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://xafs.xrayabsorption.org/tutorials.html)

<https://vimeo.com/340202552>