

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 &
```

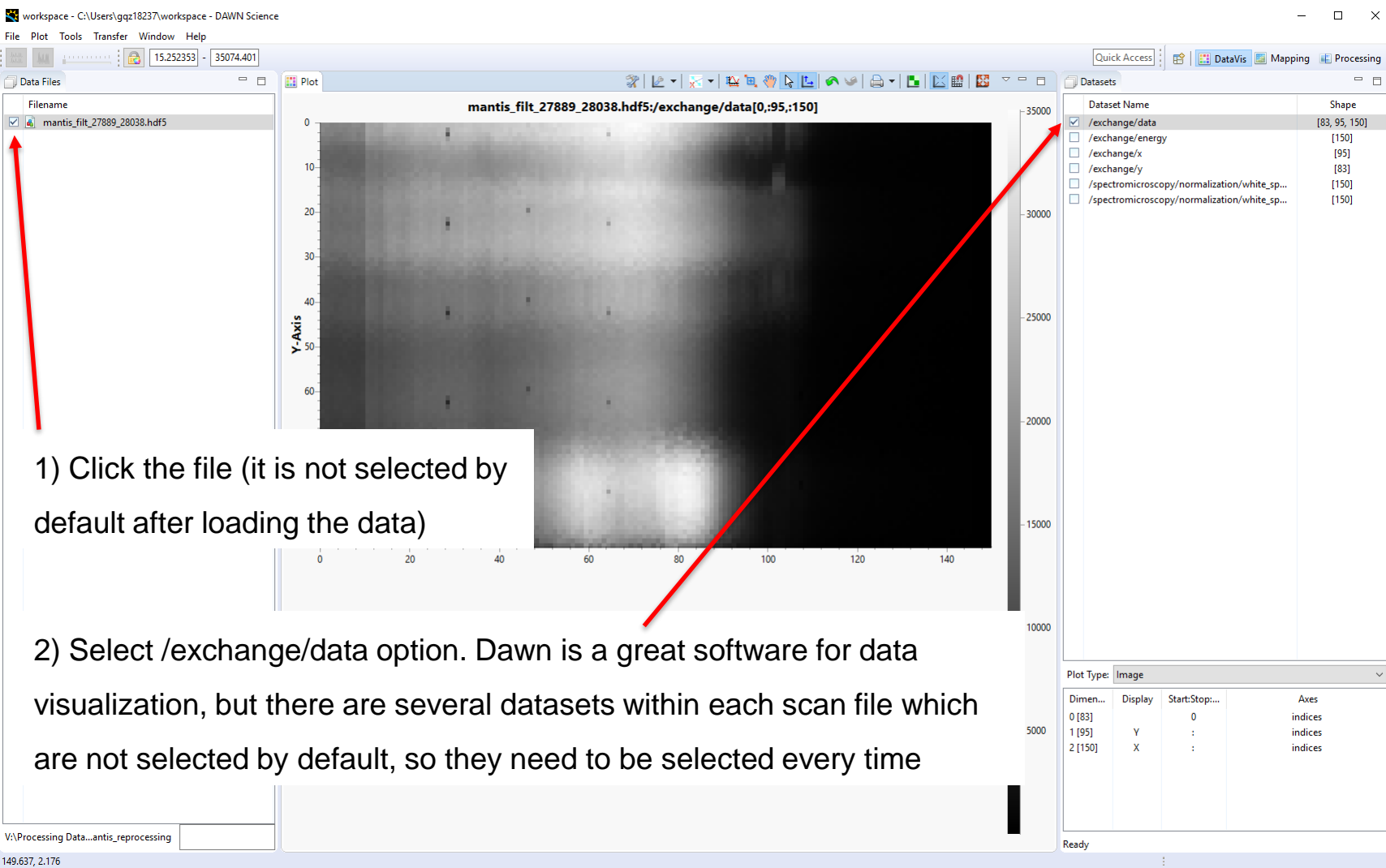
Finally, a video-tutorial on "loading DAWN for inspecting metadata from the 'i14-xxxx.nxs' files" can be found at:

<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 Science software interface. The main window displays a plot titled `mantis_filt_27889_28038.hdf5:/exchange/data[0.:95.:150]`. The plot shows a grayscale image with a vertical axis labeled `Y-Axis` ranging from 0 to 60 and a horizontal axis ranging from 0 to 140. A red arrow points from the `File` menu to the `mantis_filt_27889_28038.hdf5` file in the `Data Files` panel. Another red arrow points from the `Plot` panel to the `/exchange/data` dataset in the `Datasets` panel.

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

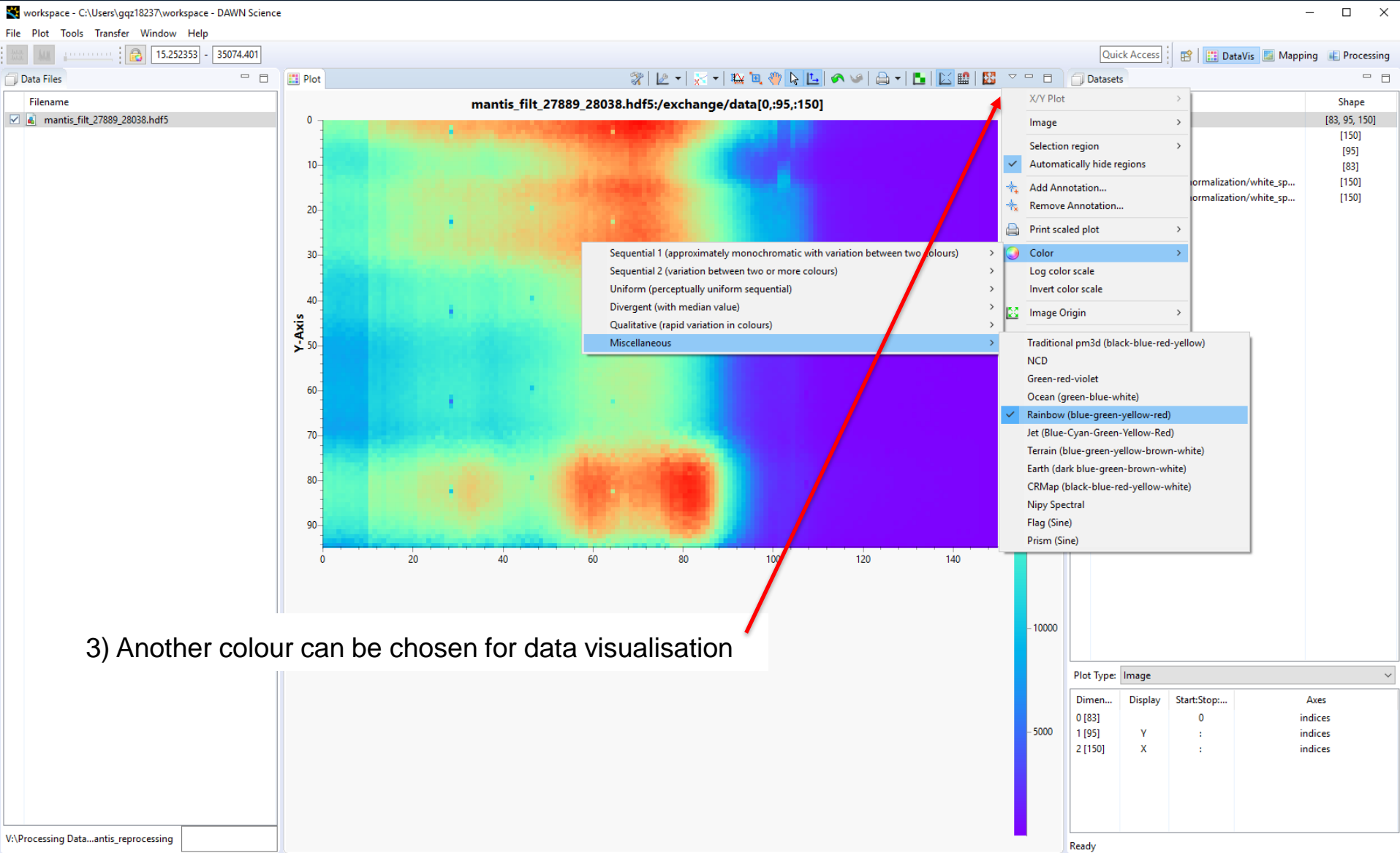
2) Select `/exchange/data` option. Dawn is a great software for data visualization, but there are several datasets within each scan file which are not selected by default, so they need to be selected every time

Dataset Name	Shape
<input checked="" type="checkbox"/> <code>/exchange/data</code>	[83, 95, 150]
<input type="checkbox"/> <code>/exchange/energy</code>	[150]
<input type="checkbox"/> <code>/exchange/x</code>	[95]
<input type="checkbox"/> <code>/exchange/y</code>	[83]
<input type="checkbox"/> <code>/spectromicroscopy/normalization/white_sp...</code>	[150]
<input type="checkbox"/> <code>/spectromicroscopy/normalization/white_sp...</code>	[150]

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

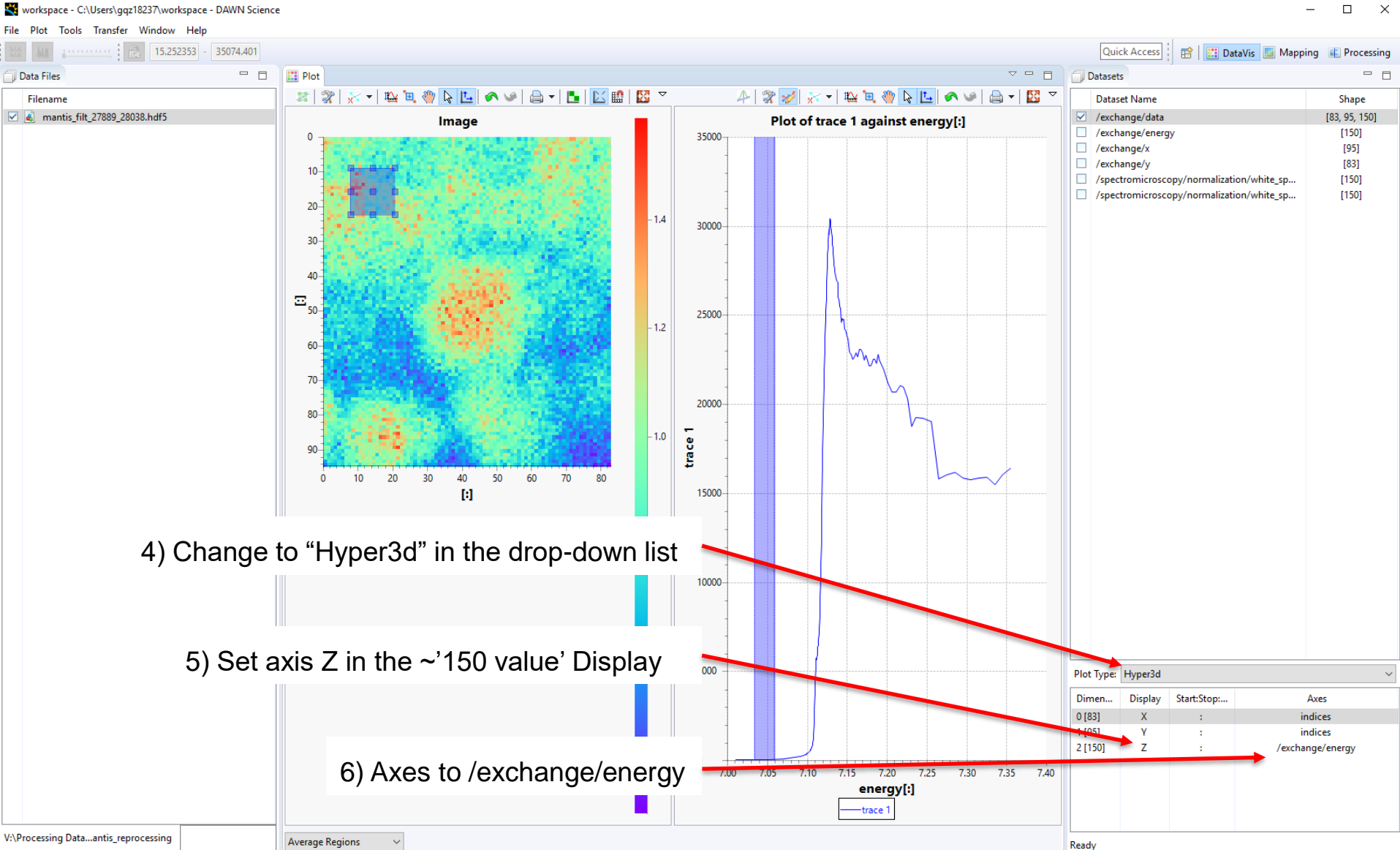
Ready

Visualisation of the data by DAWN



3) Another colour can be chosen for data visualisation

Visualisation of the data by DAWN

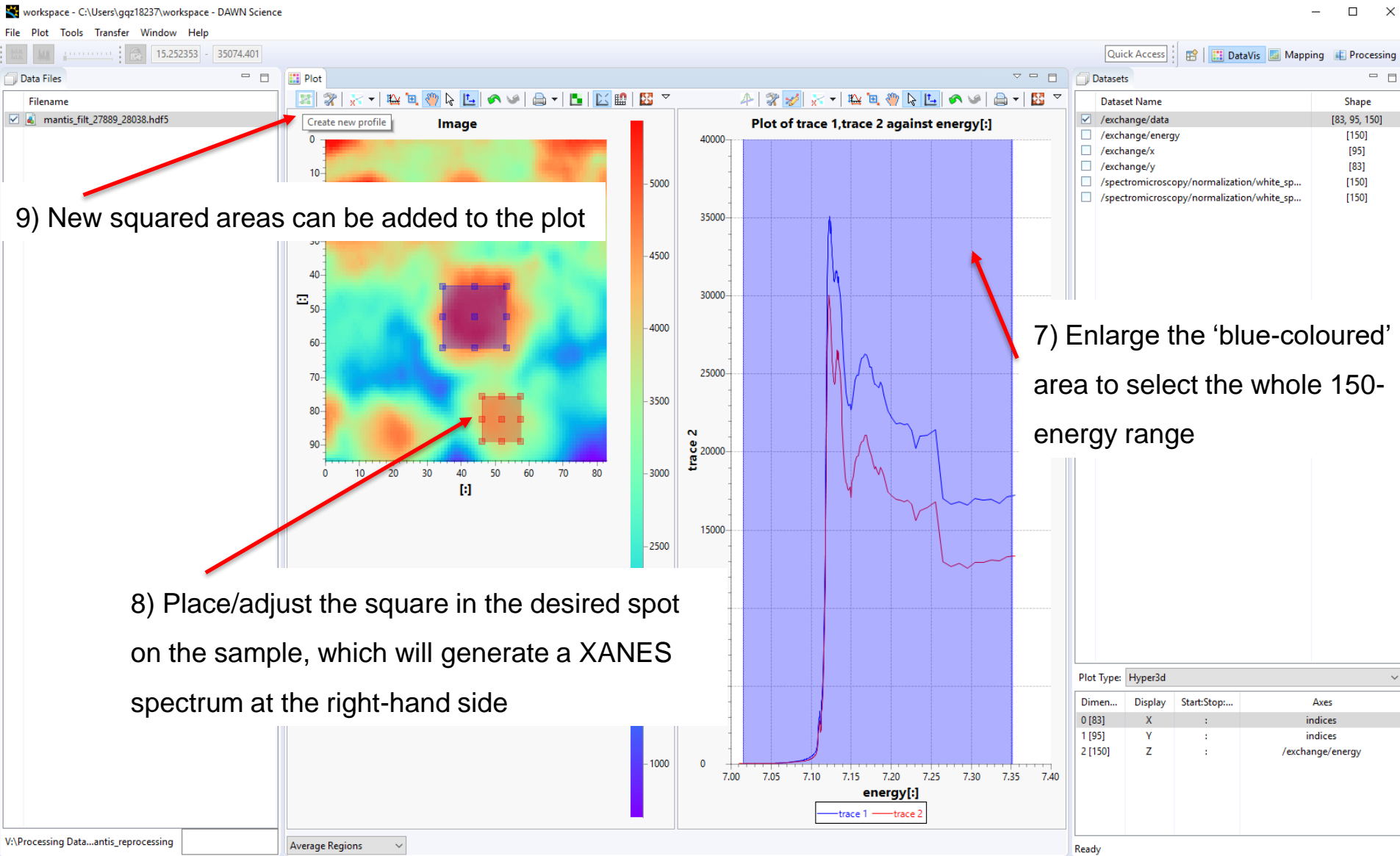


4) Change to "Hyper3d" in the drop-down list

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

6) Axes to /exchange/energy

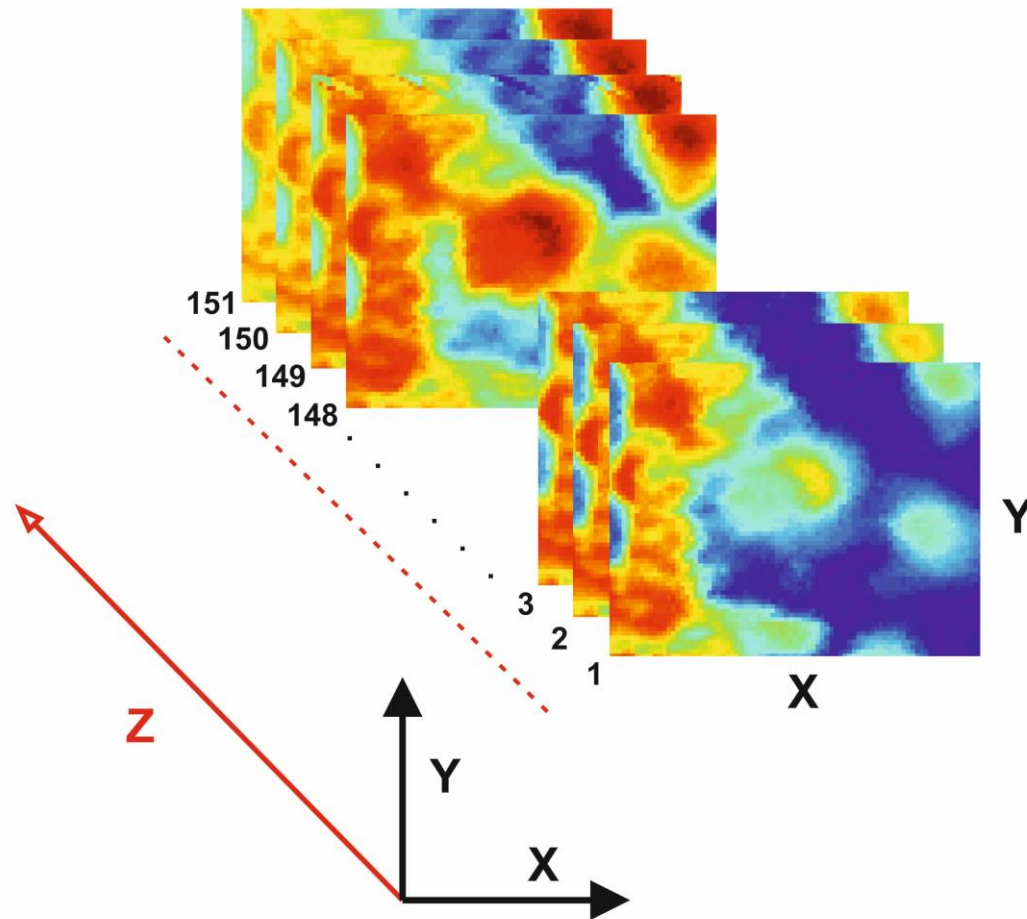
Visualisation of the data by DAWN



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 red arrow points from the 'Image' plot to a context menu that is open over the 'Plot of trace 1, trace 2 against energy' plot. The context menu includes options like 'Save screenshot as...', 'Copy to clip-board', 'Print plot', and 'Export plot data to tif/dat/csv...'. Below this, another red arrow points to an 'Export Data' dialog box. The dialog box has a 'File' field with the path 'C:\Users\gqz18237\plotdata.dat', an 'Overwrite file if it already exists' checkbox, and radio buttons for 'Format' (selected 'dat') and 'Number of files' (selected 'single'). There is also a 'single x column' checkbox. A red arrow points to the 'Format' radio buttons. In the background, another plot shows a heatmap with a region of interest outlined in red. A 'Plot Type' dropdown is set to 'Hyper3d', and 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/>



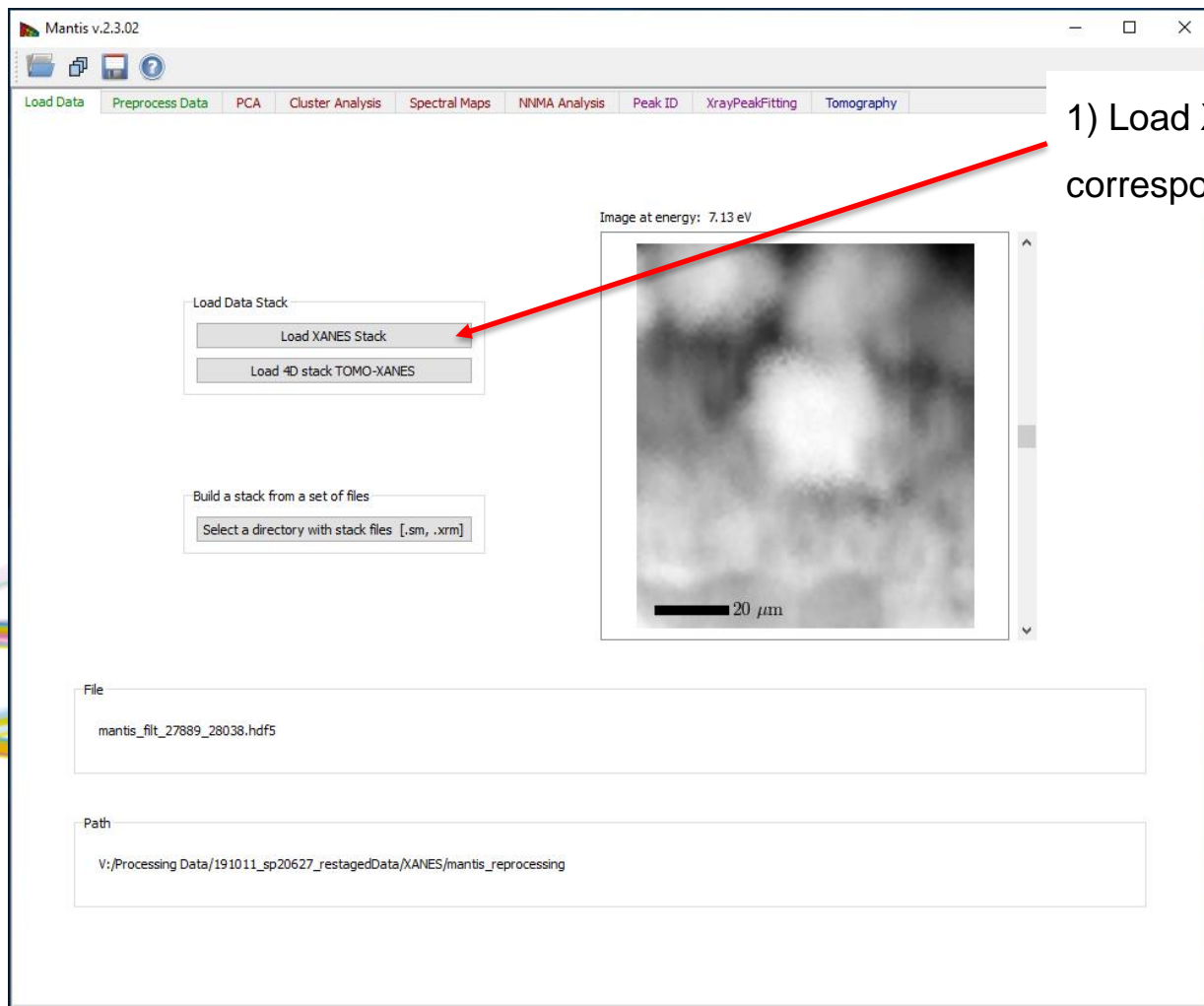
MantiS

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 options for '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' information (mantis_fit_27889_28038.hdf5), 'Image' settings (Flux selected, Scalebar checked), and 'Display settings' (Minimum: 0%, Maximum: 100%, Gamma: 1.00). The main view area 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 in the foreground, featuring a 'This Image' section with 'Set as Reference Image', 'Save Reference Image', 'Load Reference Image', and 'Remove energy from stack' buttons. It also has radio buttons for 'Automatic Alignment' (selected) and 'Manual Alignment'. The 'Automatic Alignment' section includes 'Calculate image shifts', 'Select subregion on reference', and 'Remove subregion selection' buttons, along with 'Edge Enhancement' (unchecked) and 'Prewitt' (selected) options. A 'Max shift [pixels]' dropdown is set to 0, and 'Show Cross-correlation' is unchecked. The 'Manual Alignment' section has 'Pick a point on reference image', 'This image: click on same point', and 'Apply manual shifts' buttons, with 'X manual shift' and 'Y manual shift' fields. At the bottom of the dialog are 'Save image shifts plot', 'Save image shifts', 'Load image shifts', 'Crop aligned images', 'Apply Alignment', and 'Dismiss' buttons. The dialog also displays two image windows: 'Image at energy: 7.15 eV' and 'Reference image', with 'X shift: -0.53 pixels' and 'Y shift: -0.63 pixels' shown below them. A plot of 'Image shifts' (Shifts in x, y-green [pixels] vs Photon Energy [eV]) and a 'Cross-correlation' plot are also visible.

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 top menu bar includes 'Load Data', 'Preprocess Data', 'PCA', 'Cluster Analysis', 'Spectral Maps', 'NNMA Analysis', 'Peak ID', 'XrayPeakFitting', and 'Tomography'. The main workspace is divided into several panels: 'Preprocess' (Align stack..., Limit energy range..., Clip to subregion..., Dark signal subtraction..., Save processed stack), 'Normalize' (I0 from file..., I0 from histogram..., Show I0..., Use pre-normalized data, Load Reference Images, Reset I0, Save OD data), 'Display' (File: mantis_fit_27889_28038.hdf5, Image: Flux, Optical Density, Scalebar, White, Colorbar, Display settings: Minimum: 0%, Maximum: 100%, Gamma: 1.00, Play stack movie, Save images..., Despike, Reset, Color Table...), and 'Region of Interest' (Select ROI (Lasso), Reset ROI, Set ROI As I0, Save ROI Spectrum..., ROI Dose Calculation..., Spectral ROI...). A red arrow points from the 'Save images...' button in the Display panel to a 'Save' dialog box. The dialog box shows options to save individual files: '_spectrum' (checkbox), '_image' (checkbox), 'all images' (checkbox, checked), and '.tif (data)' (checkbox, checked). The 'Filename' field is empty, and the 'Path' field is '27_restagedData/XANES/mantis_reprocessing'. The 'Save' button is highlighted.

Image at energy: 7.17 eV

Spectrum at pixel [35, ...]

Optical Density

Photon Energy [eV]

20 μm

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, Play stack movie, Save images..., Despise, 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

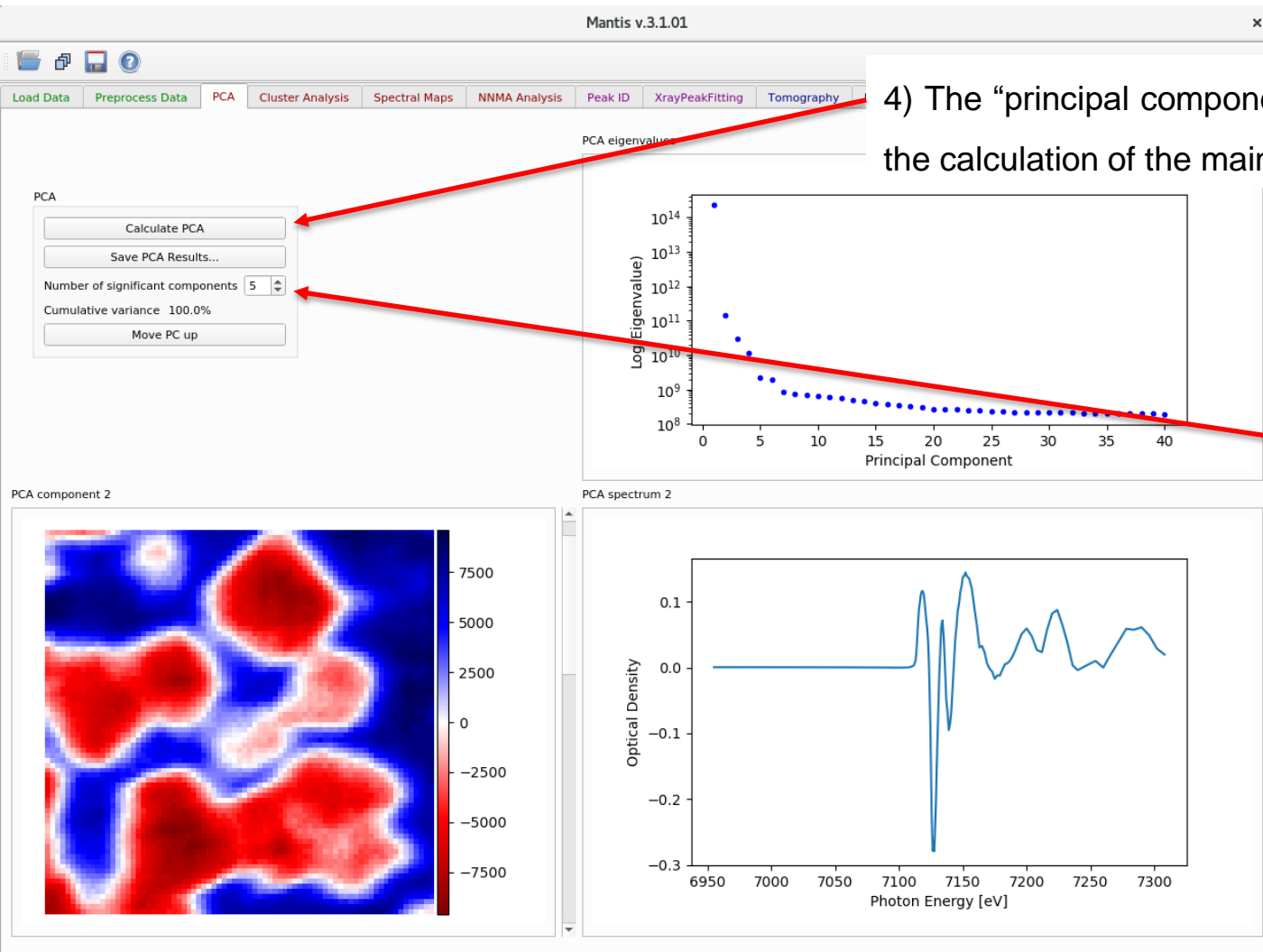
Spectrum at pixel [38, 38] or position [38.00, 38.00]

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

Mantis v.3.1.01

Cluster analysis

Calculate Clusters

Number of clusters: 5

Number of clusters found: 5

Reduce thickness effects

Divide clusters with large Sigma

PC scaling factor: 0.05

Show scatter plots...

Save CA Results...

Display

Show all spectra

Composite cluster image

Cluster 1

Cluster Error Map

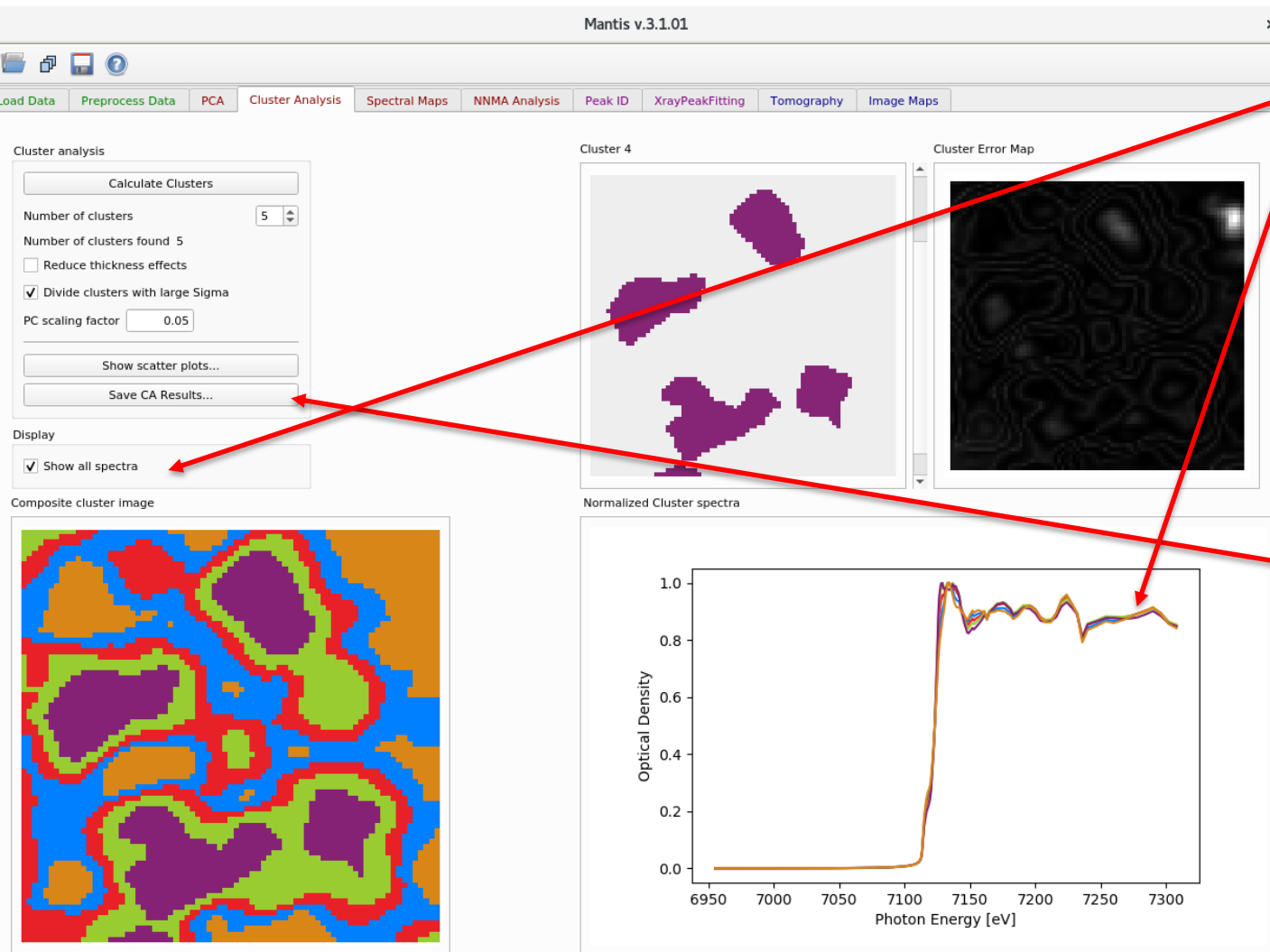
Cluster 1 spectrum

Optical Density

Photon Energy [eV]

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)

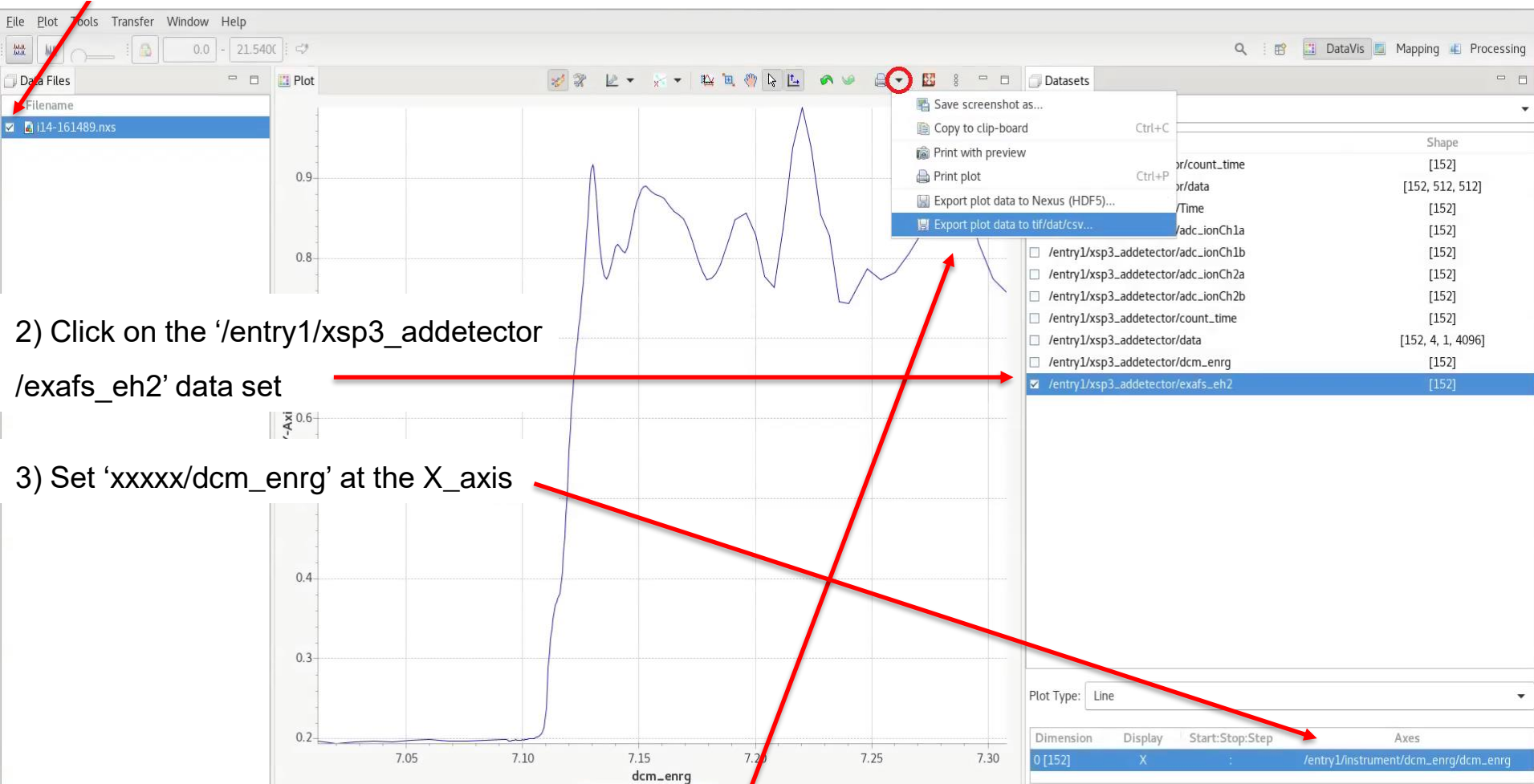
The image shows two screenshots. On the left is the Athena 0.9.26 documentation website. The top navigation bar includes 'Athena 0.9.26 documentation »' and 'previous | next'. The main content area is titled '10. Data analysis'. Below this is a 'Table Of Contents' with a list of 13 items. Red arrows point to items 2, 4, 10, and 10.1. The current Demeter version is 0.9.26. On the right is a screenshot of the Athena software interface. The window title is 'Athena [XAS data processing]'. The menu bar includes 'File', 'Group', 'Mark', 'Plot', 'Freeze', 'Merge', 'Monitor', and 'Help'. The main window is titled 'cyanobacteria'. The left sidebar lists various analysis options, with 'Linear combination fitting' highlighted. The right sidebar shows a list of parameters, with '7.03' selected.

❑ 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 displays the Athena software interface. The main window is titled 'Athena [XAS data processing]'. A dialog box titled 'Athena: Column selection' is open, showing the following settings:

- Select range: 1 2
- Energy: 1 2
- Numerator: 2
- Denominator:
- Data type: $\mu(E)$
- Energy units: eV
- Energy: tznjk.1
- $\mu(E)$: (tznjk.2) / (1)

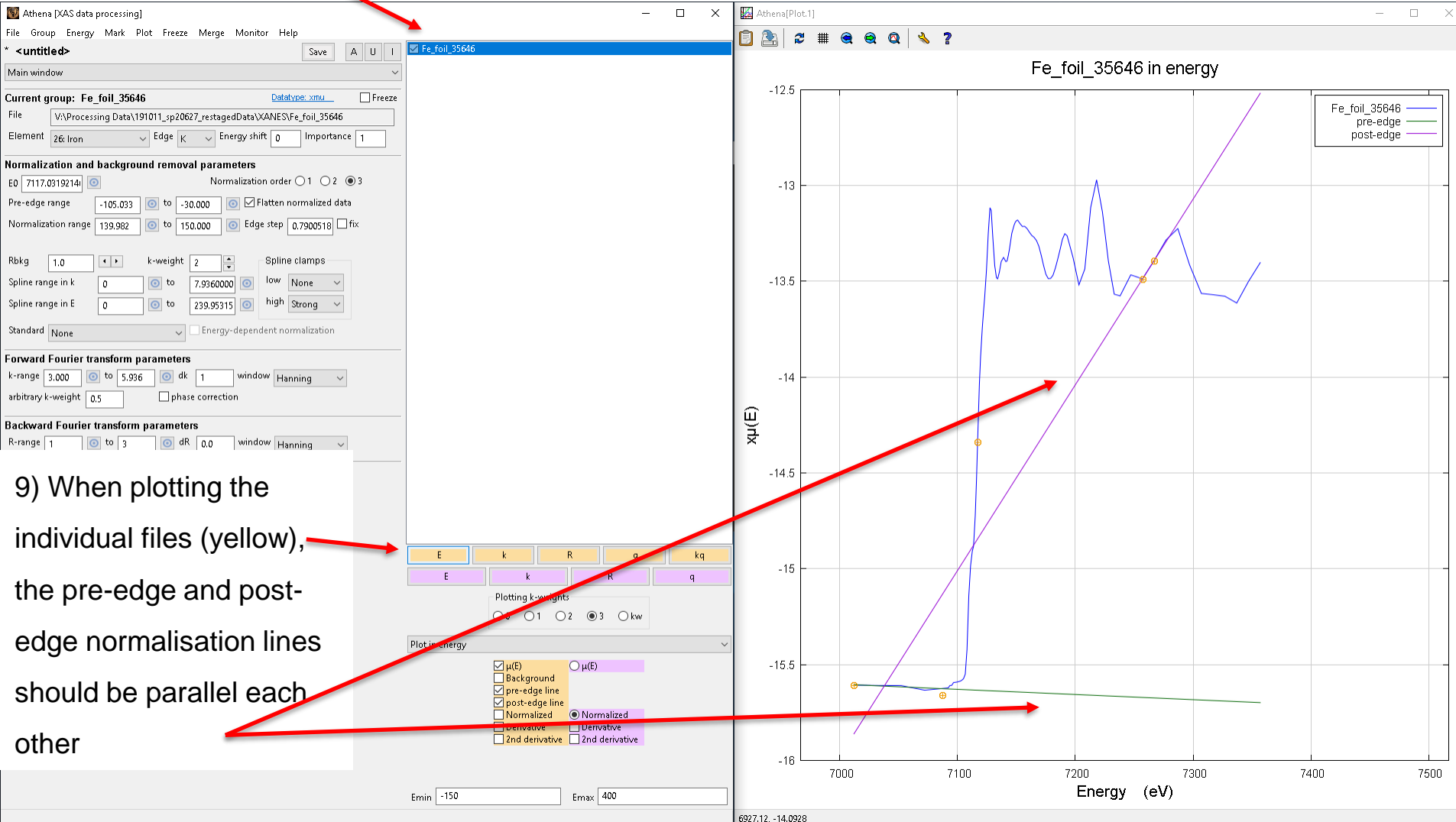
The plot window, titled 'Athena[Plot.1]', shows a plot of $\chi(E)$ versus Energy (eV) for the file 'Fe-foil_161489.dat'. The plot shows a sharp peak at approximately 7.1 eV. The y-axis ranges from 0.1 to 1.0, and the x-axis ranges from 7.05 to 7.3 eV.

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



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

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

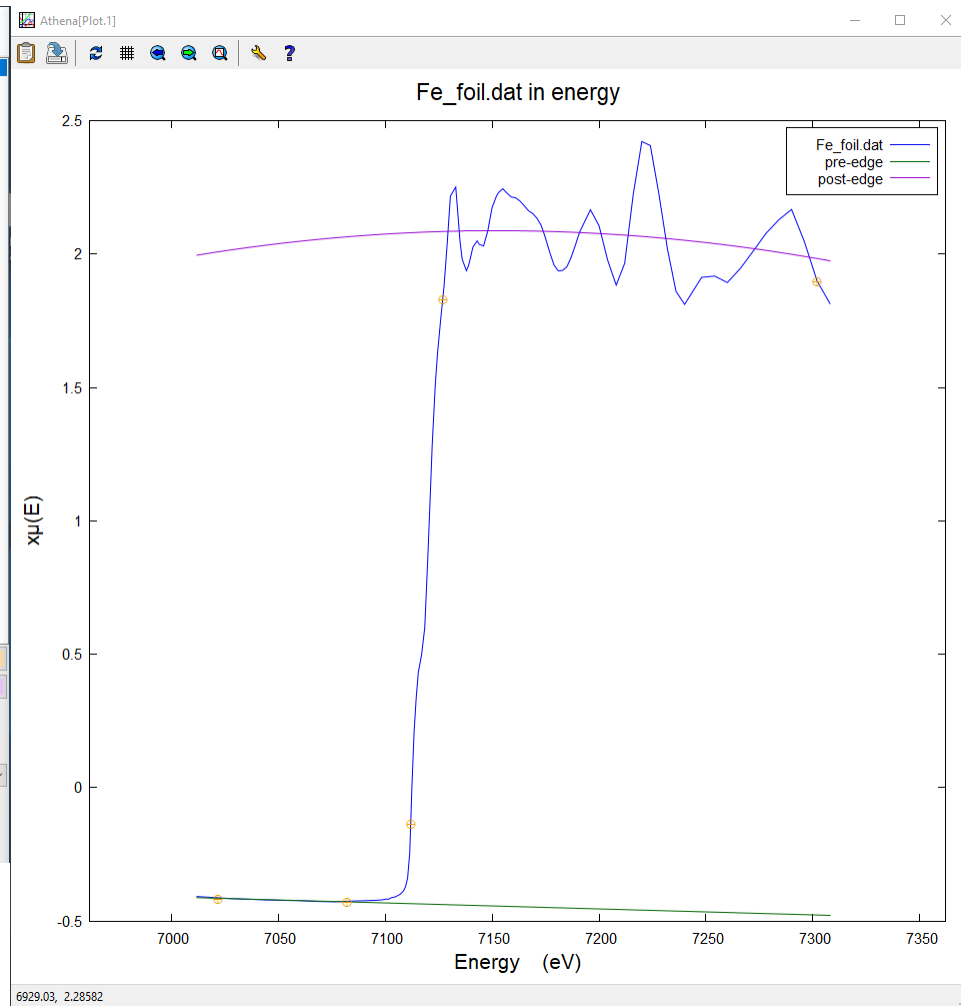
Plot in energy

$\mu(E)$ Background

pre-edge line $\mu(E)$

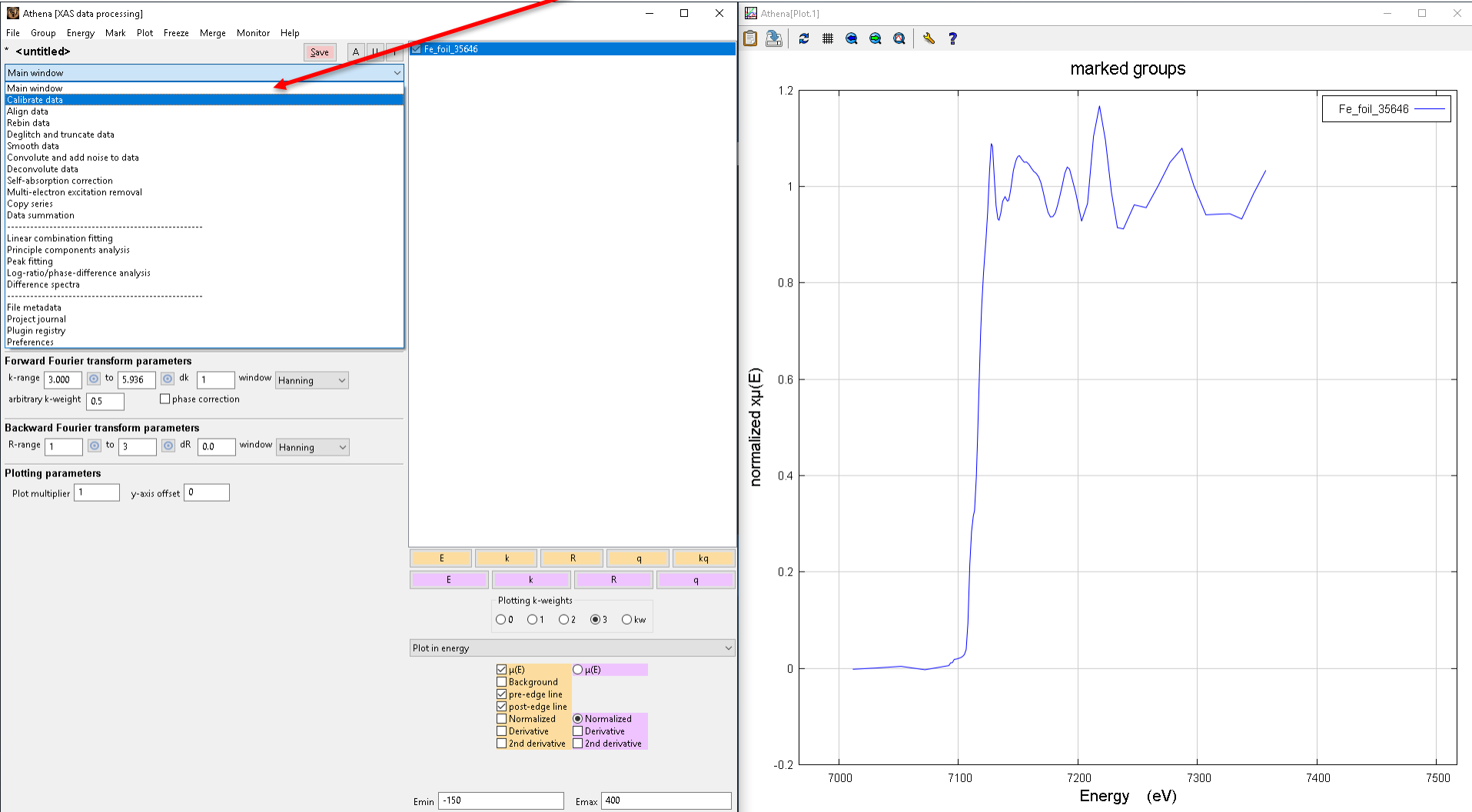
post-edge line

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



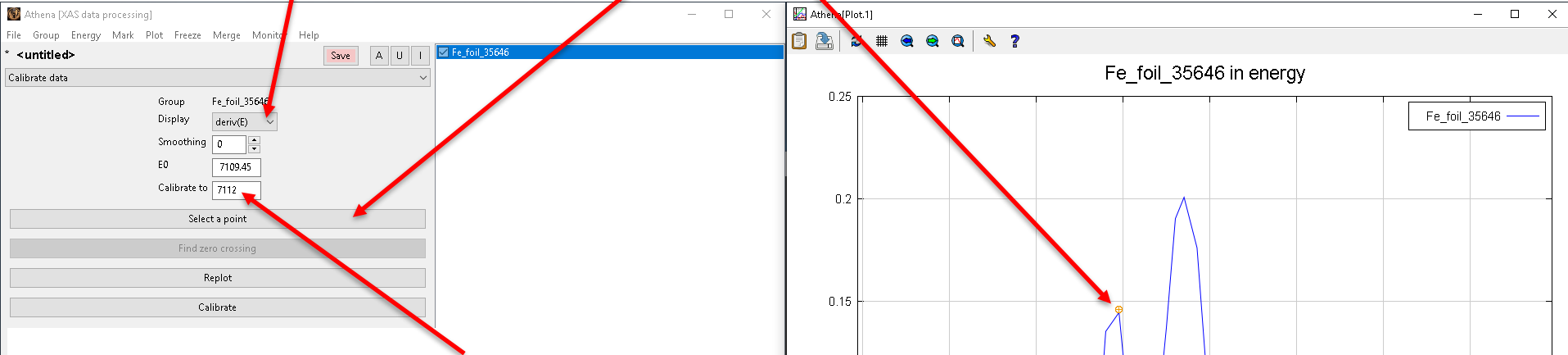
Data analysis by Athena

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



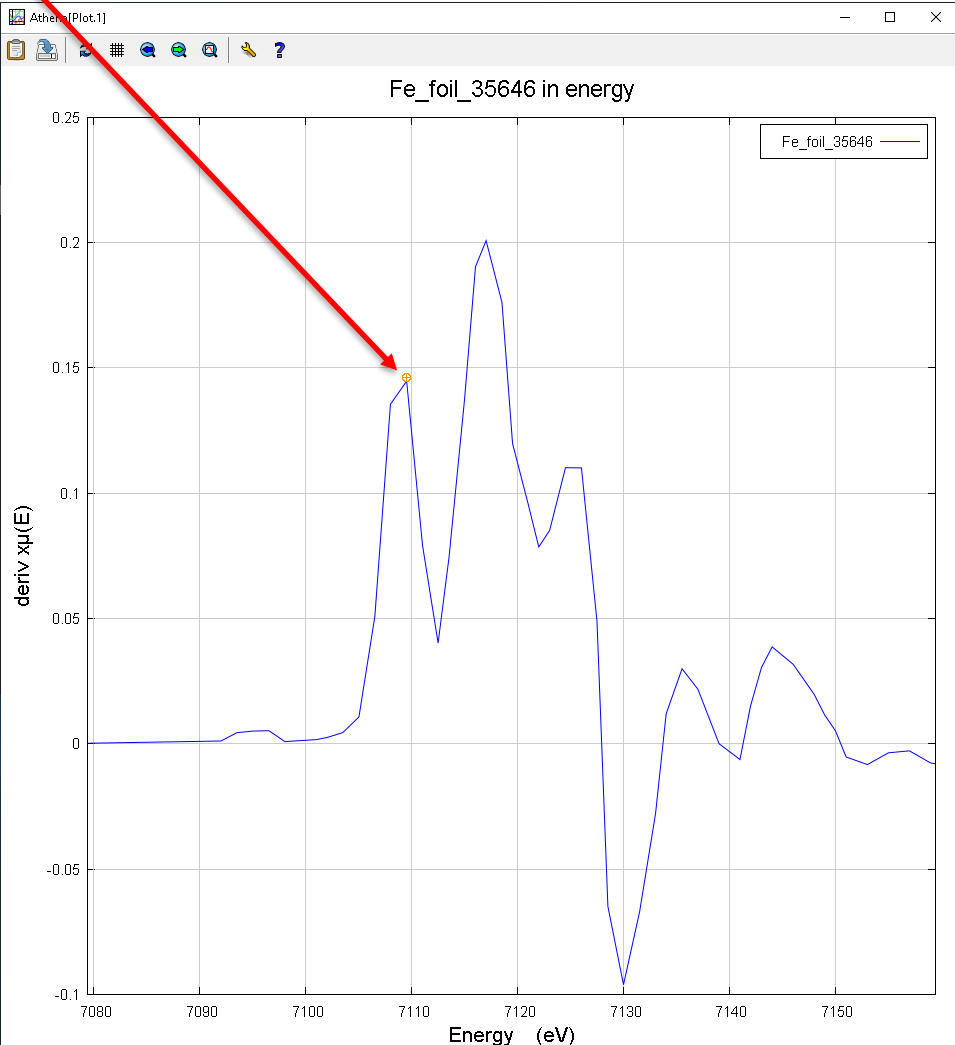
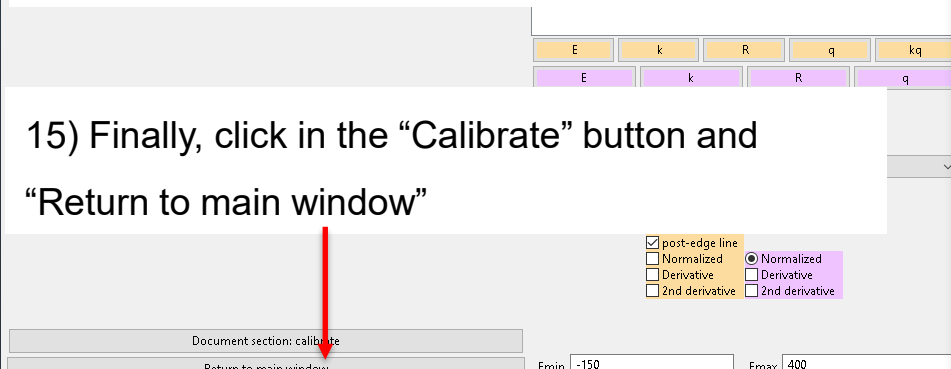
Data analysis by Athena

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: 2.5817997 fix

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

Spline range in k: 0 to 7.9960000

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

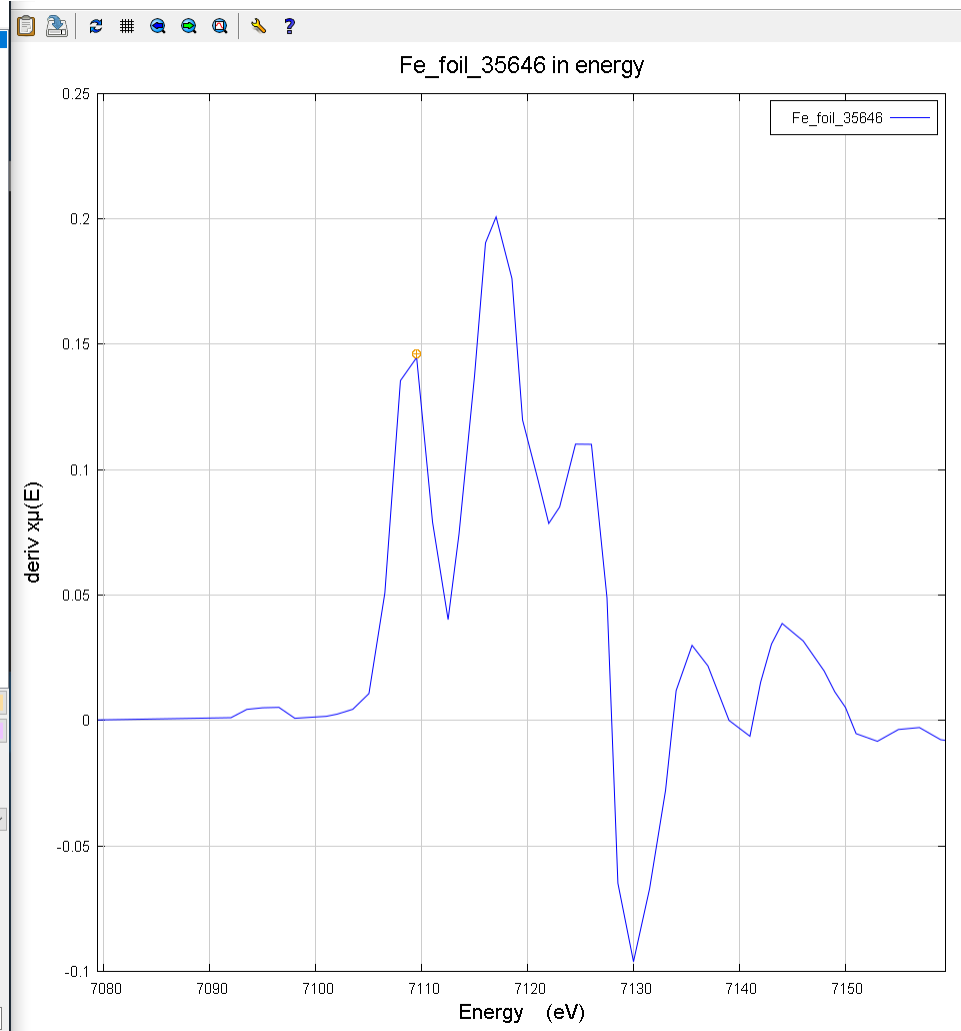
Backward Fourier transform parameters

R-range: 1 to 3 dR: 0.0 window: Hanning

Plotting parameters

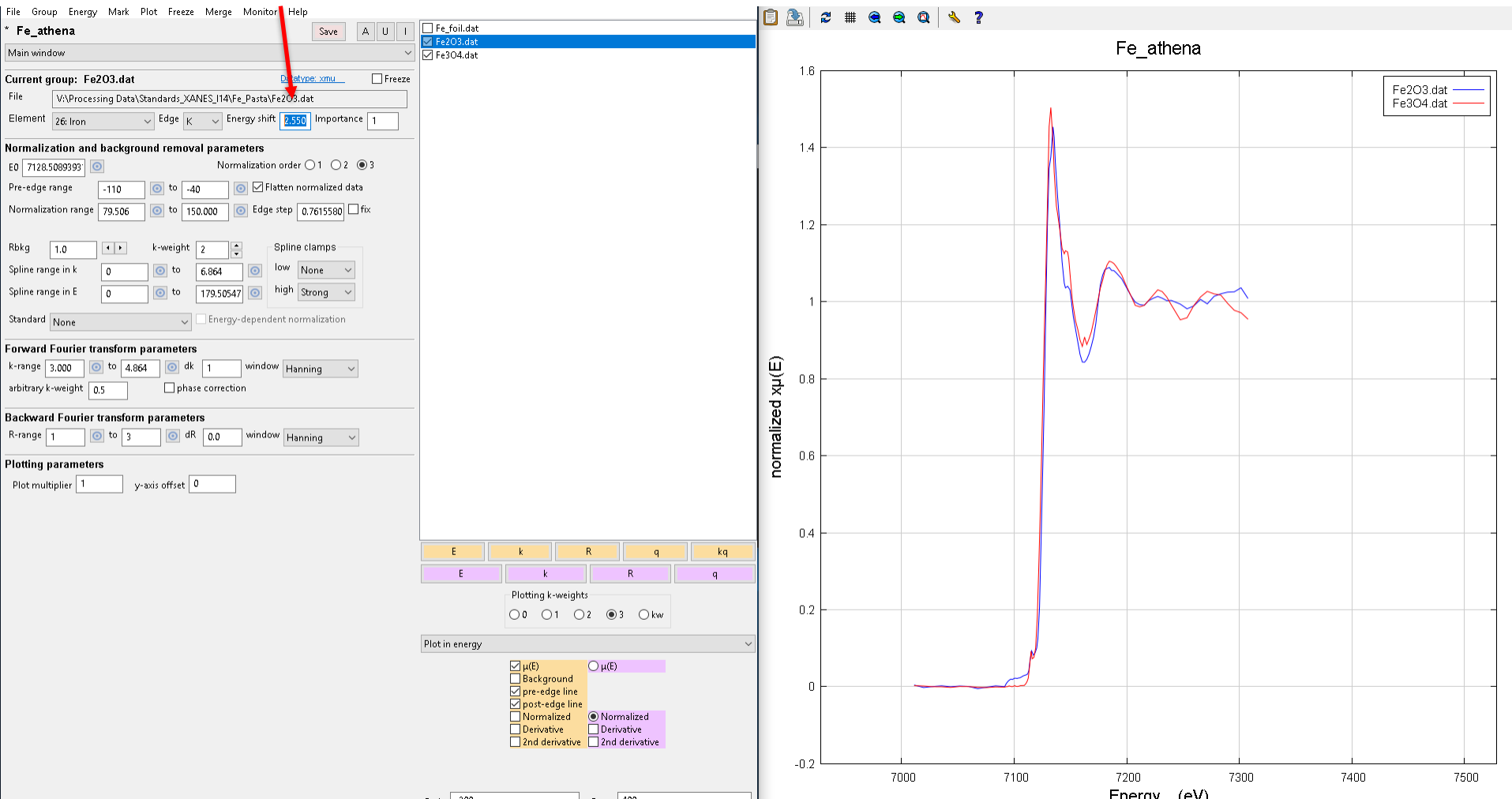
Plot multiplier: 1 y-axis offset: 0

Emin: -150 Emax: 400



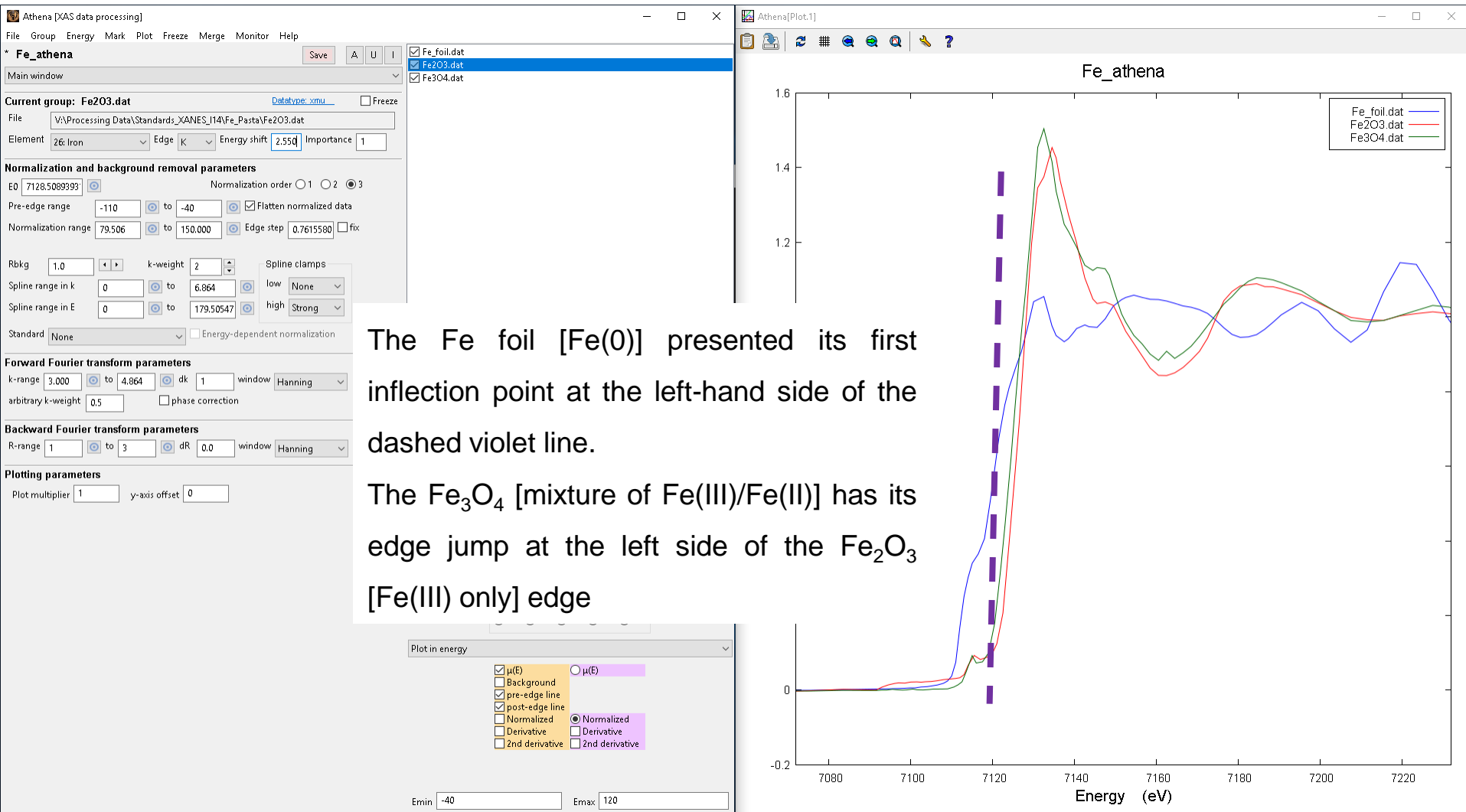
Data analysis by Athena

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



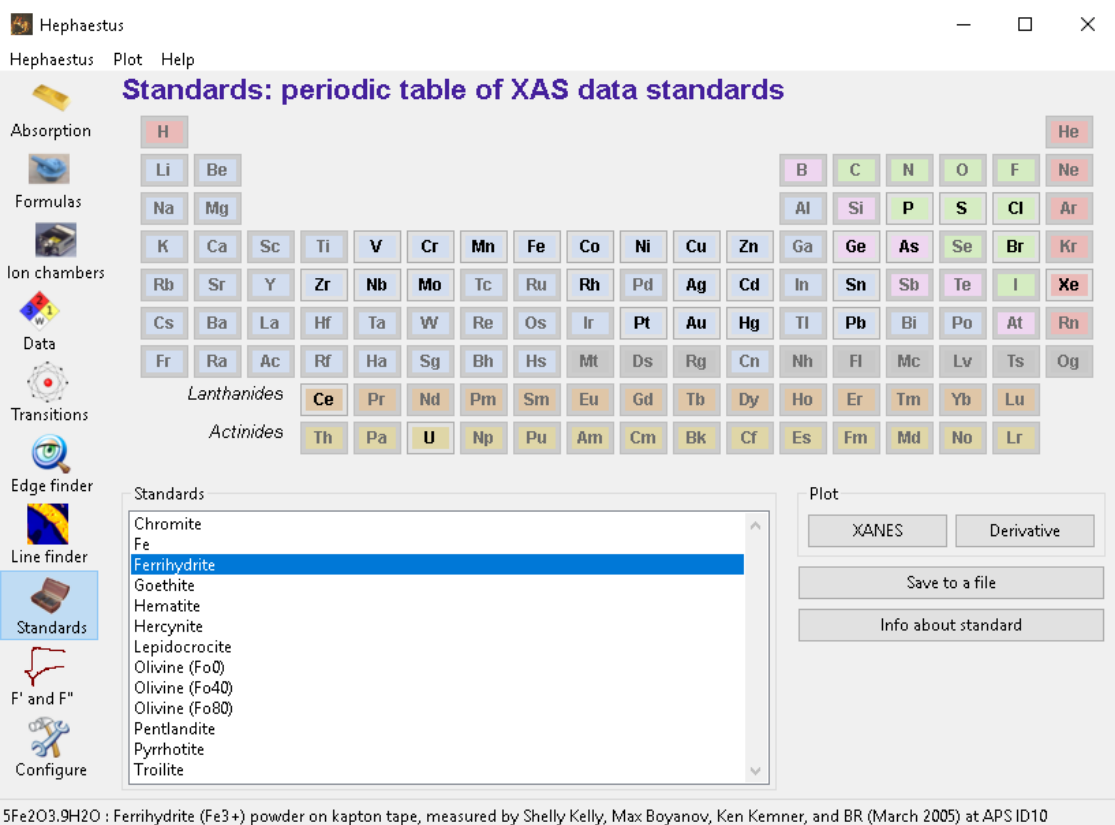
Data analysis by Athena

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



Data analysis by Athena

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



The screenshot shows the Hephaestus software interface. The main window is titled "Standards: periodic table of XAS data standards". It displays a periodic table where elements are color-coded by group. Below the table, there are sections for "Lanthanides" and "Actinides". On the left side, there is a vertical toolbar with icons for "Absorption", "Formulas", "Ion chambers", "Data", "Transitions", "Edge finder", "Line finder", "Standards", "F' and F''", and "Configure". The "Standards" section is currently active, showing a list of standards with "Ferrihydrite" selected. On the right side, there is a "Plot" section with buttons for "XANES" and "Derivative", and buttons for "Save to a file" and "Info about standard".

Standards:

- Chromite
- Fe
- Ferrihydrite**
- Goethite
- Hematite
- Hercynite
- Lepidocrocite
- Olivine (Fo0)
- Olivine (Fo40)
- Olivine (Fo80)
- Pentlandite
- Pyrrhotite
- Troilite

Plot:

XANES Derivative

Save to a file

Info about standard

5Fe2O3.9H2O : Ferrihydrite (Fe3+) powder on kapton tape, measured by Shelly Kelly, Max Boyanov, Ken Kenner, and BR (March 2005) at APS ID10

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)

The screenshot displays the Athena [XAS data processing] interface. The main window shows various processing parameters for a Hydrogen sample. A dialog box titled "Athena: Column selection" is open, showing a list of traces extracted from a plot. The "Data type" dropdown is set to "xanes", and the "Energy" dropdown is set to "10". 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 at the bottom left.

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)

#Traces extracted from Plot	Energy [eV]	trace_1	trace_2
7.355020927766984	17225.13022211674	13339.335501	
7.345003532340414	17154.33499689582	13275.782434	
7.335000497558715	16740.94850252542	13060.189773	
7.3250004867652010	16957.35497677322	13075.513160	
7.3150002829454976	16907.77615891207	12917.975431	
7.304984185085981	17046.12352936082	12937.212868	
7.294987130135263	16604.24112119809	12582.512808	
7.284973780350435	16837.33265719558	12873.533028	
7.2749786684900449	16666.64789199588	12657.409668	
7.264960785033317	17021.80051307132	12985.320718	
7.254971536386716	21471.24788306465	16811.603958	
7.244956428762254	21080.27080589178	16433.609735	
7.235946370369617	21043.110571841667	16221.001055	
7.230972667487194	20262.23374883487	15613.624330	
7.225968864228538	21385.54487141728	16661.631381	
7.220955421520214	21806.69948866748	16923.291691	
7.215959305319166	21782.56094927403	16802.282798	
7.210946841664615	21887.05309870559	16948.060198	
7.205964856046614	21820.15632426279	16959.821768	
7.200980463488876	22202.29373096818	17195.111111	
7.195976190980829	22656.82373342038	17443.455664	
7.190952378176578	23643.47515623492	18516.694193	
7.1889649658875164	24248.83412733011	18825.953674	
7.186951953441042	24479.95676658941	19033.329442	
7.184976864830595	24127.40008178002	18580.123728	
7.182969515334125	24251.00408321223	18784.769021	
7.180980018662576	24355.48291066992	19069.174058	
7.178991663369187	24318.32455204855	18906.297055	
7.176971132615006	24758.96149806467	19270.550305	
7.174978430427547	25415.17675354567	19775.821601	
7.172963574283124	25431.96067091546	20092.788111	
7.170989825216879	25900.41080150229	20453.994418	
7.168977300309421	26249.17988045121	21062.603594	
7.166979245497783	26305.01321309168	21091.985648	
7.164955776815211	26078.57387453641	20735.868664	

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 'untitled' window shows the 'Current group: plotdata_Traces2' with 'Element' set to '26: Iron' and 'Energy shift' set to '2,550'. The 'Normalization and background removal parameters' section includes 'Pre-edge range' (-100 to -30,000), 'Normalization range' (33 to 200), and 'Edge step' (21512.270). The 'Forward Fourier transform parameters' section shows 'k-range' (3,000 to 5,855) and 'dk' (1). The 'Backward Fourier transform parameters' section shows 'R-range' (1 to 3) and 'dR' (0.0). The 'Plotting parameters' section shows 'Plot multiplier' (1) and 'y-axis offset' (0). The 'Athena[Plot.1]' window shows a plot titled 'marked groups' with two traces: 'plotdata_Traces1' (blue) and 'plotdata_Traces2' (red). The plot shows a sharp peak at approximately 7,000 eV. A red arrow points from the text '22) Different regions from the same .dat file should be loaded consecutively' to the 'Energy shift' field in the Athena interface. Another red arrow points from the text '23) Remember to add the calculated energy shift for all the samples (if applicable)' to the 'Energy shift' field. A third red arrow points from the text '24) Later, it is advisable to save the XAS scans into a "sample".prj (Athena project)' to the 'Save' button in the Athena interface.

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
- Energy-dependent normalization:

The "Forward Fourier transform parameters" section includes:

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

The "Backward Fourier transform parameters" section includes:

- R-range: 1 to 3
- dR: 0.0

The "Plotting parameters" section includes:

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

The "Athena: Import from Athena project file" dialog box is open, showing the "Data group title lines" and "Plot as" options. The "Plot as" options are:

- $\mu(E)$
- $\chi(k)$
- $\chi(R)$
- $\chi(q)$
- $\text{Re}\chi(R)$
- $\text{Re}\chi(q)$
- $\text{Im}\chi(R)$
- $\text{Im}\chi(q)$

The "Import selected data" button is highlighted with a red arrow. The "Plot in energy" section is also visible, showing the "Plotting k-weights" and "Plot in energy" options.

The plot window, titled "Athena[Plot.1]", shows the XAS data for "plotdata_Traces1 in energy". 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 legend indicates "plotdata_Traces1" is plotted as a blue line.

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

The screenshot displays the Athena software interface for X-ray data processing. The main window, titled "Fe_XAS_std", is open, showing a list of data files: "Fe2O3.dat", "Fe3O4.dat", and "plotdata_Traces1". The "plotdata_Traces1" file is highlighted in blue. A red arrow points to this file. The "Main window" menu is open, showing the "Linear combination fitting" option highlighted in blue. Other options in the menu include "Principle components analysis", "Peak fitting", "Log-ratio/phase-difference analysis", "Difference spectra", "File metadata", "Project journal", "Plugin registry", and "Preferences".

Below the menu, the "Forward Fourier transform parameters" section shows the k-range set to 3.000 to 5.855 dk 1 window Hanning. The "Backward Fourier transform parameters" section shows the R-range set to 1 to 3 dR 0.0 window Hanning. The "Plotting parameters" section shows the Plot multiplier set to 1 and y-axis offset set to 0.

The plot area, titled "Fe_XAS_std", shows the normalized $\chi^2(E)$ versus Energy (eV). The x-axis ranges from 7050 to 7250 eV, and the y-axis ranges from -0.2 to 1.6. Two data series are plotted: "Fe2O3.dat" (blue line) and "Fe3O4.dat" (red line). Both series show a sharp peak at approximately 7100 eV and a smaller peak at approximately 7180 eV. The "Fe3O4.dat" series shows a slightly higher peak at 7100 eV and a slightly lower peak at 7180 eV compared to the "Fe2O3.dat" series.

At the bottom of the interface, the "Plot in energy" section is visible, with checkboxes for $\mu(E)$, Background, pre-edge line, post-edge line, Normalized, Derivative, and 2nd derivative. The "Normalized" checkbox is checked. The "Emin" is set to -80 and the "Emax" is set to 130.

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)

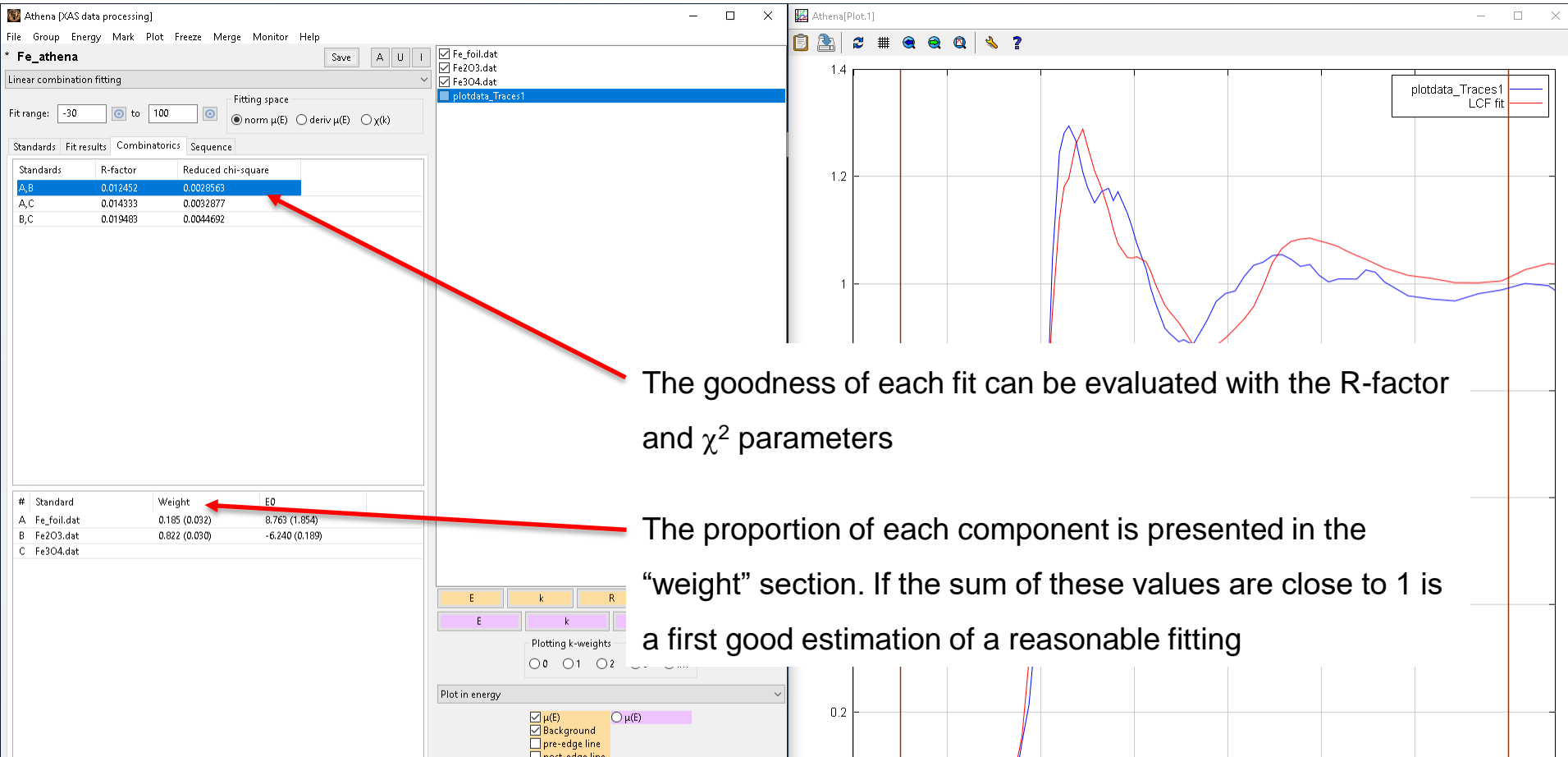
The screenshot displays the Athena software interface for XAS data processing. The main window is titled "Fe_XAS_stnd" and shows the "Linear combination fitting" (LCF) window. The "Fit range" is set from -30 to 100. The "Standards" table lists two standards: Fe2O3.dat and Fe3O4.dat, both with a weight of 0.500. The "Options" section shows "Force weights to sum to 1" unselected. The "Combinatorics" section shows "Use at most" set to 2. The "Actions" section shows "Use marked groups" selected. The plot on the right shows the normalized $x\mu(E)$ 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’

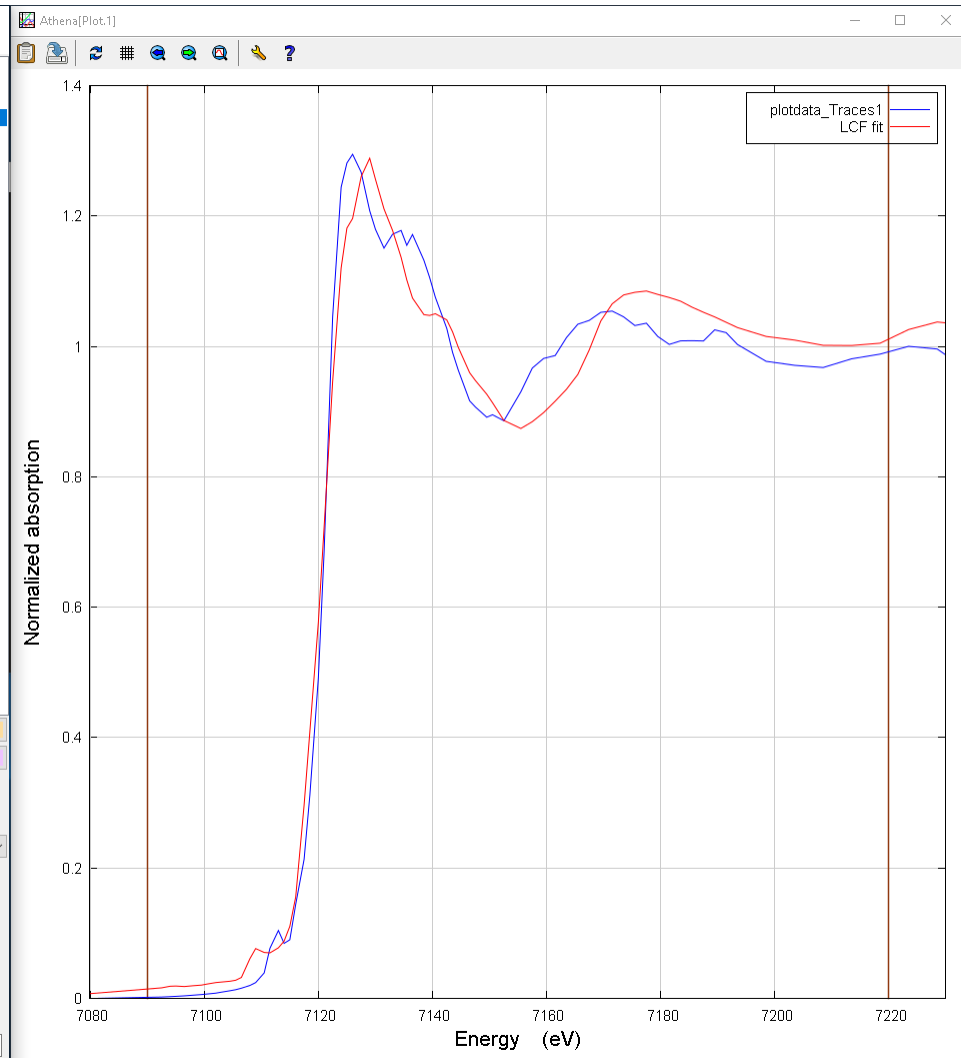
The screenshot shows the Athena [XAS data processing] window with the 'Fit Results' tab selected. The window title is 'Fe_athena'. The 'Linear combination fitting' section shows a fit range from -30 to 100. The 'Fitting space' section has 'norm $\mu(E)$ ' selected. The 'Standards' section lists 'Fe203.dat' and 'Fe_foil.dat'. The 'Fit results' section shows the following text:

```
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	

The 'Plotting k-weights' section has '3' selected. The 'Plot in energy' section has 'Normalized' selected. The 'Save fit as column data' button is highlighted with a red arrow.



Plot normalized data when plotting the current group in energy.

7070.93, 0.887273

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)

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10.1. Linear combination fitting

10.1.1. Interpreting data as a mixture of standards

ATHENA has a capability of fitting a linear combination of standard spectra to an unknown spectra. These fits can be done using normalized $\mu(E)$, derivative of $\mu(E)$, or $\chi(k)$ spectra. One use of this sort of analysis might be to interpret the kinetics of series of spectra measured during a reduction reaction. By fitting each intermediate spectrum as a linear combination of the end members, one can deduce the rate of the reaction. Another possible use would be to determine the species and quantities of standards in a heterogeneous sample.

A worked example of linear combination fitting is shown later in this manual.

To access this feature, choose Linear combination fit from the main menu. The normal parameter view will be replaced by the tool in the following figure for performing the linear combination fit.

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"/>

- ❑ 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>