



XVIII School on Synchrotron Radiation
"Gilberto Vlaic":
Fundamentals, Methods and Applications
Muggia (Trieste), Italy
21 September – 1 October September 2026



Società
Italiana
Luce di
Sincrotrone



Elettra Sincrotrone Trieste

XANES and EXAFS

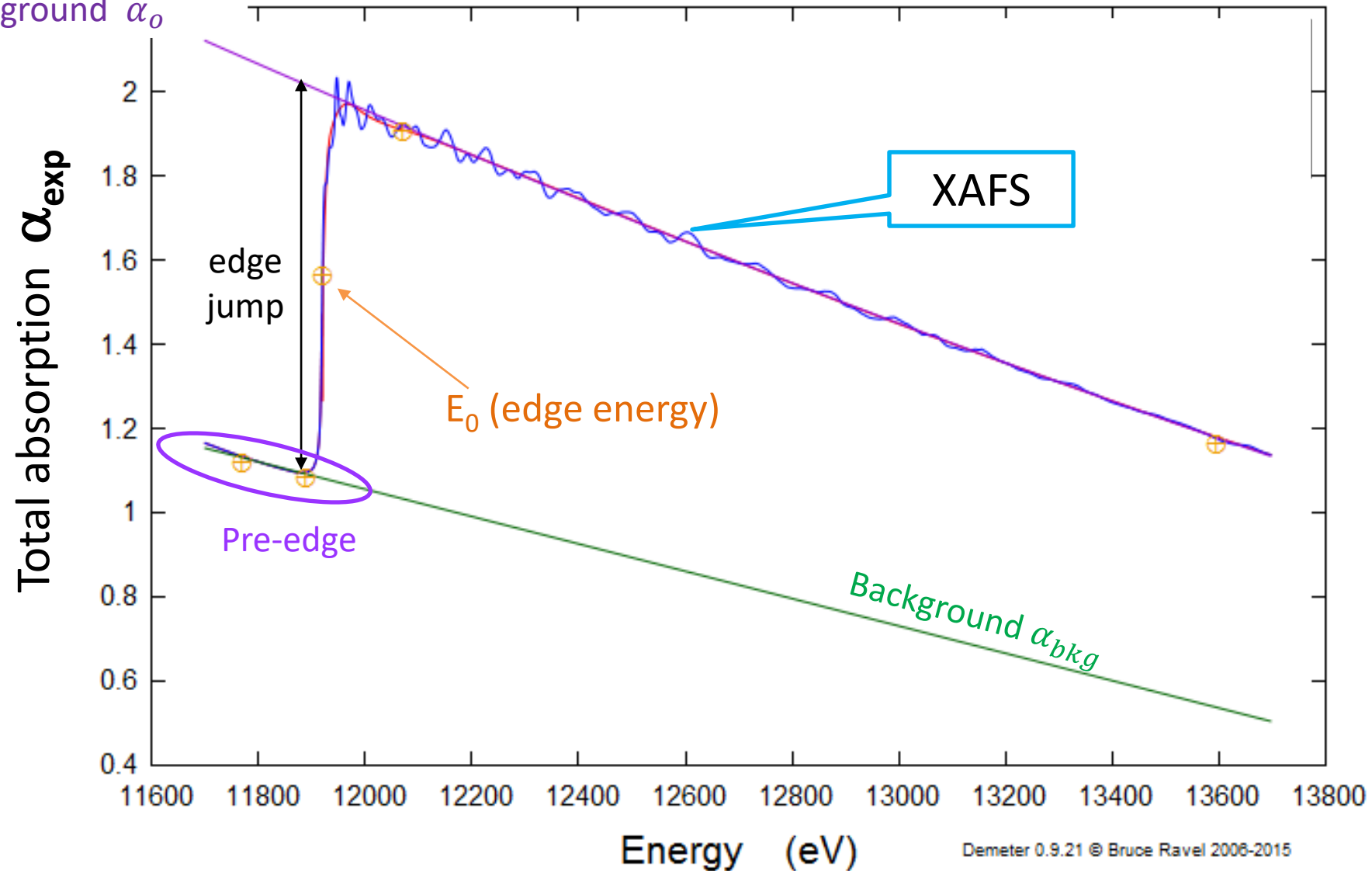
Ilaria Carlomagno

Many slides are from previous lectures of:

Prof. Carlo Meneghini
carlo.meneghini@uniroma3.it

XAS spectrum

Post edge atomic
background α_o

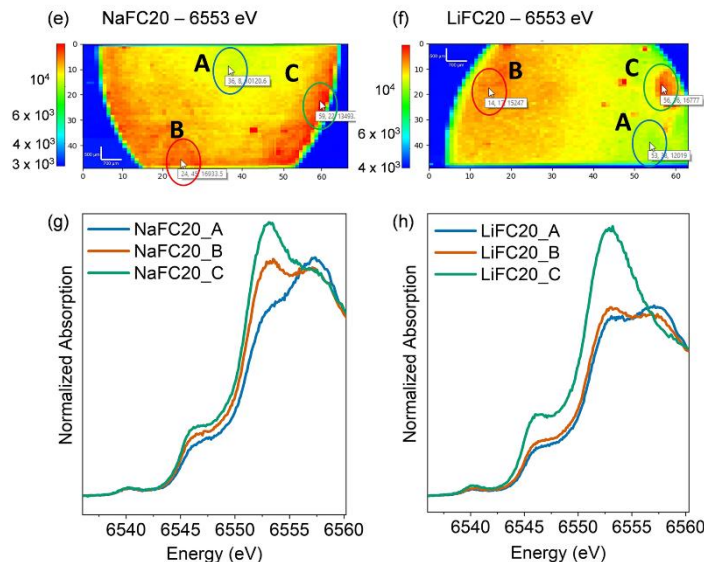


Demeter 0.9.21 © Bruce Ravel 2006-2015

XRF and XAFS...together?

Using the same (fluorescence) setup

- XRF provides spatial distribution and elemental concentration
- XRF can find the perfect spot for XAFS analysis
- XANES and EXAFS add chemical speciation and local structural characterization



MnHCF-Based Electrodes:

2D mapping above Mn K edge and then XANES in three different points.

Speciation mapping

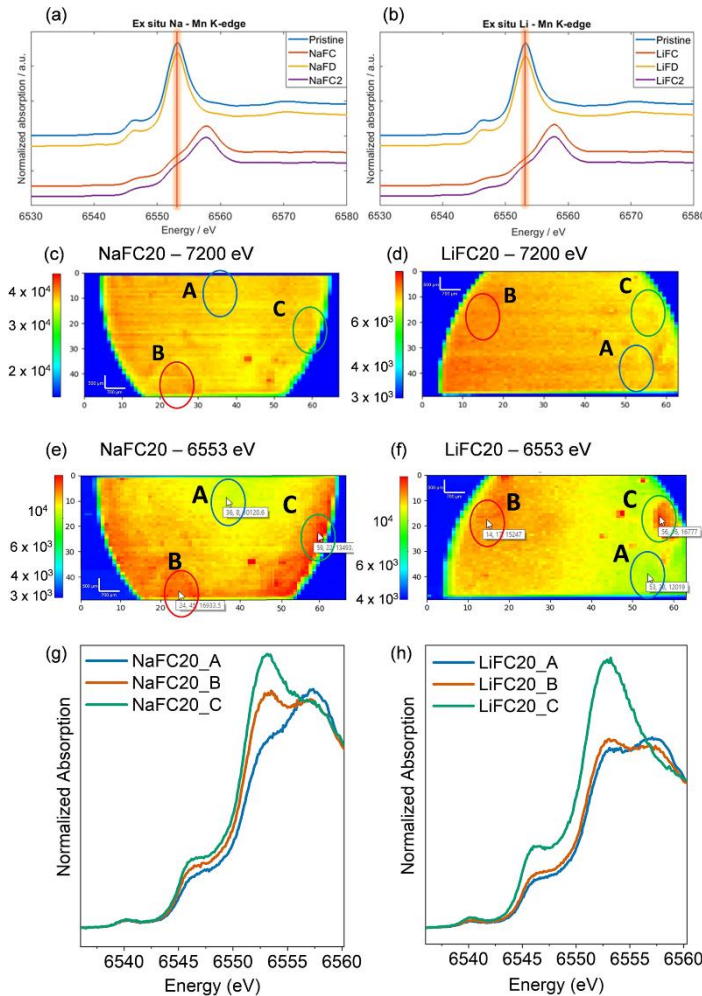
XRF and XANES can be combined to obtain the **spatial distribution of a particular species**.

To do so, you need:

- Enough **energy separation** between the white lines of the two species
- Enough **energy resolution** for your primary X-ray beam (typically $\Delta E/E = 10^{-4}$ for Si(111))

MnHCF-Based Electrodes:

Large energy gap between the K-edge energies of Mn species: tuning the excitation energy, one can see the distribution of the different species.



Detection methods

The ideal detection method is the one giving the best signal to noise ratio

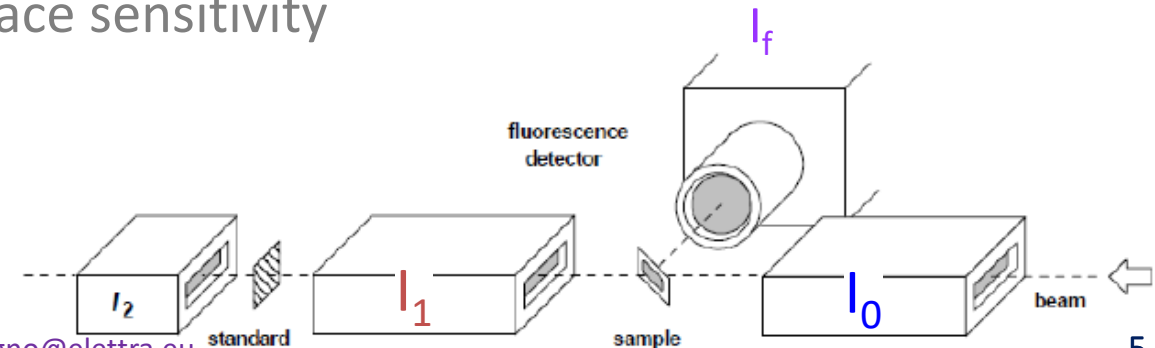
- **Fluorescence** (partial fluorescence yield PFY):
reduced background but can be time consuming.
Ideal choice for **diluted** or **thick** samples
Watch out for self absorption, detector linearity, Bragg reflections!

$$\alpha \propto \frac{I_f}{I_0}$$

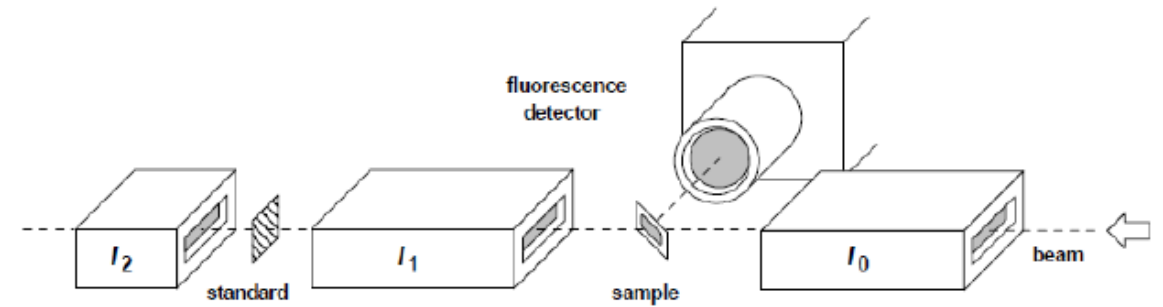
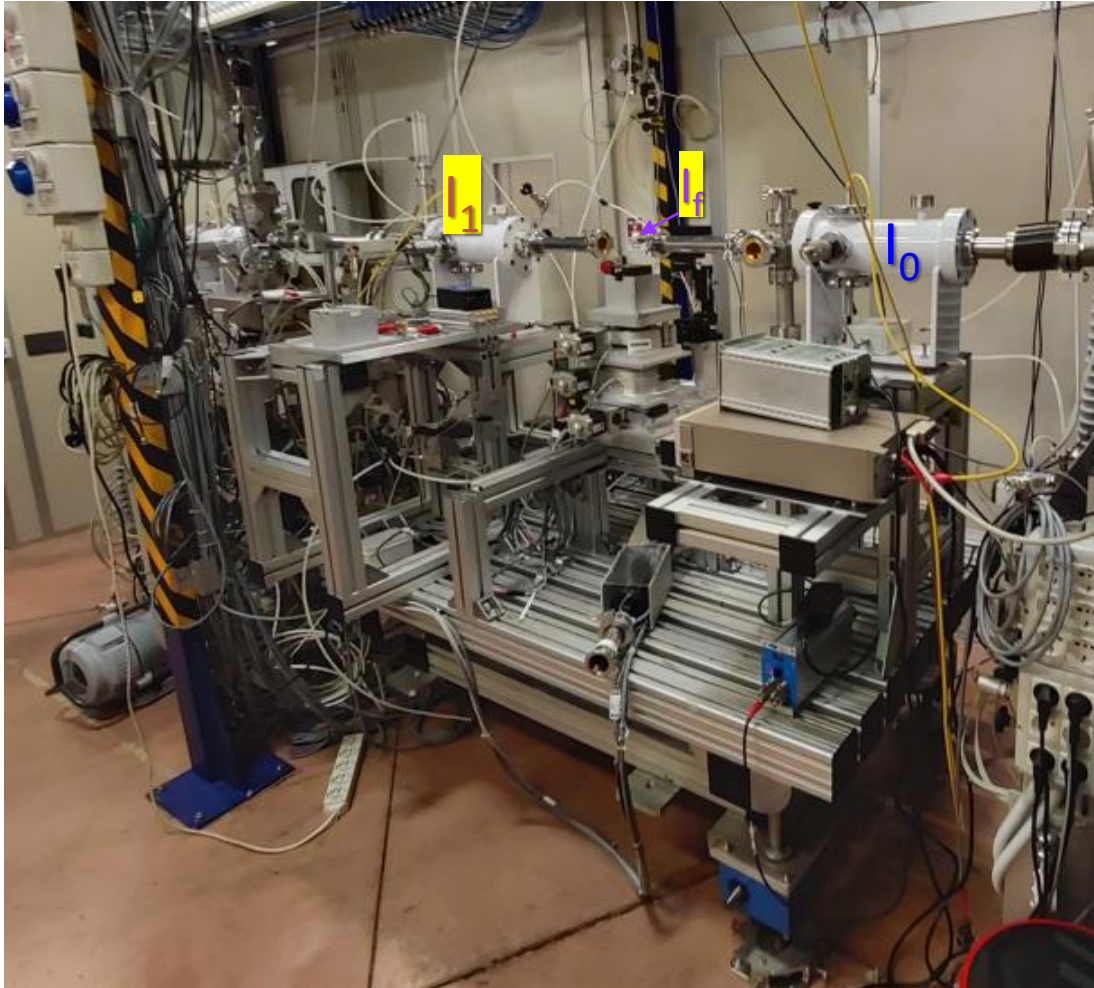
- **Transmission**: when possible, it is the best.
Needs **thin** and **concentrated** samples
Watch out for inhomogeneities!

$$\alpha = \ln \frac{I_0}{I_1}$$

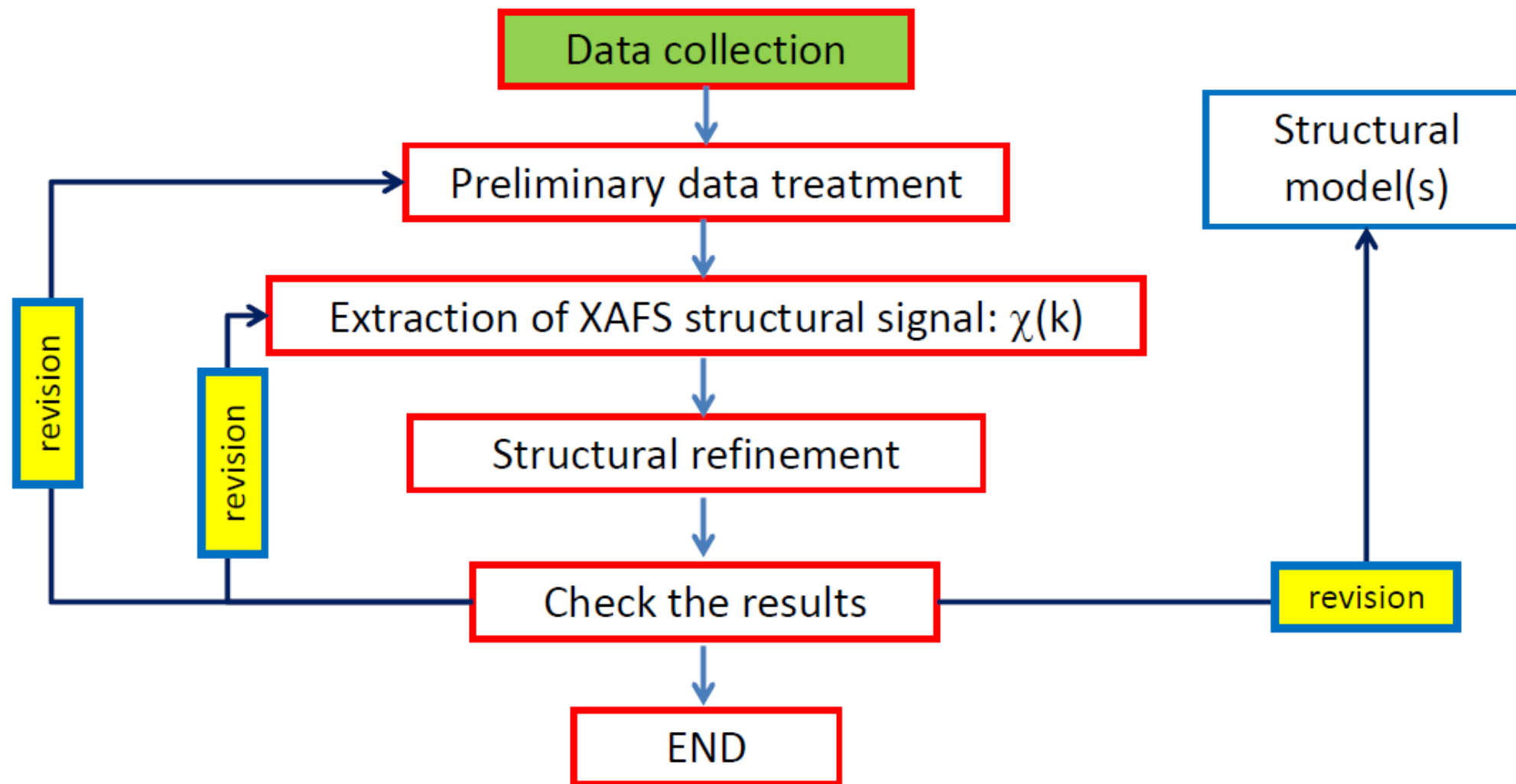
- **Electrons** (Total Electron Yield – TEY): maximum surface sensitivity

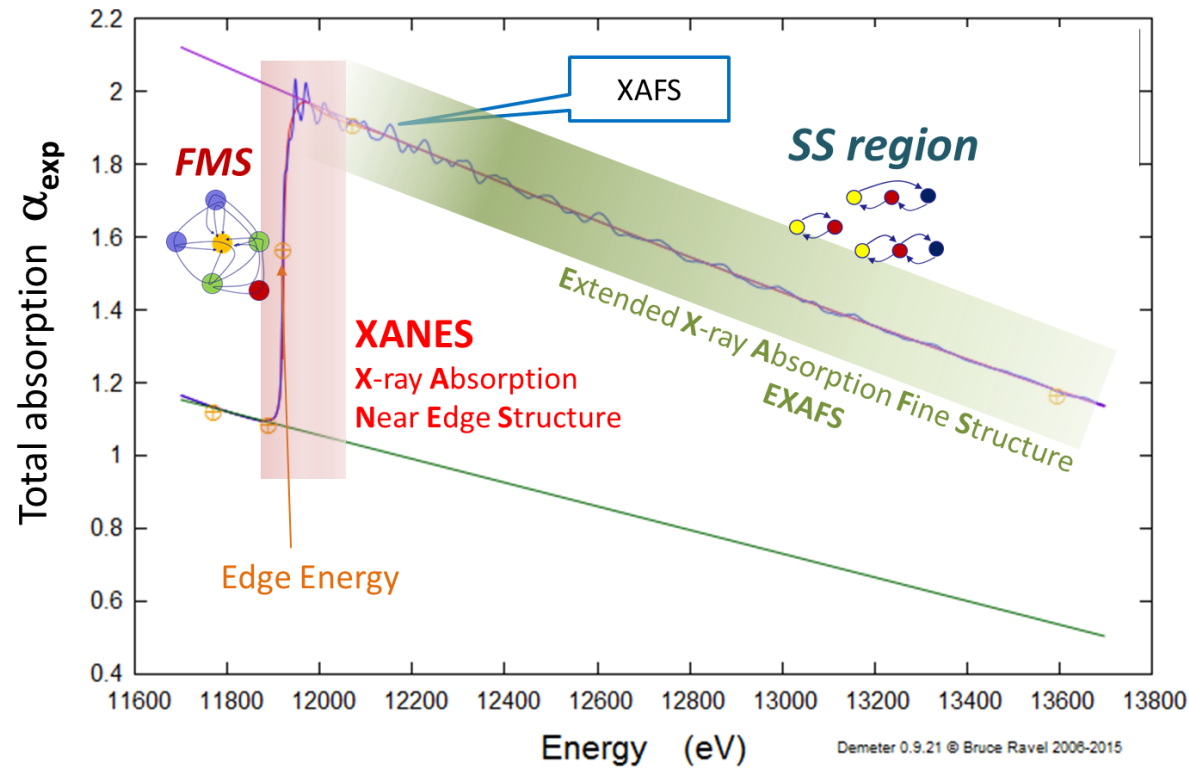


XAFS beamline @ Elettra



EXAFS experiment: our path





XANES region:

- Hard theory
- lack of an analytical expression
- long computation time



EXAFS region: simple analytical formula suited for data fitting and *easy* structural refinement

$$\chi(k) = \frac{1}{k} \sum A_j \sin(2k r_j + \psi_j)$$



$$A_j = S_o^2 \frac{N_j}{r_j} |f_j| e^{-2k^2 \sigma_j^2} e_j^{-\frac{2r_j}{\lambda}}$$

Data collection

Proposal submission + proposal evaluation + beamtime scheduling = 6 to 12 months
Difficult to have new beamtime in case of proposal failure



BEFORE experiment (proposal submission):

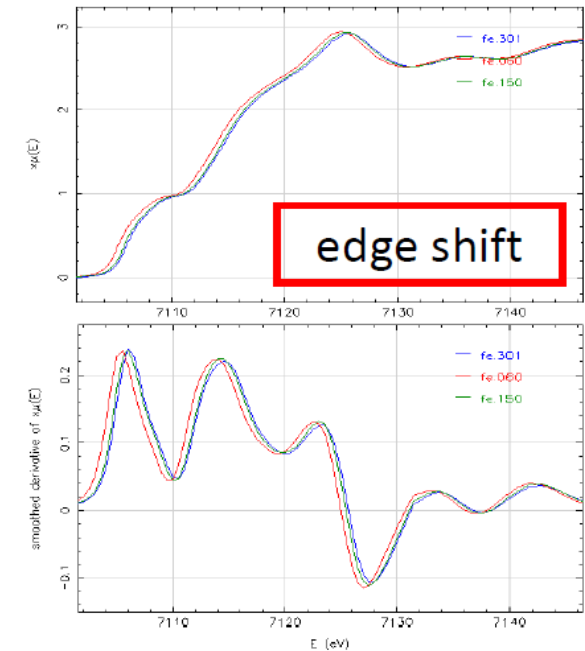
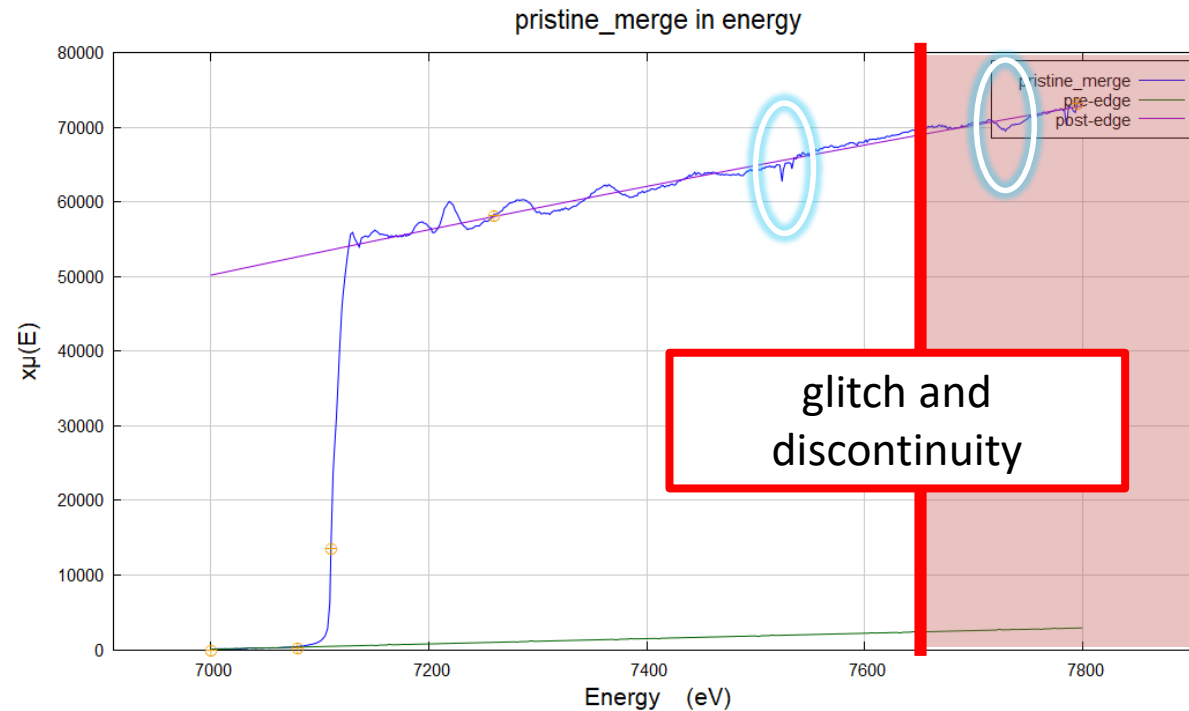
- Check the proposal submission deadlines
- Discuss your experiment with local contacts
- Choose the best set-up and sample preparation

DURING experiment:

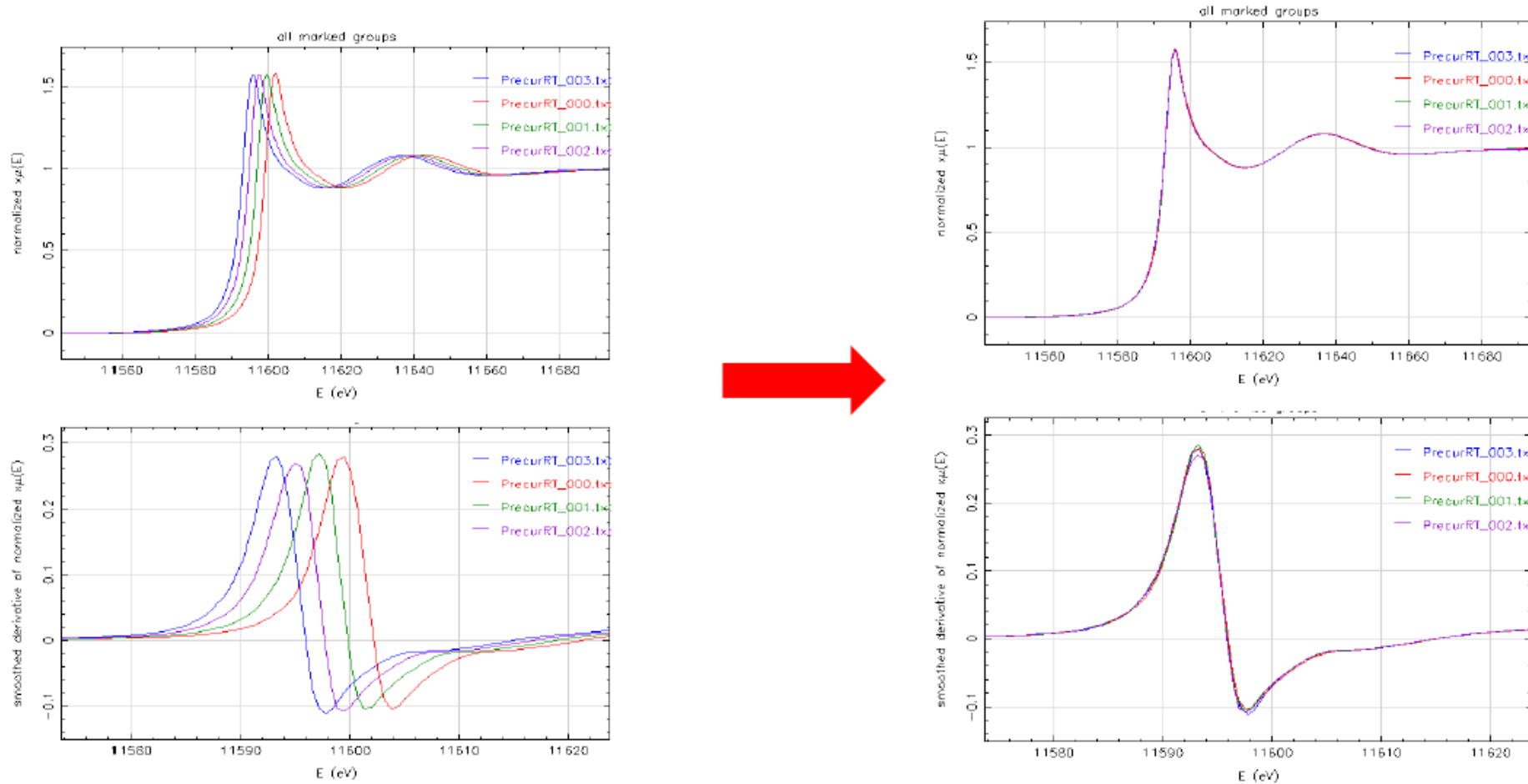
- Prepare in advance (samples, **references**, and preliminary characterization)
- Optimise data collection strategy
- Check data quality **constantly**

Preliminary data treatment

- Begins during your experiment
- Optimise energy region and number of repetitions
- Includes spectra deglitching, truncation, alignment, and merge

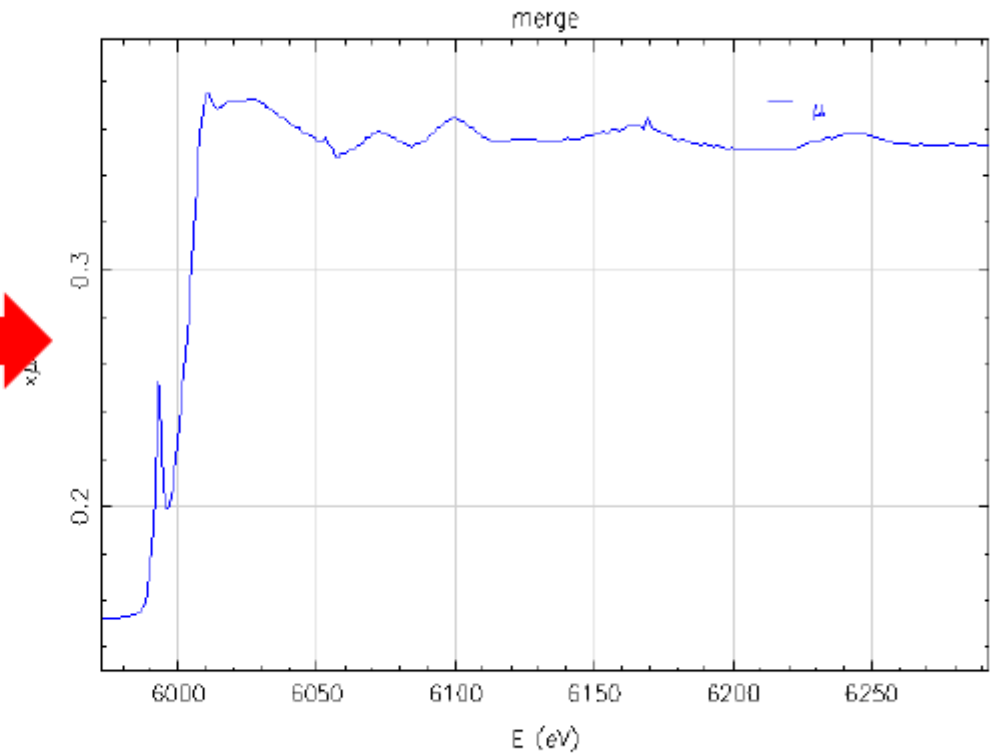
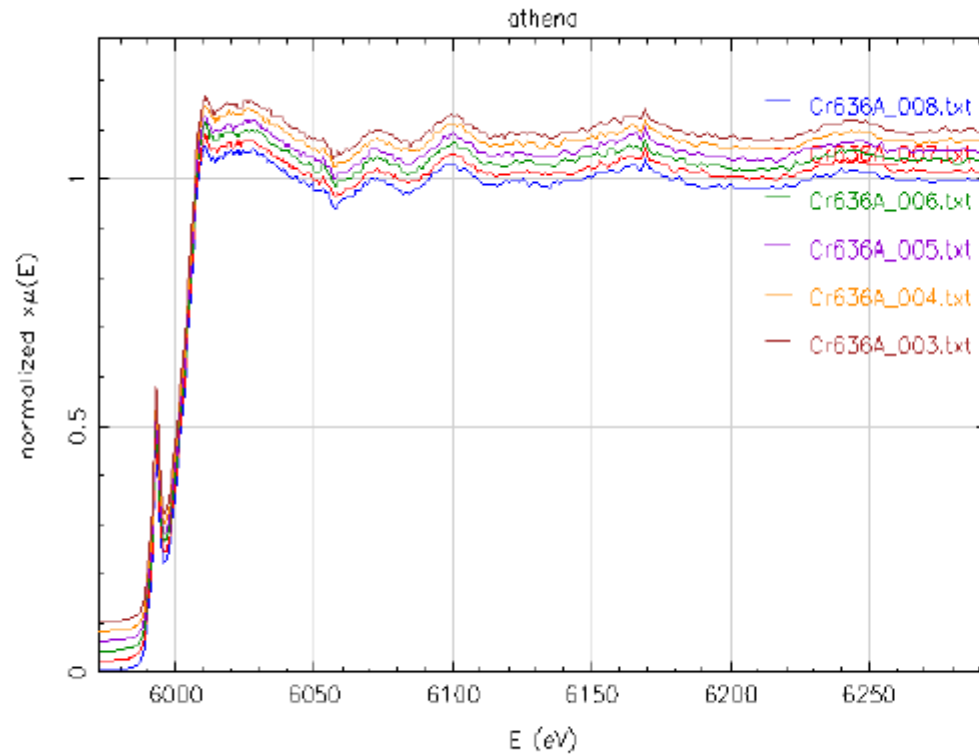


Alignment



Correct for instrumental energy shift (repeatability of monochromator positioning)

Average



Especially on ultra-diluted samples, collecting many repetitions is standard procedure.

Exercise #1



- I. Download DEMETER and install it
- II. Start Athena*
- III. Calibrate, align and average the signals

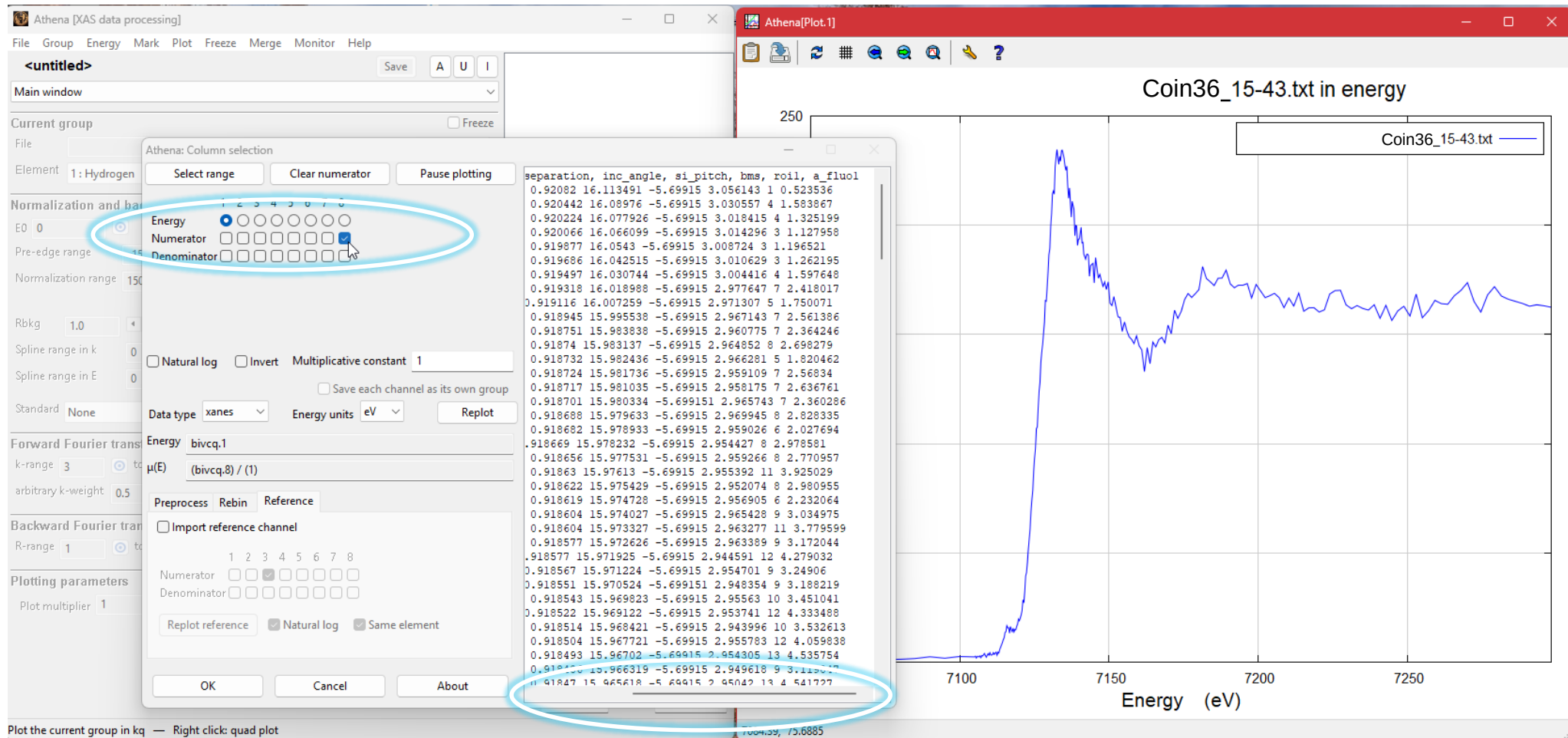
Data folder for exercises

<https://drive.elettra.eu/d/b0b0c8eb775749138b10/>

**Complete documentation available!*

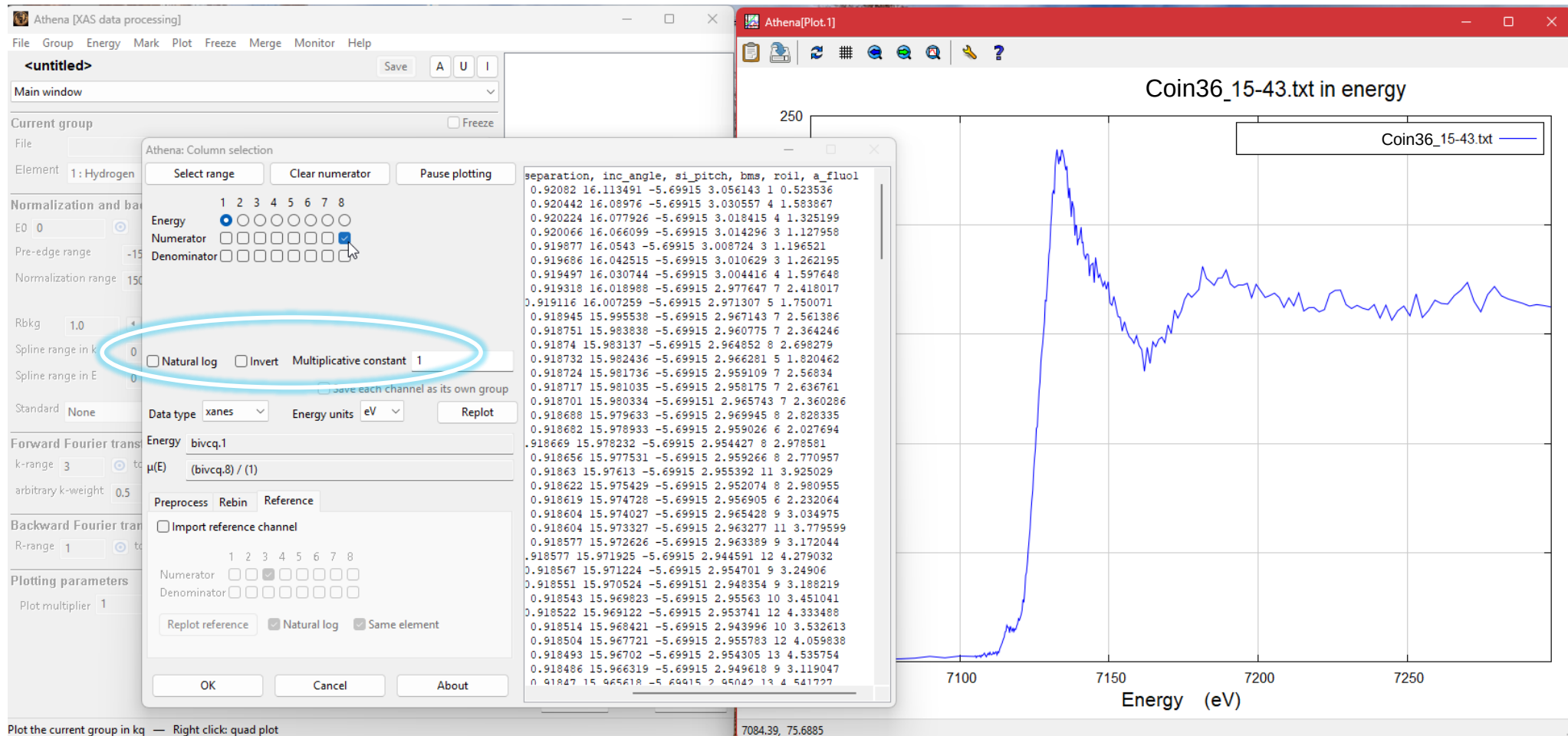
<http://bruceravel.github.io/demeter/documents/Athena/index.html>

Load data files



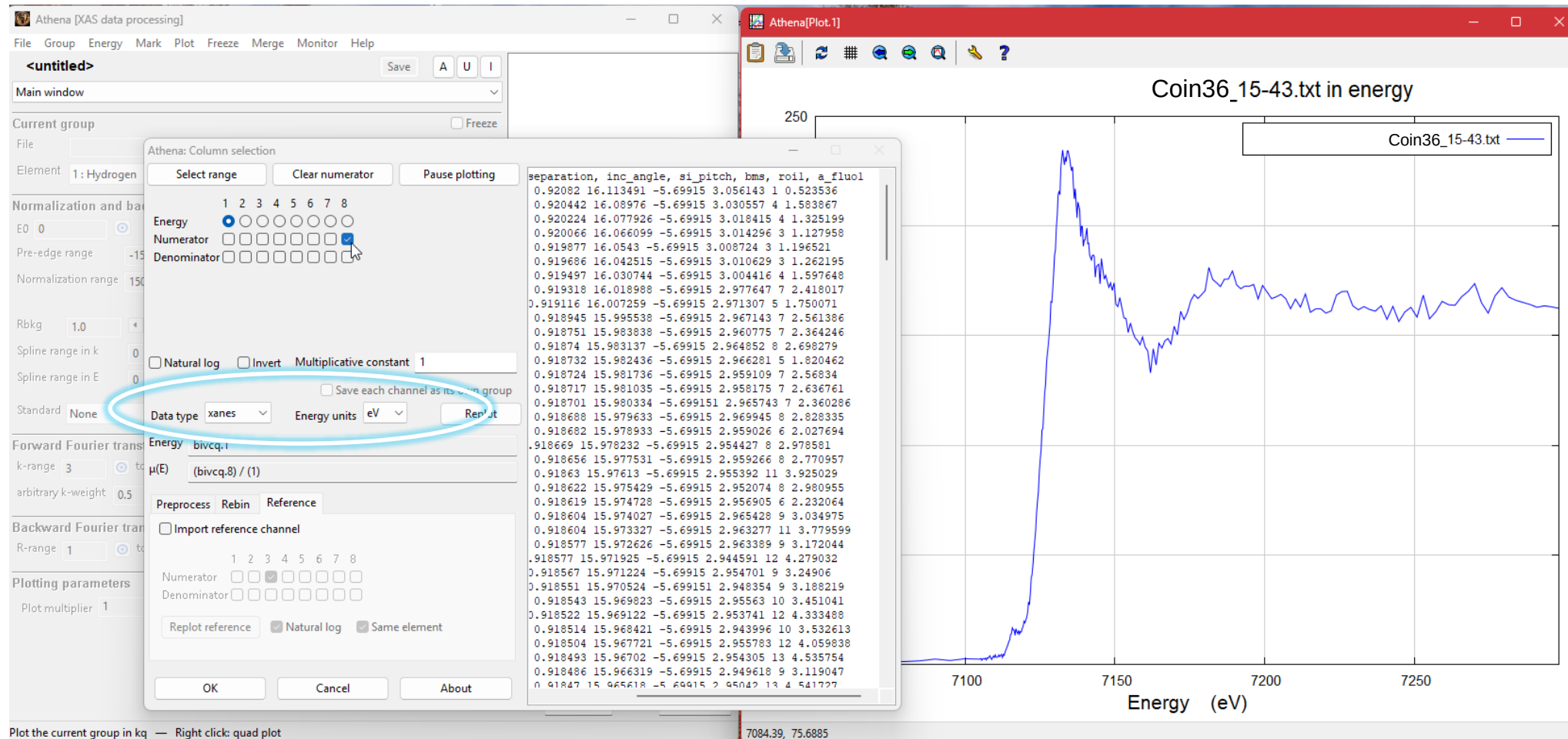
1. Start from “Coin36” datasets. Scroll the page horizontally until you find the “energy” and “a_fluo” headers, then select the corresponding columns as Energy and Numerator

Load data files



In this case, our datafile contains already the absorption, so we do not need to do anything (log or invert).

Load data files



Data type: $\mu(E)$ works for EXAFS/XANES // “xanes” works for near edge region only

Watch out for the **Energy units** in the datafile!

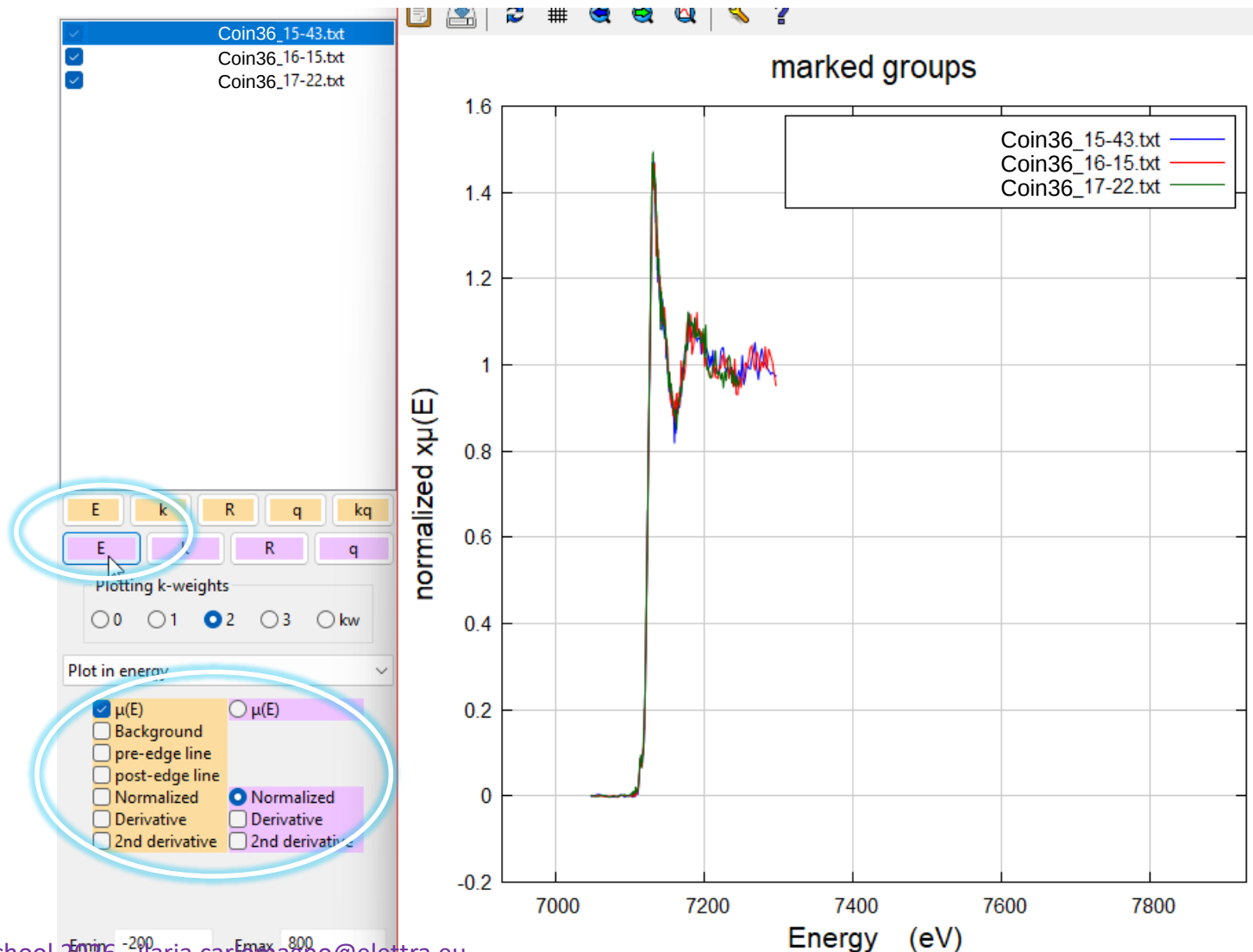
Plot all the spectra in energy

Use the “E” button to plot in energy.

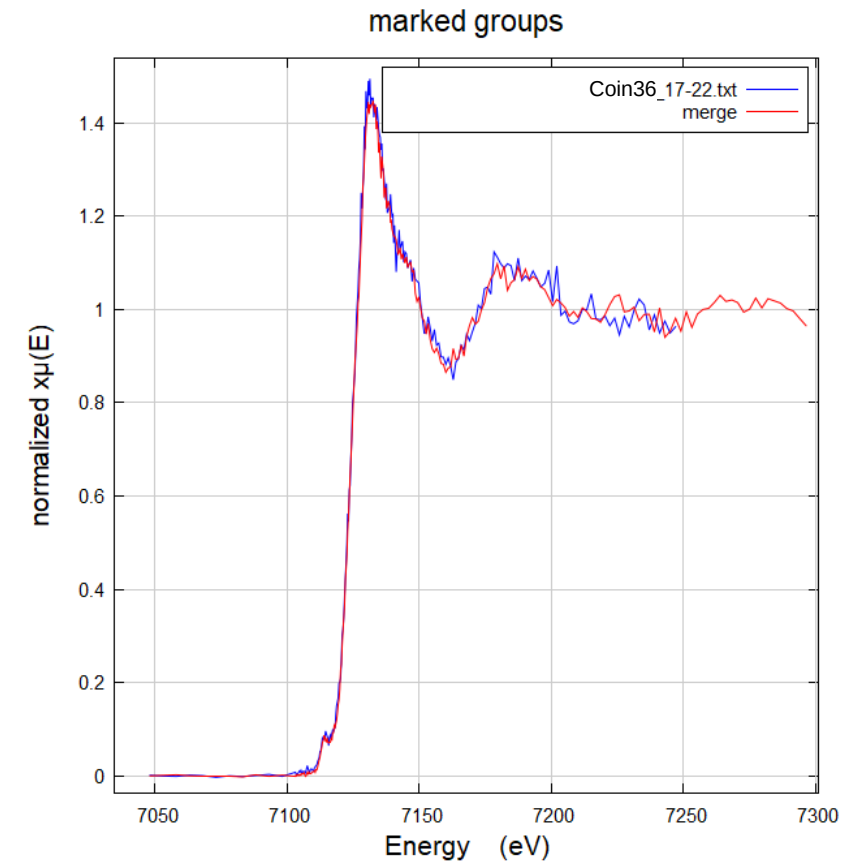
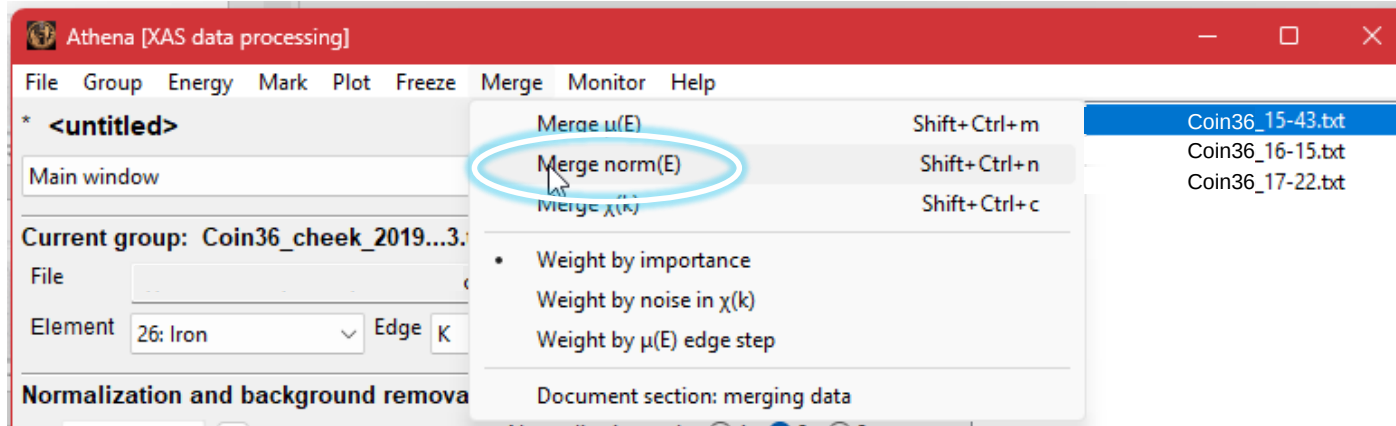
Yellow button = plot of the highlighted dataset

Pink button = plot the selected datasets

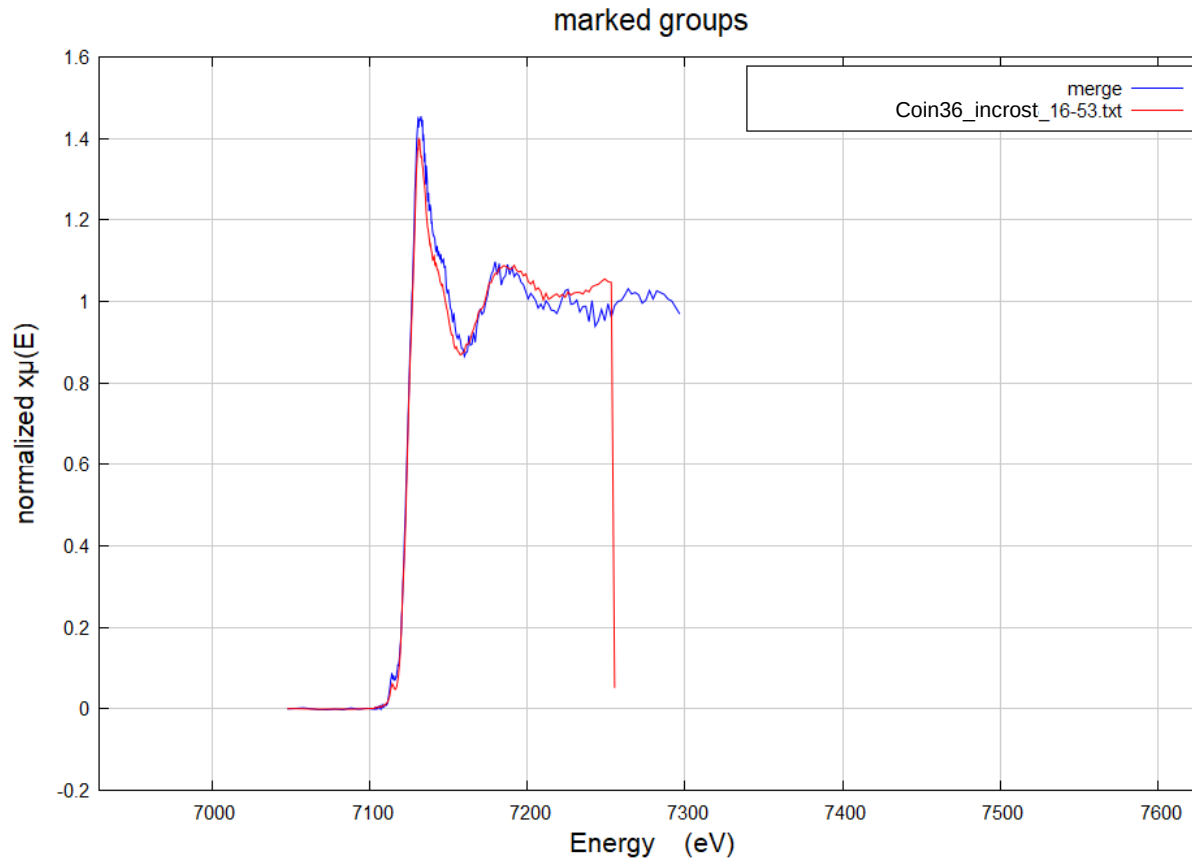
Watch out for what you are plotting!
($\mu(E)$ vs Normalized)



Merge all the spectra

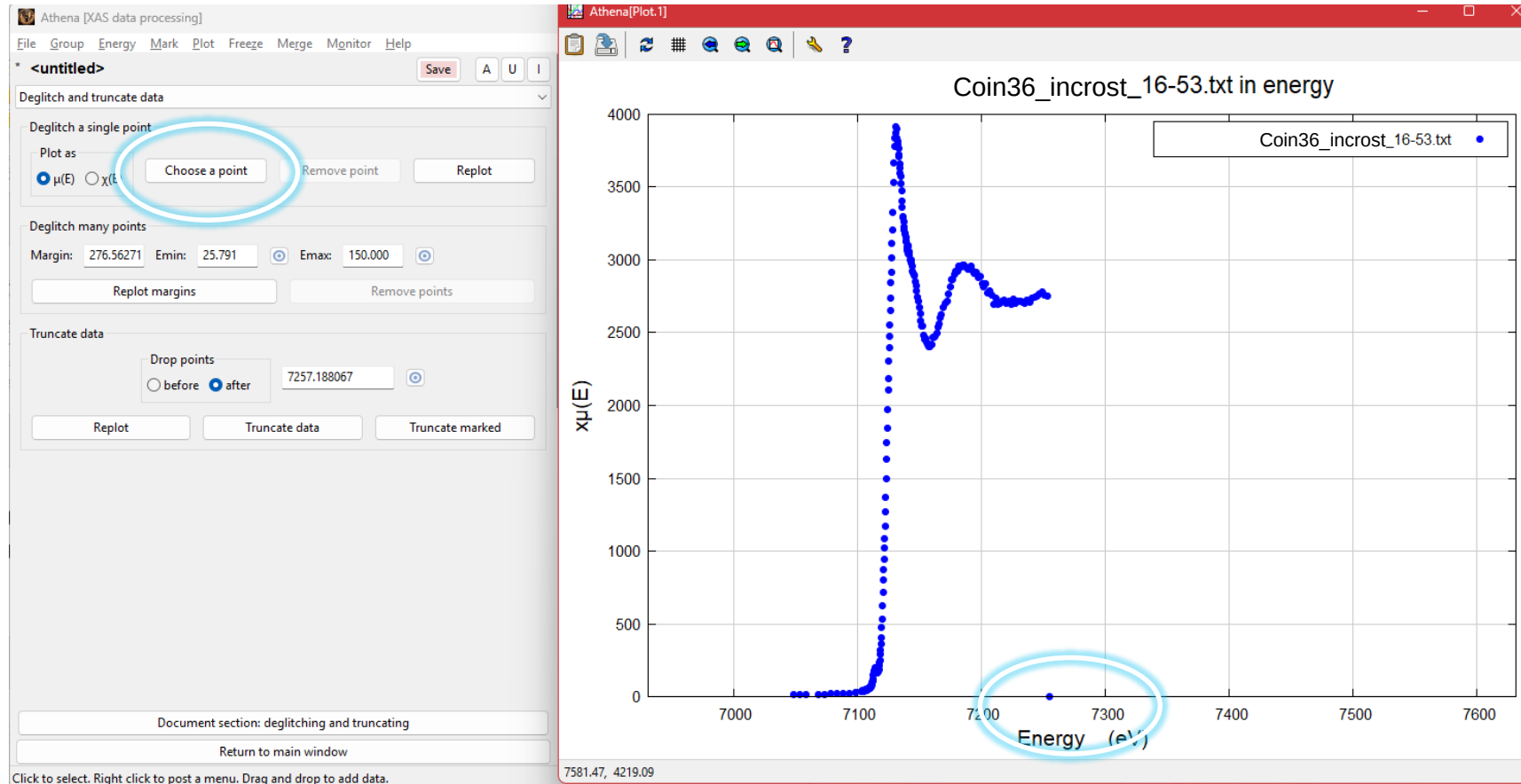


Load “Coin36_incrost_*.txt”



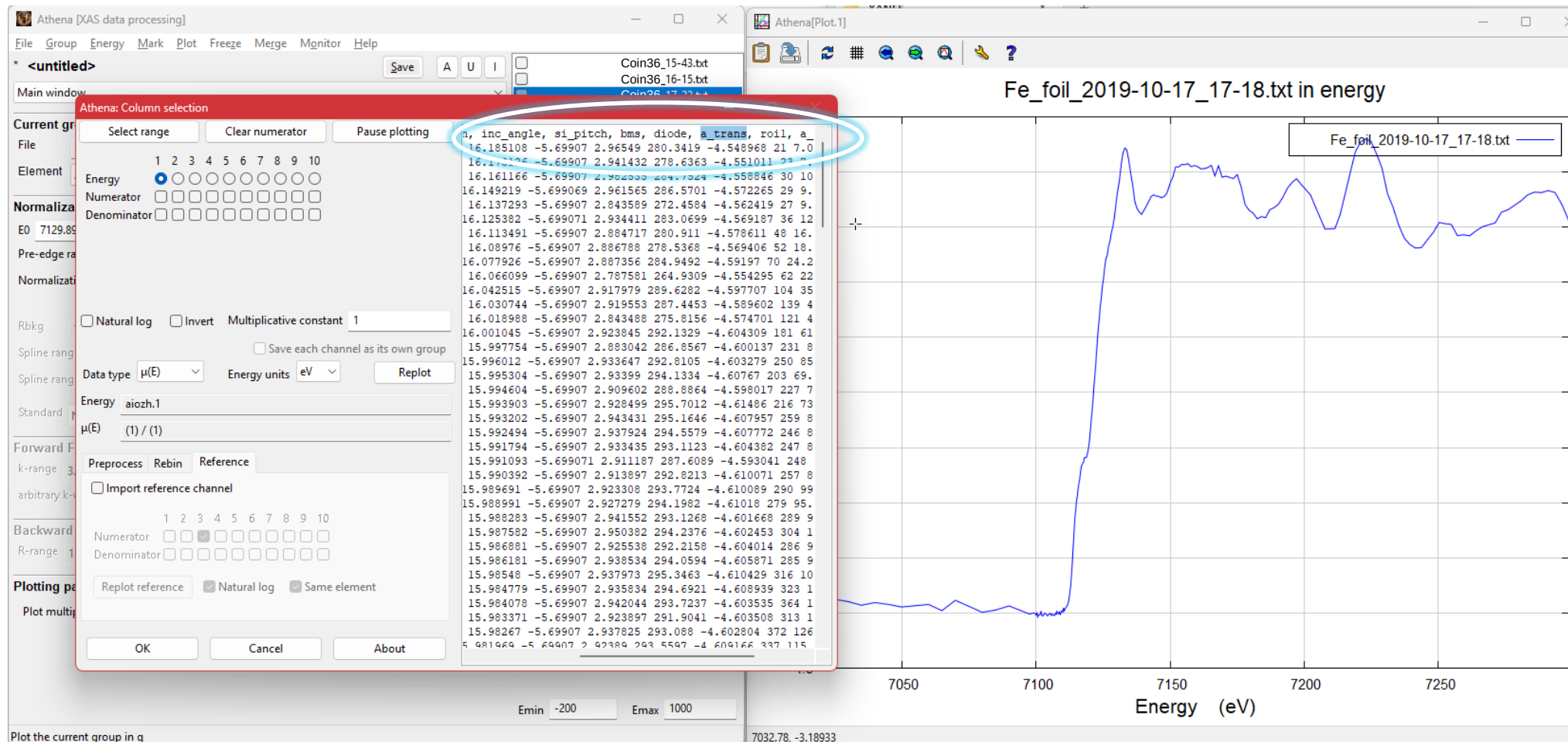
This spectrum needs some additional tweaking

Deglitch and truncate



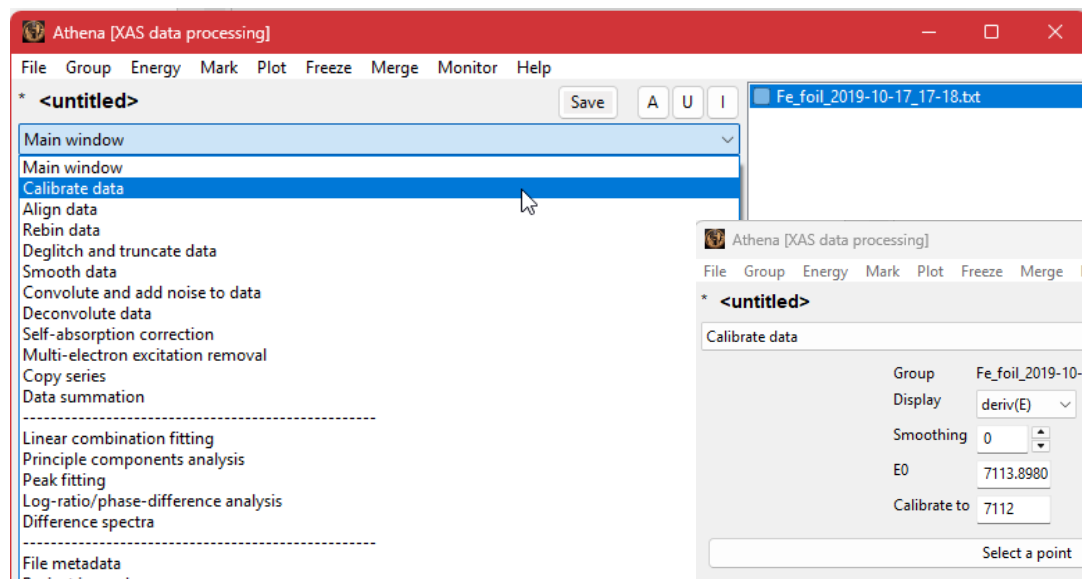
Choose a point and select the last point of the spectrum. Then **Remove point** to get rid of it.

Load reference data



Now, load the reference spectrum (Fe_foil). This time, look for the “a_trans” column!

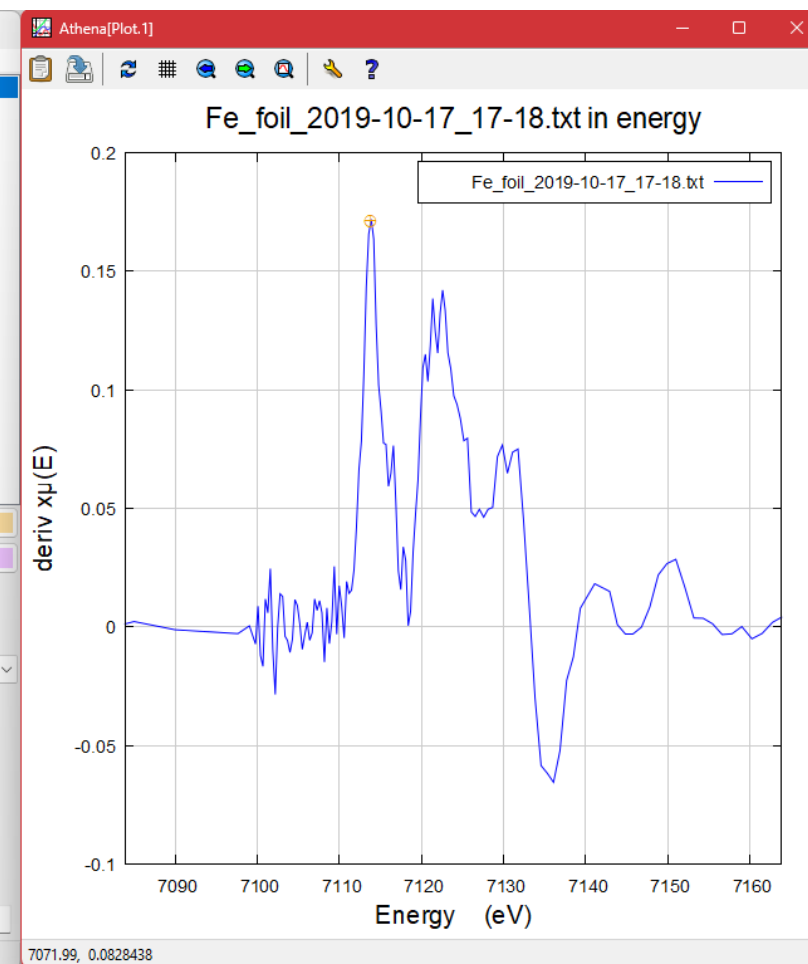
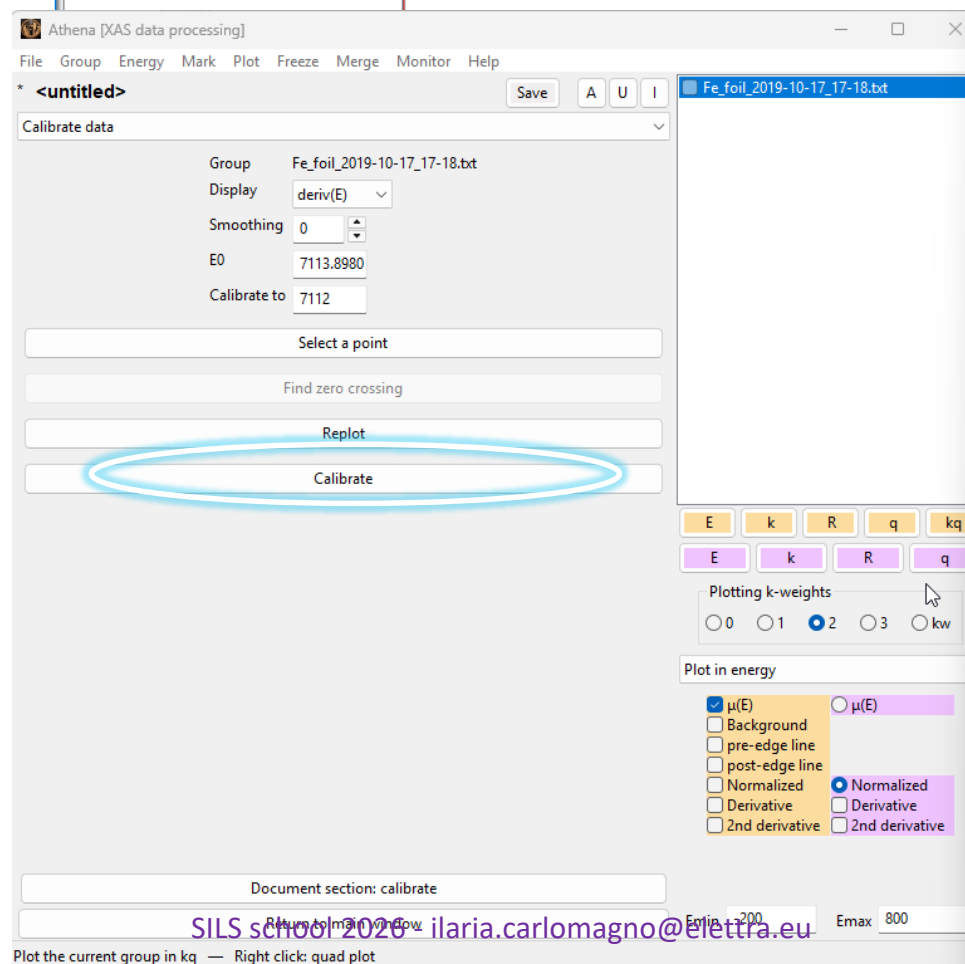
Calibrate energy (from reference)



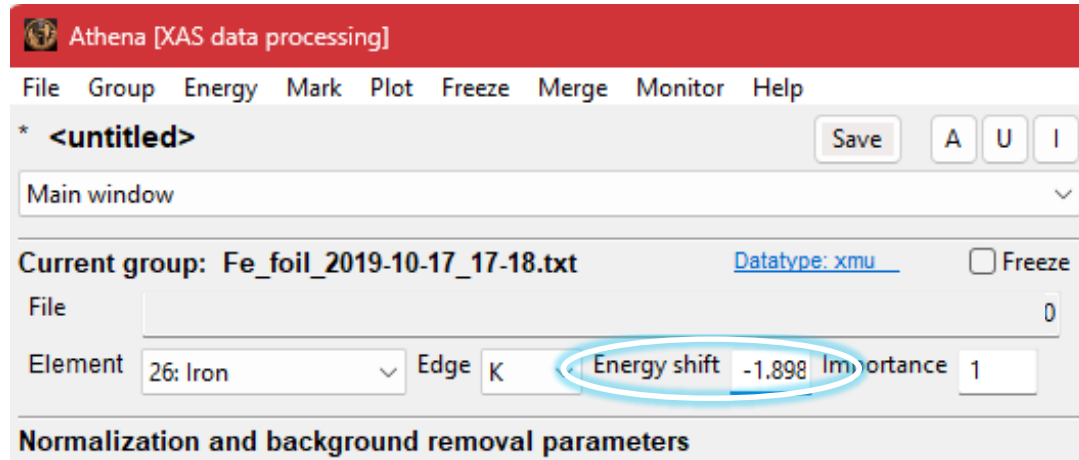
Make sure that the orange point is at the first maximum.

If not: **Select a point.**

Then **calibrate** to the expected energy (Fe K edge).



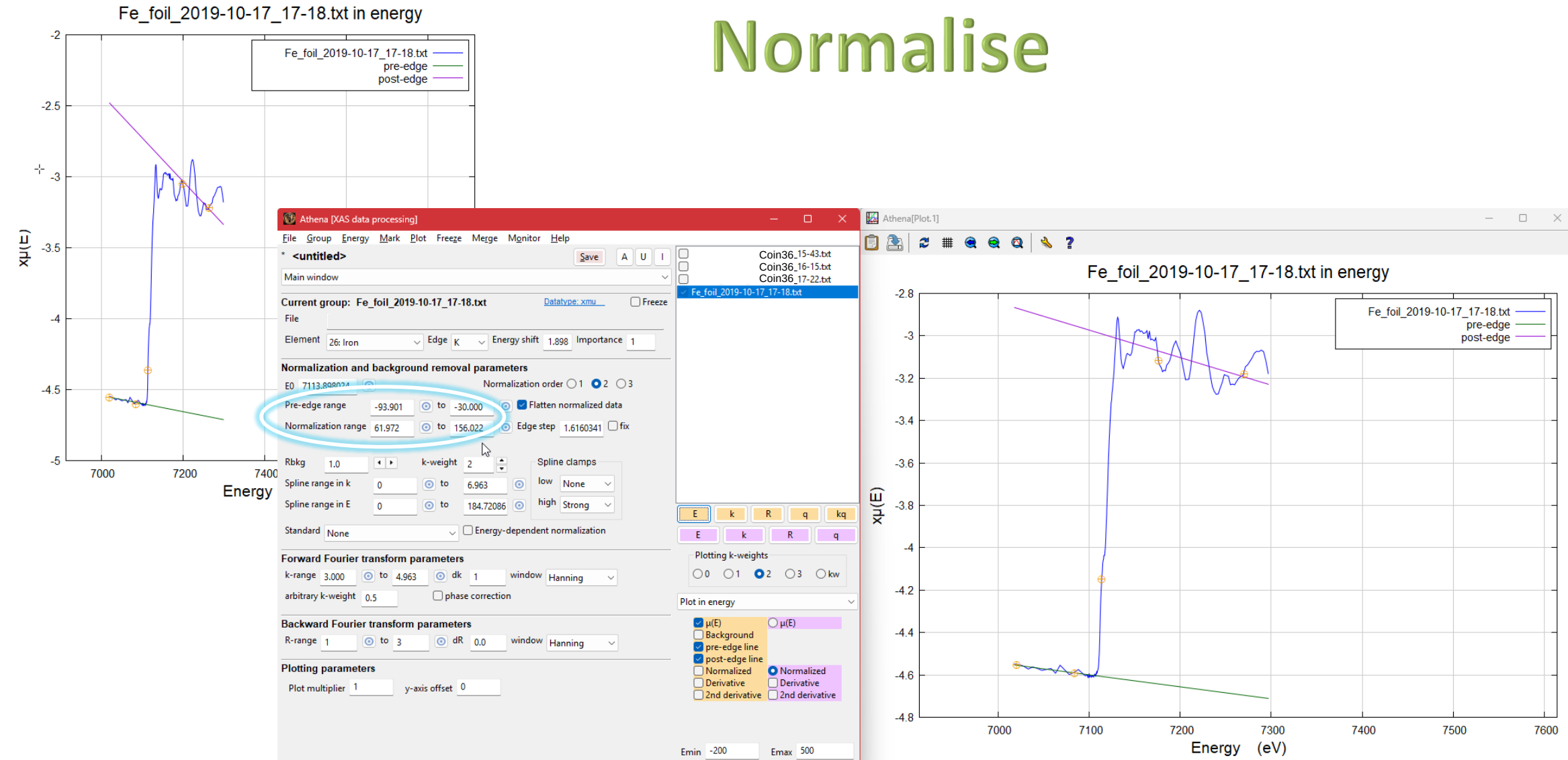
Calibrate energy (from reference)



Apply the same (instrumental) shift to the other spectra: copy and paste the **Energy shift**

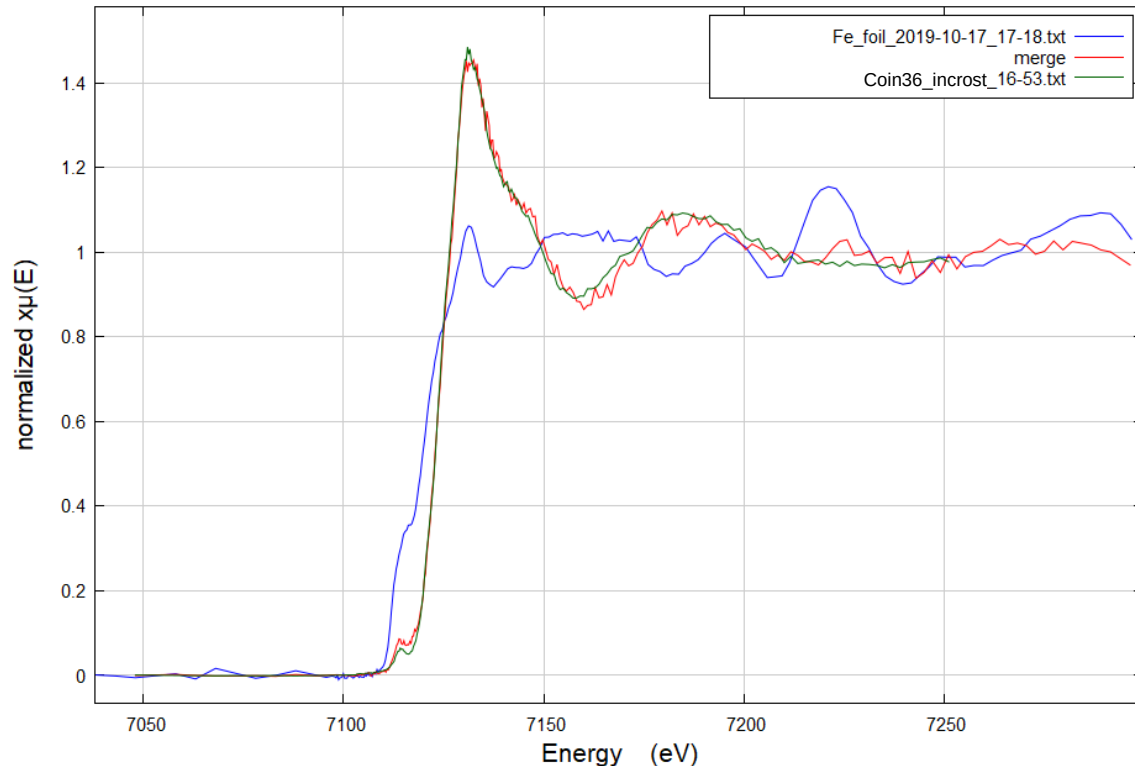
This works only for data collected in the same experimental condition (same beamline and beamtime!)

Normalise



Modify the **pre-edge range** and **normalization range** to get nice pre-edge and post-edge lines

Now you can start comparing the spectra



If we compare our spectra with the Fe foil, there is something missing...

Try loading other Fe references.

Add more references

Some beamlines can measure samples and references at the same time.

When this is the case, you can (and should!) **import reference channel**

Spectra with references will be shown in a different way in the data list:

Athena: Column selection

Select range Clear numerator Pause plotting

Energy 1 2 3 4 5 6 7 8 ☒ ☐ ☐ ☐ ☐ ☐ ☐ ☐

Numerator ☐ ☐ ☐ ☐ ☒ ☐ ☐ ☐

Denominator ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐

☐ Natural log ☐ Invert Multiplicative constant 1

☐ Save each channel as its own group

Data type xanes Energy units eV Replot

Energy okvua.1

$\mu(E)$ (okvua.5) / (1)

Preprocess Rebin Reference

☒ Import reference channel

1 2 3 4 5 6 7 8

Numerator ☐ ☐ ☐ ☐ ☐ ☒ ☐ ☐

Denominator ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐

Replot reference ☐ Natural log ☒ Same element

OK Cancel About

```
# Project Name: Parlett20195418_0
# Folder Name: Data_0
# File Name: FeO_000.txt
# Data and Time: Wednesday, 10 March, 2021
# Ring energy (GeV) = 2.0
# Ring current (mA) = 308.7
# Slits V GAP (mm) = 1.500
# Slits V CENTER (mm) = 0.000
# Slits H GAP (mm) = 4.000
# Slits H CENTER (mm) = 2.500
# Table Vertical (mm) = 0.00
# Table Horizontal (mm) = 0.00
# Huber X (mm) = 15.00
# Huber Y (mm) = 0.00
# Huber Z (mm) = 7.00
# Huber Theta (mm) = 90.00
# Ref. sample position (mm) = 118.00
# MultiSample position (mm) = 72.10
# Picoam I0 gain = 10^^
# Picoam I1 gain = 10^^
# Picoam I2 gain = 10^^
# Monochromator Crystal: Si 111
# Monochromator Energy (eV) = 8348.60
# Auxiliary Equipment = NORMAL
# Optional Parameters = ;;
#
#EnergyMot (eV) EnergyEnc (eV) I0 (counts) I1 (cc
6967.008439 6967.026701 113586.000000 1172010.0000
6972.011331 6972.043337 112940.000000 1169731.0000
6976.998833 6977.067520 112004.000000 1164655.0000
6981.993788 6982.016718 111047.000000 1159265.0000
6986.996212 6987.014584 109972.000000 1152513.0000
6992.006123 6992.019922 108401.000000 1140379.0000
6997.000503 6996.991290 108900.000000 1149792.0000
7002.002333 7002.011560 108555.000000 1150343.0000
7006.988529 7006.997769 108380.000000 1152787.0000
7012.005274 7011.949749 107968.000000 1152734.0000
```

☐ Coin36_cheek_2019-10-16_15-43.txt

☐ Coin36_cheek_2019-10-16_16-15.txt

☐ Coin36_cheek_2019-10-16_17-22.txt

☒ Fe_foil_2019-10-17_17-18.txt

☒ merge

☒ FeO_000.txt

☐ Ref FeO_000.txt

☒ Magnetite_Fe3O4_001.txt

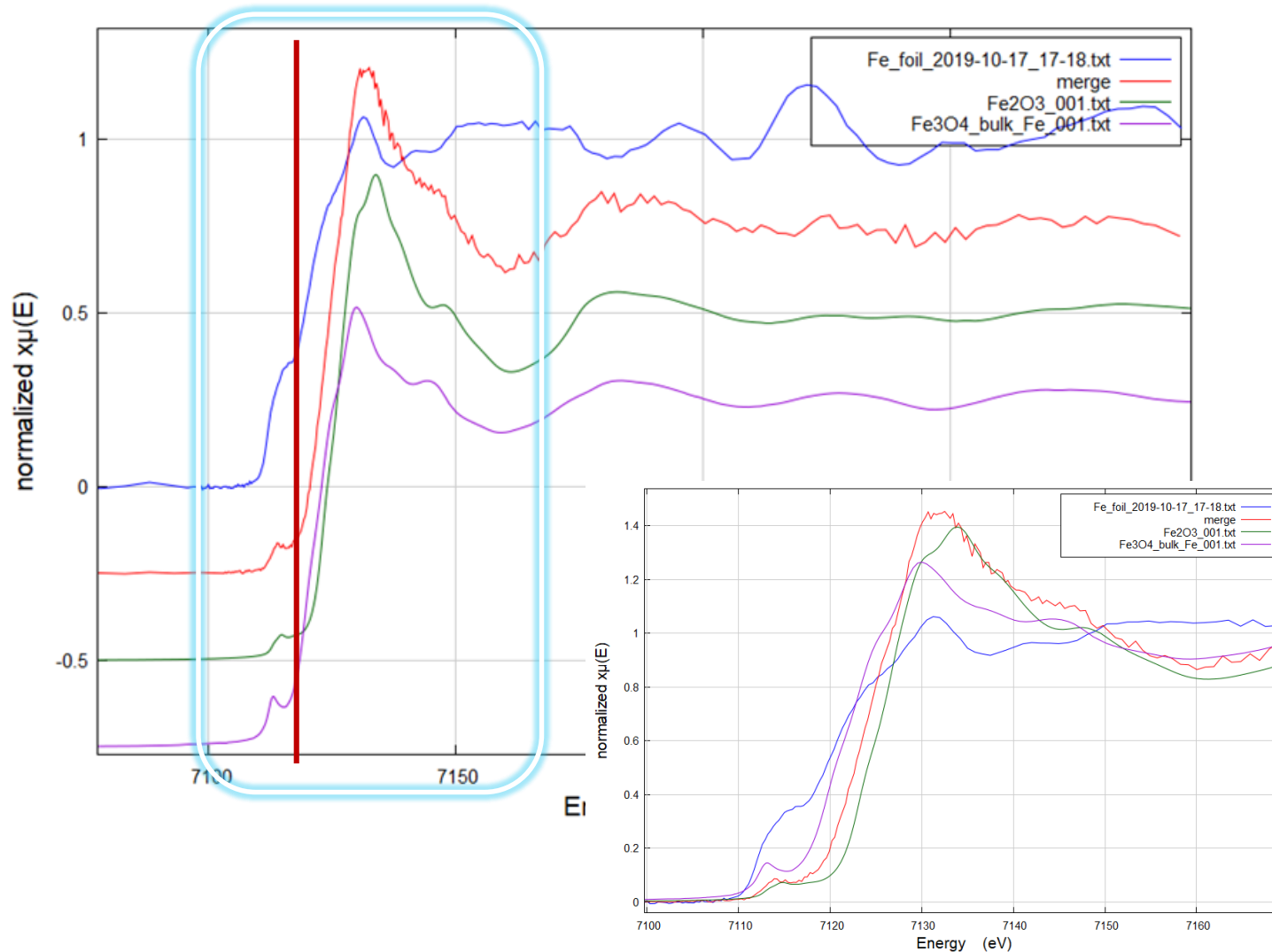
☐ Ref Magnetite_Fe3O4_001.txt

Calibrate the Ref spectra...the associated sample spectra will be automatically aligned!

E k R q kq

E k R q

Add more references



Metallic Fe starts rising before the oxides.

Fe_3O_4 - mix of Fe(III) and Fe (II) – edge jump is found at lower energy than Fe_2O_3 – which is only Fe(III).

The sample is something in between...

Exercise #2



I. Start Athena*

II. Linear Combination Fitting

Same data folder

<https://drive.elettra.eu/d/b0b0c8eb775749138b10/>

**Complete documentation available!*

<http://bruceravel.github.io/demeter/documents/Athena/index.html>

XANES: dense of electronic and structural information

XANES signal is stronger than EXAFS

- less sensitive to data statistics, sample quality, beam intensity
- can be measured on less concentrated samples
- can be measured faster than EXAFS (time resolved experiments)

Damping of XANES signal due to structural disorder is weak

- Applications to extreme condition studies: High **T**, High **P**, ...

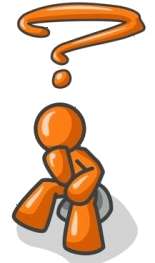
Electronic structure (DoS) and structural topology

- XANES specially sensitive to the valence state, coordination chemistry, ligand symmetry of the absorber
- Can be used as fingerprint for chemical speciation in mixtures and inhomogeneous systems

Restricted energy range around the edge

- Measurements at low energies (i.e. carbon, oxygen, nitrogen K-edges)
- Fast data collection (time resolved XAS)
- XANES Microprobes (mapping) with sub- μm resolution

XANES theory is
complex

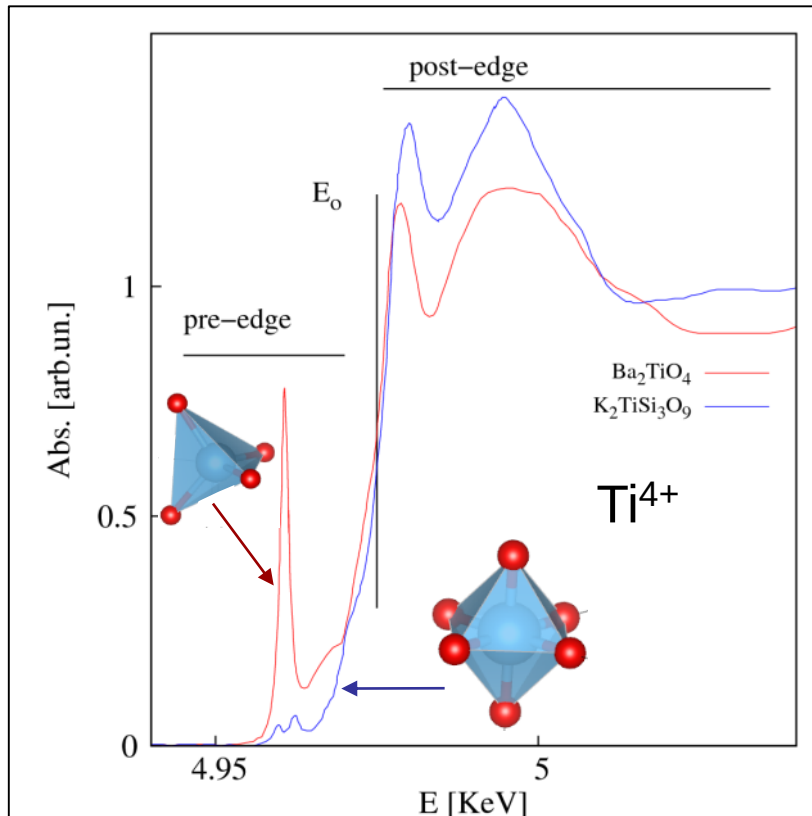


XANES are prone to simple
interpretation for simple and fast
(semi-)quantitative analysis

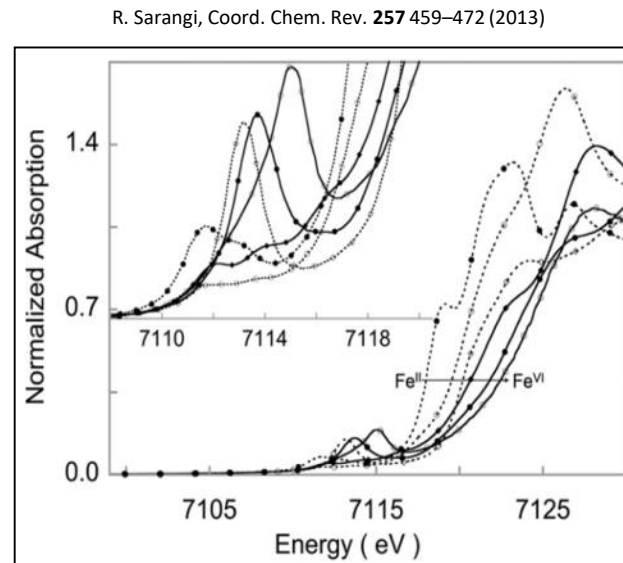
Deeper insight into the XANES region

XANES features originate from the **absorber**:

- electronic nature (**valence**)
- coordination **chemistry**
- coordination **geometry**

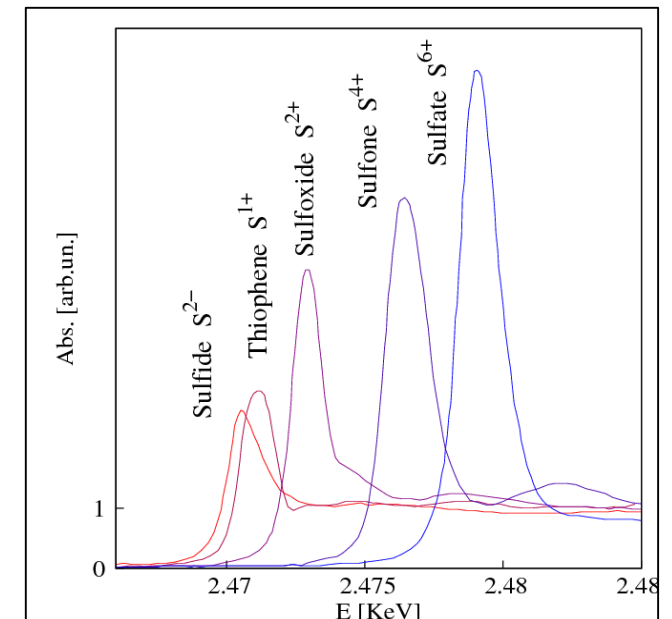


Local symmetry and XANES in Ti⁴⁺ compounds



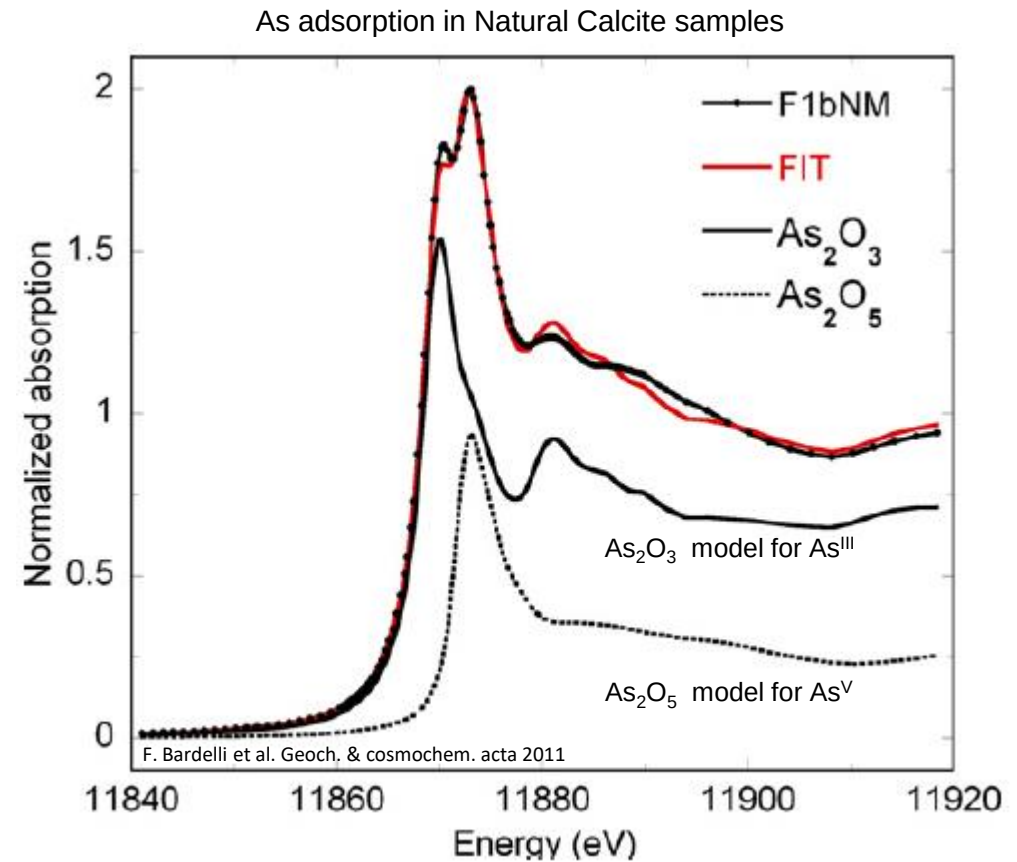
Fe K edges: representative XANES Fe in complexes

Sulphur K edges: chemical shift as a function of valence state of S-ions

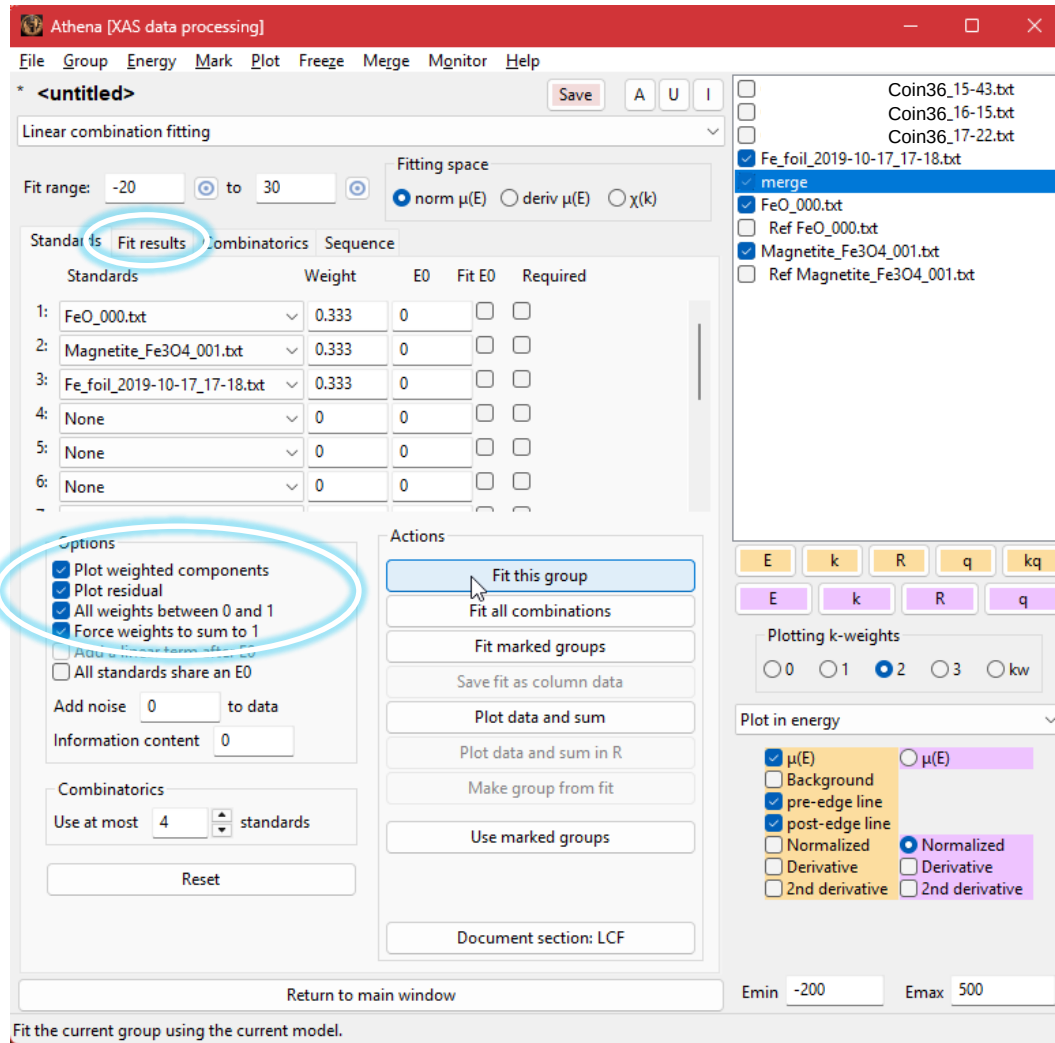


Analysis of mixtures: Linear Combination Analysis

$$\mu^{th} = \sum_j \alpha_j \mu^{ref_j}$$
$$R^2 = \sum_i (\mu^{exp}(E_i) - \mu^{th}(E_i))^2$$



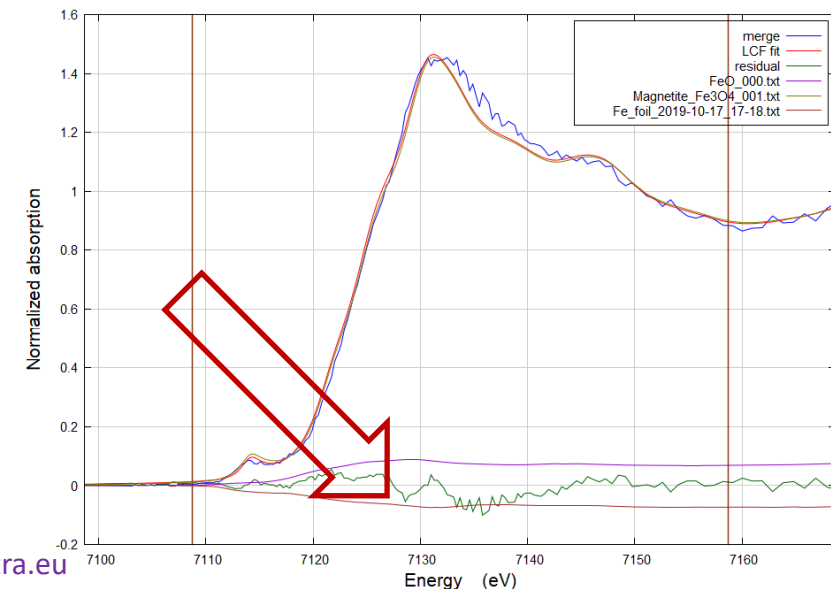
Linear Combination Fitting



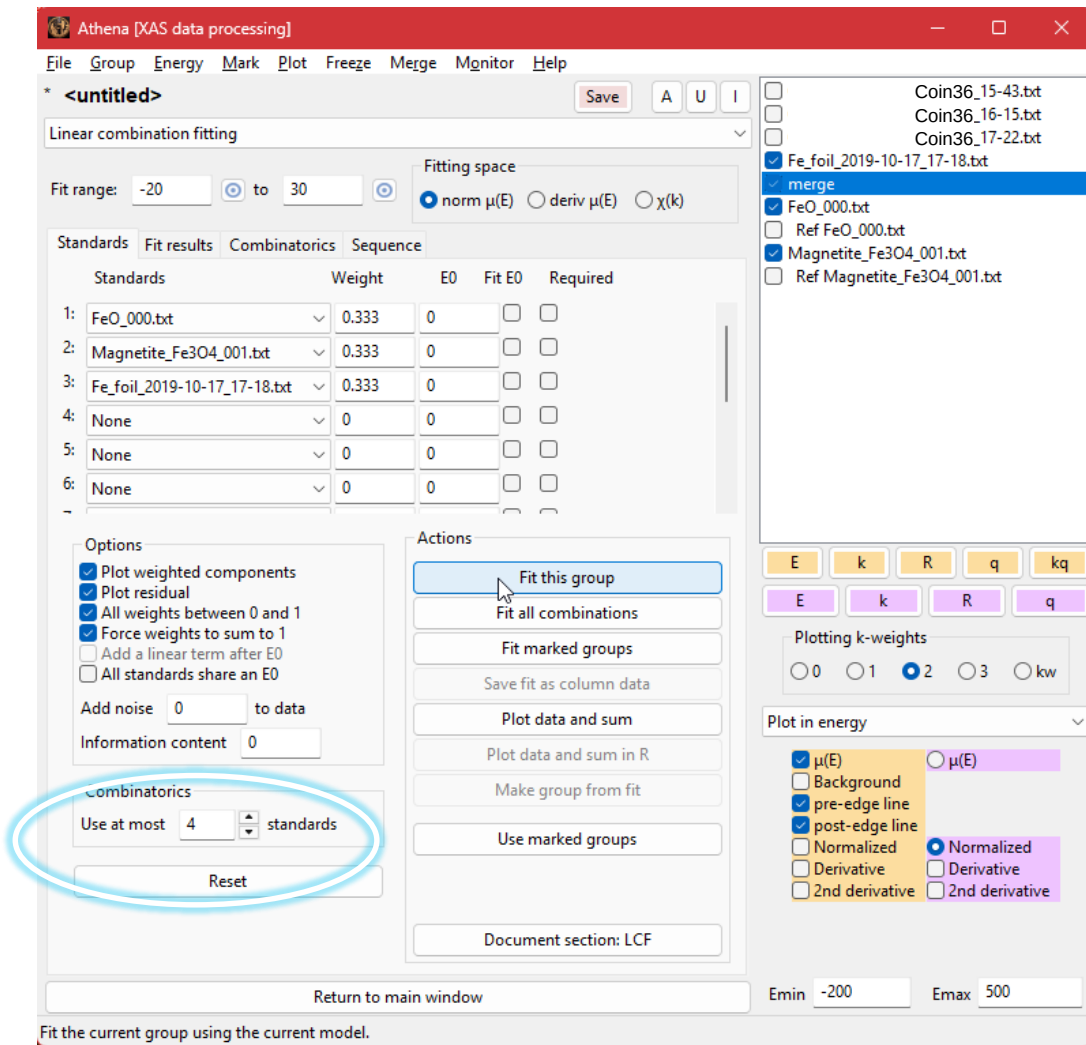
The action (fitting) is always performed on the highlighted (current) dataset

Include the references and **fit this group**.

Check the output and verify possible inconsistencies!
 χ^2 can be found under “**Fit results**”, but check also for negative contributions!



Linear Combination Fitting



How many components?

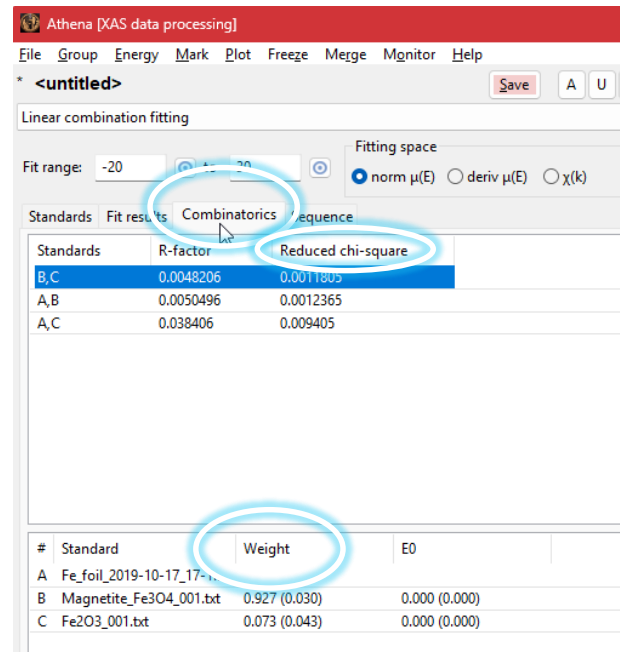
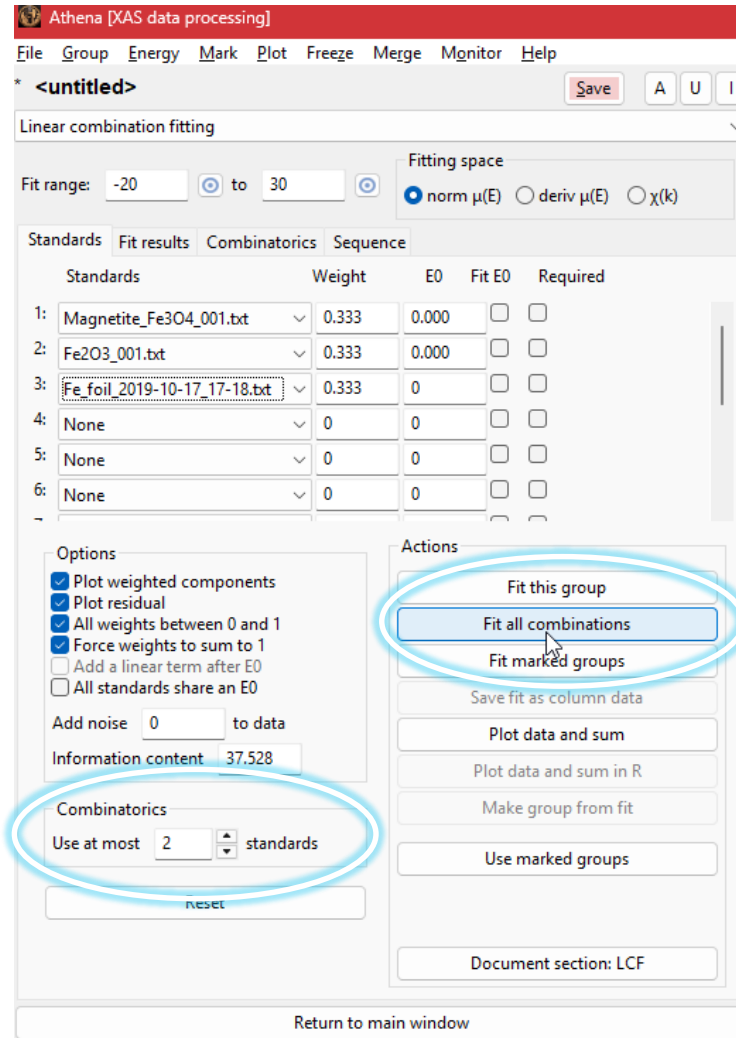
3+ standards can be selected to perform LCF if:

- the reduced χ^2 of best (n+1)-component fit decreases of $\geq 10\%$ with respect to the n-component fit
- if all the individual components account $> 5\%$

Linear Combination Fitting

Try selecting 3 references (Fe foil, Fe_2O_3 , Fe_3O_4), then fit using **at most 2** of them.

“Fit all combination”



In “combinatorics” you can easily compare all the results at once: check the reduced χ^2 and the weight of each contribution...

Linear Combination Fitting

Athena [XAS data processing]

File Group Energy Mark Plot Freeze Merge Monitor Help

* <untitled> Save A U I

Linear combination fitting

Fit range: -20 to 30 Fitting space: ☒ norm $\mu(E)$ ☐ deriv $\mu(E)$ ☐ $\chi(k)$

Standards	Weight	E0	Fit E0	Required
1: FeO_000.txt	0.641	0.000	☐	☐
2: None	0	0.000	☐	☐
3: Fe_foil_2019-10-17_17-18.txt	0.359	0.000	☐	☐
4: None	0	0	☐	☐
5: None	0	0	☐	☐
6: None	0	0	☐	☐

Options:

- ☒ Plot weighted components
- ☒ Plot residual
- ☒ All weights between 0 and 1
- ☒ Force weights to sum to 1
- ☐ Add a linear term after E0
- ☐ All standards share an E0

Add noise 0 to data

Information content 37.528

Combinatorics

Use at most 4 standards

Reset

Actions:

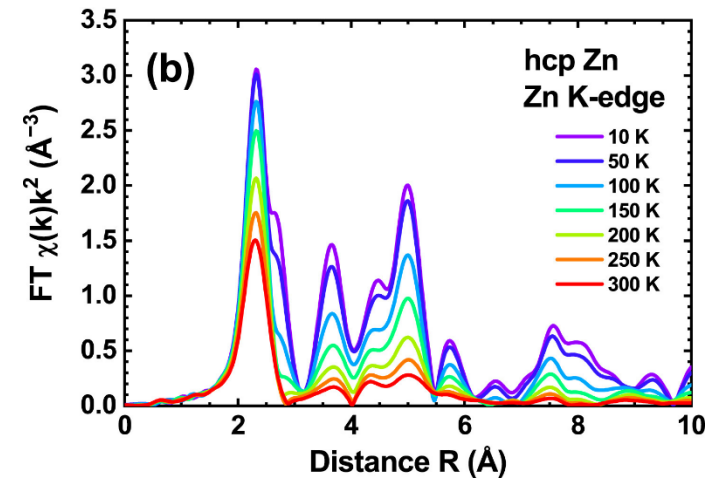
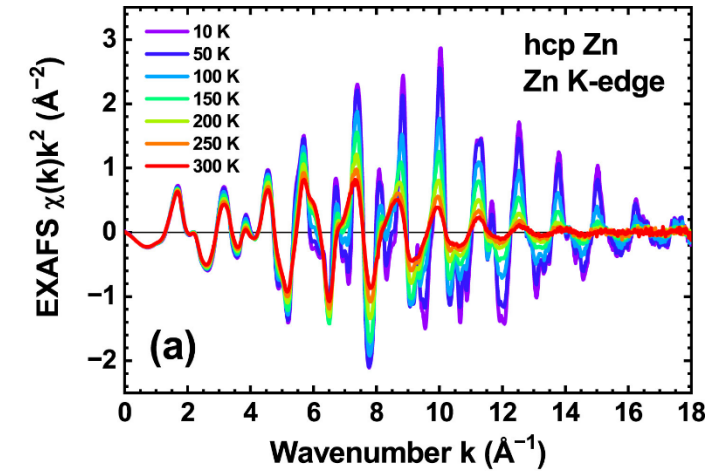
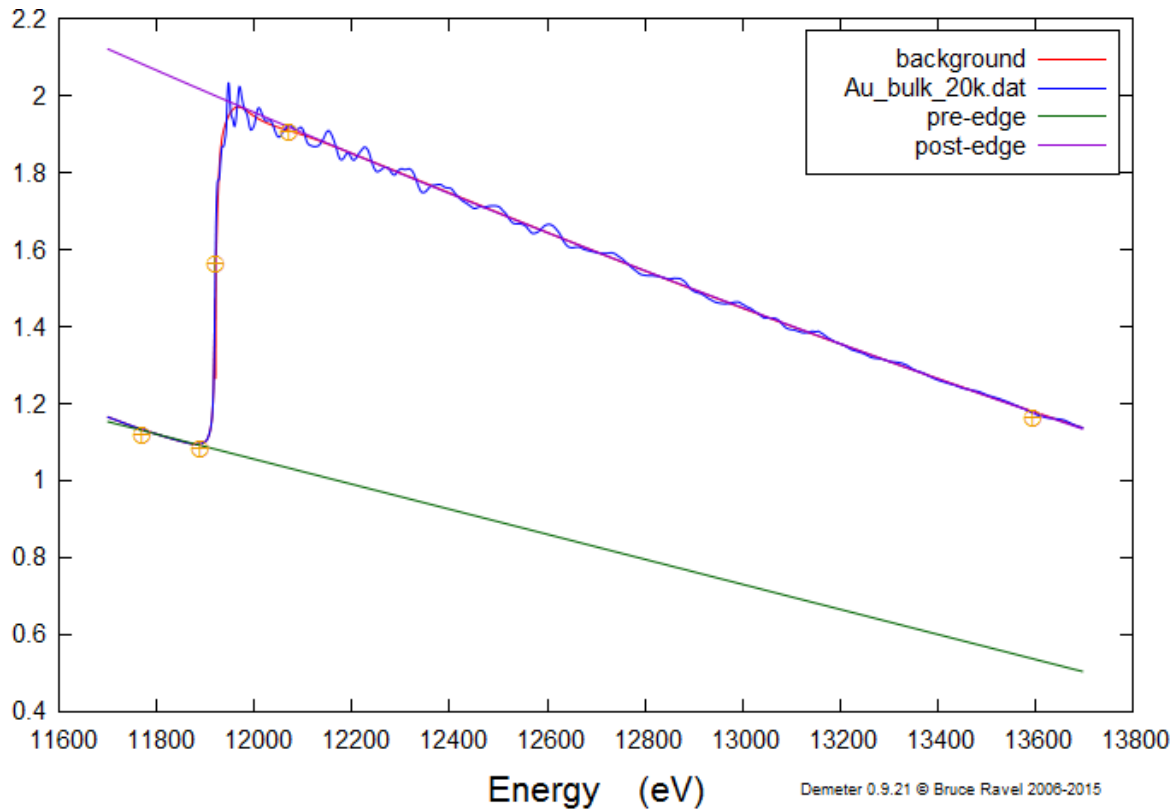
- Fit this group
- Fit all combinations
- Fit marked groups
- Save fit as column data
- Plot data and sum
- Plot data and sum in R
- Make group from fit
- Use marked groups
- Document section: LCF

Return to main window

Fit all marked groups using the current fitting model.

Once you are happy with your fit results: select the individual curve and **Save as column data**.

From XANES to EXAFS

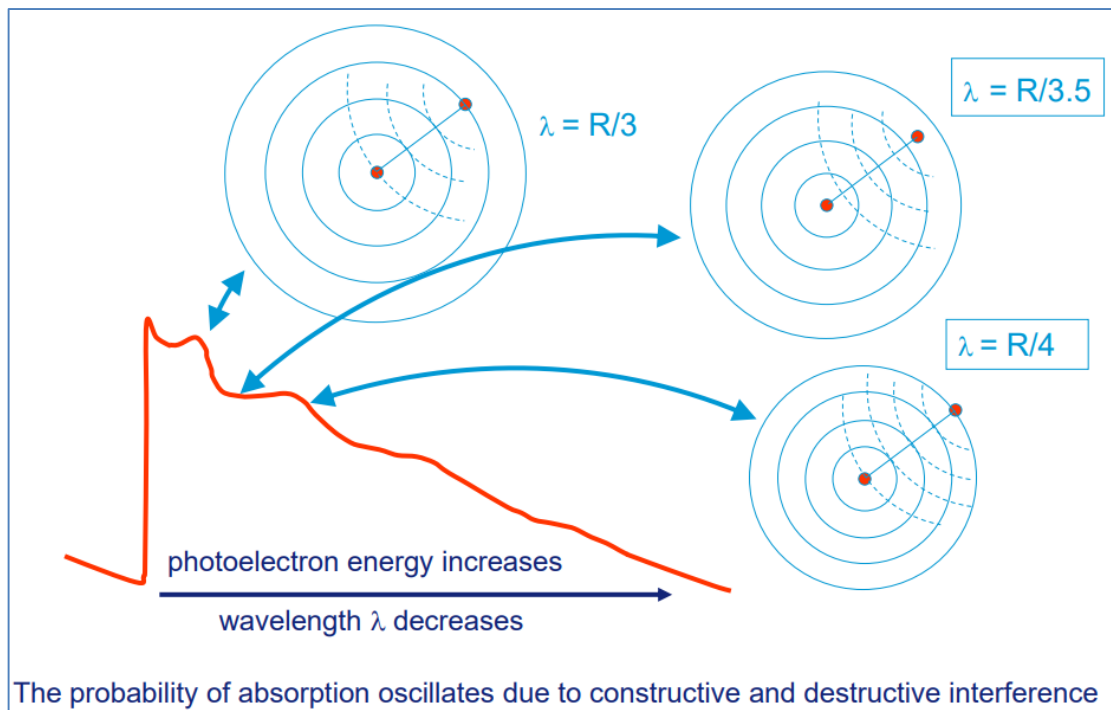


Dimitrijevs et al., Physica B: Condensed Matter 739 (2026) 418945

Why EXAFS?

EXAFS	Crystallography
Little to no sample preparation, any condition (temperature, pressure, potential...)	Needs high quality crystals
Bond length precision < 0.02 Å	Bond length precision depends on resolution
Short- to mid-range distances only	Complete 3D structure
Can't distinguish scatterers with similar Z (e.g. Fe from Co, N from C or O)	
Large uncertainty in coordination numbers	

The general idea



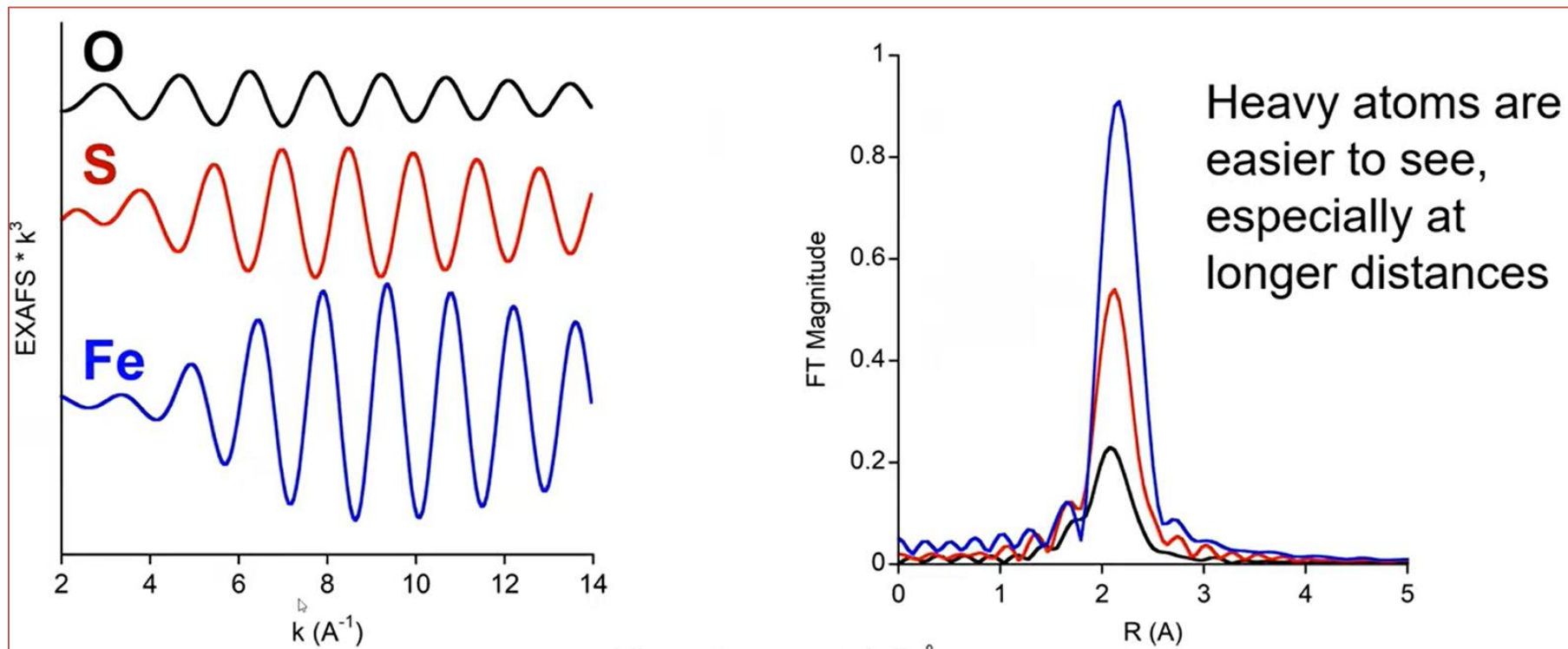
EXAFS depends on the interference effects of neighboring atoms on the photoelectron created during the absorption process.

Distance and Z affect the frequency and amplitude of the oscillations.

We can calculate the theoretical EXAFS contribution of each atom (FEFF), then add them up.

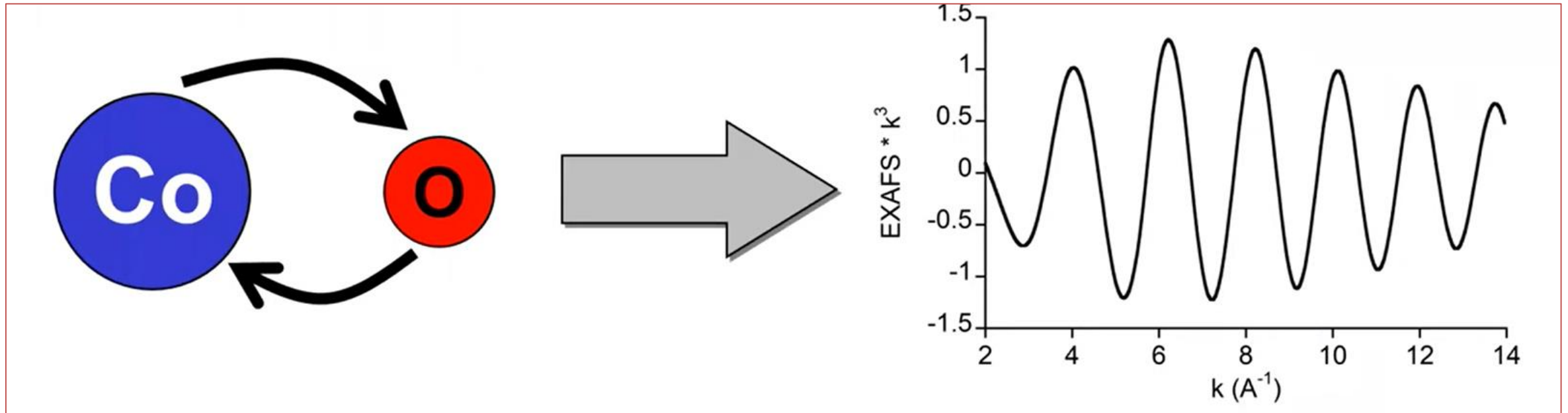
The general idea

With Z increasing, the **scattering amplitude** increases
and the **envelope** peaks at higher k



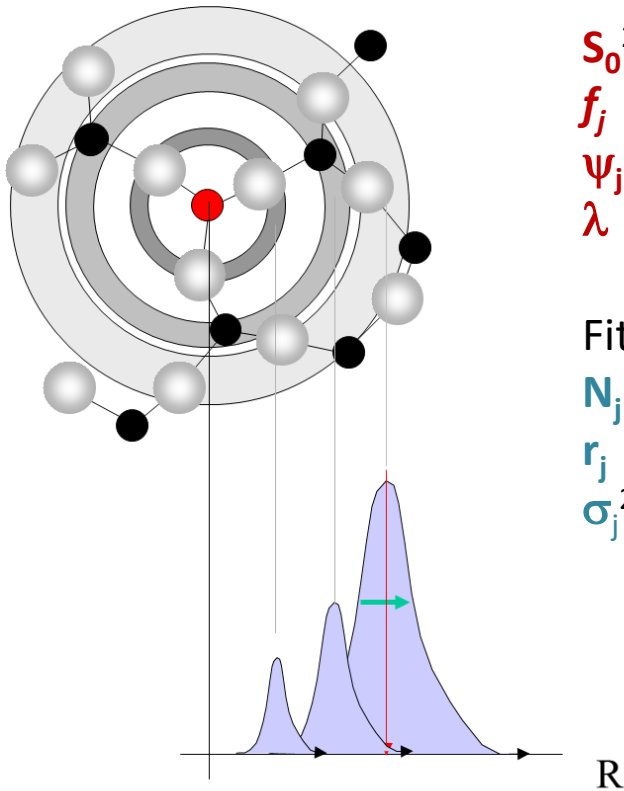
The general idea

The idea is to start from a model (we have to know something about the sample and/or to make assumptions), then **fit the scattering paths** instead of single peaks.



The EXAFS formula

$$\chi(k) = \frac{1}{k} \sum_j S_0^2 \frac{N_j}{r_j} |f_j(k)| e^{-2k^2 \sigma_j^2} e_j^{-\frac{2r_j}{\lambda}} \sin(2k r_j + \delta_j)$$



S_0^2 = many body losses

f_j = photo-electron scattering amplitude

ψ_j = photo-electron scattering phase shift

λ = photo-electron mean free path

Fitting parameters:

N_j = multiplicity (coordination number)

r_j = half path length (coordination distance)

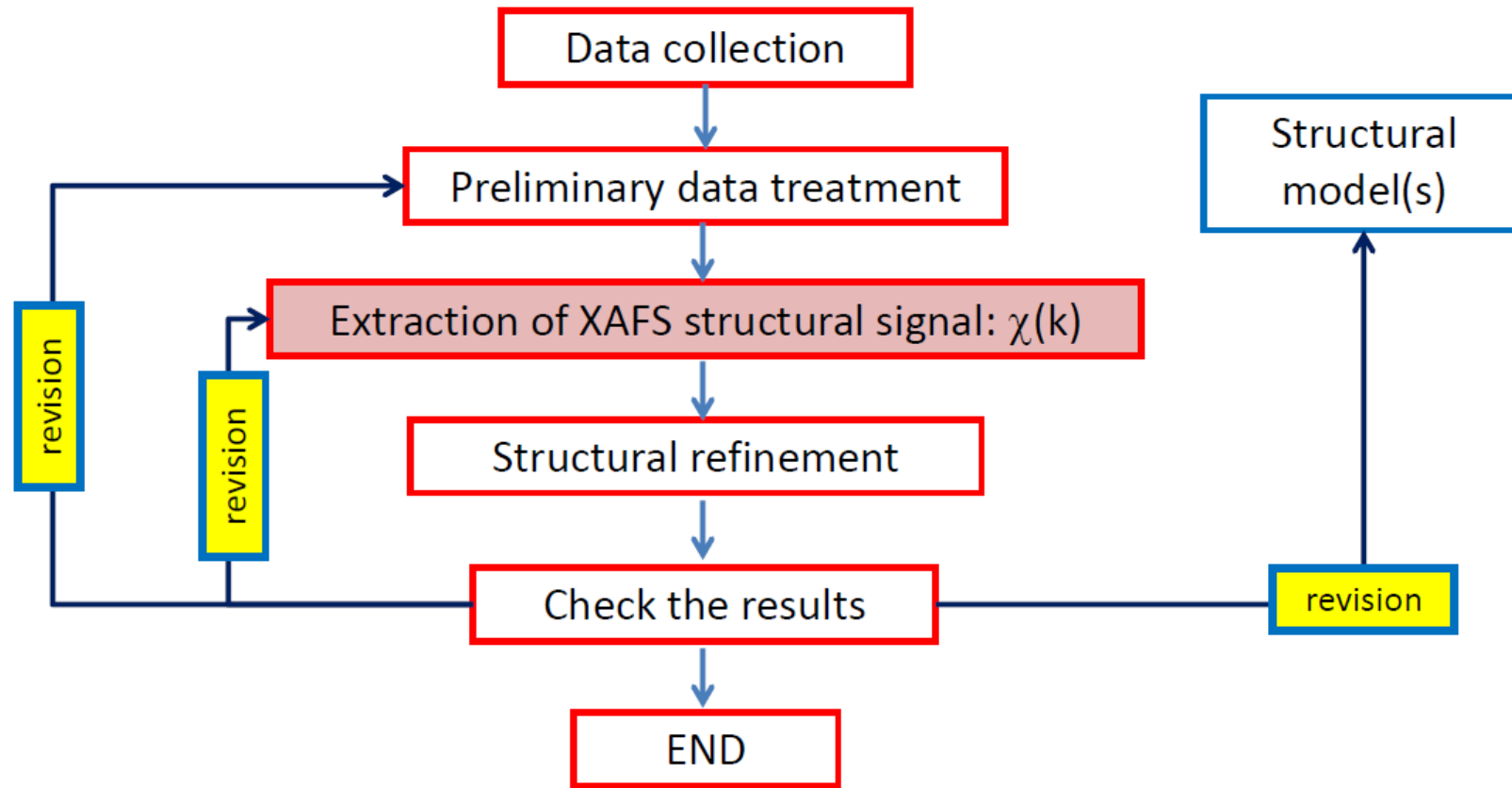
σ_j^2 = variance of the path length distribution

Debye-Waller factor

(Mean Square Relative Displacement: MSRD)

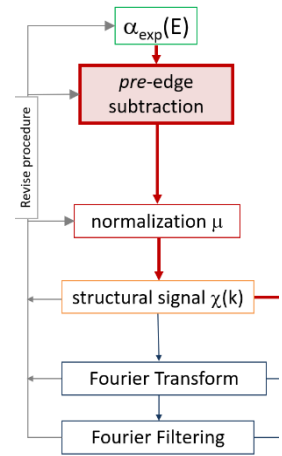
- f_j and δ_j depend on Z of the scatterer
- N_j and S_0^2 are **strongly correlated** and modulated by σ_j^2 : that's where the N large uncertainty originates!

From XANES to EXAFS

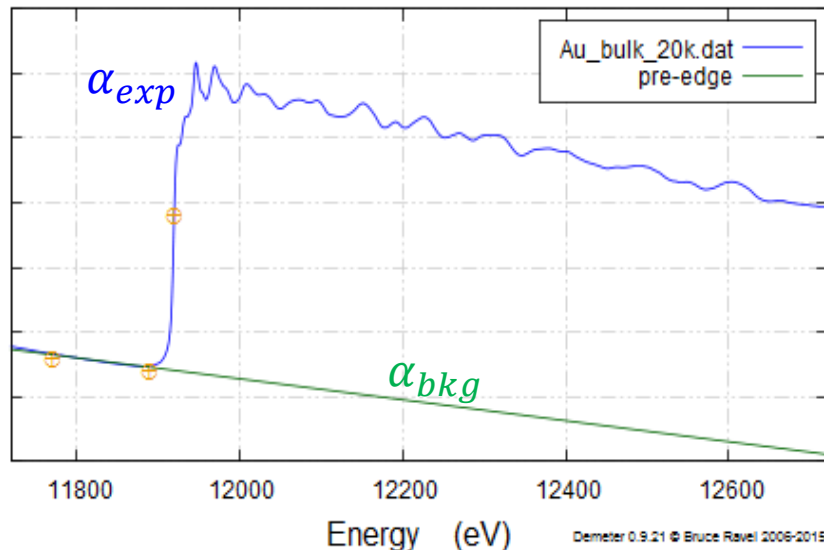


How to get the normalized $\chi(k)$

Pre-edge removal



$$\alpha_{exp} = \alpha_x + \alpha_{bkg} = \mu_x t + \alpha_{bkg} \implies \alpha_x = \mu_x t = \alpha_{exp} - \alpha_{bkg}$$



$\mu_x t$ is the absorption due to the edge

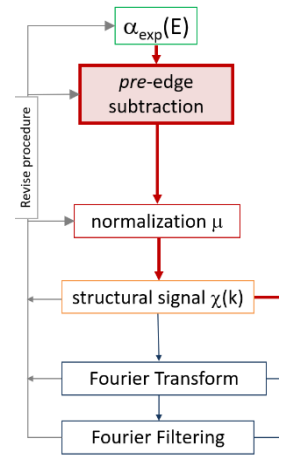
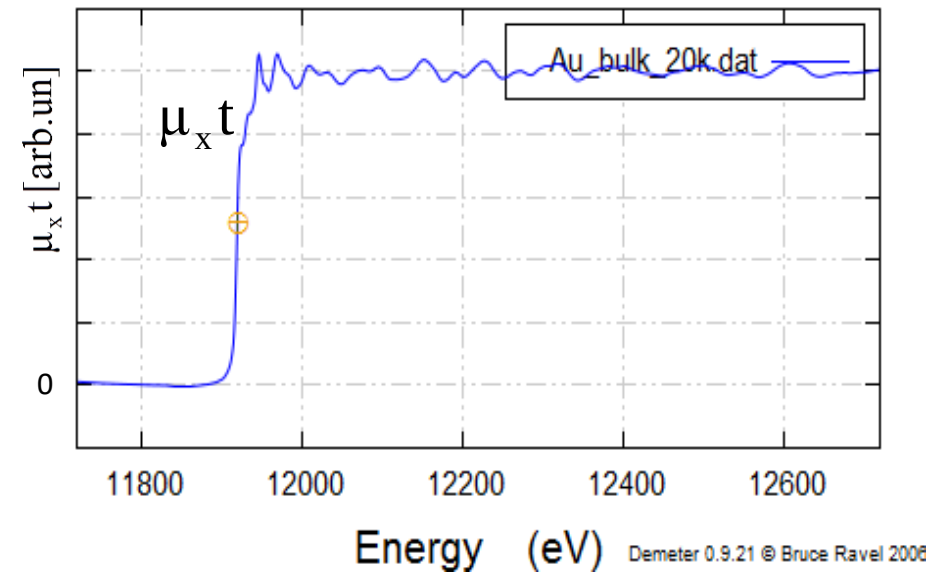
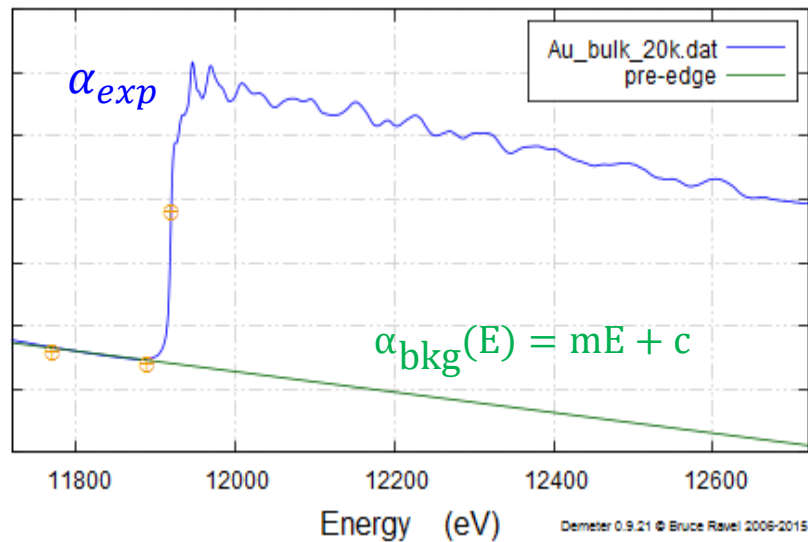
α_{bkg} is the absorption due to **everything** except $\mu_x t$

other absorption edges, air, sample holder, matrix, windows, etc...

How to get the normalized $\chi(k)$

Pre-edge removal

$$\alpha_{exp} = \alpha_x + \alpha_{bkg} = \mu_x t + \alpha_{bkg} \implies \alpha_x = \mu_x t = \alpha_{exp} - \alpha_{bkg}$$

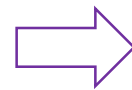
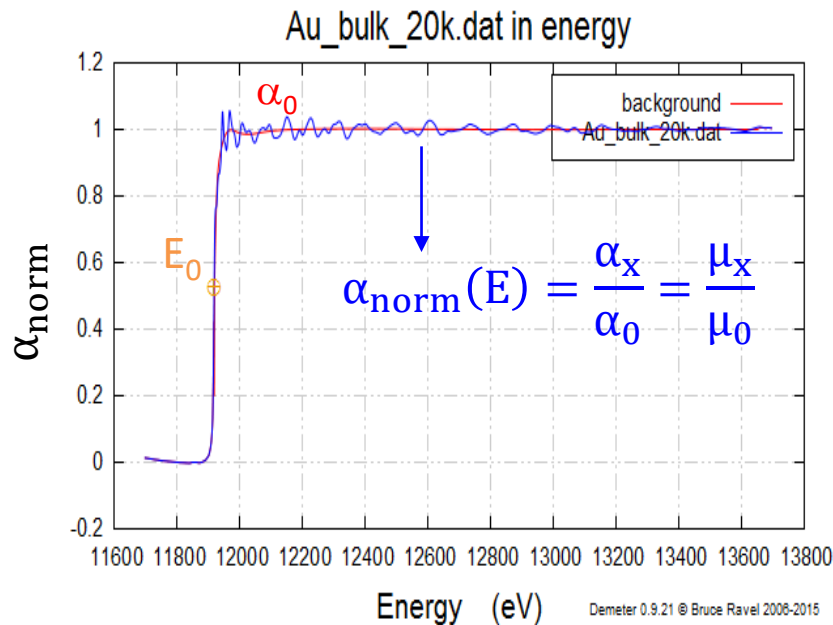


The pre-edge is simulated as a linear regression of data before the edge energy

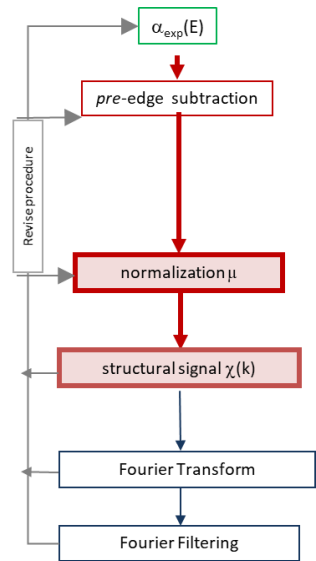
How to get the normalized $\chi(k)$

Normalization, χ and k

$$\alpha_x = \mu_x t \simeq \underbrace{\mu_0}_{\text{isolated absorber}} (1 + \underbrace{\chi}_{\text{effect of neighbours}}) t$$



$$\alpha_{\text{norm}} = \frac{\mu_0(1 + \chi)t}{\mu_0 t} = \frac{\alpha_x}{\alpha_0}$$

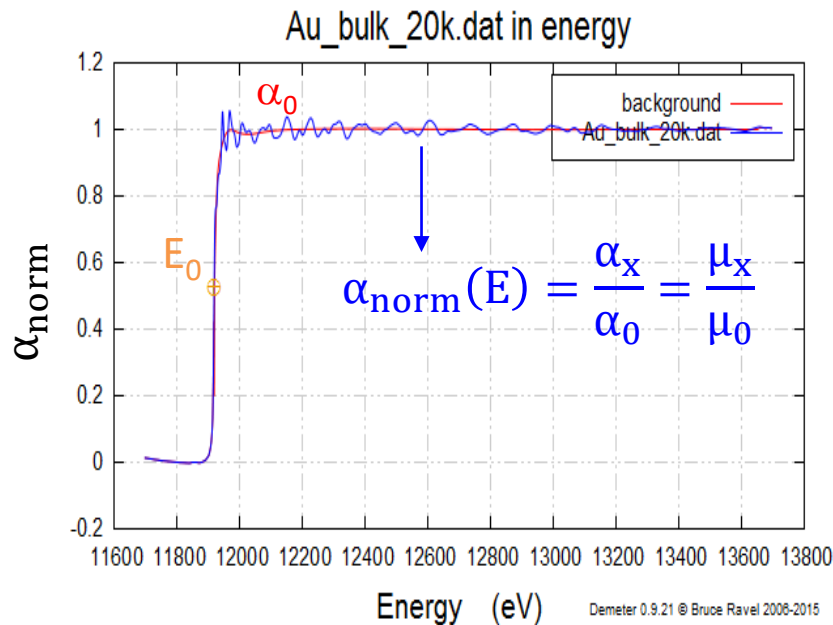
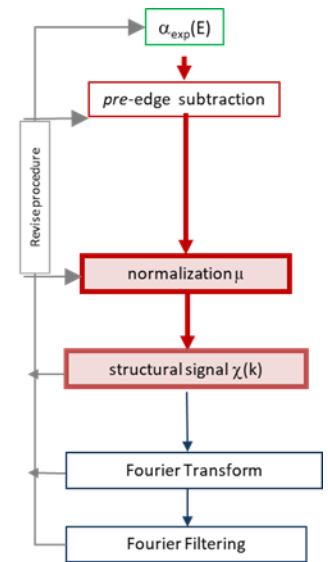


How to get the normalized $\chi(k)$

2. Normalization, χ and k

$$\alpha_{norm} = \frac{\mu_0(1 + \chi)t}{\mu_0 t} = \frac{\alpha_x}{\alpha_0}$$

$$\alpha_{norm} = (1 + \chi)$$



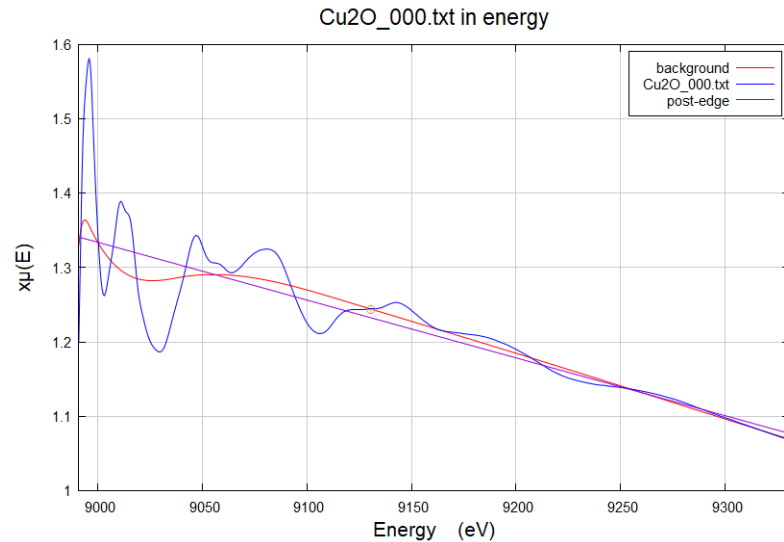
α_0 is **calculated empirically** as a smooth curve across the data.

Requirements for α_0 :

- 1) **Smooth enough** not to remove true structural features
- 2) **Structured enough** to remove not structural background structures

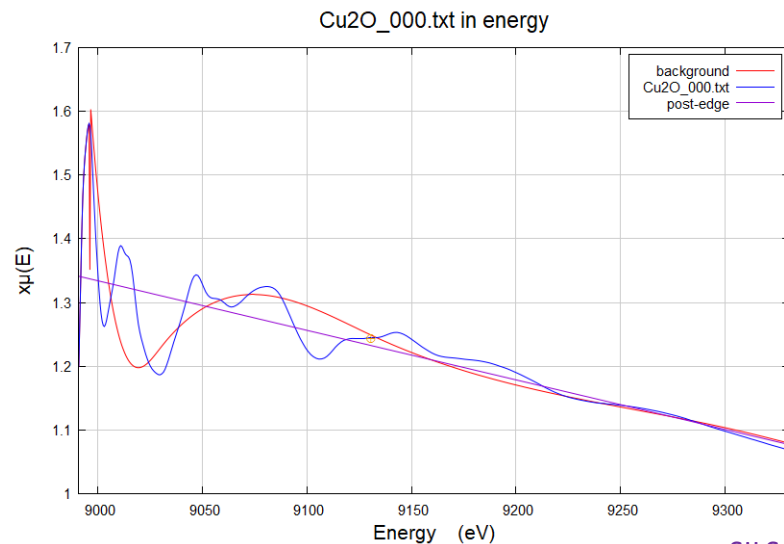
Different programs for XAFS data analysis apply different (**equivalent**) methods

Spline removal



Pre-edge background has little effect on EXAFS

Post-edge is more important: the absorber can scatter off its electrons, introducing **low frequency oscillations** that are not part of the EXAFS.



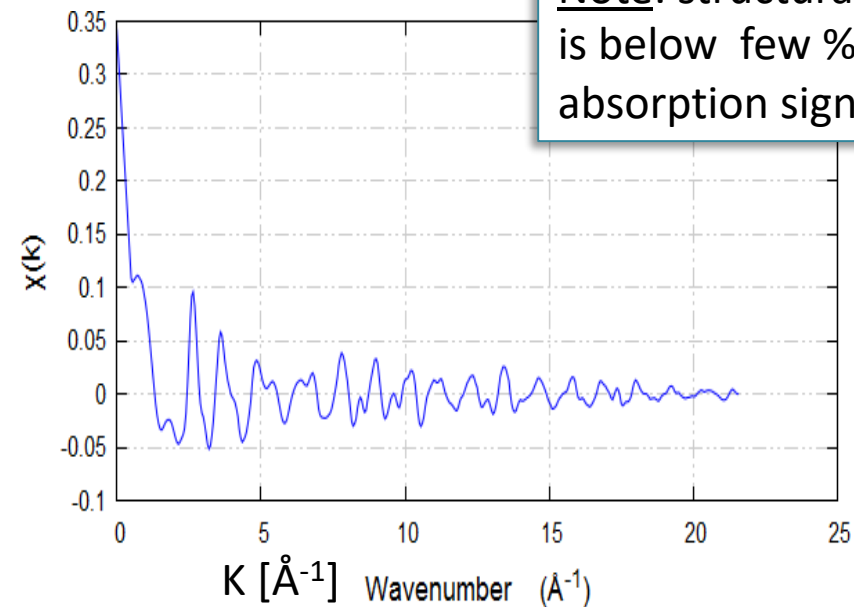
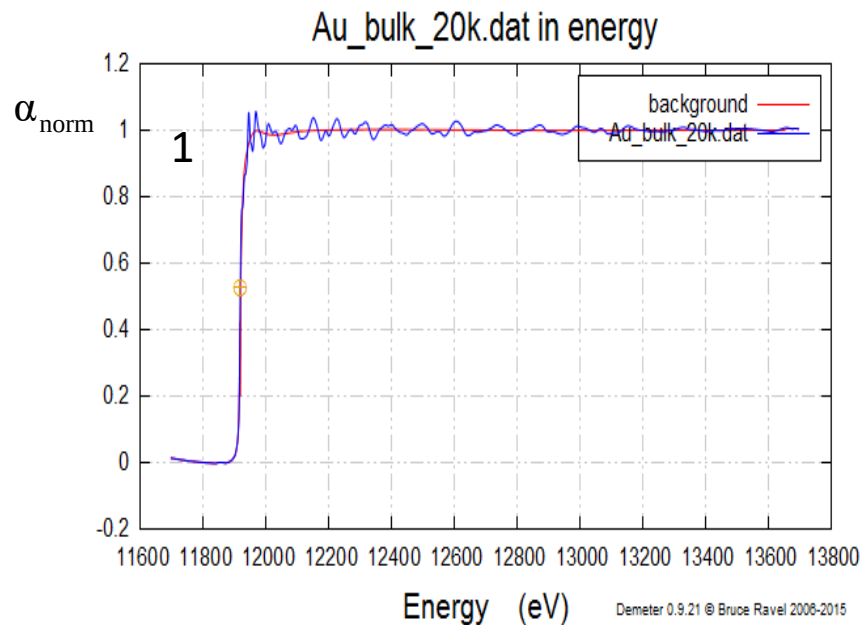
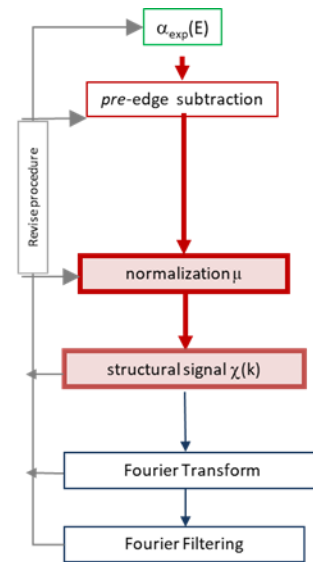
Spline removal should not remove true oscillations.

How to get the normalized $\chi(k)$

2. Normalization, χ and k

$$\alpha_{norm} = (1 + \chi)$$

$$\chi = 1 - \alpha_{norm}$$

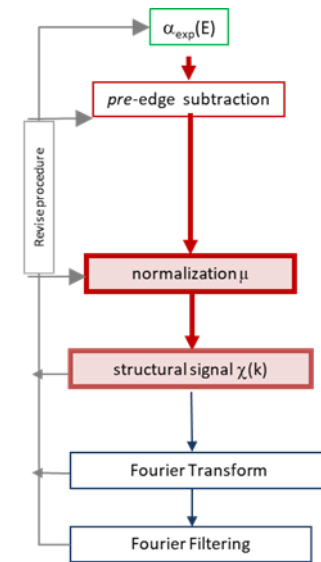
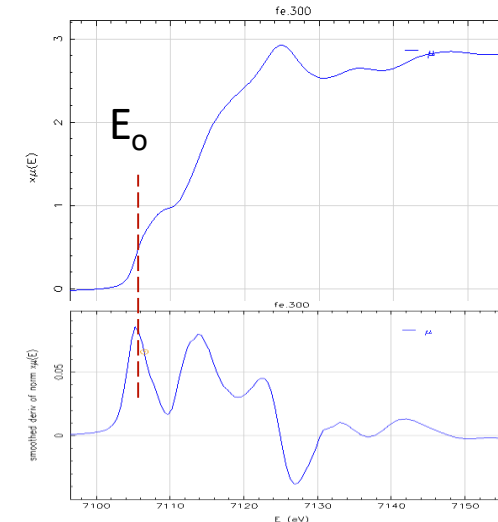
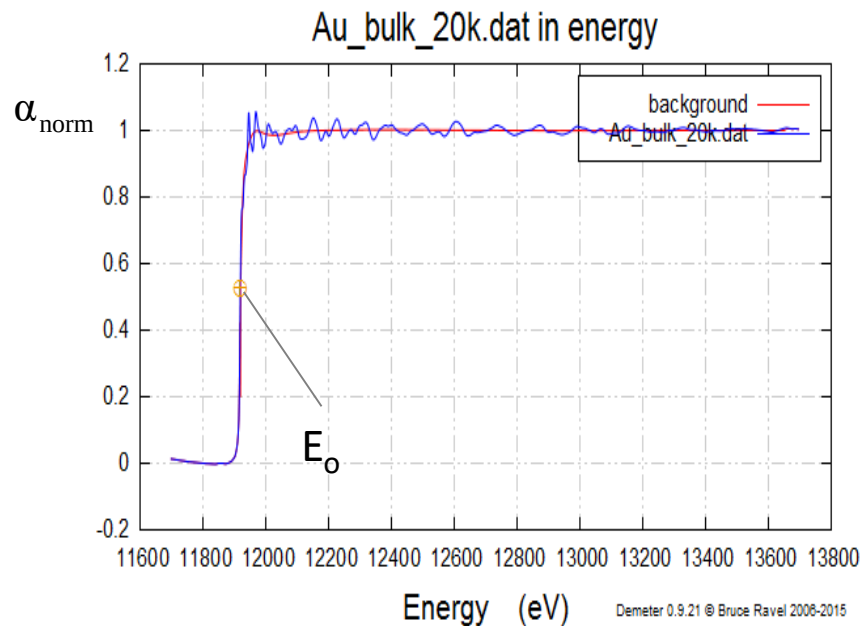


Note: structural signal (χ) is below few % of total absorption signal (μ_x)

How to get the normalized $\chi(k)$

2. Normalization, χ and **k**

$$k = \sqrt{\frac{2m(E - E_0)}{\hbar^2}} = \sqrt{0.2625(E - E_0)}$$



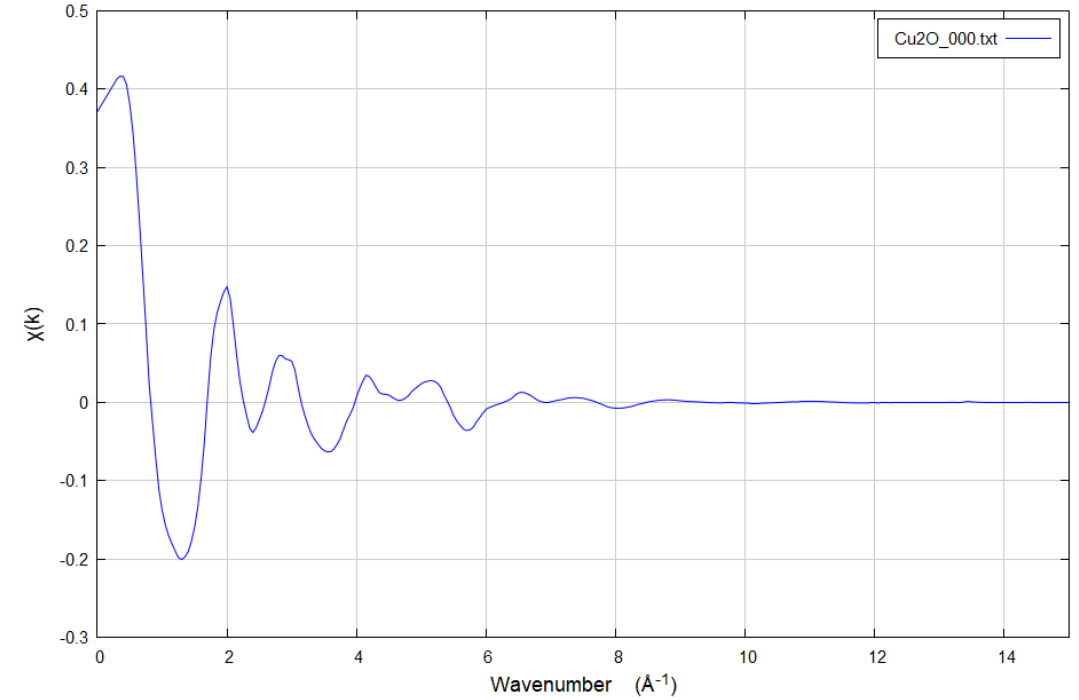
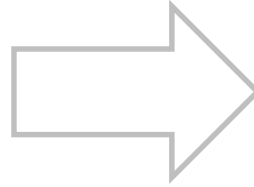
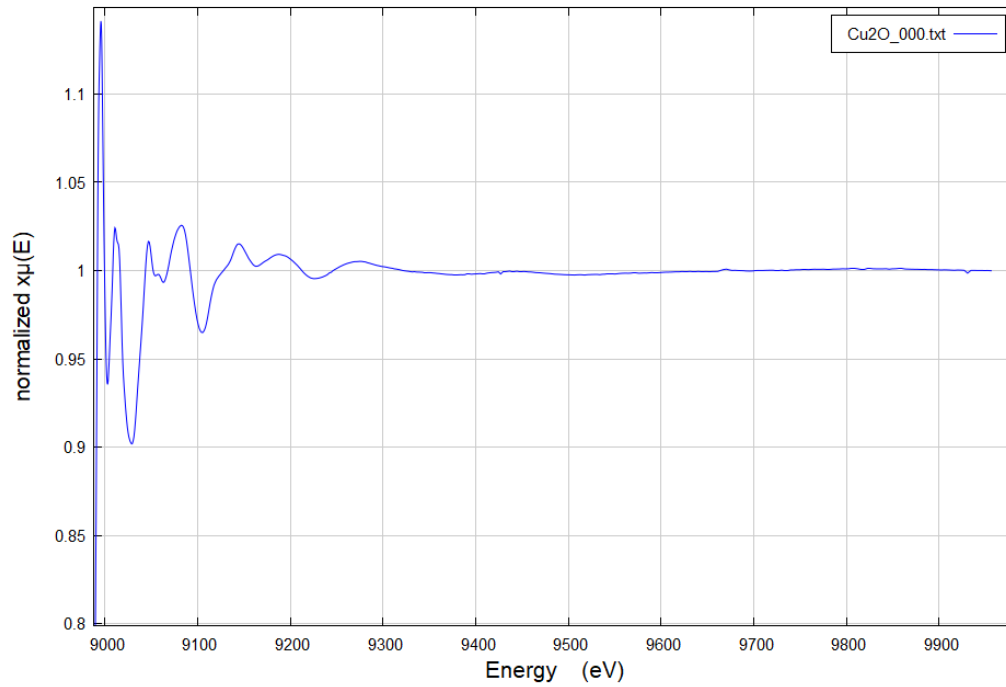
E_0 is typically chosen:

Metals: at the **first inflection point** of α_{nor}

Oxides: where $\alpha_{\text{nor}} \approx 0.5$

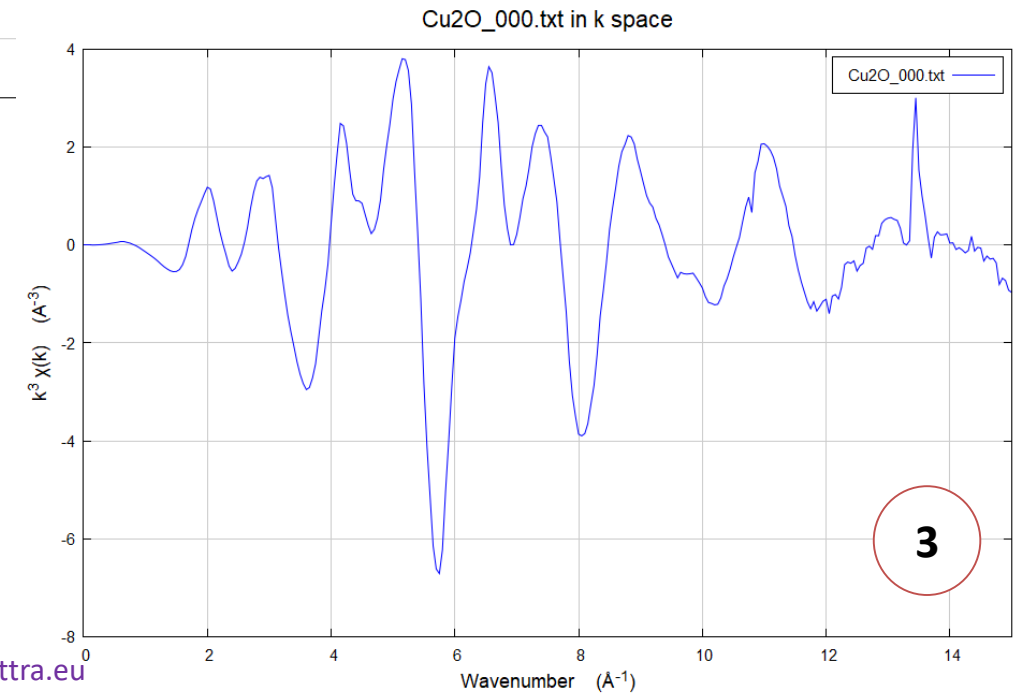
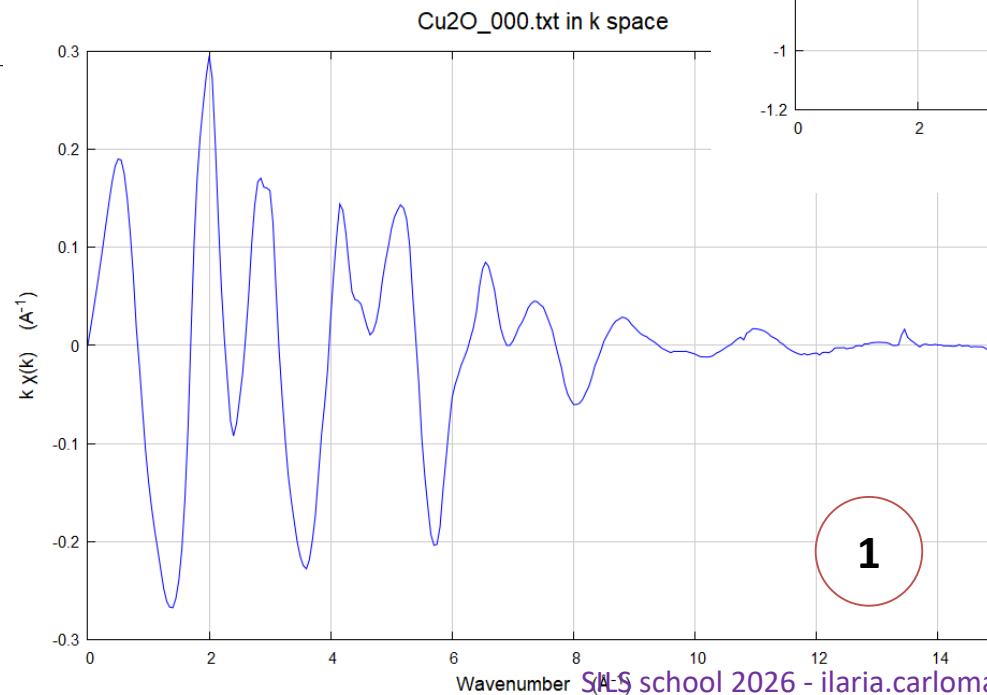
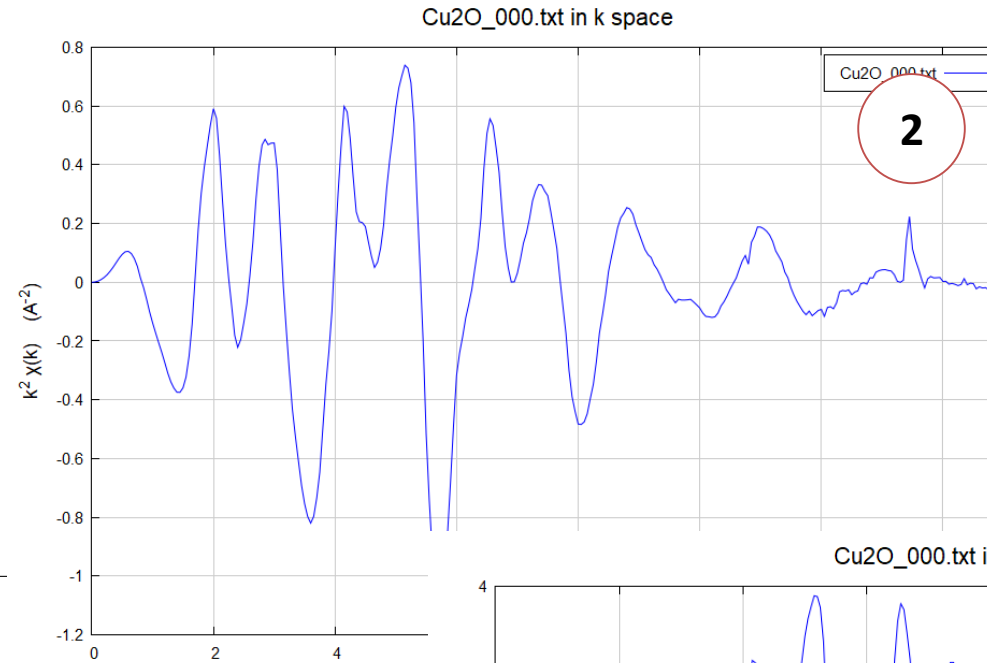
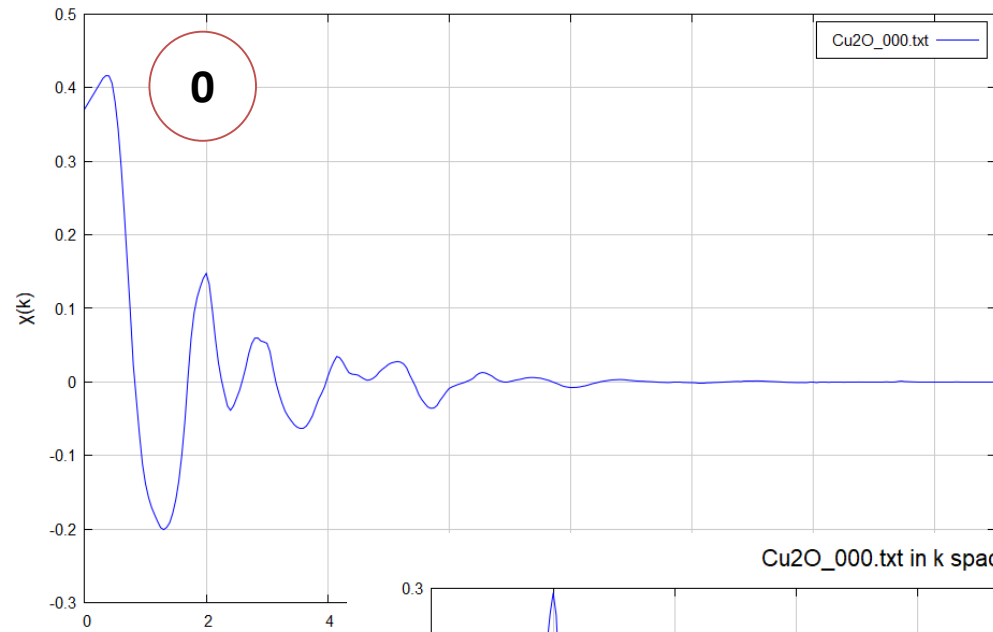
It will be refined during the analysis.

From energy to k-space

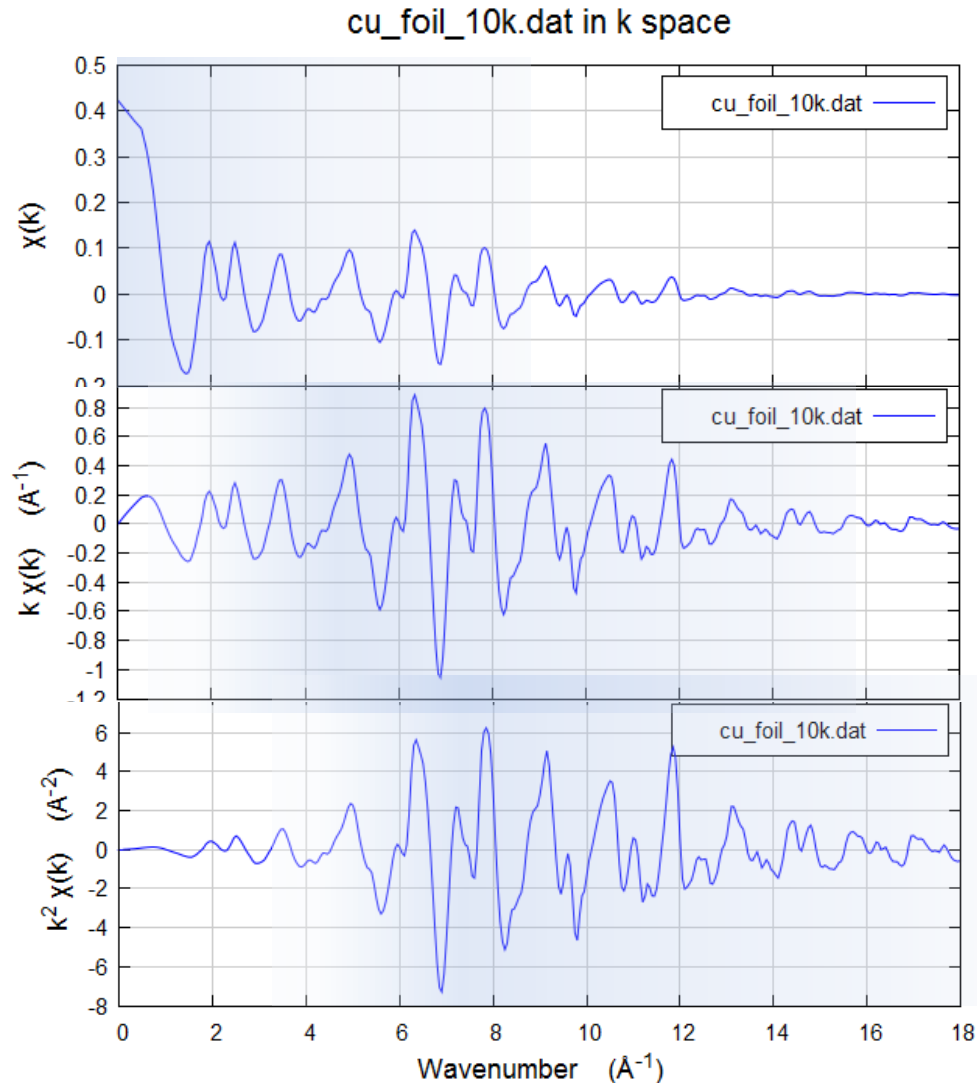


$$k = \sqrt{\frac{2m(E - E_0)}{\hbar^2}} = \sqrt{0.2625(E - E_0)}$$

Amplifying high k signal: k weights



Inspect $k^n \chi(k)$

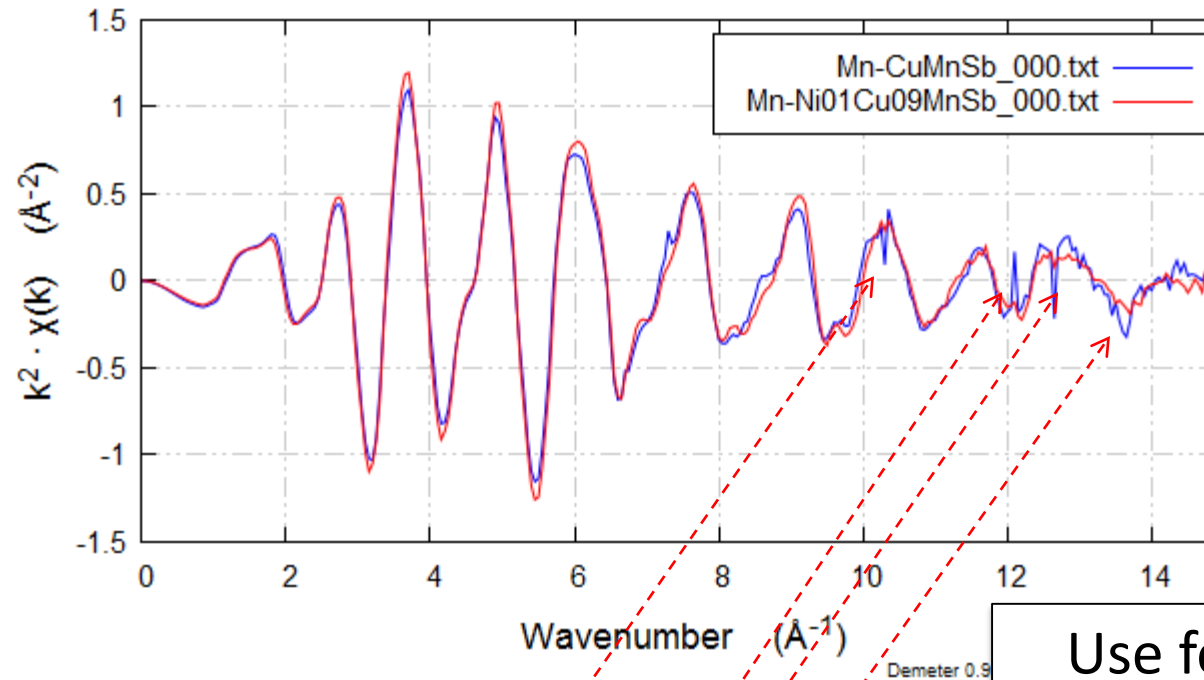


$k^n \chi(k)$ weighting highlights different features in the spectrum: high (low) n enhance high (low) k -regions

Note: low k -region ($k < 3$) is generally affected by larger inaccuracies and is difficult to analyse due to intense **multiple scattering** contributions and other not linear effects

Inspect $k^n \chi(k)$

Quality of the data



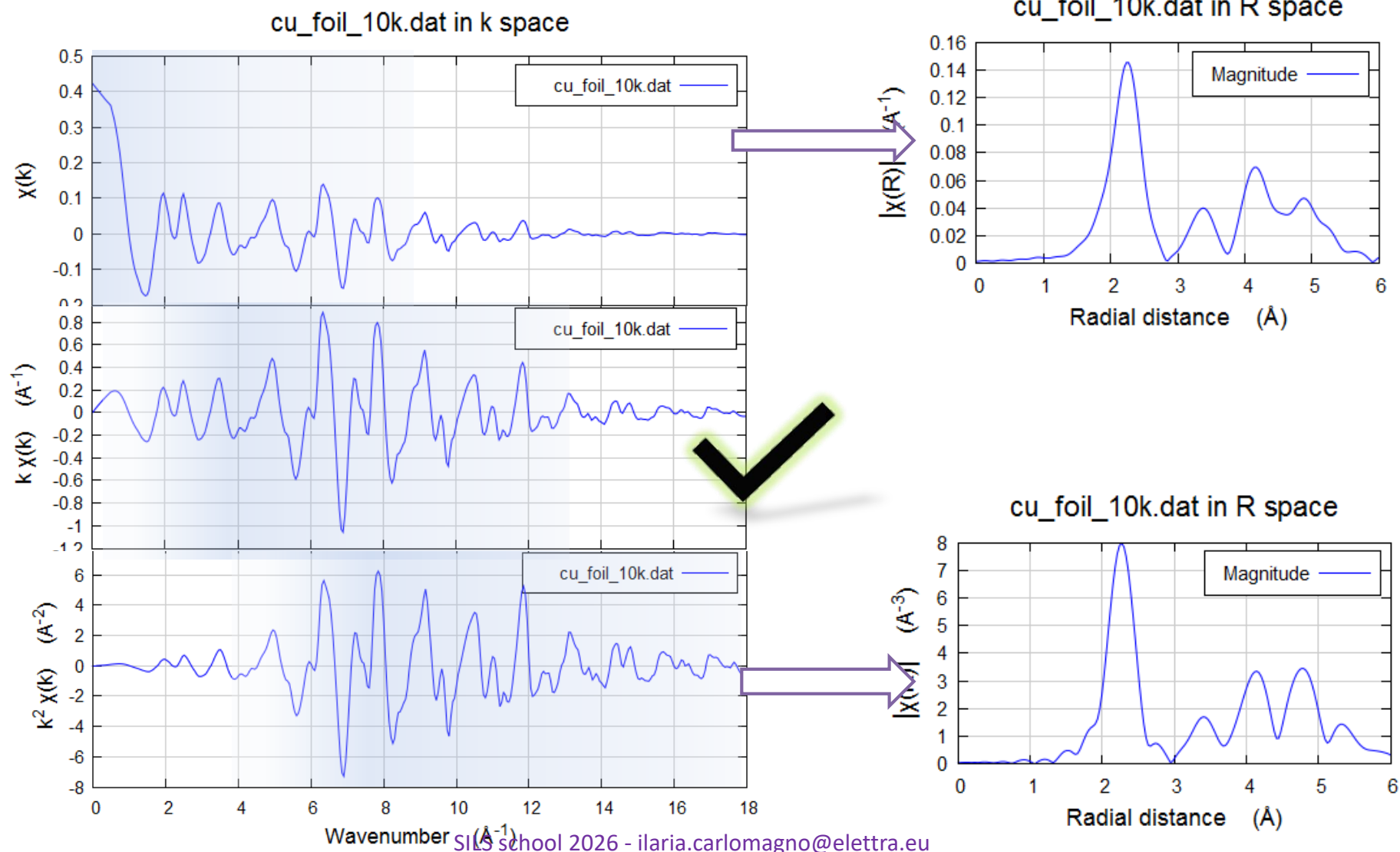
Use few/short data
free from artifacts

Check for distortions in the spectra,
correct or **cut** the data and retain only the
"good" regions"

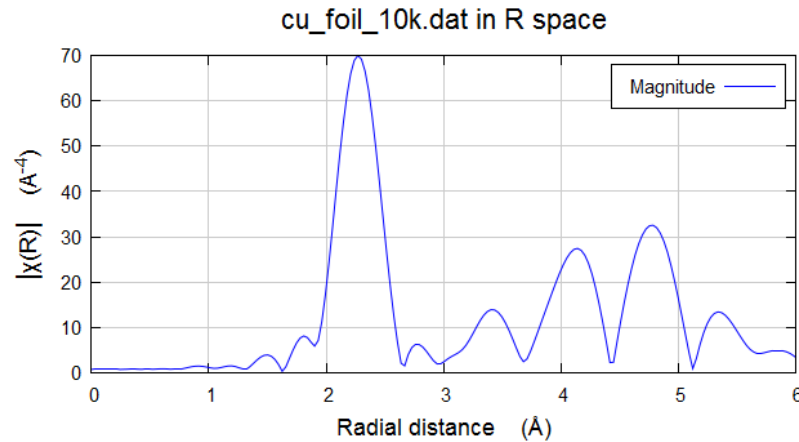
Qualitative local structure

Fourier transform

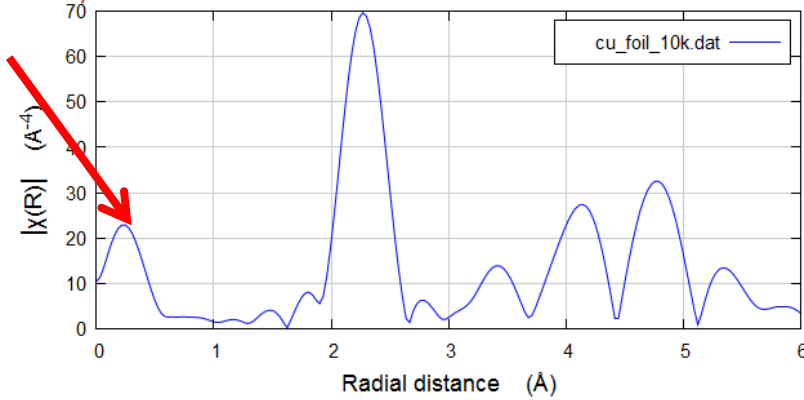
Cu



Check FT



Unphysical peak
(below 1 Å!)



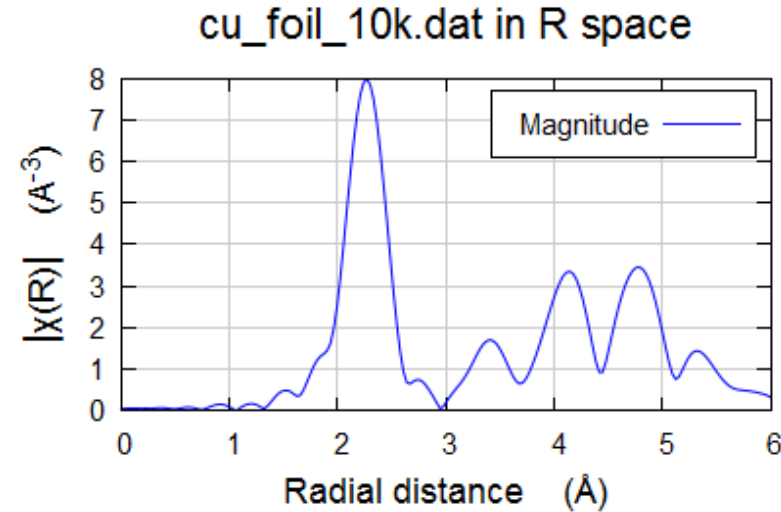
FT features:
Artifacts, distortions, noise, may suggest bad extraction, noise on the data, etc...

i.e.: intense peaks in the low R region ($\approx$ less than 1Å) likely originates from wrong extraction

Qualitative local structure

Fourier transform

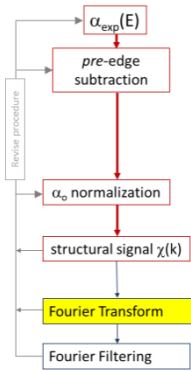
|FT| shows more intuitively the main structural features in the real space:
the FT modulus represents a **pseudo-radial distribution function** modified by the effect of amplitude, phase and mean free path parameters.



Peak positions (phase shift corrected)	= > neighbour shells
Peak amplitude and shape	= > number and type of neighbours

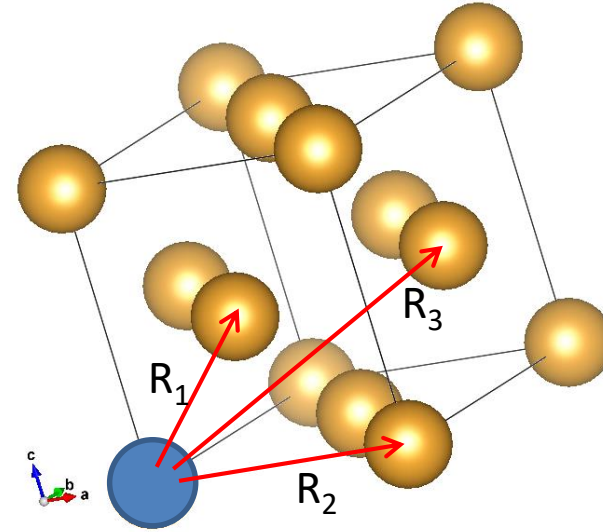
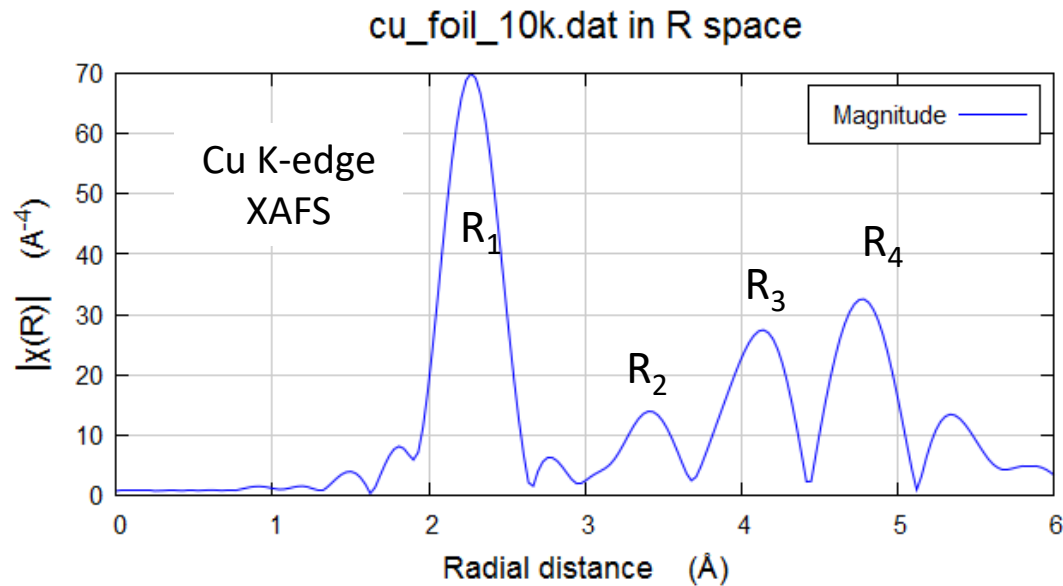
$$\chi(k) = \frac{1}{k} \sum \mathbf{A}_j \sin(2kr_j + \delta_j)$$

$$\mathbf{A}_j = S_o^2 \frac{N_j}{R_j^2} |f_j| e^{-2k^2 \sigma_j^2} e_j^{-\frac{2r_j}{\lambda}}$$



FT and expected atomic structure

Look at the FT features and compare the structure you expect



$$R_1 = \frac{a}{\sqrt{2}}$$
$$R_2 = a$$
$$R_3 = \frac{a\sqrt{6}}{2}$$
$$R_4 = \dots$$

$$N_1 = 12$$
$$N_2 = 6$$
$$N_3 = 24$$
$$N_4 = \dots$$

Let's try!

1. Cu K edge XAFS

Basic features

1. Import data
2. E, K, R, Q figures and plot parameters

Modify extraction parameters

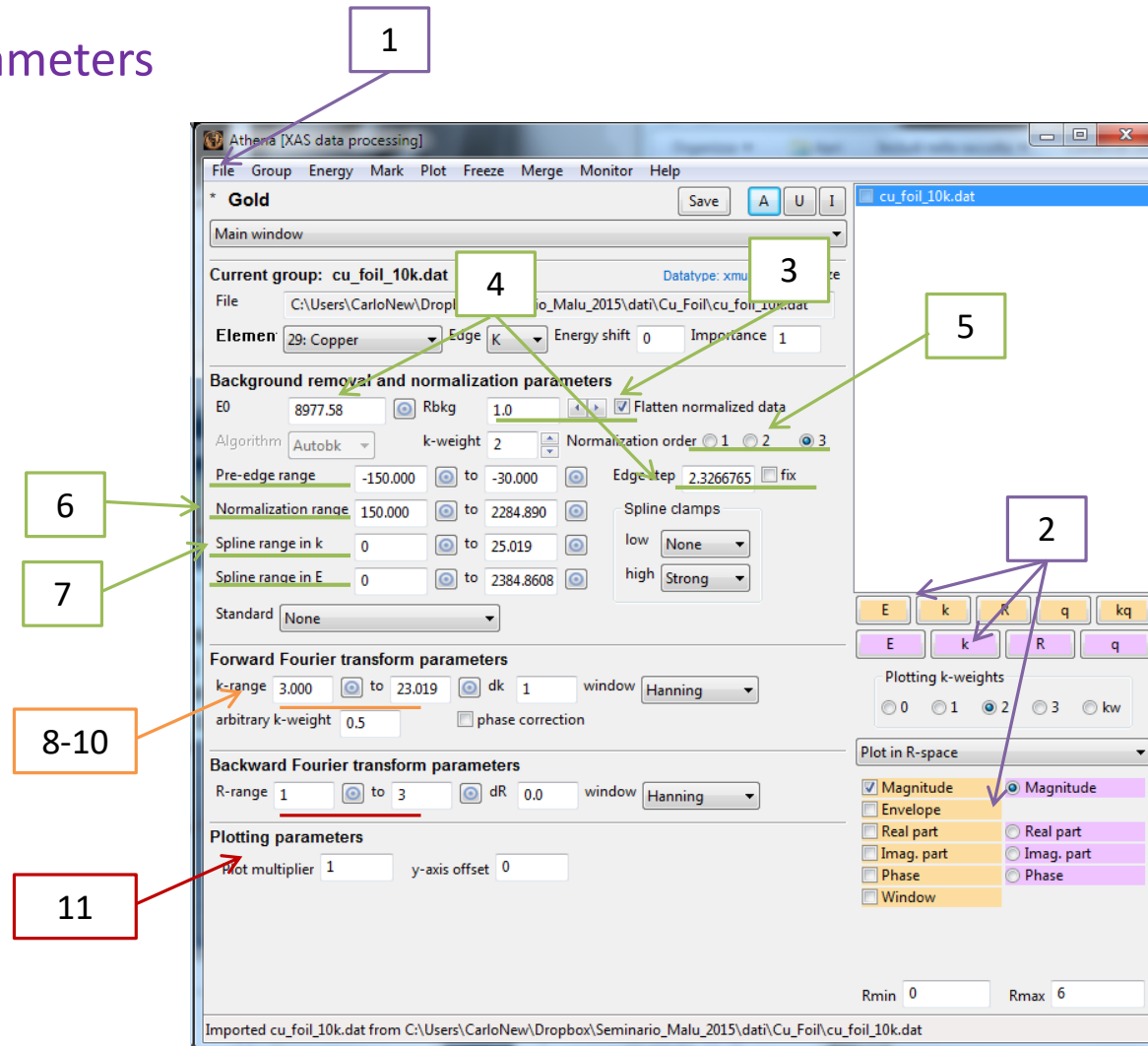
3. R_{bkg}
4. Edge Energy and step
5. Normalization order
6. Normalization range
7. Spline range

k-Weighting

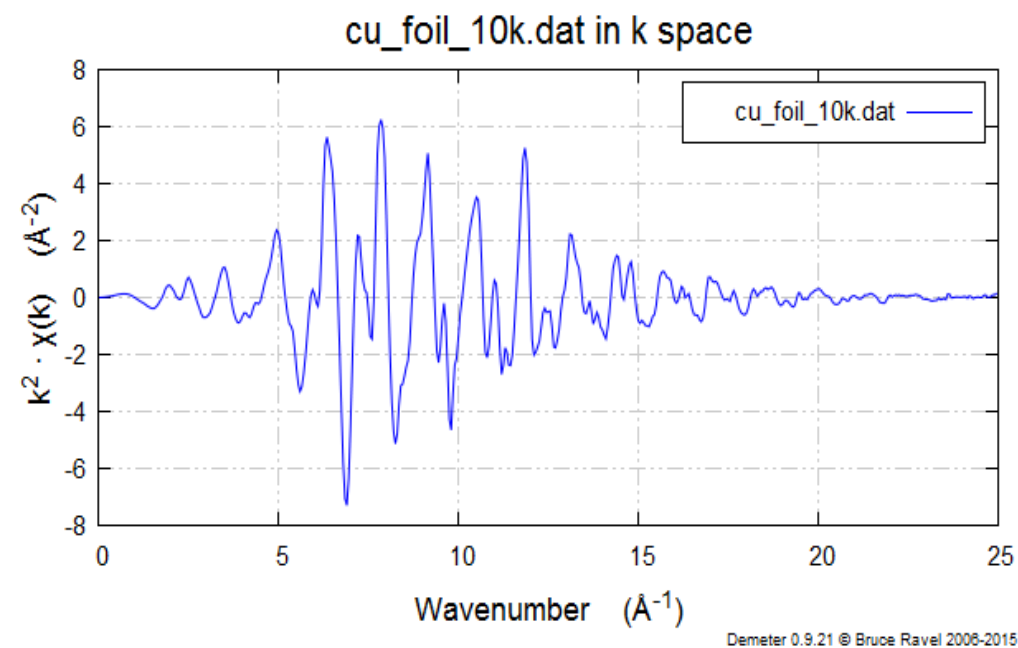
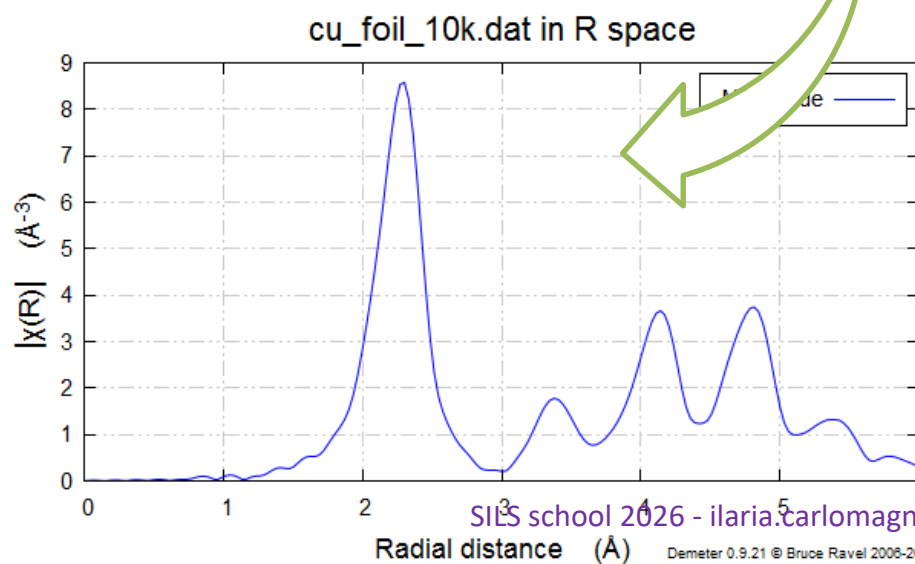
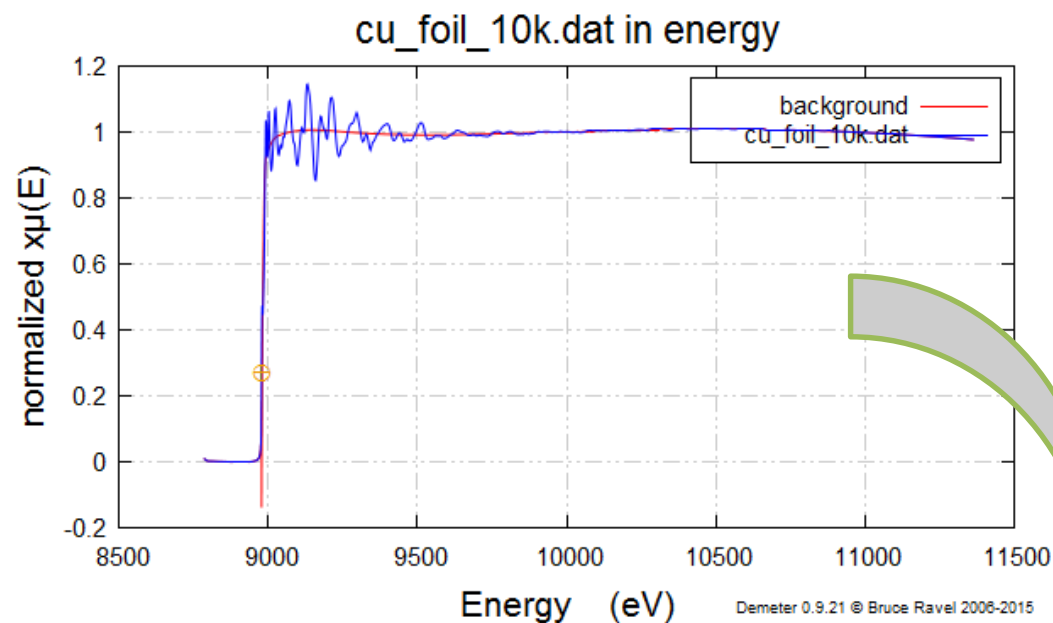
8. FT range
9. FT window
10. FT weight

Back Fourier

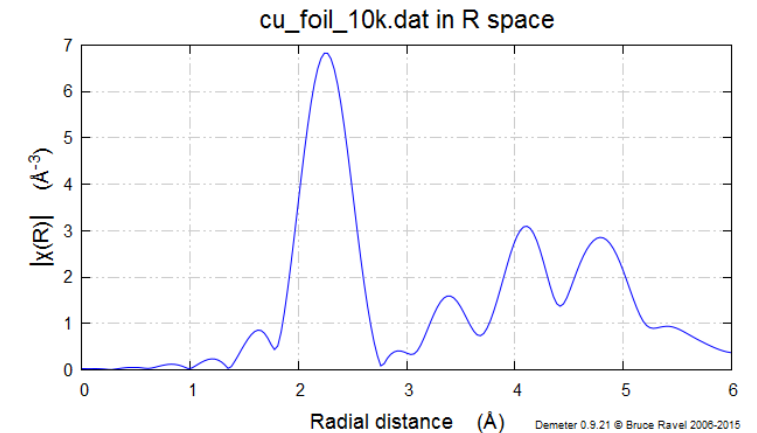
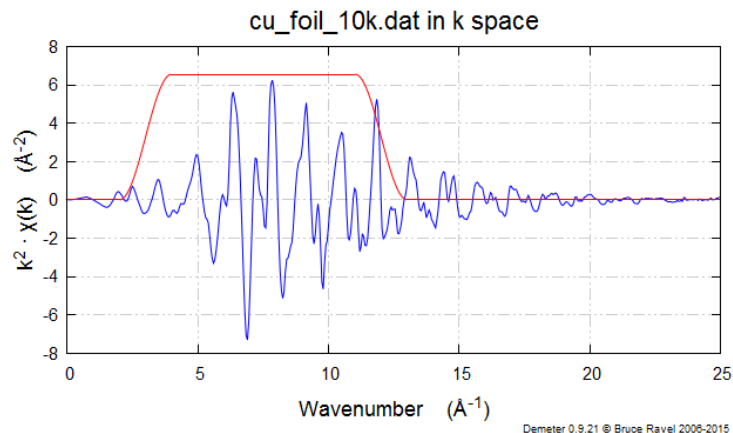
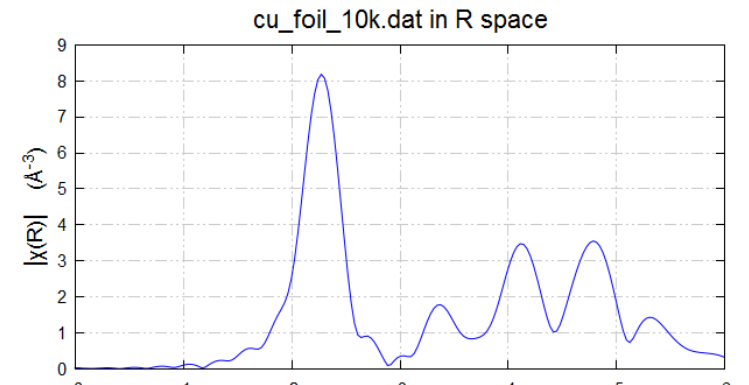
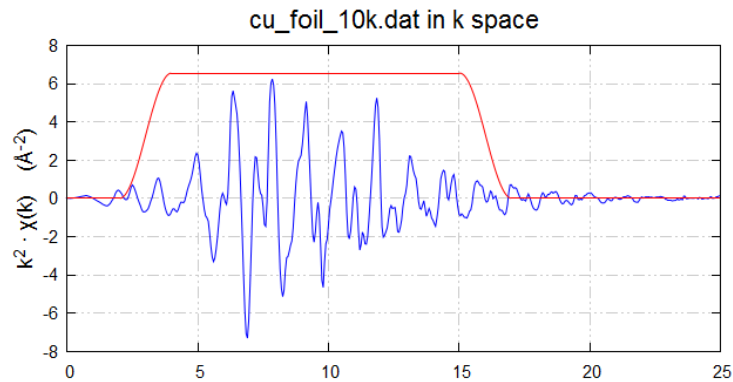
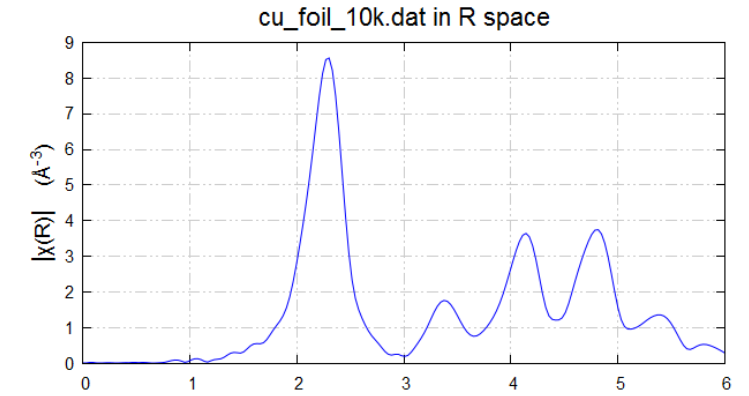
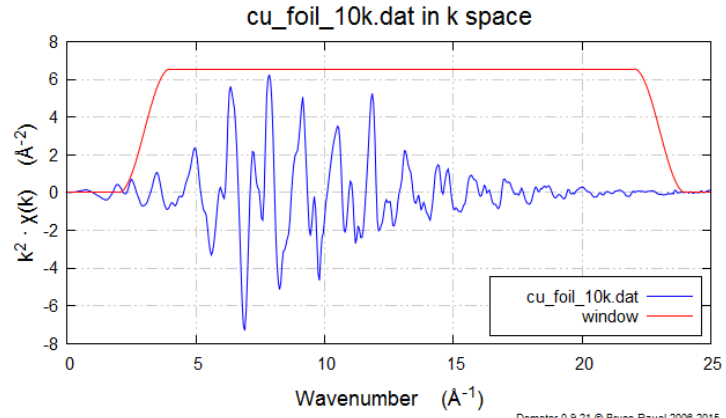
11. Range and window



Cu K edge XAFS



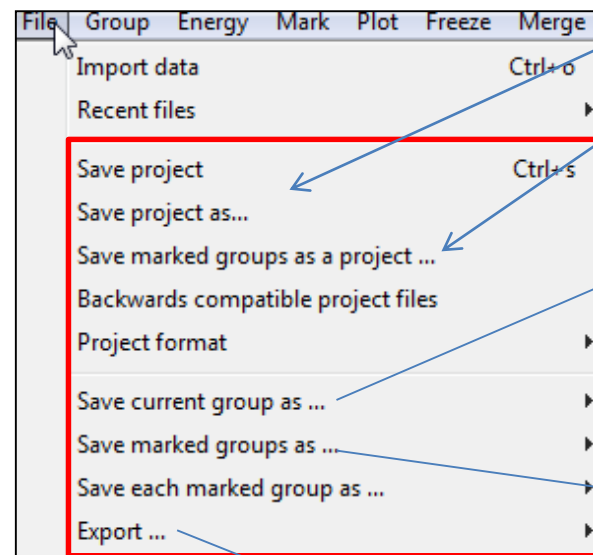
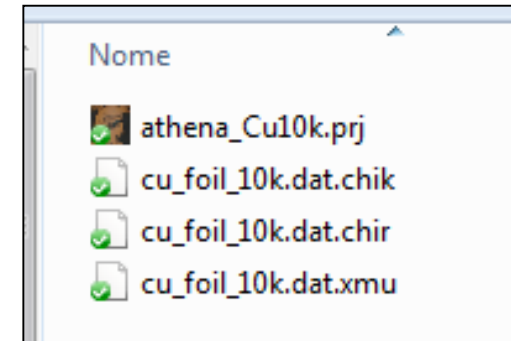
FT window



High quality data:
main FT features do not
change with windows

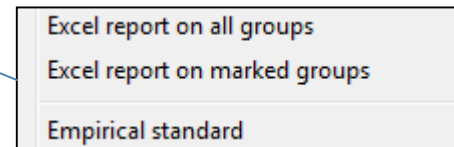
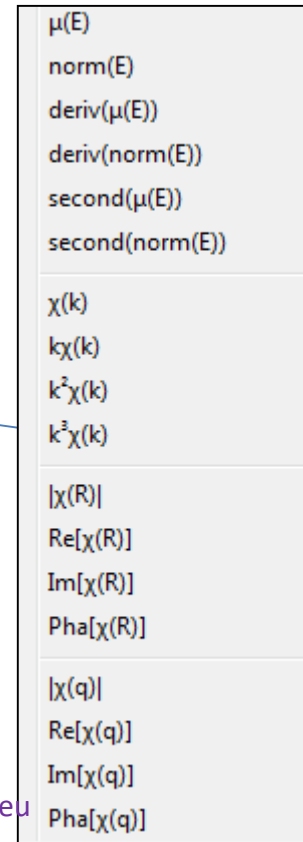
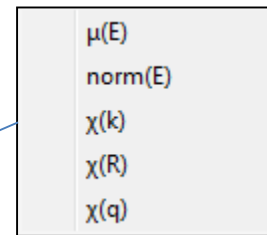
Note:

Always save data and project for future use
you have many options!



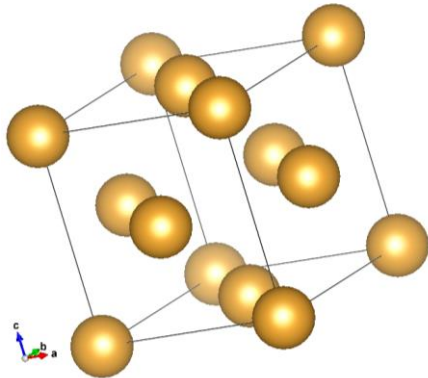
Save the entire project for reuse.

Save selected groups for separate use



Cu (fcc) and Fe (bcc) XAFS

Compare Cu and Fe EXAFS data: shows the effect of different crystallographic structure



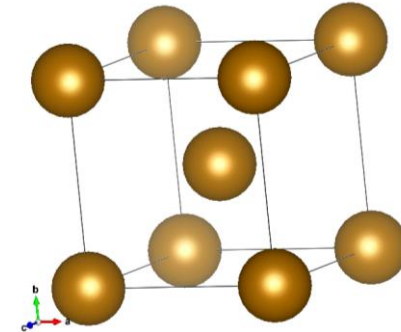
fcc
(Fm-3m)

$$\begin{aligned} R_1 &= a/\sqrt{2} & N_1 &= 12 \\ R_2 &= a & N_2 &= 6 \\ R_3 &= a\sqrt{6}/2 & N_3 &= 24 \end{aligned}$$

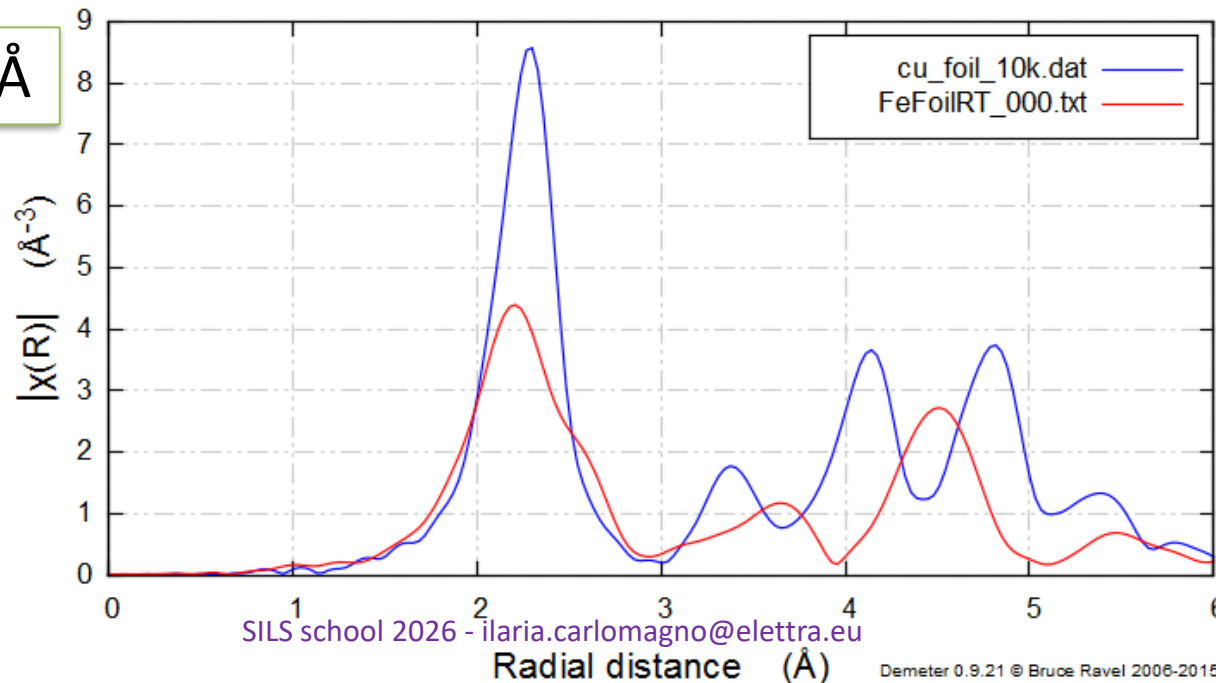
$$a_{\text{Cu}} = 3.61 \text{ \AA}$$

Bcc
(Im3m)

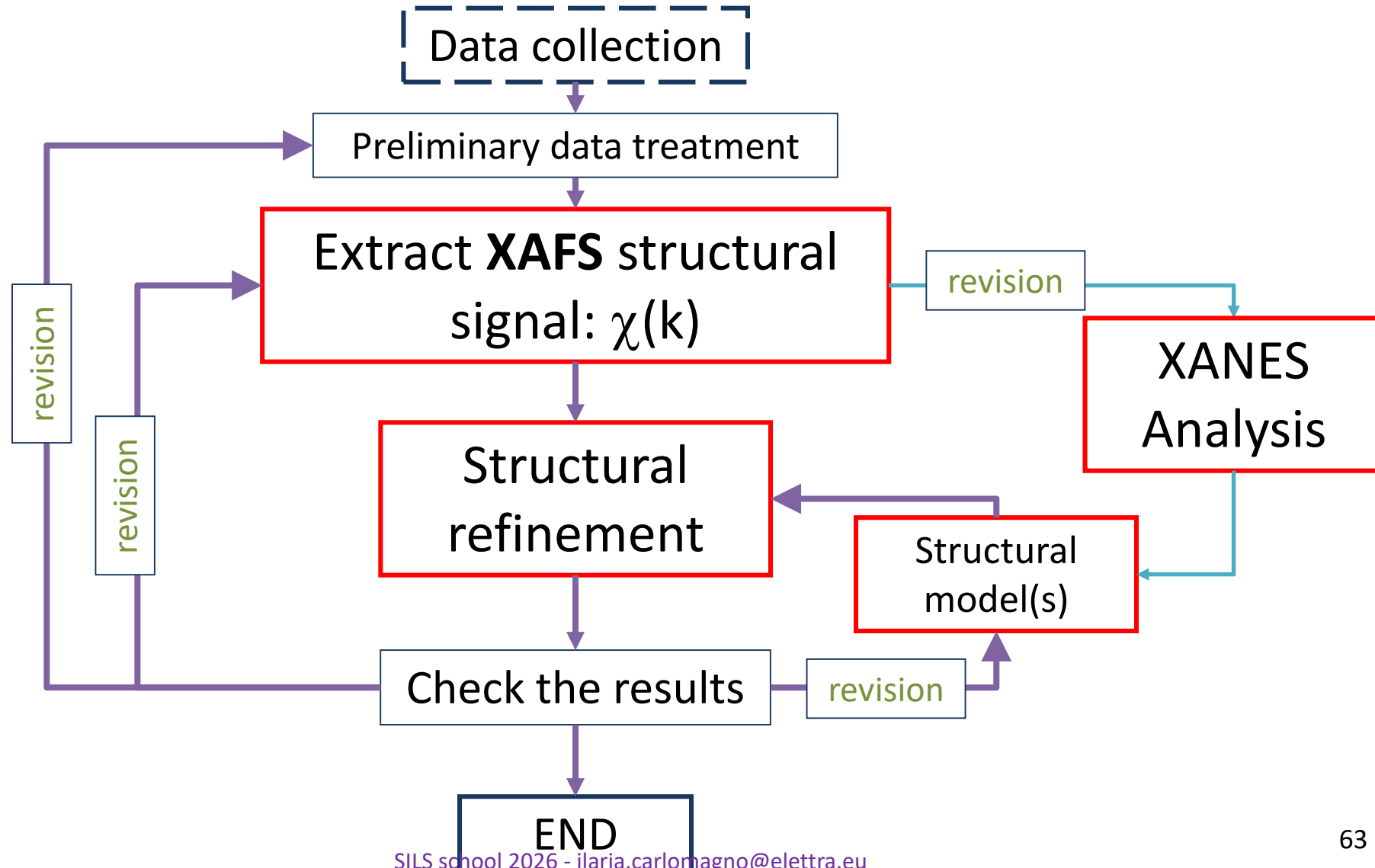
$$\begin{aligned} R_1 &= a\sqrt{3}/2 & N_1 &= 8 \\ R_2 &= a & N_2 &= 6 \\ R_3 &= a\sqrt{2} & N_3 &= 12 \end{aligned}$$



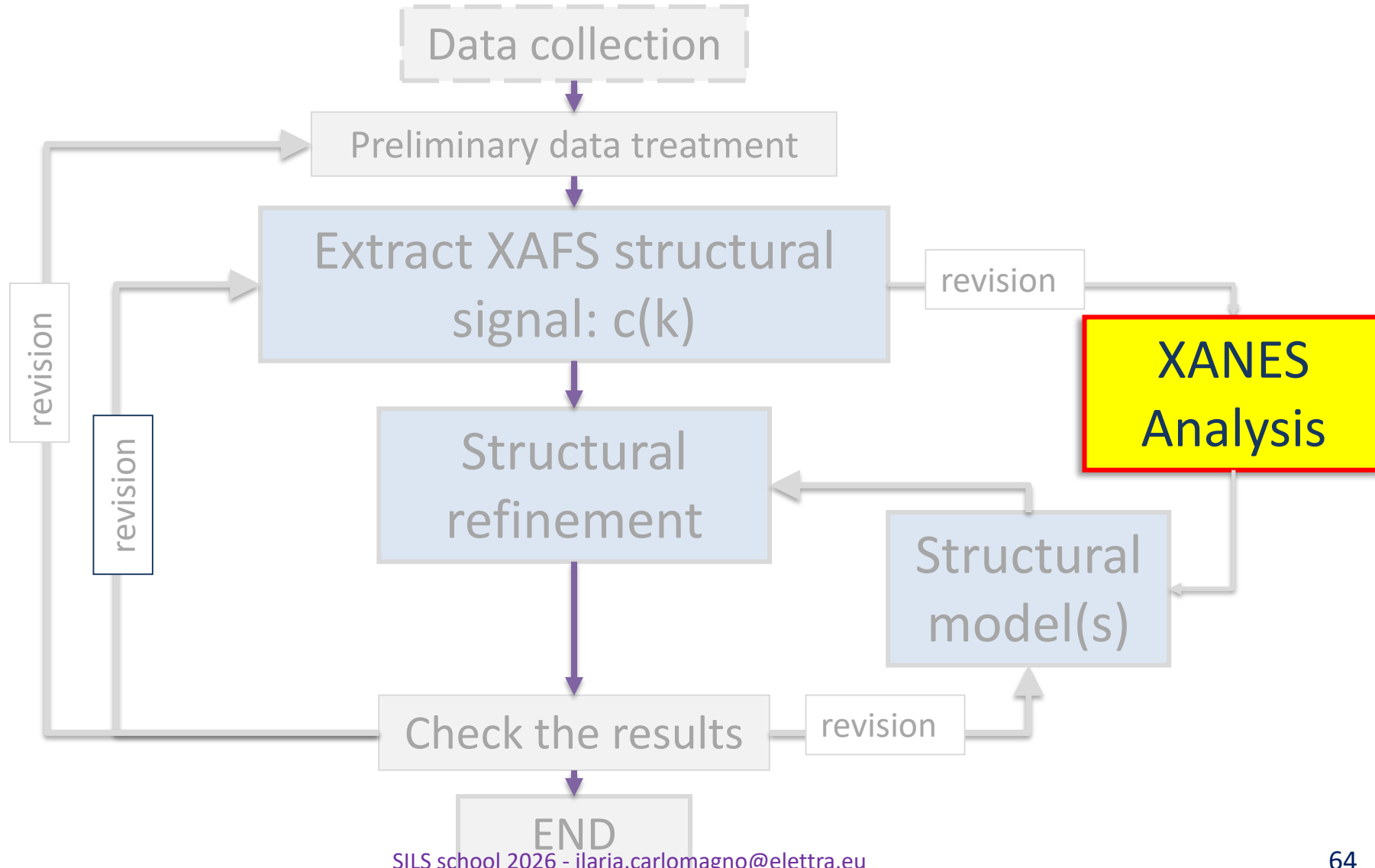
$$a_{\text{Fe}} = 2.86 \text{ \AA}$$



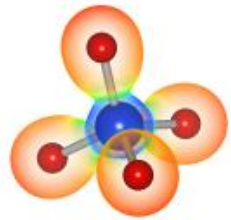
XAFS data ANALYSIS: from experimental data to structural details



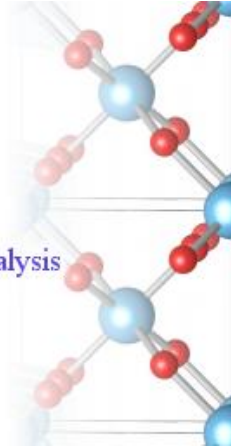
XAFS data ANALYSIS: from the experimental data to structural details



Structure Visualizers



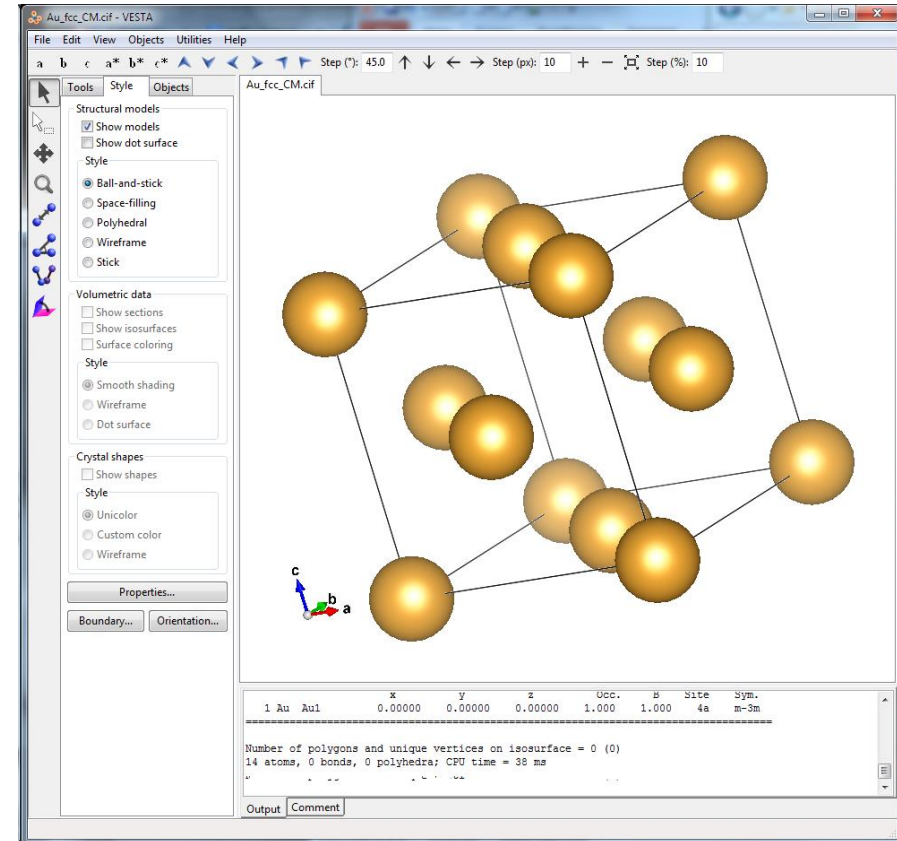
VESTA
Visualization for Electronic and STructural Analysis



<http://jp-minerals.org/soft/en/>

Google search:

Vesta Download



Crystallographic structures DBs

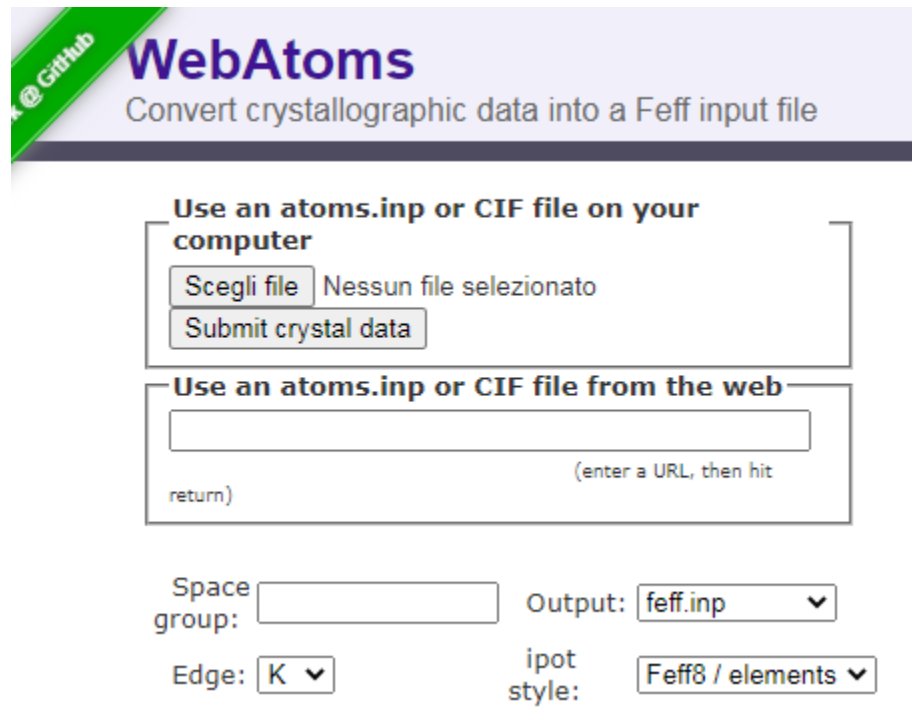
- Crystallography Open Database (COD)
crystallography.net
- WWW-MINCRYST
database.iem.ac.ru/
- ICSD (\$): inorganic chemistry database (guest)
FIZ Karlsruhe <https://icsd.fiz-karlsruhe.de/>
- Google Scholar
- ...or ask Gemini, Claude, ChatGPT!!

Often Synchrotron Facilities have
access to commercial databases.
Use them during beam-time!

Coordination geometry

Neighbour geometry around the absorber

ATOMS package
included in the Demeter (Artemis) package



The screenshot shows the WebAtoms web interface. At the top left is a green GitHub logo. The title 'WebAtoms' is in large blue font, with the subtitle 'Convert crystallographic data into a Feff input file' below it. There are two main sections for file input: 'Use an atoms.inp or CIF file on your computer' and 'Use an atoms.inp or CIF file from the web'. The first section has buttons for 'Scegli file' (which shows 'Nessun file selezionato') and 'Submit crystal data'. The second section has a text input field for a URL and a 'return)' button. Below these are two rows of dropdown menus: 'Space group:' with an empty dropdown, 'Edge:' with 'K' selected, 'Output:' with 'feff.inp' selected, and 'ipot style:' with 'Feff8 / elements' selected.

<https://millenia.cars.aps.anl.gov/webatoms/>

Note: VESTA can read or save in
ATOMS format

Software

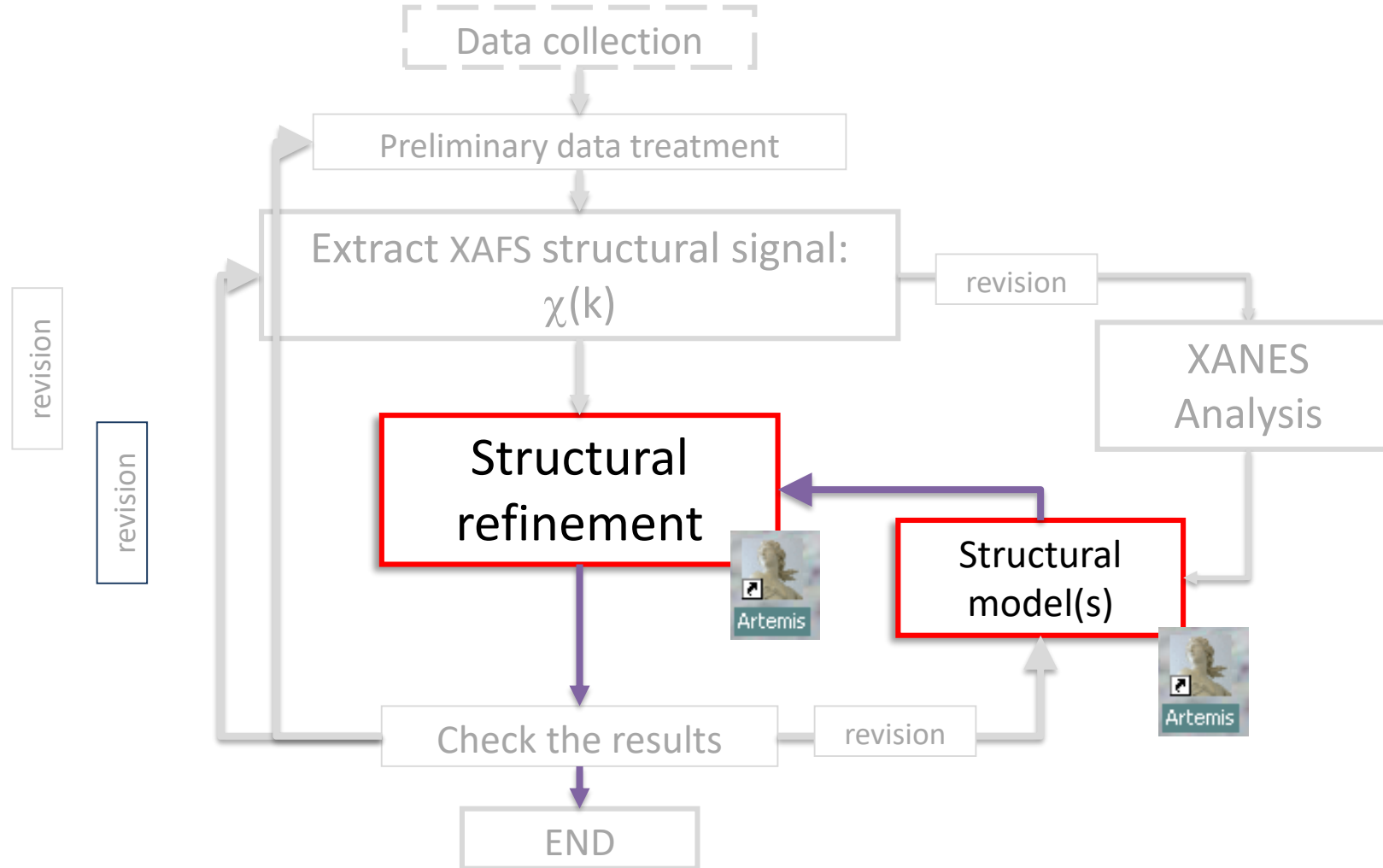
Mandatory

- Data analysis software (EXCEL, ORIGIN, Gnuplot, FitYk,)
- XAFS data analysis software

Useful:

- Database of crystallographic structures (or LLM)
- Atomic Structure visualization programs
- Coordination geometry calculation programs

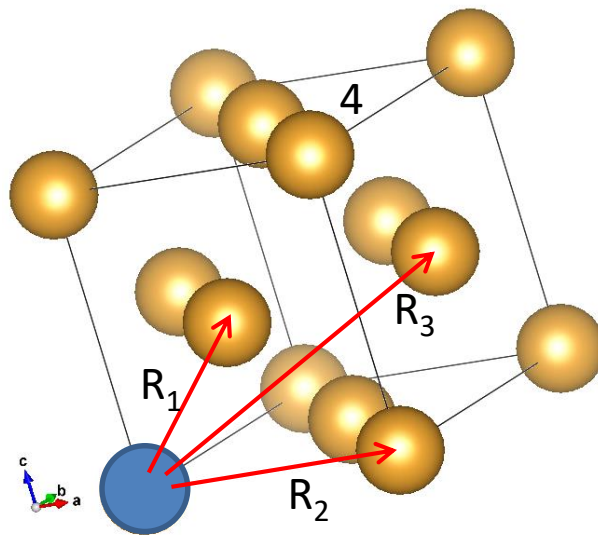
XAFS data ANALYSIS: from experimental data to structural details



Structural model & data refinement

We will start from Cu EXAFS analysis

1. search Cu metal structure (cif)
2. visualize with Vesta
3. generate a local cluster with Atoms



icsd_43493_Copper.cif

Cu metal
SPG: fcc, f m 3 m (# 220)
a = 3.61 Å
Cu 0.0 0.0 0.0

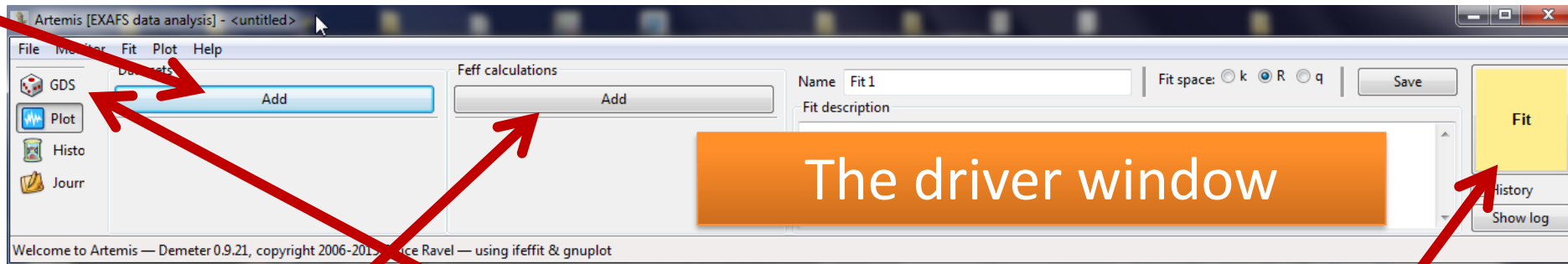
Sh	R	N	R _{Cu} (Å)
I:	$a/\sqrt{2}$	12	2.553
II:	a	6	3.610
III:	$a\sqrt{3}/2$	24	4.421
IV:	$a\sqrt{2}$	12	5.105
V:	$a\sqrt{5}/2$	24	5.708

Start Artemis (easier on wide screens!)

1

add EXAFS

$\chi(k)$ or
athena
project



2

add a
Structure
.cif or Feff
file

3

select **refinement
param.**, plot, etc...

4

Start fit

1: add $\chi(k)$ or Athena project

The screenshot displays the Artemis [EXAFS data analysis] - <untitled> window. The 'Data sets' panel on the left has an 'Add' button highlighted with a red arrow. The 'Feff calculations' panel also has an 'Add' button. The 'Fit' panel on the right shows 'Fit 1' with a 'Fit' button. A blue text box with the text 'Import the XAFS data from the Athena Project' is overlaid on the interface. Below the main window, a plot titled 'cu_foil_10k.dat in k space' shows the data $k^2 \cdot \chi(k)$ (in $\text{\AA}^{-2}$) versus Wavenumber (in $\text{\AA}^{-1}$). The plot shows a blue line for the data and a red line for the fit window. The x-axis ranges from 0 to 25, and the y-axis ranges from 0 to 1000. The plot is titled 'cu_foil_10k.dat in k space'.

Artemis [EXAFS data analysis] - <untitled>

File Monitor Fit Plot Help

Data sets: Add

Feff calculations: Add

Name: Fit 1

Fit space: ☐ k ☒ R ☐ q

Save

Fit

History

Artemis [Data] cu_foil_10k.d

Welcome to Artemis — Demeter 0.9.21, copyright 2006-2015 Bruce Ravel — using cffit & gnuplot

Data Path Marks Add

cu_foil_10k.d

Data source: o_Malu_2015\dat\ok_Cu_Foil\EXAFS_extraction\athena_Cu10k.prj, 1

Plot this data set as:

k123 R123 Rmr Rk kq

Title lines:

Fourier transform parameters:

kmin: 3.000 kmax: 23.019 dk: 1

rmin: 1 rmax: 3 dr: 0.0

Fitting k weights:

☒ 1 ☒ 2 ☒ 3 ☐ other: 0.5

Other parameters:

☒ Include in fit ☒ Plot after fit ☐ Fit background

$\epsilon(k)$: 0 ☐ Plot with phase correction

Drag paths from list and drop add paths to

[Import crystal](#)

[Start a quick](#)

[Import a structure](#)

[Import an error](#)

cu_foil_10k.dat in k space

$k^2 \cdot \chi(k)$ ($\text{\AA}^{-2}$)

Wavenumber ($\text{\AA}^{-1}$)

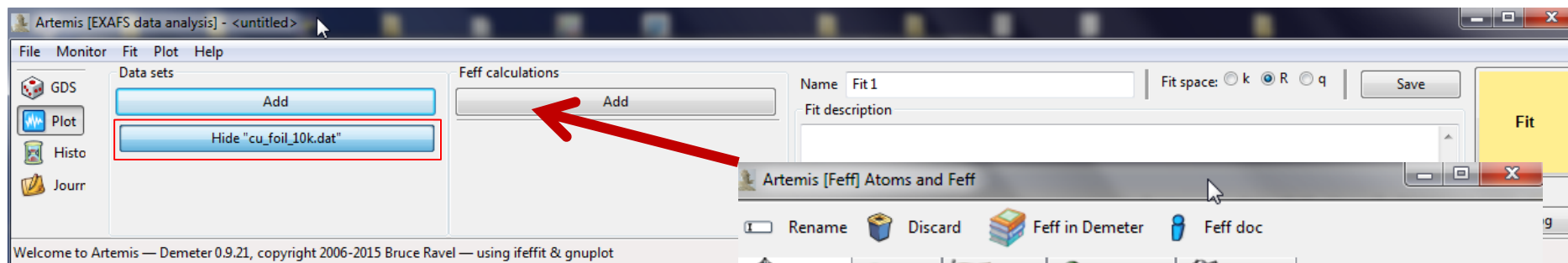
cu_foil_10k.dat

window

Demeter 0.9.21 © Bruce Ravel

SLI School 2025, data of "cu_foil_10k.dat" to Demeter

2: add model structure (cif or simple pair)



- I. Run Atoms
- II. check paths
- III. Run Feff
- IV. look at the path list

A)
generate a
local cluster

B)
calculate
amplitude and
phases for paths
in the cluster

C)
Scattering
Path list

Artemis [Feff] Atoms and Feff

Open file Save file Clear all Template Run Feff

Name: icsd_43493_Copper Margin: 0.03 Beta:

Feff input file

```

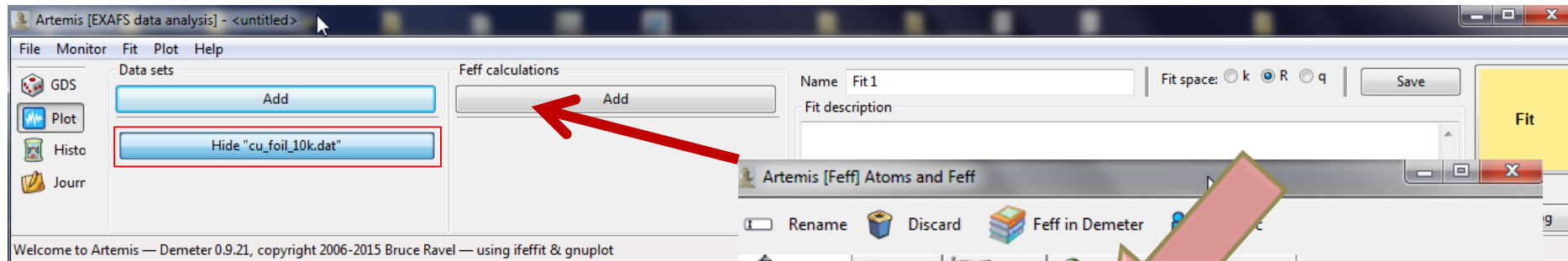
1 29 Cu

ATOMS
* x y z ipot tag distance
0.00000 0.00000 0.00000 0 Cu1 0.00000
1.80753 1.80753 0.00000 1 Cu1.1 2.55623
-1.80753 1.80753 0.00000 1 Cu1.1 2.55623
1.80753 -1.80753 0.00000 1 Cu1.1 2.55623
-1.80753 -1.80753 0.00000 1 Cu1.1 2.55623
1.80753 0.00000 1.80753 1 Cu1.1 2.55623
-1.80753 0.00000 1.80753 1 Cu1.1 2.55623
0.00000 1.80753 1.80753 1 Cu1.1 2.55623
0.00000 -1.80753 1.80753 1 Cu1.1 2.55623
1.80753 0.00000 -1.80753 1 Cu1.1 2.55623
-1.80753 0.00000 -1.80753 1 Cu1.1 2.55623
0.00000 1.80753 -1.80753 1 Cu1.1 2.55623
0.00000 -1.80753 -1.80753 1 Cu1.1 2.55623
3.61505 0.00000 0.00000 1 Cu1.2 3.61505
-3.61505 0.00000 0.00000 1 Cu1.2 3.61505
0.00000 3.61505 0.00000 1 Cu1.2 3.61505
0.00000 -3.61505 0.00000 1 Cu1.2 3.61505

```

	Core	EL	x	y	z	Tag
1	☒	Cu	0	0	0	Cu1
2	☐					
3	☐					
4	☐					
5	☐					
6	☐					

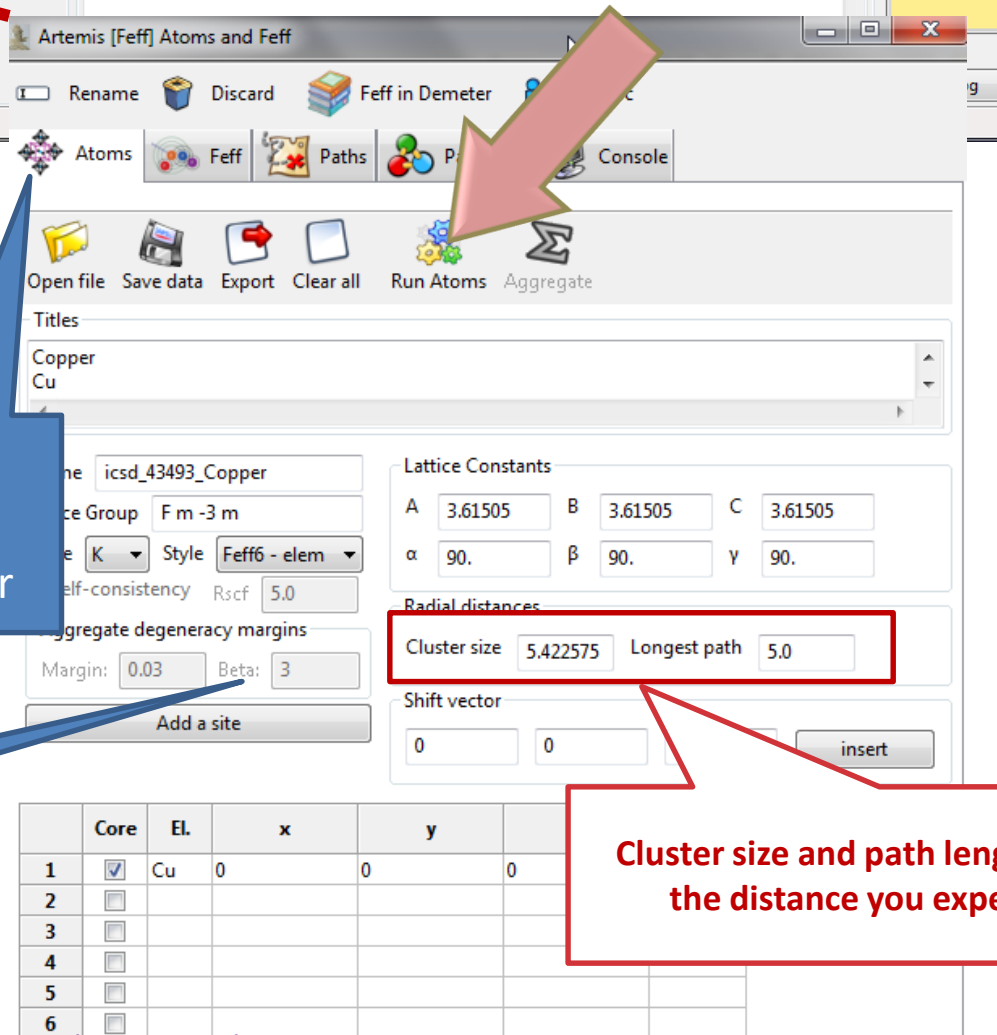
2: add model structure (cif or simple pair)



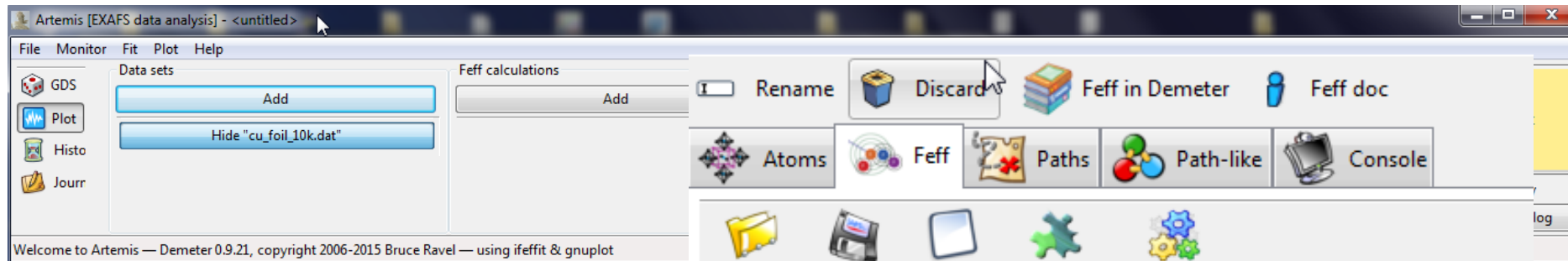
- I. **Run Atoms**
- II. check paths
- III. Run Feff
- IV. look at the path list

A)
generate a
local cluster

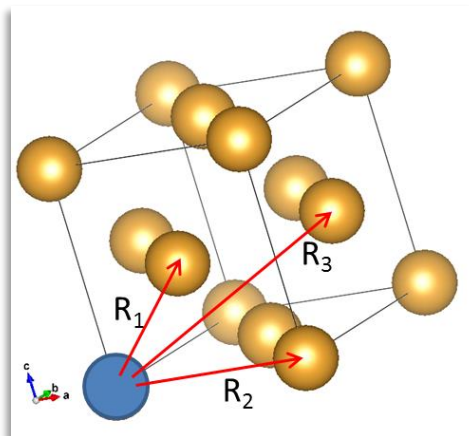
input the unit cell
parameters and cluster size



2: add model structure (cif or simple pair)



- I. Run Atoms
- II. **check scattering paths**
- III. Run Feff
- IV. look at the path list

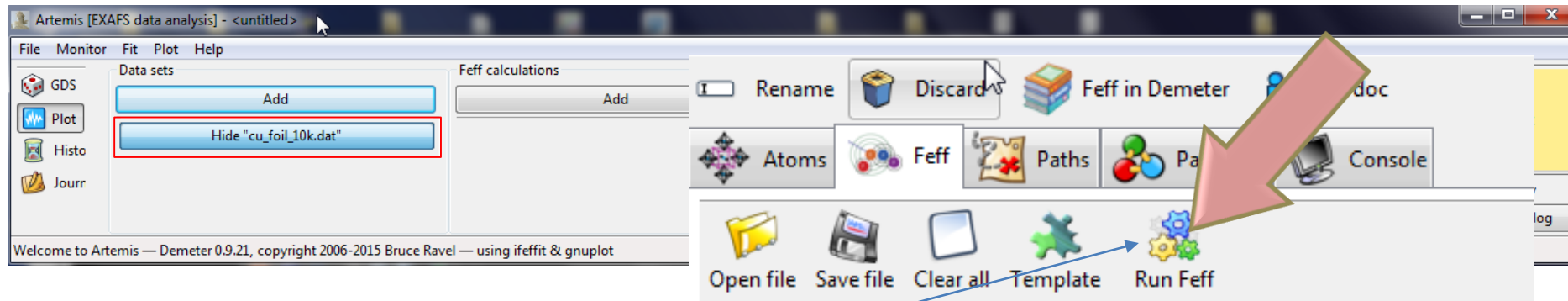


Name: Margin: Beta:

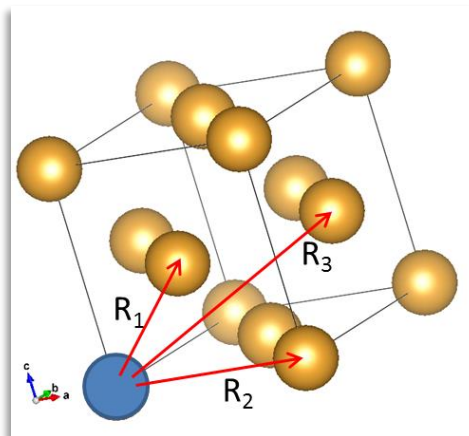
Feff input file

1	29	Cu
* this list contains 55 atoms		
ATOMS		
* x	y	z ipot tag distance
0.00000	0.00000	0.00000 0 Cu1 0.00000
1.80753	1.80753	0.00000 1 Cu1.1 2.55623
-1.80753	1.80753	0.00000 1 Cu1.1 2.55623
1.80753	-1.80753	0.00000 1 Cu1.1 2.55623
-1.80753	-1.80753	0.00000 1 Cu1.1 2.55623
1.80753	0.00000	1.80753 1 Cu1.1 2.55623
-1.80753	0.00000	1.80753 1 Cu1.1 2.55623
0.00000	1.80753	1.80753 1 Cu1.1 R_1 2.55623
0.00000	-1.80753	1.80753 1 Cu1.1 2.55623
1.80753	0.00000	-1.80753 1 Cu1.1 2.55623
-1.80753	0.00000	-1.80753 1 Cu1.1 2.55623
0.00000	1.80753	-1.80753 1 Cu1.1 2.55623
0.00000	-1.80753	-1.80753 1 Cu1.1 2.55623
3.61505	0.00000	0.00000 1 Cu1.2 3.61505
-3.61505	0.00000	0.00000 1 Cu1.2 3.61505
0.00000	3.61505	0.00000 1 Cu1.2 R_2 3.61505

2: add model structure (cif or simple pair)



- I. Run Atoms
- II. check scattering paths
- III. Run Feff**
- IV. look at the path list



Name: Margin: Beta:

Feff input file

	1	29	Cu			
* this list contains 55 atoms						
ATOMS	x	y	z	ipot	tag	distance
*	0.00000	0.00000	0.00000	0	Cu1	0.00000
	1.80753	1.80753	0.00000	1	Cu1.1	2.55623
	-1.80753	1.80753	0.00000	1	Cu1.1	2.55623
	1.80753	-1.80753	0.00000	1	Cu1.1	2.55623
	-1.80753	-1.80753	0.00000	1	Cu1.1	2.55623
	1.80753	0.00000	1.80753	1	Cu1.1	2.55623
	-1.80753	0.00000	1.80753	1	Cu1.1	2.55623
	0.00000	1.80753	1.80753	1	Cu1.1	2.55623
	0.00000	-1.80753	1.80753	1	Cu1.1	2.55623
	1.80753	0.00000	-1.80753	1	Cu1.1	2.55623
	-1.80753	0.00000	-1.80753	1	Cu1.1	2.55623
	0.00000	1.80753	-1.80753	1	Cu1.1	2.55623
	0.00000	-1.80753	-1.80753	1	Cu1.1	2.55623
	3.61505	0.00000	0.00000	1	Cu1.2	3.61505
	-3.61505	0.00000	0.00000	1	Cu1.2	3.61505
	0.00000	3.61505	0.00000	1	Cu1.2	3.61505

2: add model structure (cif or simple pair)

Artemis [EXAFS data analysis] - <untitled>

File Monitor Fit Plot Help

Data sets

Add

Hide "cu_foil_10k.dat"

Feff calculations

Add

Name: Fit1

Fit space: ☐ k ☒ R

Fit description

Rename Discard Feff in Demeter Feff

Atoms Feff Paths Path-like

Save Plot paths $\chi(k)$ $|R|$ Re Im Rank

Name of this Feff calculation: icسد_43493_Copper

Description

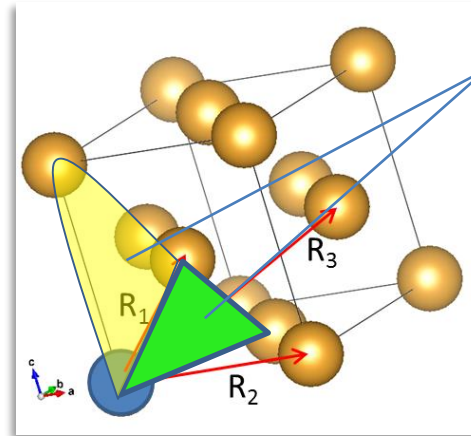
```
# TITLE Copper
# TITLE Cu
# The central atom is denoted by this token: @
# Cluster size = 6.00 Å, containing 86 atoms
# 31 paths were found within 6.000 Å
# Forward scattering cutoff 20.00
```

Scattering Paths

	Degen	Reff	Scattering path	Rank	1	Type
1	12.00	2.556	@ Cu1.1 @	10...	:	single scatte
2	6.00	3.615	@ Cu1.2 @	22.97	:	single scatte
3	8.00	3.834	@ Cu1.1 Cu1.1 @	10.56	:	acute triangl
4	48.00	4.364	@ Cu1.1 Cu1.2 @	8.54	:	other double
5	24.00	4.364	@ Cu1.1 Cu1.1 @	3.39	:	other double
6	24.00	4.428	@ Cu1.3 @	55.37	:	single scatte
7	96.00	4.770	@ Cu1.1 Cu1.3 @	21.64	:	other triang
8	48.00	4.770	@ Cu1.1 Cu1.1 @	10.61	:	other triang

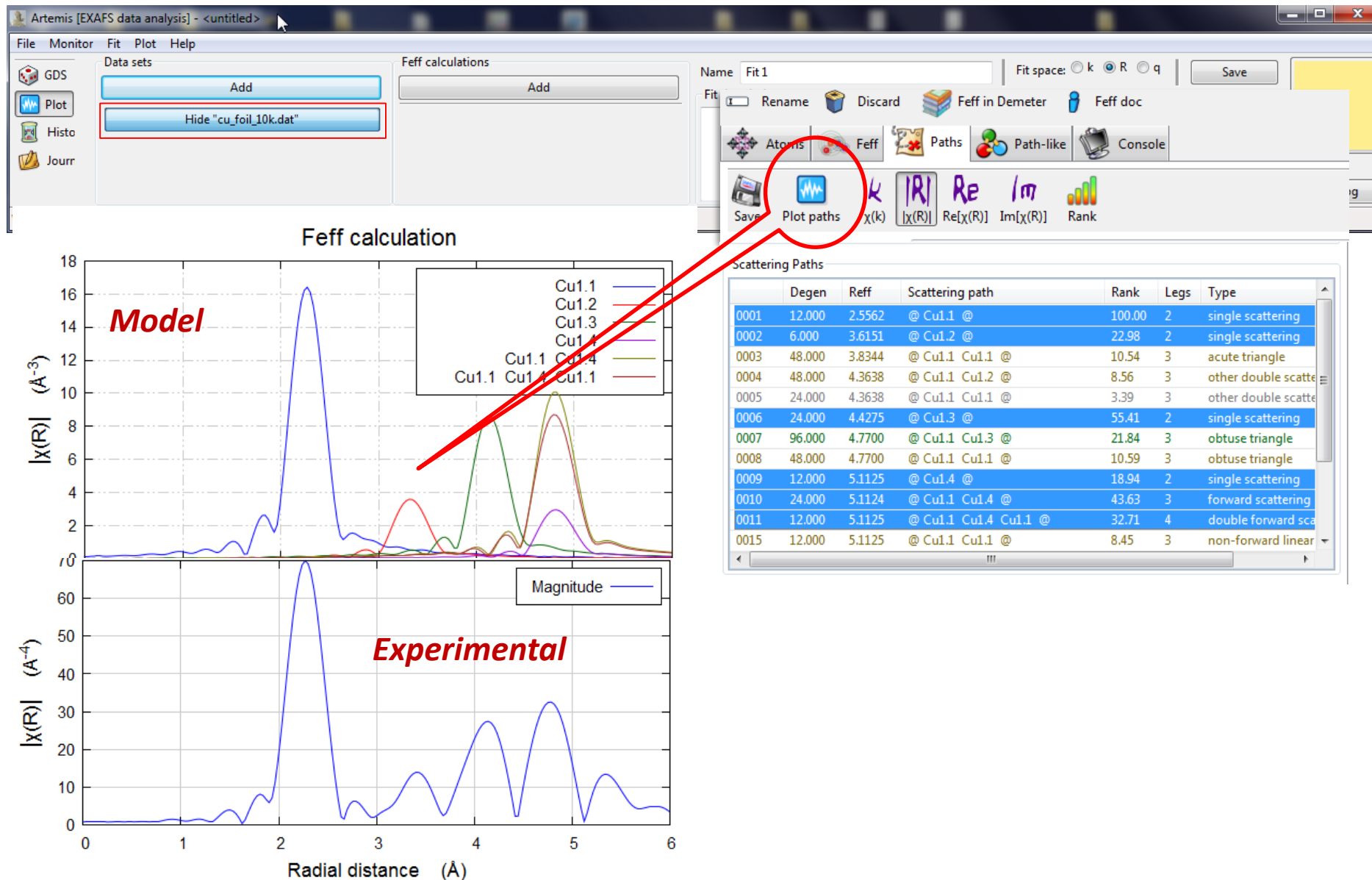
WARNING
This may crash!

Evaluate path relevance
looking at the Rank &/or at
the plots



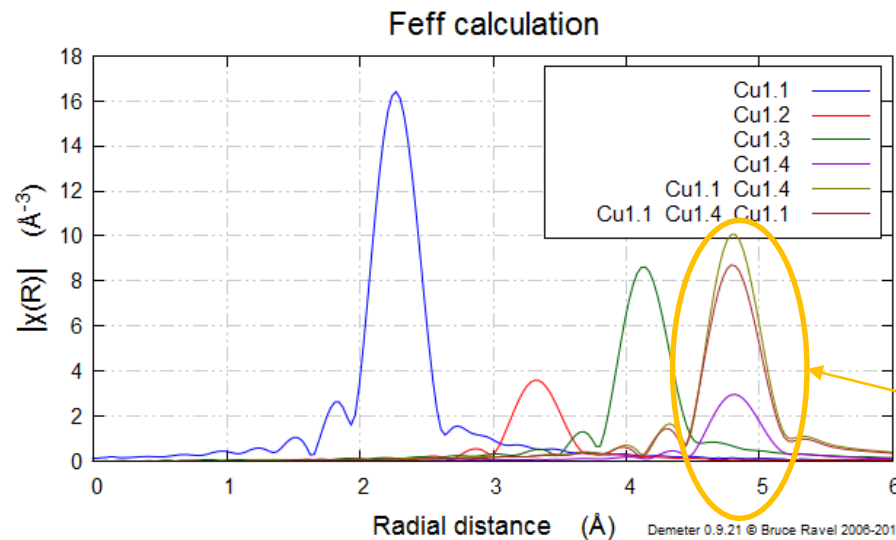
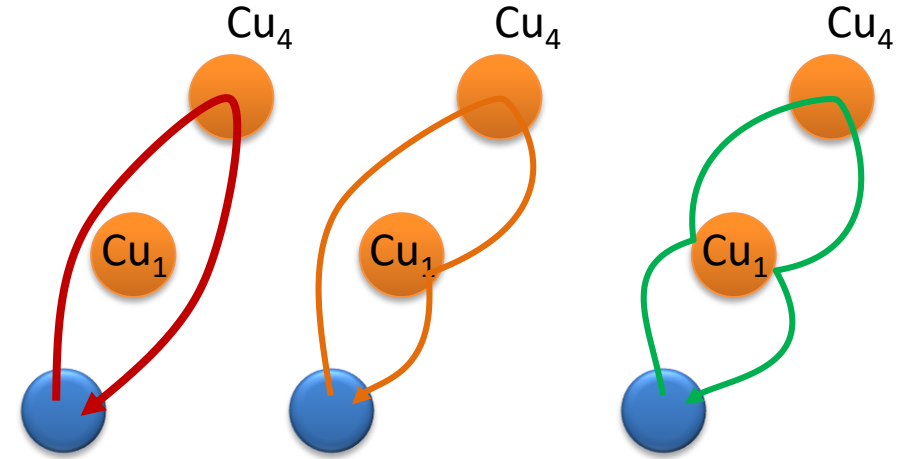
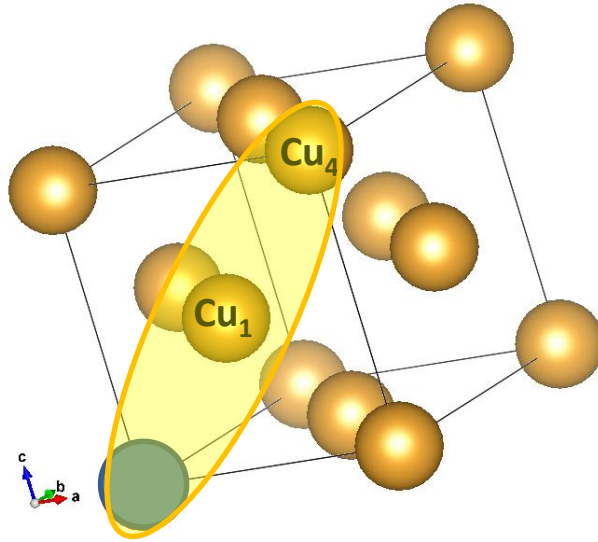
Path geometry

2: add model structure (cif or simple pair)



2: Check paths by relevance: Multiple scattering

Focusing effect enhances the multiple scattering from aligned configurations



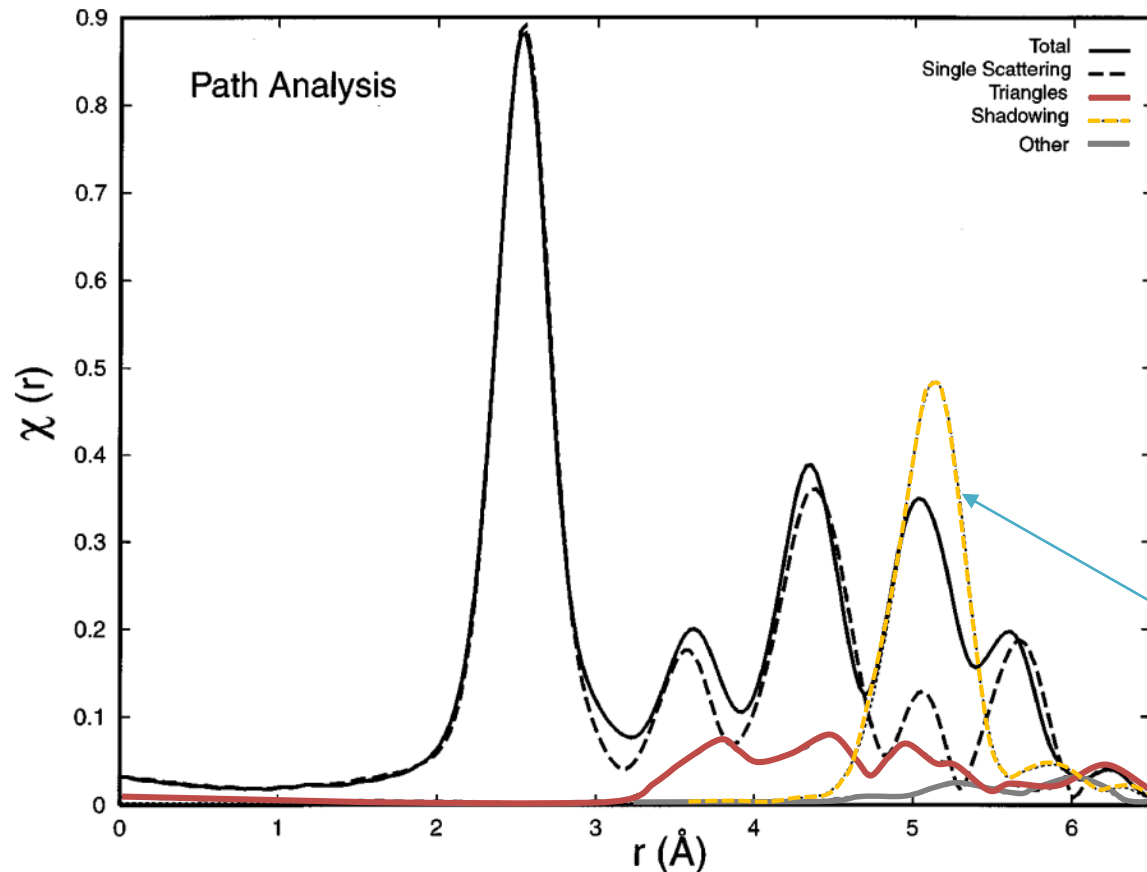
Cu1.4 Cu1.1-Cu1.4 Cu1.1-Cu1.4-Cu1.1

Scattering Paths

	Degen	Reff	Scattering path	Rank	Legs	Type
0001	12.000	2.5562	@ Cu1.1 @	100.00	2	single scattering
0002	6.000	3.6151	@ Cu1.2 @	22.98	2	single scattering
0003	48.000	3.8344	@ Cu1.1 Cu1.1 @	10.54	3	acute triangle
0004	48.000	4.3638	@ Cu1.1 Cu1.2 @	8.56	3	other double scatter
0005	24.000	4.3638	@ Cu1.1 Cu1.1 @	3.39	3	other double scatter
0006	24.000	4.4275	@ Cu1.3 @	55.41	2	single scattering
0007	96.000	4.7700	@ Cu1.1 Cu1.3 @	21.84	3	obtuse triangle
0008	48.000	4.7700	@ Cu1.1 Cu1.1 @	10.59	3	obtuse triangle
0009	12.000	5.1125	@ Cu1.4 @	18.94	2	single scattering
0010	24.000	5.1124	@ Cu1.1 Cu1.4 @	43.63	3	forward scattering
0011	12.000	5.1125	@ Cu1.1 Cu1.4 Cu1.1 @	32.71	4	double forward sca
0015	12.000	5.1125	@ Cu1.1 Cu1.1 @	8.45	3	non-forward linear

A note on the focusing effect

Contributions of some multiple-scattering paths to $\chi(k)$ for Cu.



Rev. Mod. Phys., Vol. 72, No. 3, July 2000

Single-scattering paths go from the central atom to another atom and then scatter back to the central atom.

Triangular paths hit two atoms and then return.

Shadowing paths hit another atom, and then goes straight through (in exactly the same direction) to hit another atom, before returning through the previous atom to the central atom. Because the forward scattering amplitude is large, shadowing paths can be accentuated by this “focusing” effect.

Because phase information is not retained in the figure, **destructive interference** between some of the contributions can reduce the absolute value of the total below the absolute value of some of the contributions.

Drag and drop relevant paths to the data window

Artemis [Feff] Atoms and Feff

Rename Discard Feff in Demeter Feff doc

Atoms Feff Paths Path-like Console

Save Plot paths $\chi(k)$ $|R|$ $Re[\chi(R)]$ $Im[\chi(R)]$ Rank

Name of this Feff calculation: icسد_43493_Copper

Description

```
# TITLE Copper
# TITLE Cu
# The central atom is denoted by this token: @
# Cluster size = 6.00 Å, containing 86 atoms
# 31 paths were found within 6.000 Å
# Forward scattering cutoff 20.00
```

Scattering Paths

	Degen	Reff	Scattering path	Rank	1	Type
1	12.00	2.556	@ Cu1.1 @	10...	:	single scatte
2	6.00	3.615	@ Cu1.2 @	22.97	:	single scatte
3	48.00	3.834	@ Cu1.1 Cu1.1 @	10.56	:	acute triangl
4	48.00	4.364	@ Cu1.1 Cu1.2 @	8.54	:	other double
5	24.00	4.364	@ Cu1.1 Cu1.1 @	3.39	:	other double
6	24.00	4.428	@ Cu1.3 @	55.37	:	single scatte
7	96.00	4.770	@ Cu1.1 Cu1.3 @	21.64	:	obtuse triang
8	48.00	4.770	@ Cu1.1 Cu1.1 @	10.61	:	obtuse triang
9	12.00	5.112	@ Cu1.4 @	18.92	:	single scatte
10	24.00	5.112	@ Cu1.1 Cu1.4 @	43.54	:	forward scatt

Artemis [Data] cu_foil_10k.dat

Data Path Marks Actions Debug Help

cu_foil_10k.dat CV 1 [icسد_43493_Copper] Cu1.1

Data source
trieste2021\Training\Data\Cu\EXAFS_extraction\athena_Cu10k.prj, 1

Plot this data set as
k123 R123 Rmr Rk kg

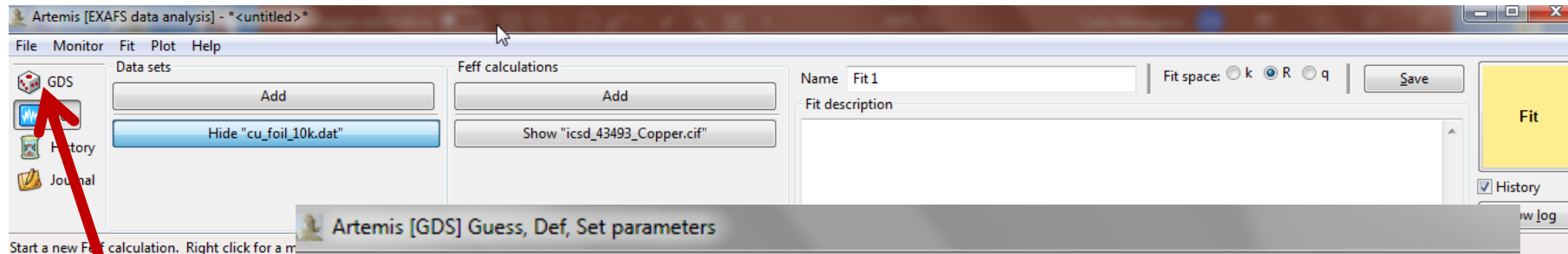
Title lines

Fourier transform parameters
kmin 3.000 kmax 23.019 dk 1
rmin 1 rmax 3 dr 0.0

Fitting k weights
☒ 1 ☒ 2 ☒ 3 ☐ other 0.5

Other parameters
☒ Include in fit ☒ Plot after fit ☐ Fit background
 $\epsilon(k)$ 0 ☐ Plot with phase correction

Define the refinement parameters



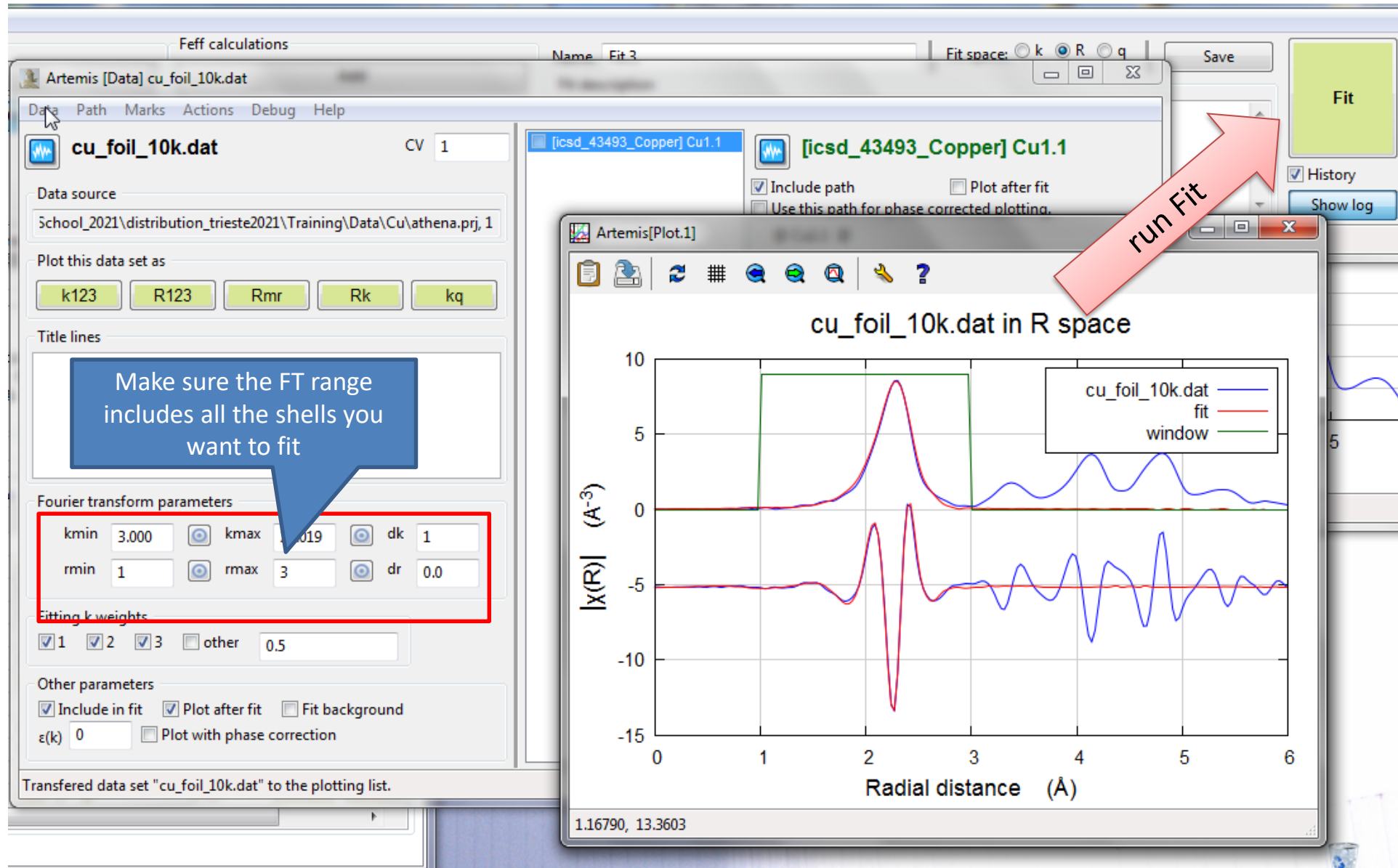
3 GDS:
define
refinement
parameters

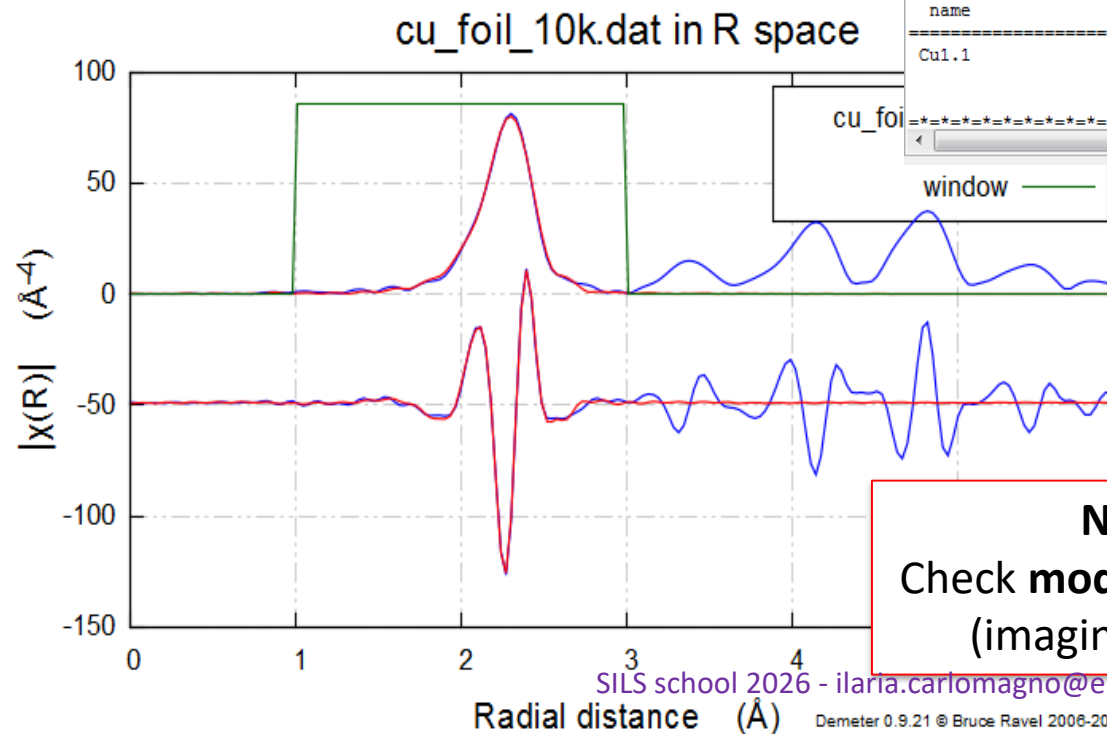
Artemis [GDS] Guess, Def, Set parameters

	Type	Name	Math expression	Evaluated
1	guess	DEo	0	
2	guess	dr_1	0	
3	guess	ss_2	0.00300	
4	guess			
5	guess			

Note:
Initialize to > 0
the σ^2 parameters

Best fit





Artemis [Log] Fit 1

```

: Athena project      = C:\Users\carlo\Dropbox\Seminario_Malu_2015\dati\ok_Cu_Foil\EXAFS_est
: name                = cu_foil_10k.dat
: k-range              = 3.000 - 23.019
: dk                   = 1
: k-window             = hanning
: k-weight             = 1,2,3
: R-range              = 1 - 3
: dR                   = 0.0
: R-window             = hanning
: fitting space        = r
: background function  = no
: phase correction     = no
: background removal   = E0: 8977.58, Rbkg: 1.0, range: [0:25.019], clamps: 0/24, kw: 2
: epsilon_k by k-weight = 1.857e-004
: epsilon_r by k-weight = 4.857e-001
: R-factor by k-weight = 1 -> 0.00421, 2 -> 0.00225, 3 -> 0.00273
    
```

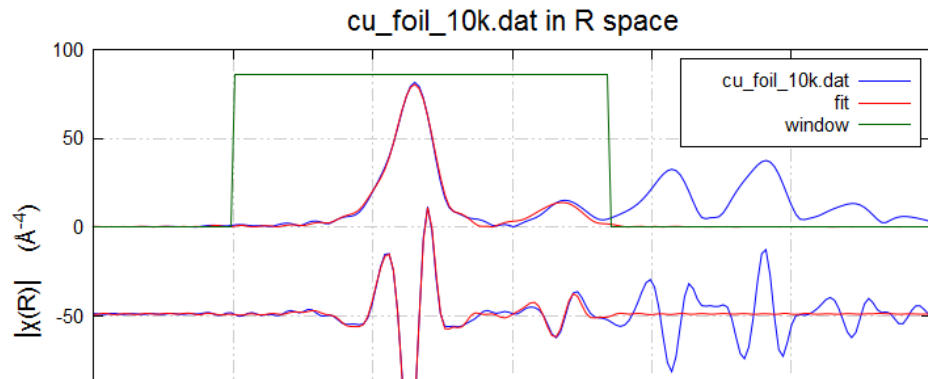
name	N	S02	sigma^2	e0	delr	Reff	R
Cu1.1	12.000	0.904	0.00345	5.419	-0.00827	2.55620	2.54793

name	ei	third	fourth
Cu1.1	0.00000	0.00000	0.00000

=====

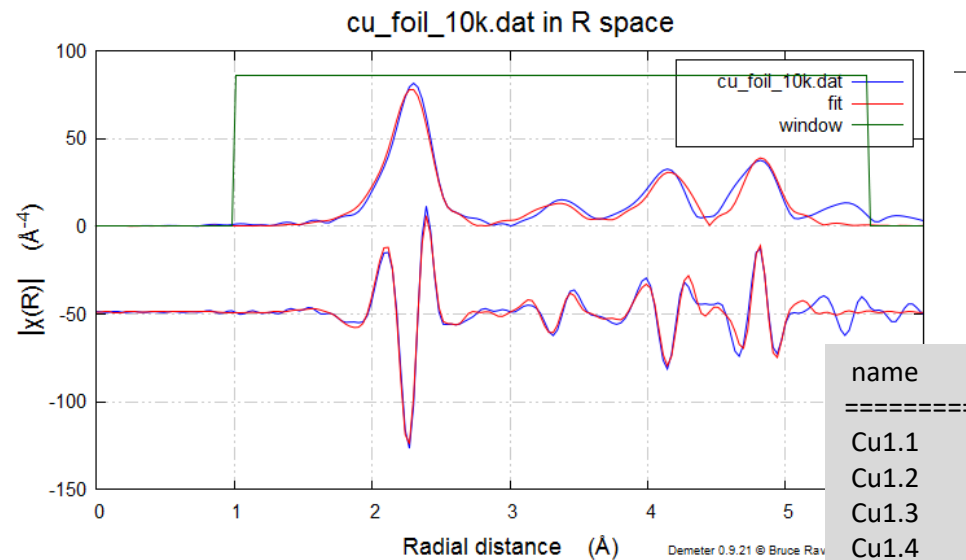
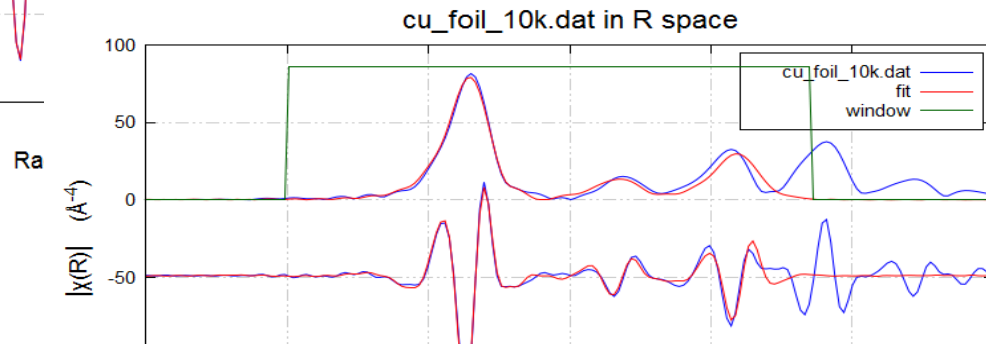
Note:
Check **modulus** and **real**
(imaginary) parts

Extend the analysis to further shells



Fourier transform parameters

kmin	3.000	☐	kmax	23.019	☐	dk	1.0
rmin	1	☐	rmax	3.7	☐	dr	0.0

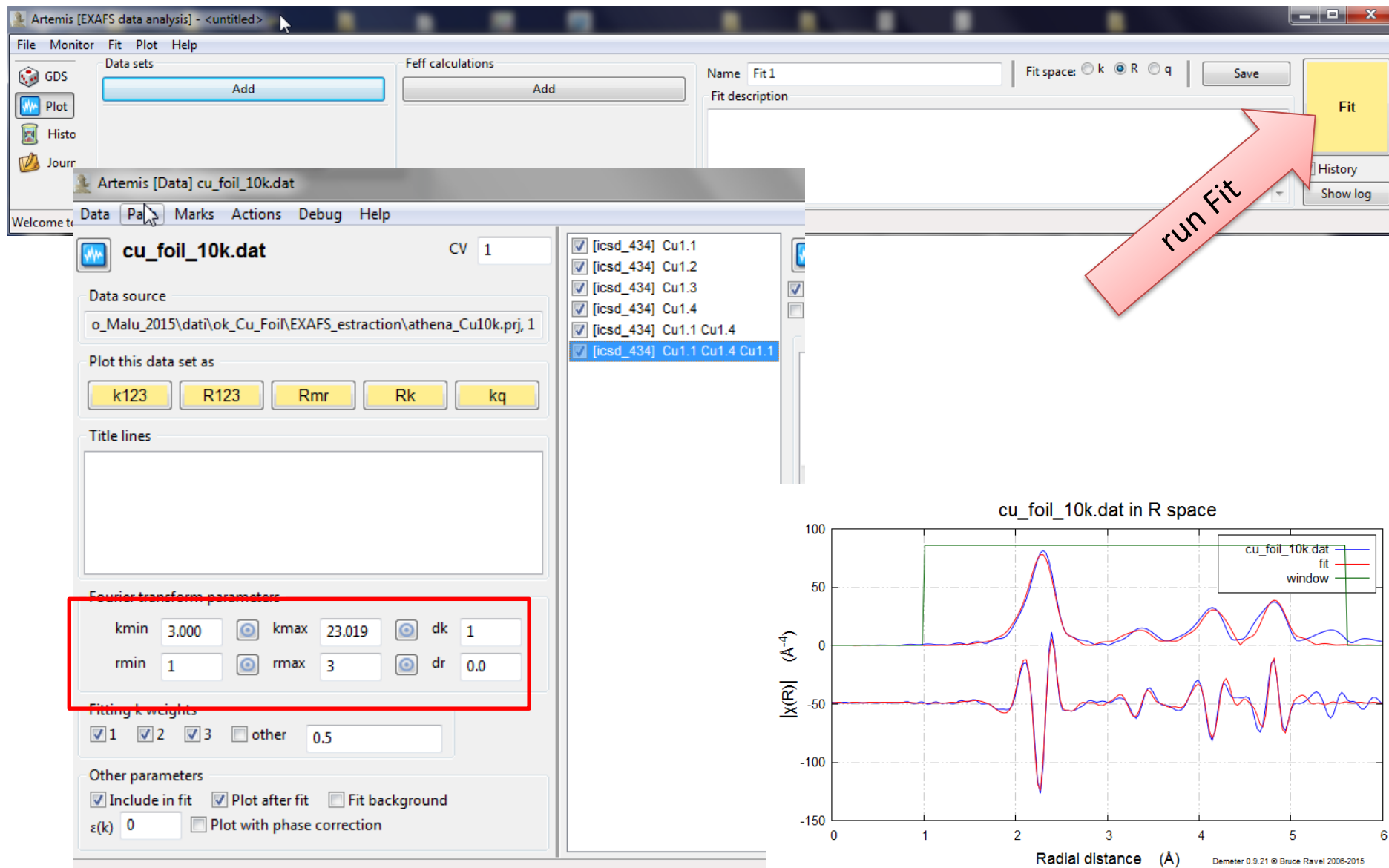


Run fit starting with first shell
then **add progressively further**
contributions

When extending your fit make sure that the
window defined in Athena is large enough!

name	N	S02	sigma^2	e0	delr	Reff	R
=====							
Cu1.1	12.000	0.980	0.00388	4.001	-0.01367	2.55620	2.54253
Cu1.2	6.000	0.980	0.00513	4.001	-0.02943	3.61510	3.58567
Cu1.3	24.000	0.980	0.00514	4.001	0.00381	4.42750	4.43131
Cu1.4	12.000	0.980	0.00092	4.001	0.00862	5.11250	5.12112
Cu1.1 Cu1.4	24.000	0.980	0.00271	4.001	0.00862	5.11250	5.12112
Cu1.1 Cu1.4 Cu1.1	12.000	0.980	0.00322	4.001	0.00862	5.11250	5.12112

Define parameters



Check best fit log file

Note:

Keep correlations among the parameters low (add physically meaningful constraints).
Far shell parameters are generally less accurate, use them with care.

Correlations between variables:

ss_42 & ss_4 --> **0.8812**

ss_1 & so2 --> **0.8229**

ss_43 & ss_42 --> **0.8137**

ss_43 & ss_4 --> **0.8062**

dr_1 & de_0 --> 0.7729

ss_43 & dr_4 --> 0.7515

dr_3 & de_0 --> 0.6824

dr_3 & dr_1 --> 0.5290

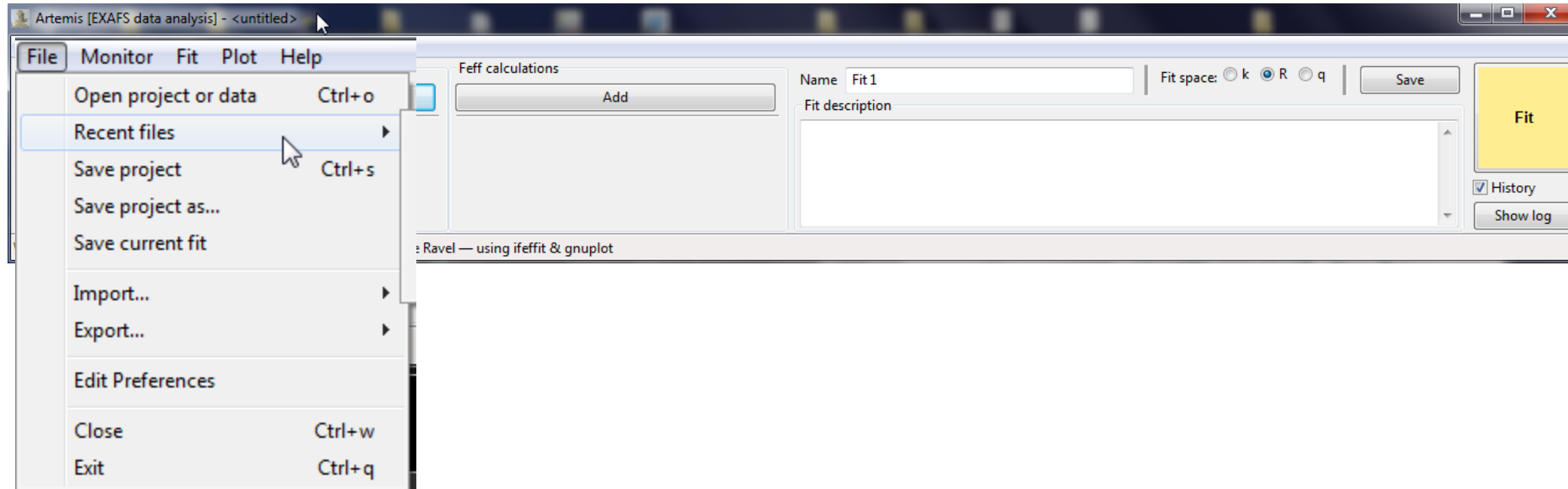
ss_3 & so2 --> 0.5153

ss_42 & so2 --> 0.4289

ss_3 & ss_1 --> 0.4239

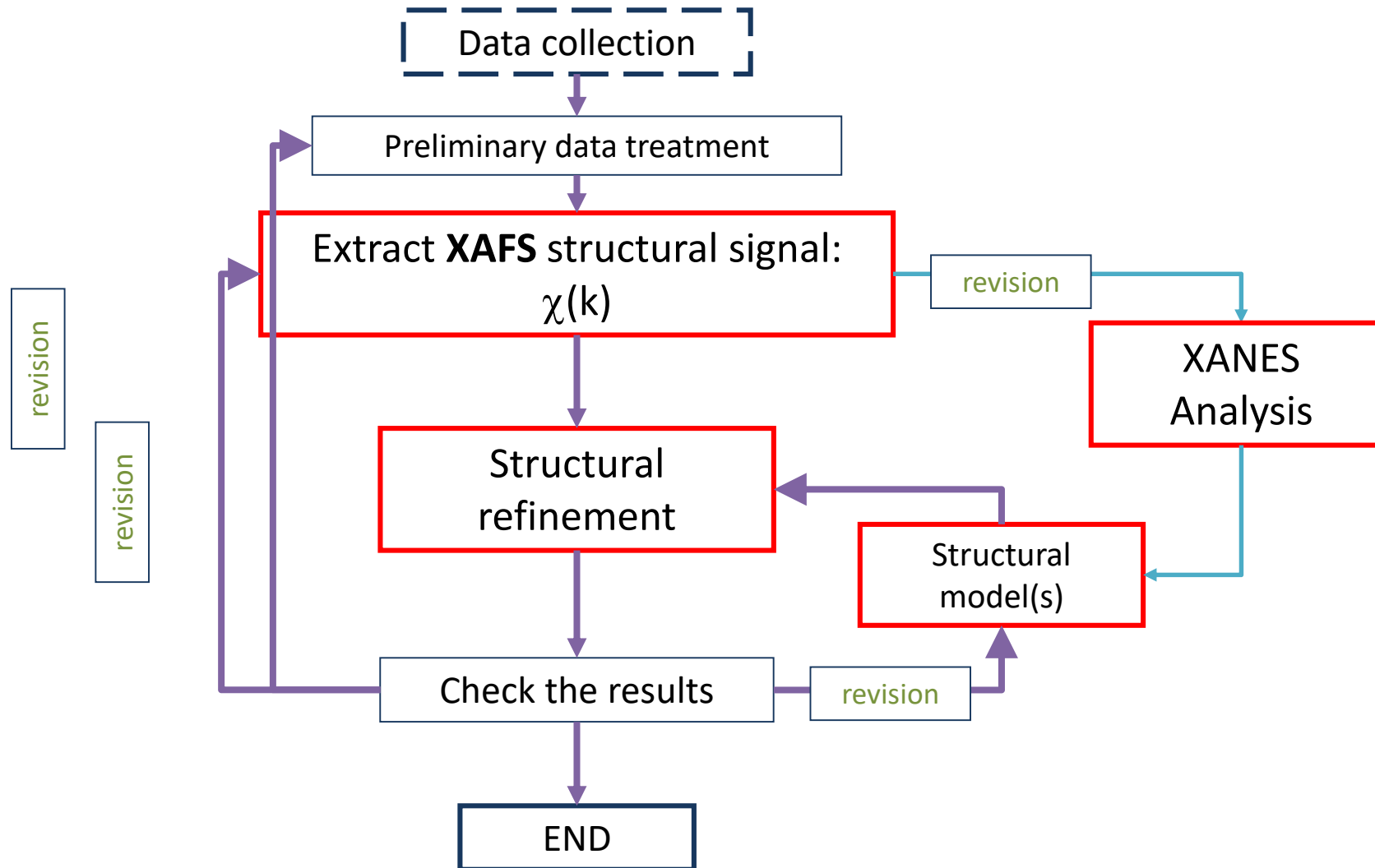
All other correlations below 0.4

5: Always save best fit and output files



6: Check your results and your hypothesis about local atomic structure... if mismatches check hypothesis, extraction, quality of the data...

XAFS data ANALYSIS: from experimental data to structural details



The end!

Additional resources and tutorials:

- <https://xafs.xrayabsorption.org/tutorials.html>
- <https://bruceravel.github.io/demeter/documents/Athena/index.html>
- EXAFS tutorial by Carlo Meneghini:
https://indico.elettra.eu/event/45/sessions/187/attachments/212/541/Meneghini_Tutorial_2024.pdf
- EXAFS tutorial by Sakura Pascarelli:
https://www.esrf.fr/files/live/sites/www/files/events/conferences/2017/User_Meeting_2017/Part%20I%20.pdf
- XANES tutorial by Sofia Diaz-Moreno: https://www.youtube.com/watch?v=hnWvc8Jm_es
- EXAFS tutorial by Chris Pollock: https://www.youtube.com/watch?v=gh_1vGVluLA&t=1391s&pp=ugUEEgJlbg%3D%3D