ROMA TRE UNIVERSITÀ DEGLI STUDI

Dipartimento di Scienze

Data analysis of XAFS data

1. XAFS data analysis and related software
2. **From XAS to XAFS: data treatment procedures**
3. Training: EXAFS data refinement
4. Training: XANES data analysis

https://tinyurl.com/SRSelettra2021

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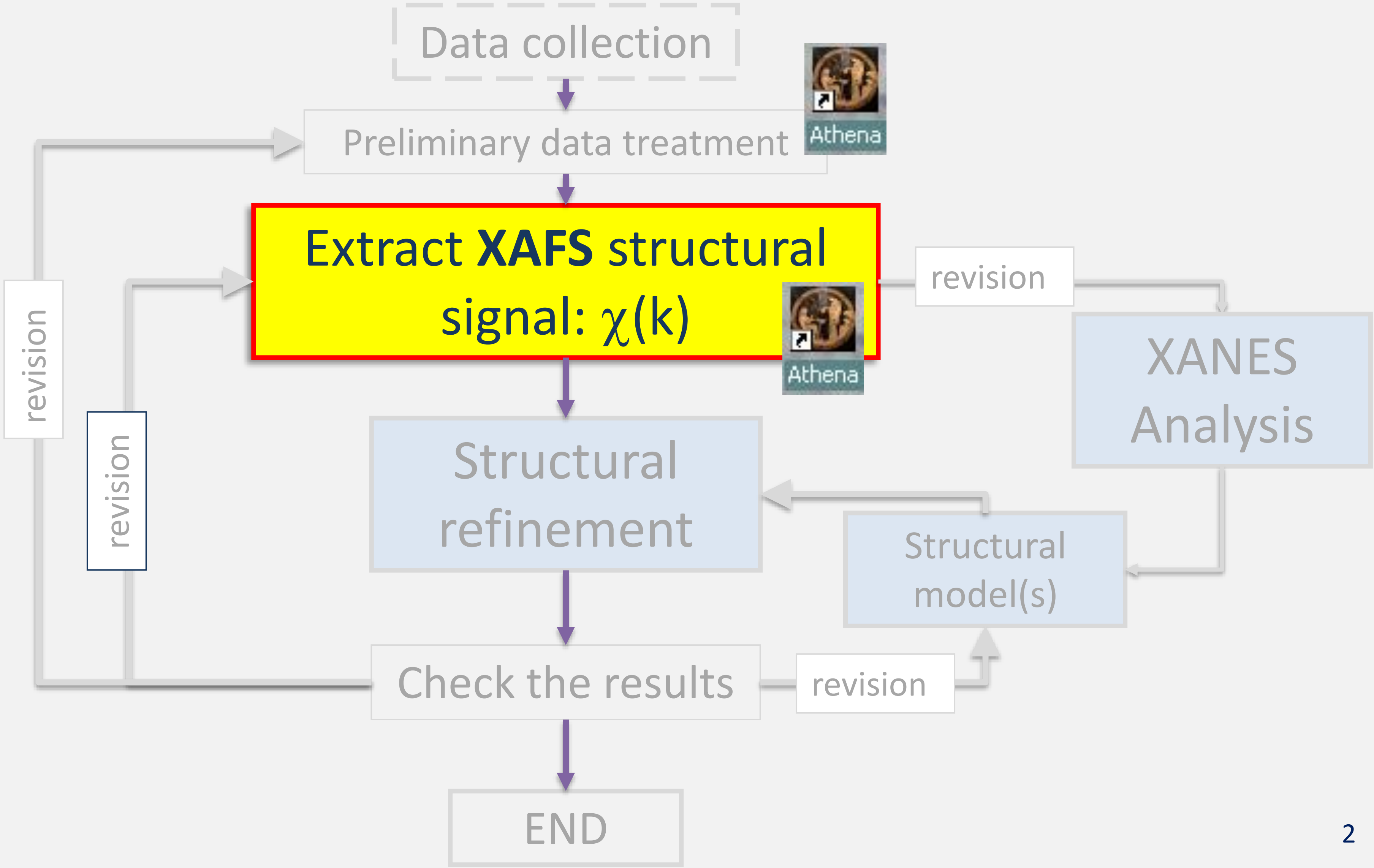
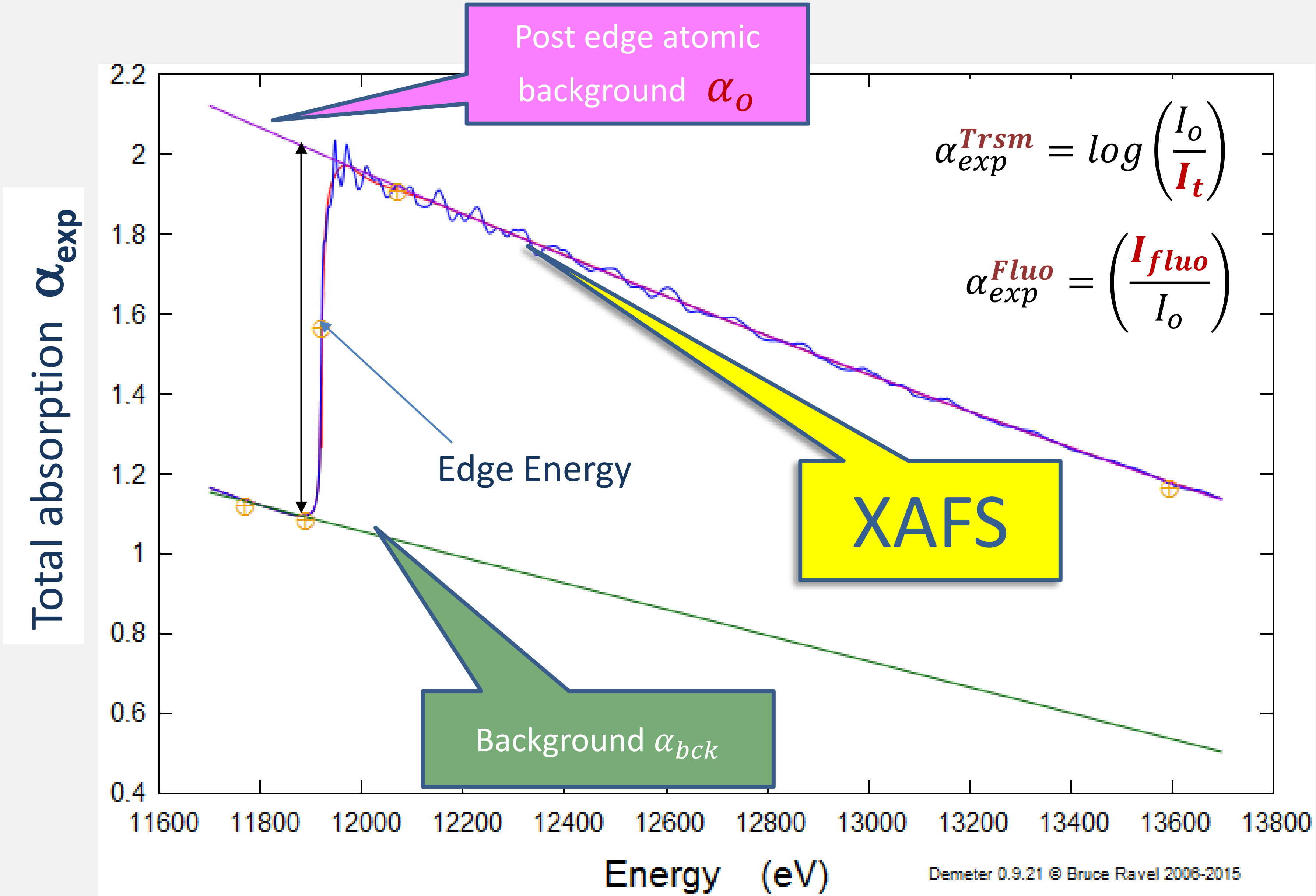
1st on-line School on Synchrotron Radiation "Gilberto Vlaic": Fundamentals, Methods and Application

XAFS data ANALYSIS: from the experimental data to structural details



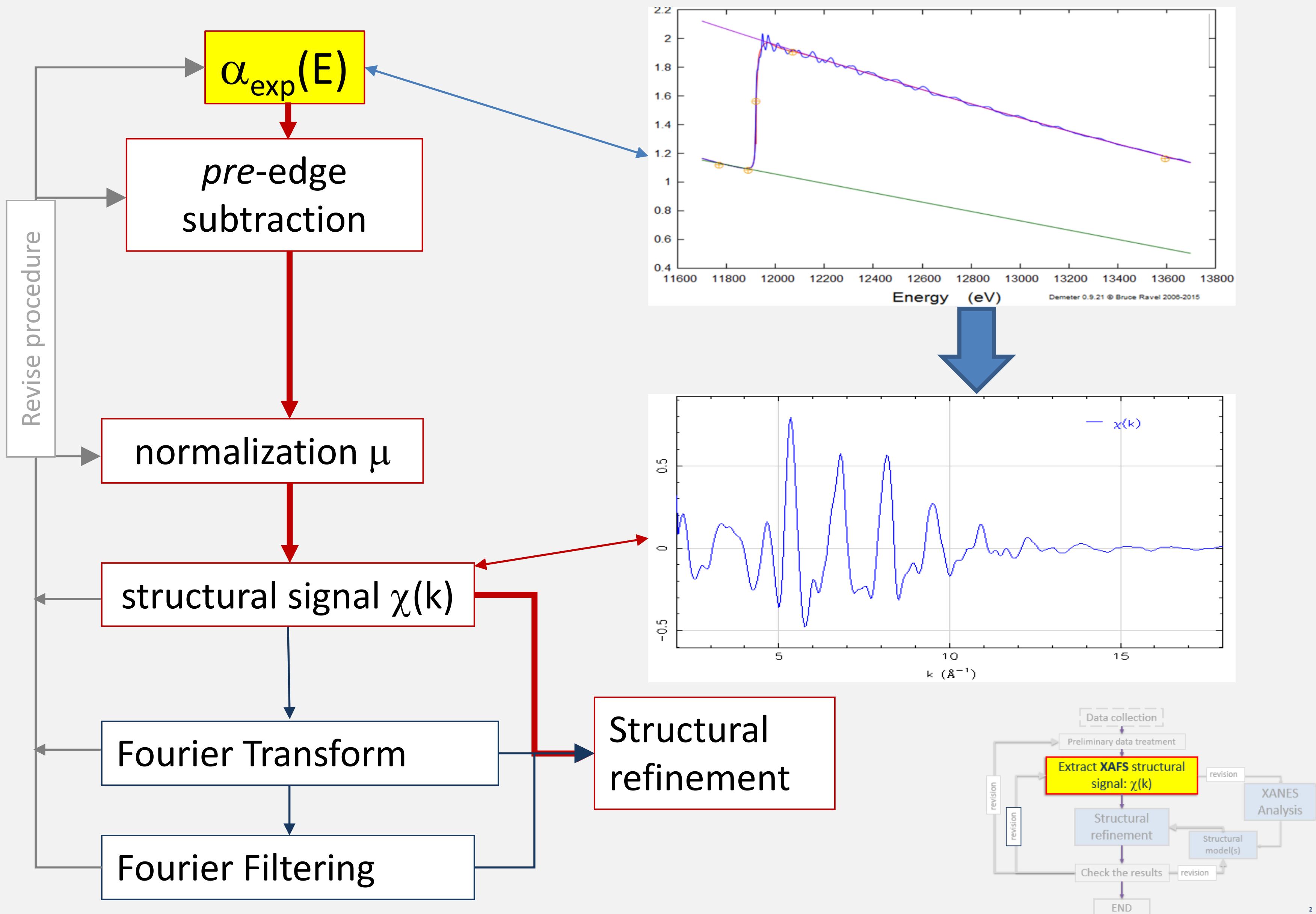
Characteristics of a XAS spectrum



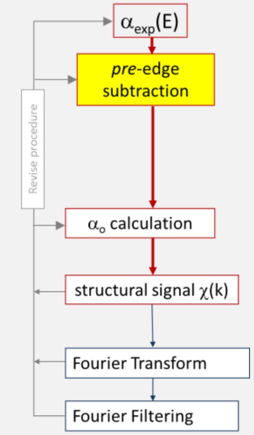
$$\alpha_{exp}^{Trsm} = log\left(\frac{I_o}{I_t}\right)$$

$$\alpha_{exp}^{Fluo} = \left(\frac{I_{fluo}}{I_o}\right)$$

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



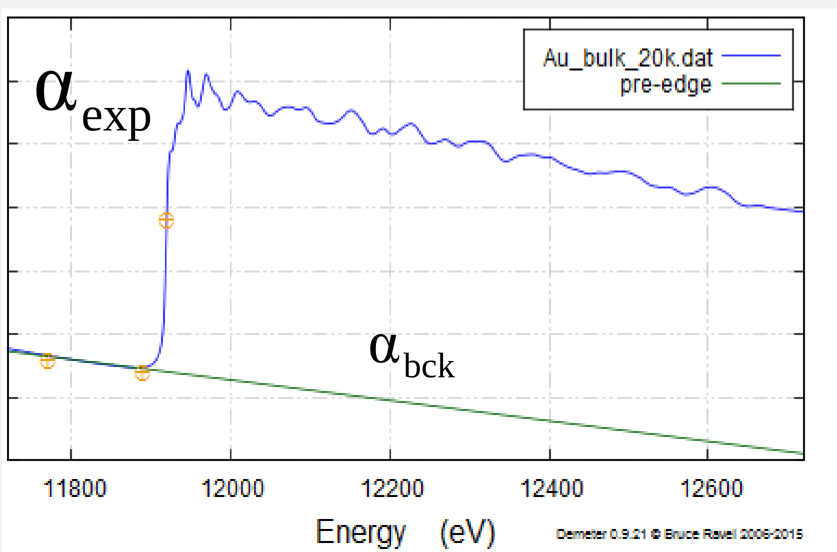
How to get the normalized $\chi(k)$



1. Remove pre-edge

$$\alpha_{exp} = \alpha_x + \alpha_{bck} = \mu_x t + \alpha_{bck}$$

$$\alpha_x = \mu_x t = \alpha_{exp} - \alpha_{bck}$$

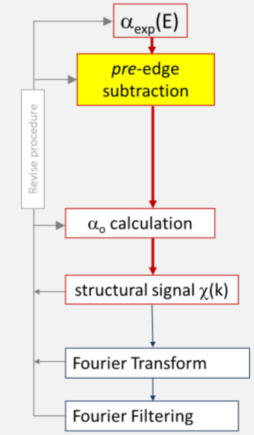


$\mu_x t$ is the absorption due to the edge we are working on

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

Everything being: other absorption edges, air, sample holder, matrices, chamber windows, etc…

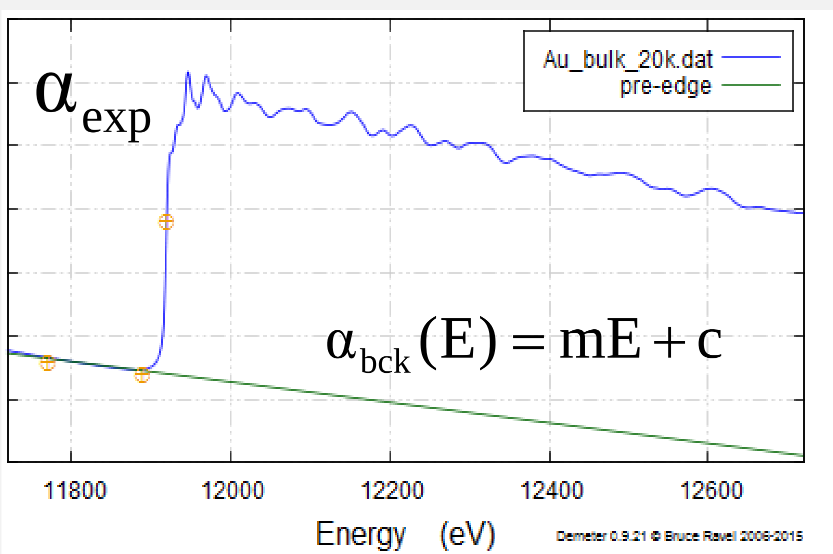
How to get the normalized $\chi(k)$

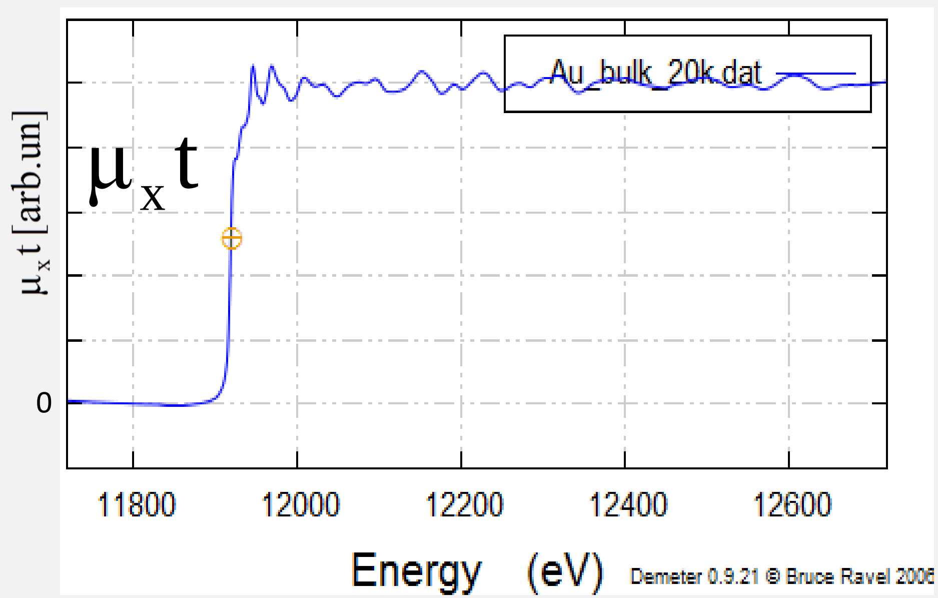


1. Remove pre-edge

$$\alpha_{exp} = \alpha_x + \alpha_{bck} = \mu_x t + \alpha_{bck}$$

$$\alpha_x = \mu_x t = \alpha_{exp} - \alpha_{bck}$$

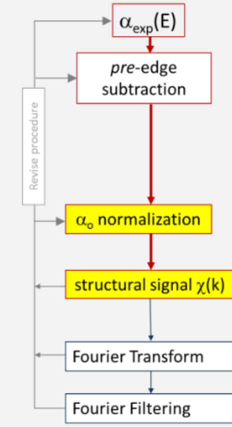




The pre-edge is simulated as a linear regression of data before the edge energy

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

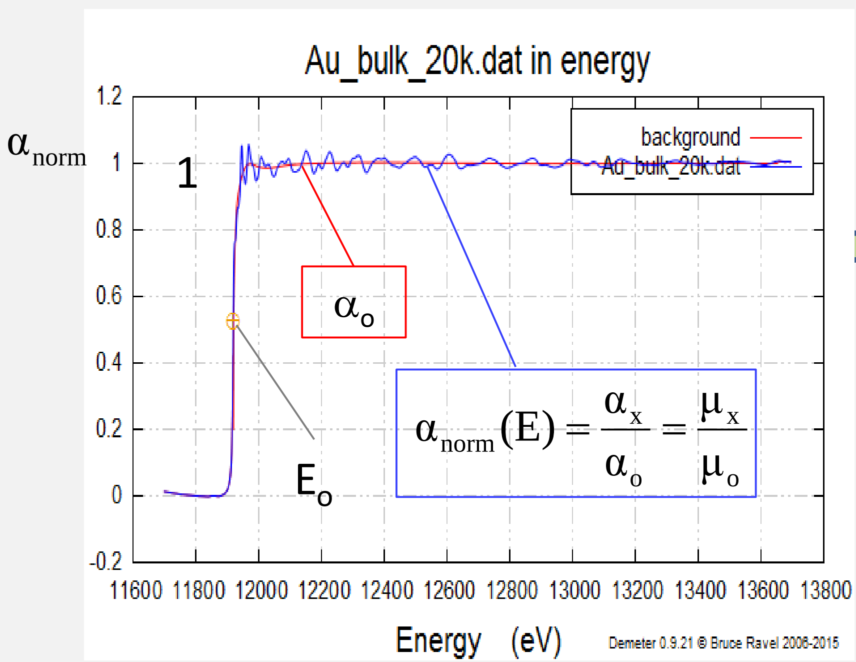
2. Normalization, χ and k



$$\alpha_x = \mu_x t \simeq \mu_o (1 + \chi) t$$

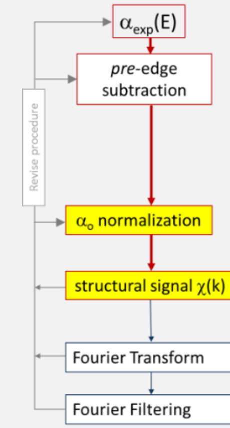
effect of Neighbours

isolated absorber



$$\alpha_{norm} = \frac{\mu_o (1 + \chi) t}{\mu_o t} = \frac{\boldsymbol{\alpha_x}}{\boldsymbol{\alpha_o}}$$

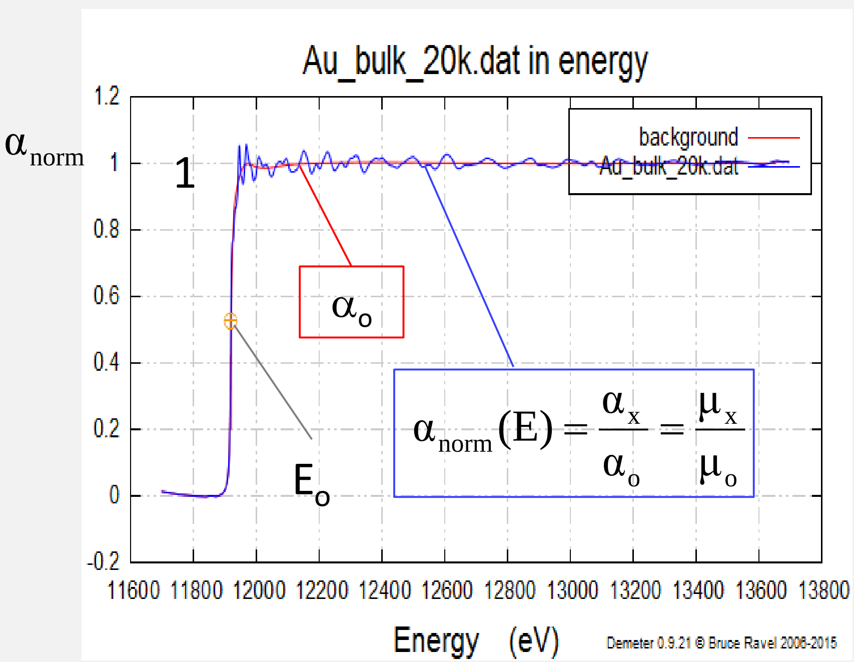
How to get the normalized $\chi(k)$



2. **Normalization**, χ and k

$$\alpha_{norm} = \frac{\mu_o(1+\chi)t}{\mu_o t} = \frac{\alpha_x}{\alpha_o}$$

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

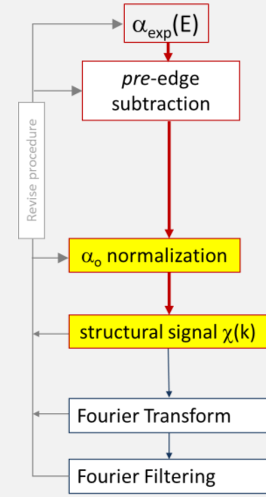


α_o is calculated empirically as a smooth curve across the data.
*Different programs for XAFS data analysis apply different (**equivalent**) methods*

Requirements for α_o :
1) **Smooth enough** to not remove true structural features
2) **Structured enough** to remove not structural background structures

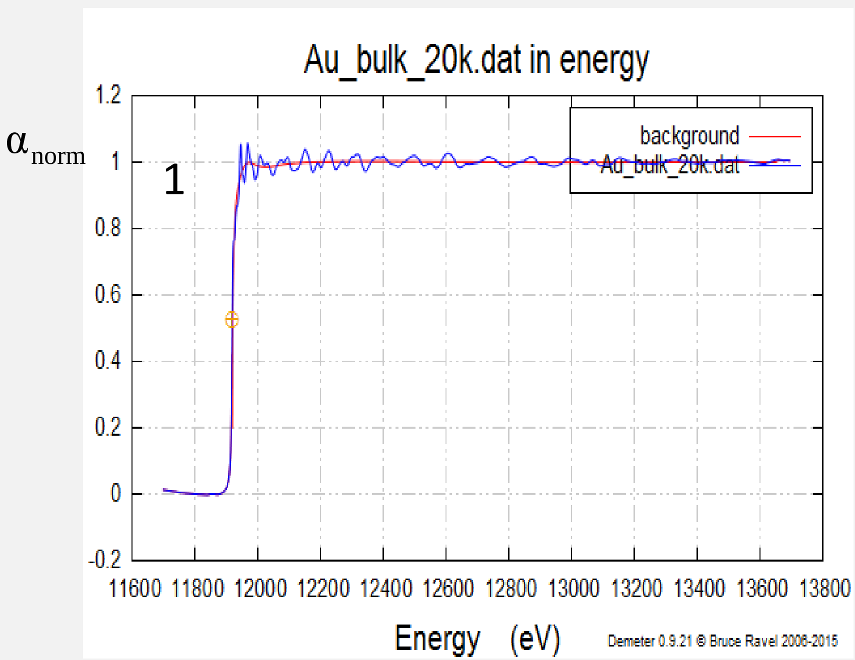
How to get the normalized $\chi(k)$

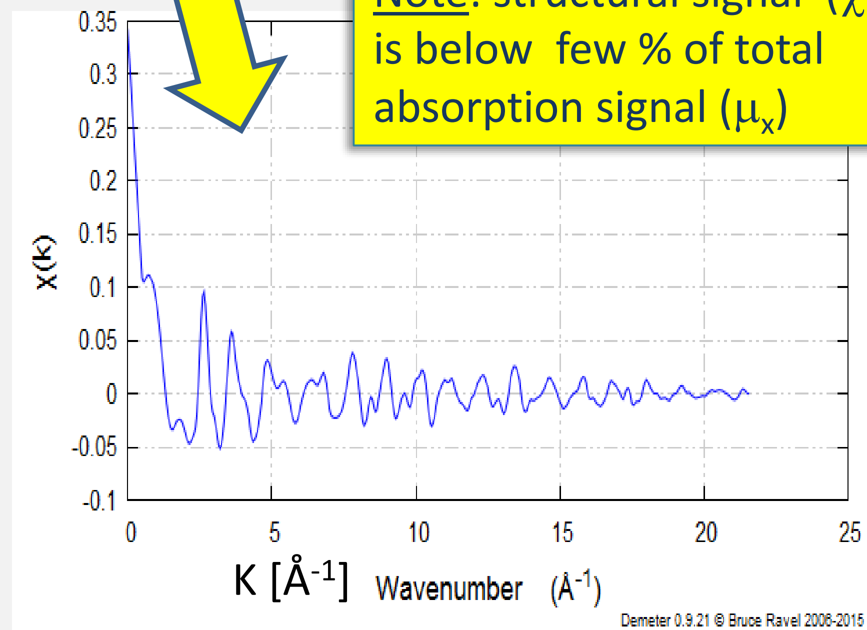
2. Normalization, χ and k



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

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



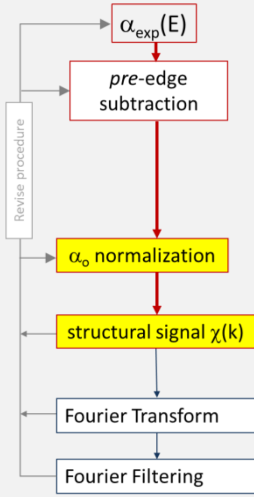


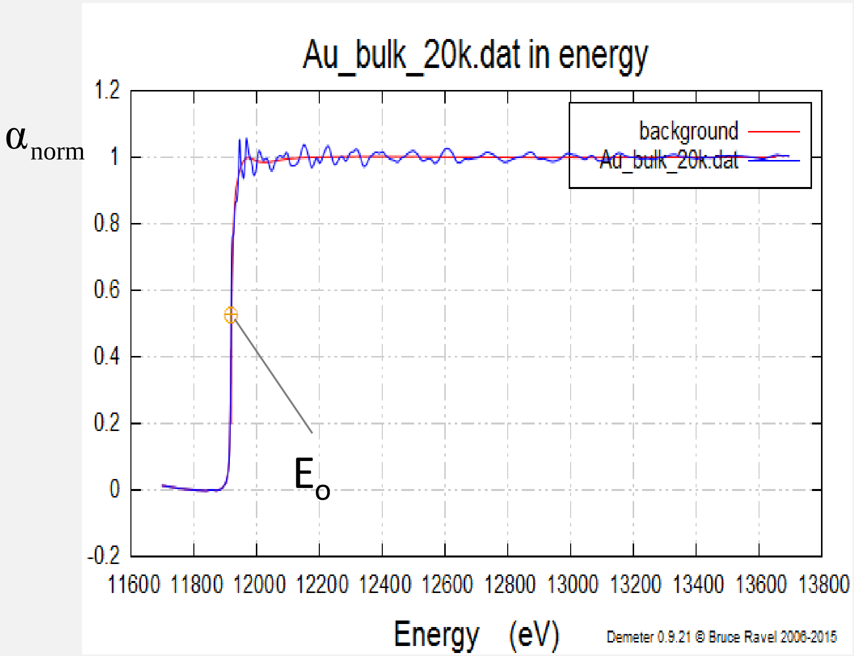
<u>Note</u>: structural signal (χ) is below few % of total absorption signal (μ_x)

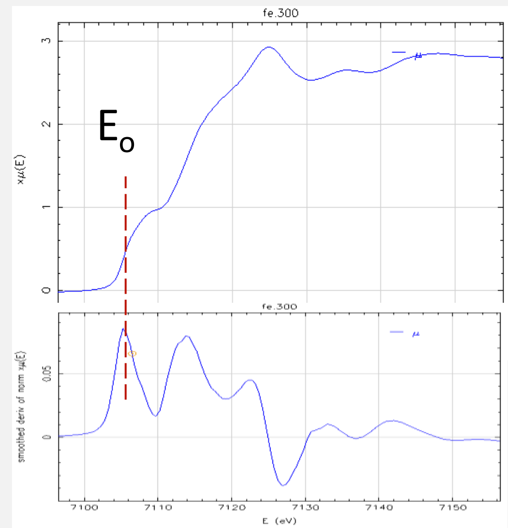
How to get the normalized χ(k)

2. Normalization, χ and **k**

$$k = \sqrt{[2m(E_{h\nu} - E_0)/\hbar^2]}$$

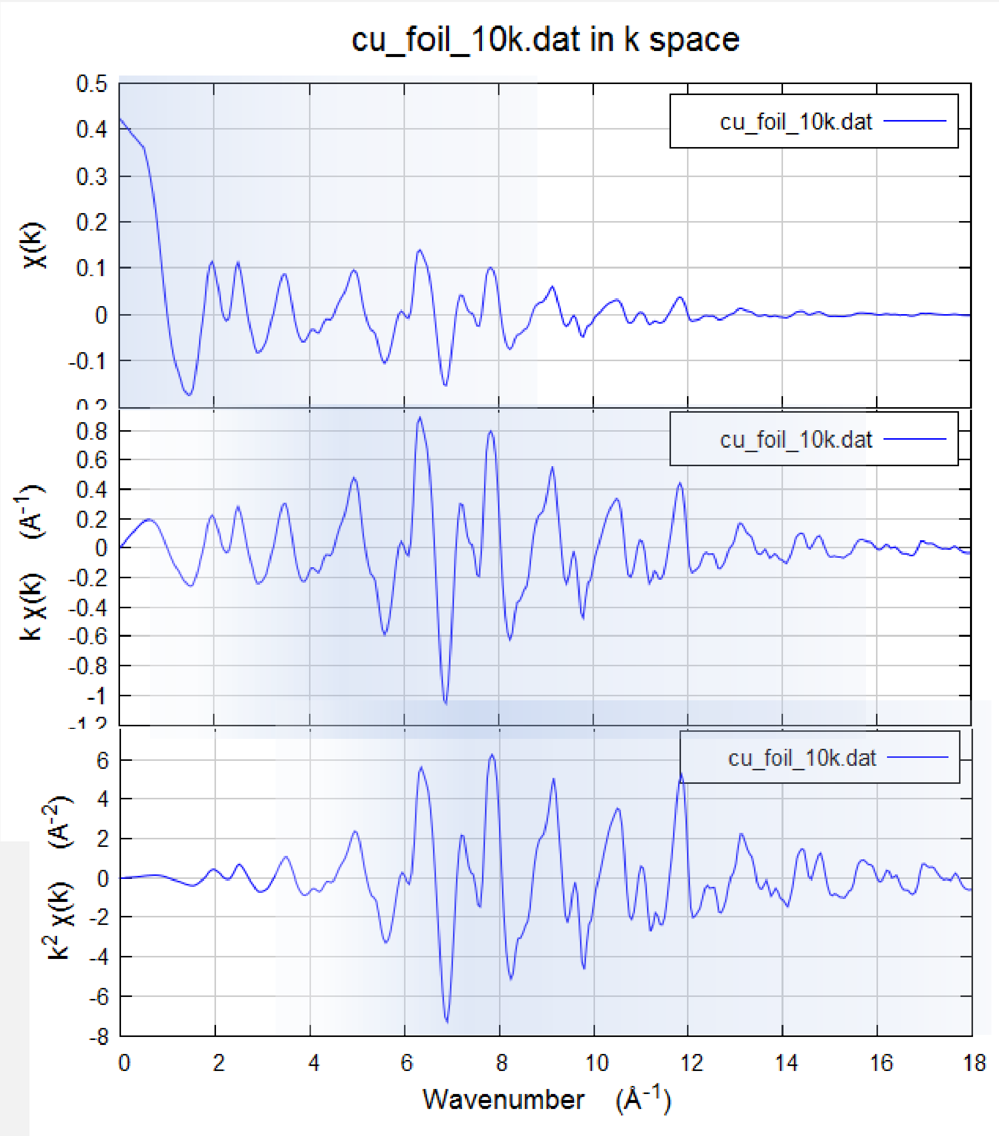






Edge energy is selected at the **first inflection point of α_{nor} or** where α_{nor} =0.5
It will be refined during the analysis.

Check your data: Inspect $k^n\chi(k)$

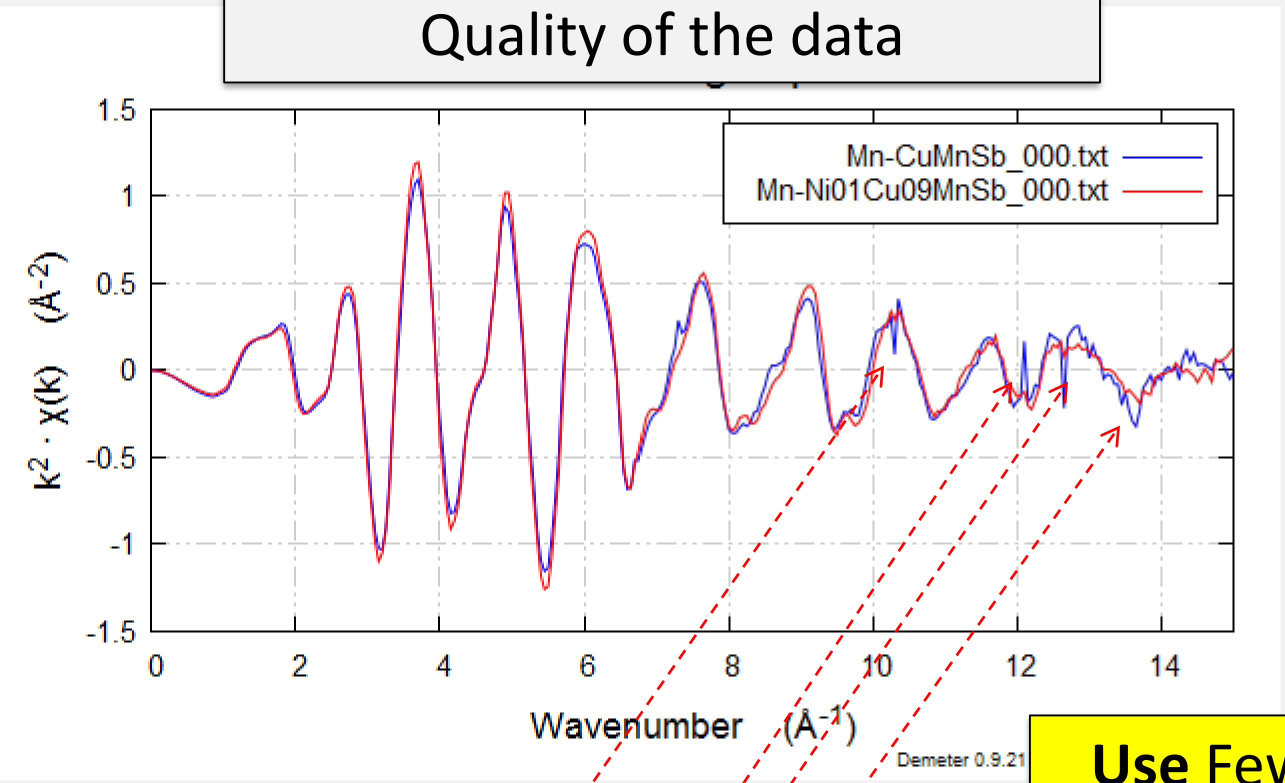


$k^n\chi(k)$ weighting highlights different features in the spectrum: high (low) n enhance high (low) k-regions

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



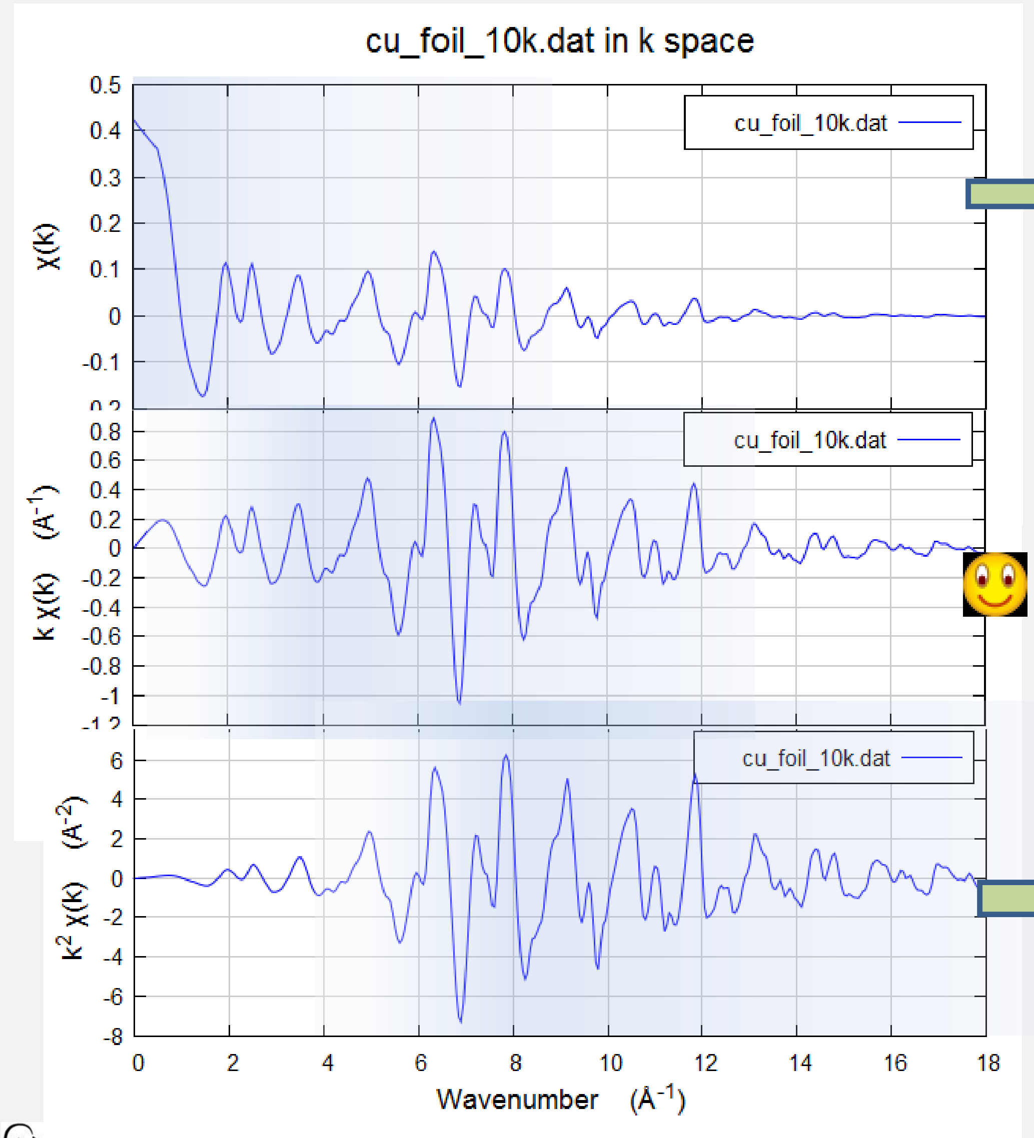
Check for distortions in the spectra, **correct** or **cut** the data and retain only the "good" regions

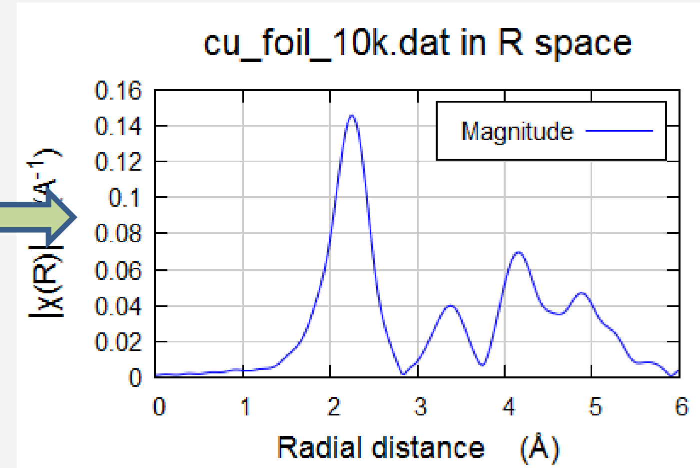
Use Few/short data **free from artifacts**

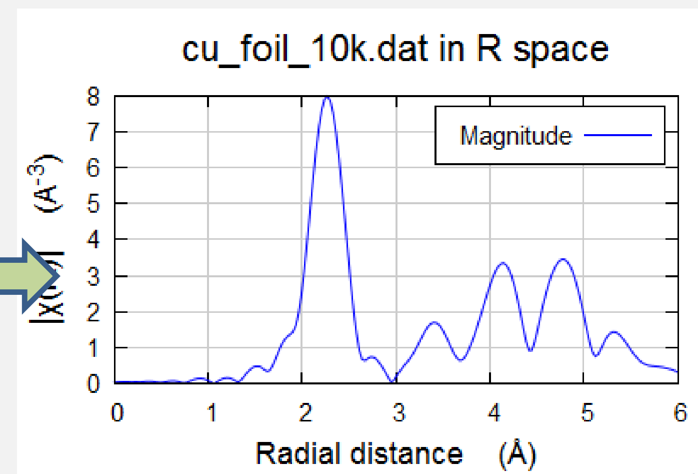
Qualitative local structure

3. Fourier transform

Cu



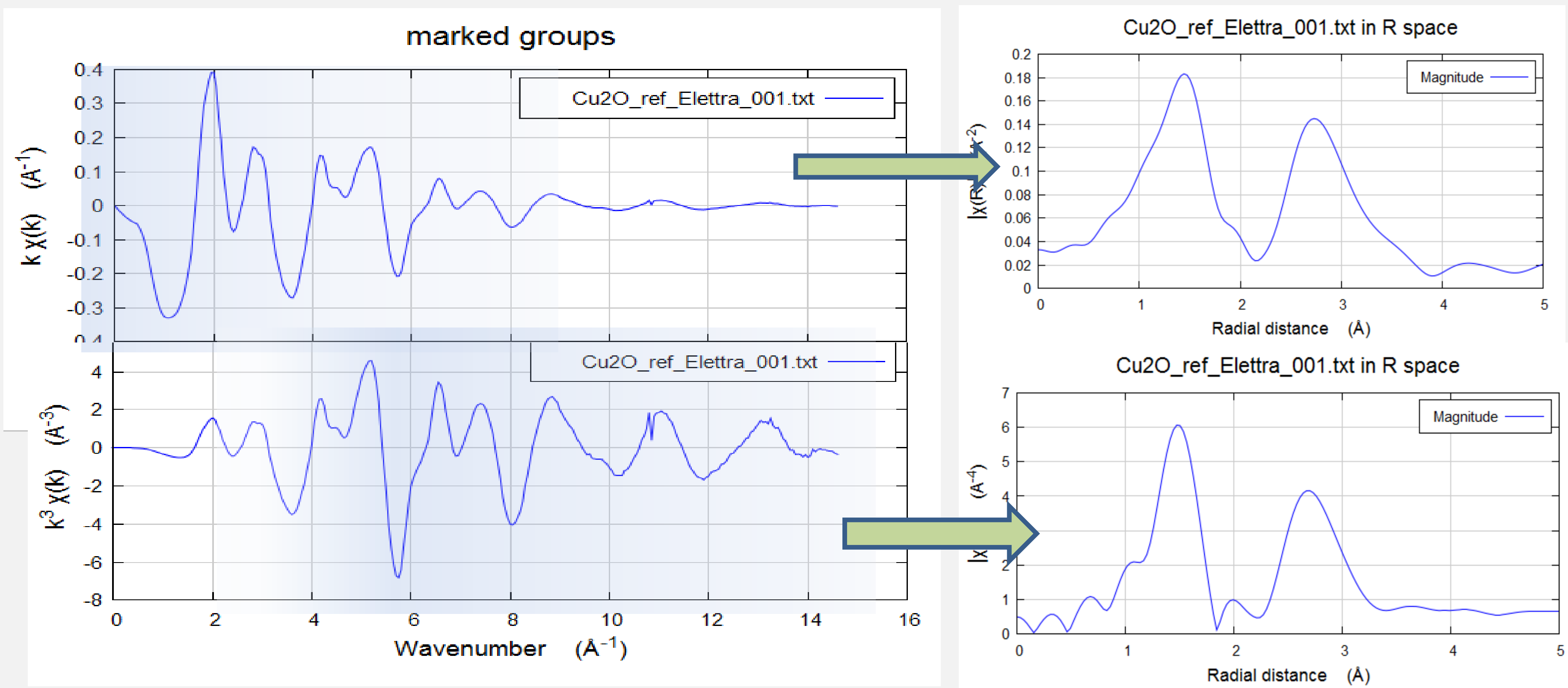




Qualitative local structure

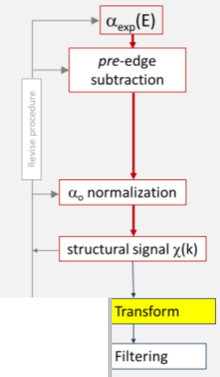
3. Fourier transform

Cu_2O

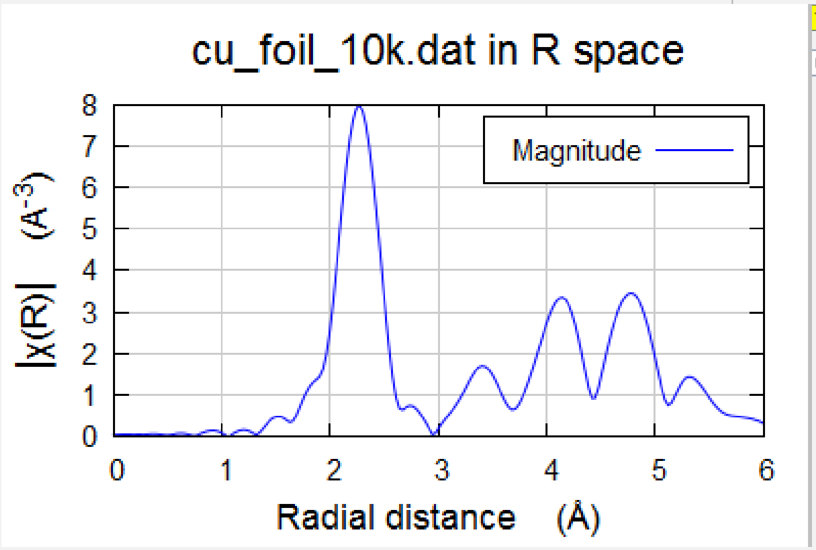


Qualitative local structure

3. 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.

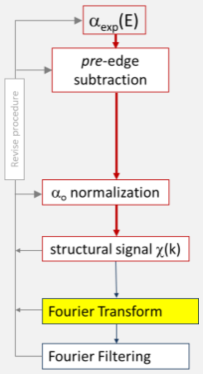


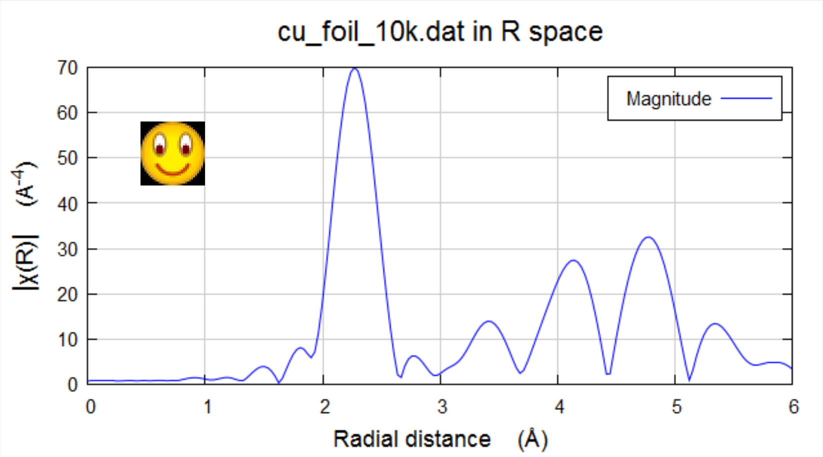
Peak positions (phase shift corrected) => neighbour shells
Peak amplitude and shape => number and type of neigbours

$$\chi(k) = \frac{1}{k}\sum \boldsymbol{A_j} \sin\left(2kr_j + \psi_j\right)$$

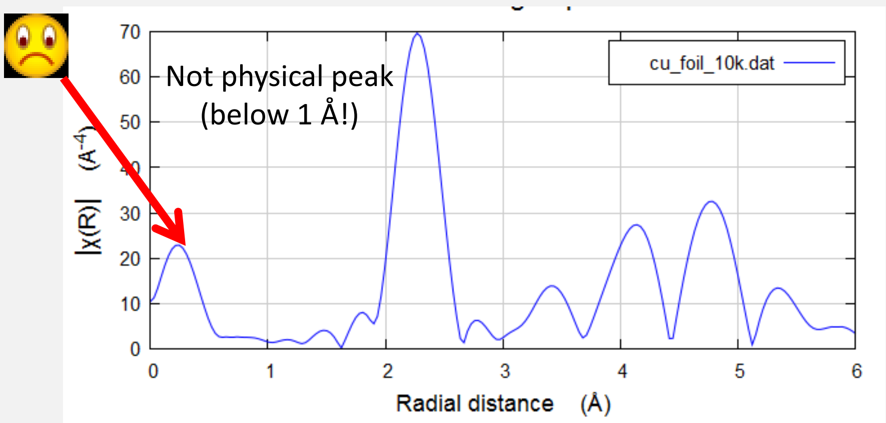
$$\boldsymbol{A_j} = S_o^2 \frac{N_j}{R_j}\left|f_j\right| e^{-2k^2\sigma_j^2}\, e_j^{-\frac{2r_j}{\lambda}}$$

Check FT





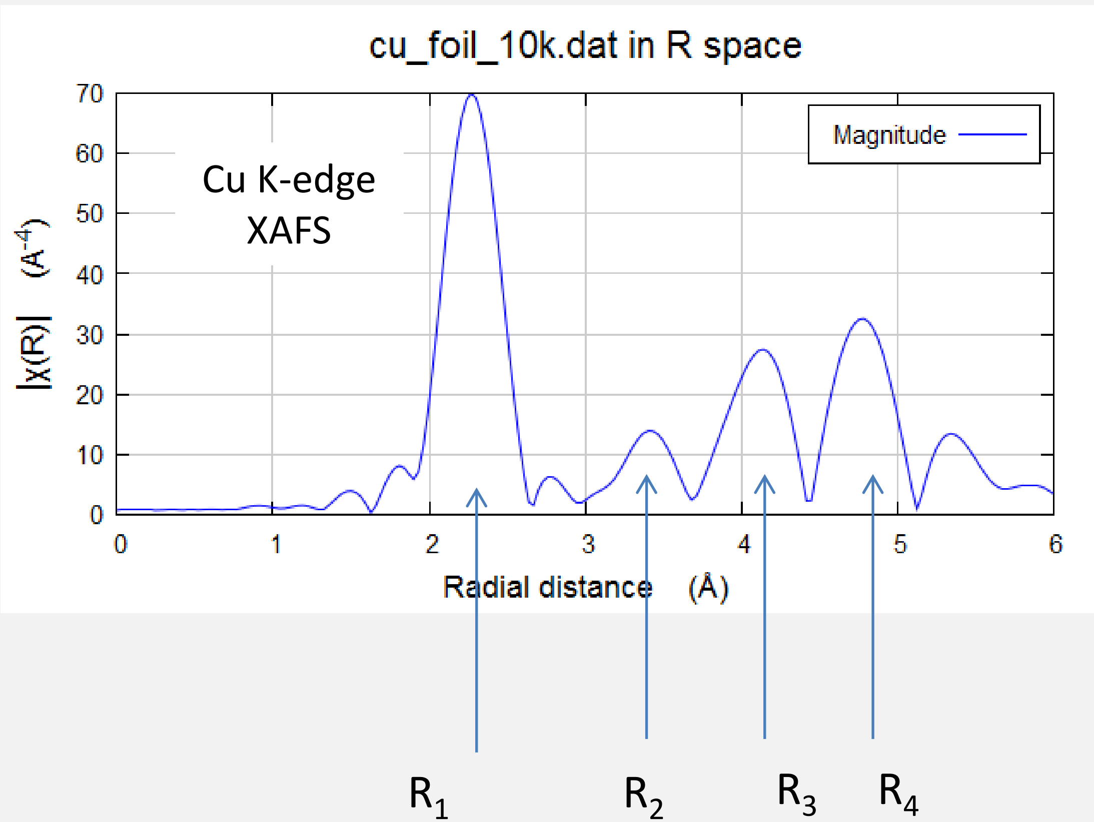
FT features:
Artifacts, distortions, noise, may suggests bad extraction, noise on the data, etc…

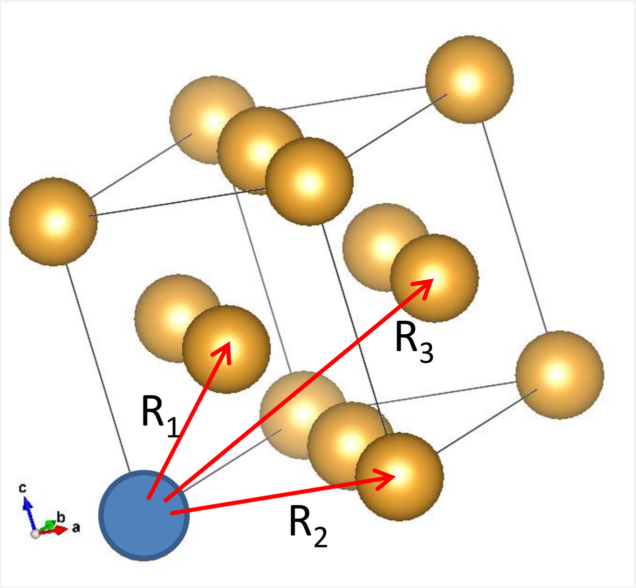


i.e.: intense peaks in the low R region (≈ less than 1Å) may signify errors in the extractions

FT and expected atomic structure

Look at the FT features and compare the structure you expect





$$R_1 = a\sqrt{2} \qquad N_1 = 12$$

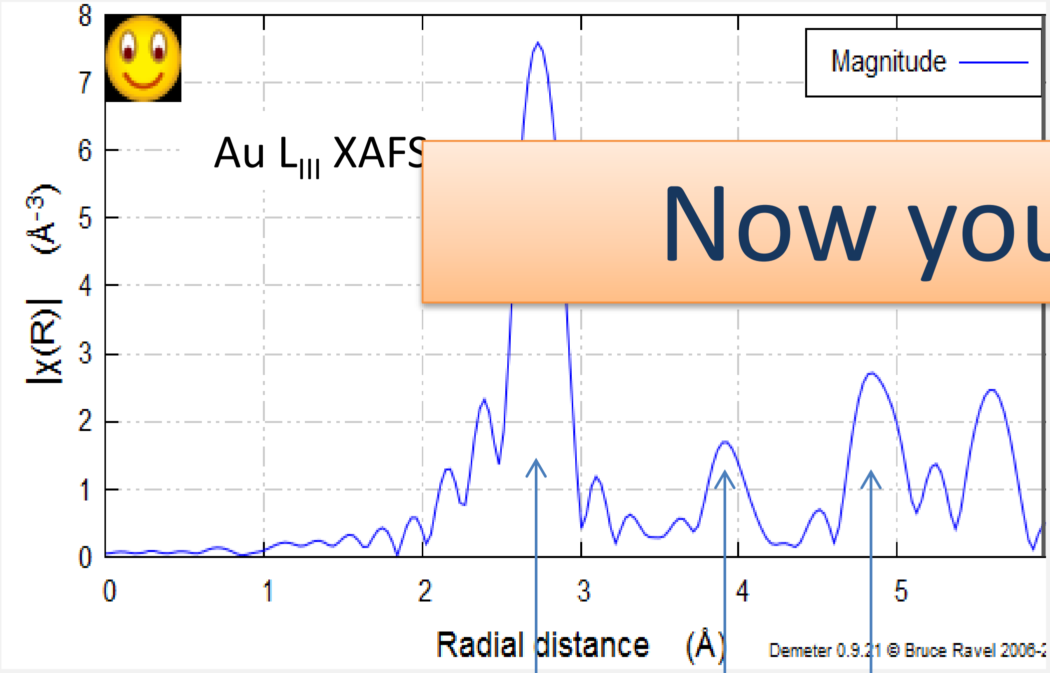
$$R_2 = a \qquad N_2 = 6$$

$$R_3 = \frac{a\sqrt{6}}{2} \qquad N_3 = 24$$

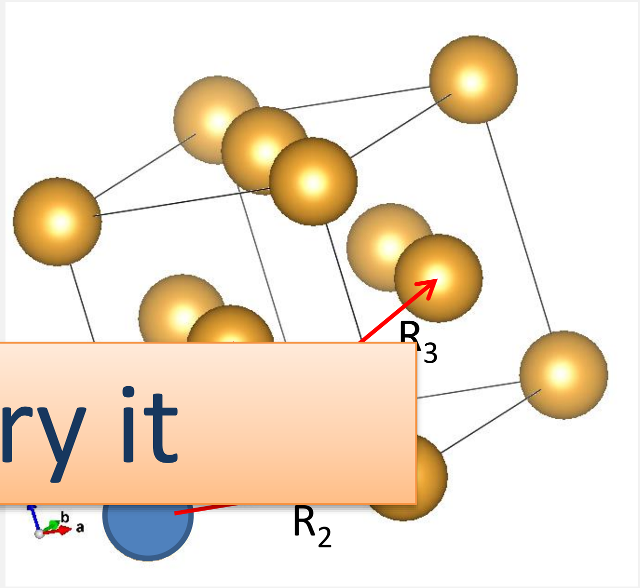
$$R_4 = \cdots. \qquad N_4 = \ldots$$

FT and expected atomic structure

Look at the FT features and compare the structure you expect



R_1 R_2 R_3



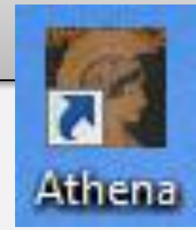
Now you try it

$$R_1 = a/\sqrt{2} \qquad N_1 = 12$$
$$R_2 = a \qquad N_2 = 6$$
$$R_3 = a\sqrt{6}/2 \qquad N_3 = 24$$

Exercises

I. Download Demeter and install it

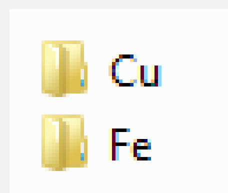
II. Start Athena



Examples and exercises Download **Data** folder

Copper (**Cu**) metal foil

Iron (**Fe**) metal foil



1. Cu K edge XAFS

Basic features

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

Modify extraction parameters

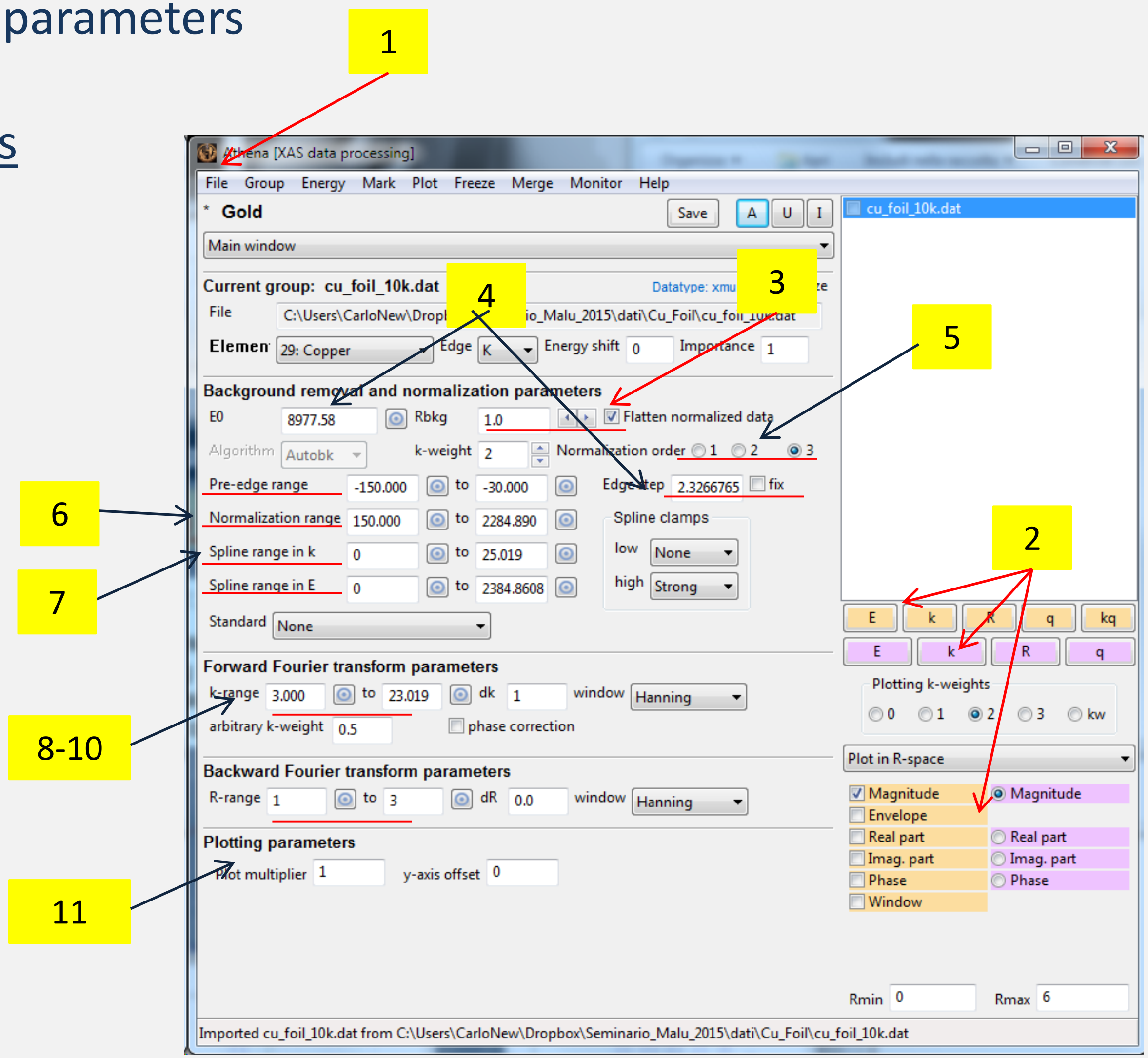
3. R_{bkg}
4. Edge Energy and step
5. Normalization order
6. Normalization range
7. Spline range

k-Weighting

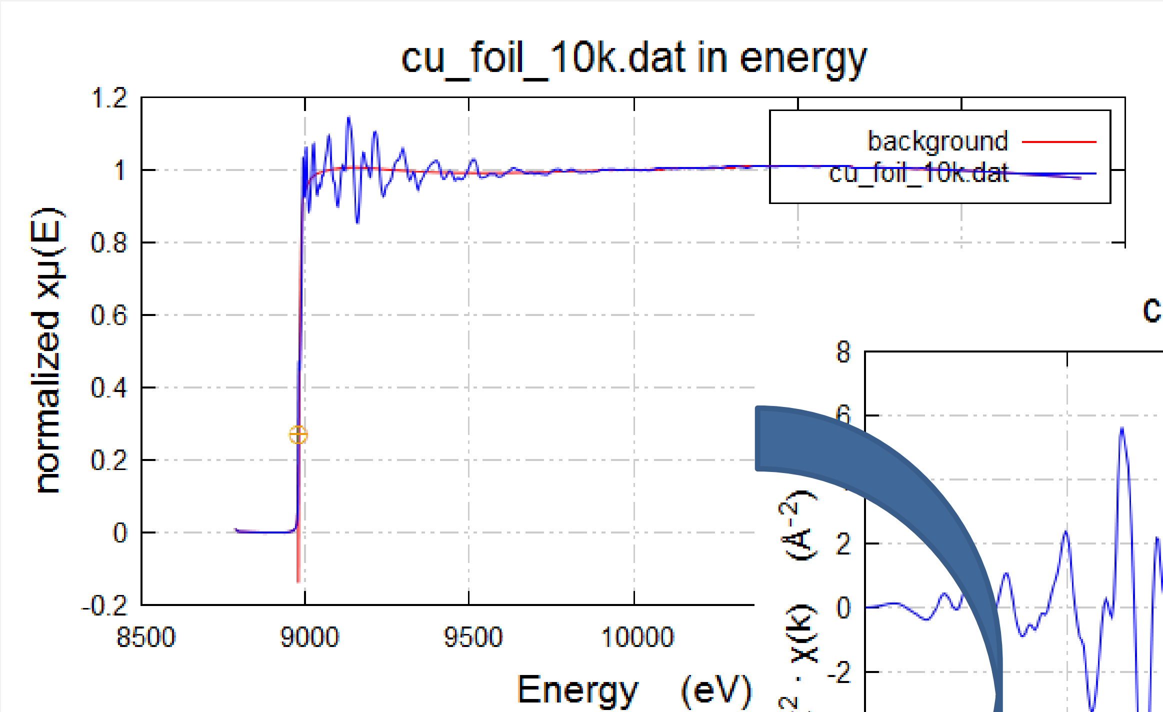
8. FT range
9. FT window
10. FT weight

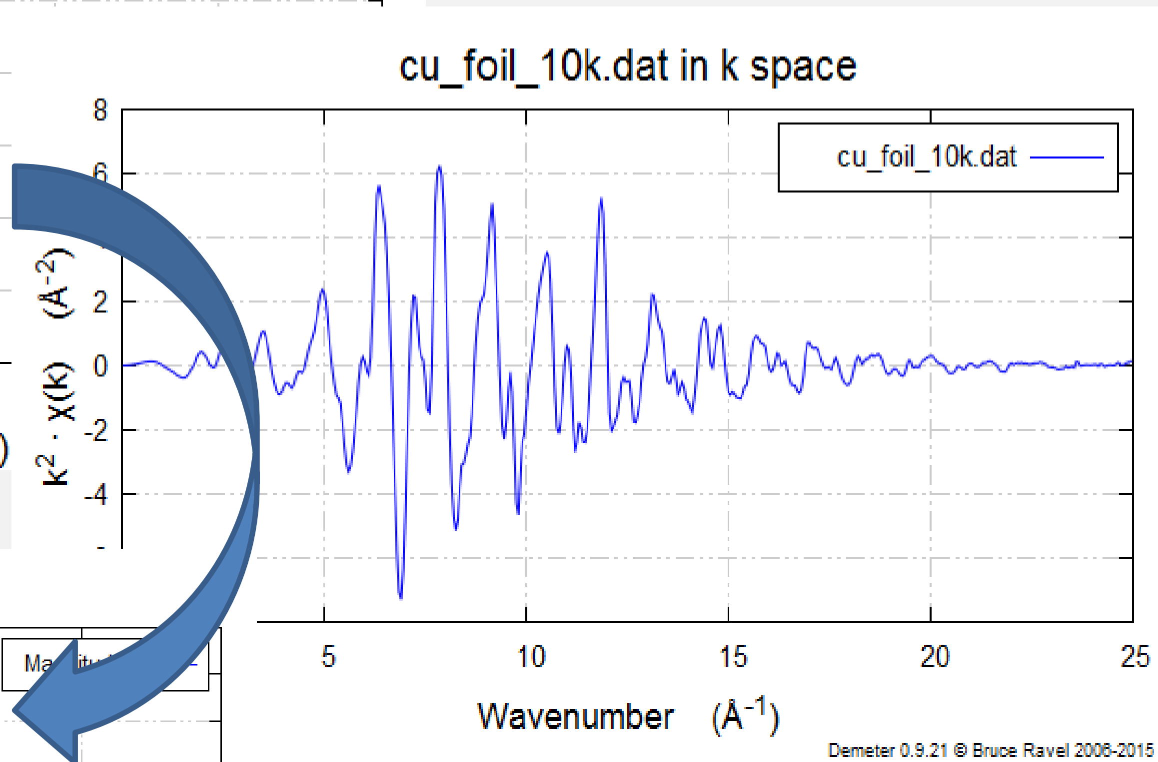
Back Fourier

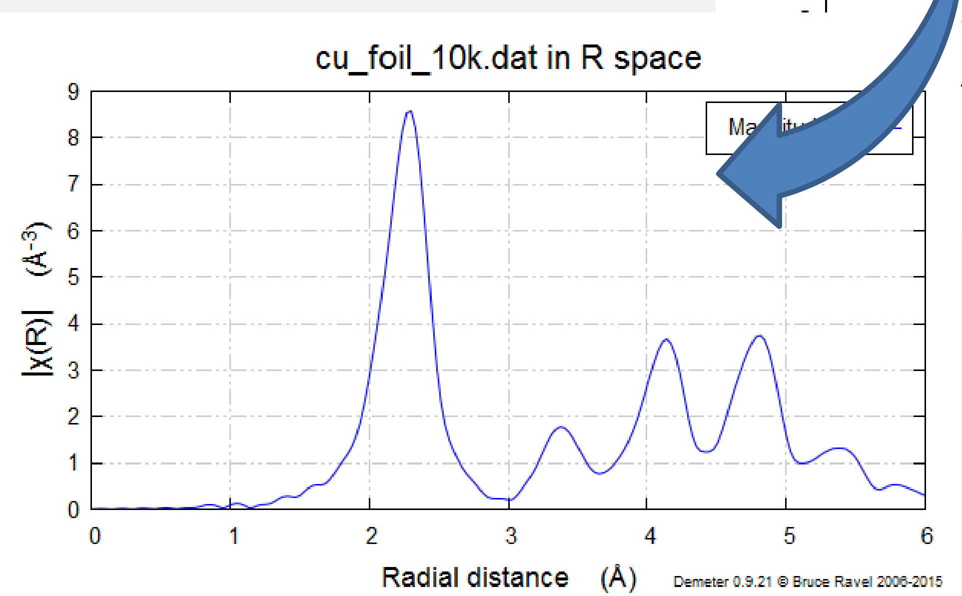
11. Range and window



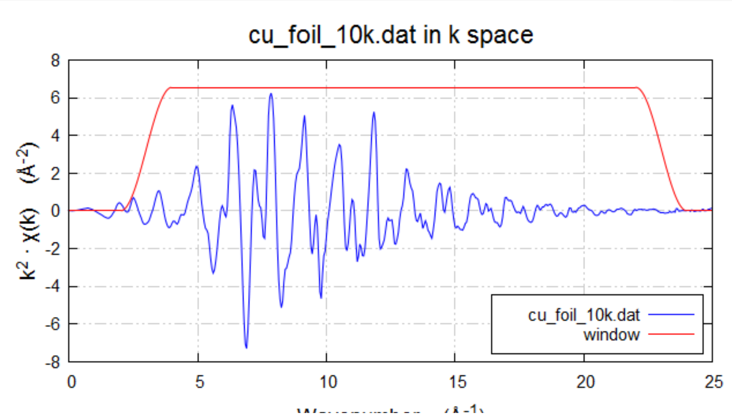
Cu K edge XAFS

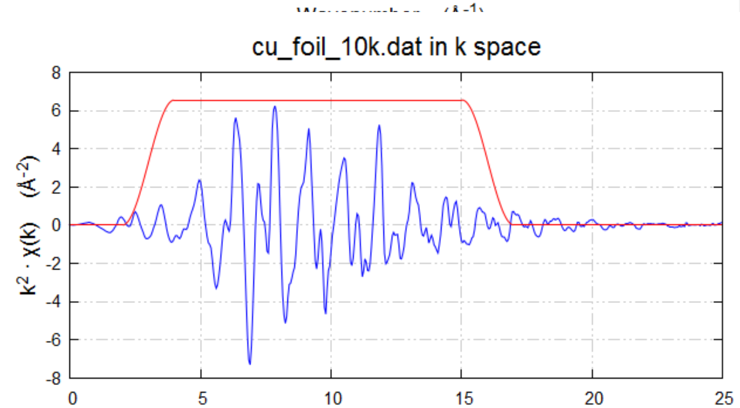


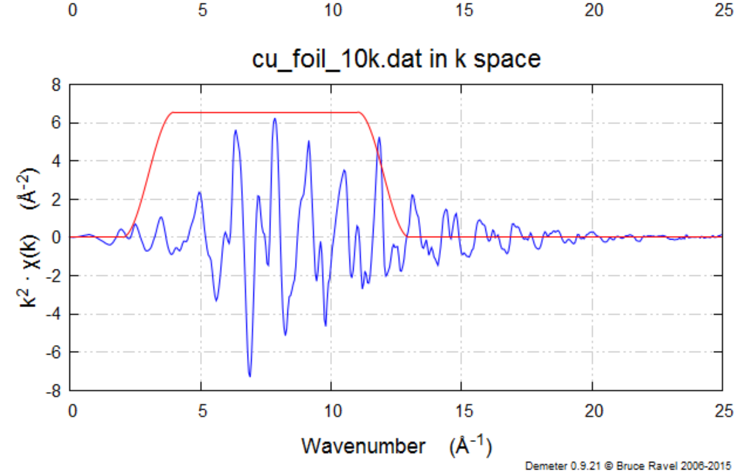




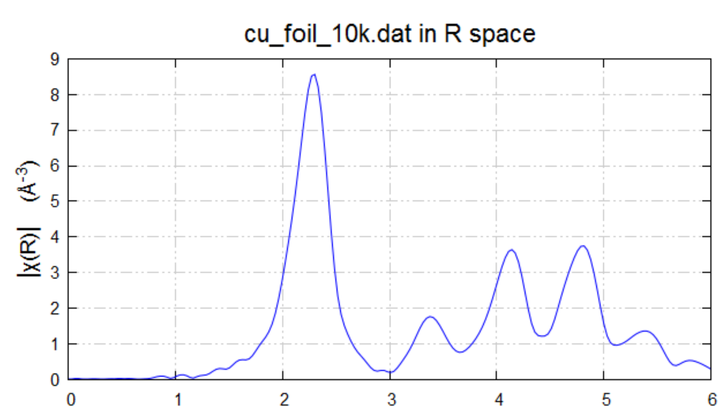
FT window

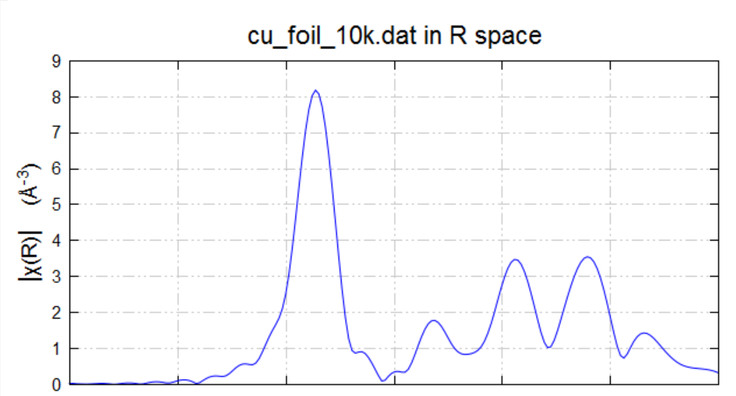


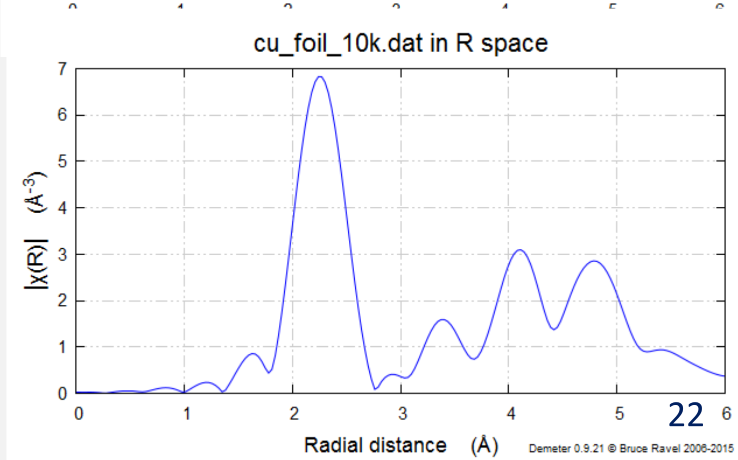




High quality data: main FT features does not change with windows

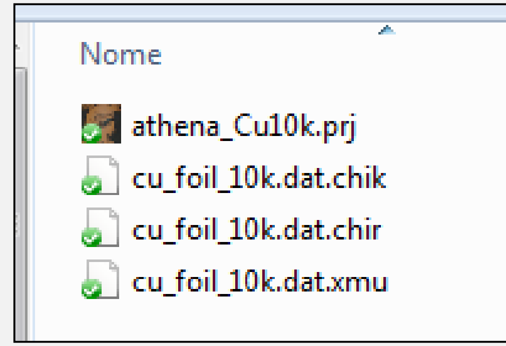


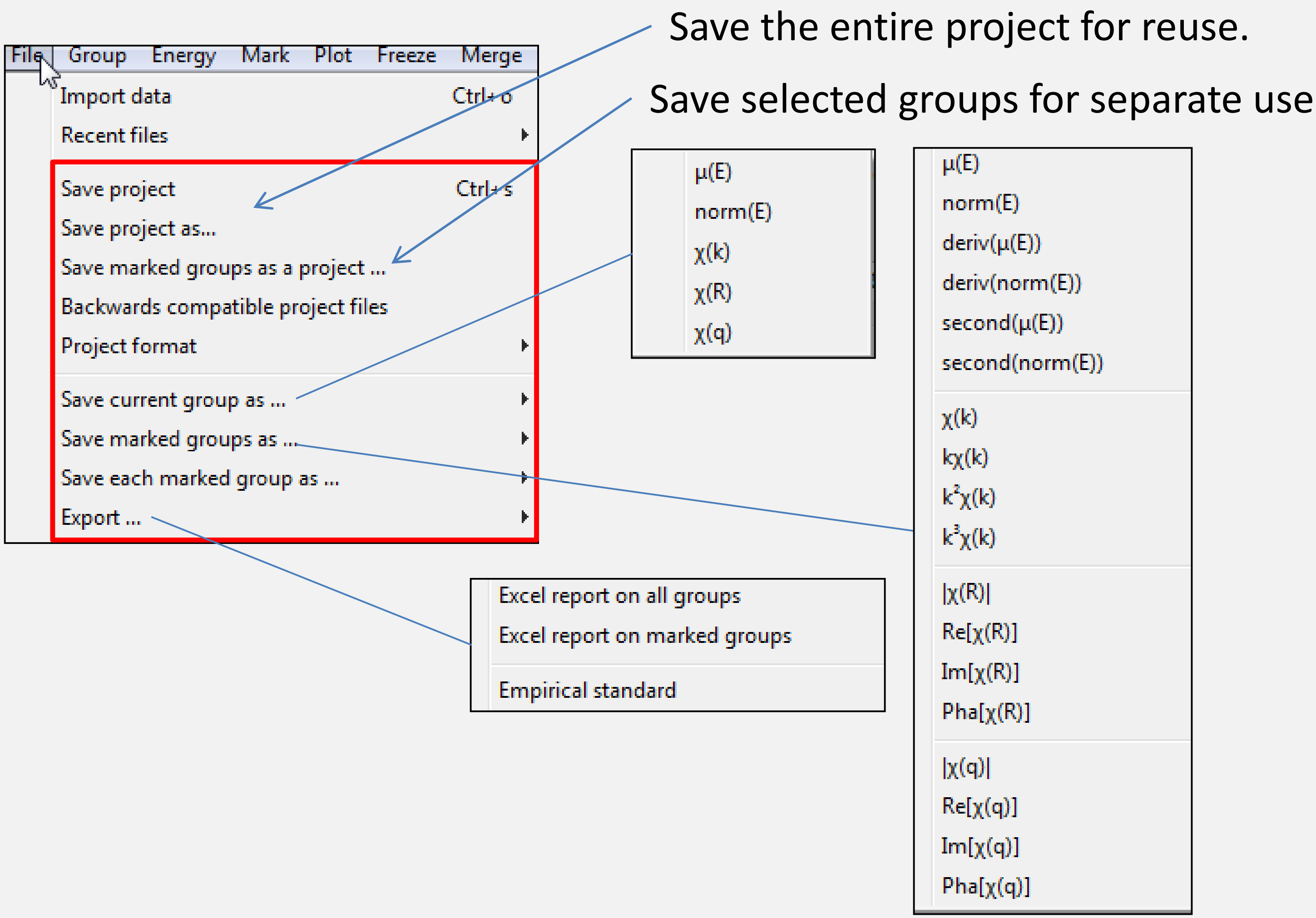




Note:

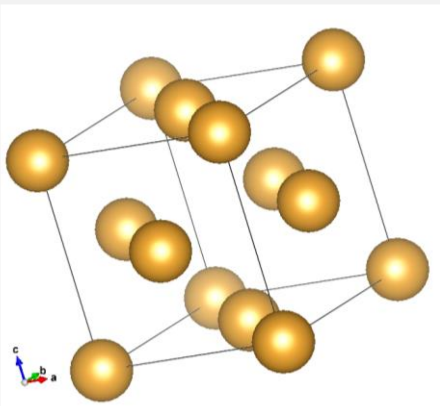
Always save data and project for future use you have many options!





3. Cu (fcc) and Fe (bcc) XAFS

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



fcc (Fm-3m)

$R_1 = a/\sqrt{2}$ $N_1 = 12$

$R_2 = a$ $N_2 = 6$

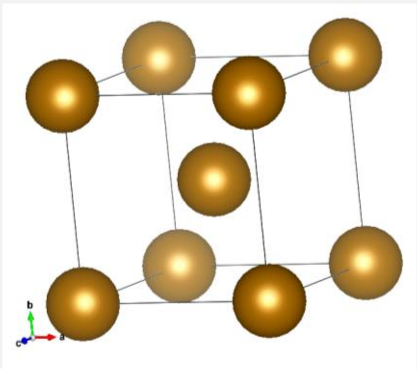
$R_3 = a\sqrt{6}/2$ $N_3 = 24$

Bcc (Im3m)

$R_1 = a\sqrt{3}/2$ $N_1 = 8$

$R_2 = a$ $N_2 = 6$

$R_2 = a\sqrt{2}$ $N_3 = 12$



a_{Cu}= 3.61 A

a_{Fe}=2.86 A