INTRODUCTORY COURSE
Synchrotron EXAFS & XANES techniques for Chemical Speciation on Environmental Systems

VIPER for EXAFS data treatment

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Cerdanyola del Vallès 09.10.14

Program

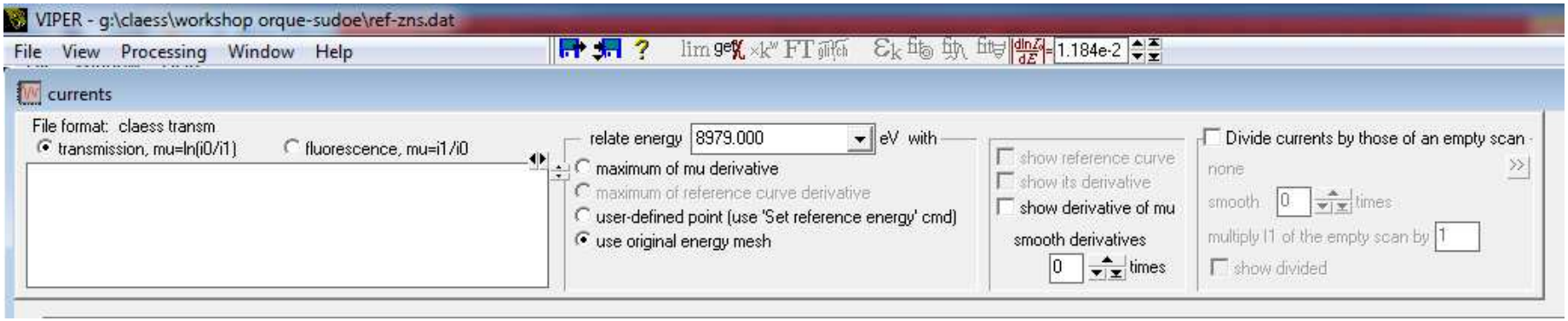
Grup de Tècniques de Separació en Química
UAB

VIPER:

- Author: Konstantin Klementiev
- Version: 26 Feb 2013, build 1109

Visual Processing in EXAFS researches

Program interface:



Steps to follow for data analysis

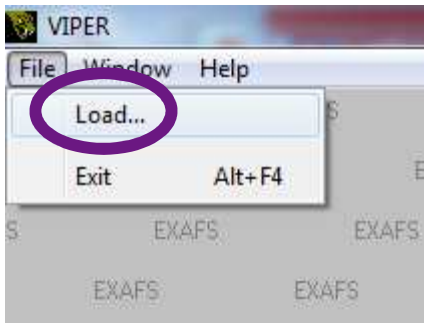
ALBA

Grup de Tècniques de Separació en Química UAB

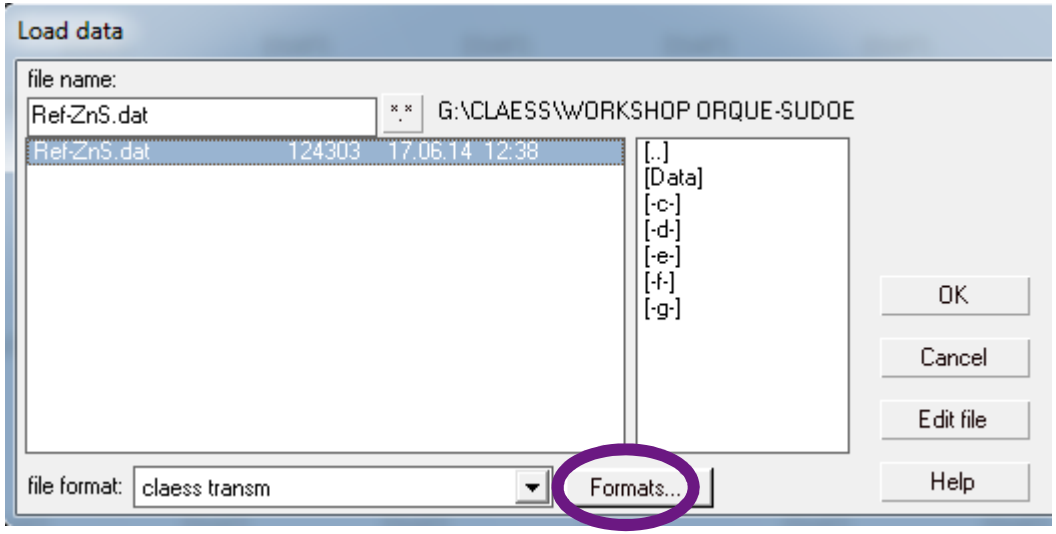
1. Load files
    - 1.1 Choose correct data format
    - 1.2 Choose transmission or fluorescence
    - 1.3 Energy Calibration
2. Get χ
3. Configure line colour
4. Deglitch
5. Set E_0
6. Subtract Pre-edge background
7. Set Kmin and Kmax
8. Normalize Post-edge
9. Combine repetitions
10. Back Fourier Transform
11. Fit
    - 11.1 Create input file with Atoms
    - 11.2 Create paths with feff6
    - 11.3 Load path
    - 11.4 Add more shells if needed
    - 11.5 Start
    - 11.6 Change S_0^2 if needed
    - 11.7 Add constraints
    - 11.8 Start Again
12. Get the error of the fit
13. Save

1. Loading files

- Select the file

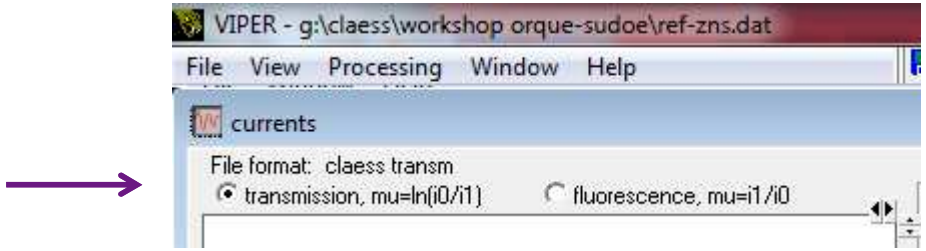


- Choose correct data format

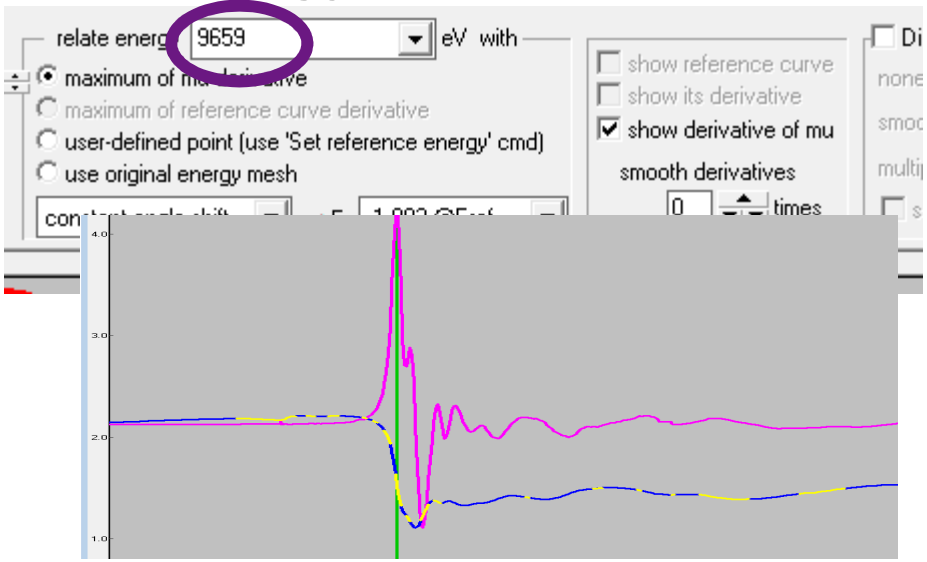


1. Loading files

- Choose transmission or fluorescence



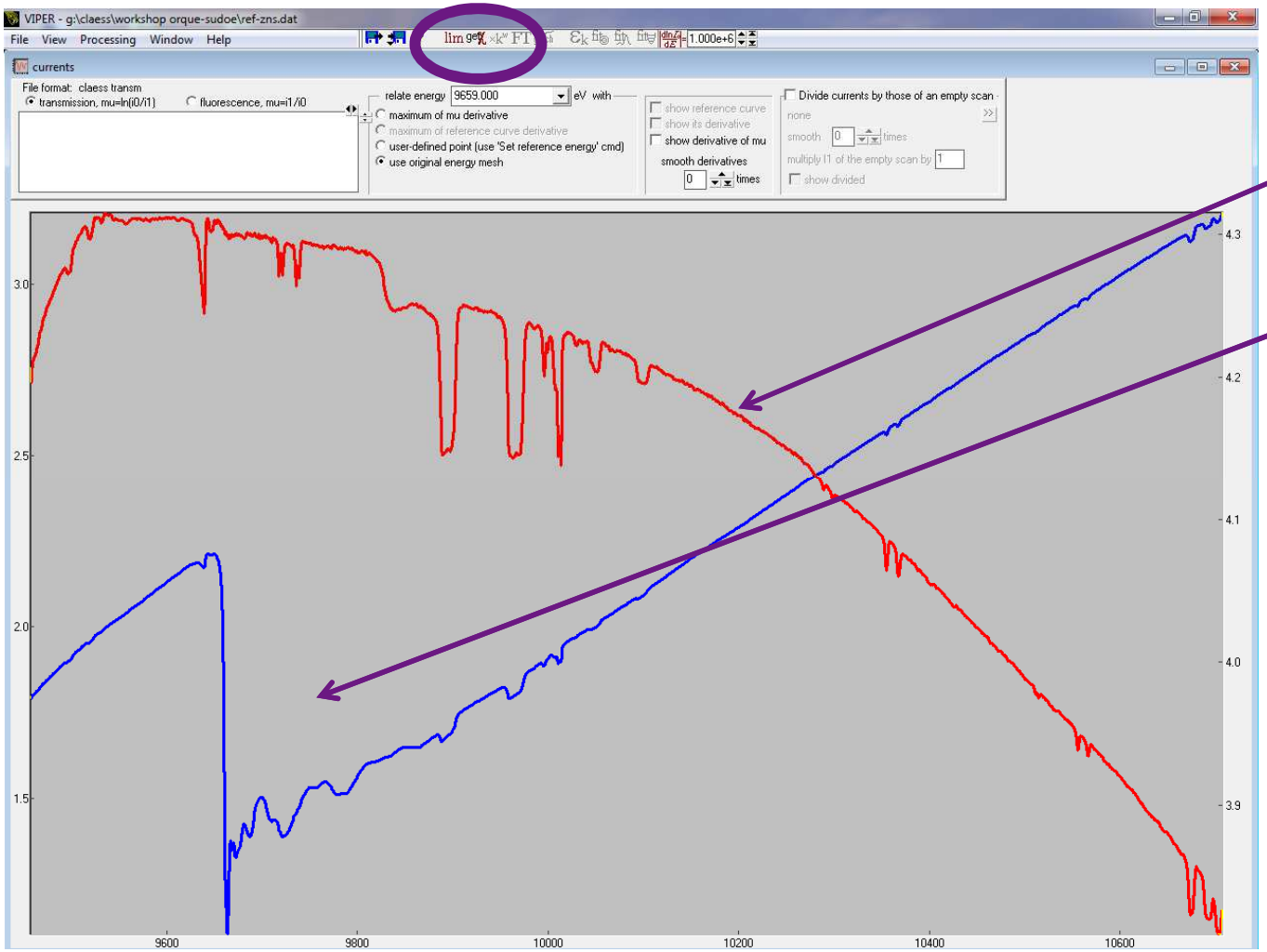
- Energy calibration



Different ways:

- Selecting the theoretical energy for the element
- Selecting the maximum of the μ derivative
- Using a foil as a reference (before I_2)
- Selecting a point in the spectra

2. Get χ



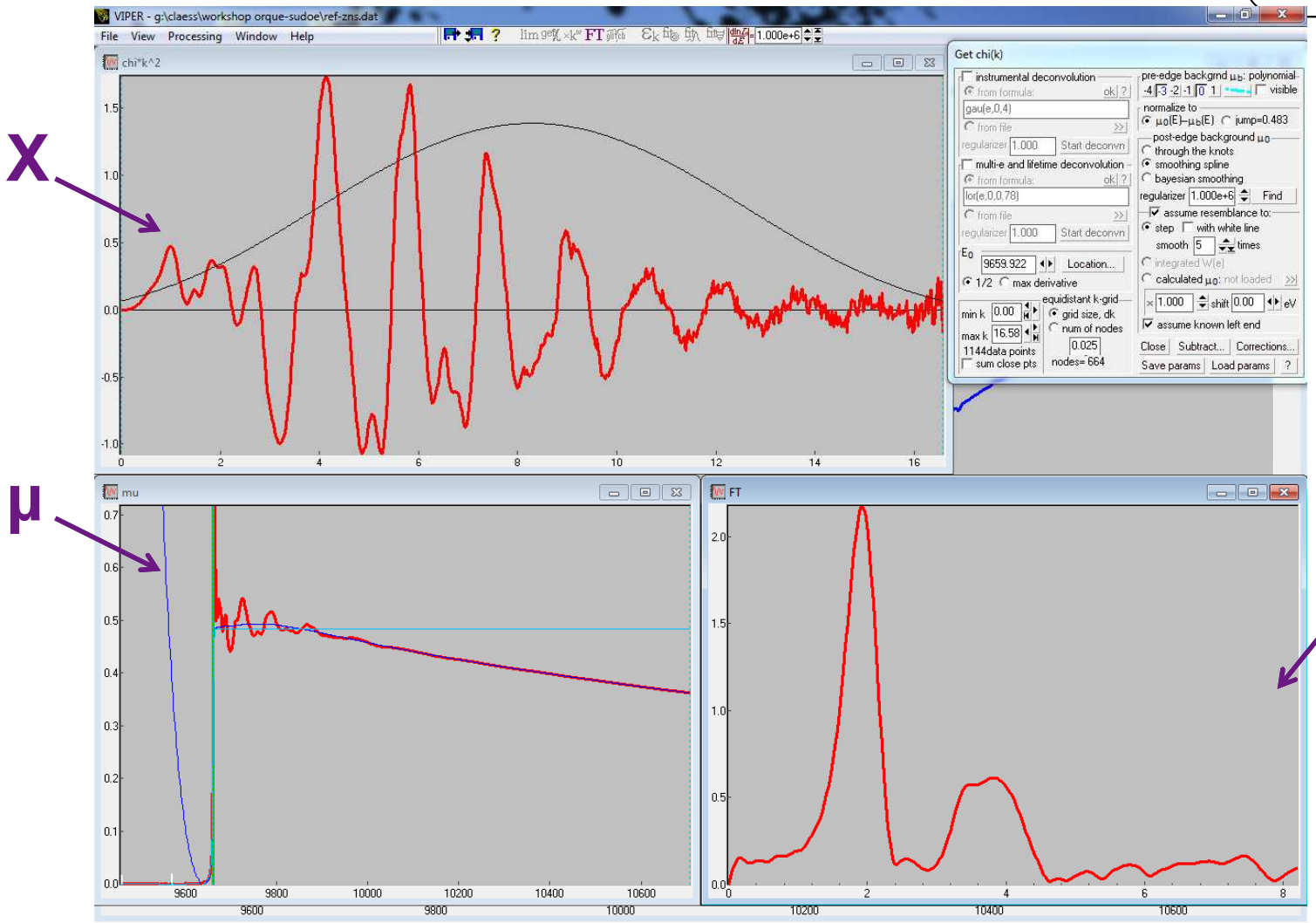
I_0

I_1

To see μ spectra

Press button:

2. Get χ


VIPER - g:\claess\workshop orque-sudoe\ref-zns.dat
File View Processing Window Help
chi*k^2
Get chi(k)
instrumental deconvolution
multi-e and lifetime deconvolution
pre-edge backgrnd μb: polynomial
normalize to
post-edge background μ0
through the knots
smoothing spline
bayesian smoothing
assume resemblance to:
step
with white line
integrated W(e)
calculated μ0: not loaded
assume known left end
Close
Subtract...
Corrections...
Save params
Load params
mu
FT
χ
μ
FT

2. Get χ

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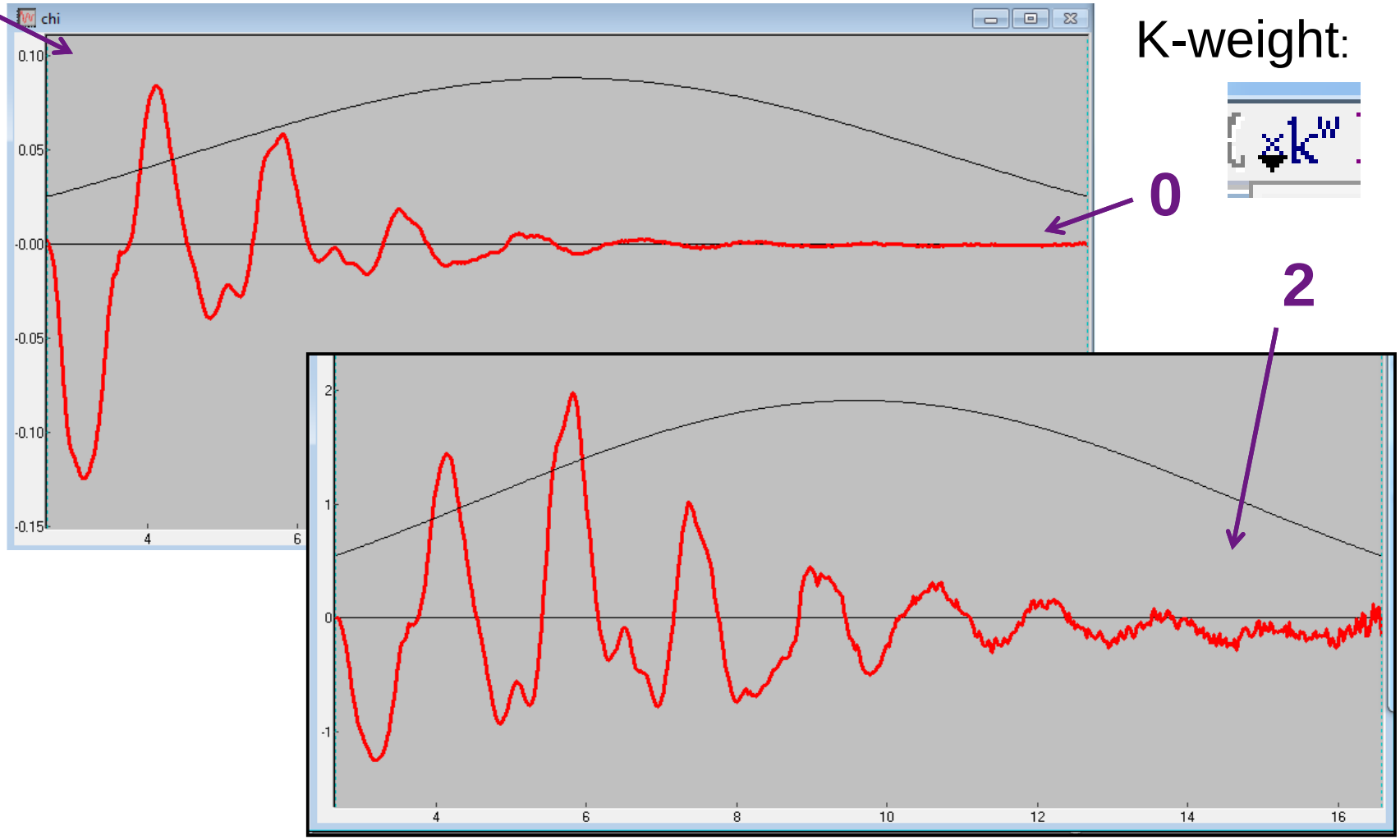
χ

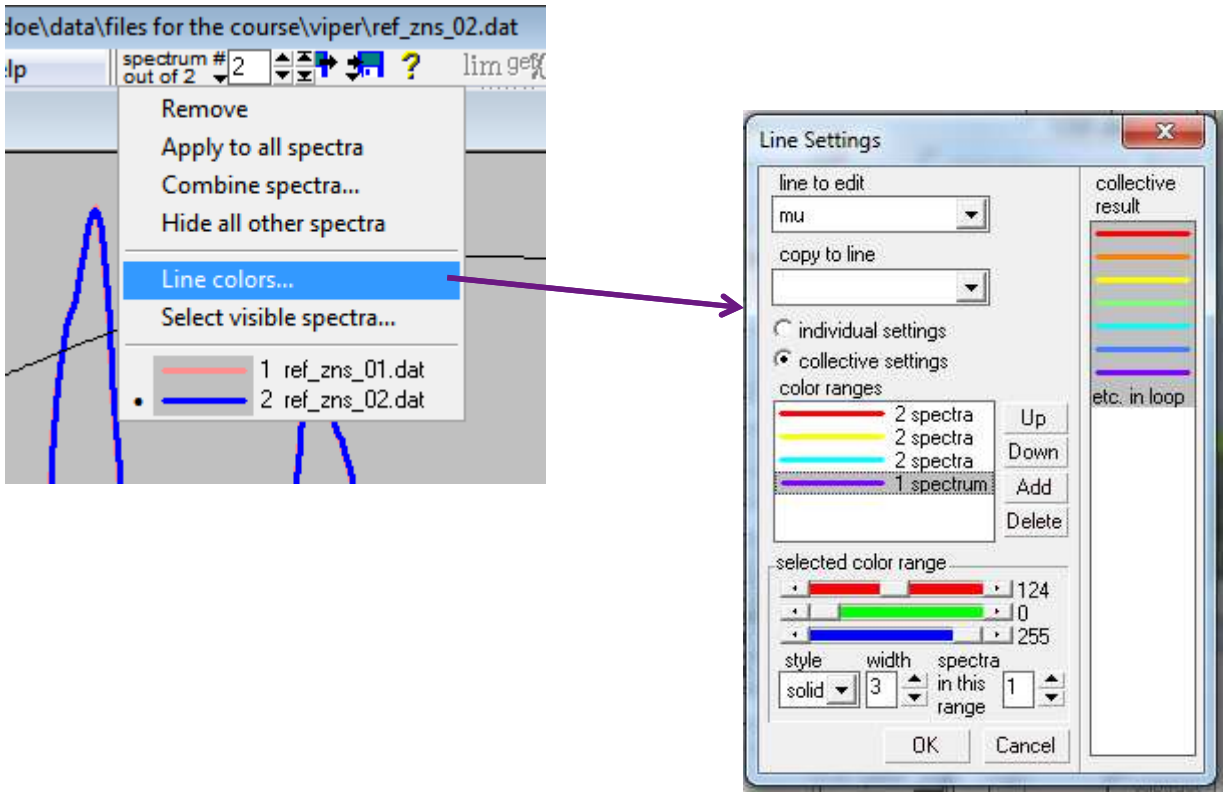


K-weight:

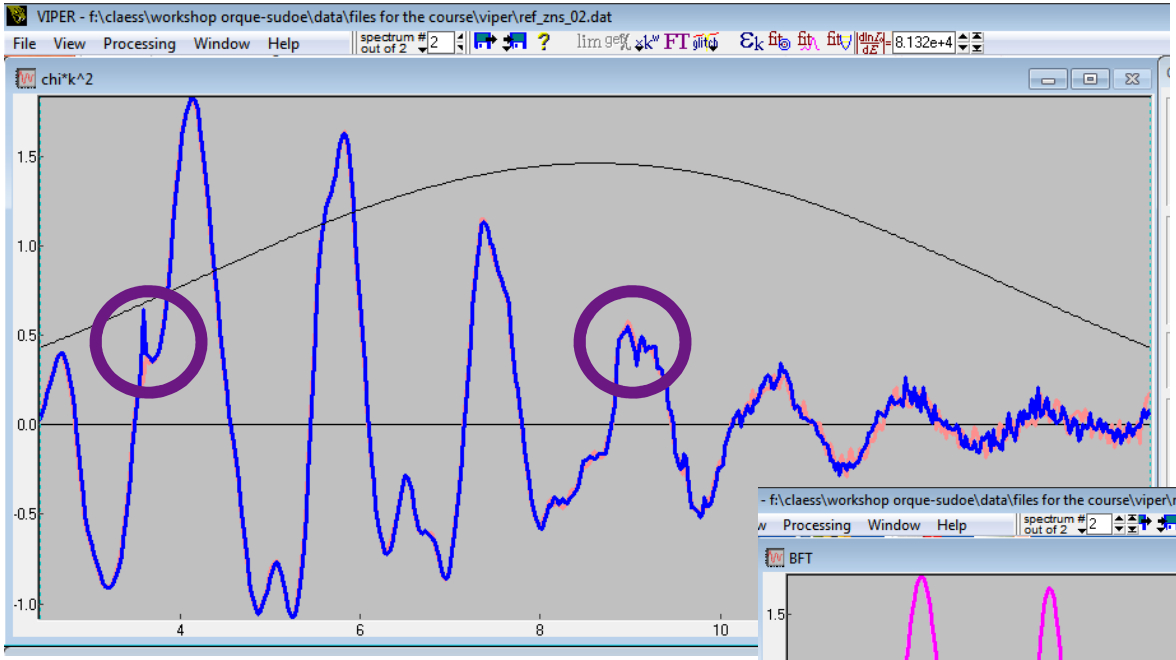
0

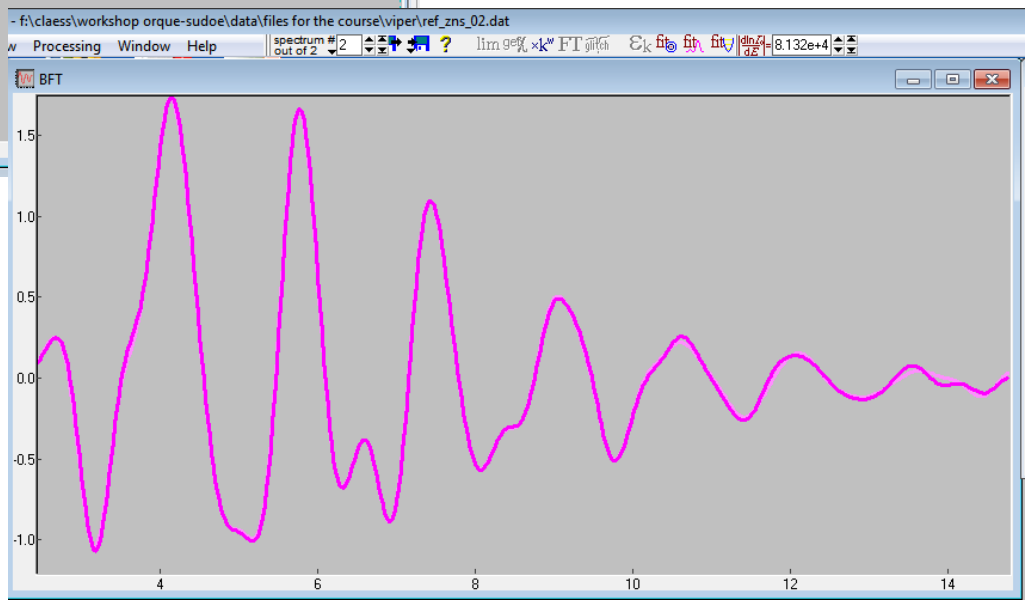
2

3. Line Colour



4. Deglitch

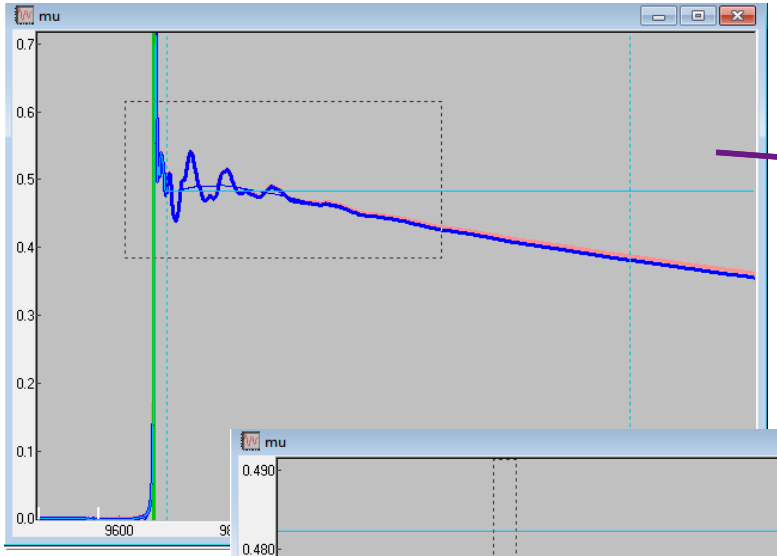




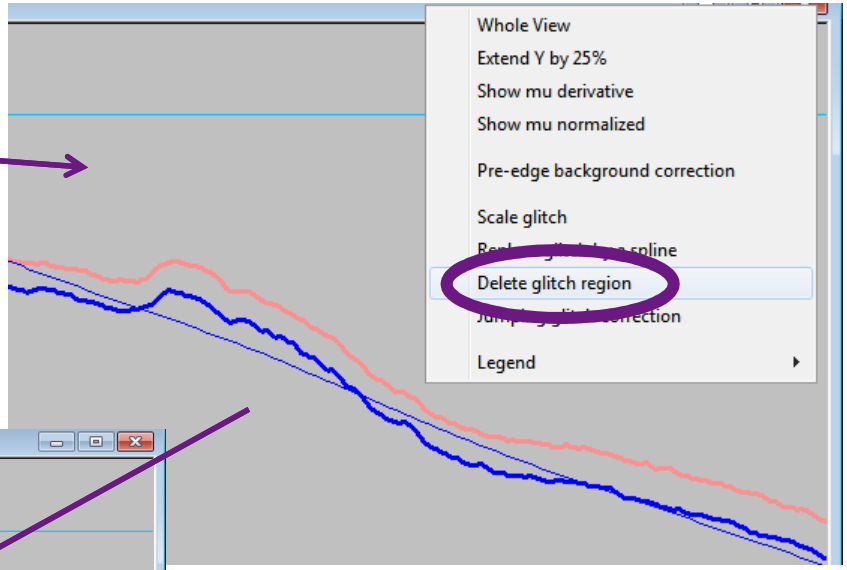
- Glitch: Abrupt change in intensity due to the monochromator
- They can be removed

4. Deglitch

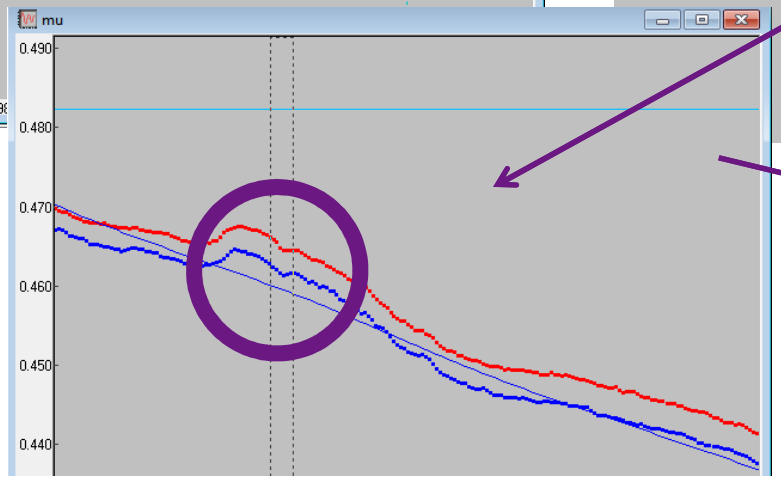
- Zoom



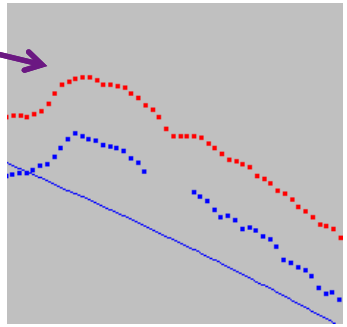
- Right click→ delete glicht region



- Select region by dragging

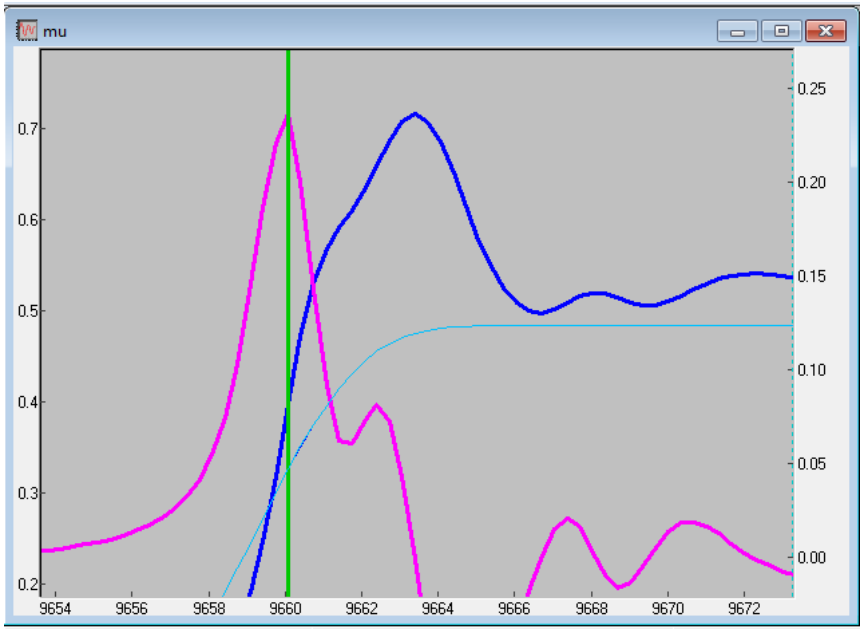


- Delete points



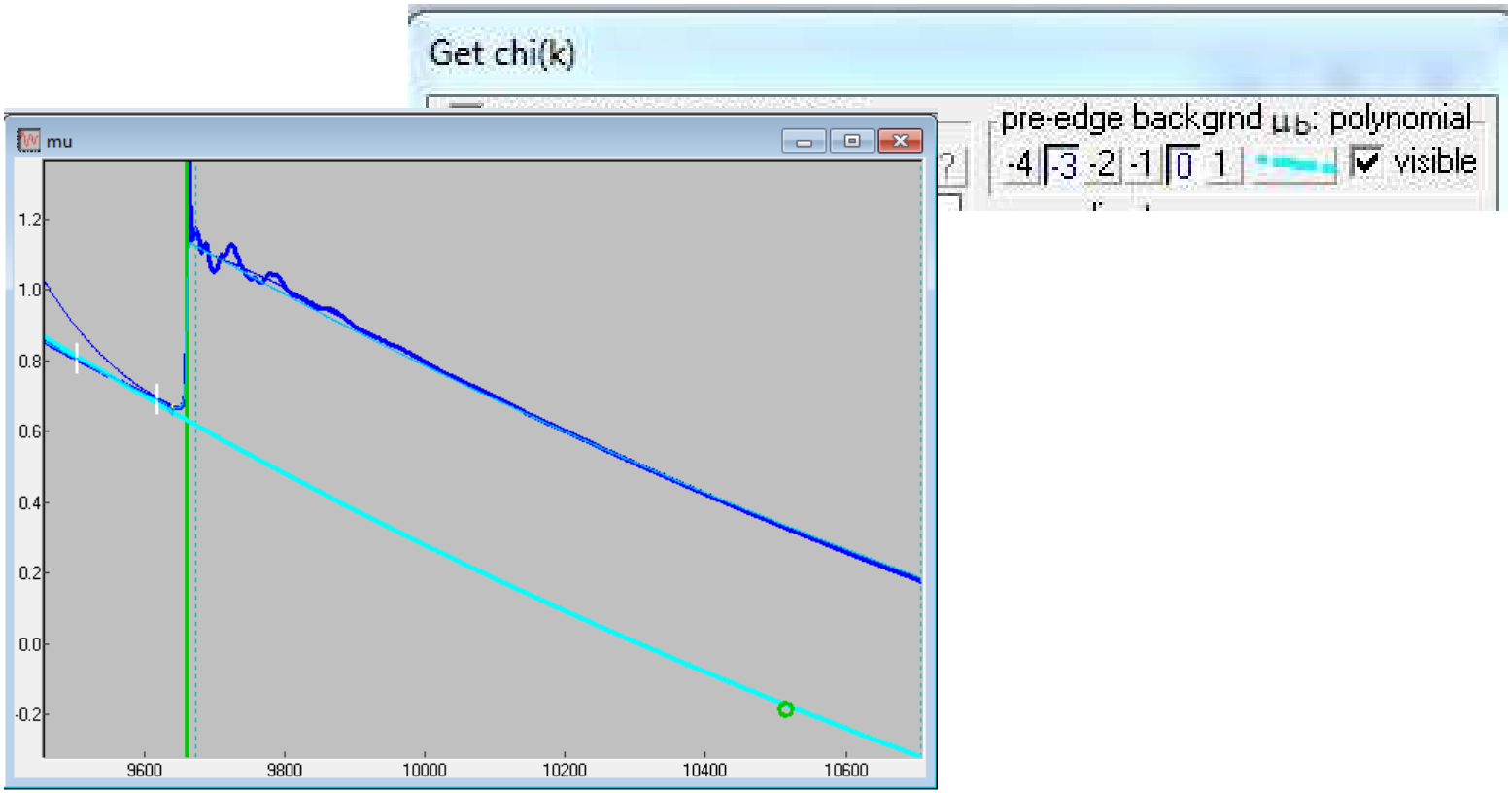
5. Set E_o

- On μ window→ right click → show μ derivative
- Place the green line on top of the first peak of the pink derivative



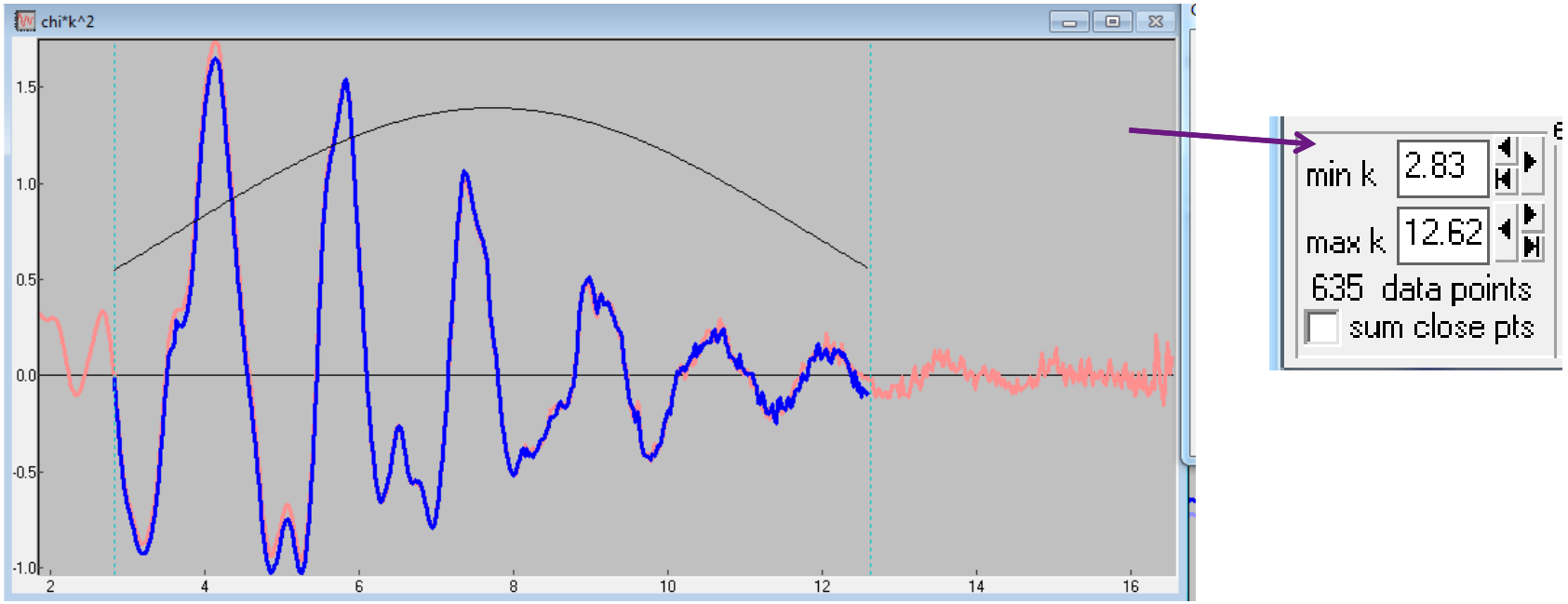
6. Subtract Pre-edge

- Subtract background selecting a region in the pre-edge and drawing polynomial line through it



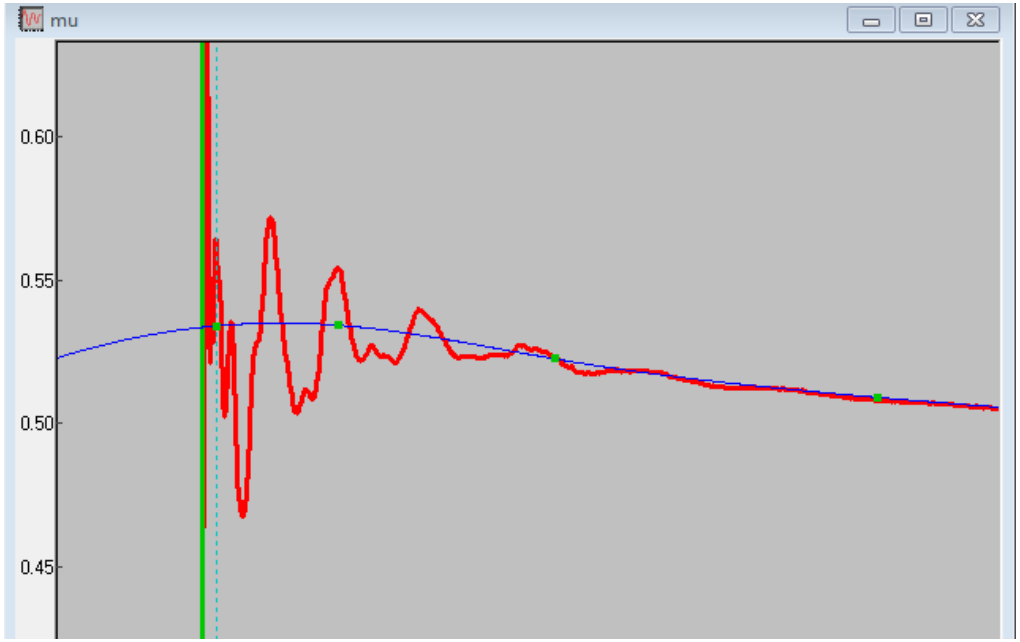
7. Set Kmin + Kmax

- Seen in both μ and χ as green dotted lines
- Set them where χ=0 and oscillations are visible over noise



8. Post-edge

- Get a straight line to avoid introducing artificial oscillations



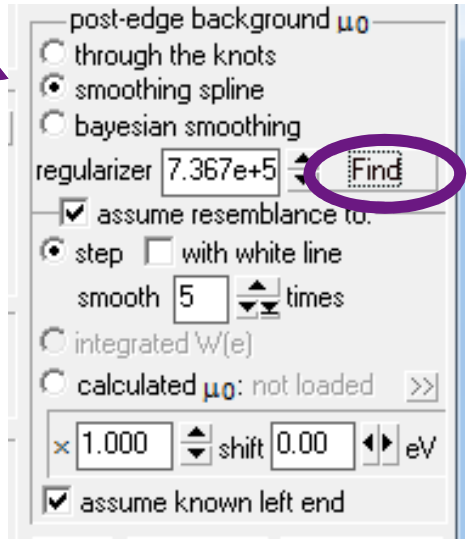
3 ways:

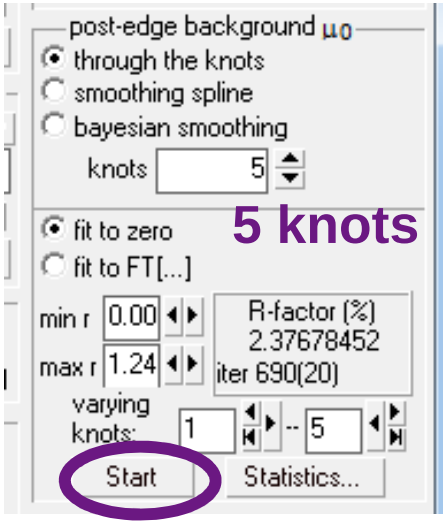
- Through the knots
- Smoothing spline
- Bayesian smoothing

8. Post-edge

3 ways:

- Through the knots
- Smoothing spline
- Bayesian smoothing

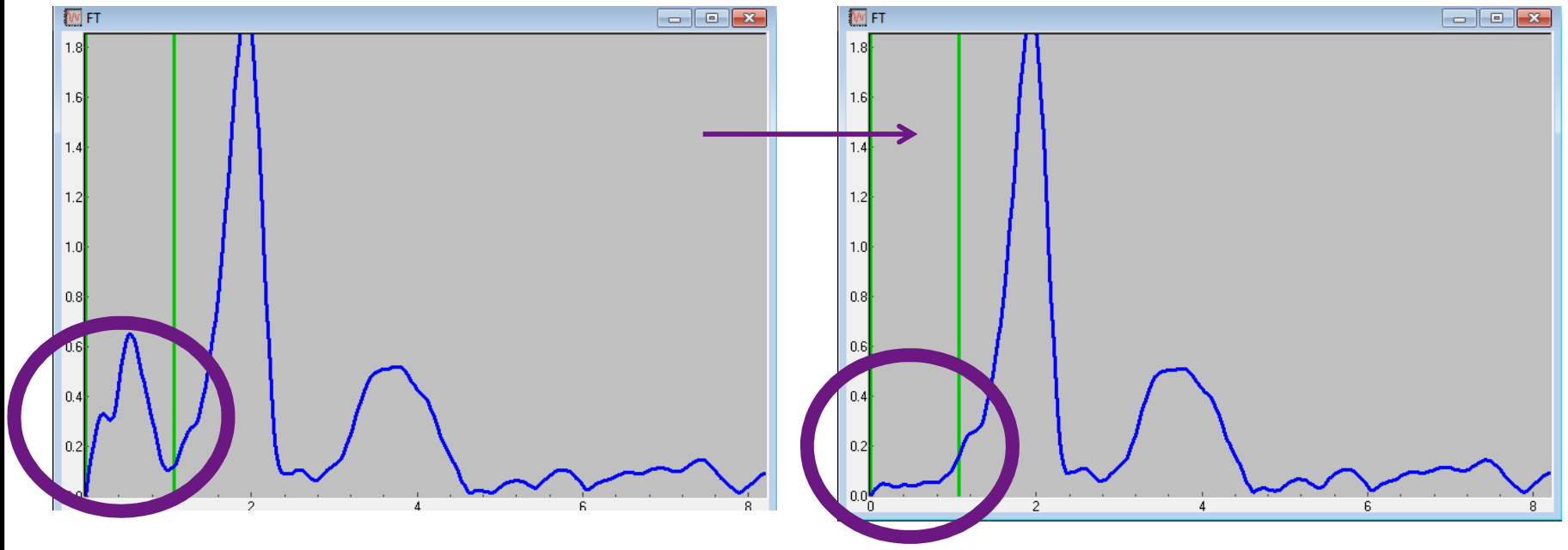




5 knots

8. Post-edge

- In the FT, minimize signal before the first shell



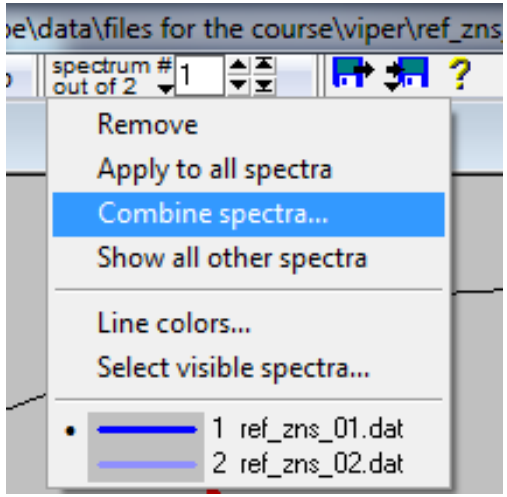
9. Combine spectra

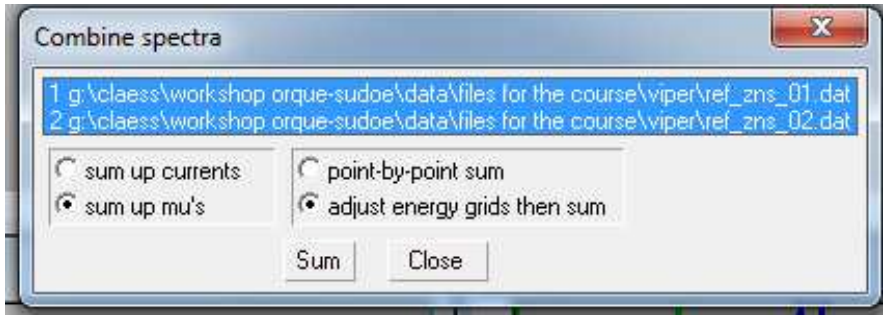
- 1 scan, different channels of the detector

  →Sum up currents+ point-by-point sum

- Different scans, 1 sample

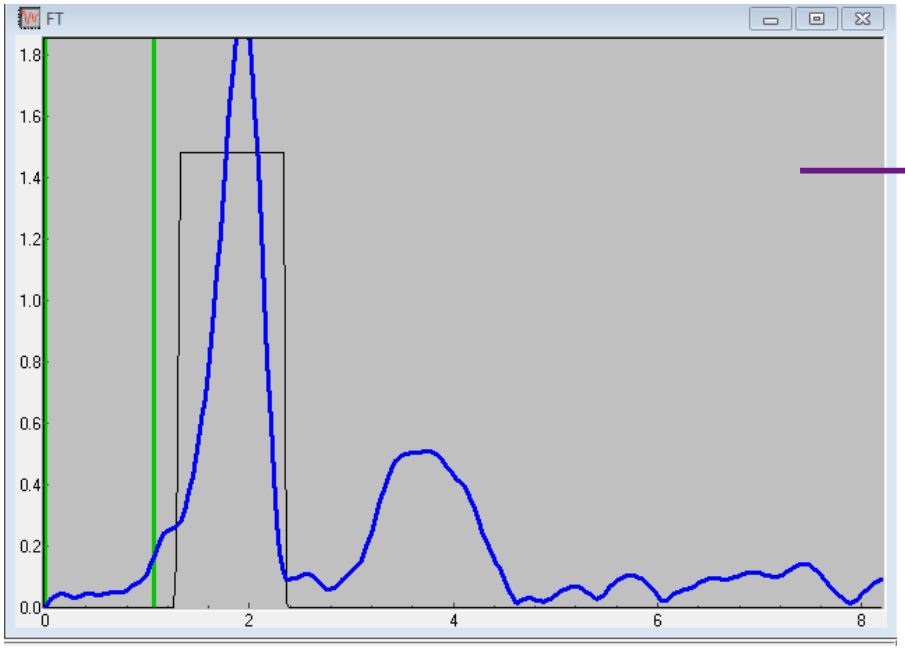
  → Sum up mu's + adjust energy grids then sum

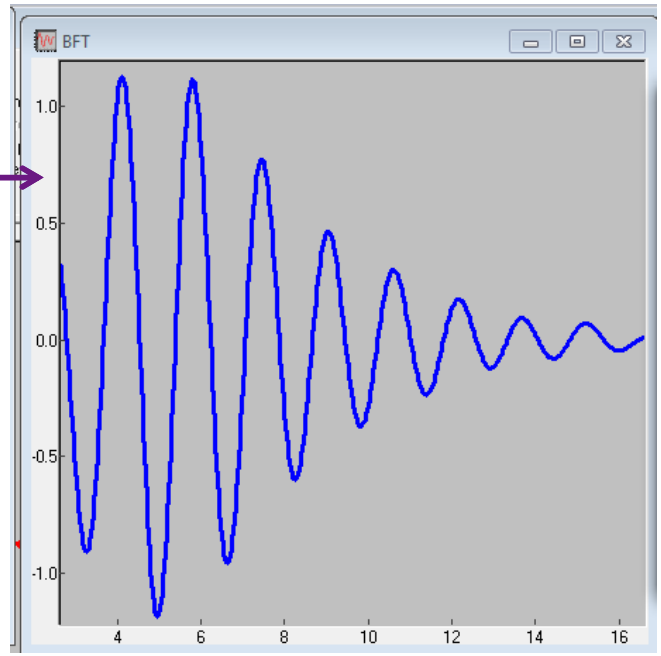




10. Back Fourier

- Press 
- Select the first peak in the Fourier Transform window
- The BFT frequencies must be similar to the ones in χ





11. Fit

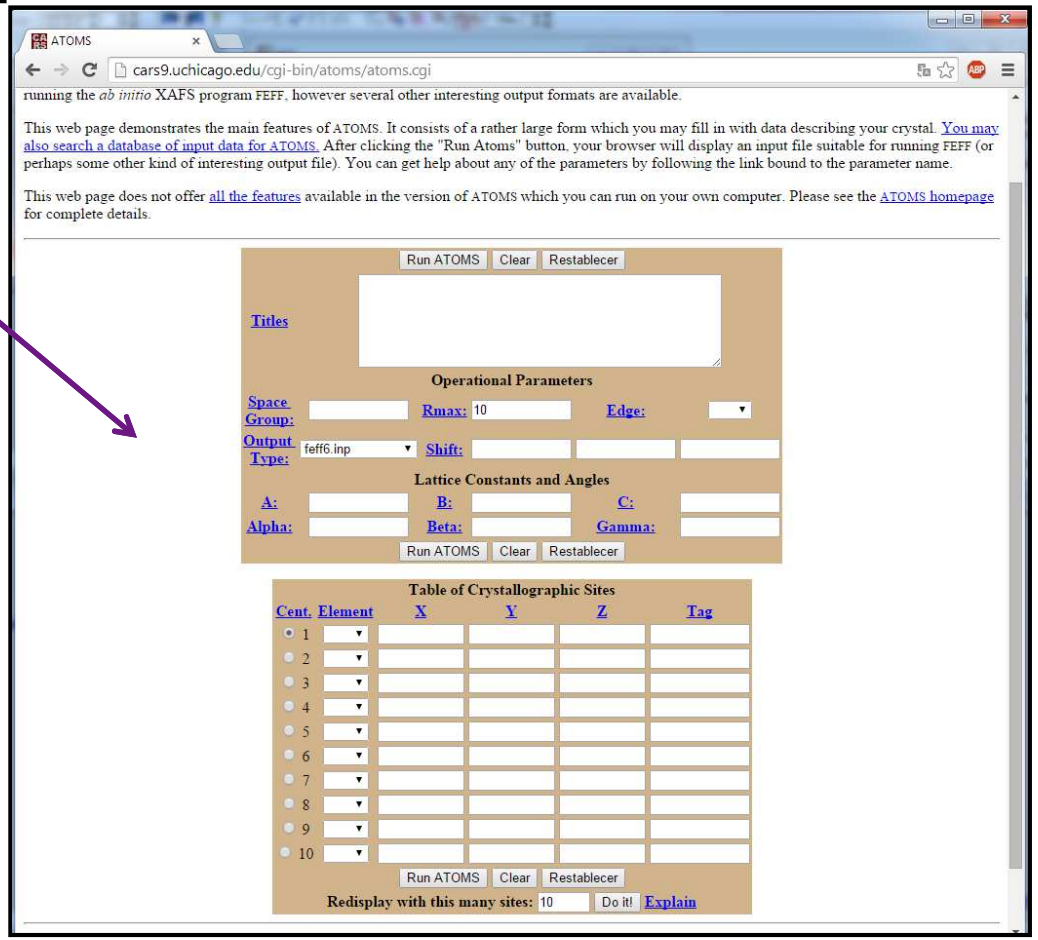
11.1 Create input file with Atoms

- Find the crystal structure data of the compound
- Several databases online for free:
  - Web Atoms
  - American Mineralogist Crystal Structure Database (AMCSD)
  - Crystallography Open Database (COD)
- Paid databases:
  - Inorganic Crystallographic Structure Database (ICSD)
  - etc

11. Fit

11.1 Create input file with Atoms

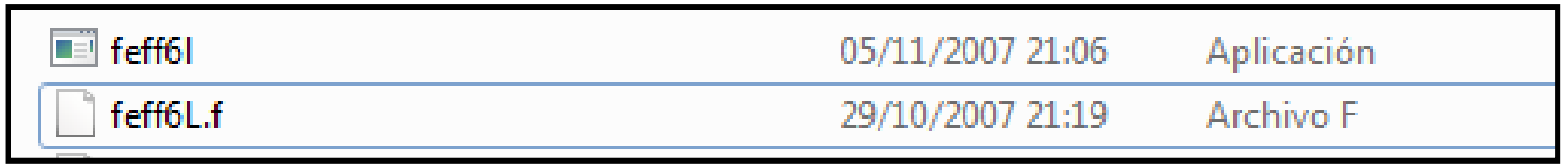
- Web atoms
  (google it)
- Fill it
- Run



11. Fit

11.2 Create paths with feff6

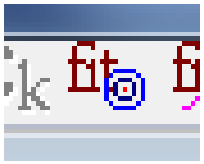
- Copy-paste the data atoms provide in a notepad
- Save it as feff.inp inside a labeled folder
- Copy in the same folder the files:

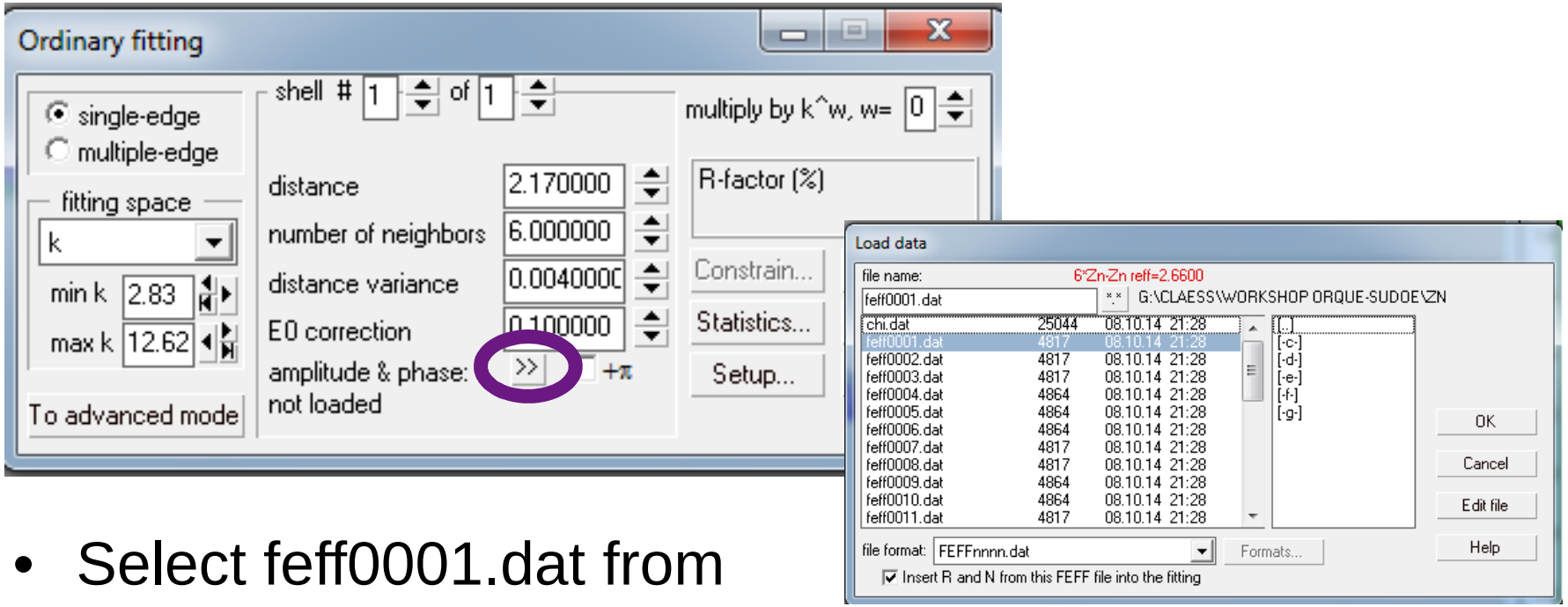


- Double click on the application
- It will run and create new files, called paths in the same folder

11. Fit

11.3 Load path

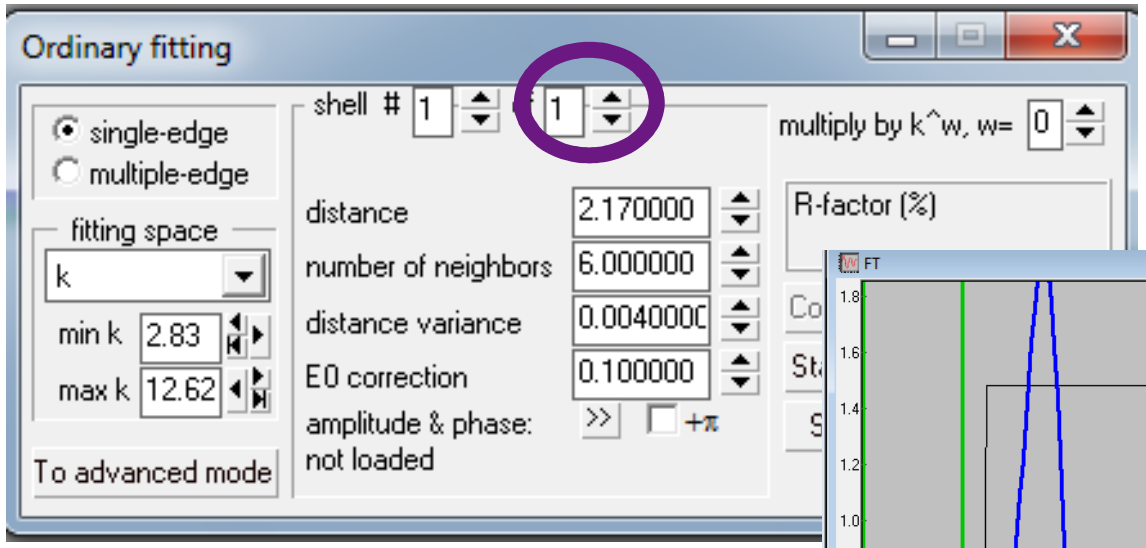
- In the BFT window press: 
- New window:

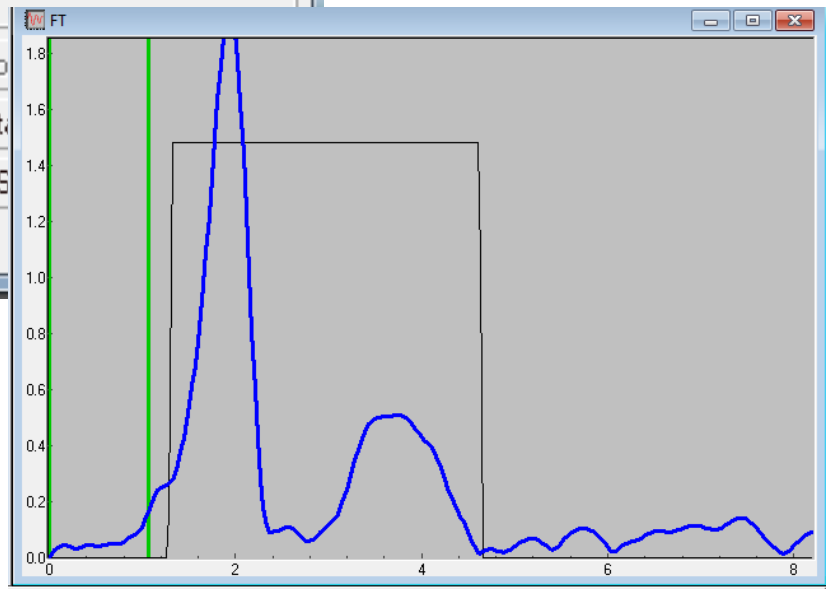


- Select feff0001.dat from

11. Fit

11.4 Add more shells if needed

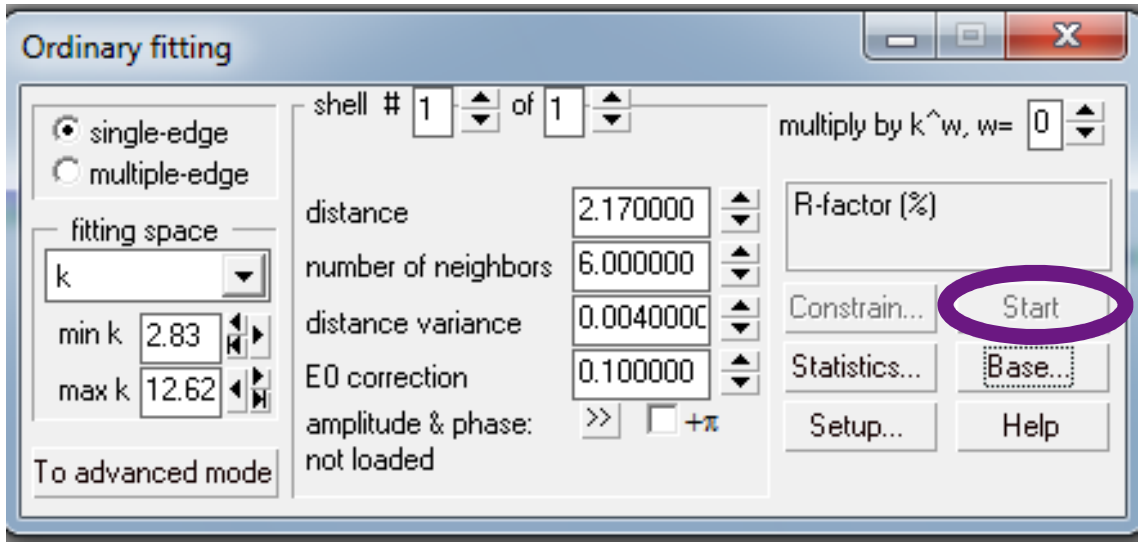




1 shell=
1 coordination sphere

11. Fit

11.5 Start



Distance (r) = bond leght
Number of neighbors (n) = coordination number
Distance variance (σ^2) =Debye-Waller Factor
E0 correction (e) = energy shift

11. Fit

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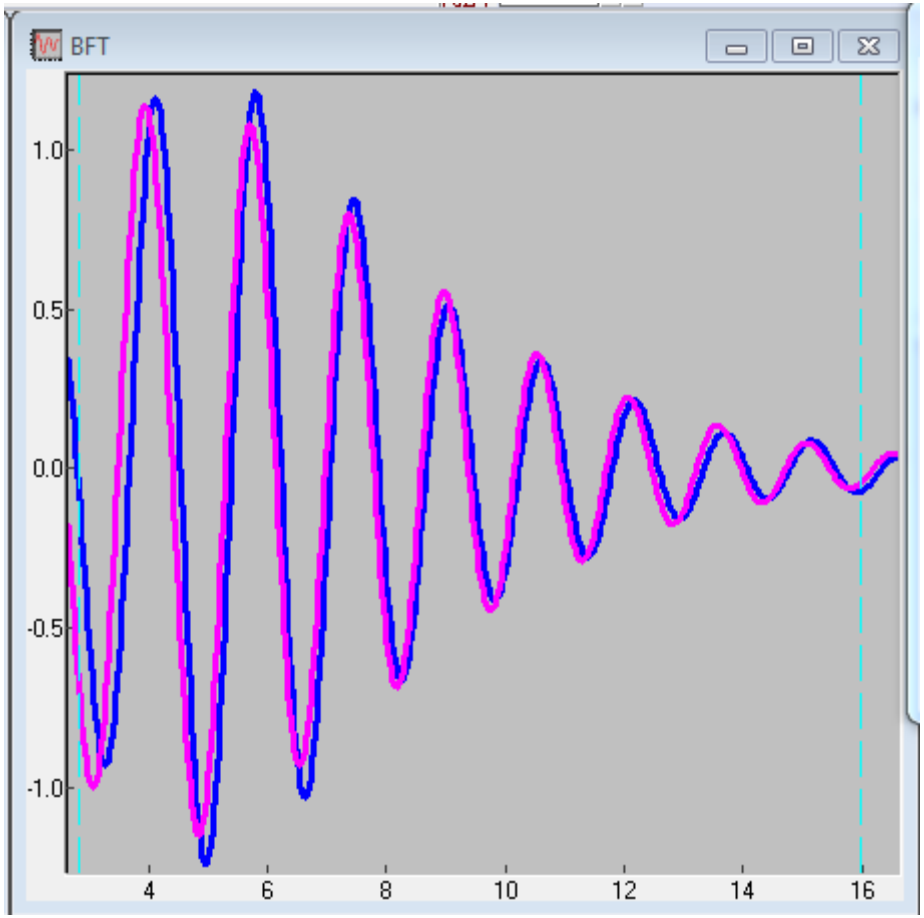
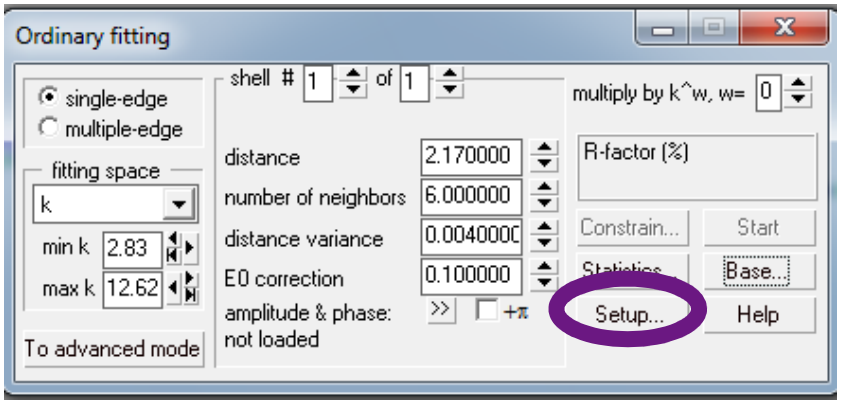
11.5 Start

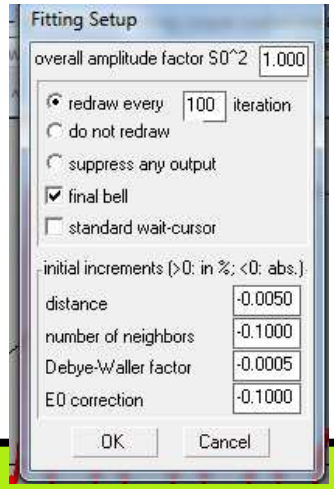


11. Fit

11.6 Change S_0^2 if needed

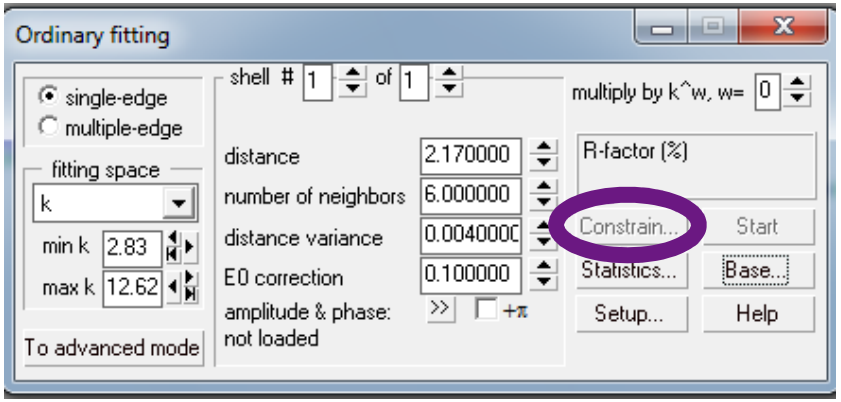
- Sometimes the obtained coordination number (n) is lower than it should
- You need to change the amplitud, S_0^2
- Divide: $\frac{obtained\ n}{theoric\ n}$
- Use the result as the new S_0^2 , change it in Setup





11. Fit

11.7 Add constraints if needed



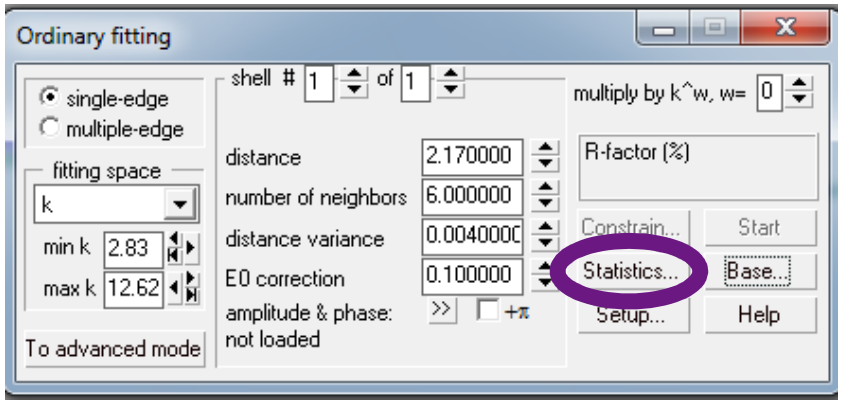
For example:
If you fit two shells use
e2=e1

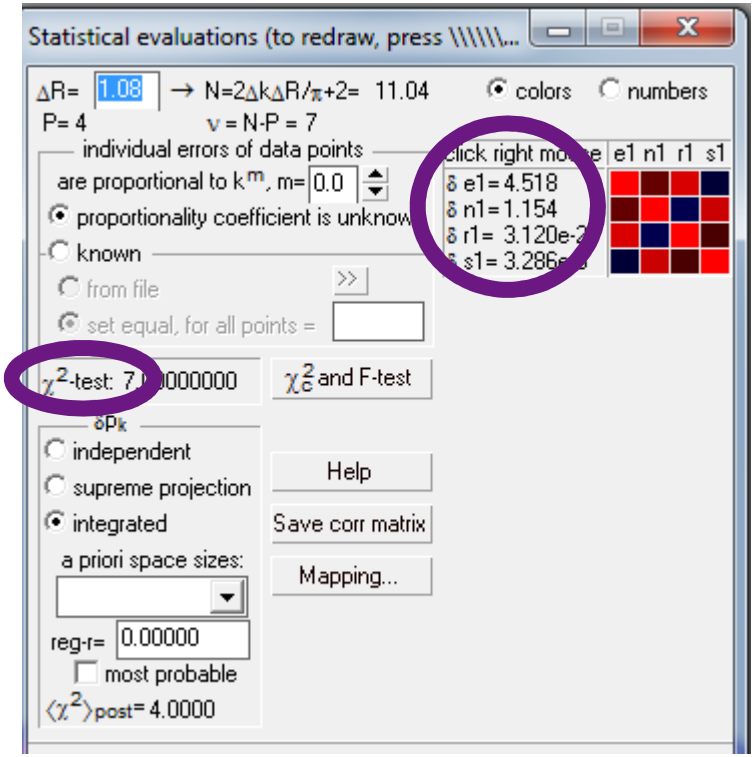
11.8 Start again the fit

12. Fit Error

12. Get the error of the fit

- Press statistics
- Copy deltas to a new file
- Check χ^2 value for the goodness of the fit





13. Save

13. Save everything in a project