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UAB

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

VIPER for EXAFS data treatment

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Grup de Tècniques de Separació - Universitat Autònoma de Barcelona

Cerdanyola del Vallès 09.10.14

Program



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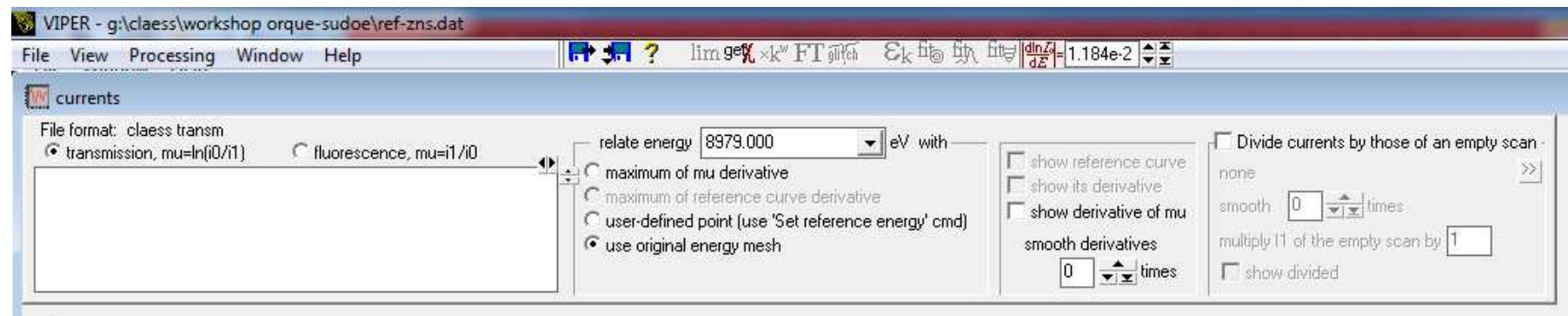
VIPER:

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



Visual Processing in
EXAFS researches

Program interface:



Steps to follow for data analysis

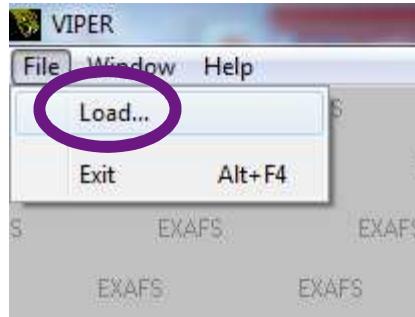


1. Load files
 - 1.1 Choose correct data format
 - 1.2 Choose transmission or fluorescence
 - 1.3 Energy Calibration
2. Get x
3. Configure line colour
4. Deglitch
5. Set E_0
6. Subtract Pre-edge background
7. Set Kmin and Kmax
8. Normalize Post-edge
9. Combine repetitions
10. Back Fourier Transform
11. 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

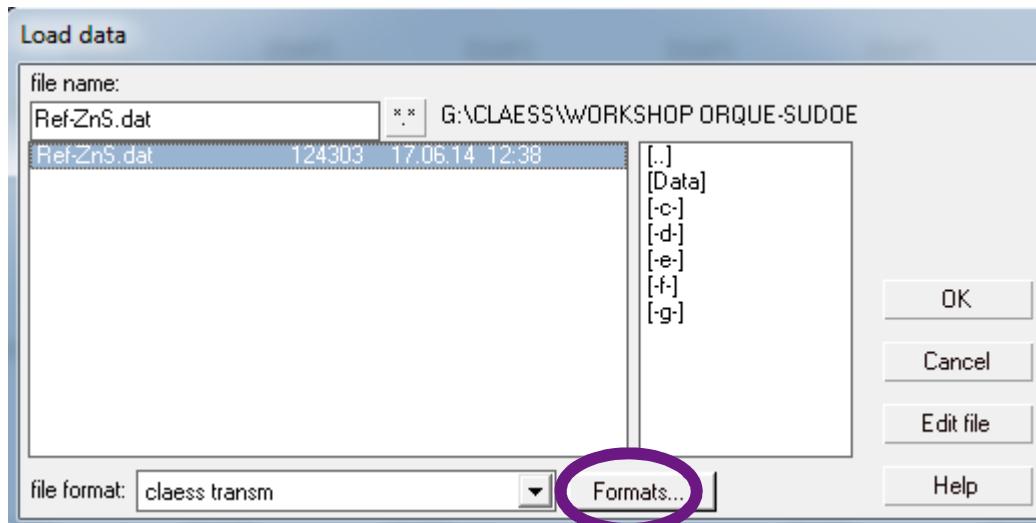
1. Loading files



- Select the file



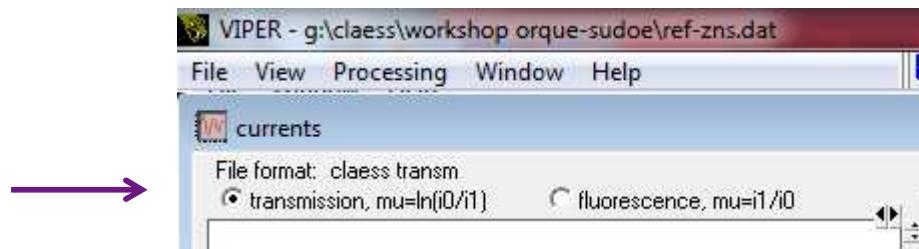
- Choose correct data format



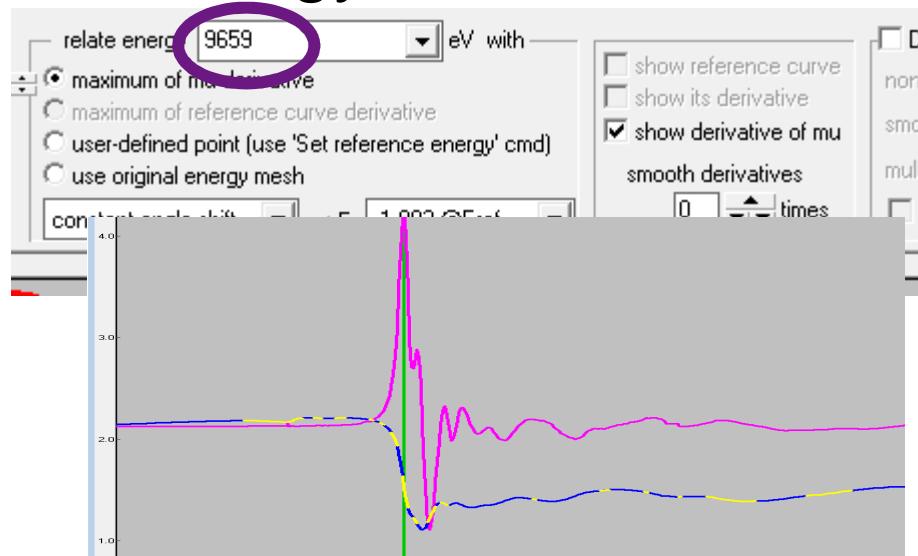
1. Loading files



- Choose transmission or fluorescence



- Energy calibration



Different ways:

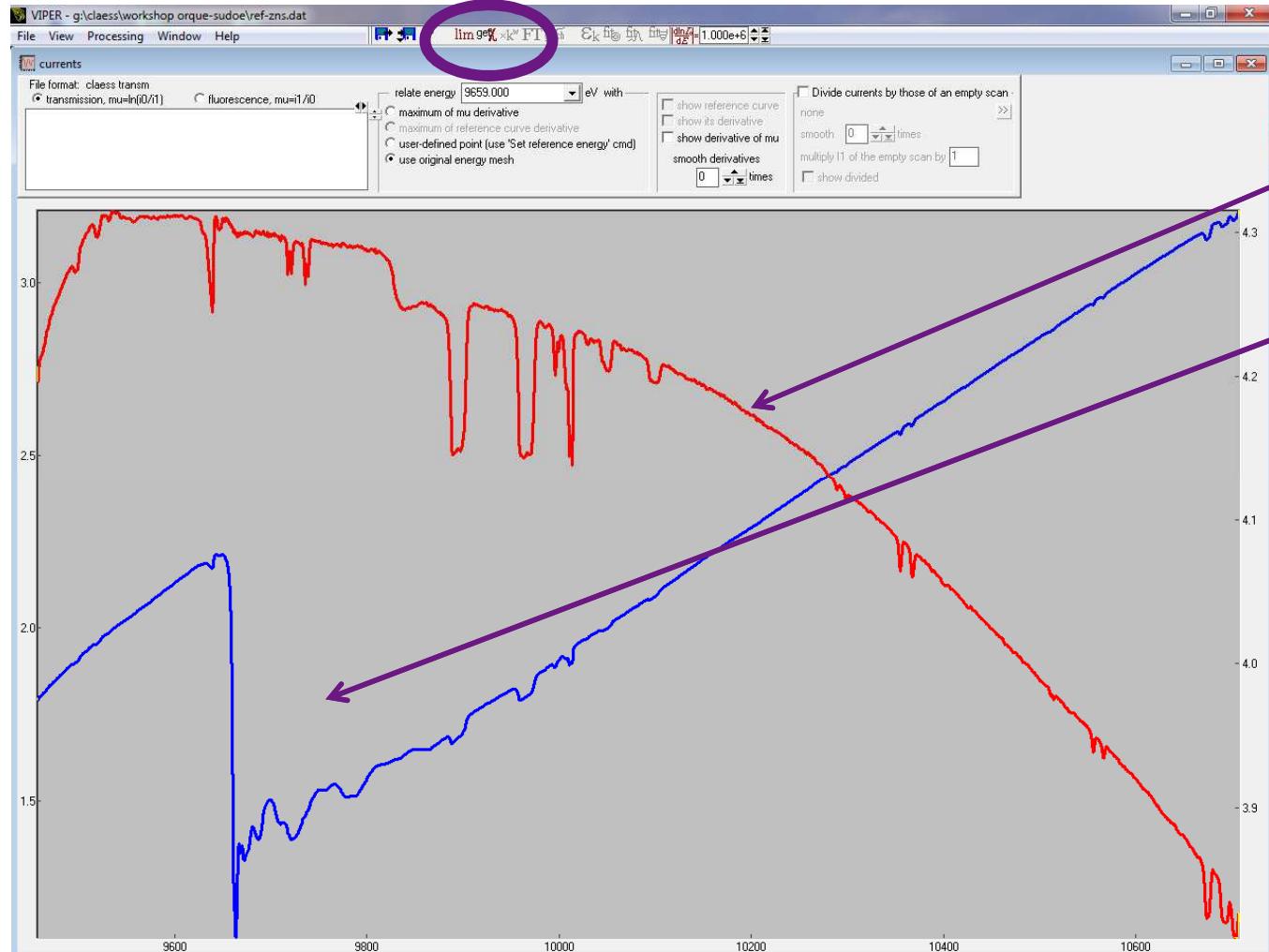
- Selecting the theoretical energy for the element
- Selecting the maximum of the μ derivative
- Using a foil as a reference (before I_2)
- Selecting a point in the spectra



2. Get x



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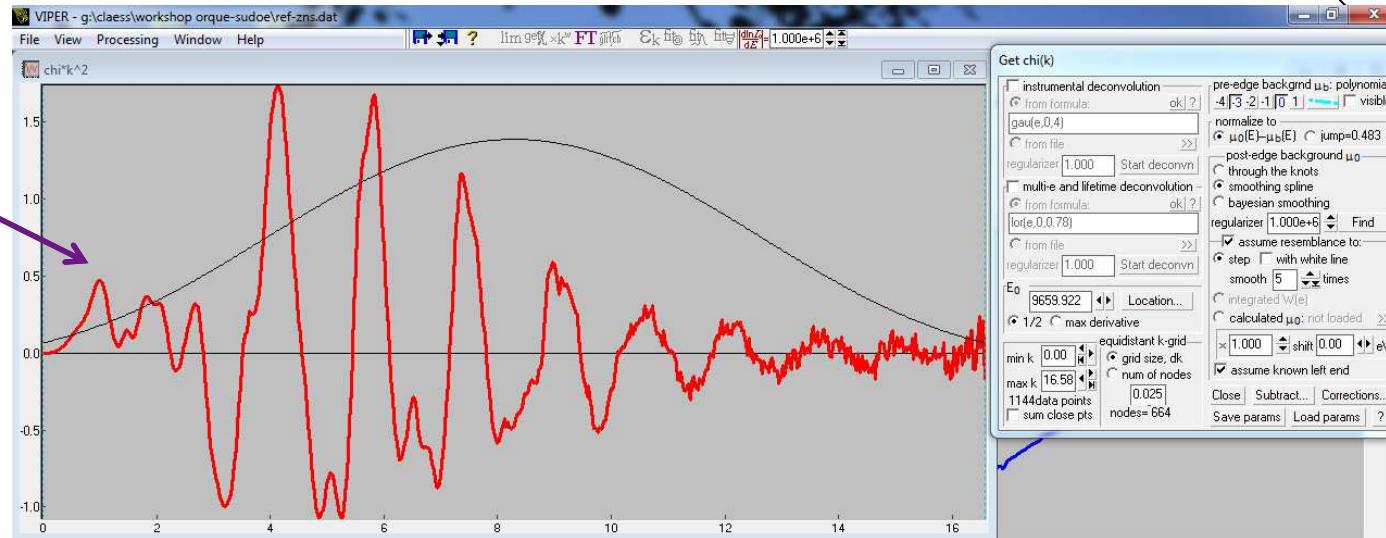


2. Get x

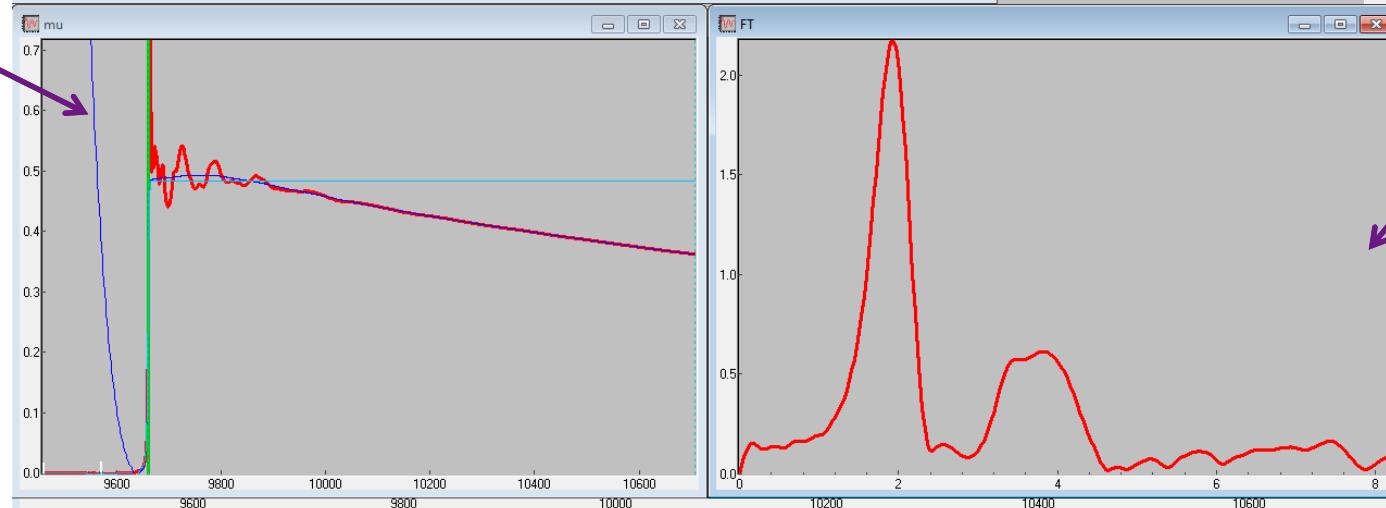


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X



μ



FT

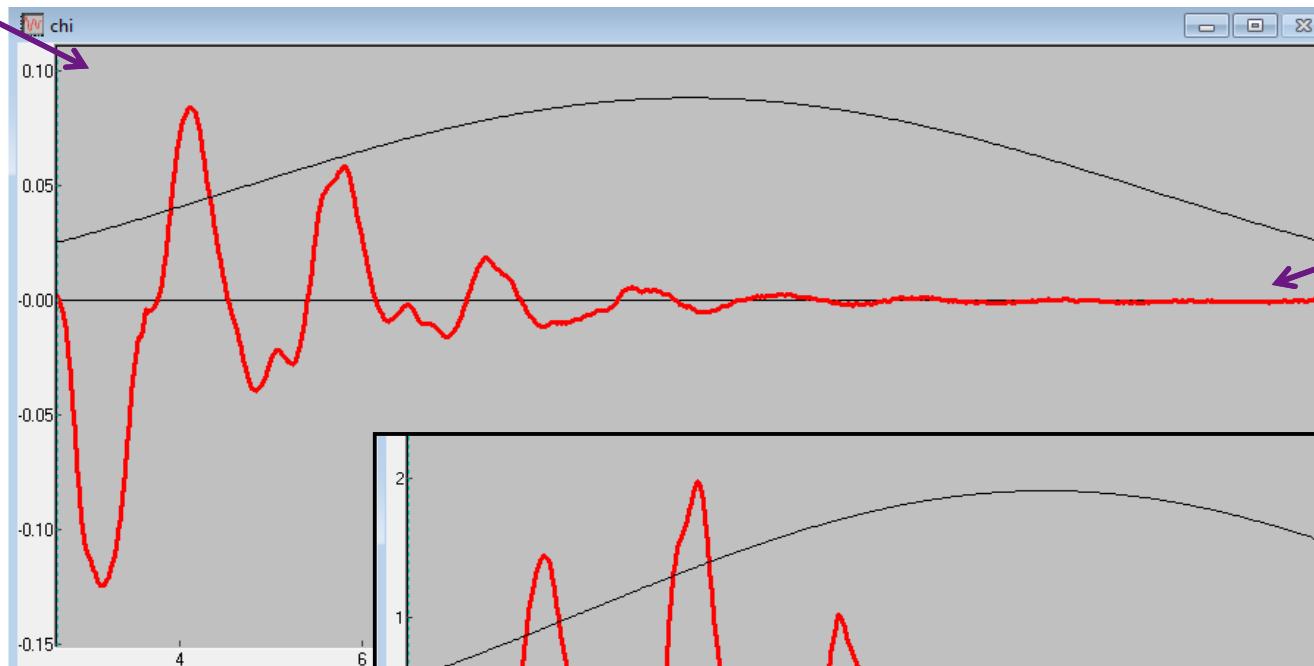


2. Get x



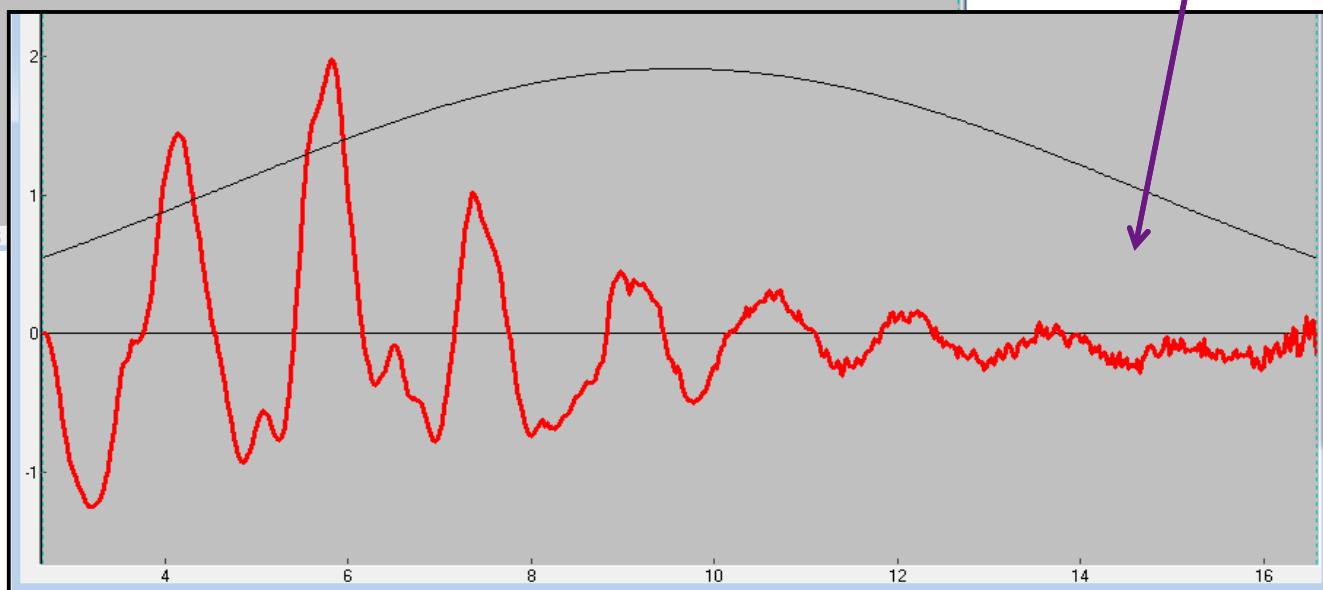
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X

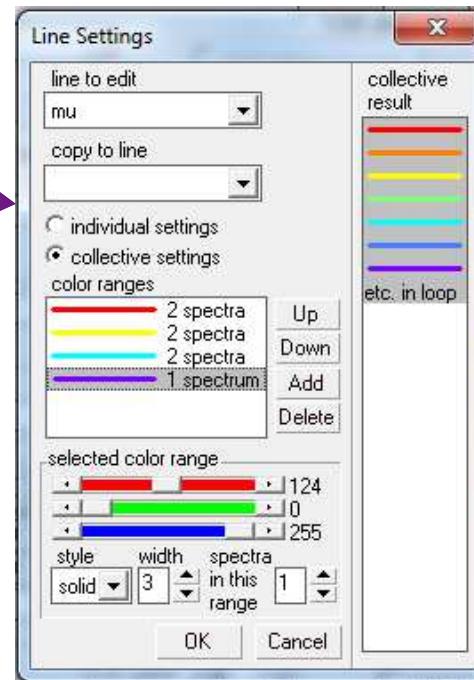
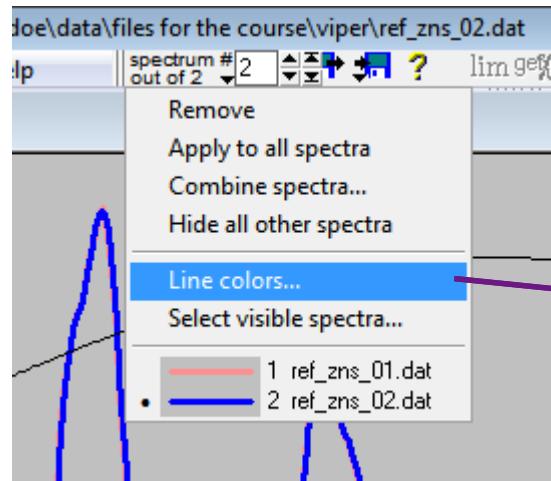


0

2



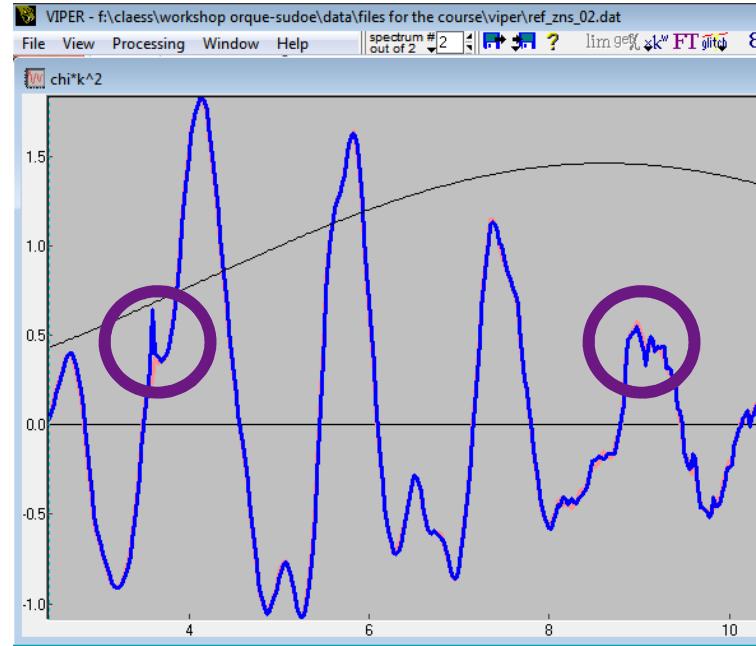
B. Line Colour



4. Deglitch



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

4. Deglitch



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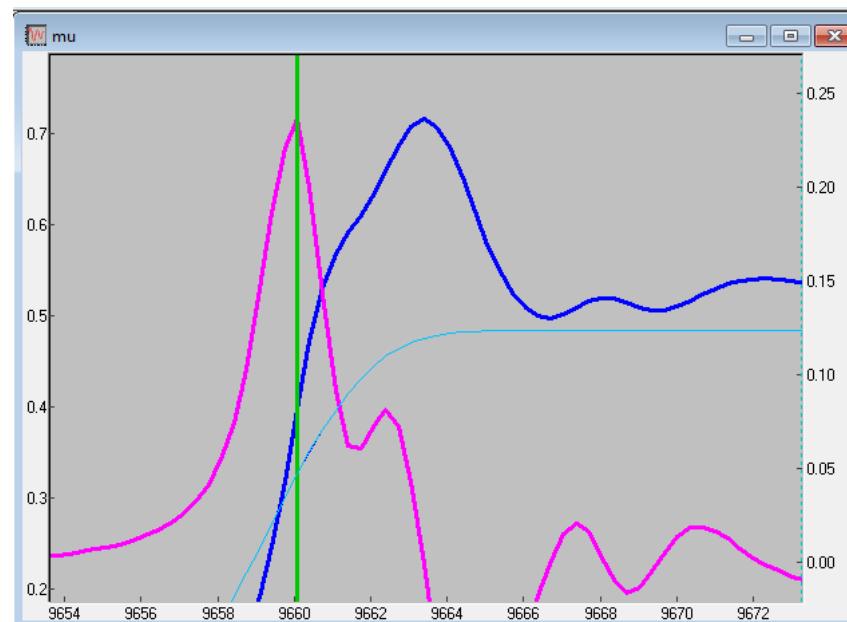
- Zoom
 - Right click → delete glicht region
 - Select region by dragging
 - Delete points
-



5. Set E_o



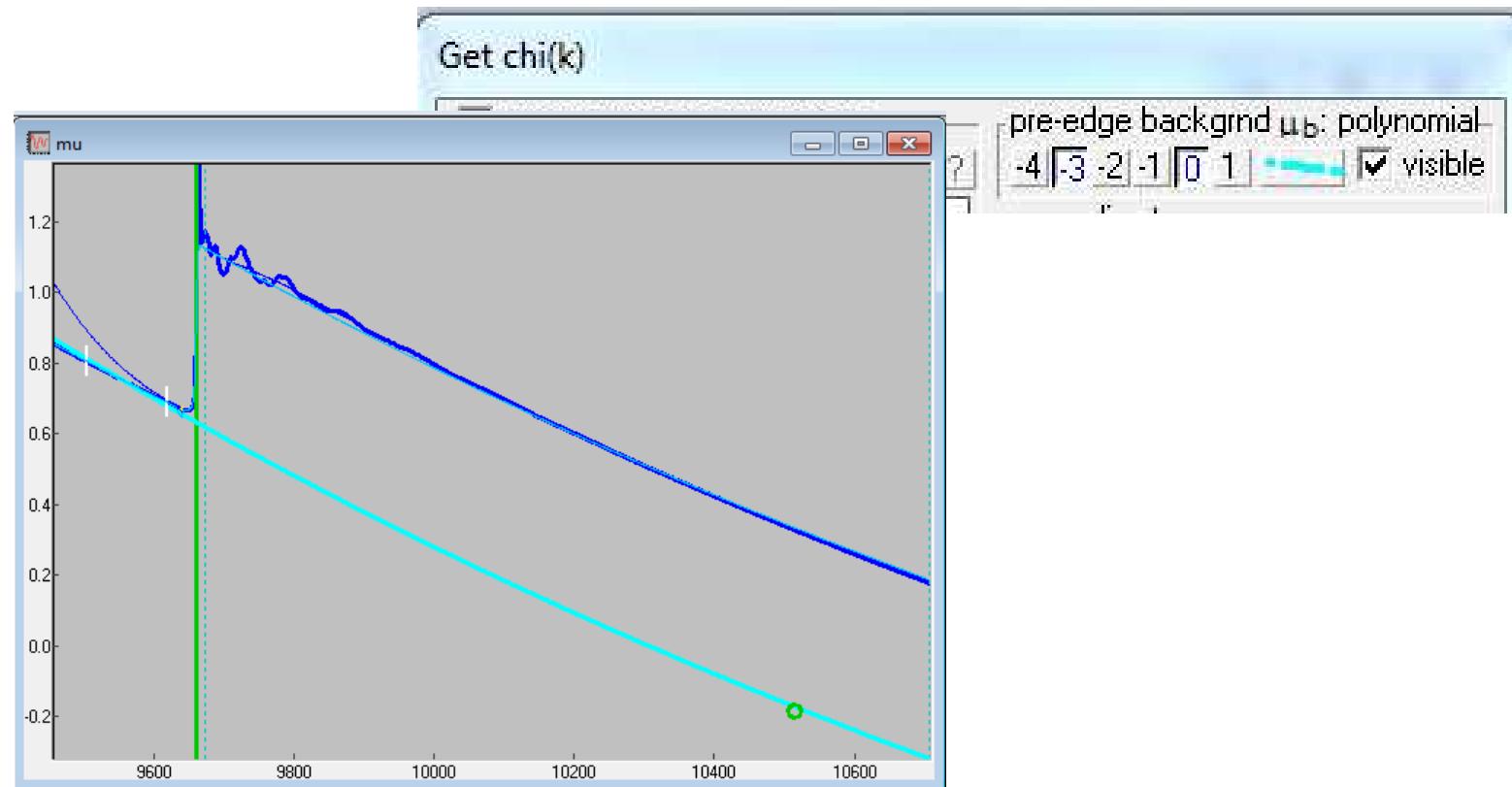
- On μ window → right click → show μ derivative
- Place the green line on top of the first peak of the pink derivative



6. Subtract Pre-edge



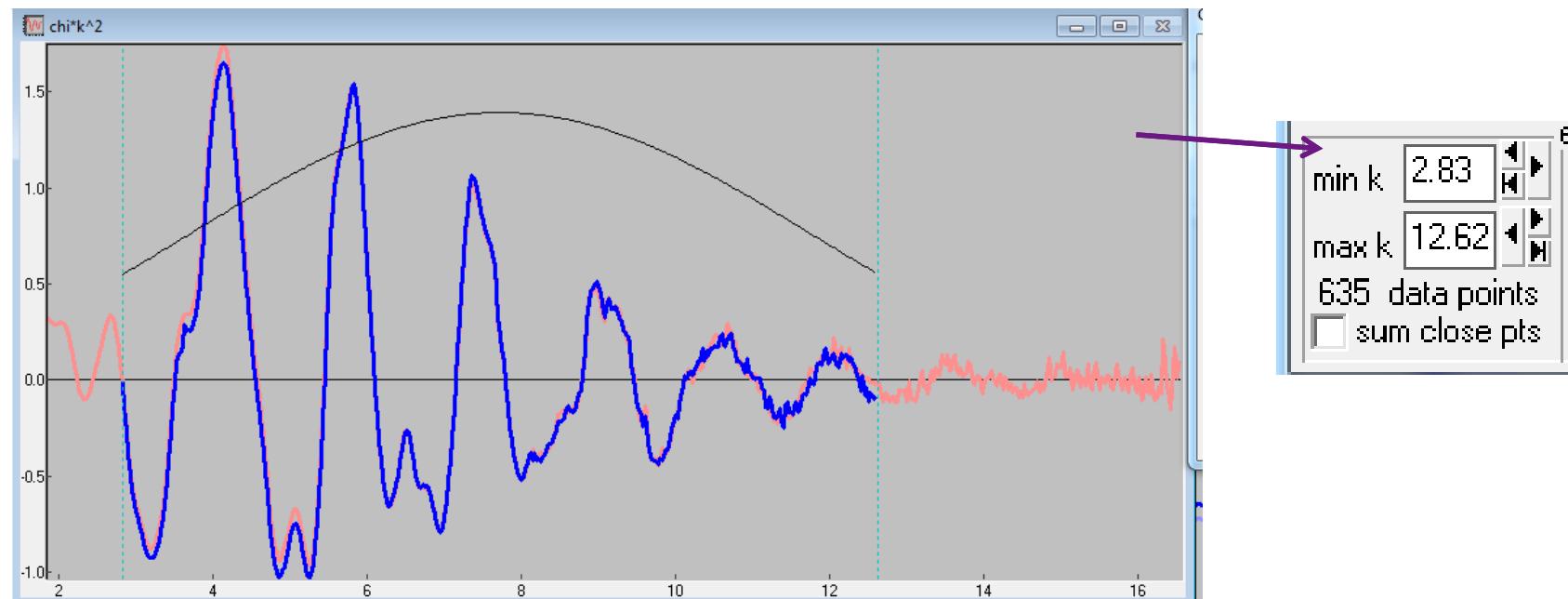
- Subtract background selecting a region in the pre-edge and drawing polynomial line through it



7. Set Kmin + Kmax



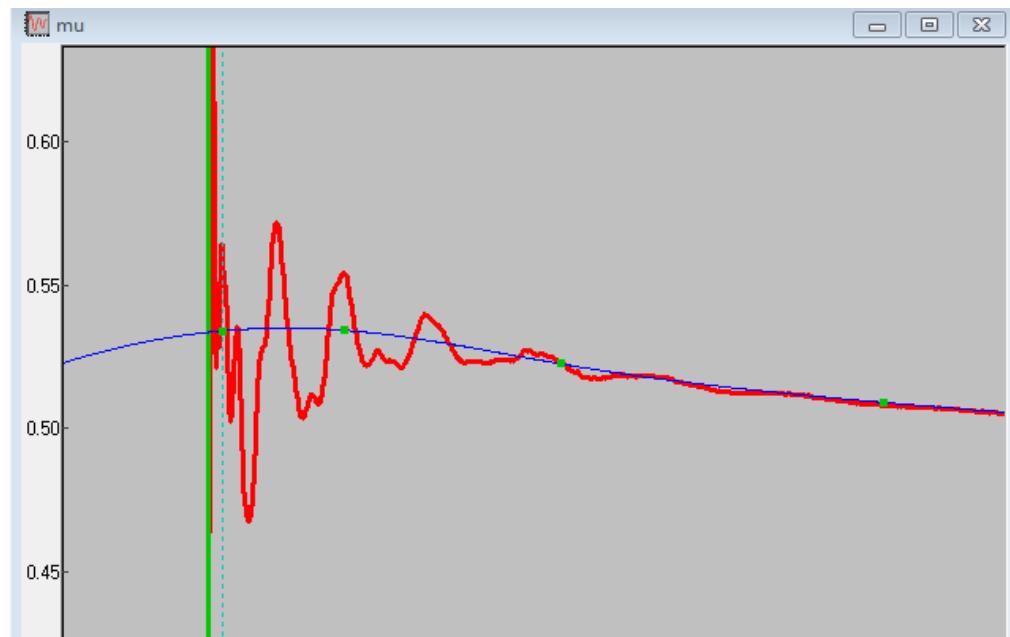
- Seen in both μ and χ as green dotted lines
- Set them where $\chi=0$ and oscillations are visible over noise



8. Post-edge



- Get a straight line to avoid introducing artificial oscillations



3 ways:

- Through the knots
- Smoothing spline
- Bayesian smoothing

8. Post-edge



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3 ways:

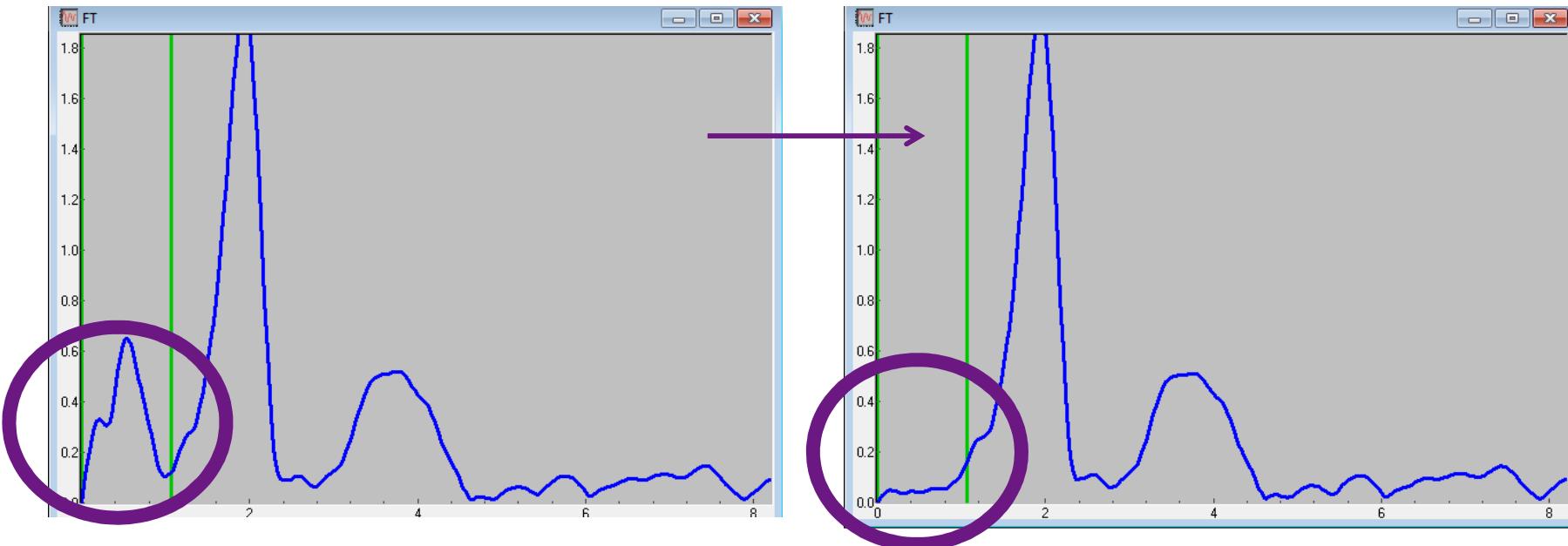
- Through the knots
- Smoothing spline
- Bayesian smoothing

8. Post-edge



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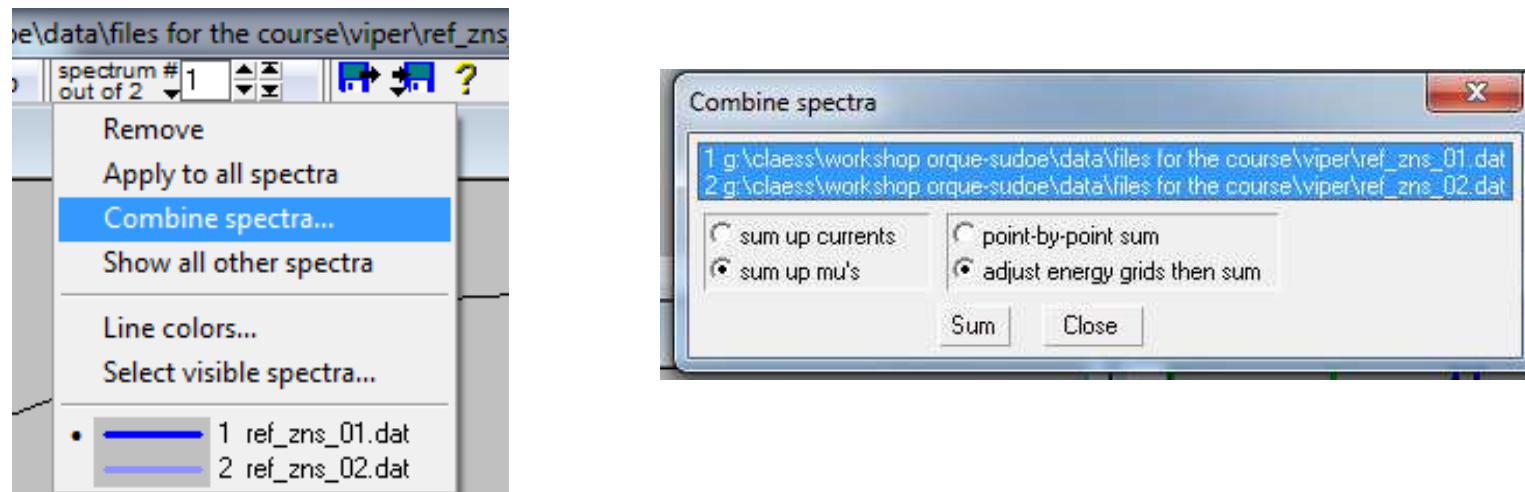
- In the FT, minimize signal before the first shell



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

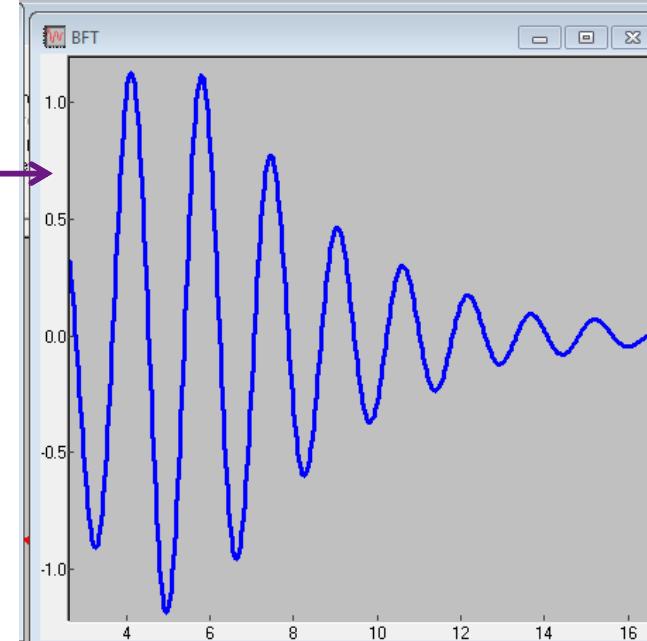
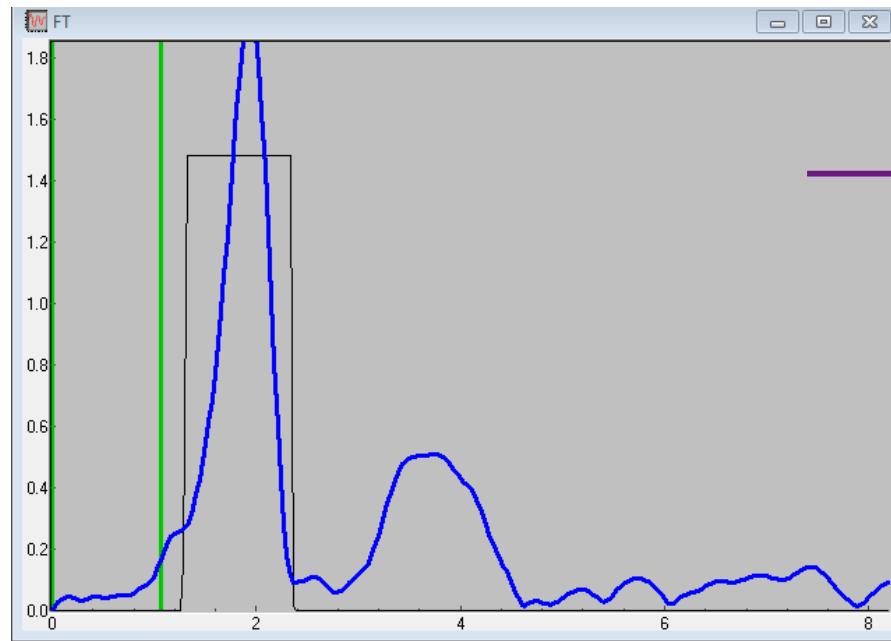


10. Back Fourier



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





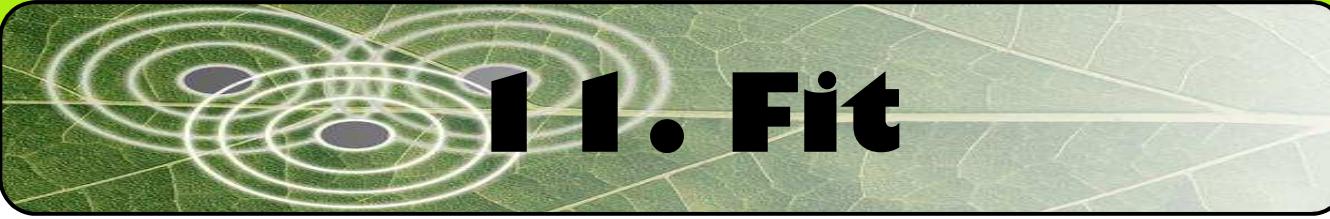
11. Fit



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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



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11.1 Create input file with Atoms

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

The screenshot shows the ATOMS web interface at cars9.uchicago.edu/cgi-bin/atoms/atoms.cgi. The page title is "ATOMS". The main content area contains the following text:

running the *ab initio* XAFS program FEFF, however several other interesting output formats are available.

This web page demonstrates the main features of ATOMS. It consists of a rather large form which you may fill in with data describing your crystal. You may also search a database of input data for ATOMS. After clicking the "Run Atoms" button, your browser will display an input file suitable for running FEFF (or perhaps some other kind of interesting output file). You can get help about any of the parameters by following the link bound to the parameter name.

This web page does not offer all the features available in the version of ATOMS which you can run on your own computer. Please see the [ATOMS homepage](#) for complete details.

The interface includes several input fields and buttons:

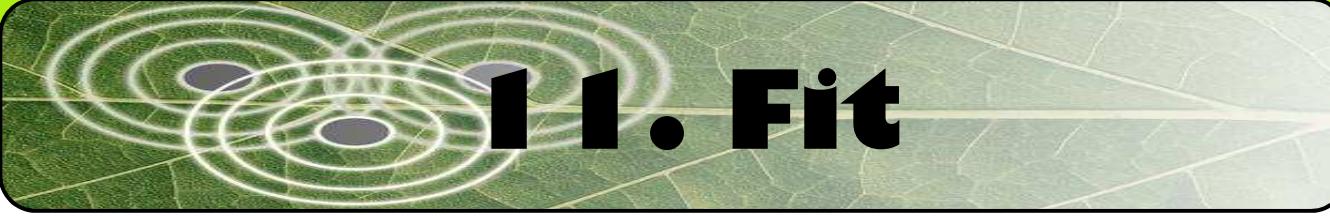
- Operational Parameters:** Space Group: Rmax: Edge:
- Output Type:** feff6.inp
- Lattice Constants and Angles:** A: B: C:
Alpha: Beta: Gamma:
- Run ATOMS** **Clear** **Restablecer**

Table of Crystallographic Sites

| Cent. | Element | X | Y | Z | Tag |
|------------------------------------|---------|---|---|---|-----|
| <input checked="" type="radio"/> 1 | | | | | |
| <input type="radio"/> 2 | | | | | |
| <input type="radio"/> 3 | | | | | |
| <input type="radio"/> 4 | | | | | |
| <input type="radio"/> 5 | | | | | |
| <input type="radio"/> 6 | | | | | |
| <input type="radio"/> 7 | | | | | |
| <input type="radio"/> 8 | | | | | |
| <input type="radio"/> 9 | | | | | |
| <input type="radio"/> 10 | | | | | |

Run ATOMS **Clear** **Restablecer**

Redisplay with this many sites: **Do it!** [Explain](#)



11. Fit



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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:

| | | |
|----------|------------------|------------|
| feff6I | 05/11/2007 21:06 | Aplicación |
| feff6L.f | 29/10/2007 21:19 | Archivo F |

- 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:

The screenshot shows the BFT software interface. On the left, the main window has the title "Ordinary fitting". It contains several input fields and buttons:

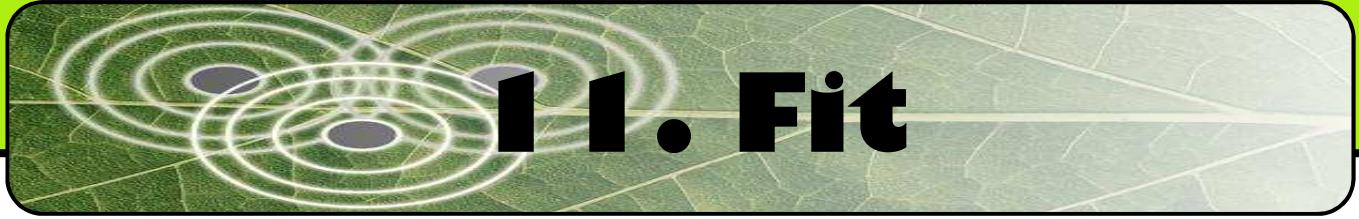
- "shell # 1 of 1" with spinners.
- "multiply by k^w, w= 0" with a spinner.
- "R-factor (%)" button.
- "Constrain..." button.
- "Statistics..." button.
- "Setup..." button.
- "Load data" button.
- "To advanced mode" button.

On the right, there is a "Load data" dialog box:

| file name: | 6^Zn-Zn refl=2.6600 |
|--------------|----------------------|
| feff0001.dat | 25044 08.10.14 21:28 |
| feff0001.dat | 4817 08.10.14 21:28 |
| feff0002.dat | 4817 08.10.14 21:28 |
| feff0003.dat | 4817 08.10.14 21:28 |
| feff0004.dat | 4864 08.10.14 21:28 |
| feff0005.dat | 4864 08.10.14 21:28 |
| feff0006.dat | 4864 08.10.14 21:28 |
| feff0007.dat | 4817 08.10.14 21:28 |
| feff0008.dat | 4817 08.10.14 21:28 |
| feff0009.dat | 4864 08.10.14 21:28 |
| feff0010.dat | 4864 08.10.14 21:28 |
| feff0011.dat | 4817 08.10.14 21:28 |

Buttons at the bottom of the dialog box include "OK", "Cancel", "Edit file", and "Help".

- Select feff0001.dat from

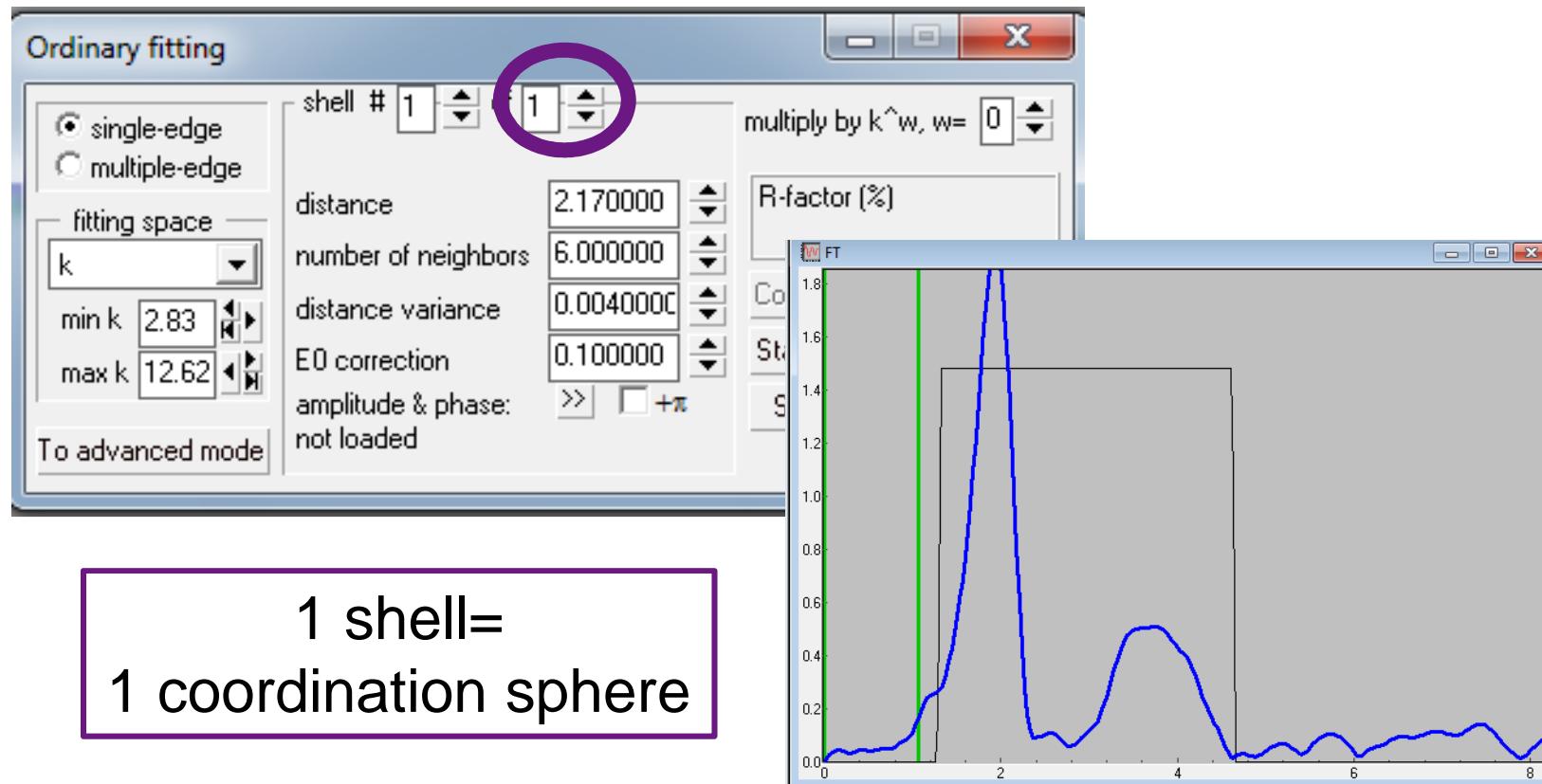


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11.4 Add more shells if needed

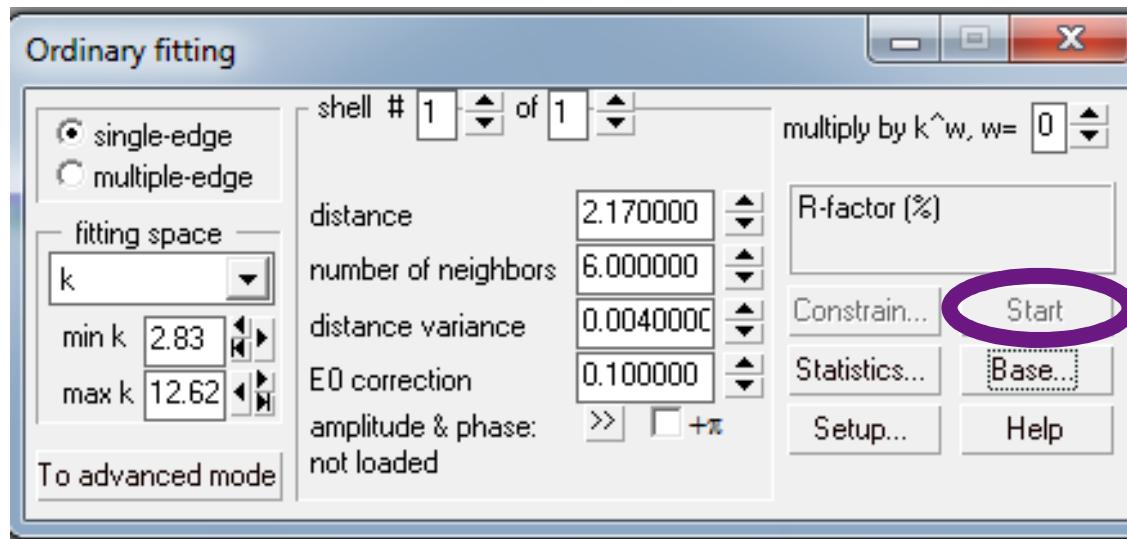


1 shell=
1 coordination sphere



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11.5 Start

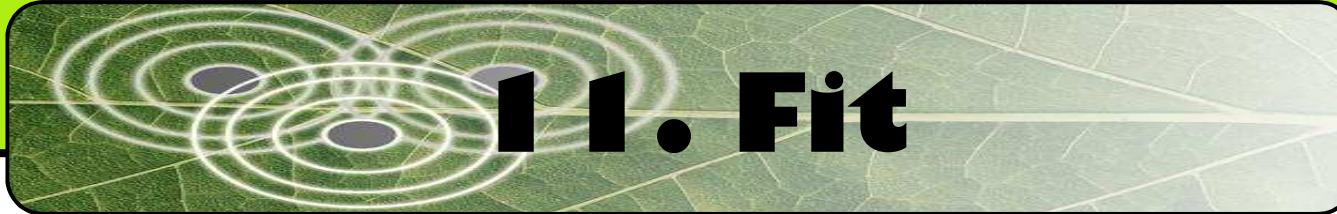


Distance (r) = bond length

Number of neighbors (n) = coordination number

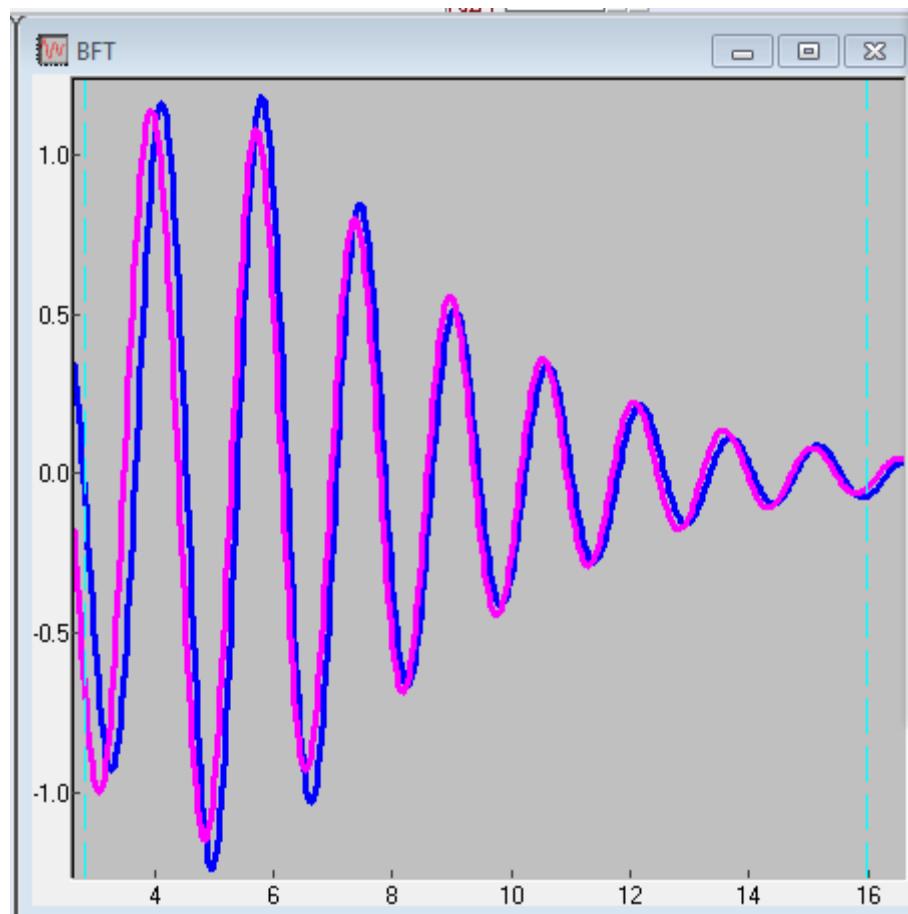
Distance variance (σ^2) = Debye-Waller Factor

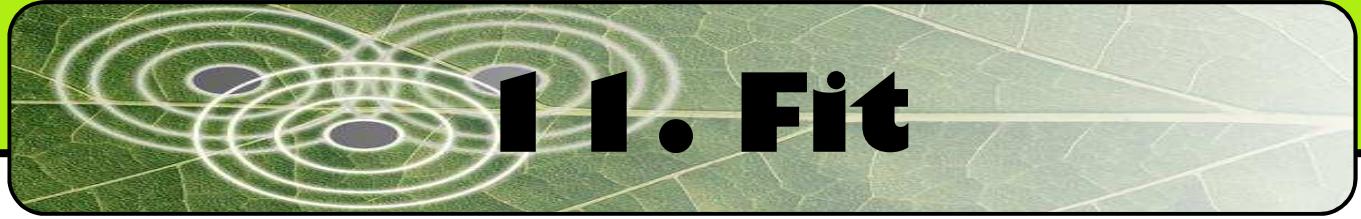
E0 correction (e) = energy shift



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11.5 Start

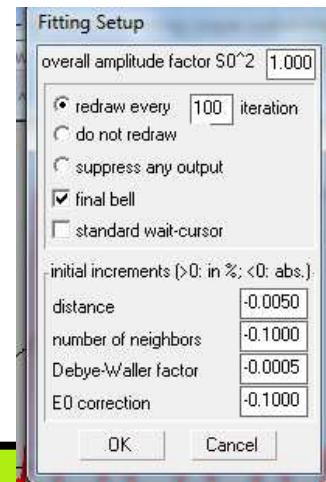
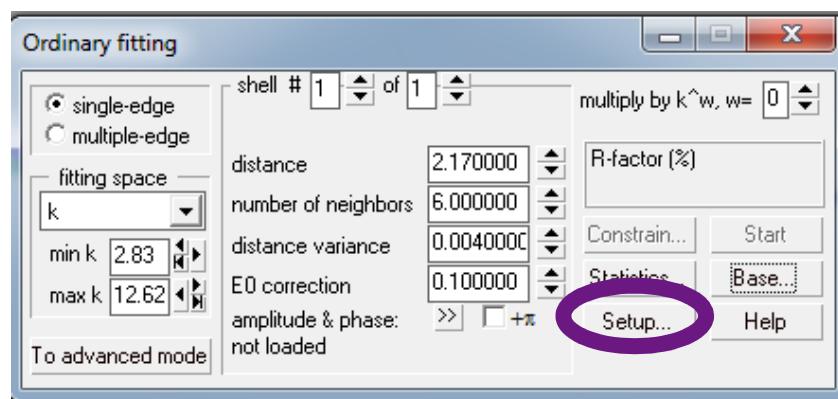


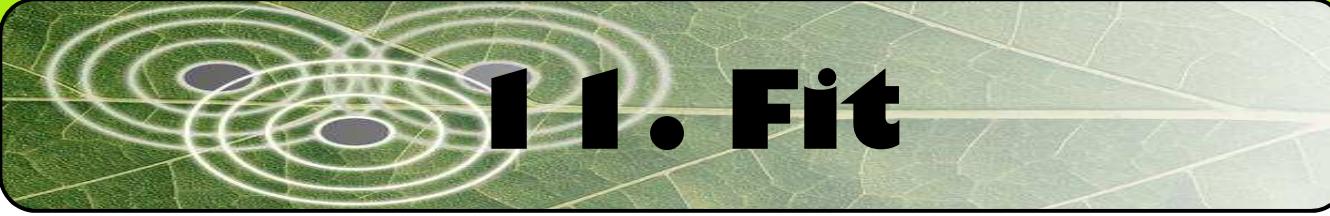


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11.6 Change S_0^2 if needed

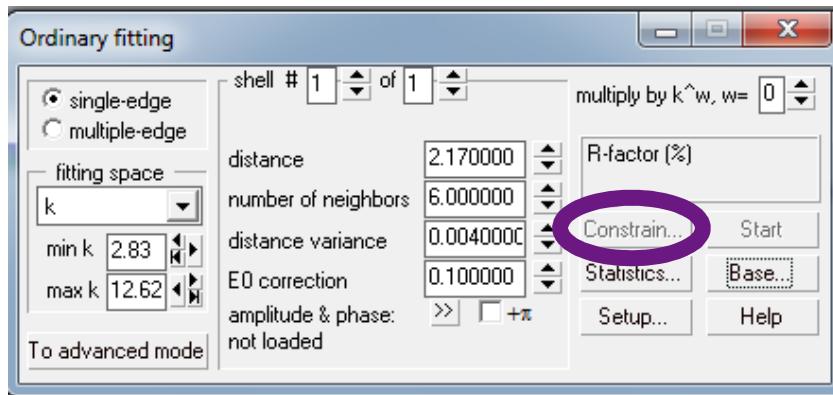
- Sometimes the obtained coordination number (n) is lower than it should
- You need to change the amplitud, S_0^2
- Divide: $\frac{\text{obtained } n}{\text{theoric } n}$
- Use the result as the new S_0^2 , change it in Setup





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11.7 Add constraints if needed



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

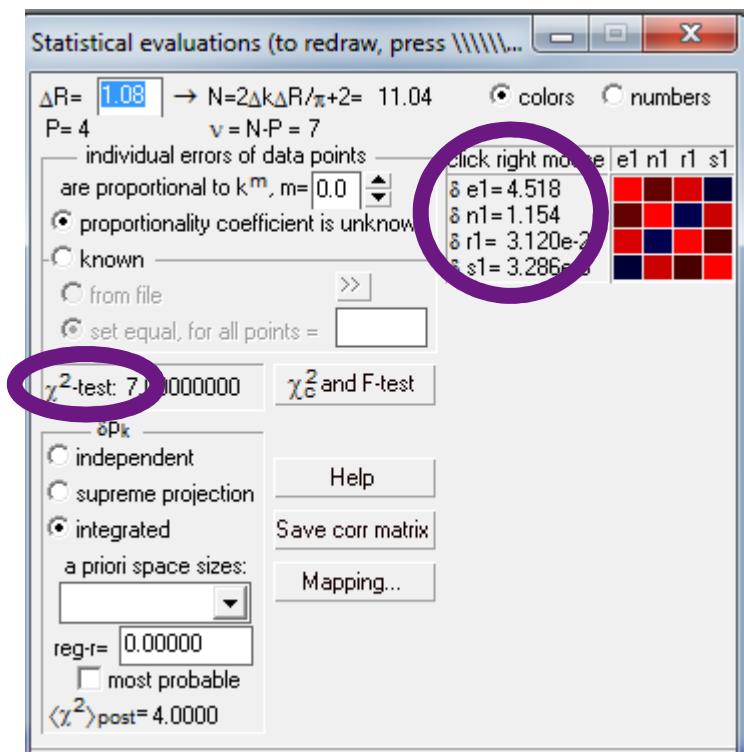
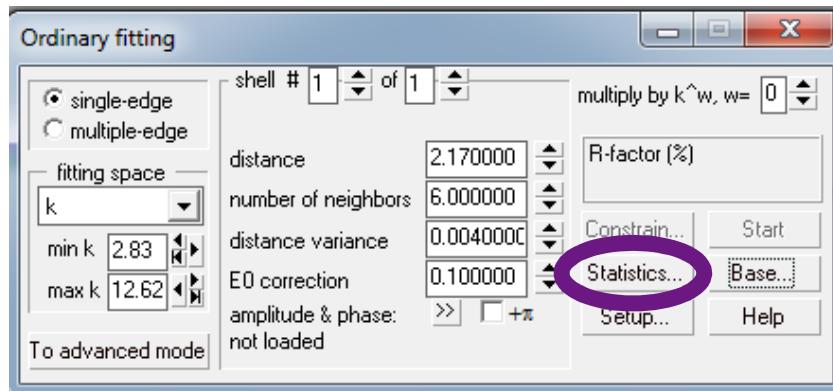
11.8 Start again the fit

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



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13. Save everything in a project

