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

XANDA for XANES data treatment

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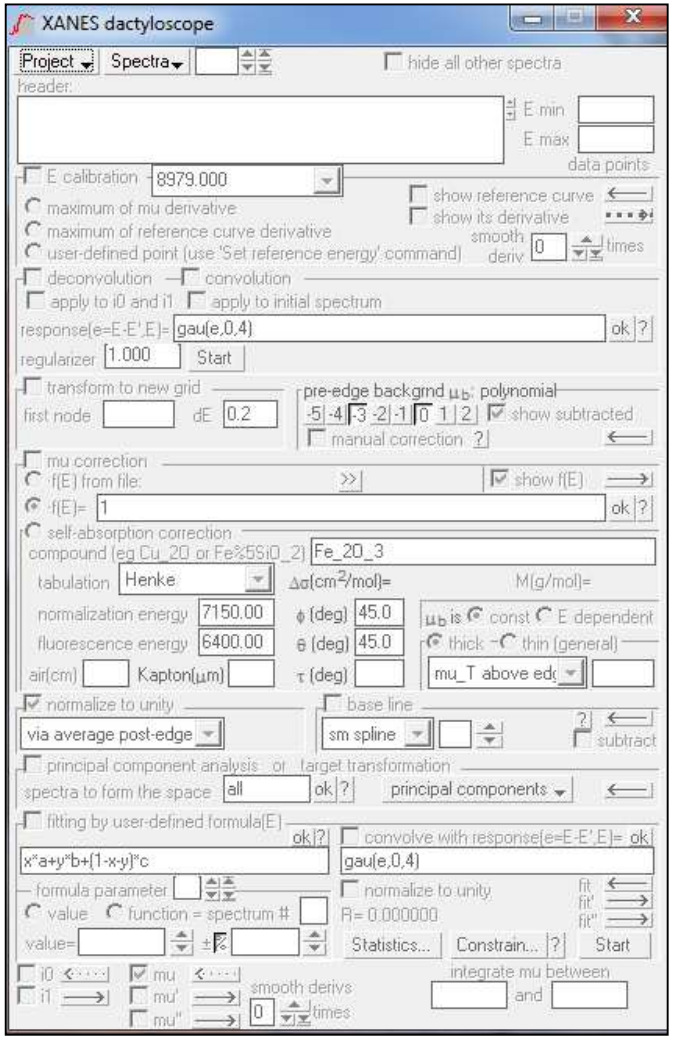
Cerdanyola del Vallès 07.10.14

Program

Grup de Tècniques de Separació en Química
UAB

Program interface:



XANES dactyloscope:

- Author: Konstantin Klementiev
- Version: 15 Feb 2013, build 604

A program for quick and rigorous XANES analysis for Windows

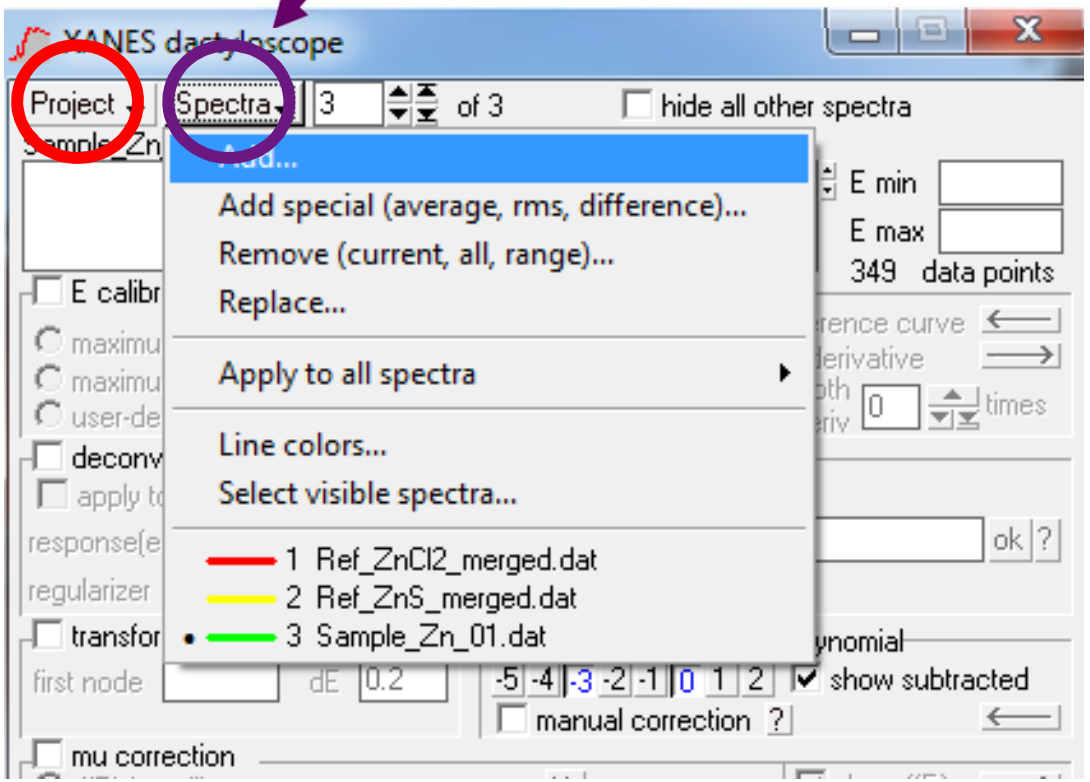
Steps to follow for data analysis

1. Load files
    - 1.1 Choose correct data format
    - 1.2 Choose transmission or fluorescence
    - 1.3 Configure line colour if needed
2. Corrections
    - 2.1 Energy calibration
    - 2.2 Self-absorption
3. Subtract Pre-edge background
4. Normalize Post-edge
5. Merge
6. Set limits
7. Fitting by user-defined formula
8. Determine the error of the fit
9. Save

1. Loading files

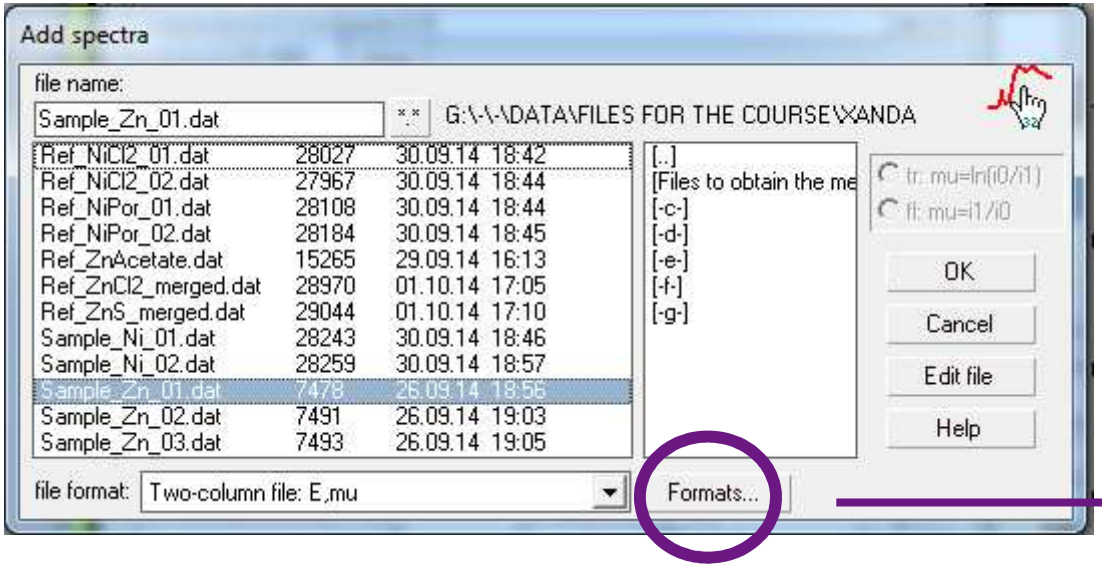
Load single or multiple spectra → .dat files

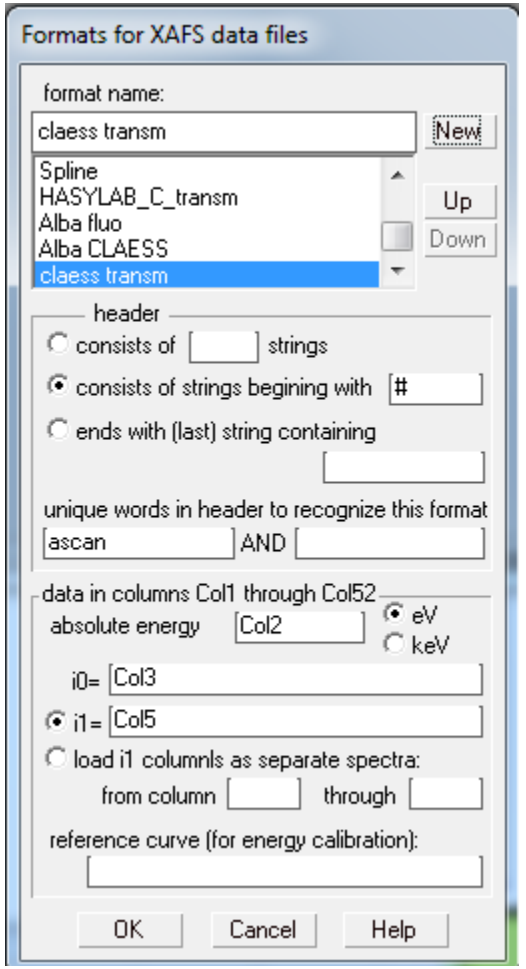
Load an already created XANES project → .xpj files



1. Loading files

- 1.1 Choose correct data format



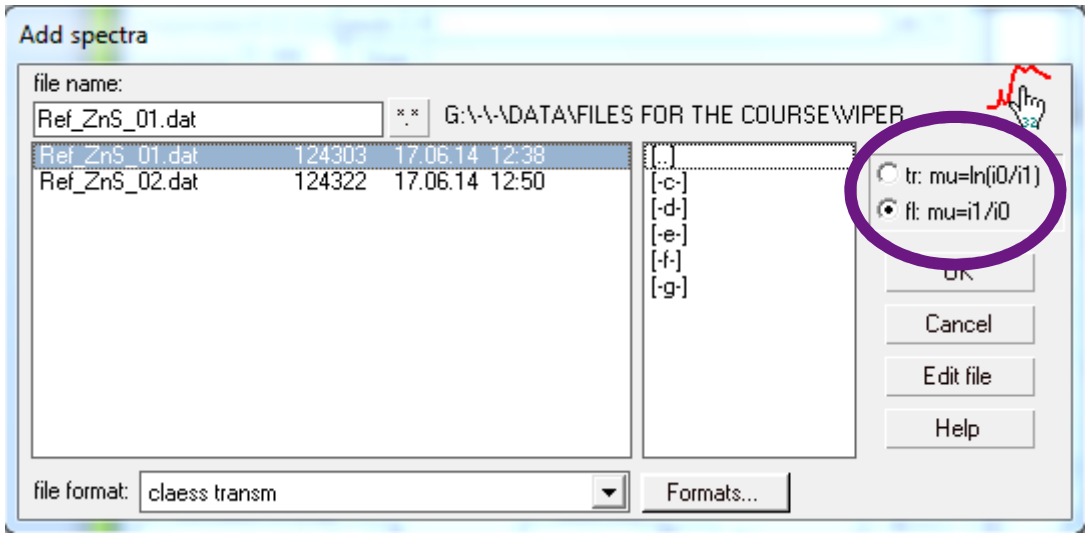


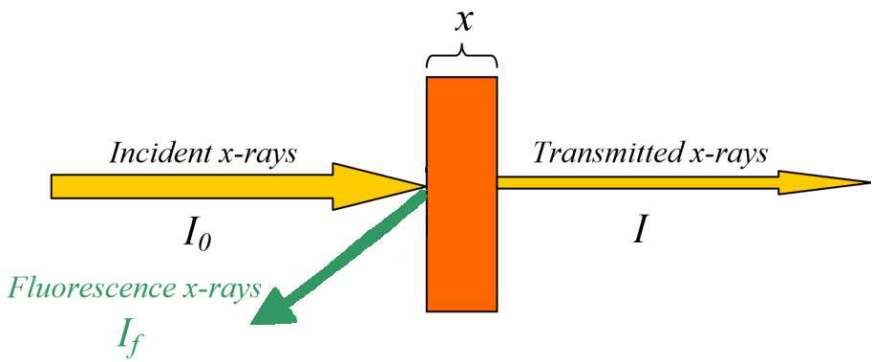
Every file with raw data has many columns with multiple information. Ask the beamline scientists which are the columns to use as E, i0 and i1.

Several beamline formats are already written in the program

1. Loading files

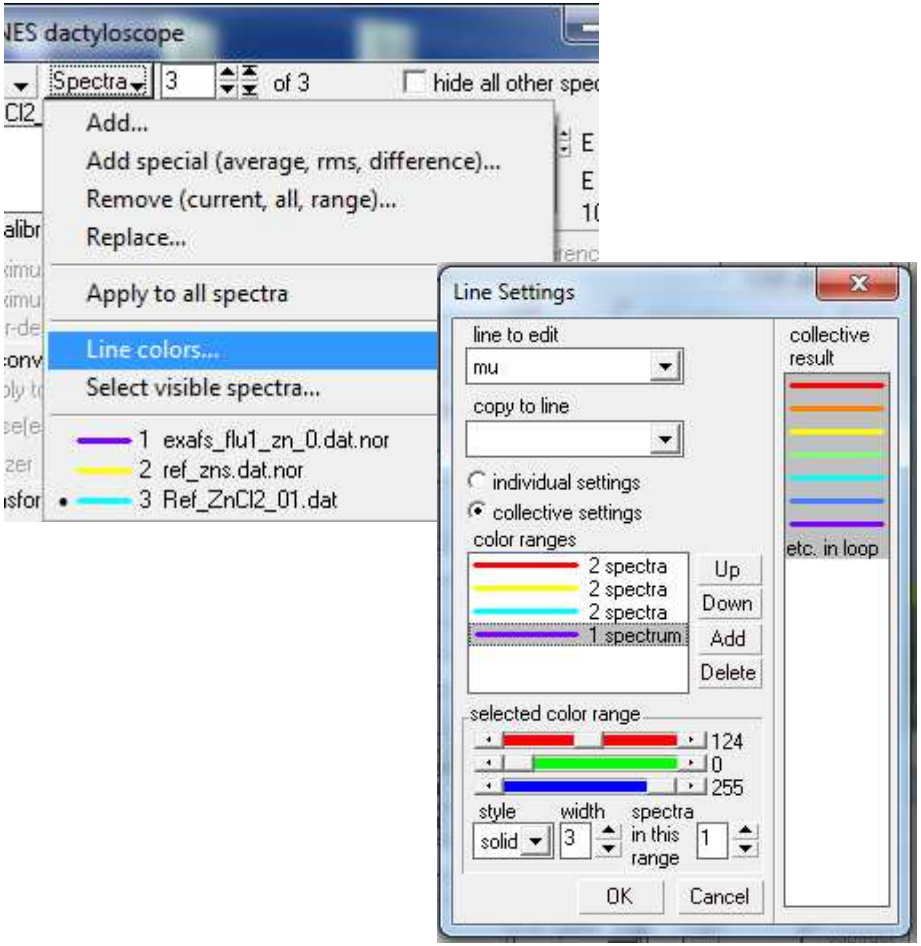
- 1.2 Choose transmission or fluorescence

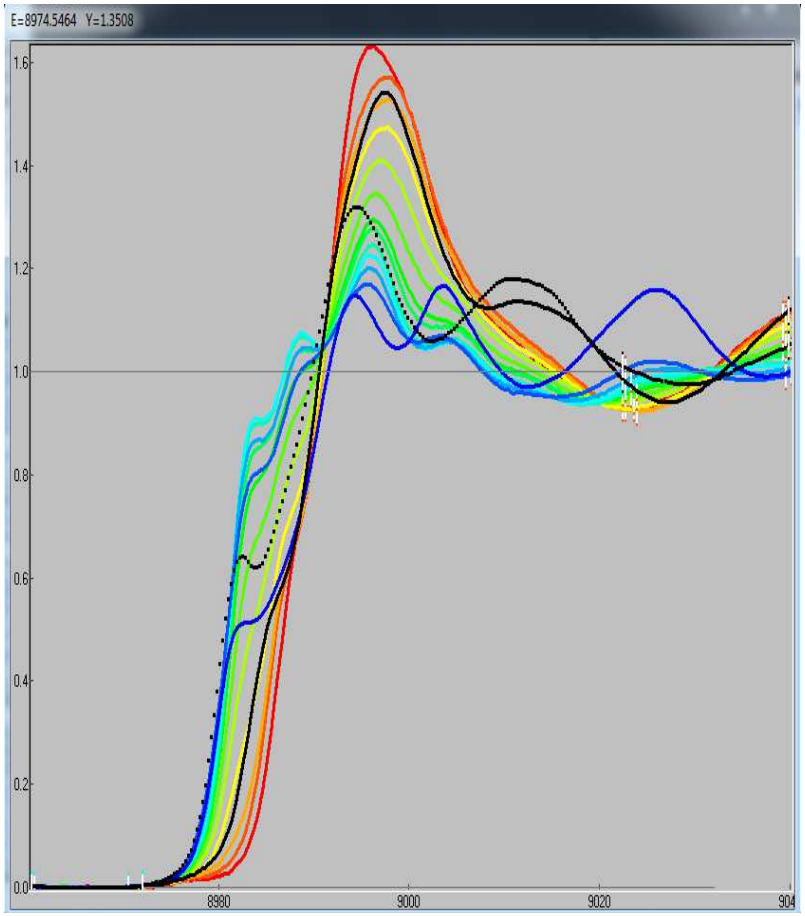




1. Loading files

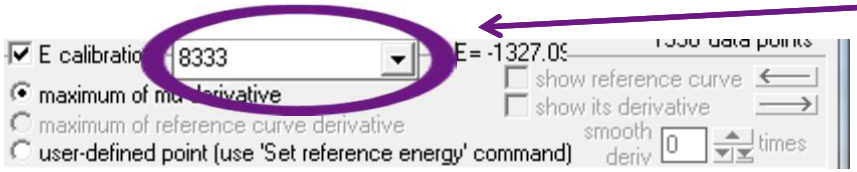
- 1.3 Configure line colour if needed





2. Corrections

- 2.1 Energy calibration

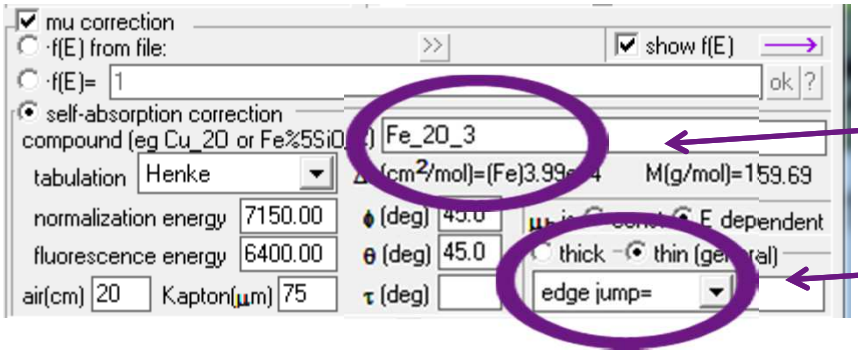


The jumps of the species must be aligned in E

Different ways:
Selecting the theoretical energy for the element
Using a foil as a reference, placed before I_2
Selecting the maximum of the μ derivative
Selecting a chosen point in the spectra

- 2.2 Self-absorption correction

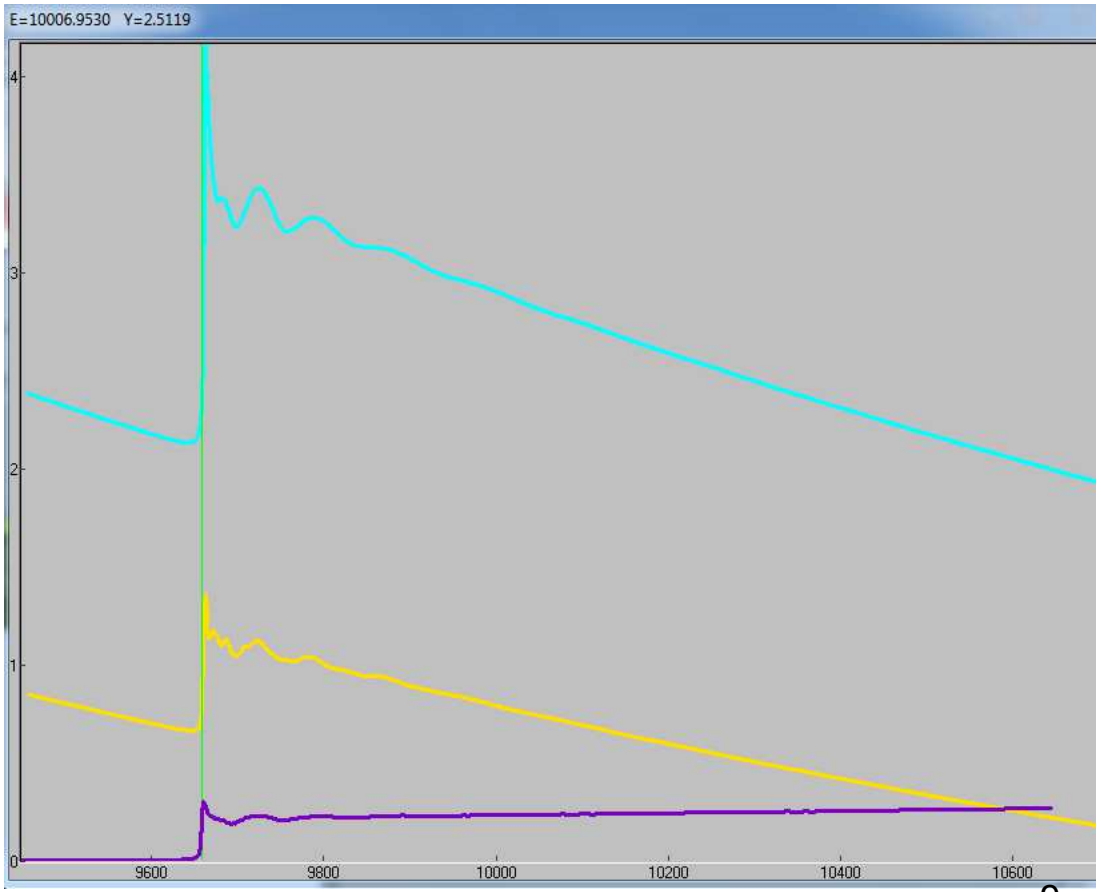
(only if needed)



Enter sample information:
compound chemical formula
and
physical thickness or edge jump

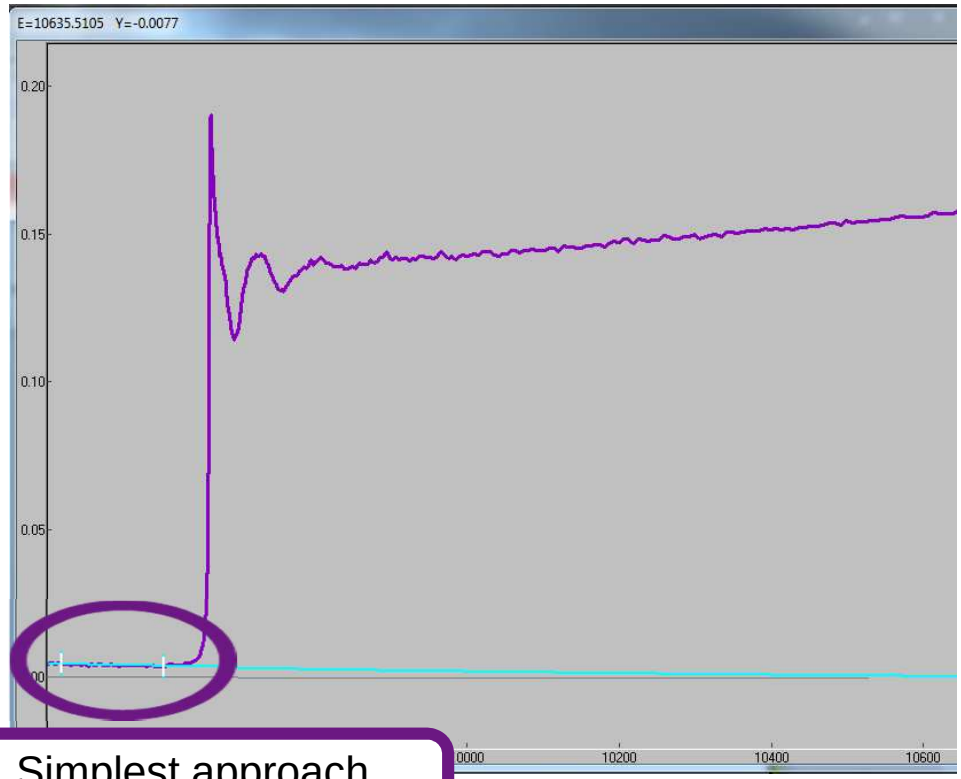
2. Corrections

Before pre-edge and post-edge normalization, the spectra are difficult to compare.



3. Pre-edge background

The background has to be subtracted from the spectra. It is found by drawing a polynomial line over a pre-edge region.



Usual polynomials:

Lineal: aE+b → Simplest approach, good for Fluorescence

Victoreen: $aE^{-3}+bE^{-4}$ → Complex approach, good for Transmission

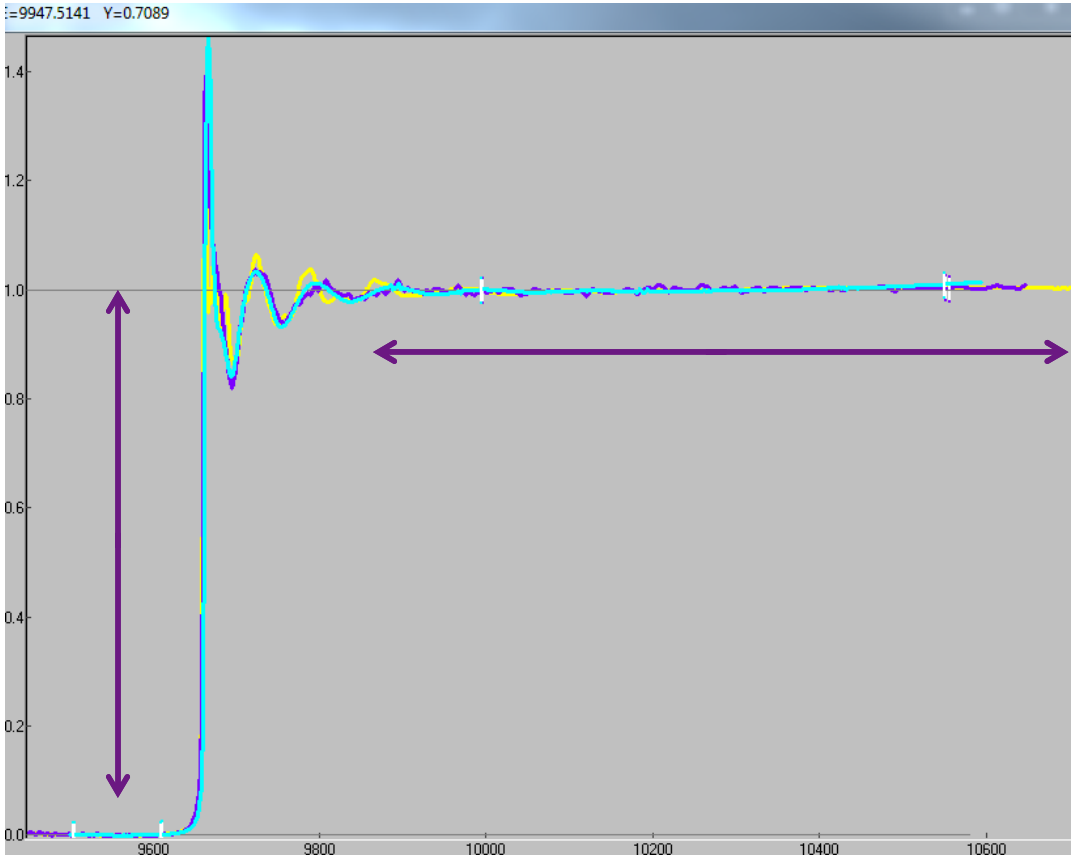
Modified Victoreen: $aE^{-3}+b$ → Complex approach, good for Transmission

4. Normalize Post-edge

Divide the spectra by a constant

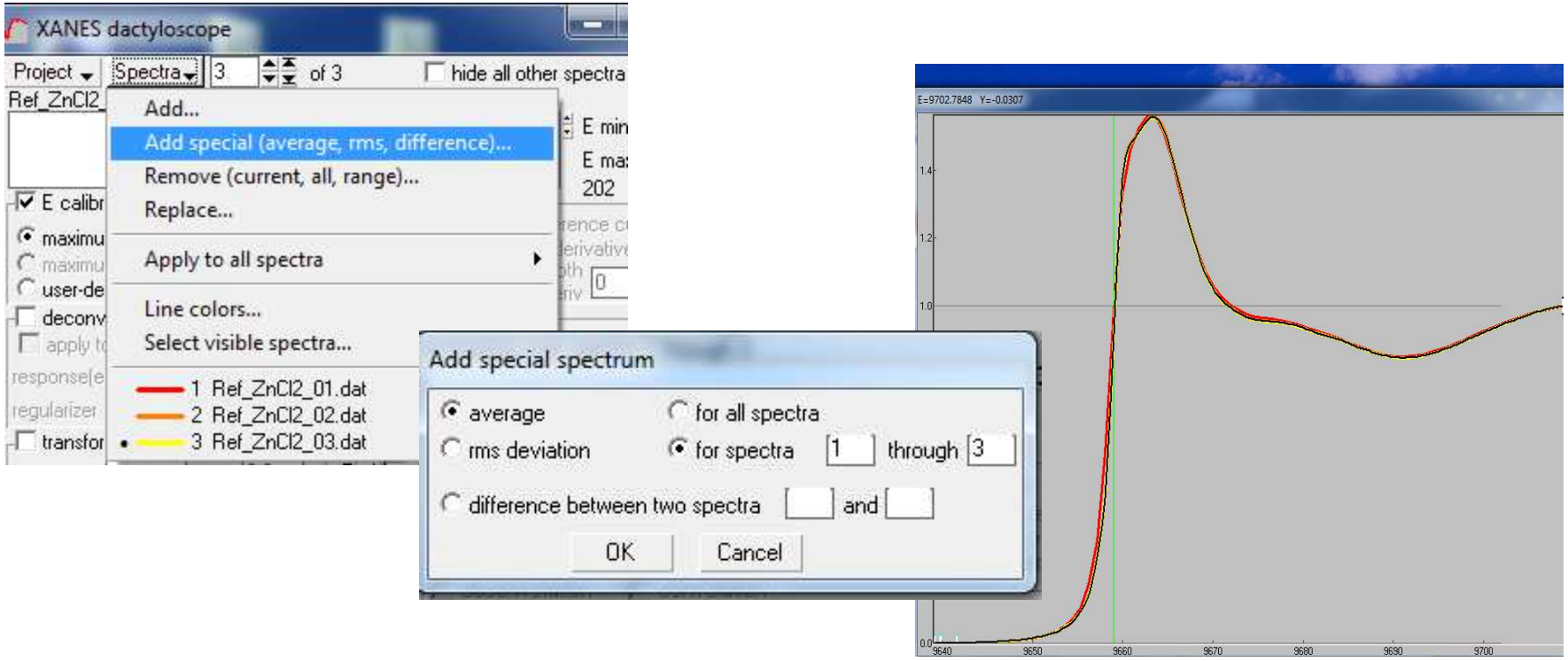
Use the height of the jump

The mean value of the post-edge equals 1



5. Merge

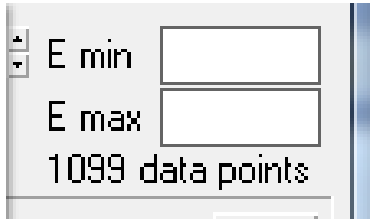
- Combine repetitions from the same sample



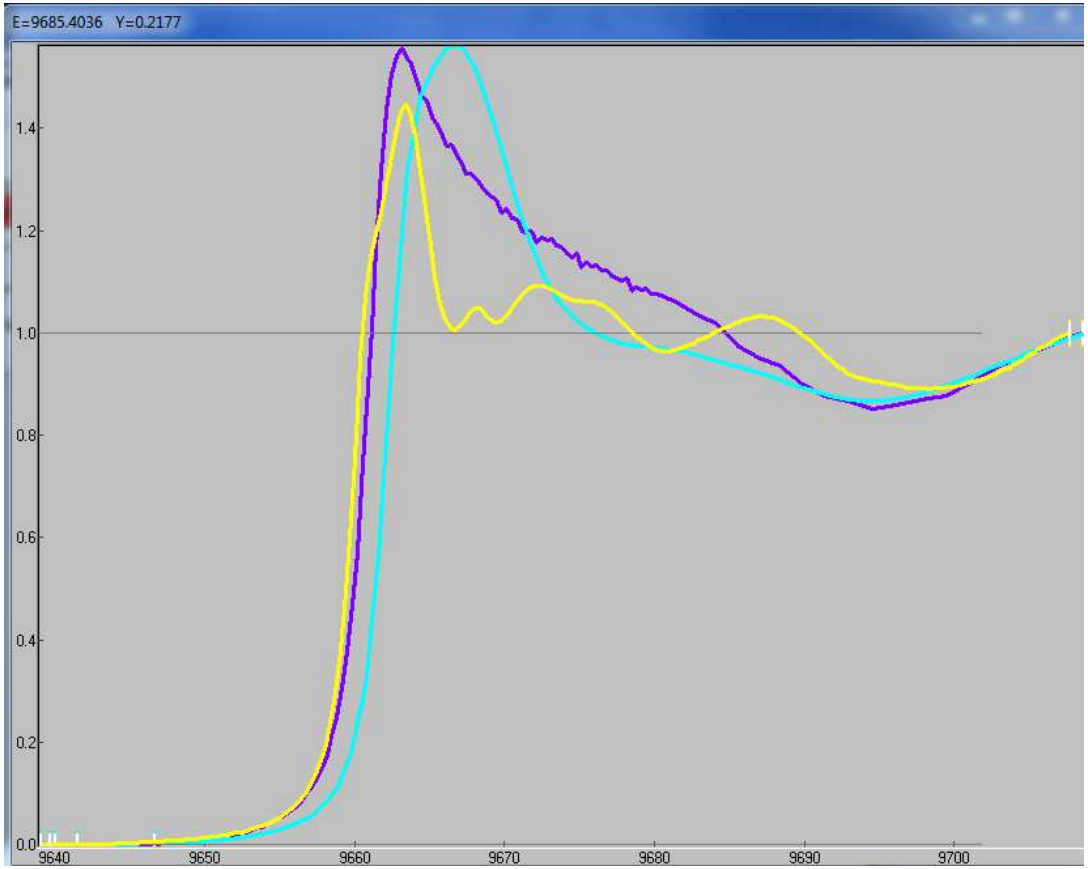
6. Set limits

For the fit, a small region is needed

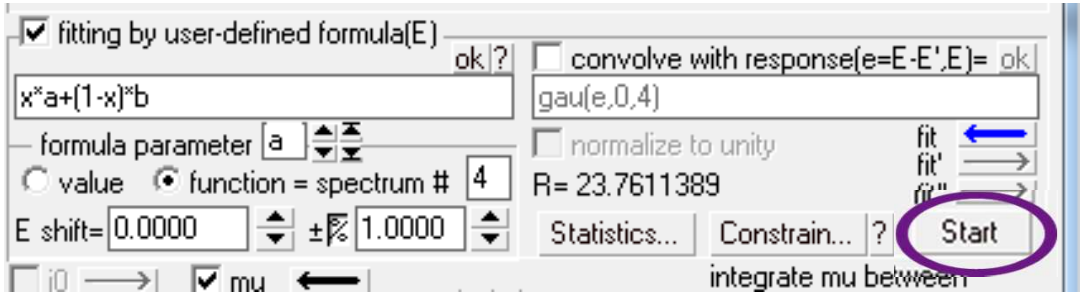
Set:

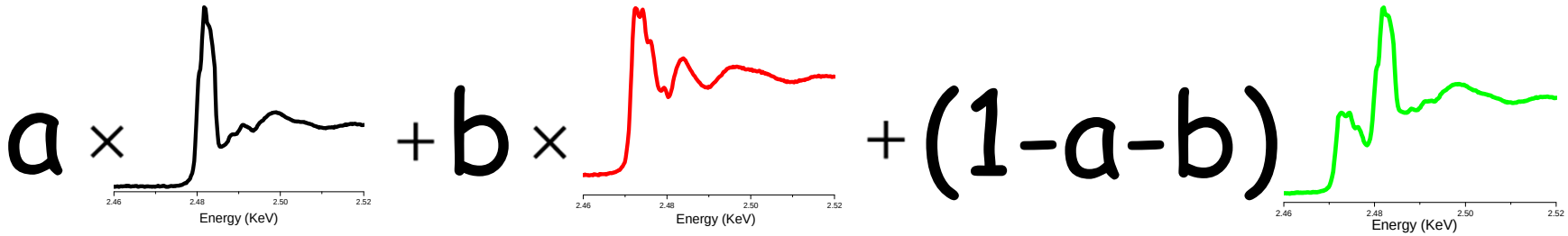


For XANES, normally -20ev and +50eV around the edge



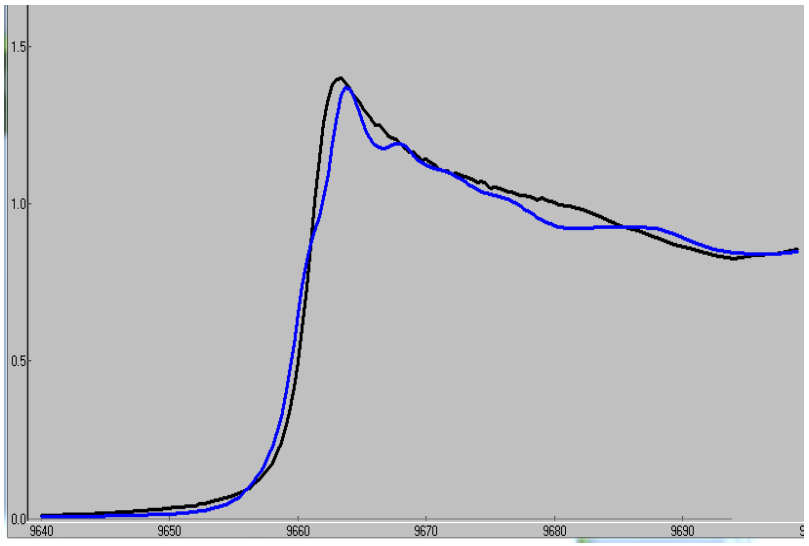
7. Fitting

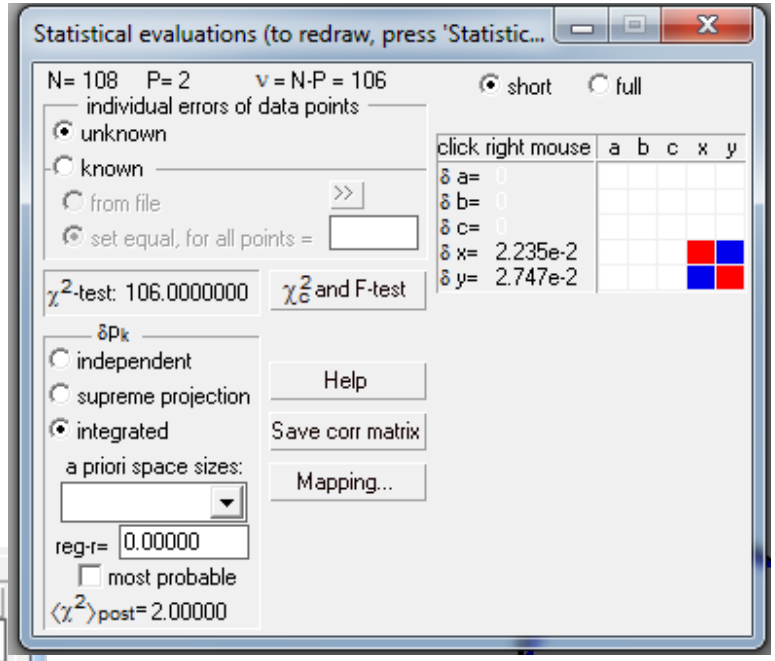


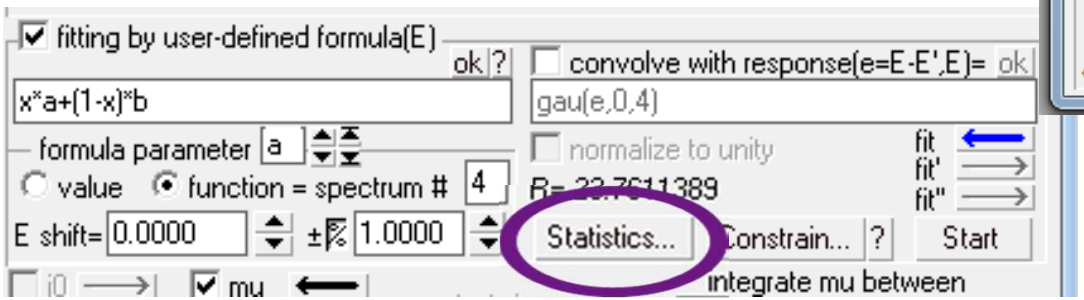


- Write a formula such as a*w+b*x+(1-a-b)*y, where w, x and y are reference spectra and a and b are values for the weight. (do not use z in a formula, it's the energy)
- Add constrains so the values of a and b are between 0 and 1: ain0..1 and bin0..1
- Add the desired initial increment for the fit (±value, in absolute numbers or percentage).
- In order to obtain a good fit, it is important to have the appropriate references.
- Press Start. It may take time.

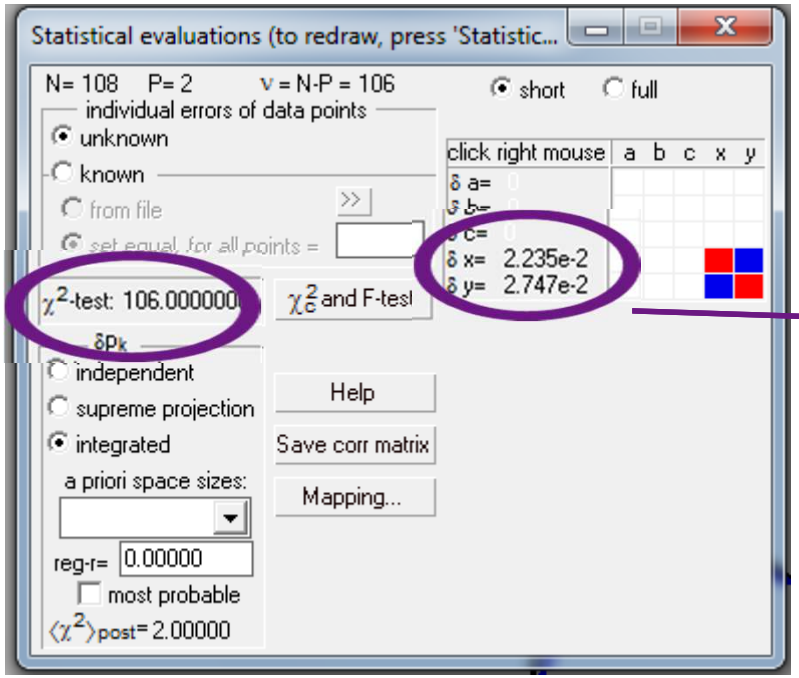
8. Errors


1.5
1.0
0.5
0.0
9640
9650
9660
9670
9680
9690


Statistical evaluations (to redraw, press 'Statistic...
N= 108 P= 2 ν = N-P = 106
short full
individual errors of data points
unknown
known
from file
set equal, for all points =
click right mouse a b c x y
δ a=
δ b=
δ c=
δ x= 2.235e-2
δ y= 2.747e-2
χ²-test: 106.0000000
χ²c and F-test
δpk
independent
supreme projection
integrated
a priori space sizes:
Help
Save corr matrix
Mapping...
reg-r= 0.00000
most probable
⟨χ²⟩post= 2.00000

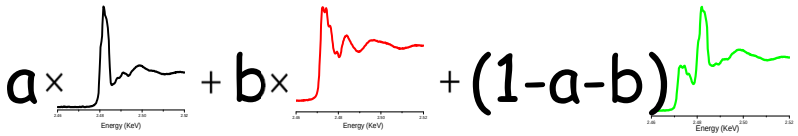

fitting by user-defined formula(E)
ok ?
x*a+(1-x)*b
formula parameter a
value
function = spectrum # 4
E shift= 0.0000
± 1.0000
convolve with response(e=E-E',E)= ok
gau(e,0,4)
normalize to unity
fit
fit'
fit''
Statistics...
Constrain...
?
Start
integrate mu between
mu

8. Errors



Right click and copy deltas.
Paste in a document
The ratios of the components and their confidence intervals are obtained.

a=3.0377e-1±2.416e-2 b=2.6122e-2±3.751e-2
w=0.0000±0000000 x=0.0000±0000000 y=0.0000±0000000



$$\chi^2 = \sum_i^N \left(\frac{d_i - t_i(k,p)}{\sigma_i}\right)^2$$

Goodness of the fit.
The lowest the better

9. Save

- Save everything as a project

- Save each spectra separately