

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>

Contacts:

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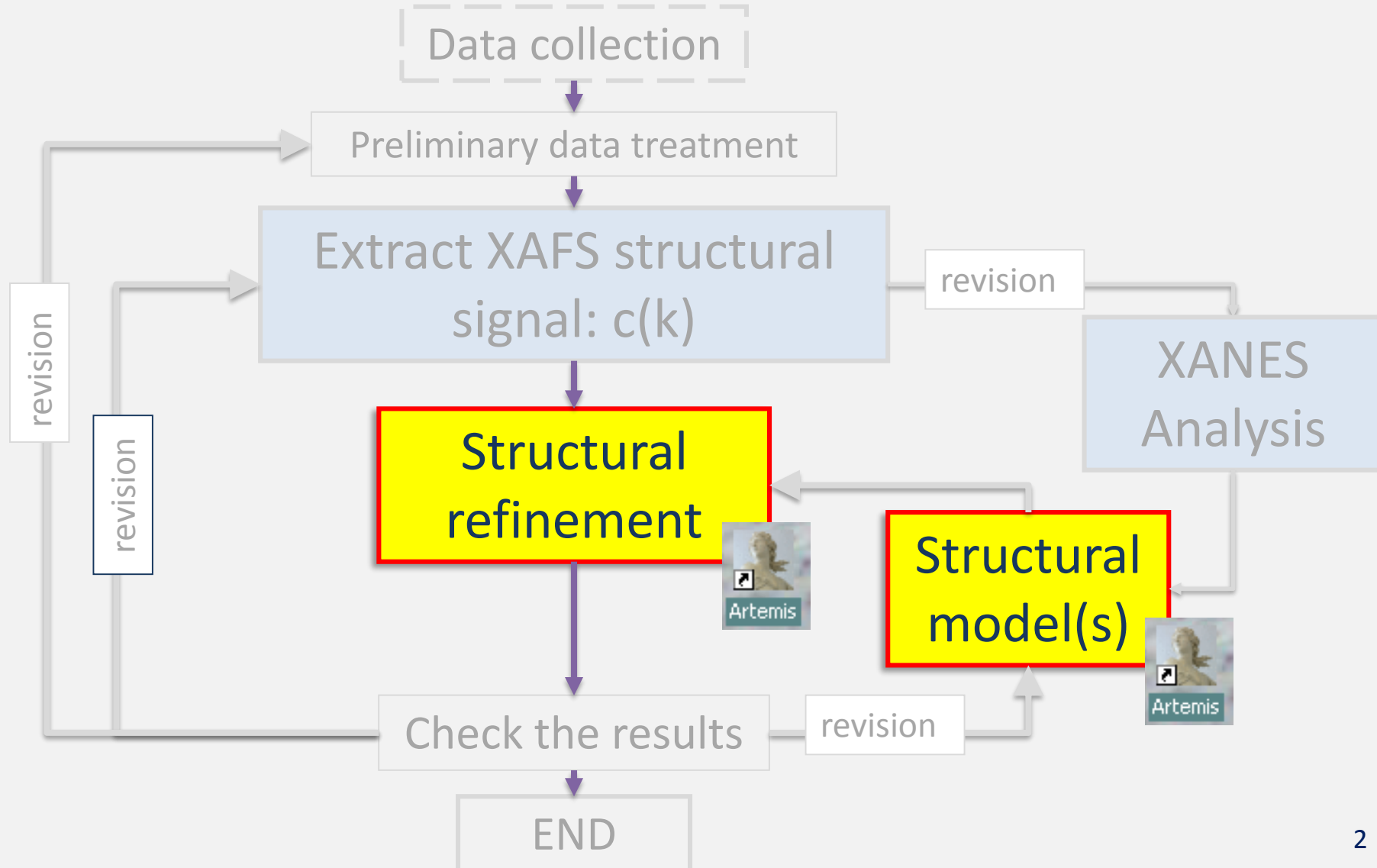
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1st on-line School on Synchrotron Radiation "Gilberto Vlaic":
Fundamentals, Methods and Application

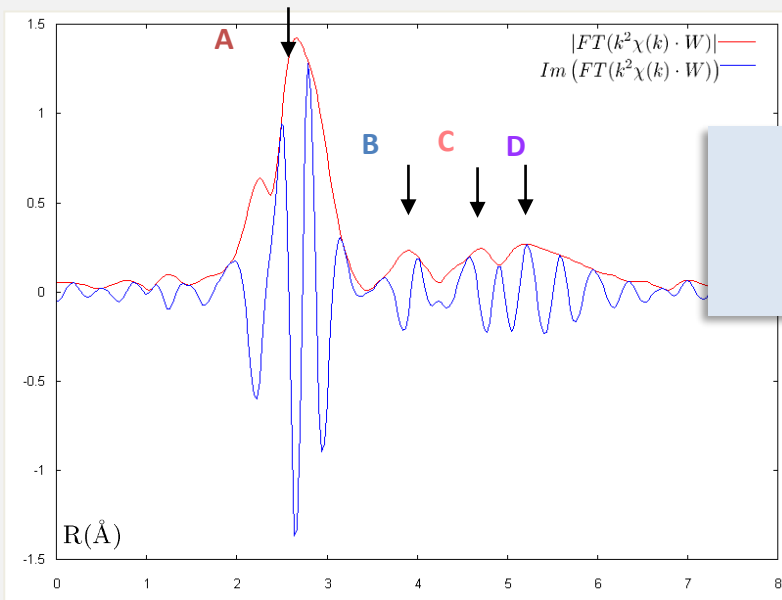
from the experimental data to structural details



The EXAFS standard formula

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

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

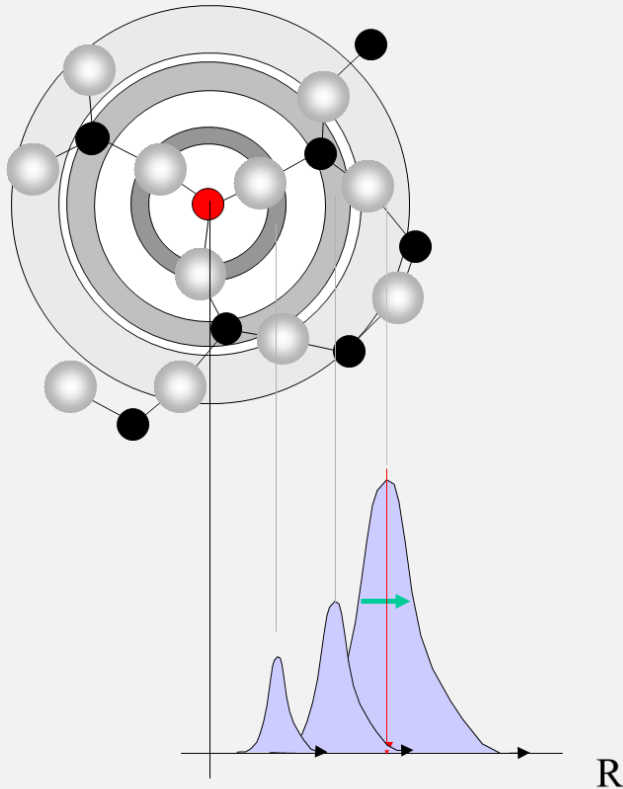


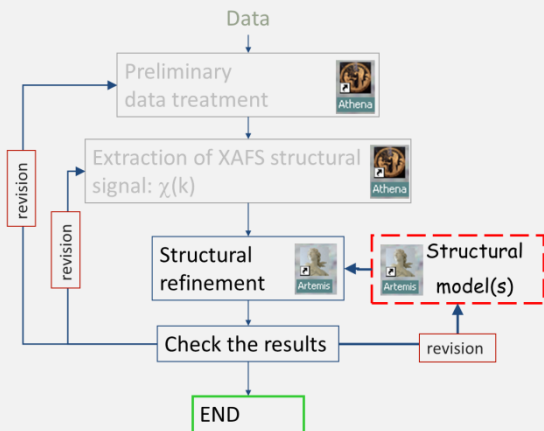
parameters

- f_j = photo-electron scattering amplitude
- ψ_j = photo-electron scattering phase
- λ = photo-electron mean free path
- S_o^2 = many body losses

Structure

- N_j = multiplicity (coordination number)
- r_j = half path length (coordination distance)
- σ_j^2 = variance of the path length distribution
(Mean Square Relative Displacement: MSRD)

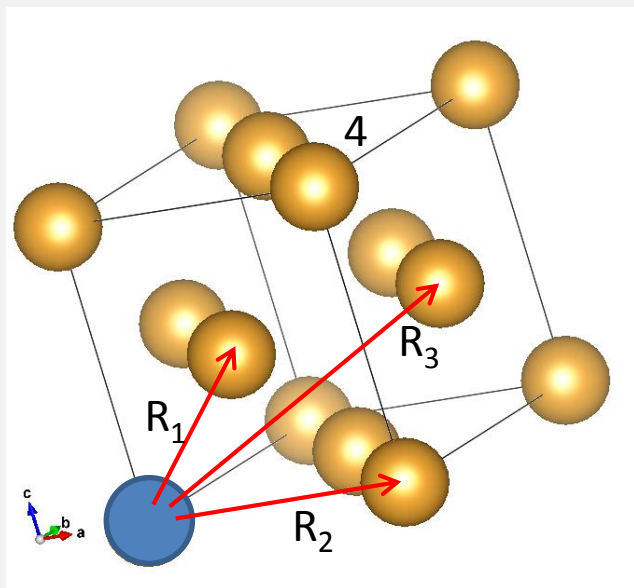




Structural model & Data refinement

We will analyze Cu EXAFS firstly

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



icsd_43493_Copper.cif

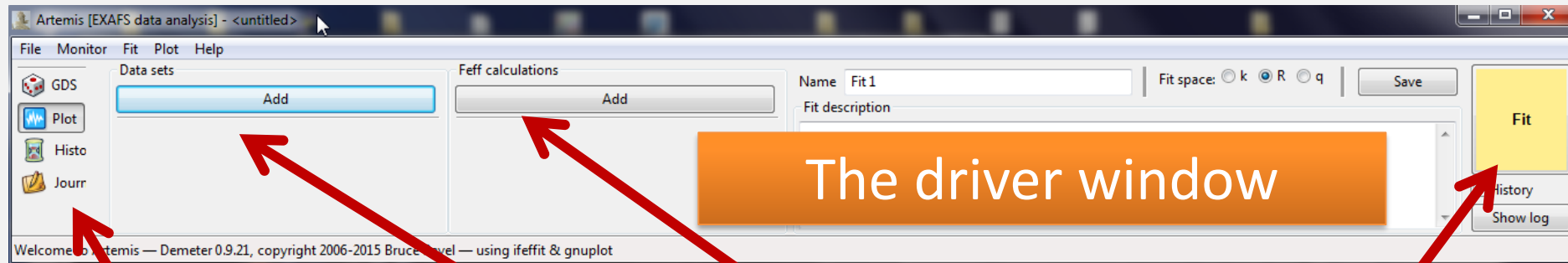
Cu metal
 SPG: fcc, f m 3 m (# 220)
 $a = 3.61 \text{ \AA}$
 Cu 0.0 0.0 0.0

Sh	R	N	$R_{Cu}(\text{\AA})$
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 Arthemis

better on la larger monitor!



3:select
**refinement
pram.**, plot,
etc...

1:add
EXAFS
 $\chi(k)$ or
athena
project

2:add a
Structure
.cif or Feff
file

4
Start fit



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

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 description

Fit

History

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

Artemis [Data] cu_foil_10k.d

Data Path Marks Add

cu_foil_10k.d

Data source: o_Malu_2015\data\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](#)

Transferred data set "cu_foil_10k.dat" to the plotting list.

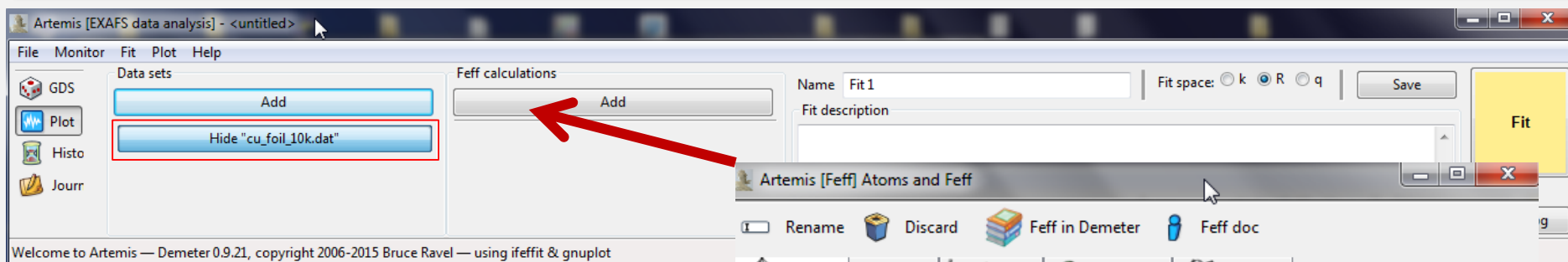
cu_foil_10k.dat in k space

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

Wavenumber (\AA^{-1})

Demeter 0.9.21 © Bruce Ravel

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

Rename Discard Feff in Demeter Feff doc

Atoms Feff Paths Path-like Console

Open file Save data

Titles

Copper Cu

Name: icisd_43493_Copper Margin: 0.03 Beta:

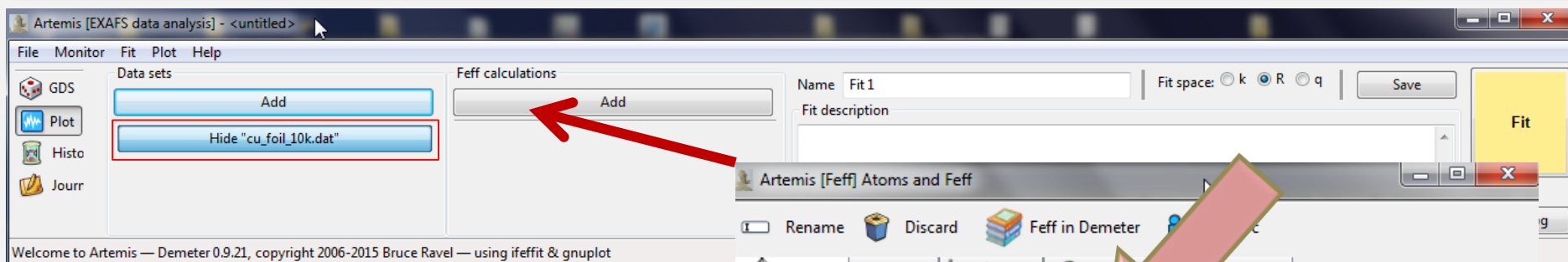
Feff input file

```
1 29 Cu

ATOMS * this list contains 55 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
```

	Core	EL	x	y	z	Tag
1	<input checked="" type="checkbox"/>	Cu	0	0	0	Cu1
2	<input type="checkbox"/>					
3	<input type="checkbox"/>					
4	<input type="checkbox"/>					
5	<input type="checkbox"/>					
6	<input type="checkbox"/>					

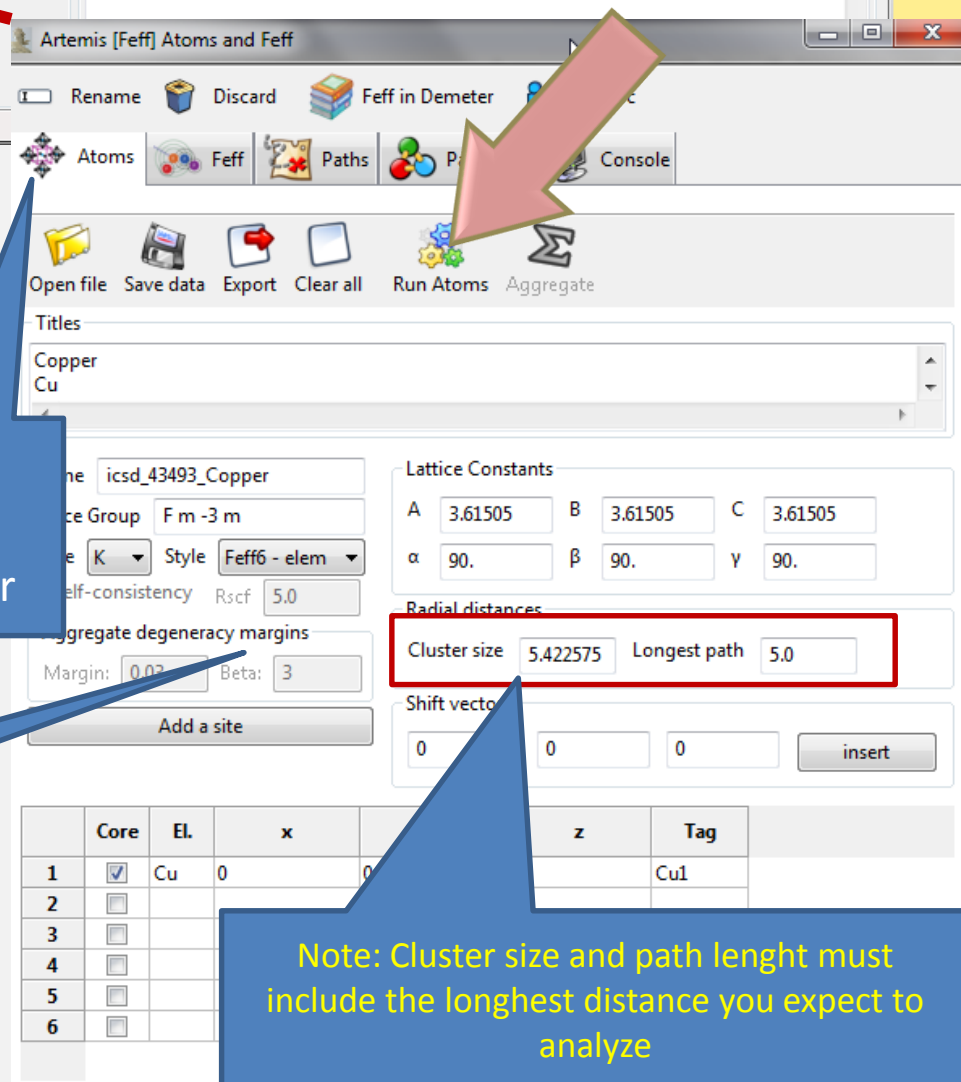
2: add model structure (cif or simple pair)



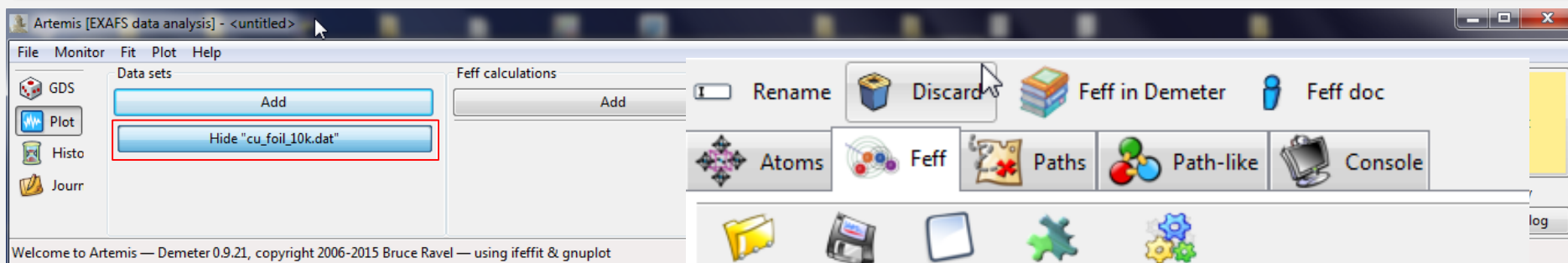
- I. Run Atoms
- II. check paths
- III. Run Feff
- IV. look at the path list

A)
generate a
local cluster

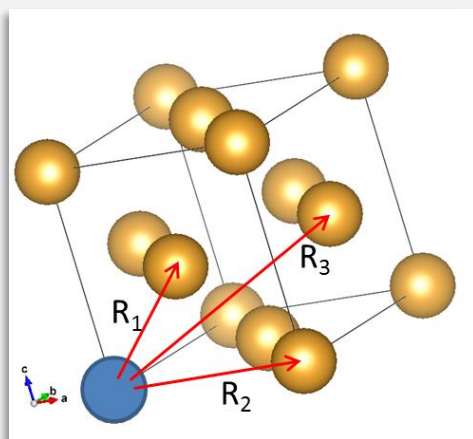
A)
input the unit cell
parameters and cluster size



2: add model structure (cif or simple pair)



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

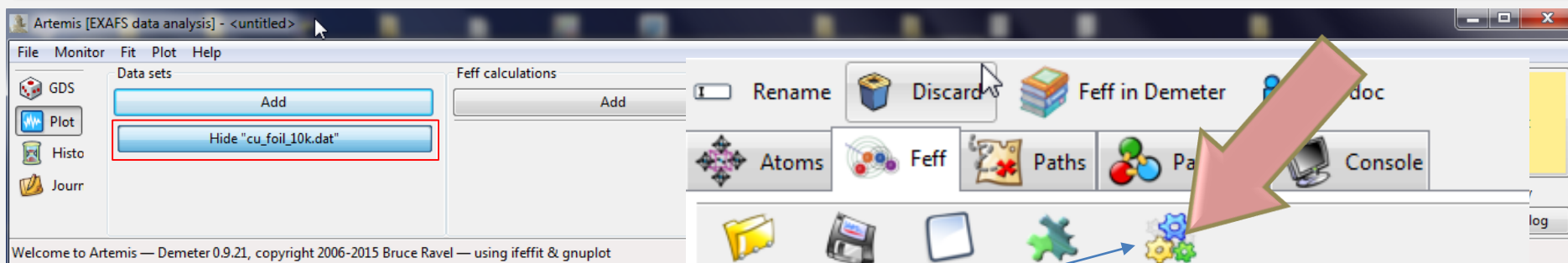


Name: Margin: Beta:

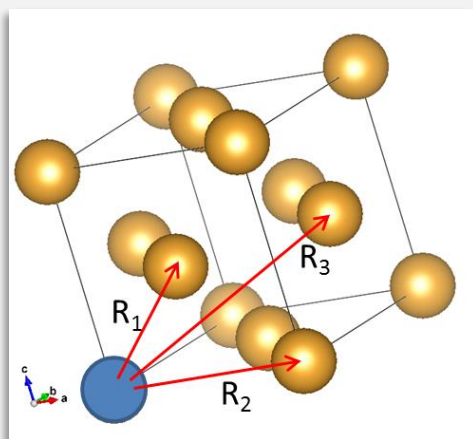
Feff input file

	1	29	Cu		
ATOMS					
*	x	y	z	iprot 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)



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



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

2: add model structure (cif or simple pair)

WARNING
This may
crashes!

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 doc

Atoms Feff Paths Path-like Consistent

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

Name of this Feff calculation: icstd_43493_Copper

Description

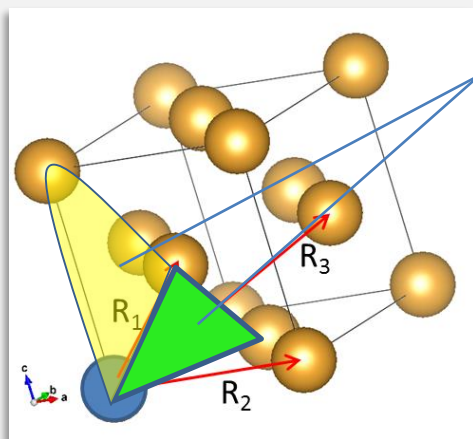
TITLE Copper
TITLE Cu
The central atom is denoted by this token: @
Cluster size = 6.00 Å, containing 86 atoms
31 paths were found within 6.000 Å
Forward scattering cutoff 20.00

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

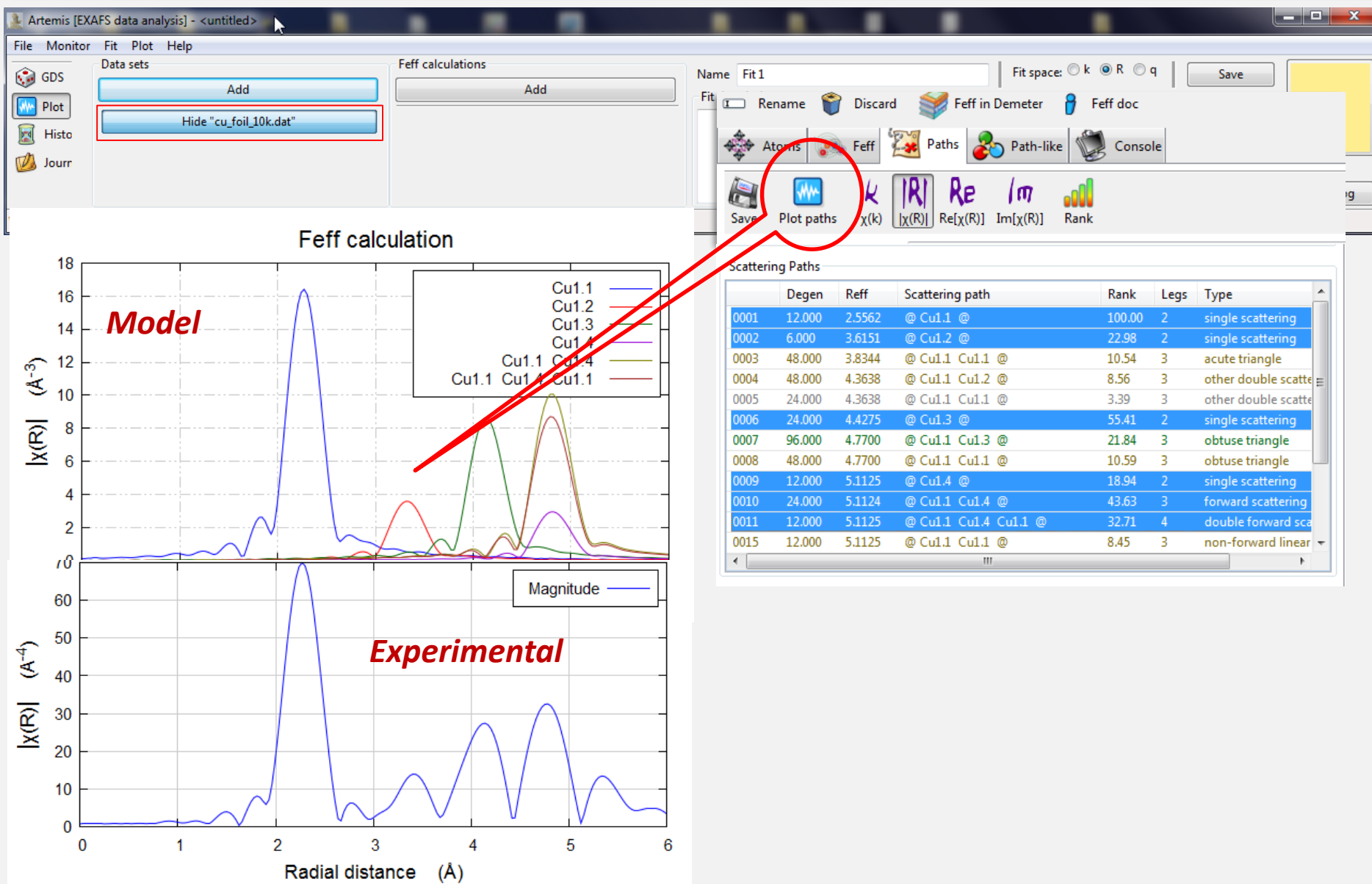
Welcome to Artemis — Demeter 0.9.21, copyright 2006-2015 Bruce Ravel — using ifeffit & gnuplot

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



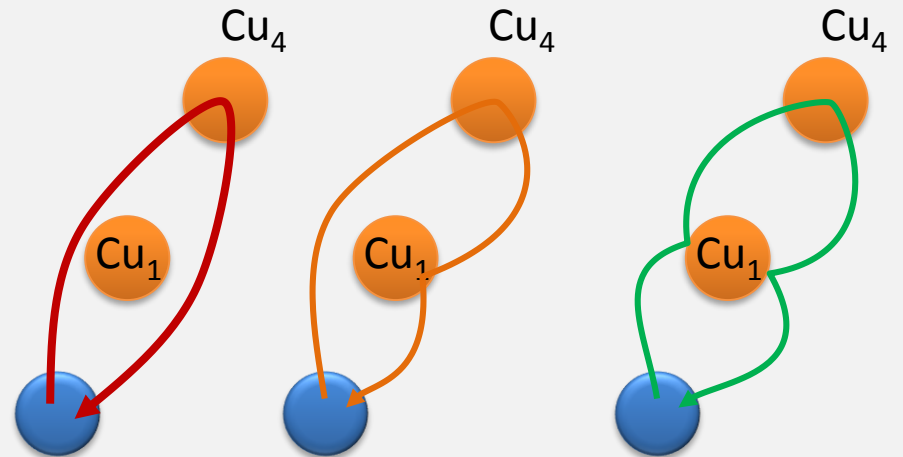
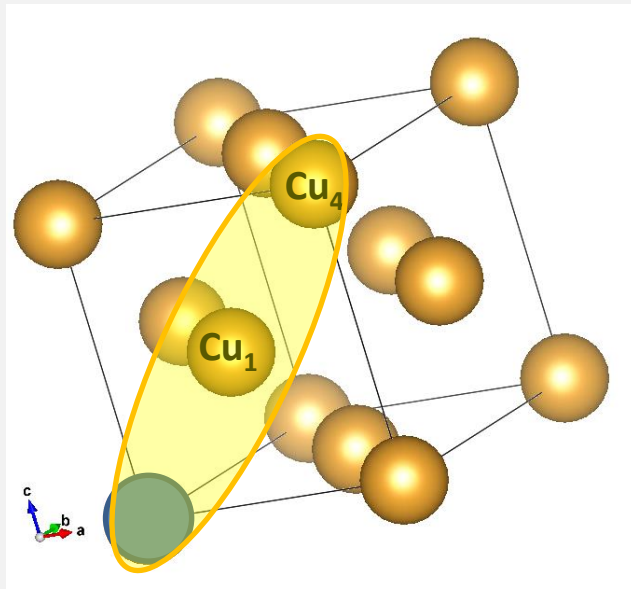
Path geometry

2: add model structure (cif or simple pair)



2: Check paths by relevance: **Multiple scattering**

Focusing effect enhances the multiple scattering from aligned configurations

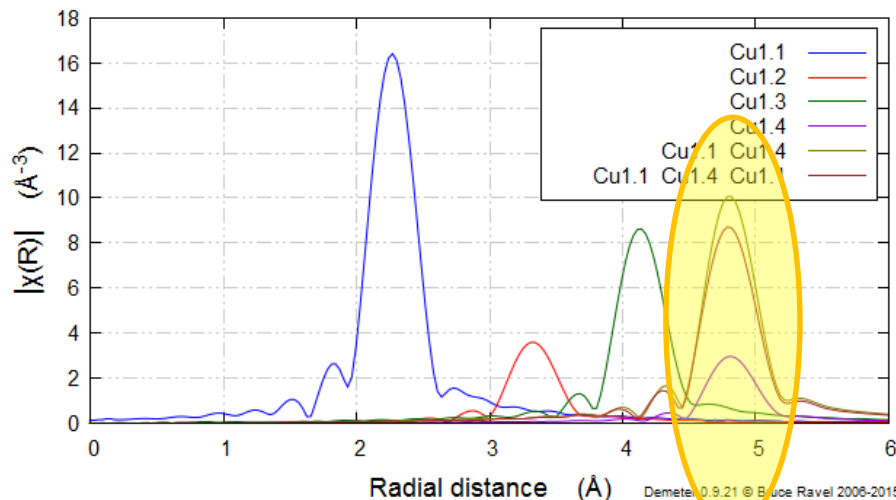


Cu1.4

Cu1.1-Cu1.4

Cu1.1-Cu1.4-Cu1.1

Feff calculation



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 @	10.39	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

2: drag and drop relevant paths to the data window

The screenshot displays the Artemis software interface, which is used for EXAFS data analysis. The main window, titled 'Artemis [Data] cu_foil_10k.dat', shows the 'Data' tab selected. The 'Data source' is set to 'trieste2021\Training\Data\Cu\EXAFS_extraction\athena_Cu10k.prj, 1'. The 'Plot this data set as' section includes buttons for 'k123', 'R123', 'Rmr', 'Rk', and 'kg'. The 'Title lines' section is empty.

Below the main window, the 'Artemis [Feff] Atoms and Feff' window is open. It shows the 'Paths' tab selected. The 'Name of this Feff calculation' is 'icsd_43493_Copper'. The 'Description' section contains the following text:

```
# 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
```

The 'Scattering Paths' table is visible, showing a list of paths with columns for Degen, Reff, Scattering path, Rank, and Type. The first row is highlighted in blue:

Degen	Reff	Scattering path	Rank	Type
1	12.00	@ Cu1.1 @	10.11	single scatter
2	6.00	@ Cu1.2 @	22.97	single scatter
3	48.00	@ Cu1.1 Cu1.1 @	10.56	acute triangle
4	48.00	@ Cu1.1 Cu1.2 @	8.54	other double
5	24.00	@ Cu1.1 Cu1.1 @	3.39	other double
6	24.00	@ Cu1.3 @	55.37	single scatter
7	96.00	@ Cu1.1 Cu1.3 @	21.64	obtuse triangle
8	48.00	@ Cu1.1 Cu1.1 @	10.61	obtuse triangle
9	12.00	@ Cu1.4 @	18.92	single scatter
10	24.00	@ Cu1.1 Cu1.4 @	43.54	forward scatter

A blue arrow indicates the process of dragging a path from the 'Scattering Paths' table in the 'Atoms and Feff' window to the 'Data' window. The path selected is '[icsd_43493_Copper] Cu1.1'.

2: Define parameters

NOTE: DE and So2 should be the same for absorbers in the same phase

The screenshot shows the software interface for [icsd_43493_Copper] Cu1.1. It includes checkboxes for 'Include path' and 'Plot after fit', and a checkbox for 'Use this path for phase corrected plotting'. Below these, a table shows scattering data for (1) single scattering, high (100.00). The table has columns x, y, z, and ipo, with values 1.807530, 1.807530, 0.000000, and 0.000000 respectively. A right-click context menu is open over the 'DEo' parameter, showing options: 'Guess DEo', 'Def DEo', 'Set DEo', 'Lguess DEo', and 'Skip DEo'. A yellow callout points to the right-click action, and a blue callout points to the 'DEo' parameter.

x	y	z	ipo
1.807530	1.807530	0.000000	
0.000000	0.000000	0.000000	

Reff=2.556, nleg=2, degen=12

Label: N=12, S0²=1

Parameters: DEo, dr_1, ss_2, ...

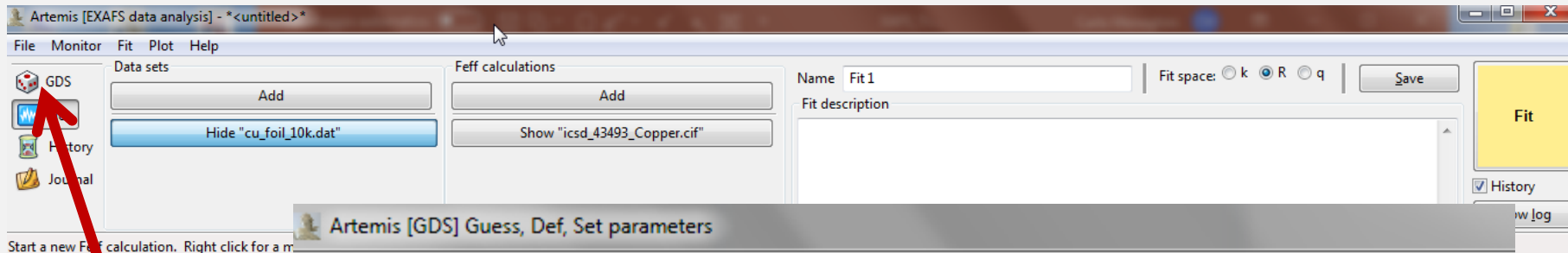
Context menu options for DEo:

- Guess DEo
- Def DEo
- Set DEo
- Lguess DEo
- Skip DEo

right click

You must provide a name to each parameter you want refine

3: Define the refinement parameters



Start a new Feff calculation. Right click for a m

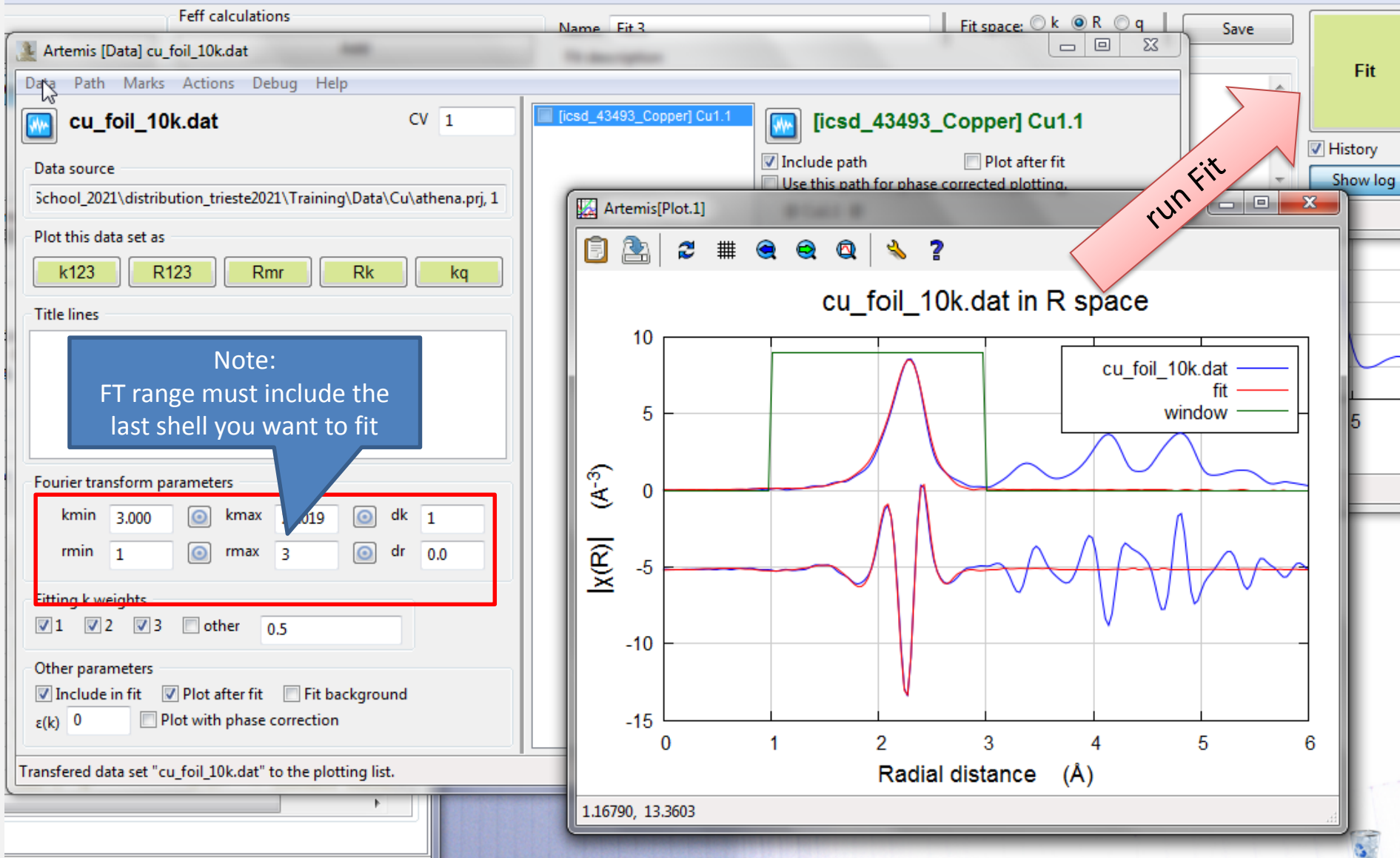
Artemis [GDS] Guess, Def, Set parameters				
	Type	Name	Math expression	Evaluated
1	guess	DEo	0	
2	guess	dr_1	0	
3	guess	ss_2	0.00300	
4	guess			
5	guess			

Note:
Initialize to > 0
the σ^2 parameters

3 GDS:
define
refinement
parameters



3: Best fit



4: FIT



```
Artemis [Log] Fit 1

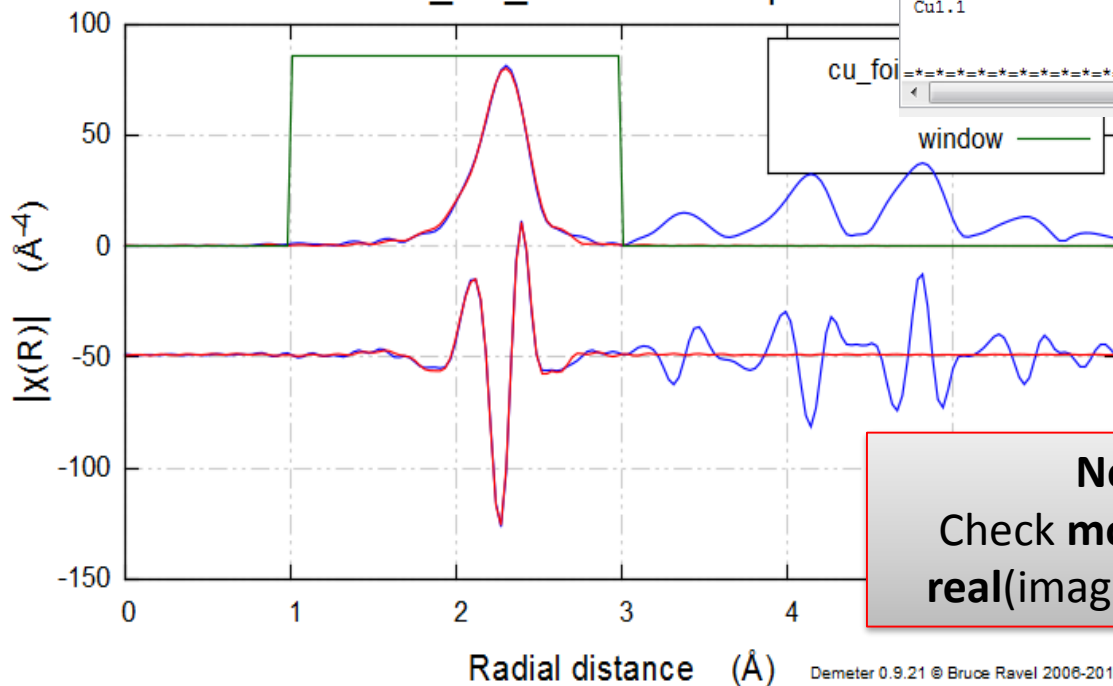
: Athena project      = C:\Users\carlo\Dropbox\Seminario_Malu_2015\dati\ok_Cu_Foil\EXAFS_est
: name                = cu_foil_10k.dat
: k-range              = 3.000 - 23.019
: dk                   = 1
: k-window             = hanning
: k-weight             = 1,2,3
: R-range              = 1 - 3
: dR                   = 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

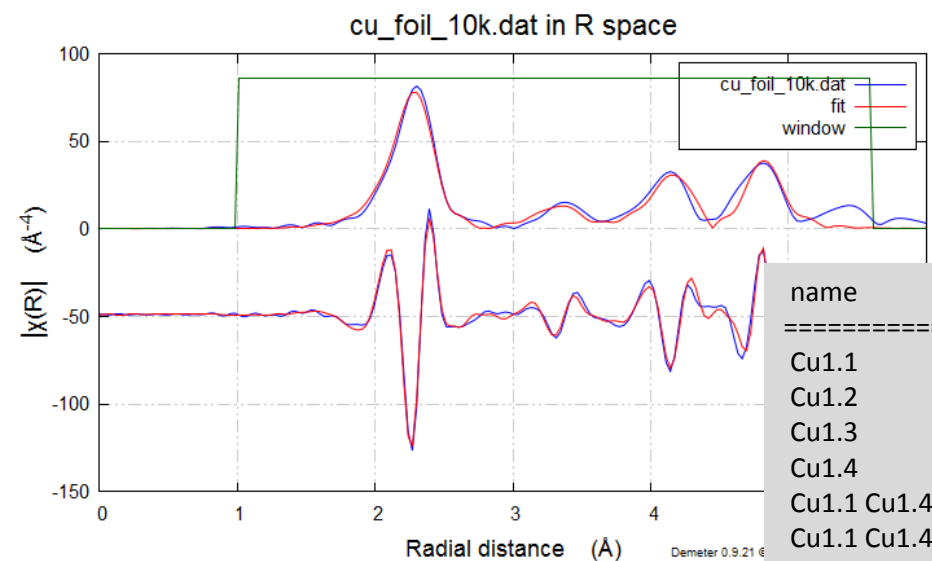
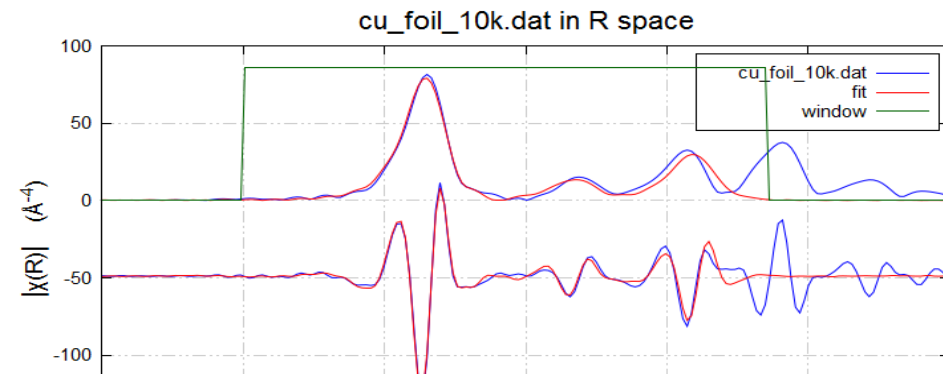
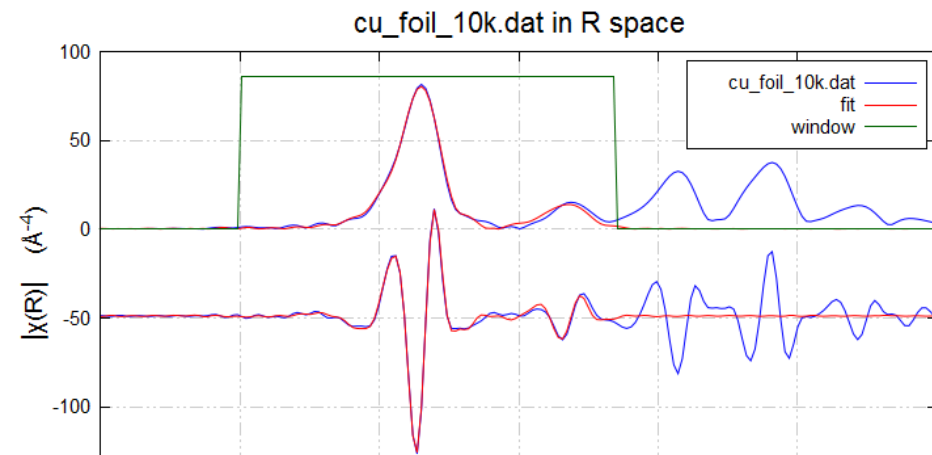
```
=====
<=====
```

cu_foil_10k.dat in R space



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

4: FIT, extend the analysis to further shells



Fourier transform parameters

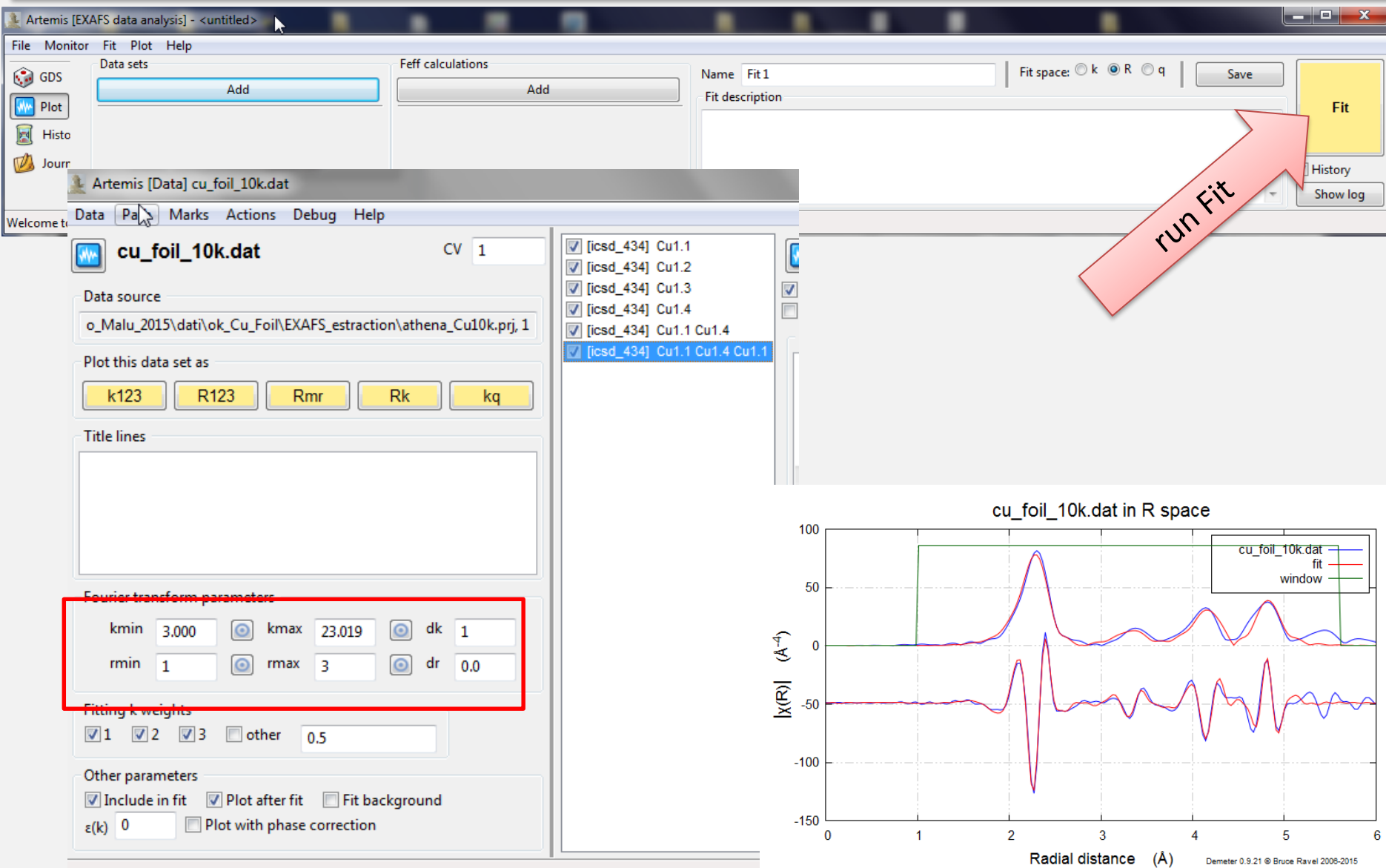
kmin	3.000	<input checked="" type="checkbox"/>	kmax	23.019	<input checked="" type="checkbox"/>	dk	1
rmin	1	<input checked="" type="checkbox"/>	rmax	3.7	<input checked="" type="checkbox"/>	dr	0.0

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

Note:
extend your fit but: a large enough windows must be defined in the Athena project

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

3: Define parameters



4: Check best fit log file

Note:

Keep correlations among the parameters low, maybe add physical meaning constraints.

Far away shell parameters are generally less accurate, use them with care

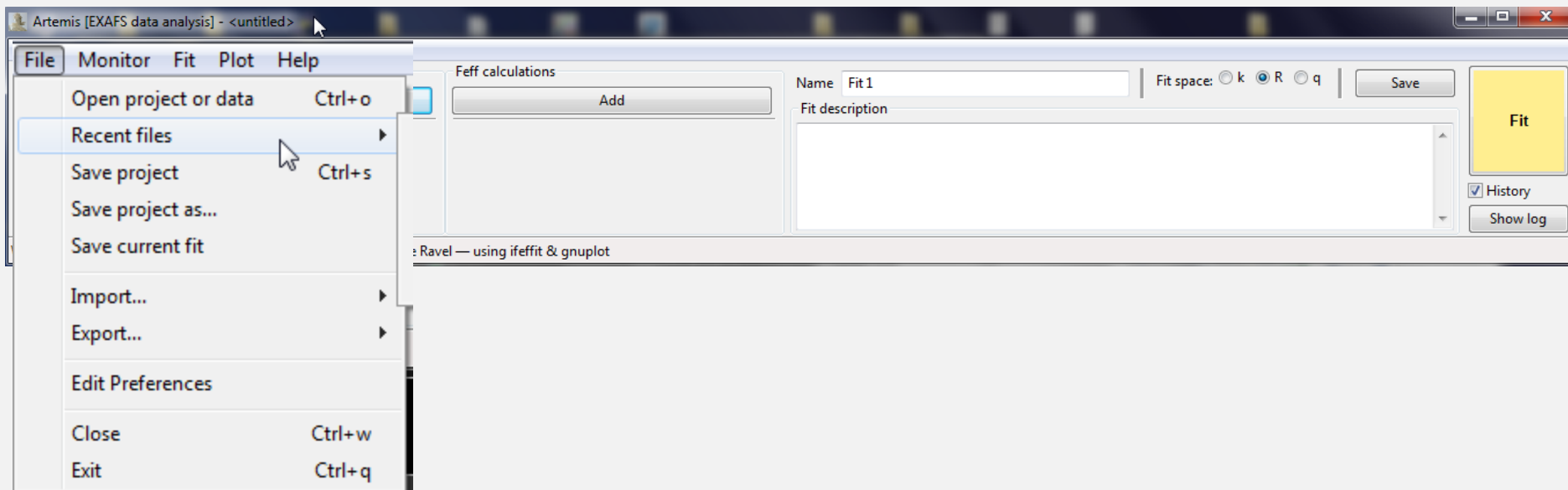
Correlations between variables:

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...



Your Exercises

Analyze the Fe structure

