Basics of EXAFS Data Processing

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Honeywell

Data processing overview

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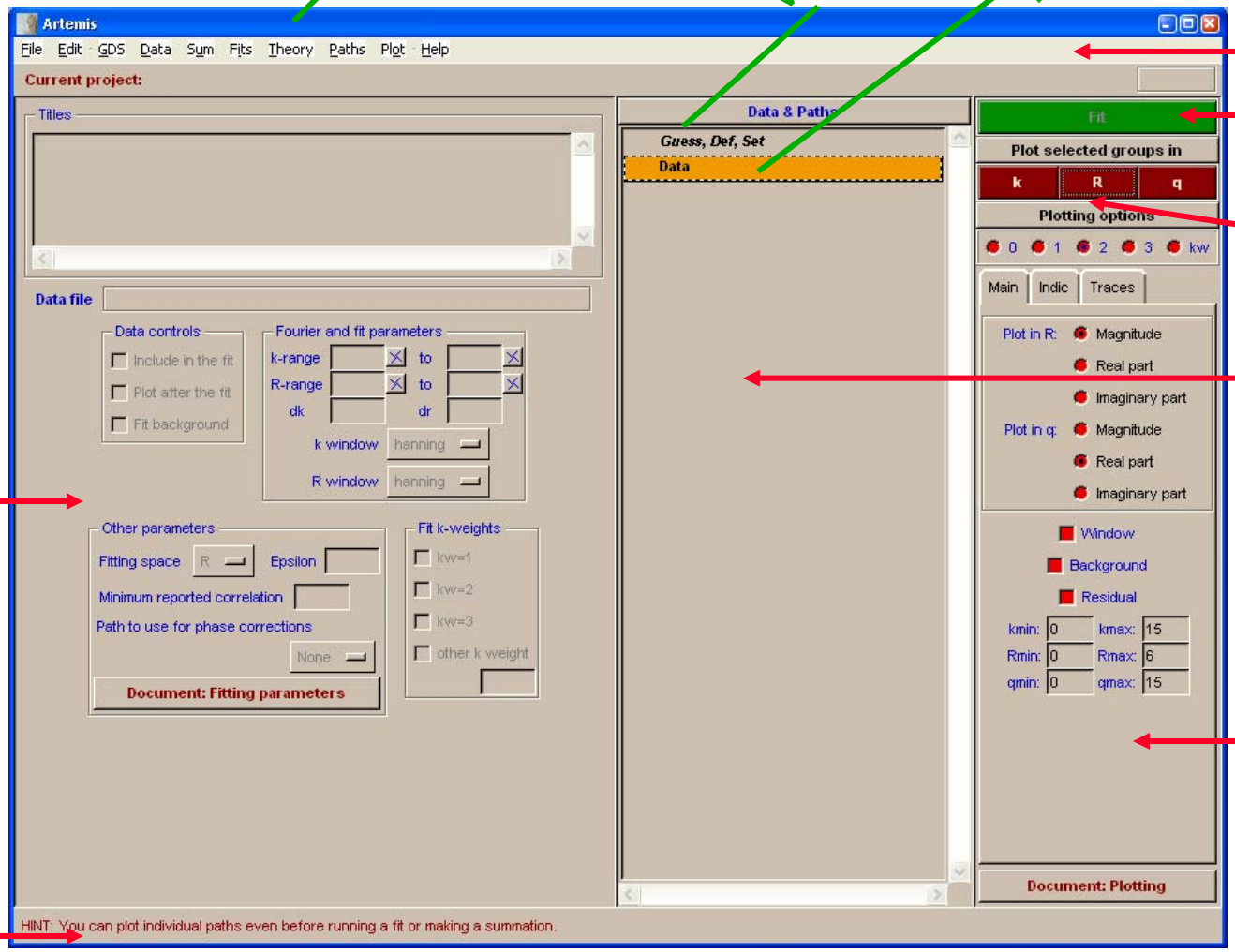
- **Introduction to Artemis**
- **Modeling Cu foil**
- **Background subtraction using theory**
- **Modeling U to determine neighboring atom type**
- **Multiple data set modeling**

- M Newville. "IFEFFIT: interactive EXAFS analysis and FEFF fitting." J. Synch. Rad. **8**: pp 322-324, 2001. http://cars9.uchicago.edu/ifeffit/
- J J Rehr."*Ab initio* multiple scattering X-ray absorption fine structure and X-ray absorption near edge structure code". University of Washington**:** pp, 1995. http://leonardo.phys.washington.edu/feff/
- E A Stern, M Newville, B Ravel, Y Yacoby and D Haskel. "The UWXAFS analysis package: Philosophy and details." Physica B **208 & 209**: pp 117-120, 1995.

Artemis



Project Name:

Menus:

Fit: optimize variables

Plot and Do buttons:

Data display area: Changes depending on selected information from Data and Paths list

Data and Paths: Changes data display area

Plotting parameters

Echo Area: Messages from Artemis

Reading in χ Data

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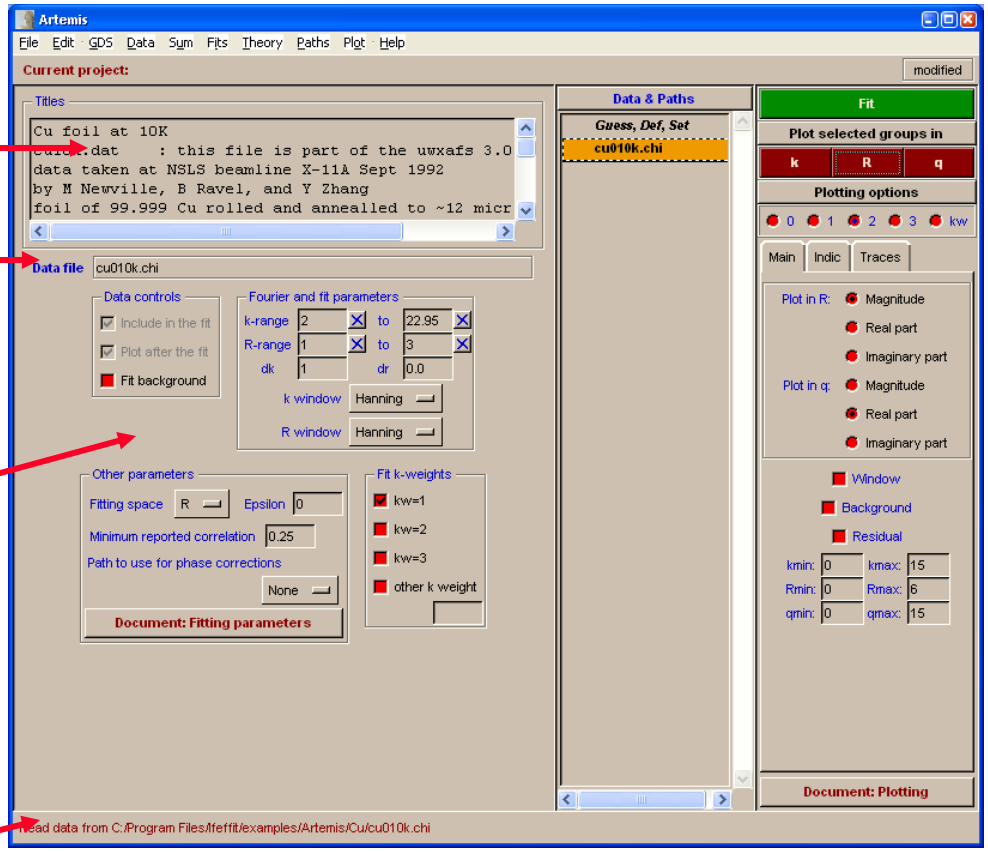
➢File: open data file: F:\Ifeffit\examples\Artemis\Cu\cu010k.chi

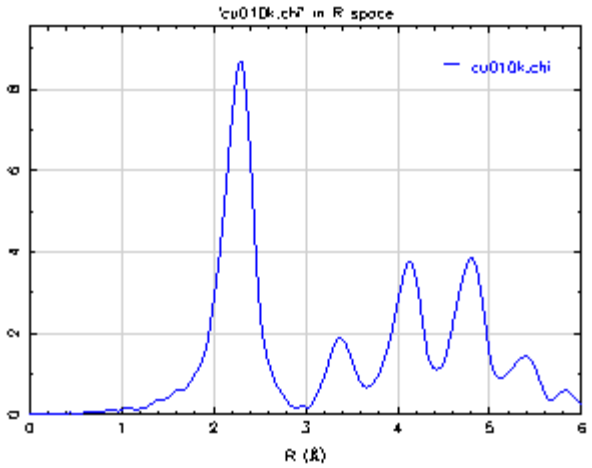
Title lines from data file

data file name

Fourier transform parameters



Message from Artemis



Data plotted in R-space

Reading Data from Athena Project

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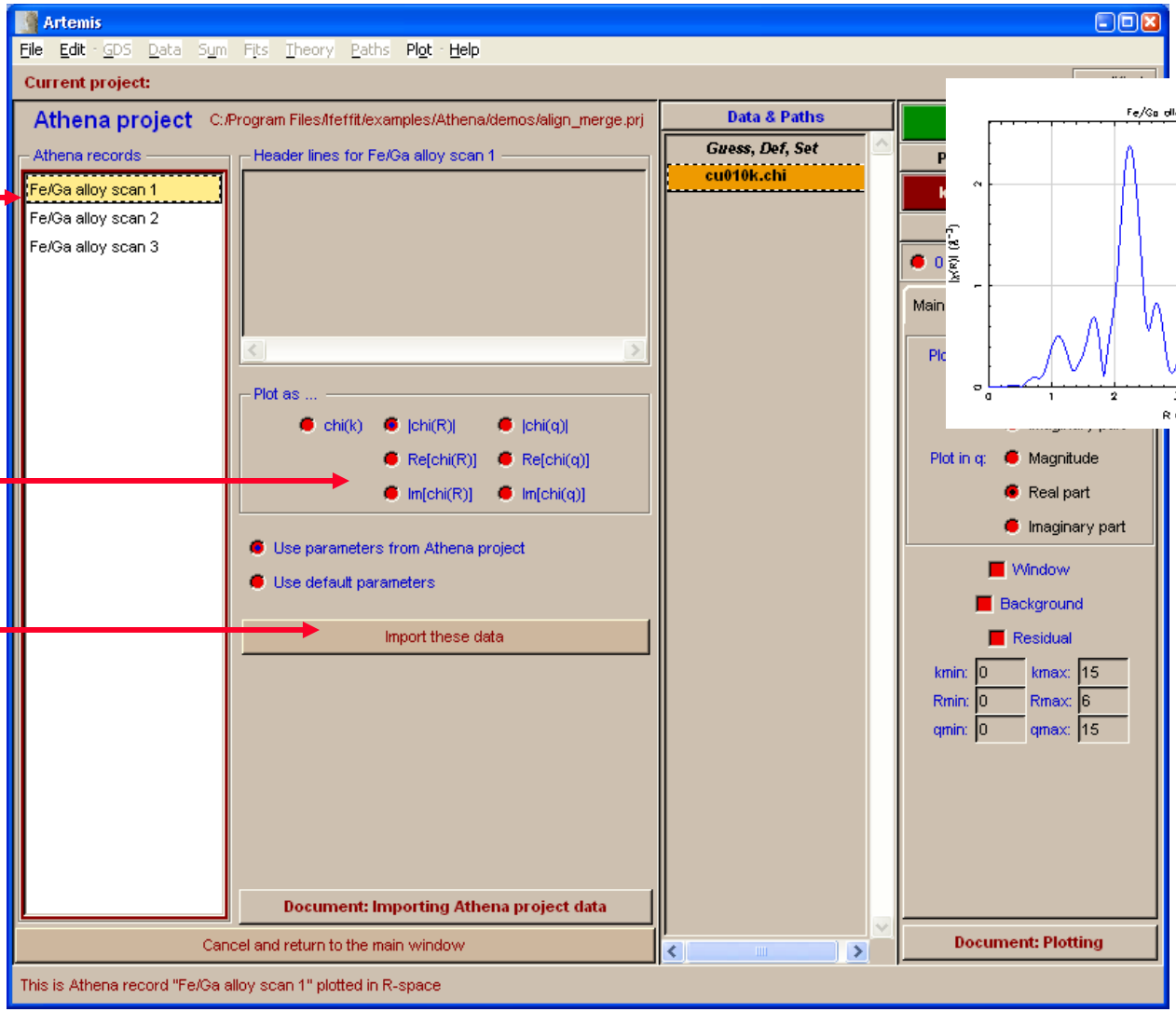
➢File: open data file: F:\Ifeffit\examples\Athena/demos/align_merge.prj

list of data from Athena

Plot the data

Import the selected data set



Atoms page

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➢File: open file: F:\lfeffit\examples\Artemis\Cu\atoms.inp

Atoms Tab

Title lines

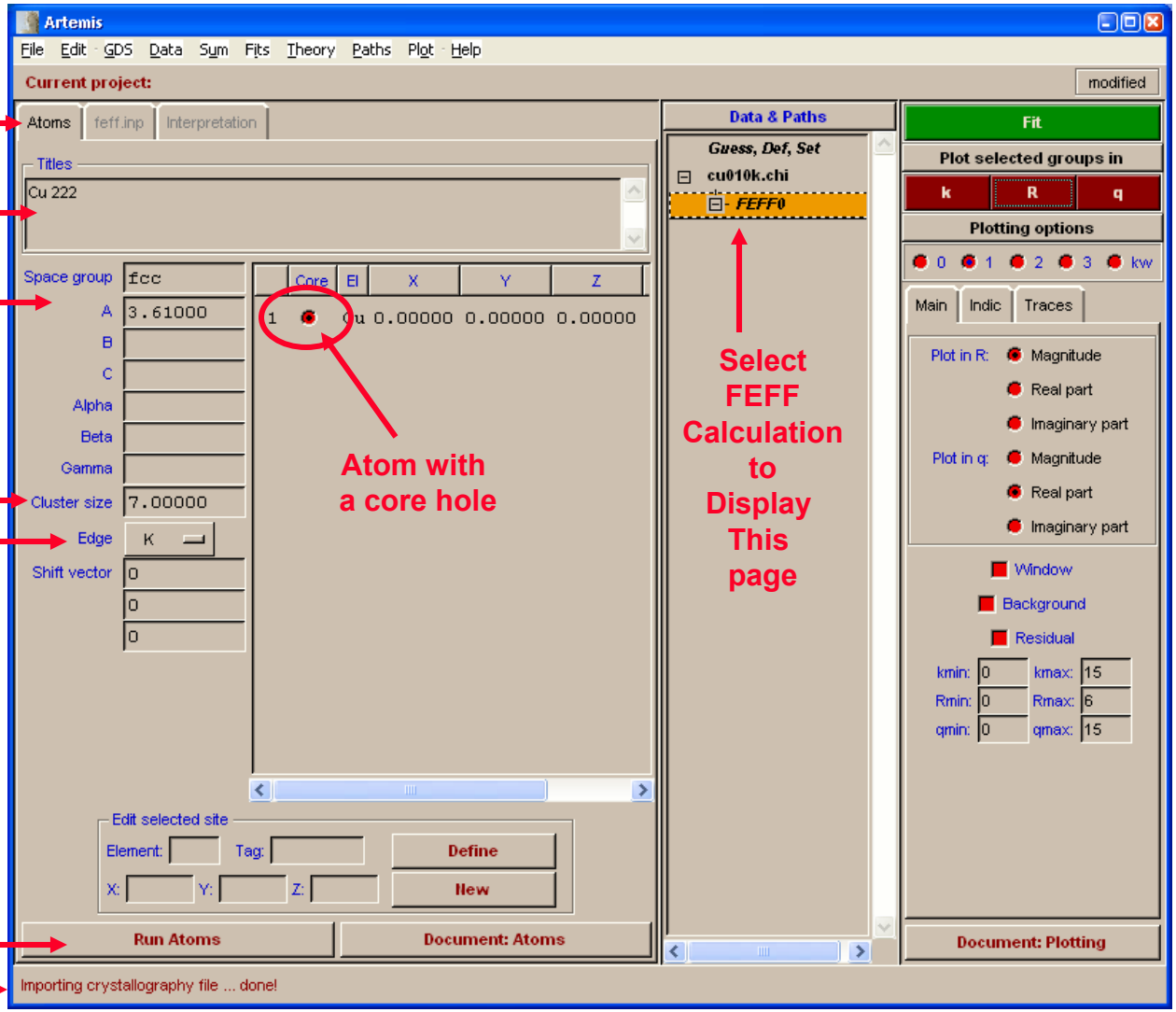
Structural Information

Always much larger than paths used in fit

Absorption edge

Make input (feff.inp) for theoretical calculation (FEFF)

Message from Artemis



Theory input page (feff.inp)

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➢Click “Run Atoms”

Feff.inp Tab

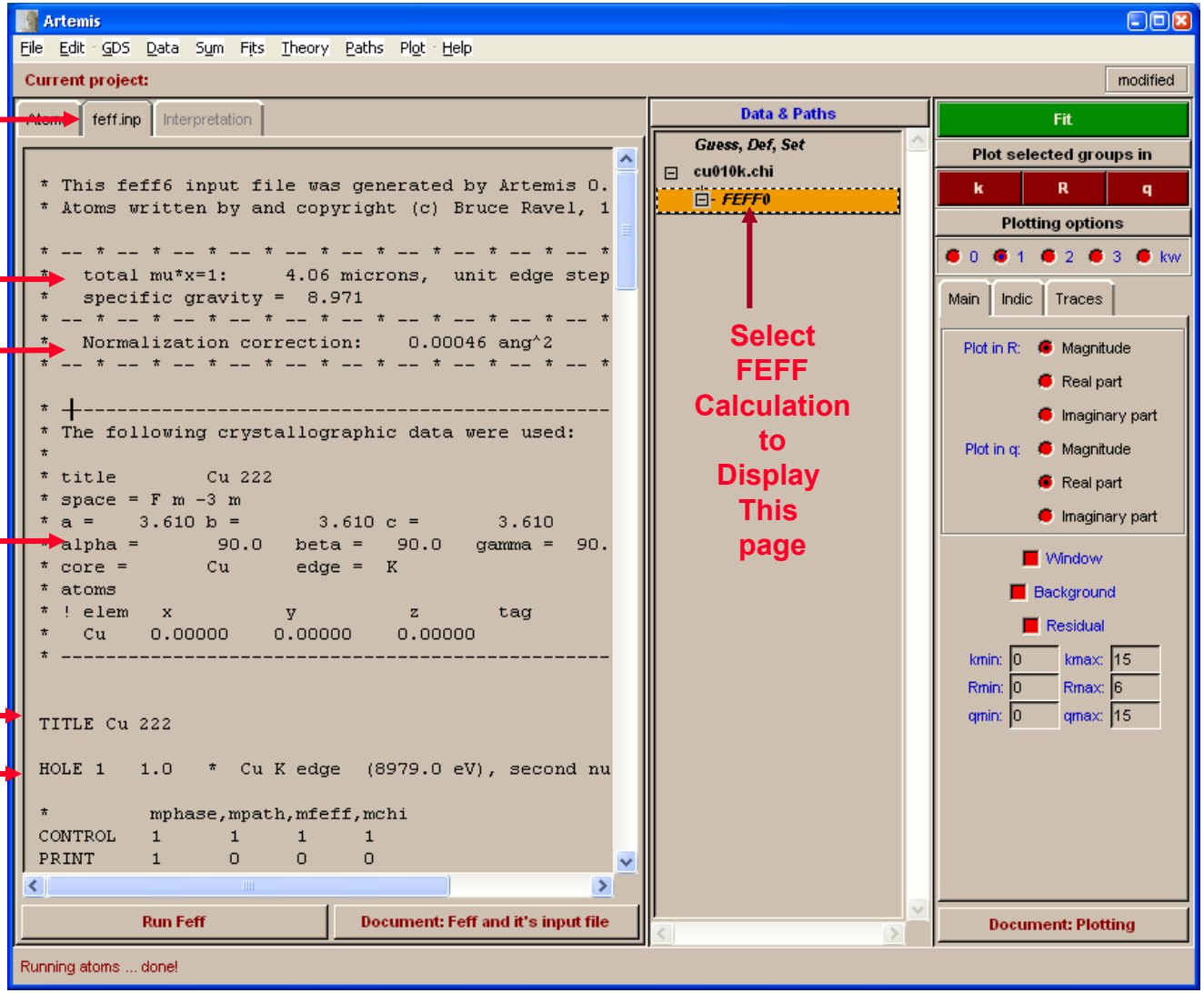
Absorption lengths

normalization correction

Crystallographic information From atoms.inp

title lines

hole number



Theory input page (feff.inp)

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➢Scroll feff.inp page downward

Feff.inp Tab

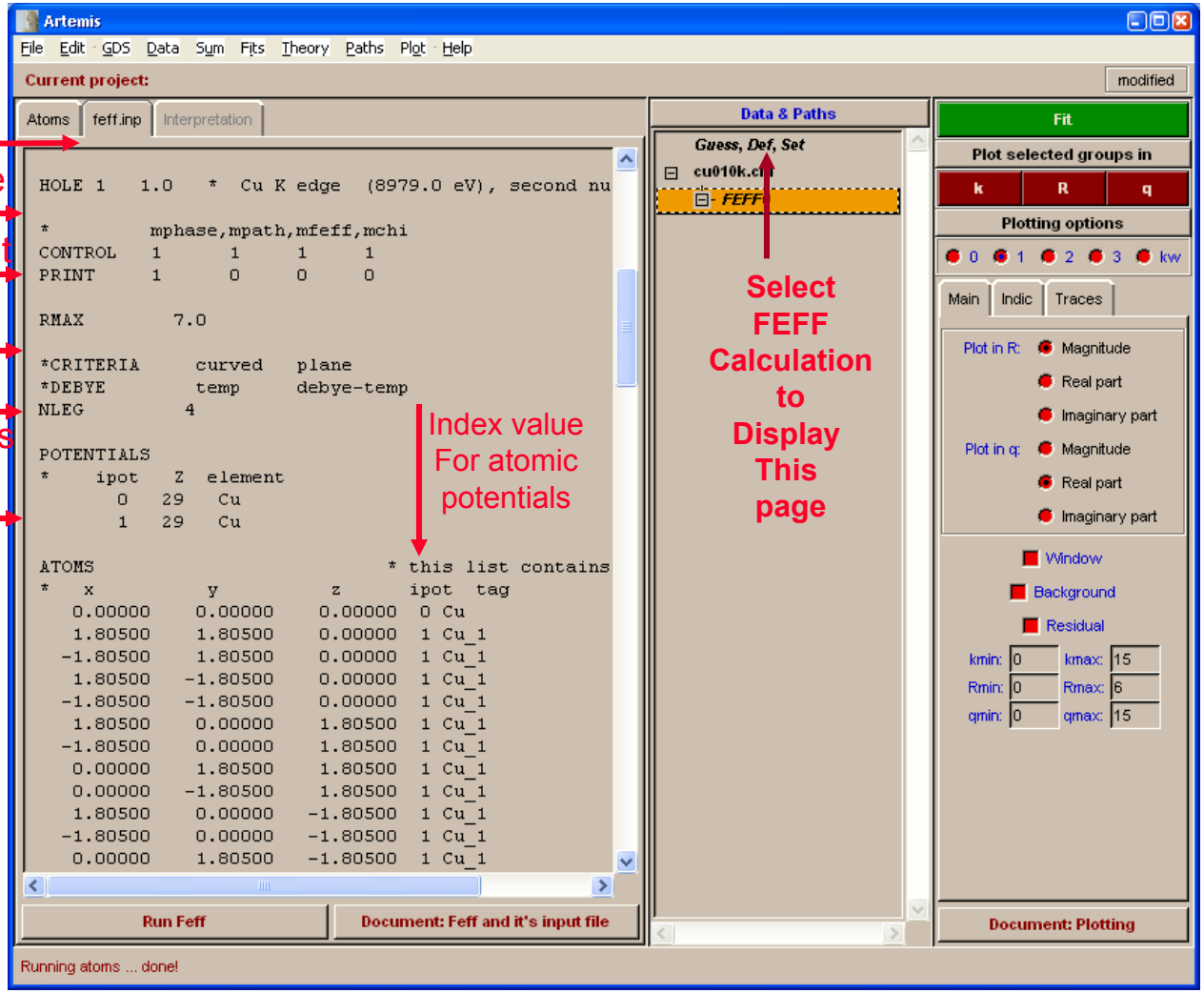
Core Hole and S02 value

Toggle switches for different FEFF modules and output max path length

Control over the number of output paths

Atomic potential index list

List of atomic positions



Theory input page (feff.inp)

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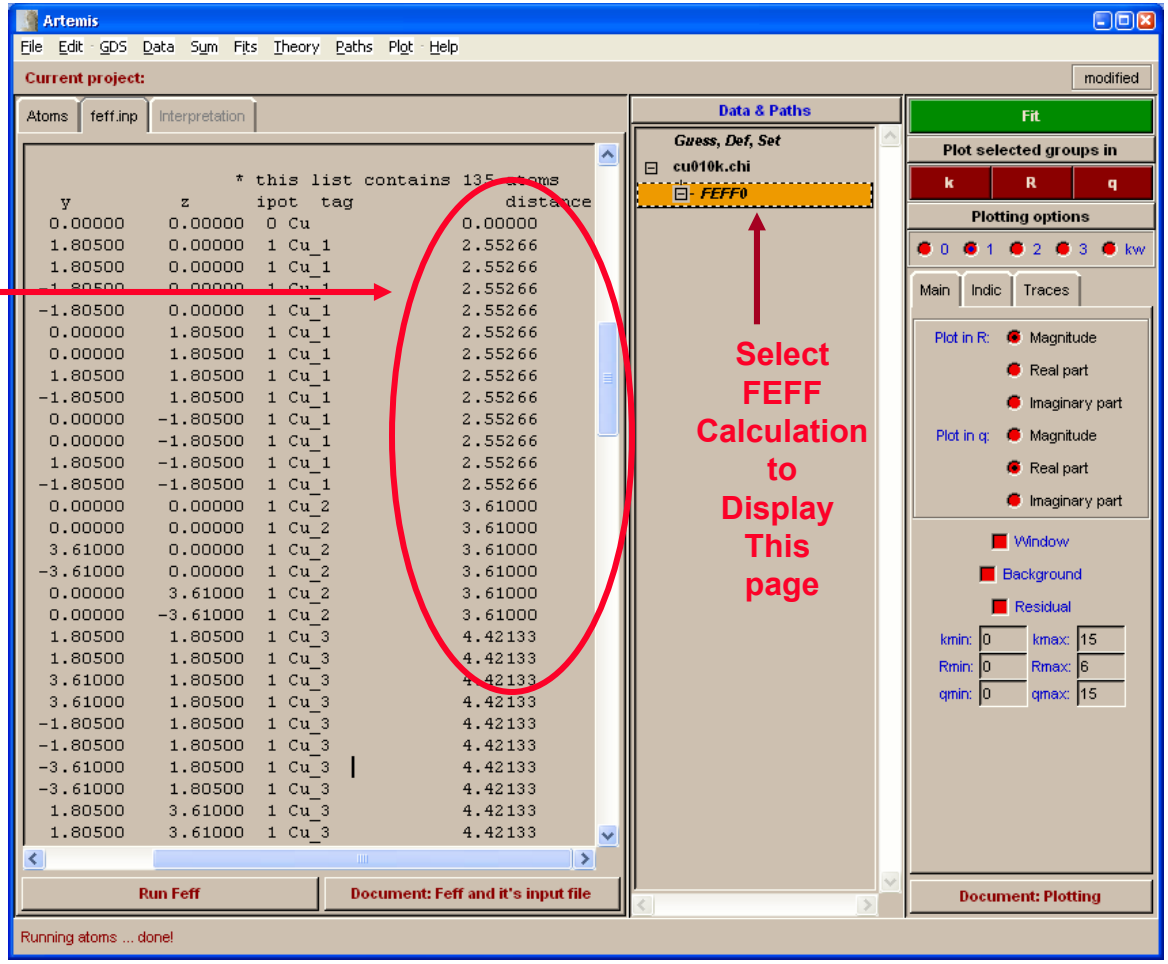
- Scroll feff.inp page downward and to the right

Check that the atomic Distances from the absorbing atom Are reasonable



Running Feff

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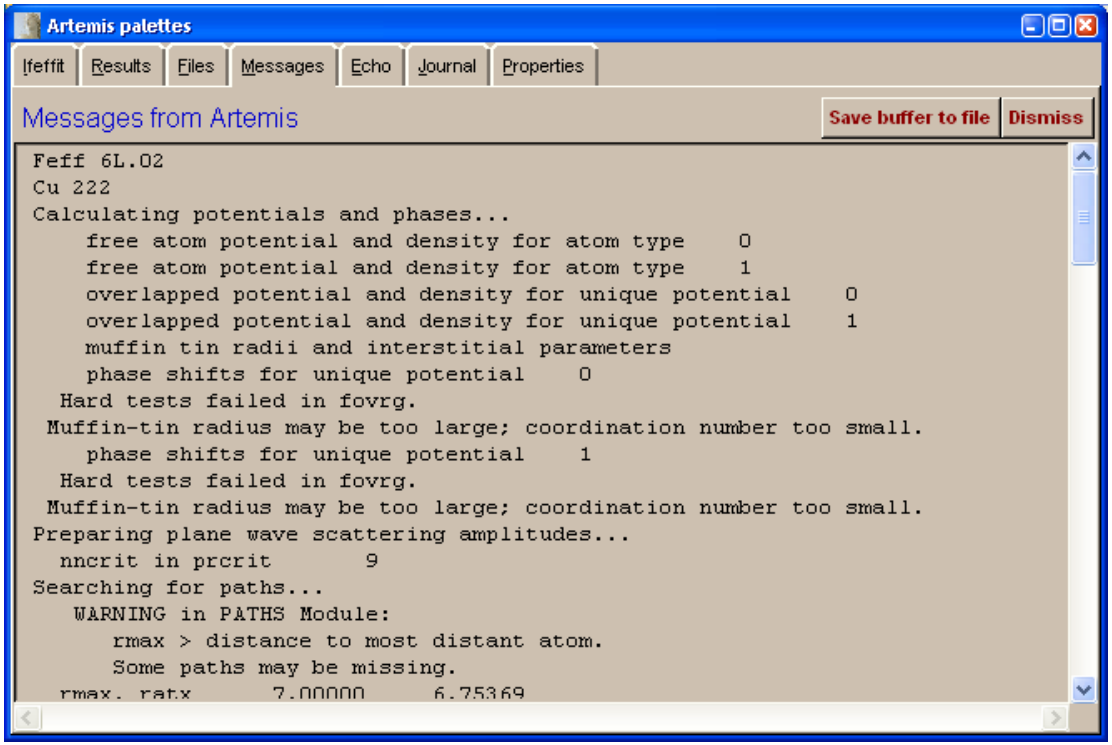
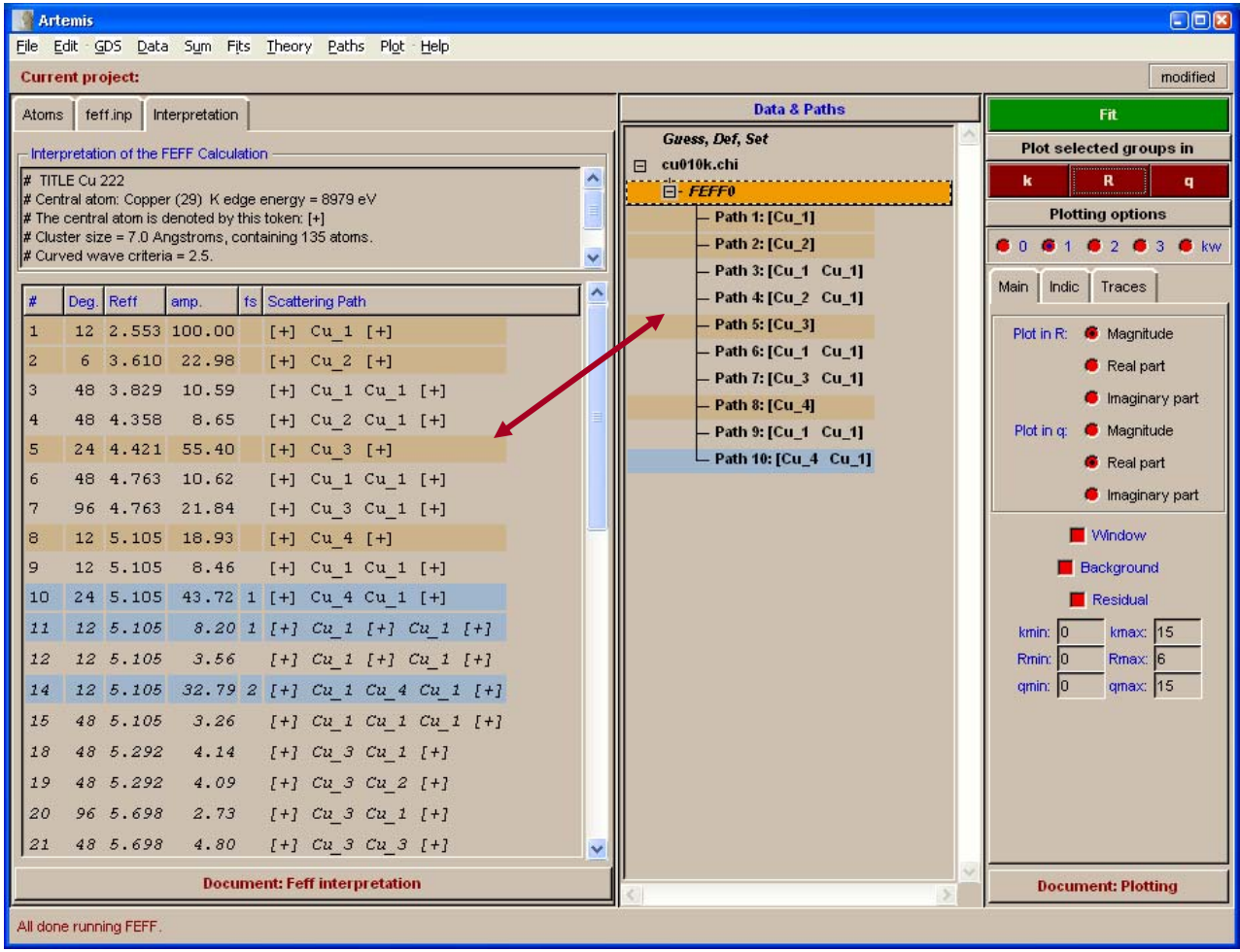
Text messages during Feff calculation

- **Artemis Palettes**
  - **Ifeffit: Shows the interface from Artemis to Ifeffit. Artemis is just a nice interface to Ifeffit. Ifeffit does the work!**
  - **Results: Shows the results page from a fit of the theory to the data.**
  - **Files: Shows data files.**
  - **Messages: Shows output from Feff.**
  - **Echo: Shows entire message from Artemis Echo area**
  - **Journal: Useful place to make notes.**
  - **Properties: Notes about the current project file.**



Feff paths: feffxxxx.dat

- **Degen:** Degeneracy of the path (number of identical scattering paths)
- **reff:** Inital half path length (bond length for single scattering path)
- **amp:** Estimate of amplitude of path relative to first path.
- **fs:** Number of forward scattering events.
- **scattering path:** atoms scattering photoelectron, [+] symbol represents core atom.



S I Zabinsky, J J Rehr, A Ankudinov, R C Albers and M J Eller. "Multiple-scattering calculations of X-ray-absorption spectra." Phys. Rev. B **52**(4): pp 2995-3009, 1995.

Path Description

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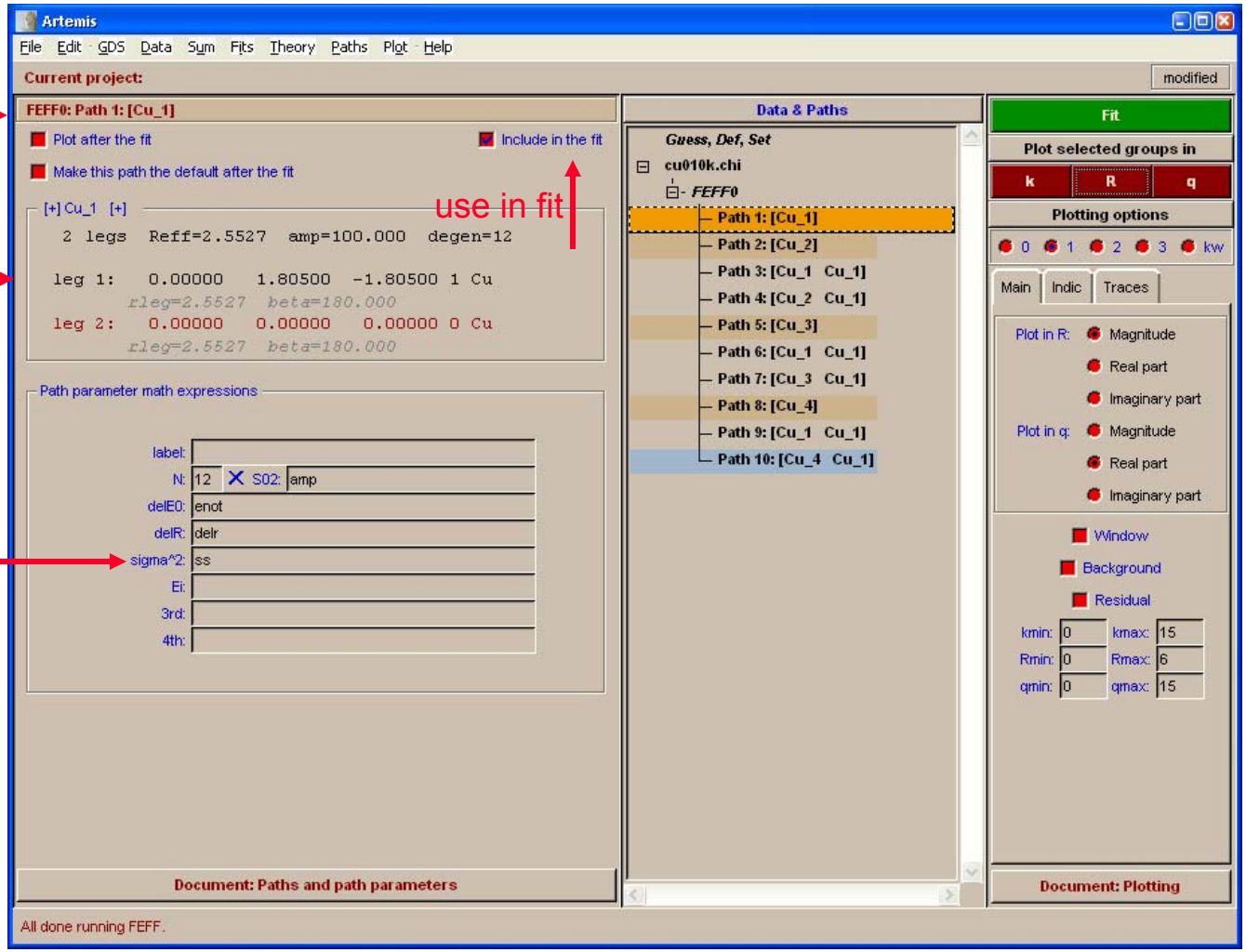
➢Click on Path1 in the Data & Paths list

Feff calculation

Path description

parameters



The EXAFS Equation

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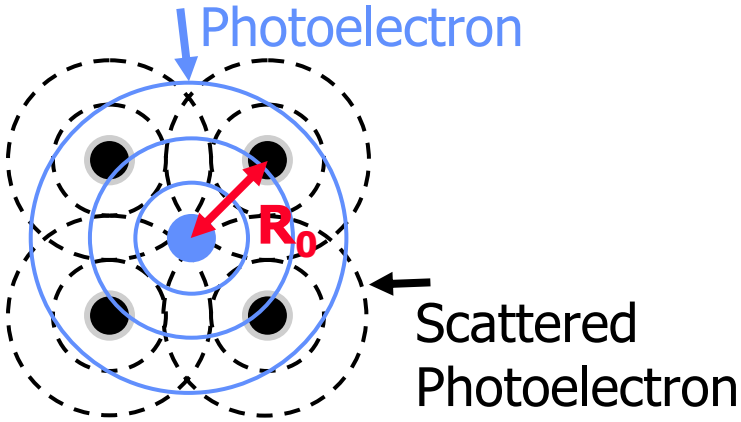
- E. A. Stern and S M Heald Basic principles and applications of EXAFS. Handbook of Synchrotron Radiaction. E. E. Koch. New York, North-Holland. **10:** pp 995-1014, 1983.
- E. A. Stern. "Theory of the extended x-ray-absorption fine structure." Phys Rev B **10**(8): pp 3027-3037, Oct 1974.
- E A Stern. "Structural determination by X-ray Absorption." Contemp. Phys **19**(4): pp 239-310, 1978.



$$\chi(k) = \Sigma_i \chi_i(k)$$

with each path written as:

$$\chi_i(k) = \left(\frac{(N_i S_0^2) F_i(k)}{kR_i^2} \sin(2kR_i + \varphi_i(k)) \exp(-2\sigma_i^2 k^2) \exp(-2R_i/\lambda(k)) \right)$$

$$R_i = R_0 + \Delta R$$

$$k^2 = 2\, m_e(E - E_0)/\, \hbar$$

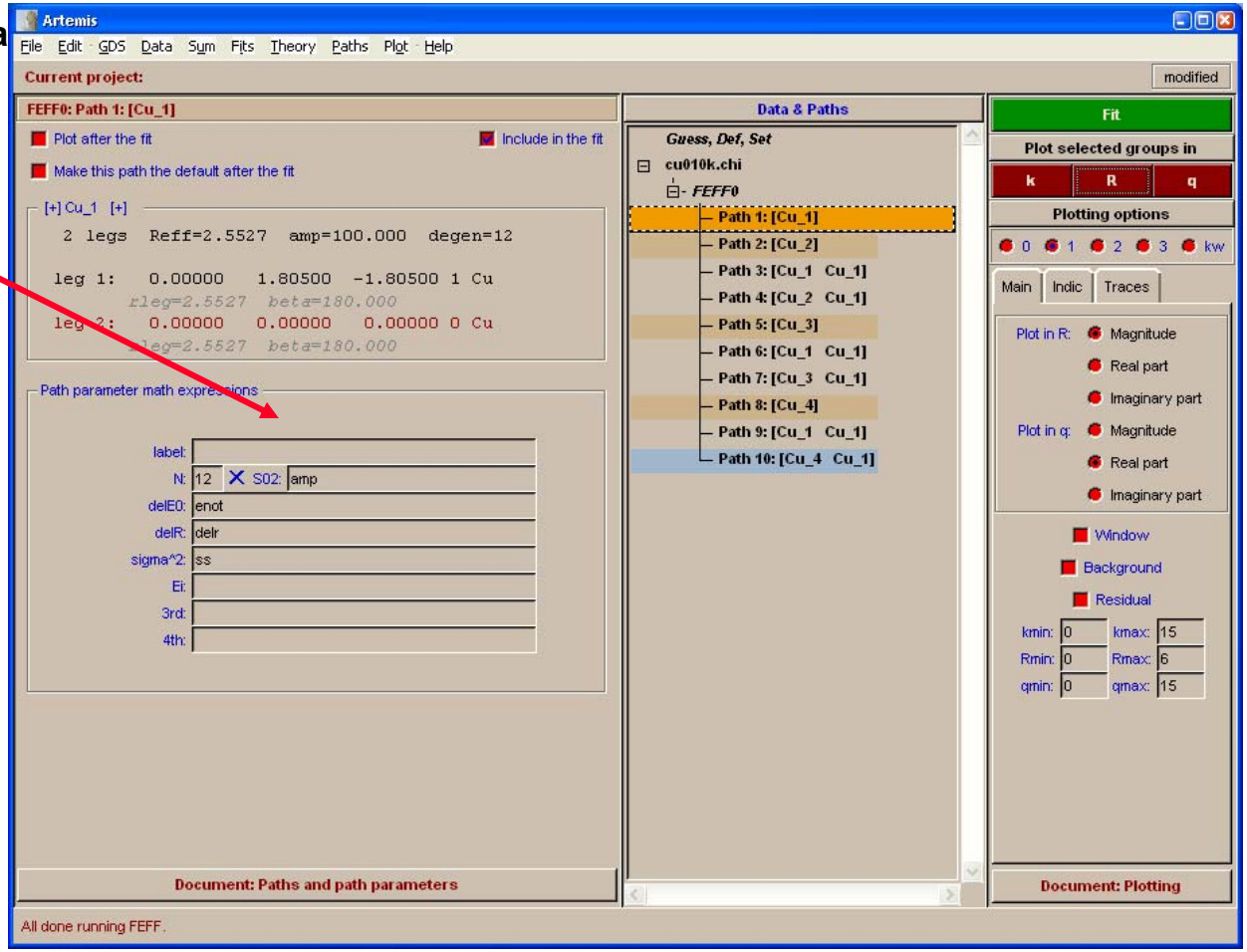
$F_i(k)$ effective scattering amplitude
$\varphi_i(k)$ effective scattering phase shift
$\lambda(k)$ mean free path
R_0 initial path length

N_i degeneracy of path
S_0^2 passive electron reduction factor
E_0 energy shift
ΔR change in half-path length
σ_i^2 mean squared displacement

Feff calculates the theoretical scattering amplitude F(k) effective, hence the name Feff.

Path Parameters

- **To produce a theoretical model each path must have a value for each of these parameters.**
- **Artemis makes a guess for these expressions.**
- **These values can originate from numbers or math expressions.**



S02 passive electron reduction factor

N degeneracy of path

E0 energy shift

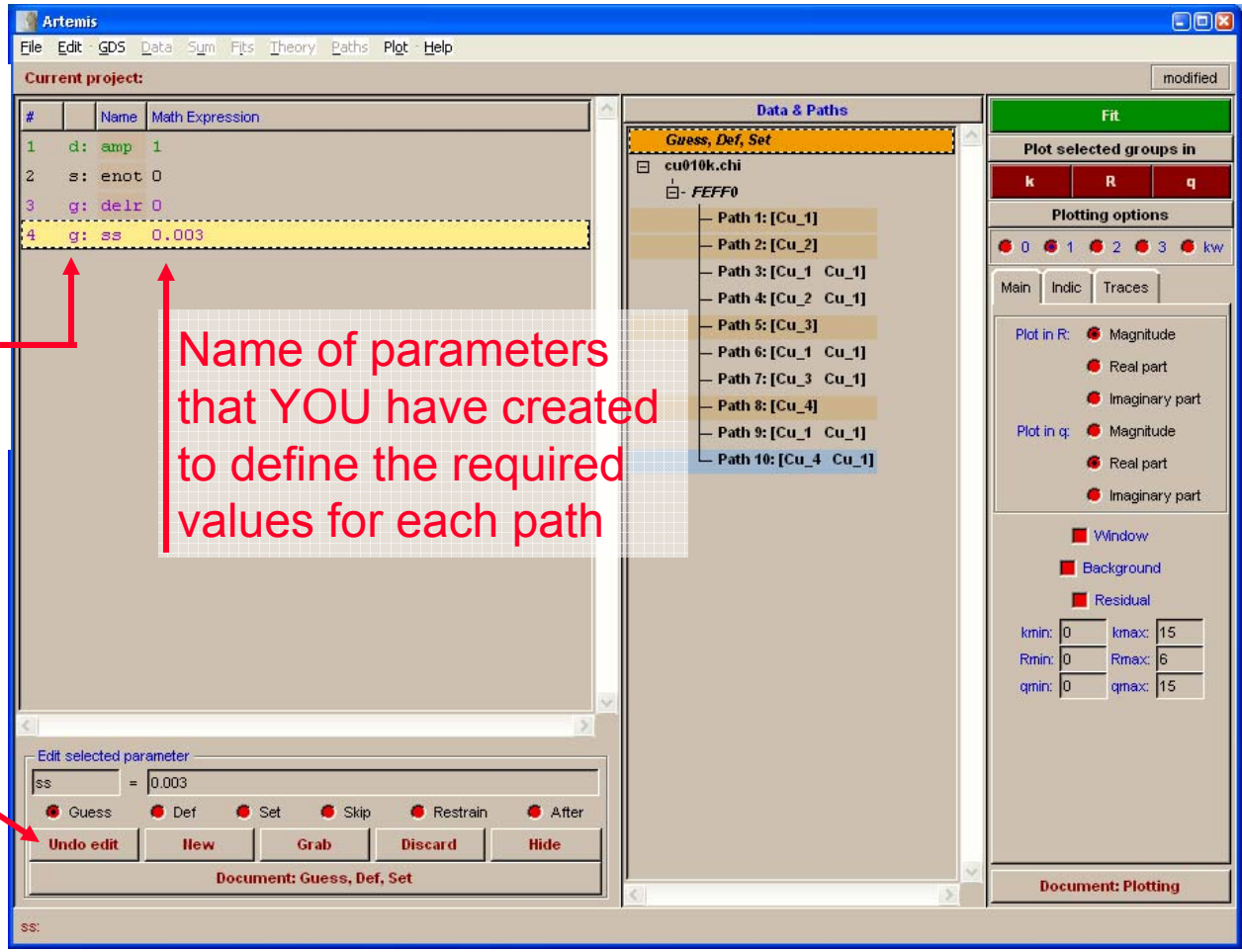
delR change in half-path length

sigma^2 mean squared displacement of the half path length

➢Click “Guess, Def, Set” in the Data & Paths list

EXAFS parameters

- **Guess:** Optimize parameter in fit.
- **Def:** Define a parameter to a given value but re-evaluate it during the fit
- **Set:** Set a value to a given value. Determine value once.
- **Skip:** Do not use this parameter.
- **Restrain:** penalty that can be added to the fit.



- **Define:** Define parameter to value given.
- **New:** Make a new parameter.
- **Grab:** Set the value to the best-fit value from the fit.
- **Discard:** remove parameter.
- **Hide:** Hide editing area

➢Click “Fit” to optimize these values

Fit Results

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Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit — Raw log file — Save — Dismiss

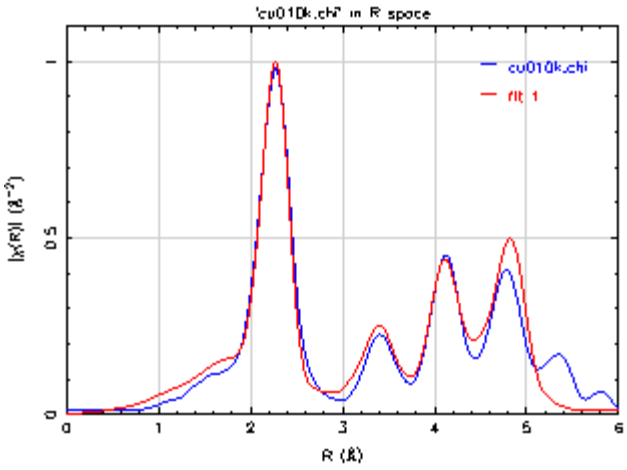
```
Project title    :  Fitting cu010k.chi
Comment          :  Fit #1
Prepared by      :
Contact          :
Started          :  12:38:31 on 26 June, 2009
This fit at      :  14:41:50 on 27 June, 2009
Environment      :  Artemis 0.8.013 using Windows XP, perl 5.008008, Tk 804.027, a
Data sets        :  "cu010k.chi"
Fit label        :  fit 1
Figure of merit  :  1

============================================================

Independent points           =      21.187500000
Number of variables          =       4.000000000
Chi-square                   =    1078.130989819
Reduced Chi-square           =      62.727621226
R-factor                     =       0.010391311
Measurement uncertainty (k)  =       0.000914479
Measurement uncertainty (R)  =       0.003911955
Number of data sets          =       1.000000000


Guess parameters +/- uncertainties  (initial guess):
  amp              =     0.8626890   +/-       0.0435050     (1.0000)
  enot             =     4.8357170   +/-       0.4306520     (0.0000)
  ss               =     0.0031660   +/-       0.0003500     (0.0030)
  alpha            =    -0.0030710   +/-       0.0010070     (0.0000)


Cor[illegible]bles:
                   -->  0.8711
                   -->  0.7818
All other correlations are below 0.25
```

Statistical quality of Fit

Initial guessed value

Your parameters

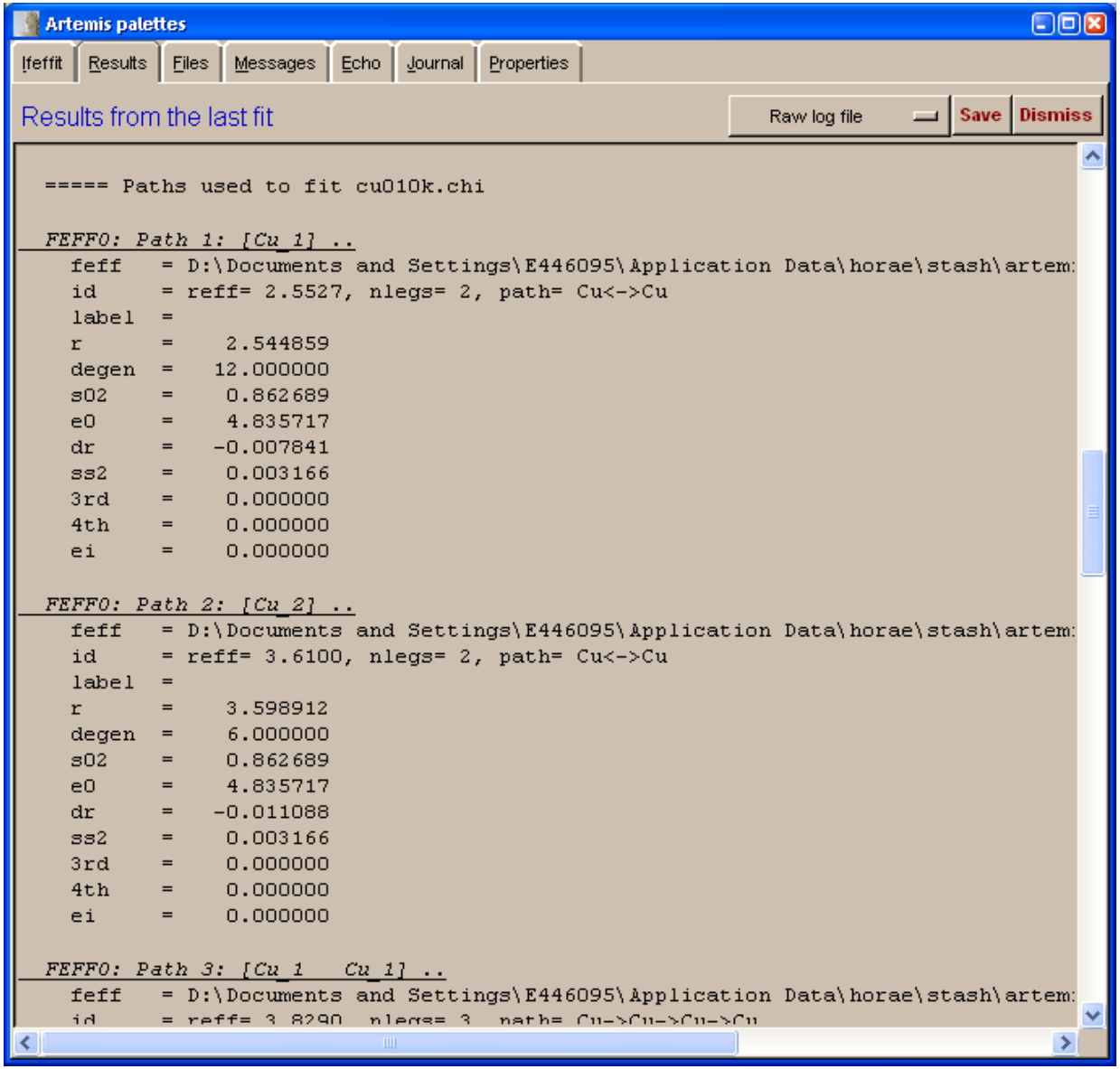
Best fit values with uncertainties



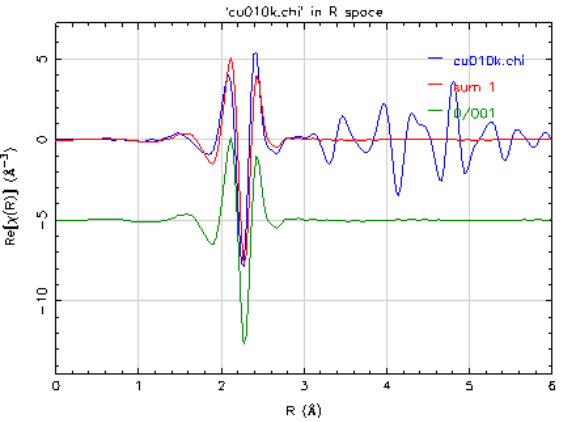
Fit Results

The value used for each EXAFS parameter for each path included in the fit.

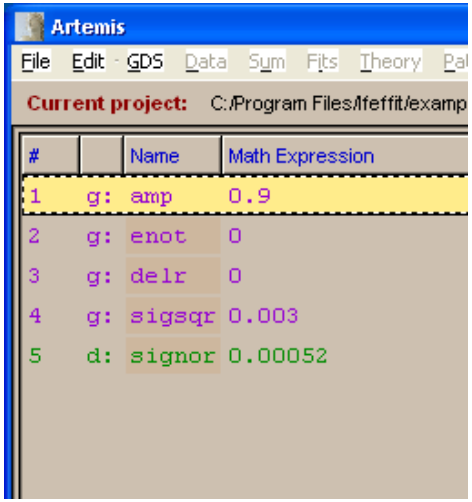
Used as a diagnostic tool when models do not work as expected



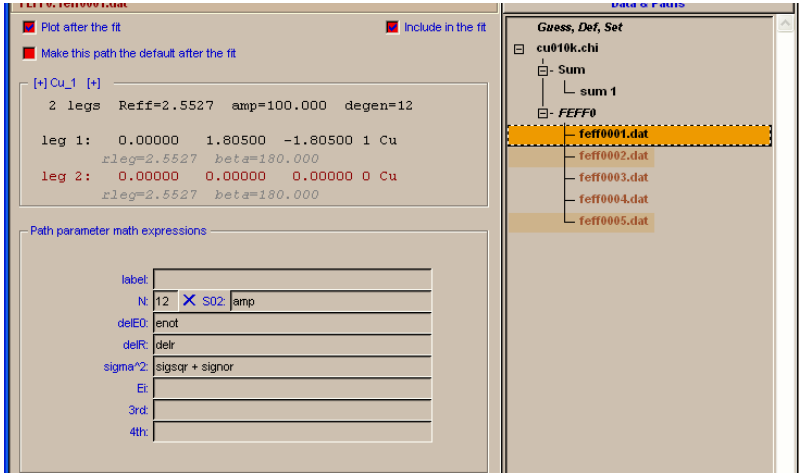
Example: Cu foil



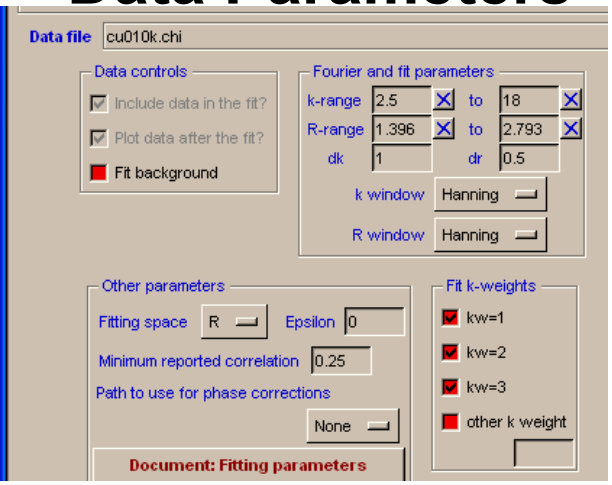
GSD Info



Path Info



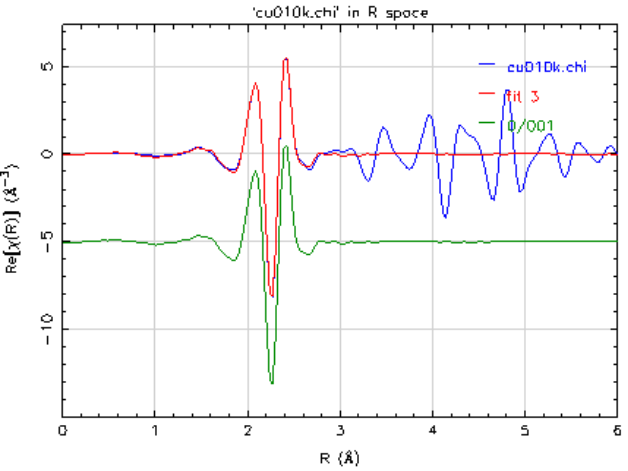
Data Parameters



- Example from c:\Program Files\Ifeffit\examples\Artemis
- Set up variables for first shell and give them reasonable values
- Sum paths to see how the theory corresponds to the data
- Notice strong signal due to 1st Cu neighbor is in about the right place for the data. Overall shape is similar
- Make sure that fit range is appropriate and choose kw for fit

File Number

Cu First Shell Model Results



Results Info

```
Independent points               =      13.579101562
Number of variables              =       4.000000000
Chi-square                       =     259.769677739
Reduced Chi-square               =      27.118375982
R-factor                         =       0.001326741
Measurement uncertainty (k)      =       0.001130698
Measurement uncertainty (R)      =       0.004441269
Number of data sets              =       1.000000000


Guess parameters +/- uncertainties  (initial guess):
  amp                =      0.9087470   +/-       0.0209050
  enot               =      5.4877270   +/-       0.2674280
  delr               =     -0.0045840   +/-       0.0012840
  sigsqr             =      0.0029450   +/-       0.0001320

Def parameters (using "FEFF0: feff0001.dat"):
  signor             =      0.0005200
```

GSD Info

Artemis

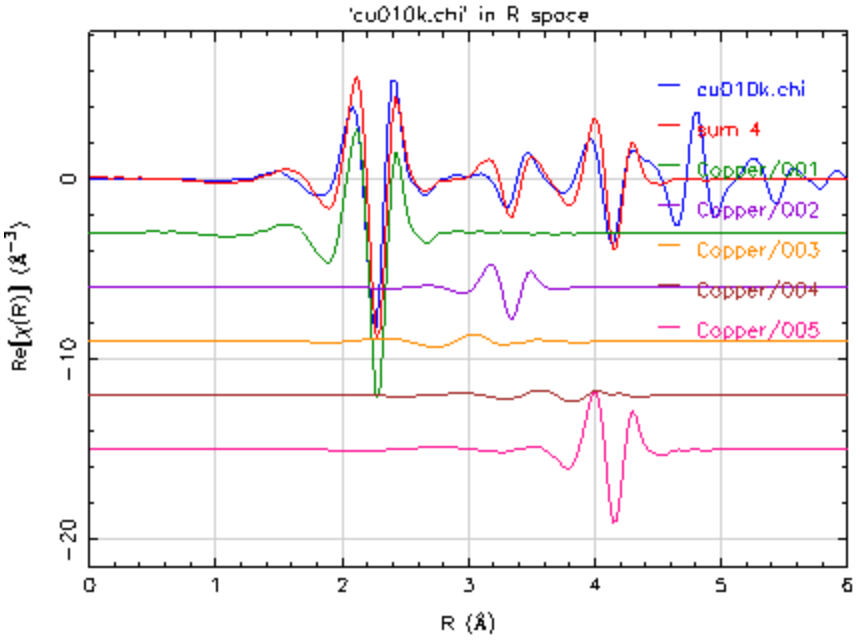
File Edit GDS Data Sum Fits Theory Paths Plot He

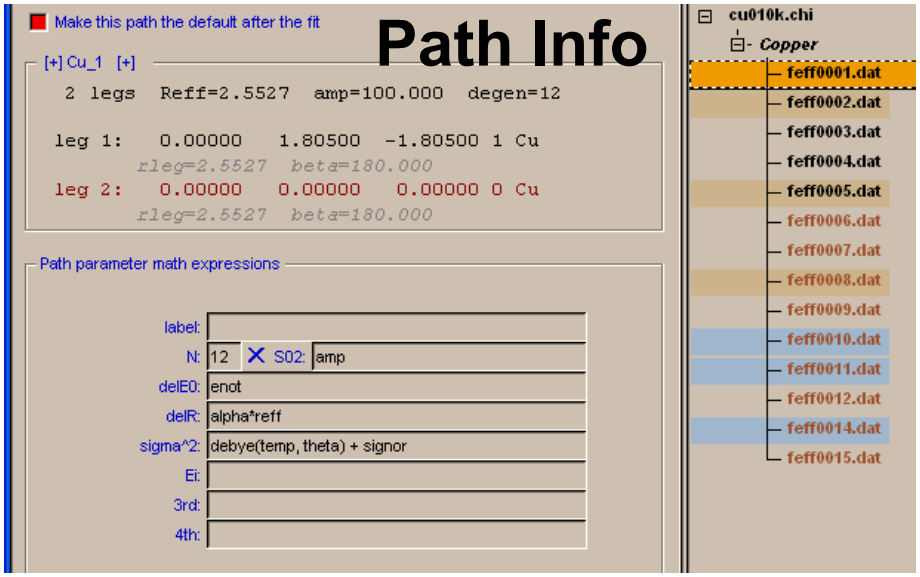
Current project: C:/Program Files/Ifeffit/examples/Artemis/Cu

#		Name	Math Expression
1	g:	amp	0.908747 (0.020905)
2	g:	enot	5.487727 (0.267428)
3	g:	delr	-0.004584 (0.001284)
4	g:	sigsqr	0.002945 (0.000132)
5	d:	signor	0.00052

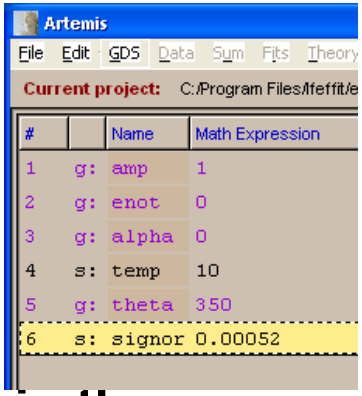
- Data and Model are in good agreement
- GSD values are reasonable

Cu: Three Shell Model



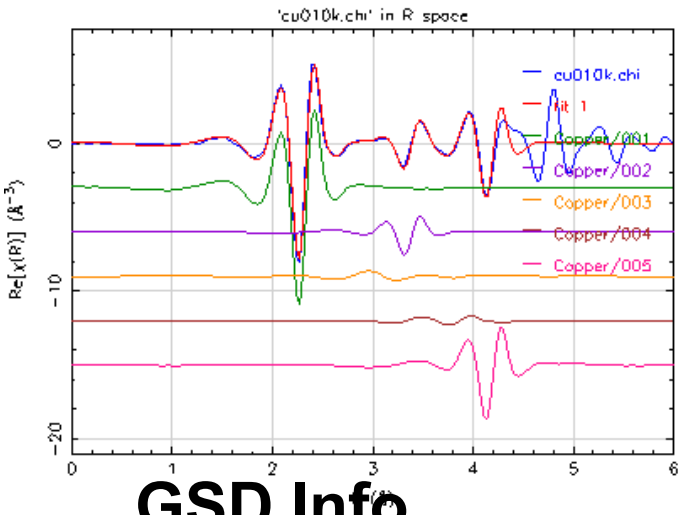


GSD Info

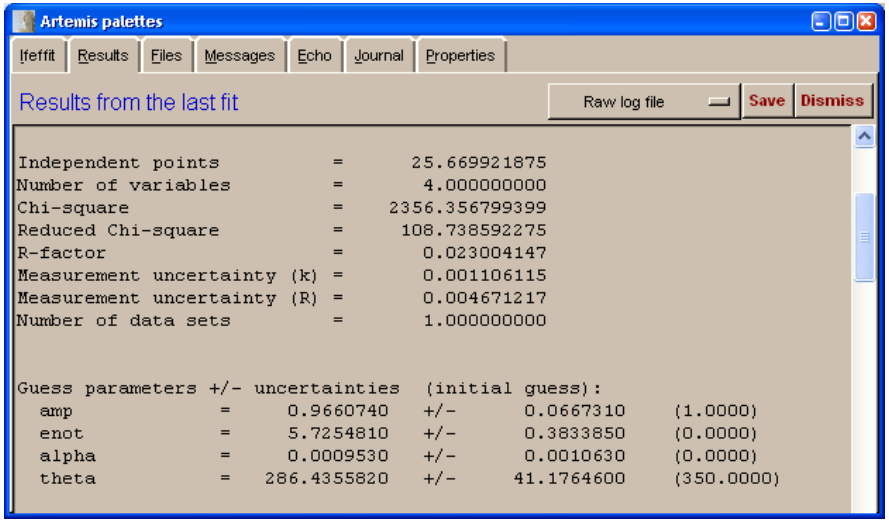


- Use alpha to define path lengths
- Use Debye temperature to define σ^2 values
- Each Single scattering path fits into a specific signal in the data

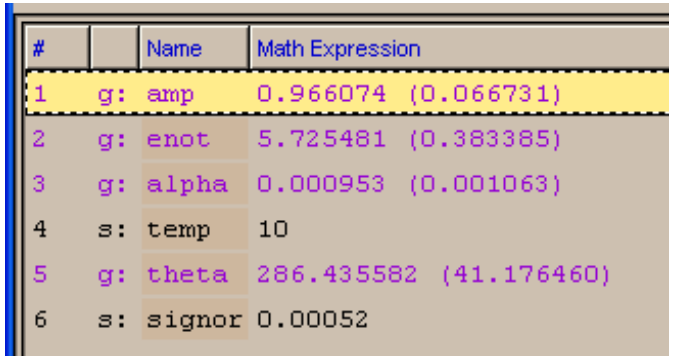
Fitting results for three shell fit to Cu



Results Info



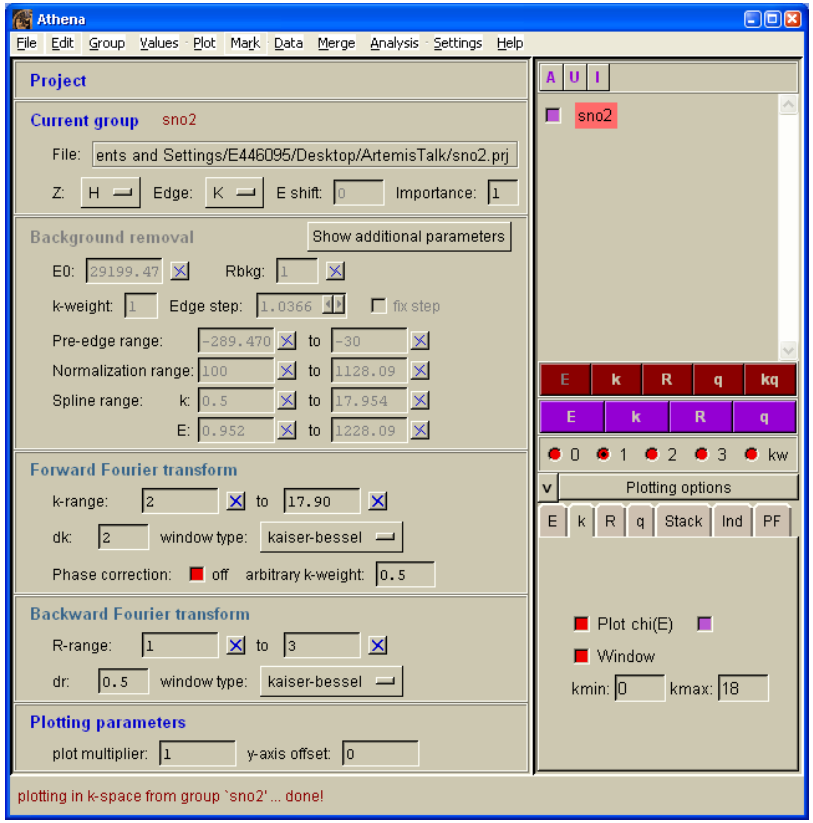
GSD Info

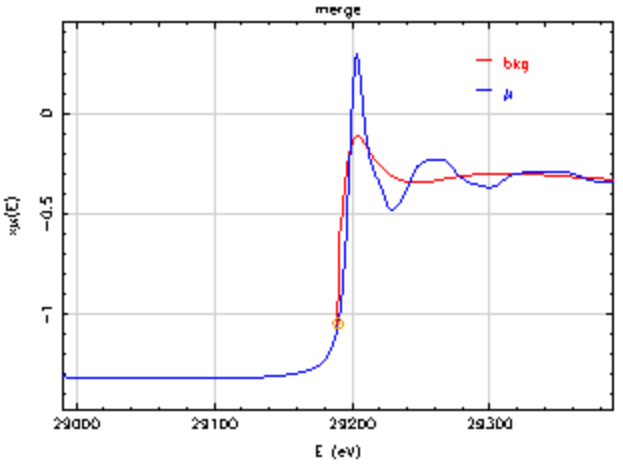


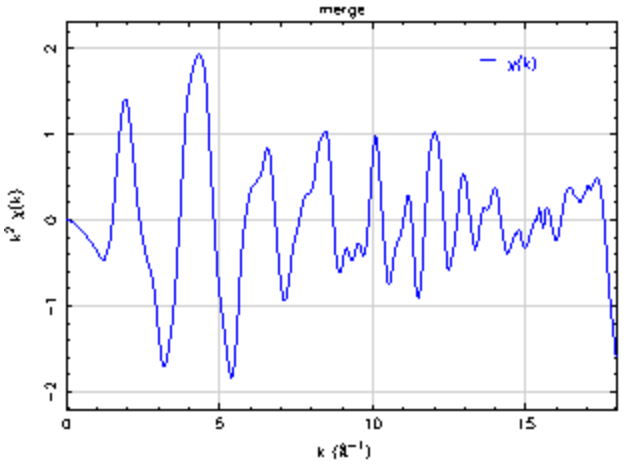
#		Name	Math Expression
1	g:	amp	0.966074 (0.066731)
2	g:	enot	5.725481 (0.383385)
3	g:	alpha	0.000953 (0.001063)
4	s:	temp	10
5	g:	theta	286.435582 (41.176460)
6	s:	signor	0.00052

- There is more to this example on your computer
- Modeling more shells and modeling temperature dependent spectra series

Example #2: Align SnO_2 EXAFS Spectra to Theory







- **E0 is somewhere on the edge**
- **Using default parameters**

M Newville, B Ravel, D Haskel and E A Stern. "Analysis of multiple scattering XAFS data using theoretical standards." Physica B 208 & 209: pp 154-156, 1995.

Fit of first Shell

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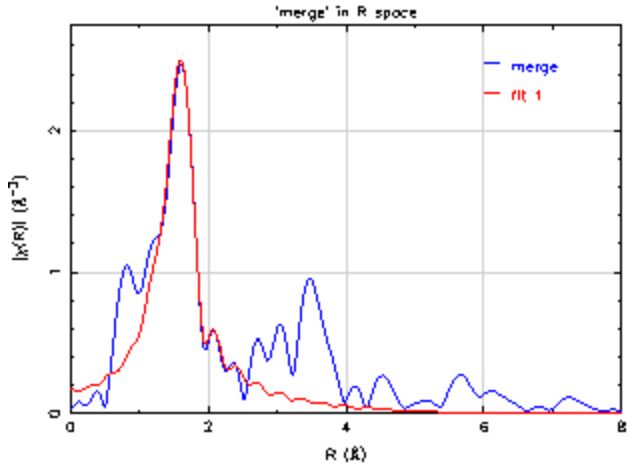
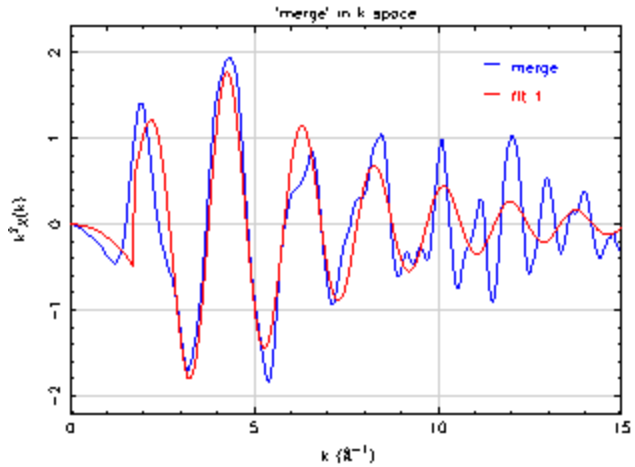
Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit — Raw log file — Save — Dismiss

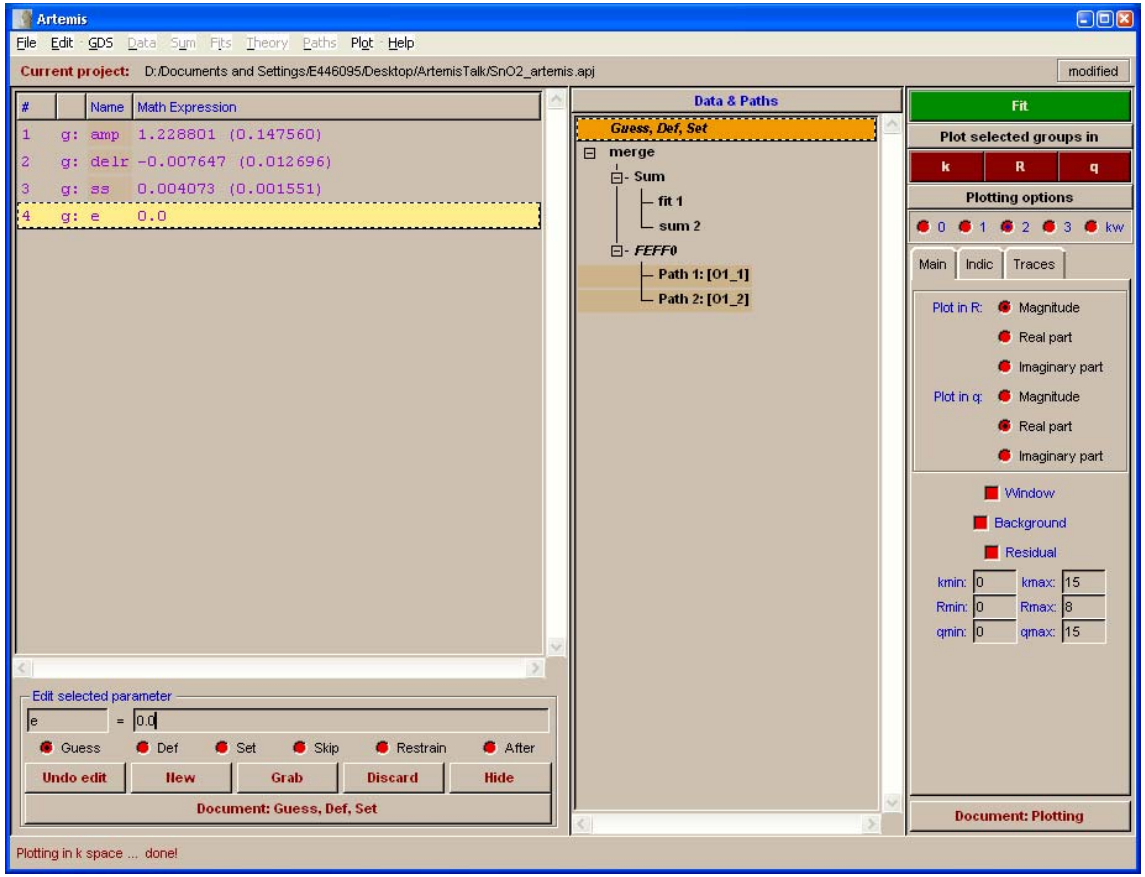
```
Project title    :  Fitting chi.chi
Comment          :  Fit #1
Prepared by      :  skelly@little.er.anl.gov
Contact          :
Started          :  09:28:09 on 11 May, 2004
This fit at      :  14:33:08 on 28 June, 2009
Environment      :  Artemis 0.8.013 using Windows XP, perl 5.008008, Tk 804.027, a
Data sets        :  "merge"
Fit label        :  fit 1
Figure of merit  :  1

============================================================

Independent points           =      12.437500000
Number of variables          =       4.000000000
Chi-square                   =  144652.749867735
Reduced Chi-square           =   17144.029613954
R-factor                     =       0.063204981
Measurement uncertainty (k)  =       0.000180334
Measurement uncertainty (R)  =       0.000432656
Number of data sets          =       1.000000000


Guess parameters +/- uncertainties  (initial guess):
  amp             =     1.2288010   +/-       0.1475600     (1.0000)
  delr            =    -0.0076470   +/-       0.0126960     (0.0000)
  ss              =     0.0040730   +/-       0.0015510     (0.0000)
  e               =    11.5113960   +/-       1.6084920     (0.0000)


Correlations between variables:
        delr and e           -->  0.8451
         amp and ss          -->  0.8057
All other correlations are below 0.25
```

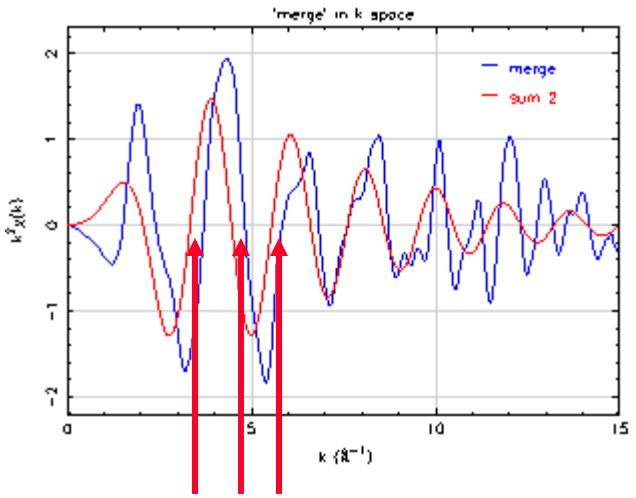




- **E0 could be smaller**
- **Background needs to be adjusted**
- **More information:**
- **M Newville, B Ravel, D Haskel and E A Stern. "Analysis of multiple scattering XAFS data using theoretical standards." Physica B 208 & 209: pp 154-156, 1995.**

Create first shell model with ΔE=0

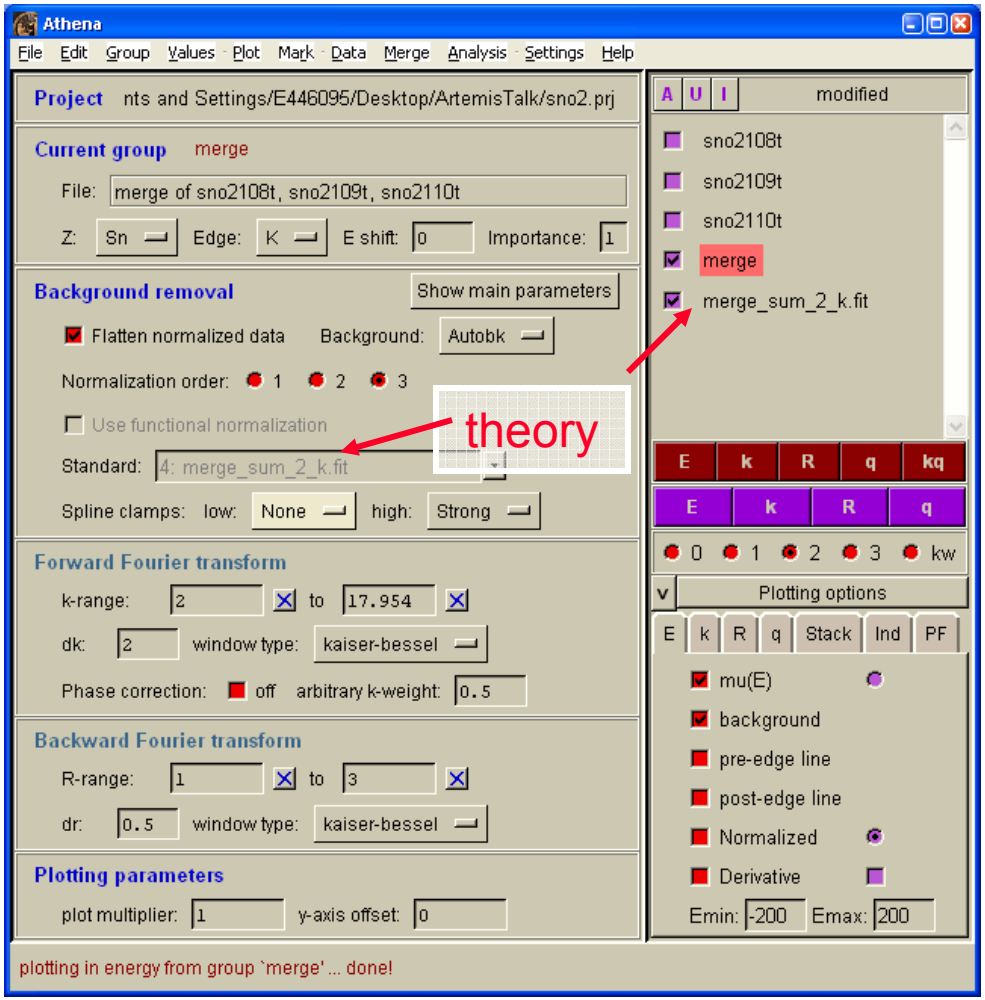


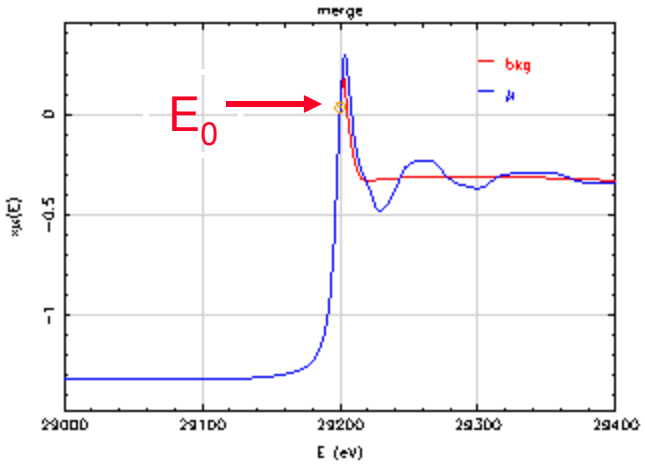


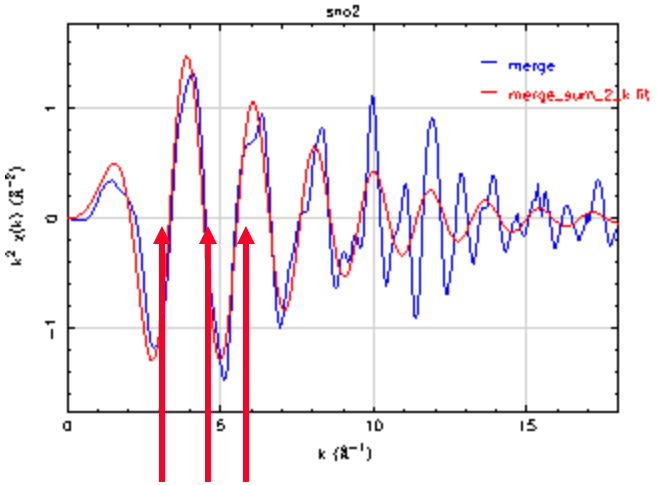
Data and theory are not aligned

- **Set parameters to their best-fit values by using the Grab button.**
- **Set e=0.**
- **Sum all paths to make model spectrum with E=0.**

Background corrected and energy aligned by using theory



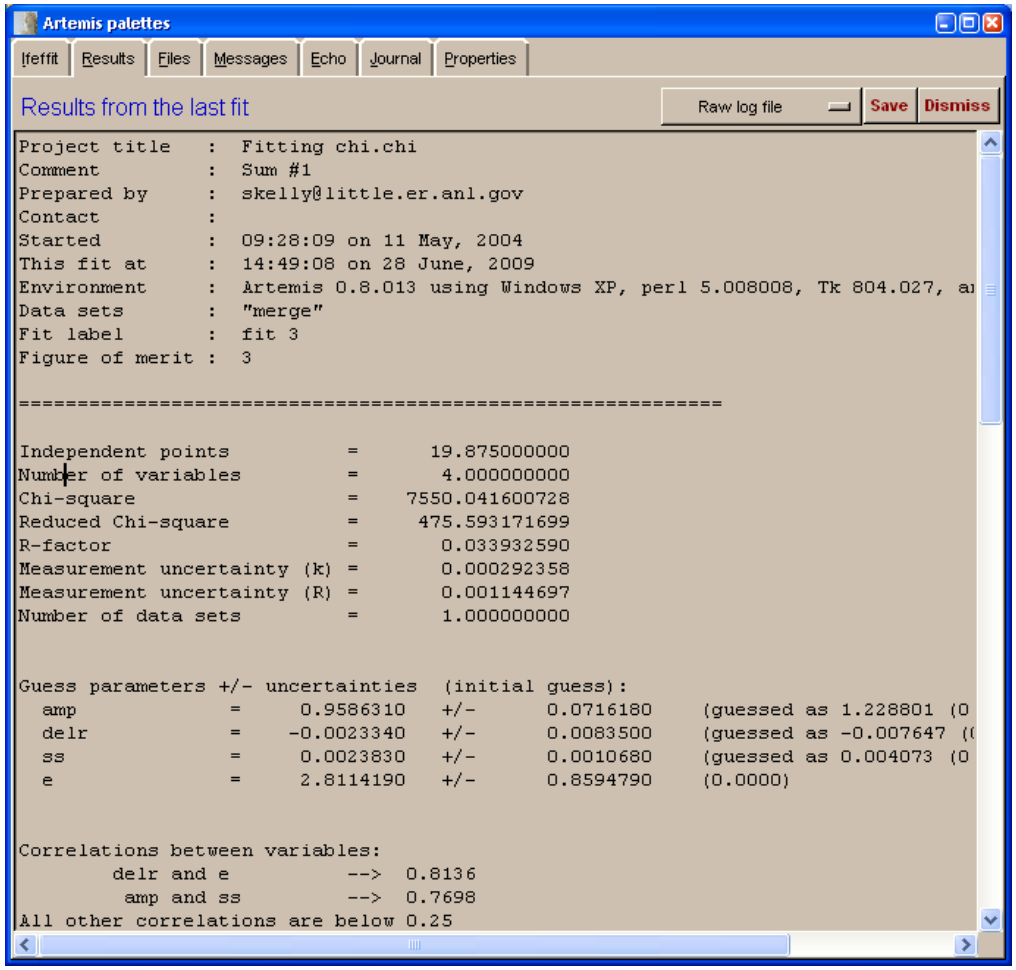


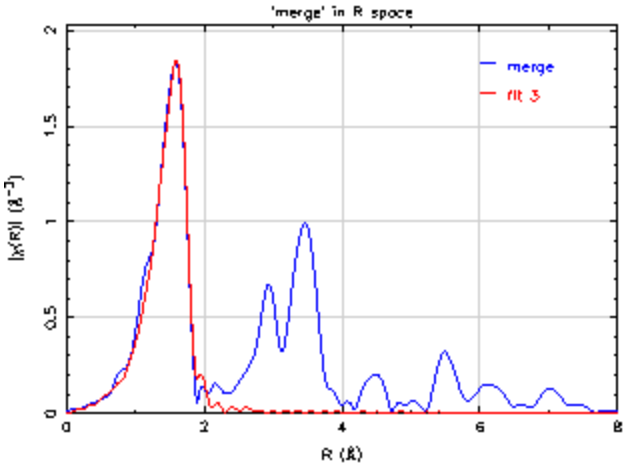


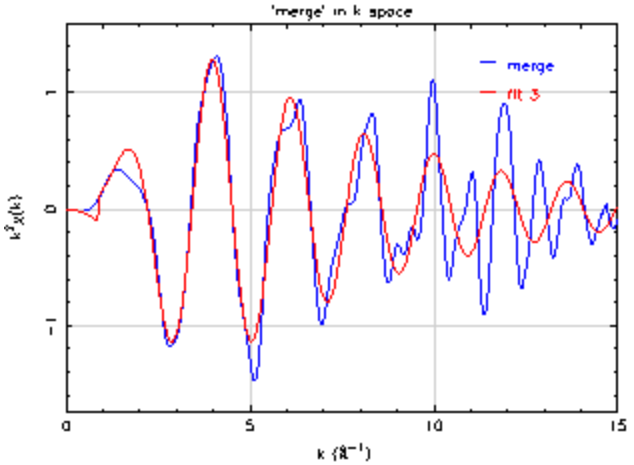
- **Read the theory into Athena**
- **Select your data and use the theory as a standard**
- **Adjust E0 so that the data and theory are aligned at low k values**

Data and theory are aligned

Fit to first shell with background adjusted



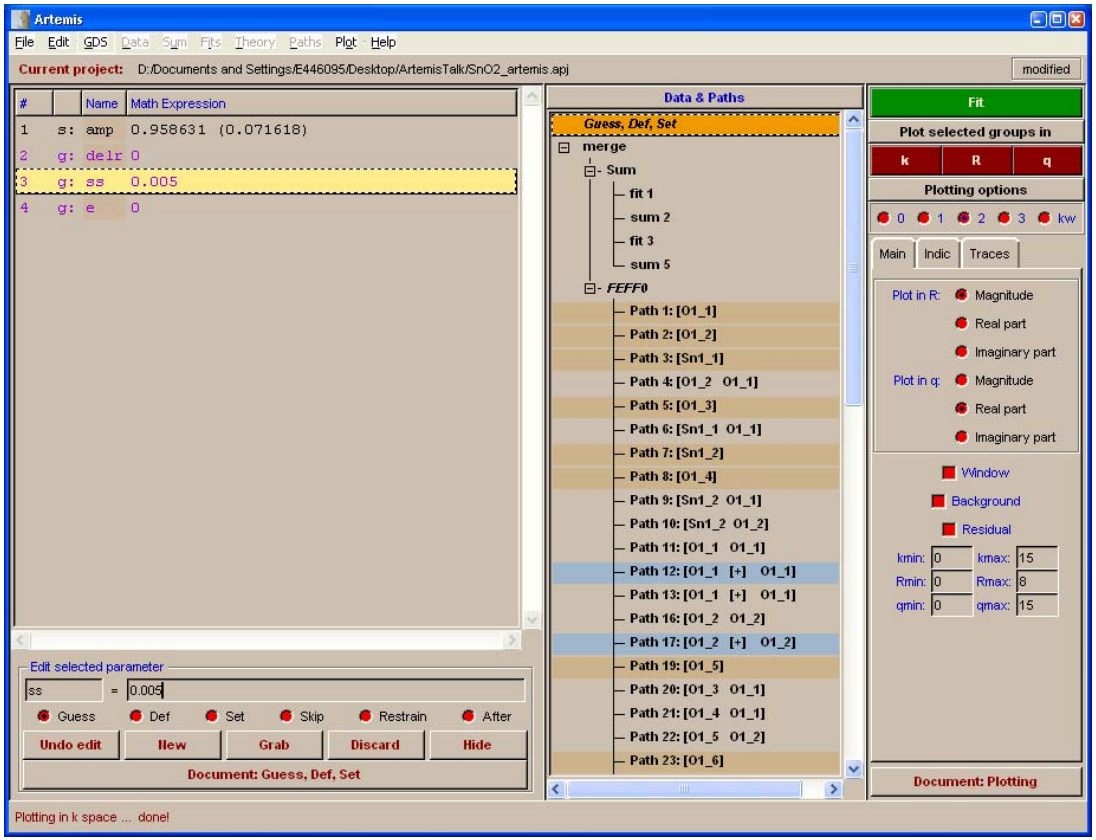


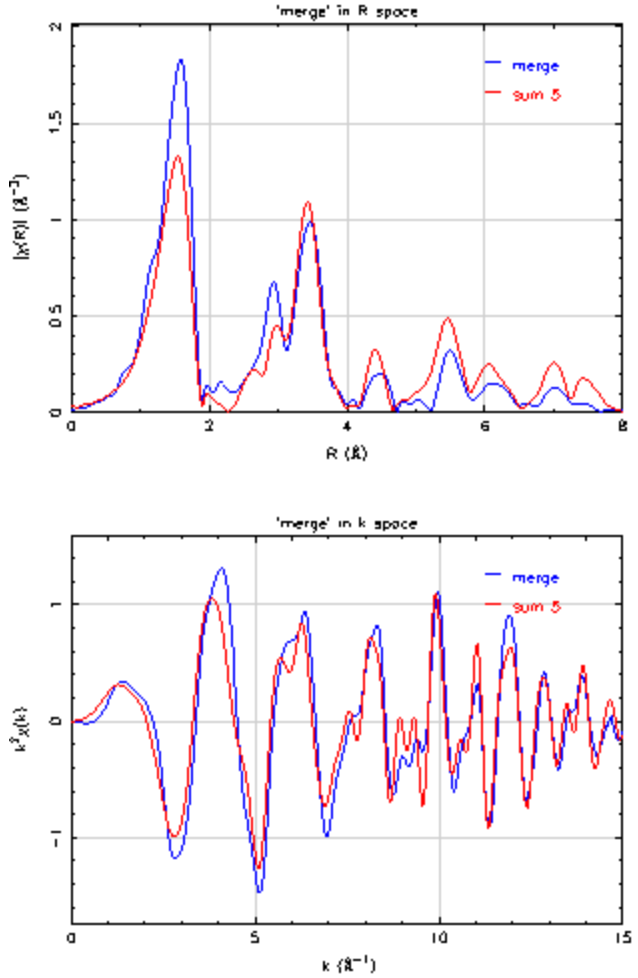


- **Read the chi(k) data back into Artemis and fit the first shell**

Example 3: Modeling a metal-oxide (SnO_2)

- Compare the sum of all 69 paths to the data

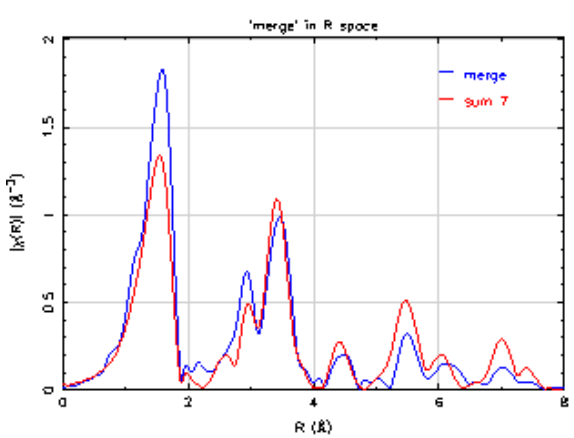


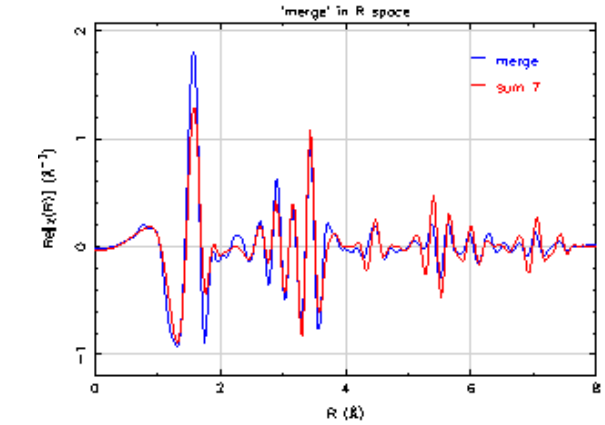


File Number

How many paths are required?

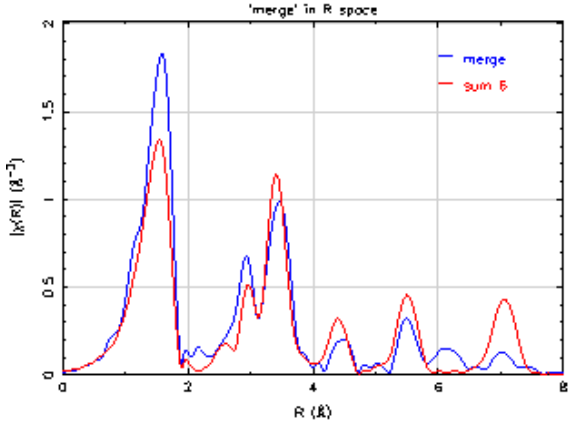
Minimum amplitude 5%
30 of 69 paths

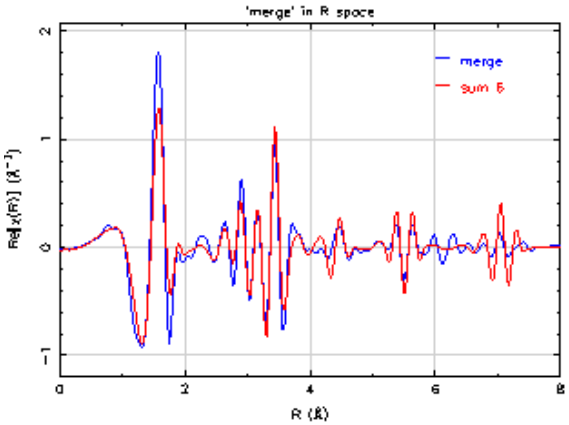




All data is present in model

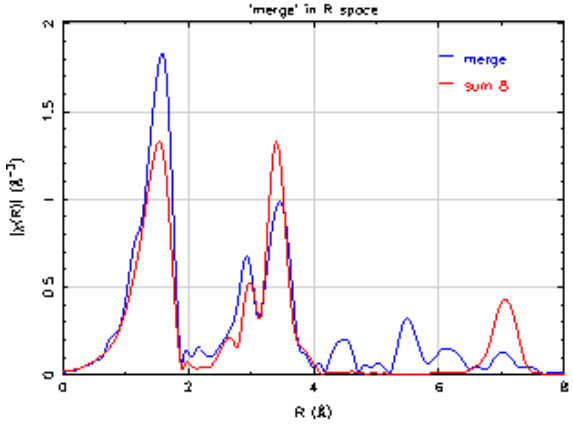
Minimum amplitude 10%
18 of 69 paths

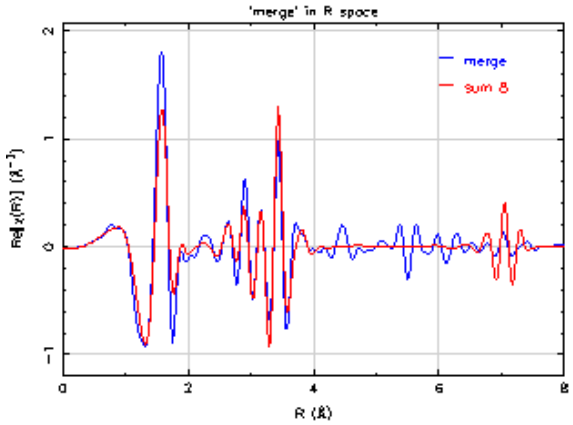




missing a little of the data

Minimum amplitude 15%
8 of 69 paths





missing a lot of the data

Models to consider:

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- Δr:
  - Symmetric expansion term: Alpha * reff.
  - Grouped depending on distance and atom types
  - Related to unit cell dimensions
- ΔE:
  - Energy shifts that depend on atom type
  - One energy shift for all paths
  - Two energy shifts, one for first shell and another for all other shells
- σ^2:
  - Grouped depending on distance and atom types.
  - Use a Debye or Einstien model, with one or more characteristic temperatures.
  - Each shell with independent value.
  - Separate structural disorder from thermal disorder components.
- S02:
  - One S02 for all paths.
  - Approximate S02 from standards.
- N:
  - Determined by the crystal structure.
  - Fit a data series were N is expected to change.

Some EXAFS references

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- Multiple edges, structural information: **B Ravel, E. Cockayne, M. Newville and K. M. Rabe. "Combined EXAFS and first-principles theory study of Pb1-xGexTe." Phys. Rev. B** 60**(21): pp 14632–14642, Dec 1999**
- Structural information, bond angles: **A. I. Frenkel, E A Stern, A. Voronel, M. Qian and M Newville. "Solving the structure of disordered mixed salts." Phys. Rev. B** 49**(17 – 1): pp 11662–11674, May 1994.**
- Model two phases, then combined to model a mixture: **S. Kelly, R. Ingalls, F. Wang, B. Ravel and D. Haskel. "X-ray-absorption fine-structure study of the B1-to-B2 phase transition in RbCl." Phys. Rev. B** 57**(13): pp 7543–7550, April 1998**
- Determine neighbor atom types and number using standards: **S. D. Kelly, K. M. Kemner, J. B. Fein, D. A. Fowle, M. I. Boyanov, B. A. Bunker and N. Yee. "X-ray absorption fine-structure determination of pH dependent U-bacterial cell wall interactions." Geochem. Cosmo. acta.** 66**(22): pp 3855-3871, Nov 2002.**
- Multiple techniques: **P G Allen, J J Bucher, D L Clark, N M Edelstein, S A Ekberg, J W Gohdes, E A Hudson, N Kaltsoyannis, W W Lukens, M P Neu, P D Palmer, T Reich, D K Shuh, C D Tait and B D Zwick. "Multinuclear NMR, Raman, EXAFS, and X-ray diffraction studies of uranyl carbonate complexes in near-neutral aqueous solution. X-ray structure of [C(NH2)3]6[(UO2)3(CO3)6] 6.5H20." Inorg. Chem.** 34**: pp 4797-4807, 1995.**
- Pressure dependent data: **A. I. Frenkel, F. M. Wang, S. Kelly, R. Ingalls, D. Haskel, E. A. Stern and Y. Yacoby, "Local structural changes in KNbO3 under high pressure", *Physical Review B*** 56**, 10869, 1997.**
- Temperature dependent data: **D. Haskel, E.A. Stern, D.G. Hinks, A.W. Mitchell, J.D. Jorgensen, J.I. Budnick, "Dopant and Temperature Induced Structural Phase Transitions in $La_{2-x}Sr_xCuO_4$" Physical Review Letters,** 76 **(3) pg 439**
- Multiple edges, structural disorder: **S. Calvin, E. E. Carpenter, B. Ravel, V. G. Harris and S. A. Morrison. "Multiedge refinement of extended x-ray-absorption fine structure of manganese zinc ferrite nanoparticles." Phys. Rev. B** 66**: pp 224405, 2002.**
- Structural information from XANES and EXAFS: **B. Ravel, E. A. Stern, R. I. Vedrinskii and V. Kraizman. "Local structure and the phase transitions of BaTiO3." FERROELECTRICS** 206**(1-4): pp 407-430, 1998.**
- Temperature dependence and nanoparticles: **A. I. Frenkel, C. W. Hills and R. G. Nuzzo. "A view from the inside: Complexity in the atomic scale ordering of supported metal nanoparticles." JOURNAL OF PHYSICAL CHEMISTRY B** 105**(51): pp 12689-12703, 2001.**
- **Kelly, S. D.; Hesterberg, D.; Ravel, B. Analysis of soils and minerals using X-ray absorption spectroscopy. In Methods of soil analysis, Part 5 -Mineralogical methods; Ulery, A. L., Drees, L. R., Eds.; Soil Science Society of America: Madison, WI, USA, 2008; pp 367-463.**

The Model for SnO_2 data

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- Δr: symmetric expansion term: alpha * reff.
- ΔE: Energy shifts that depend on atom type:
  - The first shell; Eo1,
  - All other oxygen scattering events: Eo2
  - All tin scattering events: Esn
- σ^2: Grouped depending on distance and atom types.
- S_0^2: one for all paths.
- N: determined from the crystal structure.

Final Model for SnO_2

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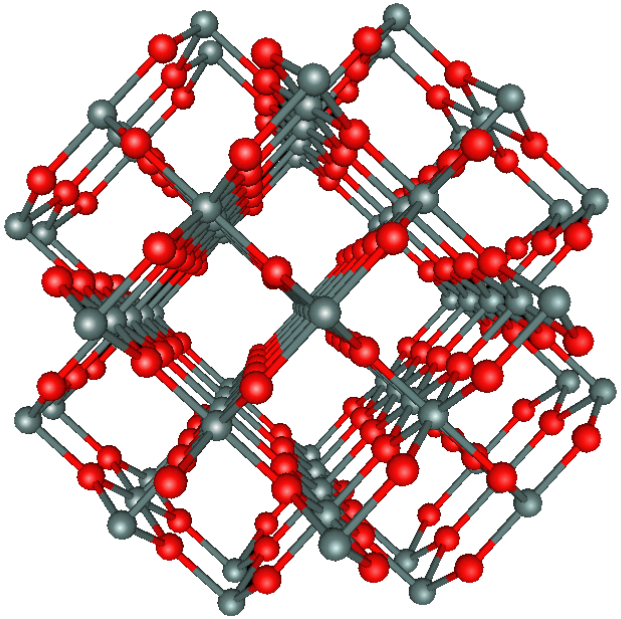
Includes all the atoms shown here



Path	*N*	*reff*	*ΔR*	σ^2	*ΔE*
Sn-O1	4	2.0519	**Alpha**·reff	**σ^2o1**	**ΔEo1**
Sn-O2	2	2.0567	Alpha·reff	σ^2o1	ΔEo1
Sn-Sn1	2	3.1864	Alpha·reff	**σ^2sn1**	**ΔEsn**
Sn-O3	4	3.5906	Alpha·reff	**σ^2o3**	**ΔEo2**
Sn-Sn2	8	3.7093	Alpha·reff	**σ^2sn2**	ΔEsn
Sn-Sn2-01	8	3.9090	Alpha·reff	**σ^2sn2o1**	0.5·ΔEsn+0.5·ΔEo1
Sn-Sn2-02	8	3.9090	Alpha·reff	σ^2sn2o1	0.5·ΔEsn+0.5·ΔEo1
Sn-O5	8	4.2414	Alpha·reff	**σ^2o5**	ΔEo2
Sn-Sn3	4	4.7373	Alpha·reff	**σ^2sn3**	ΔEsn
Sn-O7	8	4.8006	Alpha·reff	**σ^2o7**	ΔEo2
Sn-Sn4	8	5.7092	Alpha·reff	**σ^2sn4**	ΔEsn
Sn-Sn5	8	5.8365	Alpha·reff	σ^2sn4	ΔEsn
Sn-Sn5-01	8	5.8405	Alpha·reff	σ^2sn4	0.5·ΔEsn+0.5·ΔEo1
Sn-Sn5-04	8	5.8405	Alpha·reff	σ^2sn4	0.5·ΔEsn+0.5·ΔEo2
Sn-O1-Sn5-O1	4	5.8444	Alpha·reff	σ^2sn4	0.33·ΔEsn+0.66·ΔEo1
Sn-O4-Sn5-O4	4	5.8444	Alpha·reff	σ^2sn4	0.33·ΔEsn+0.66·ΔEo2
Sn-Sn6-Sn1	4	6.3728	Alpha·reff	**σ^2sn6**	ΔEsn
Sn-Sn1-Sn6-Sn1	2	6.3728	Alpha·reff	σ^2sn6	ΔEsn
Sn-Sn7	4	6.6995	Alpha·reff	**σ^2sn7**	ΔEsn
Sn-Sn7-O2	4	6.6995	Alpha·reff	σ^2sn7	0.5·ΔEsn+0.5·ΔEo1
Sn-Sn7-O6	4	6.6995	Alpha·reff	σ^2sn7	0.5·ΔEsn+0.5·ΔEo2
Sn-Sn7-O6-O2	4	6.6995	Alpha·reff	σ^2sn7	0.33·ΔEsn+0.33·ΔEo1+0.33·ΔEo2
Sn-O6-Sn7-O2	4	6.6995	Alpha·reff	σ^2sn7	0.33·ΔEsn+0.33·ΔEo1+0.33·ΔEo2
Sn-O2-Sn7-O6-O2	4	6.6995	Alpha·reff	σ^2sn7	0.25·ΔEsn+0.50·ΔEo1+0.25·ΔEo2
Sn-O6-Sn7-O6-O2	4	6.6995	Alpha·reff	σ^2sn7	0.25·ΔEsn+0.25·ΔEo1+0.50·ΔEo2
Sn-Sn8	8	7.4187	Alpha·reff	**σ^2sn8**	ΔEsn
Sn-Sn8-Sn2	16	7.4187	Alpha·reff	σ^2sn8	ΔEsn
Sn-Sn2-Sn-Sn2	8	7.4187	Alpha·reff	4·σ^2sn2	ΔEsn
Sn-Sn2-Sn8-Sn2	8	7.4187	Alpha·reff	σ^2sn8	ΔEsn
Sn-Sn9	16	7.6578	Alpha·reff	**σ^2sn9**	ΔEsn

- **One S02-value was also determined in the fit.**
- **30 paths used in final model.**
- **There are a total of 18 parameters in this model and 56 independent points in the data.**

Final Model and Fit to SnO_2

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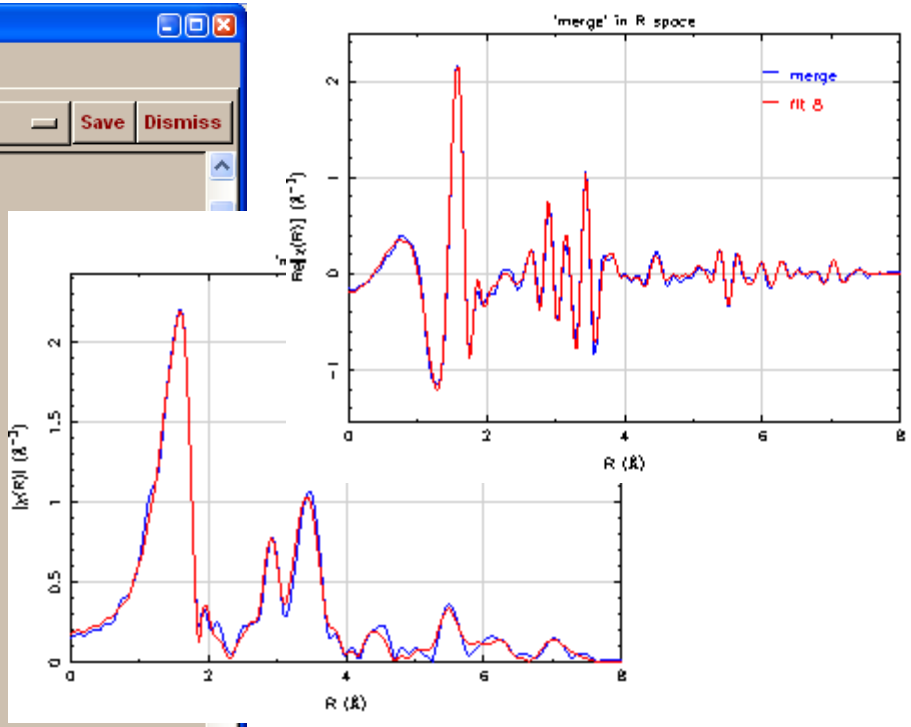
Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit — Raw log file — Save — Dismiss

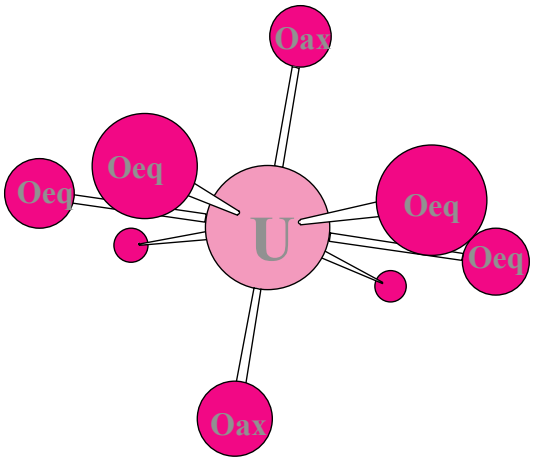
```
============================================================

Independent points               =      70.494140625
Number of variables              =      18.000000000
Chi-square                       =   19369.502641930
Reduced Chi-square               =     368.984088725
R-factor                         =       0.016091320
Measurement uncertainty (k)      =       0.000149981
Measurement uncertainty (R)      =       0.000587235
Number of data sets              =       1.000000000


Guess parameters +/- uncertainties  (initial guess):
  amp                =     1.0512750   +/-       0.0230720     (1.0731)
  eo1                =     2.7995790   +/-       0.2075060     (3.4017)
  esn                =     1.3666090   +/-       0.4750070     (2.6664)
  eo2                =     3.5177280   +/-       0.8663380     (5.0268)
  alpha              =    -0.0009020   +/-       0.0007570     (-0.0001)
  sso1               =     0.0031980   +/-       0.0002910     (0.0034)
  sso2               =     0.0184130   +/-       0.0070080     (0.0203)
  sso4               =     0.0055760   +/-       0.0020580     (0.0066)
  sso6               =     0.0059160   +/-       0.0036660     (0.0066)
  sssn2o1            =     0.0035050   +/-       0.0010600     (0.0040)
  sssn1              =     0.0035740   +/-       0.0002840     (0.0038)
  sssn2              =     0.0053430   +/-       0.0002450     (0.0054)
  sssn3              =     0.0070690   +/-       0.0014830     (0.0071)
  sssn4              =     0.0073380   +/-       0.0006350     (0.0077)
  sssn5              =     0.0084460   +/-       0.0026580     (0.0090)
  sssn6              =     0.0069210   +/-       0.0024670     (0.0066)
  sssn7              =     0.0087330   +/-       0.0016900     (0.0086)
  sssn8              =     0.0106090   +/-       0.0044800     (0.0096)


Correlations between variables:
```

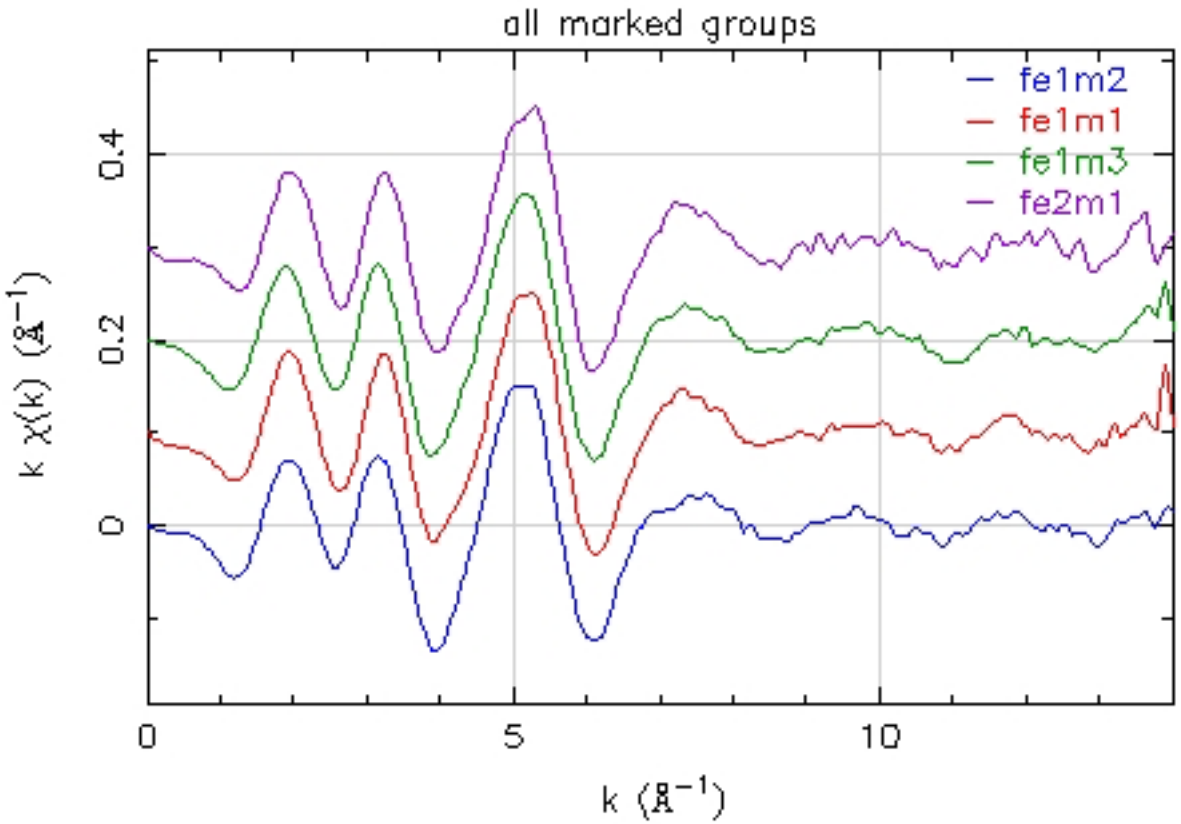


- **Typical values for EXAFS parameters**
- **Δr: less than 0.5 Å**
- **ΔE: less than 10 eV**
- **σ^2: 0.003 to 0.020 Å^2**
- **S02: 0.70 to 1.10**

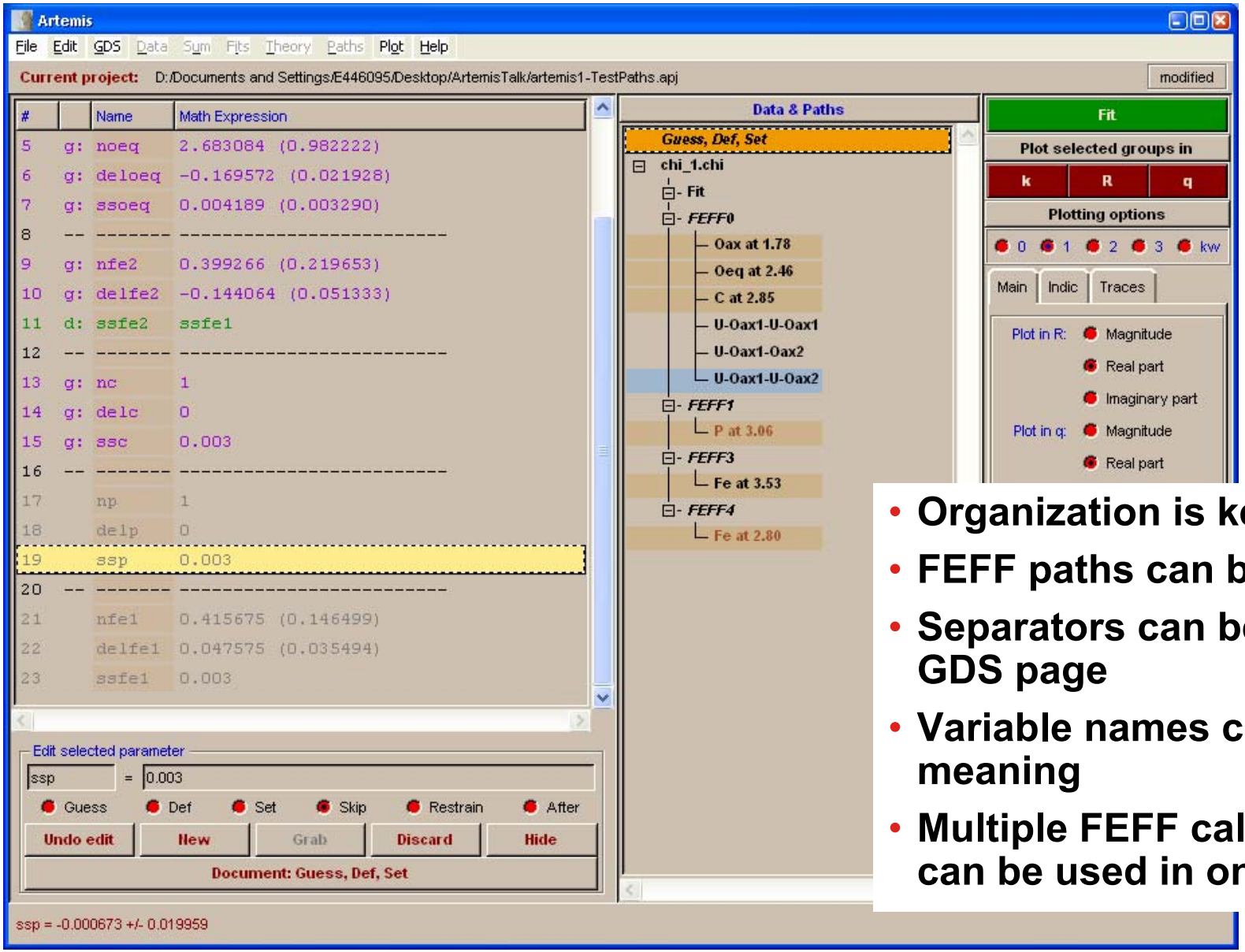
Example 4: Determining 2nd shell atom type and number

- Uranyl in equilibrium with a mixture of Fe-oxides and different microbial components.
- Possible second shell atoms, O, C, P, Fe, U.
- 30-50 combinations of these atoms were tested.
- Three of these tests are shown here:
  - C and Fe,
  - P and Fe,
  - Fe and Fe



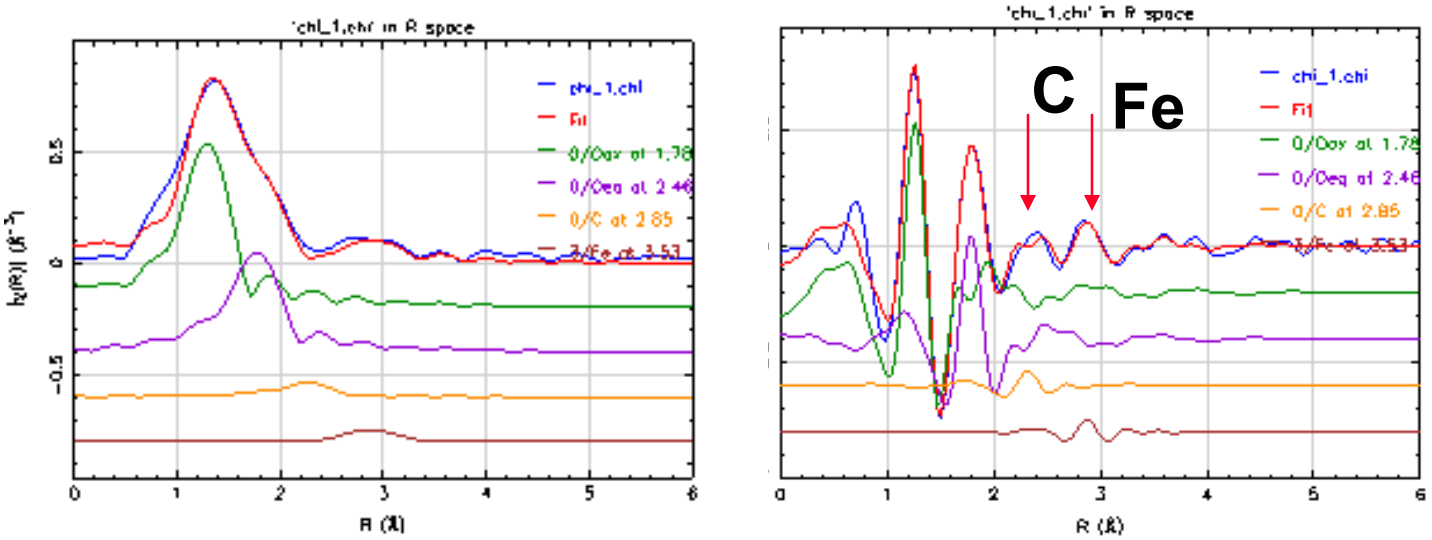


Setting up a Uranyl Model



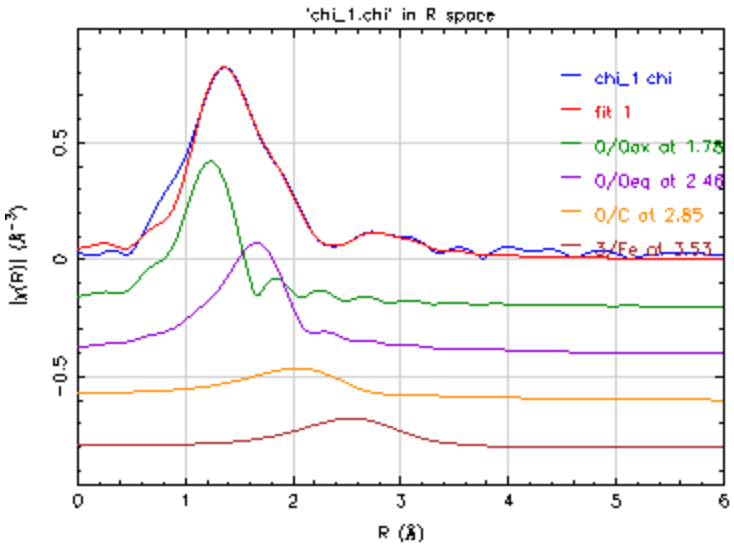
- Organization is key
- FEFF paths can be renamed
- Separators can be added to GDS page
- Variable names can have meaning
- Multiple FEFF calculations can be used in one model

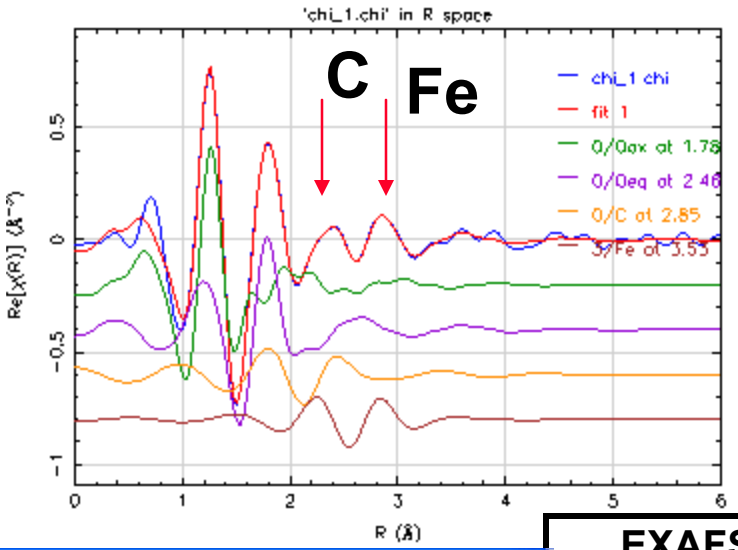
Test data for C and Fe shells



- **Place C and Fe shells in a "good" spot**
  - **set parameters – no fitting**
  - **monitor results and fit spectra**
  - **Needs to be done using real or imaginary part of FT, can not always be done using magnitude of FT.**

Fit Results using C and Fe shells





Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit — Column view — Save — Dismiss

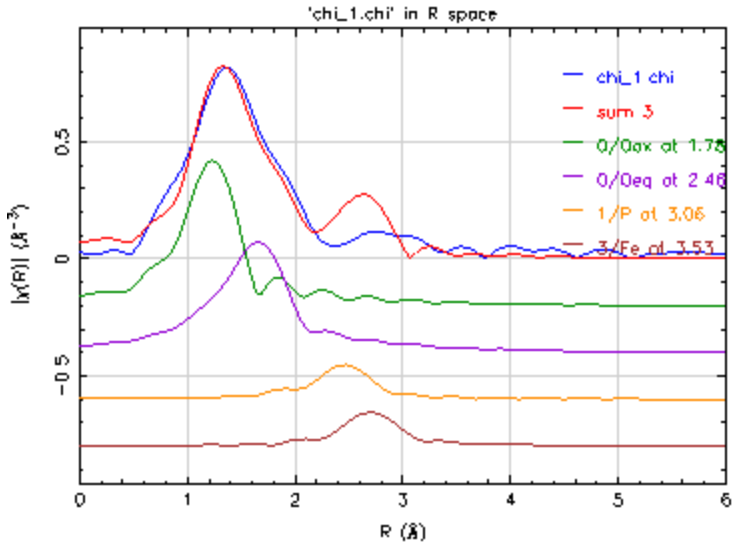
R-factor for this data set = 0.00203

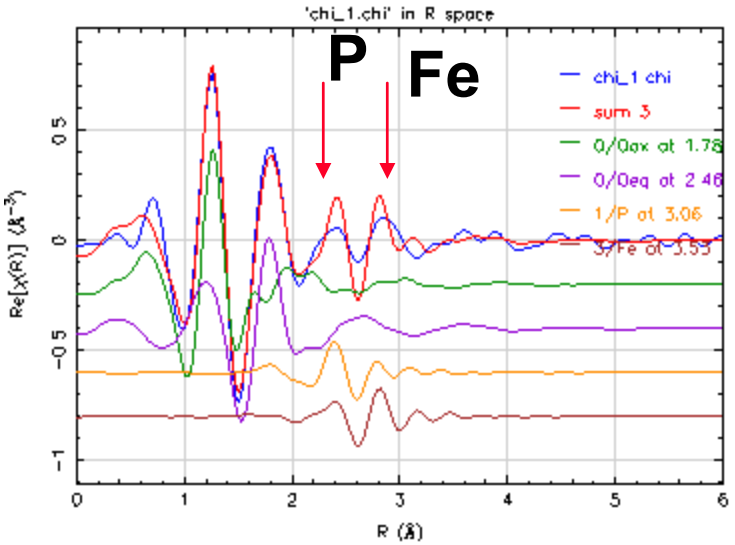
path	degen	amp	sigma^2	e0	reff	delta_R	R
"FEFF0: Oax at 1.78"	1.00000	2.000	0.00386	-8.79362	1.75510	-0.00517	1.74993
"FEFF0: Oeq at 2.46"	1.00000	5.719	0.01094	-8.79362	2.46160	-0.21986	2.24174
"FEFF0: C at 2.85"	1.00000	21.896	0.03066	-8.79362	2.85250	0.01219	2.86469
"FEFF0: U-Oax1-U-Oax1"	1.00000	2.000	0.01546	-8.79362	3.51020	-0.01035	3.49985
"FEFF0: U-Oax1-Oax2"	1.00000	2.000	0.00773	-8.79362	3.51580	-0.01035	3.50545
"FEFF0: U-Oax1-U-Oax2"	1.00000	2.000	0.00386	-8.79362	3.51580	-0.01035	3.50545
"FEFF3: Fe at 3.53"	1.00000	20.066	0.03957	-8.79362	3.52570	-0.26212	3.26358

EXAFS Parameters	
σ^2-values	-
Distances	+
Coordination numbers	-
ΔE-value	-

Signals became broad and unrealistic

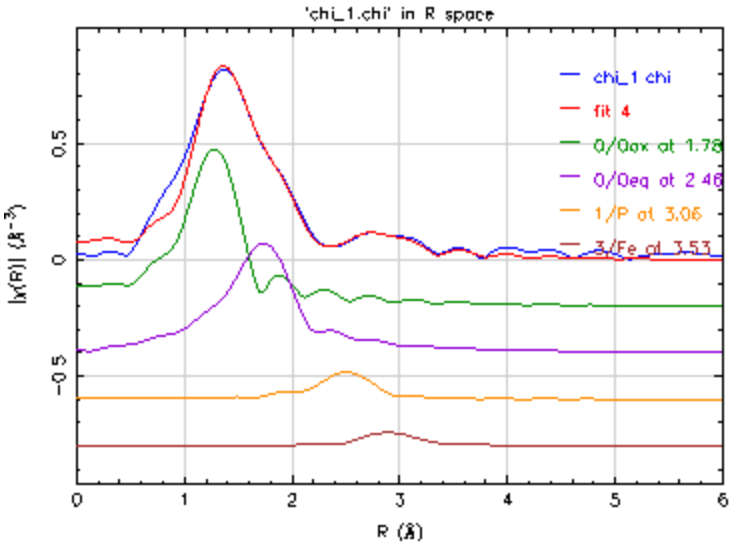
Test data for P and Fe shells

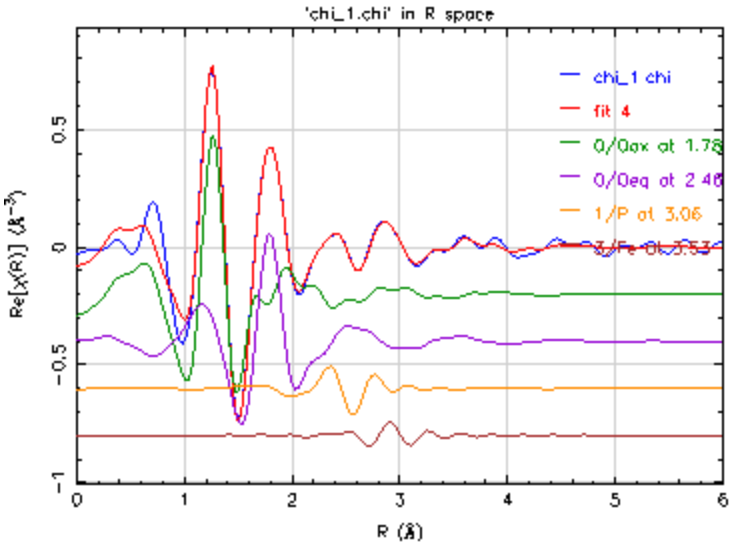




- Place P and Fe shells in a "good" spot
  - set parameters – no fitting
  - monitor results and fit spectra

Fit Results using P and Fe shells





Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

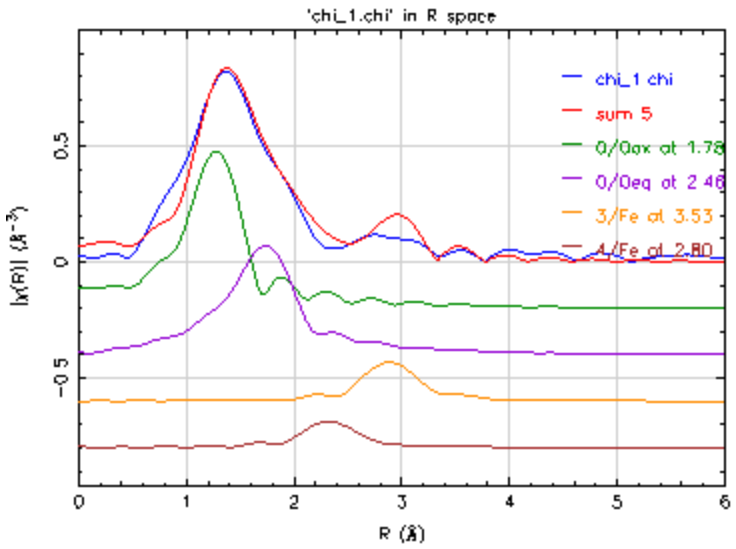
Results from the last fit — Column view — Save — Dismiss

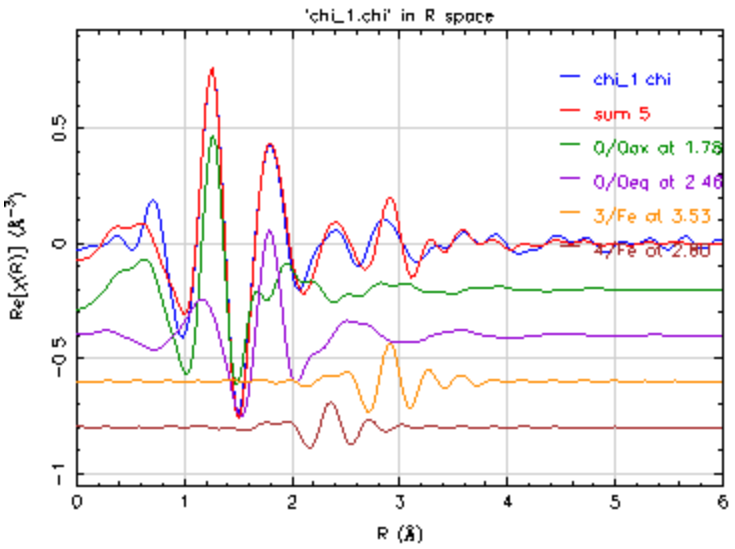
R-factor for this data set = 0.00264

path	degen	amp	sigma^2	e0	reff	delta_R	R
"FEFF0: Oax at 1.78"	1.00000	2.000	0.00351	-1.12310	1.75510	0.02066	1.77576
"FEFF0: Oeq at 2.46"	1.00000	4.615	0.00941	-1.12310	2.46160	-0.17609	2.28551
"FEFF0: U-Oax1-U-Oax1"	1.00000	2.000	0.01405	-1.12310	3.51020	0.04132	3.55152
"FEFF0: U-Oax1-Oax2"	1.00000	2.000	0.00702	-1.12310	3.51580	0.04132	3.55712
"FEFF0: U-Oax1-U-Oax2"	1.00000	2.000	0.00351	-1.12310	3.51580	0.04132	3.55712
"FEFF1: P at 3.06"	1.00000	0.835	0.00398	-1.12310	3.05550	0.00987	3.06537
"FEFF3: Fe at 3.53"	1.00000	0.276	0.00139	-1.12310	3.52570	-0.12876	3.39694

EXAFS Parameters	
σ^2-values	-
Distances	+
Coordination numbers	+
ΔE-value	+

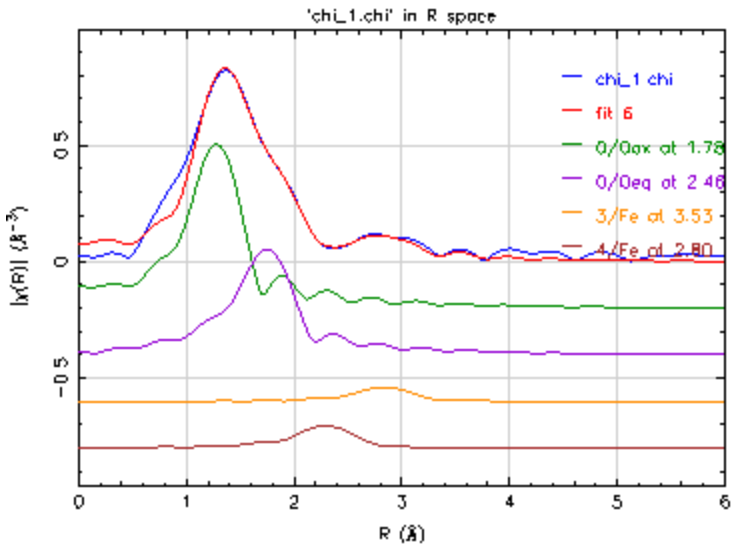
Test data for Fe and Fe shells

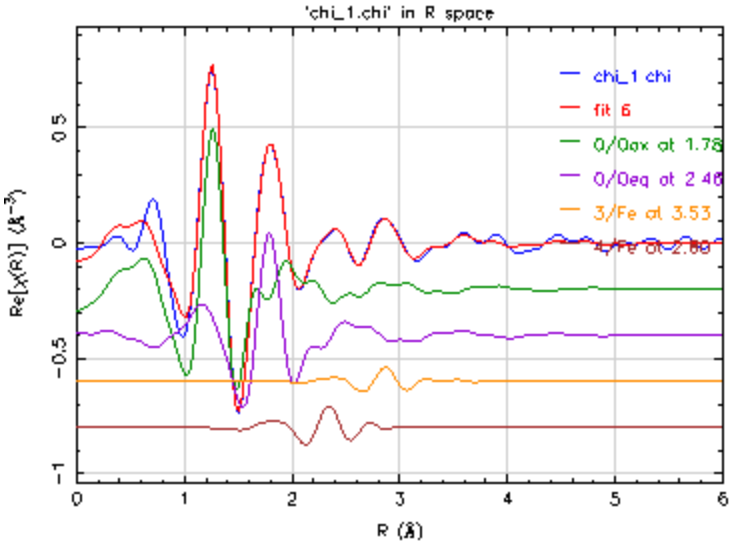




- **Place Fe and Fe shells in a "good" spot**
  - **set parameters – no fitting**
  - **monitor results and fit spectra**

Fit Results using Fe and Fe shells





Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit — Column view — Save — Dismiss

R-factor for this data set = 0.00254

path	degen	amp	sigma^2	e0	reff	delta_R	R
"FEFF0: Oax at 1.78"	1.00000	2.000	0.00320	-0.59470	1.75510	0.01998	1.77508
"FEFF0: Oeq at 2.46"	1.00000	3.235	0.00594	-0.59470	2.46160	-0.17897	2.28263
"FEFF0: U-Oax1-U-Oax1"	1.00000	2.000	0.01280	-0.59470	3.51020	0.03995	3.55015
"FEFF0: U-Oax1-Oax2"	1.00000	2.000	0.00640	-0.59470	3.51580	0.03995	3.55575
"FEFF0: U-Oax1-U-Oax2"	1.00000	2.000	0.00320	-0.59470	3.51580	0.03995	3.55575
"FEFF3: Fe at 3.53"	1.00000	0.670	0.00830	-0.59470	3.52570	-0.16117	3.36453
"FEFF4: Fe at 2.80"	1.00000	0.669	0.00830	-0.59470	2.80090	0.03782	2.83872

EXAFS Parameters	
σ^2-values	+
Distances	+
Coordination numbers	+
ΔE-value	+

Comparison of Models

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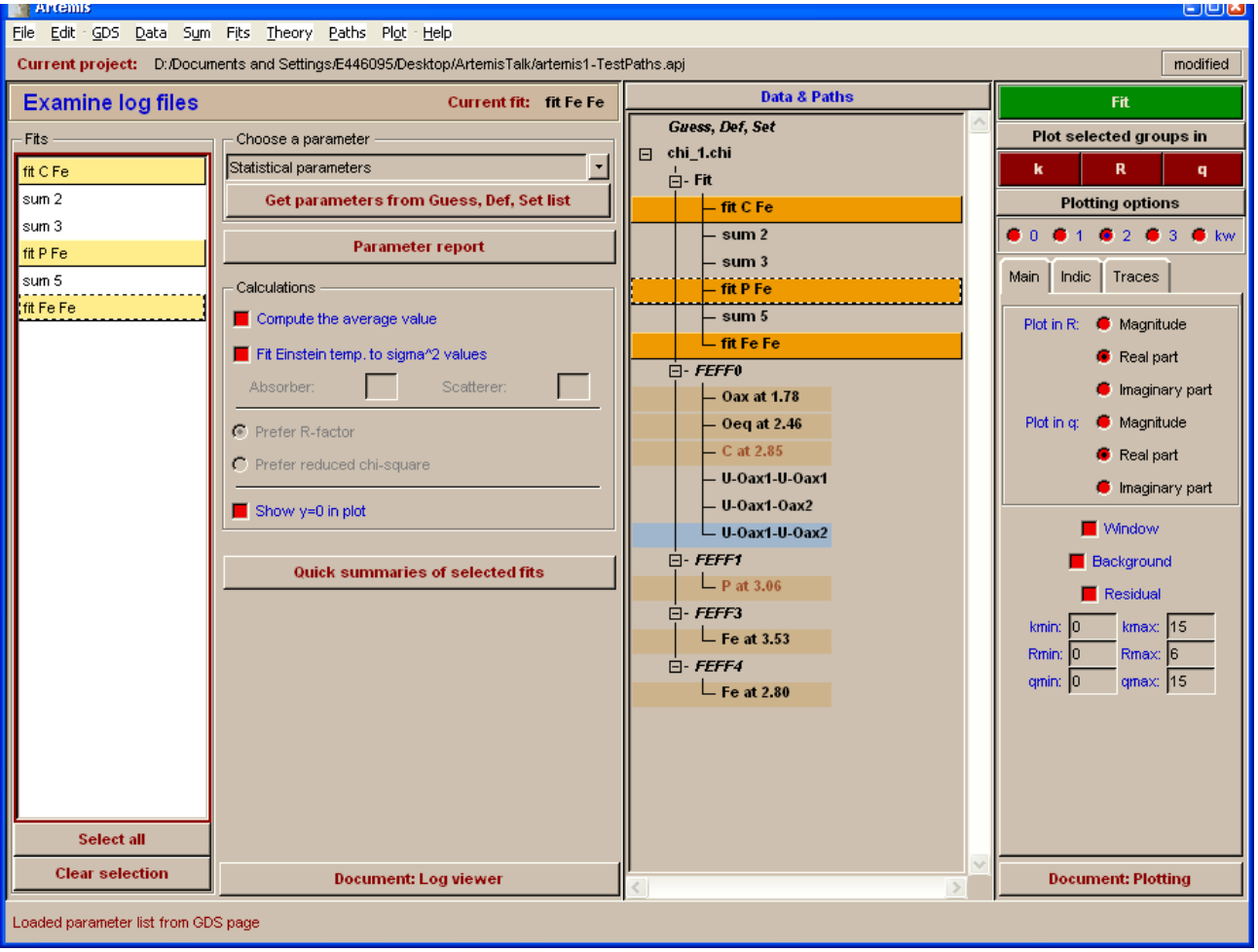
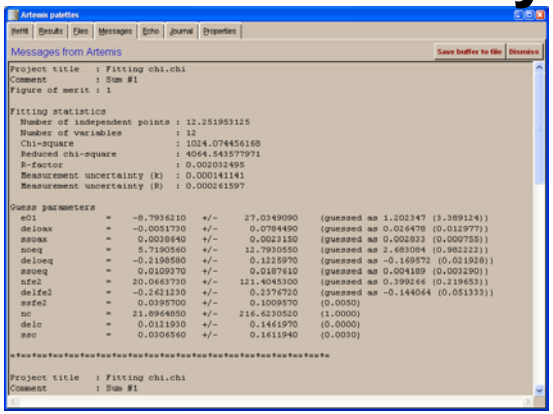
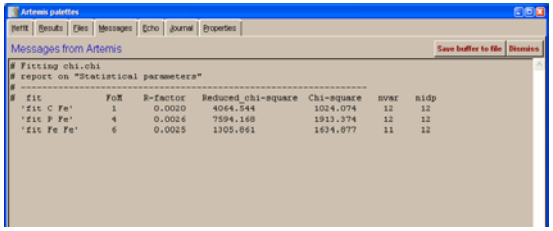


Quick summary



Parameter Report



- **Models are saved in Artemis and can be compared or re-instated later**

Multiple data set fit

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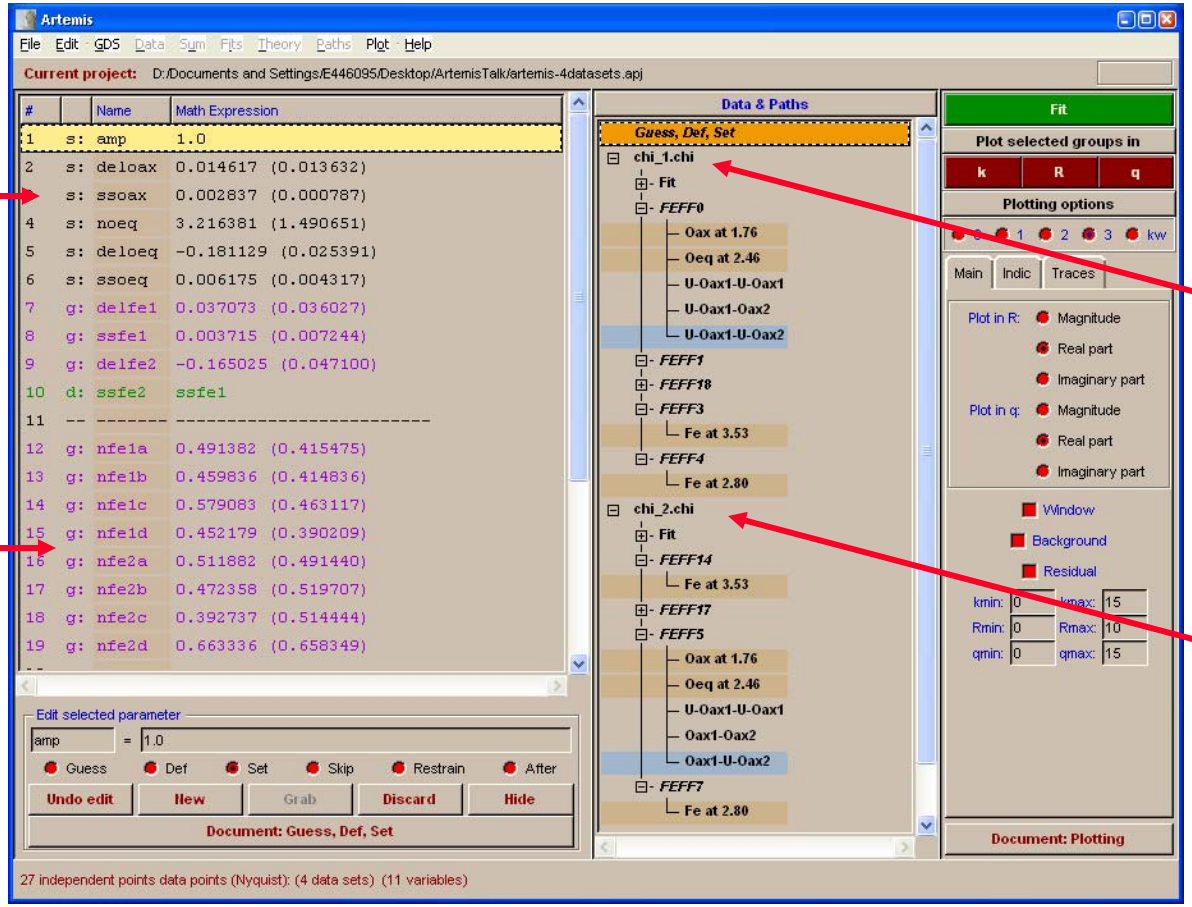
Common variables for all 4 data sets

variables for each data set



Data set 1

Data set 2

- Fit 4 data sets
- Use K-weights of 1, 2 and 3
- Fit different N values for each data set
- Data series is needed to accurately determine the number of Fe atoms in the 2nd and 3rd shells

Modeling a Data Series

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Artemis palettes

Ifeffit | Results | Files | Messages | Echo | Journal | Properties

Results from the last fit

```
Environment      :  Artemis 0.8.013 using Windows XP, perl 5.008008, Tk 80
Fit label        :  fit Fe Fe
Data sets        :  "chi_1.chi", "chi_2.chi", "chi_3.chi", "chi_4.chi"
Figure of merit :  7

============================================================


Fitting statistics
  Number of independent points : 26.578125000
  Number of variables          : 11
  Chi-square                   : 9490.351885016
  Reduced chi-square           : 609.210151094
  R-factor                     : 0.057174889
  Measurement uncertainty (k)  : 0.000196055
  Measurement uncertainty (R)  : 0.000220316

Guess parameters
  delfe1          =     0.0422810   +/-      0.0123250     (guessed as 0.0
  ssfe1           =     0.0050600   +/-      0.0047000     (guessed as 0.0
  delfe2          =    -0.1704970   +/-      0.0137850     (guessed as -0.
  nfe1a           =     0.5063030   +/-      0.2914520     (guessed as 0.4
  nfe1b           =     0.5505380   +/-      0.3310320     (guessed as 0.4
  nfe1c           =     0.6513330   +/-      0.3598810     (guessed as 0.5
  nfe1d           =     0.4671820   +/-      0.2862200     (guessed as 0.4
  nfe2a           =     0.5683350   +/-      0.3168750     (guessed as 0.5
  nfe2b           =     0.5138540   +/-      0.3234190     (guessed as 0.4
  nfe2c           =     0.4784870   +/-      0.3155270     (guessed as 0.3
  nfe2d           =     0.7617610   +/-      0.4179530     (guessed as 0.6

Def parameters (using "FEFF0: Oax at 1.76")
```