Brief tutorial on X ray powder diffraction data analysis

- Essential bibliography
- X-ray powder diffraction (XRPD): brief summary
- Qualitative Analysis: evaluate your pattern and look for possible phase(s)
- Quantitative Analysis: Rietveld refinement

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Warning

These notes represent an introduction to x-ray powder diffraction analysis, far from exhaustive but intended to drive the Reader, who has collected its first XRD data, through the different steps that will bring him to:

i. inspect the diffractograms in order to check the data quality and obtain preliminary rough information about sample nature, crystallinity, etc...;

ii. compare the experimental diffractograms with those of models and reference compounds, in order to make preliminary hypothesis about sample structure and composition;

iii. perform the full pattern structural refinement (Rietveld method) in order to achieve a first quantitative understanding of the crystallographic structure of the samples.

However the Reader must keep in mind that XRPD data analysis is far from automatic, instead it is a complex procedure requiring competence and experience, and often tumbles across a slow learning process via trial and error process.

Repository

Grado_2013_XRD_tutorial.pdf
https://db.tt/7UXhsrWR

XRPD_tutorial_Grado2013.zip
https://db.tt/wopyl8TS

Essential bibliography XRPD

- B.E. Warren, **X-Ray Diffraction** (Addison-Wesley, **1990**).
- H.P. Klug and L.E. Alexander, X-Ray Diffraction Procedures (Wiley Interscience, **1974**).
- B.D. Cullity, Elements of X-Ray Diffraction (Wiley, **1978**).
- Modern Powder Diffraction Reviews in Mineralogy, Vol. 20 Mineralogical Society of America, (**1989**).
- Fundamentals of Crystallography IUCr Texts on Crystallography -2 C. Giacovazzo, (Oxford Science Publication, 1992.
- The Rietveld Method IUCr Monographs on Crystallography - 5 R.A. Young, Editor Oxford Science Publication, 1993.
- X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials H.P Klug and L.E. Alexander Wiley-Interscience, 1974, 2nd edition.
- Defects and Microstructure Analysis by Diffraction R.L. Snyder, J. Fiala and H.J. Bunge, IUCr Monographs on Crystallography, Vol 10, Oxford Science Publications, 1999.
- Diffraction Analysis of the Microstructure of Materials
- Diffraction Analysis of the Microstructure of Materials E. J. Mittemeijer, P. Scardi Springer (2004)

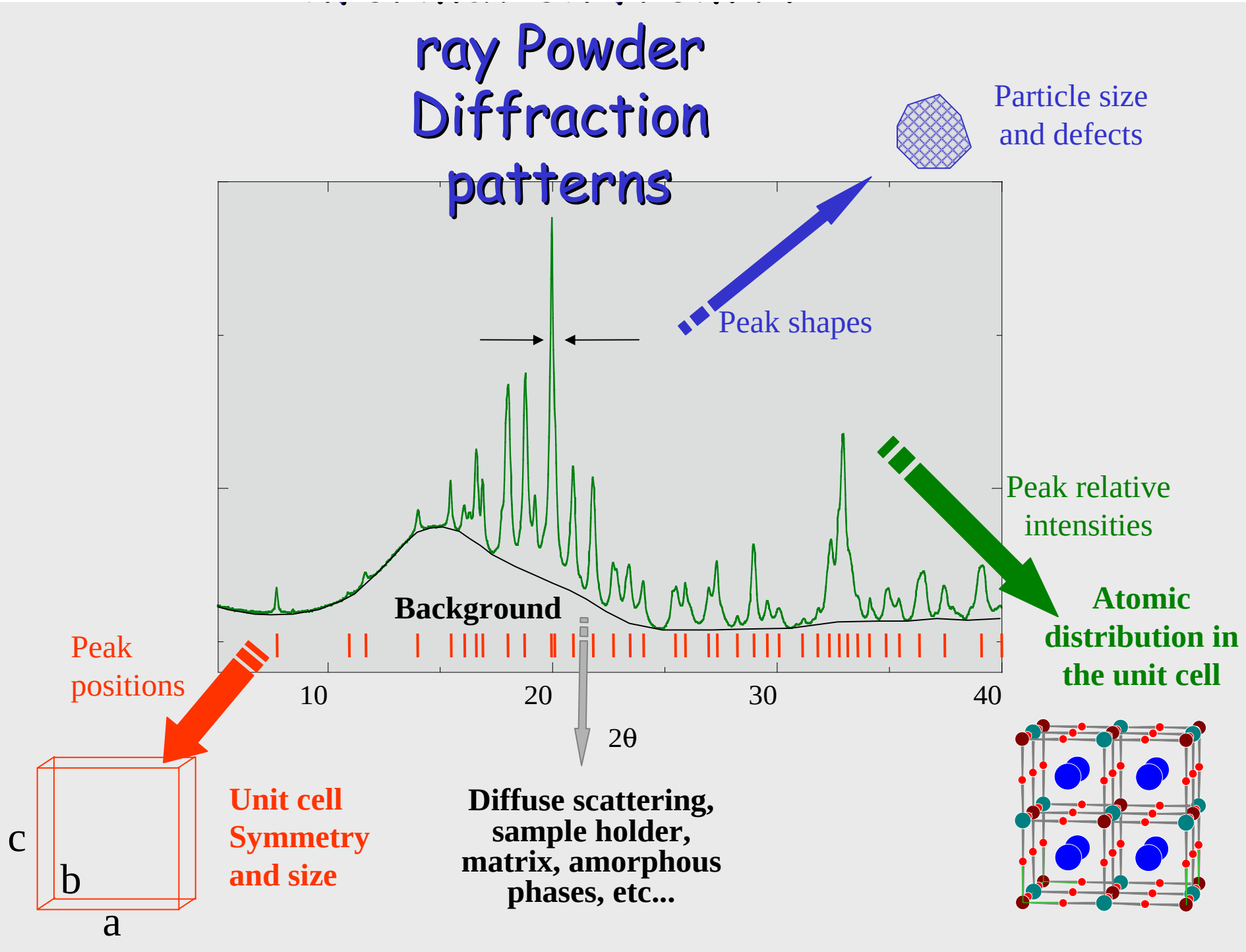
On line resources

http://epswww.unm.edu/xrd/resources.htm → A resource page for XRD

http://www.ccp14.ac.uk/ → The collaborative computational projects

http://www.icdd.com/ → International centre for diffraction data

ray Powder Diffraction patterns



NOTE

Ab initio recognition and structural refinement of crystallogrphic structure of unknown phase(s) (i.e.: direct methods) is a hardly complex task**

It is easier (and it is often the case) to refine the crystallographic structure (and phase composition) of a sample exploiting the a-priori knowledge you may have about your sample, that is: starting from models, hypothesis, patterns database, etc...

** Ab initio structure determination from Powder diffraction data

•Harris, K.D.M., M. Tremayne, and M. Kariuki. Contemporary Advances in the Use of Powder X-Ray Diffraction for Structure Determination, Angew. Chem. Int. Ed. 40 (2001) 1626-1651.

•Giacovazzo, C. Direct Methods and Powder Data: State of the Art and Perspectives, Acta Crystallogr. A52 (1996) 331-339.

•Scardi, P., et al. International Union of Crystallography Commission for Powder Diffraction. http://www.iucr.org/iucr-top/comm/cpd/

XRPD experiment has gone!

We have data....

2008_04_28_AU_SI_FE_MULTI_PRISTINE_E_D27.3_1.LOG - Blocco note

File Modifica Formato Visualizza ?

7058.3	6637.87	6637.5	27.3	2.348691E9	5.093221E7	7.3	
7063.16	6633.3	6633	27.3	2.393167E9	5.068094E7	7.3526E-10	30
7068.25	6628.53	6628.5	27.3	2.360922E9	5.18502E7	7.319033E-10	30
7073.23	6623.86	6623.55	27.3	2.407742E9	5.19483E7	7.3038E-10	30
7078.22	6619.19	6619.05	27.3	2.379208E9	5.14425E7	7.335633E-10	30
7083.22	6614.51	6614.1	27.3	2.359907E9	5.242249E7	7.3719E-10	30
7088.23	6609.84	6609.6	27.3	2.356088E9	5.266497E7	7.3772E-10	30
7093.13	6605.27	6605.1	27.3	2.342297E9	5.303566E7	7.3482E-10	30
7098.15	6600.59	6600.15	27.3	2.317491E9	5.265541E7	7.3115E-10	30
7099.22	6599.6	6599.25	27.3	2.443618E9	5.28878E7	7.325067E-10	30
7100.18	6598.7	6598.35	27.3	2.464316E9	5.354216E7	7.370267E-10	30
7101.25	6597.71	6597.45	27.3	2.5068E9	5.342406E7	7.365033E-10	30
7102.22	6596.82	6596.55	27.3	2.387894E9	5.346274E7	7.324167E-10	30
7103.18	6595.92	6595.65	27.3	2.35998E9	5.40353E7	7.315033E-10	30
7104.14	6595.03	6594.75	27.3	2.376211E9	5.416876E7	7.3519E-10	
7105.21	6594.03	6593.85	27.3	2.381363E9	5.377292E7	7.377567E-	
7106.18	6593.14	6592.95	27.3	2.392809E9	5.454645E7	7.335033E-	
7107.25	6592.14	6592.05	27.3	2.492729E9	5.376432E7	7.300567E-	
7108.21	6591.25	6591.15	27.3	2.427193E9	5.474856E7	7.339067E-	
7109.18	6590.35	6590.25	27.3	2.272422E9	5.560243E7	7.387567E-	
7110.25	6589.36	6589.35	27.3	2.334179E9	5.529091E7	7.348767E-	
7111.22	6588.46	6588.45	27.3	2.471662E9	5.730961E7	7.3075E-10	
7112.18	6587.57	6587.55	27.3	2.454253E9	6.138133E7	7.330133E-	
7113.26	6586.57	6586.2	27.3	2.479547E9	6.535016E7	7.374733E-	
7114.22	6585.68	6585.3	27.3	2.314557E9	6.697034E7	7.361667E-	

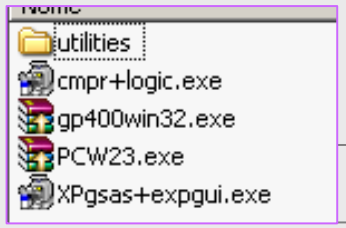
And now?

www.fis.uniroma3.it/~meneghini/software.html

XRPD_tutorial.exe

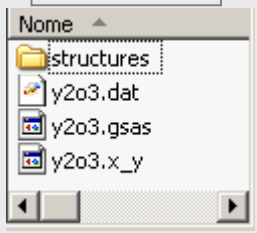
Download and install:
XRD_tutorial.exe

XRD_tutorial folder

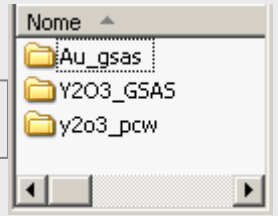


Programs

Data



Analysis



Gp400win32.exe

expand

gnuplot

bin

wgnuplot.exe

VESTA

PCW23.exe

GSAS

CMPR

Y2O3_PCW

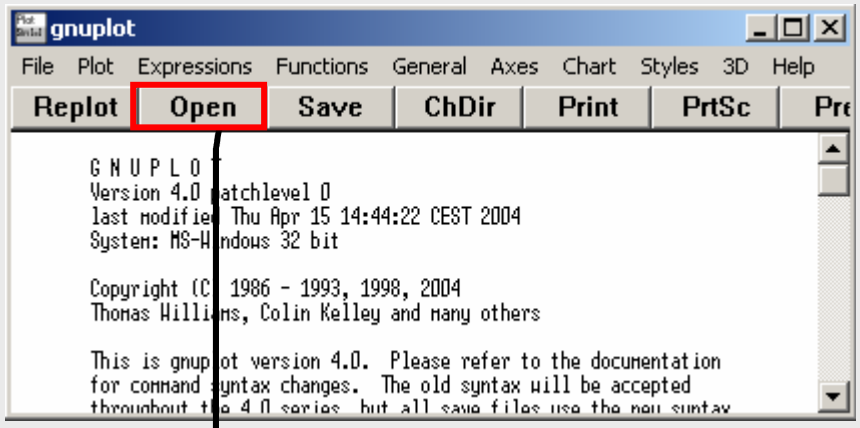
Y2O3_GSAS

Au_GSAS

Firstly: get a look to the data!

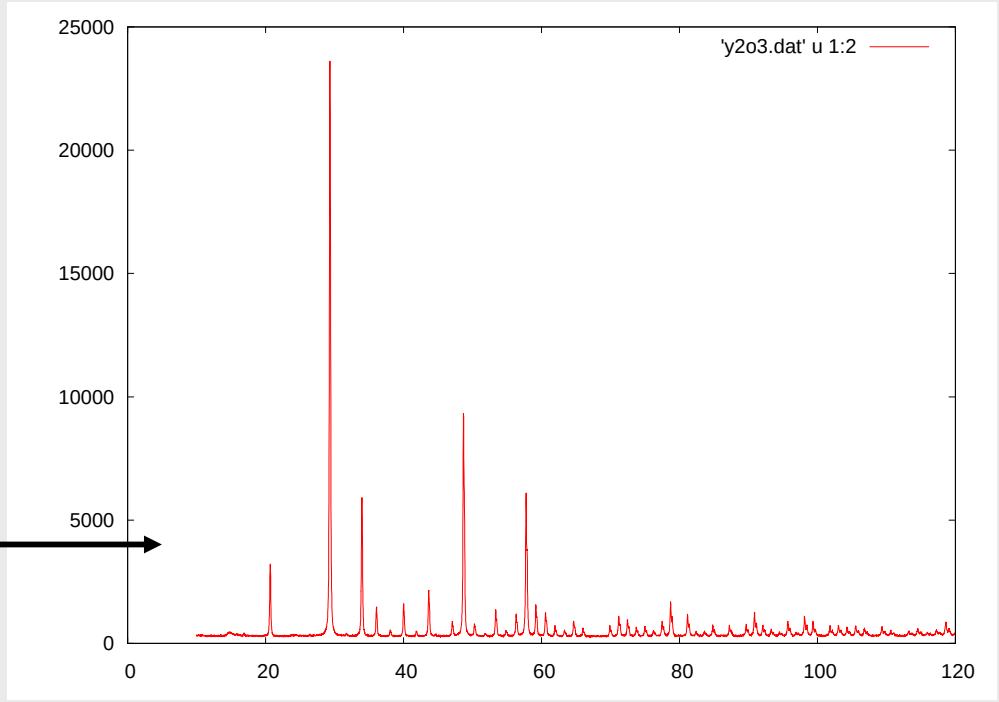
More information on Gnuplot at:

www.gnuplot.info



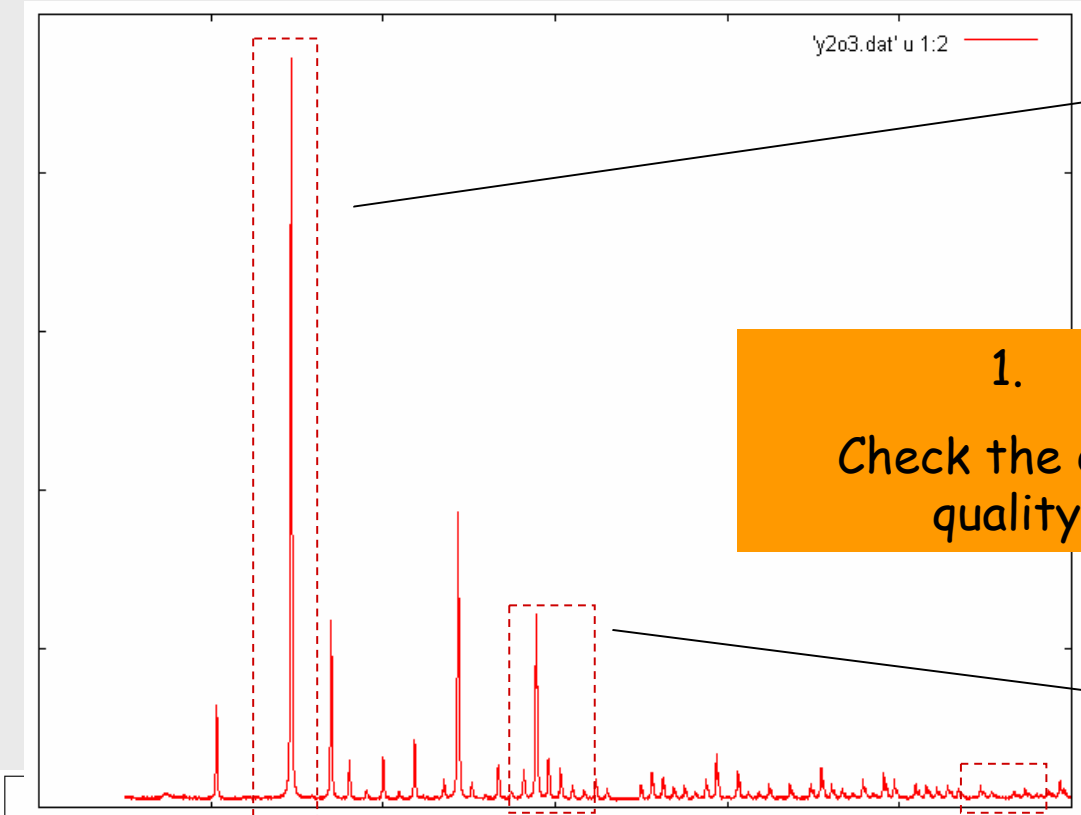
.../data

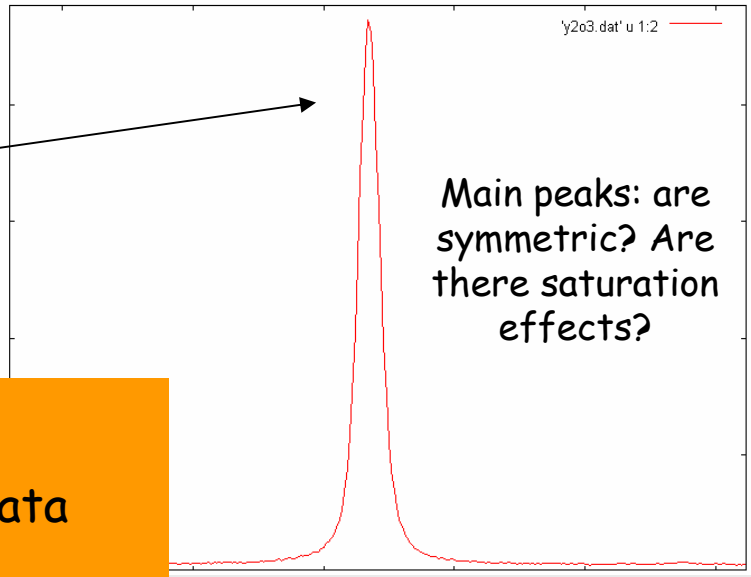
plo_y2O3.plt




```
gnuplot> pl [:][:]  'y2o3.dat' u  1:2  w l
```

plot — x-range, y-range — file — using x:y columns — with lines

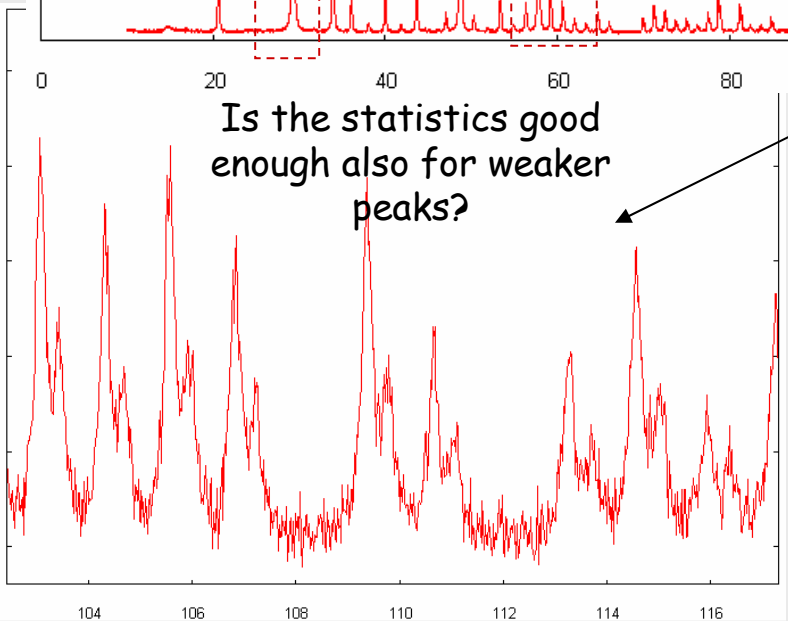




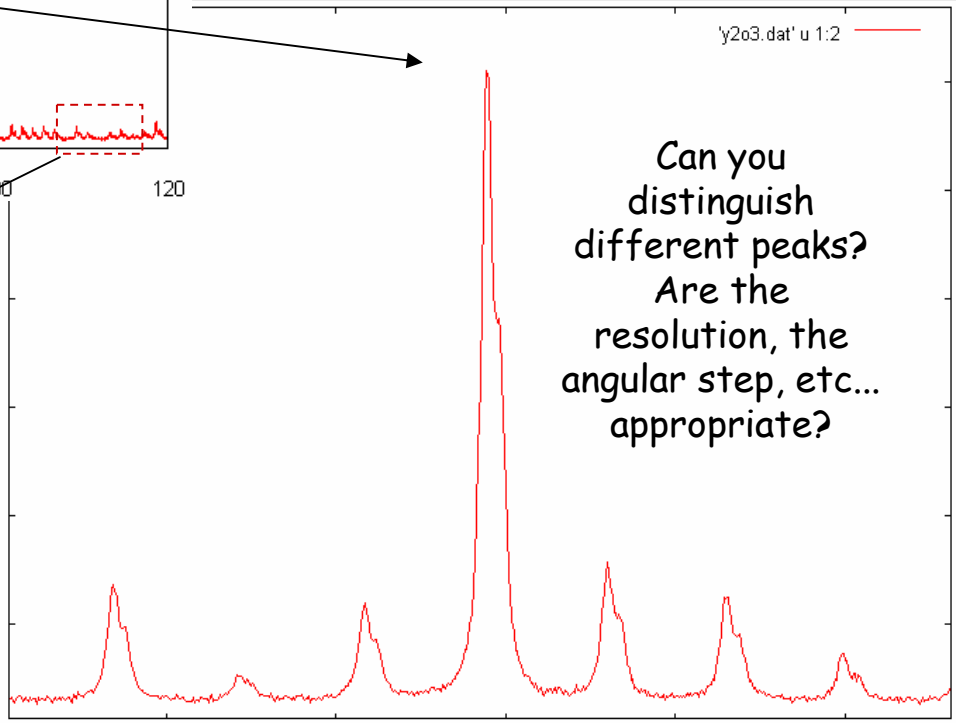
Main peaks: are symmetric? Are there saturation effects?

1.

Check the data quality



Is the statistics good enough also for weaker peaks?

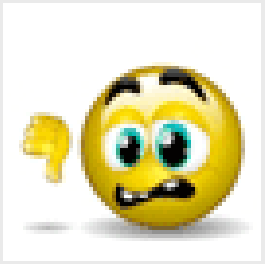


Can you distinguish different peaks? Are the resolution, the angular step, etc... appropriate?

If the patterns are good...

...go ahead

If not...



Note: Data collection on S.R. is definitively faster than in laboratory but:

6-12 months from proposal submission to experiment . . .

!!! (if you are lucky) !!!

...consider to recollect the XRPD patterns

SECOND: compare your data with models based on your a priori knowledge on the sample

Compare your diffractograms with patterns expected for compounds of similar composition

Look for the structure of know compounds on database!

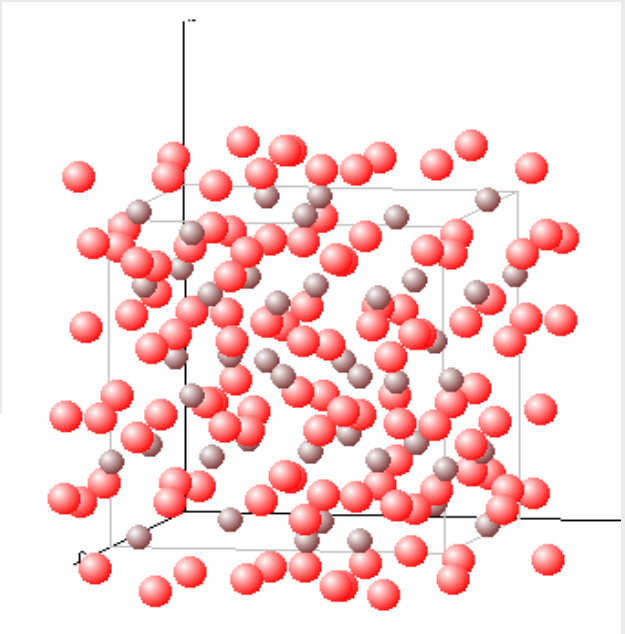
http://database.iem.ac.ru/mincryst/

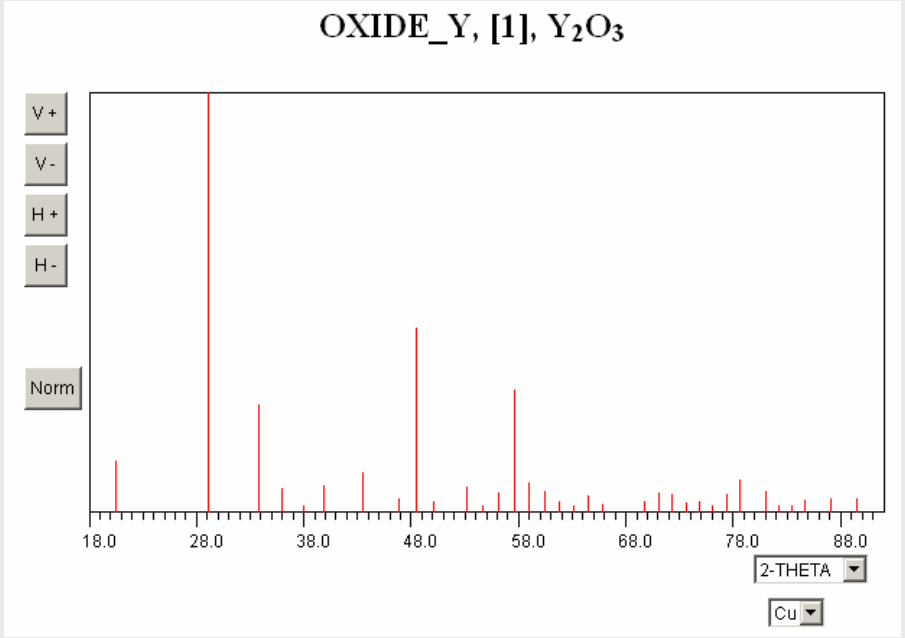
http://webmineral.com/

http://barns.ill.fr/

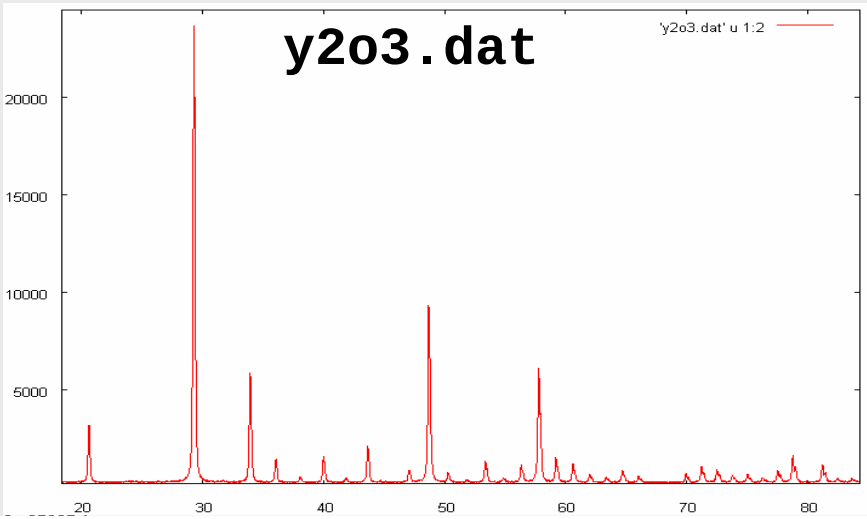
Note:
SR facilities have often access to private DataBase closed to your institution!

http://database.iem.ac.ru/mincryst/



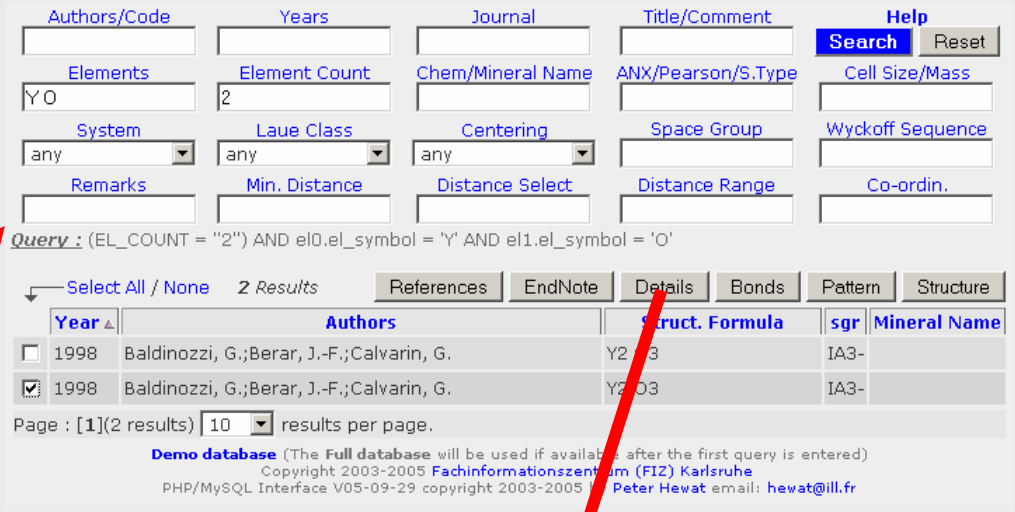


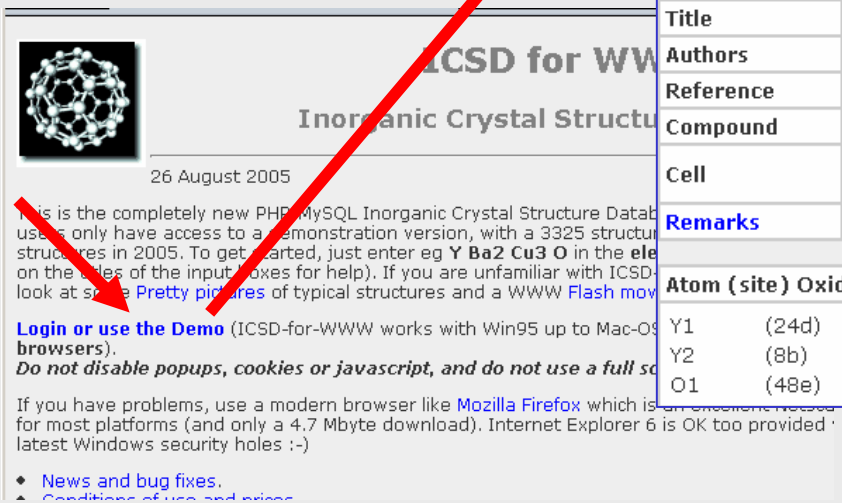

```
OXIDE_Y

1

Y(2)O(3)


Cubic   I a3   Z = 16

R =0.082

6 .7 .93

Ref.Str.:
         Paton M.G., Maslen E.N. (1965)
        * Acta Cryst., 19, 307-310
        R: D.T.N.


                    Initial data from file TRANS.DAT

  Lattice parameters (cub. angs.,degr.):

              a =  10.6040   alpha =   90.00
              b =  10.6040   beta  =   90.00
              c =  10.6040   gamma =   90.00

  Unit cell volume (cub. angs.) =  1192.36
```

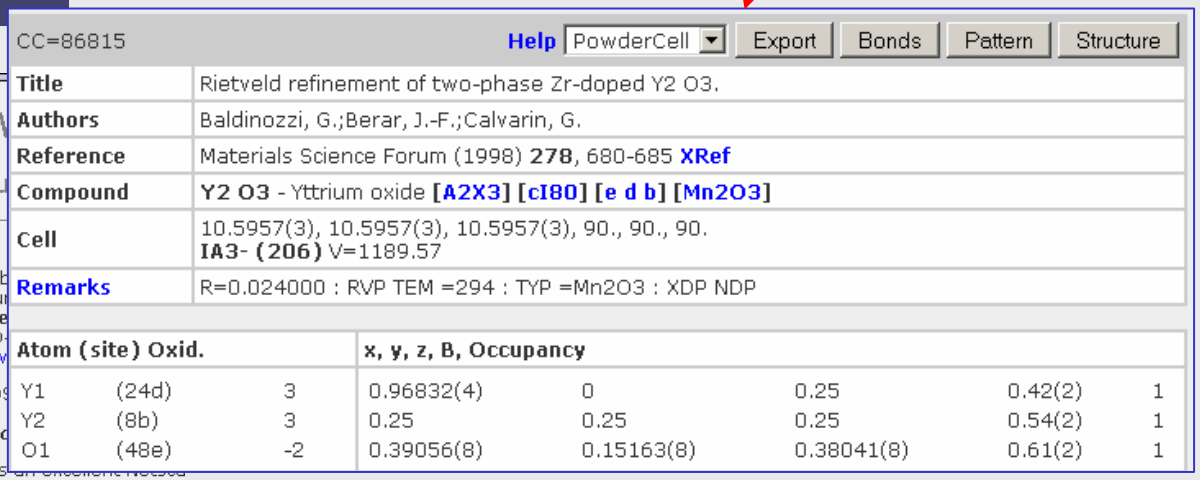


ICSD public version

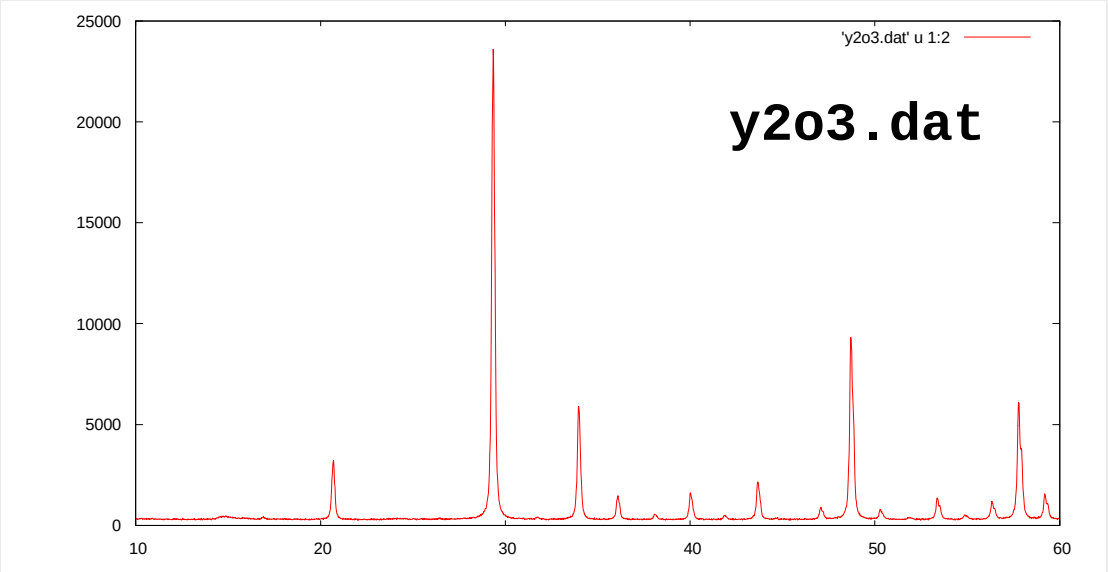
http://barns.ill.fr

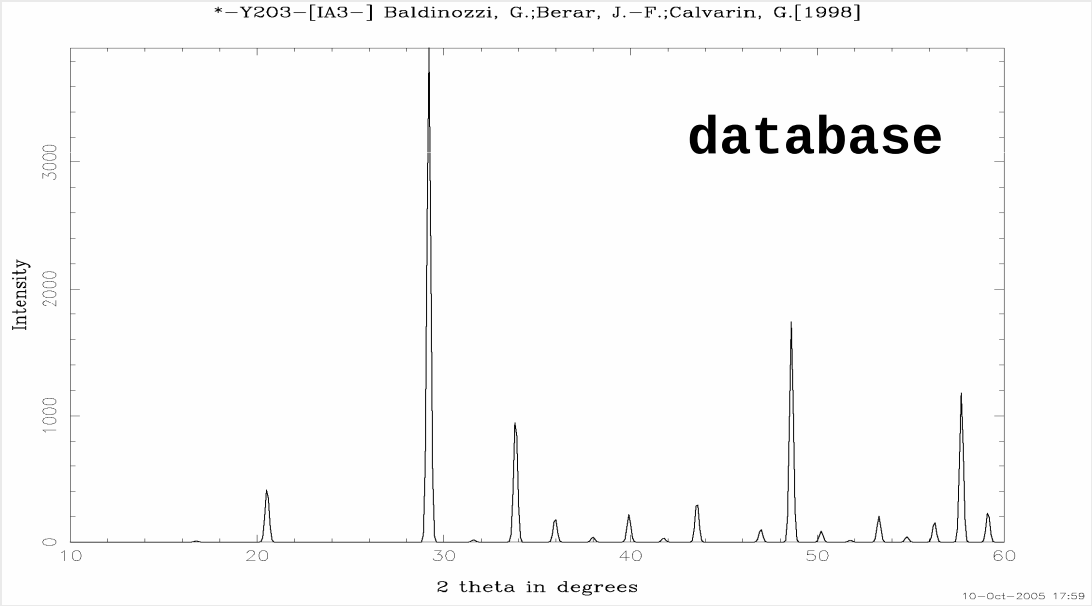






Your data



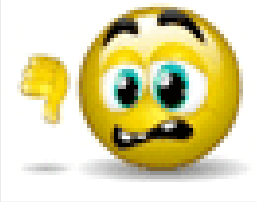


Literature Data

They reasonably match!

go ahead!

If not... maybe your sample is wrong



Go deeper into the data

ICSD Details

1 entry selected.
CC=Collection Code: [AB2X4]=ANX Form: [cF56]=Pearson: [e d a]=Wyckoff Symbol: [Al2MgO4]=Structure Type:
Click the ANX, Pearson or Wyckoff Symbol to find structures with that symbol.

CC=86815 **Help** PowderCell Export Bonds Pattern Structure

Title	Rietveld refinement of two-phase Zr-doped Y2 O3.
Authors	Baldinozzi, G.;Berar, J.-F.;Calvarin, G.
Reference	Materials Science Forum (1998) **278**, 680-685 **XRef**
Compound	**Y2 O3** - Yttrium oxide **[A2X3] [cI80] [e d b] [Mn2O3]**

save file: icsd_86815.**cel**

CC=86815 **Help** CIF Export Bonds Pattern Structure Jmol

Title	Rietveld refinement of two-phase Zr-doped Y2 O3.
Authors	Baldinozzi, G.;Berar, J.-F.;Calvarin, G.
Reference	Materials Science Forum (1998) **278**, 680-685 **Link XRef SCOPUS SCIRUS Google**
Compound	**O3 Y2** - Yttrium oxide **[A2X3] [cI80] [e d b] [Mn2O3]**
Cell	10.5957(3), 10.5957(3), 10.5957(3), 90., 90., 90. **IA3- (206)** V=1189.57
Remarks	R=0.024000 : RVP TEM =294 : TYP =Mn2O3 : XDP NDP

Atom (site) Oxid.			**x, y, z, B, Occupancy**				
Y1	(24d)	3	0.96832(4)	0	0.25	0.42(2)	1
Y2	(8b)	3	0.25	0.25	0.25	0.54(2)	1
O1	(48e)	-2	0.39056(8)	0.15163(8)	0.38041(8)	0.61(2)	1

save file: icsd_86815.**cif**

icsd_86815.cel

```
CELL 10.595700 10.595700 10.595700 90.000000 90.000000 90.000000
natom   3
Y1      39 0.968320 0.000000 0.250000
Y2      39 0.250000 0.250000 0.250000
O1      8 0.390560 0.151630 0.380410
rgnr 206
```

icsd_86815.**cel**

icsd_86815.**cif**

icsd_86815.cif

```
##############################################################################
# Baldinozzi, G.;Berar, J.-F.;Calvarin, G. (1998)
# Materials Science Forum 278, 680-685
# Rietveld refinement of two-phase Zr-doped Y2 O3
#
# CIF by ICSD-for-WWW,  Copyright 2003 FIZ-Karlsruhe & A.W.Hewat
# NOT TO BE PUBLISHED IN ANY FORM. See http://icsd.ill.fr/icsd/c
##############################################################################

data_86815-ICSD
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'Y2 O3'
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```
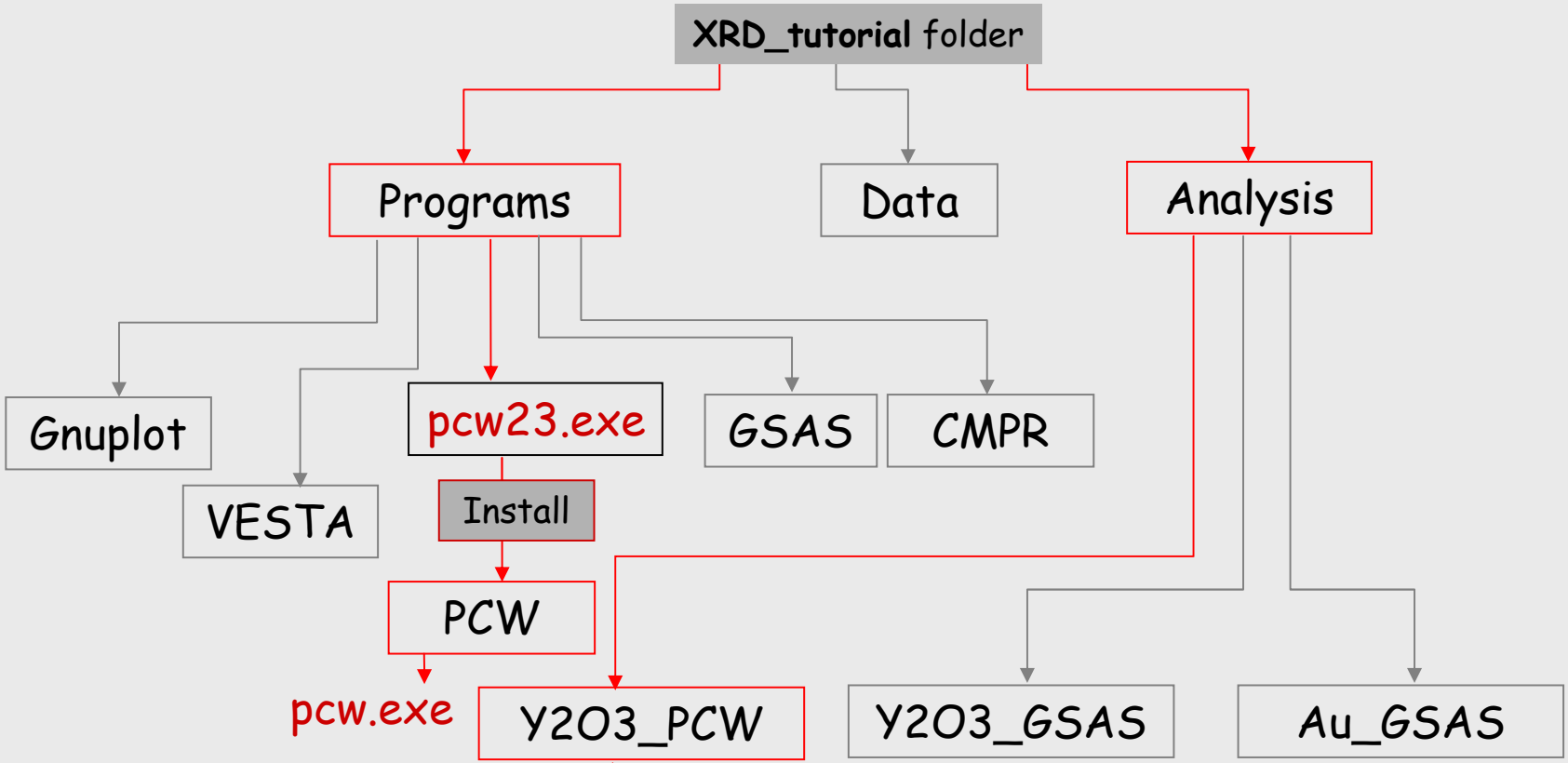

PowderCell

PCW



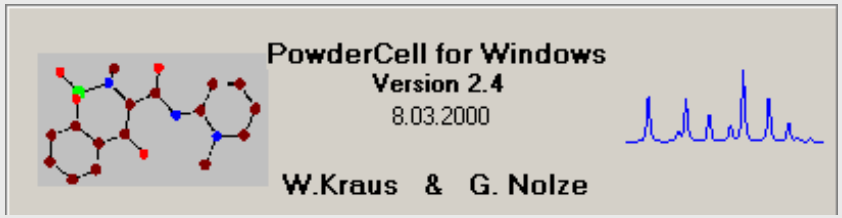
PowderCell is a simple to handle program allowing:

-structural visualization,

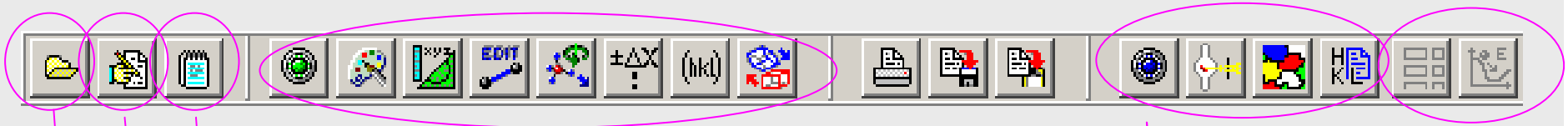
-theoretical XRPD pattern calculation

-Rietveld refinement

- etc...



http://users.omskreg.ru/~kolosov/bam/a_v/v_1/powder/details/pcwindex.htm



generate the pattern

Play with the structure

look at the whole cell

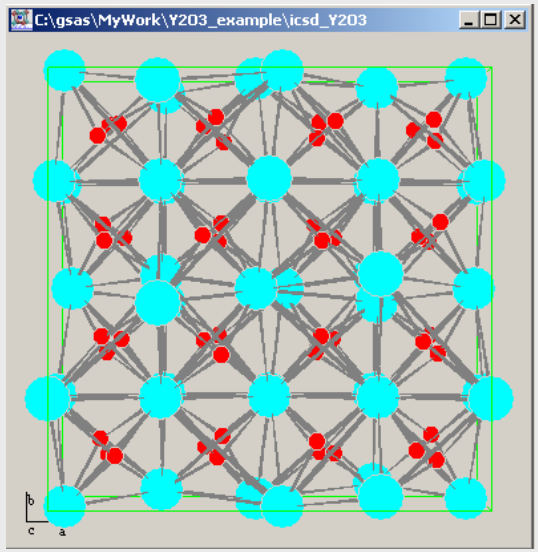
pattern refinement

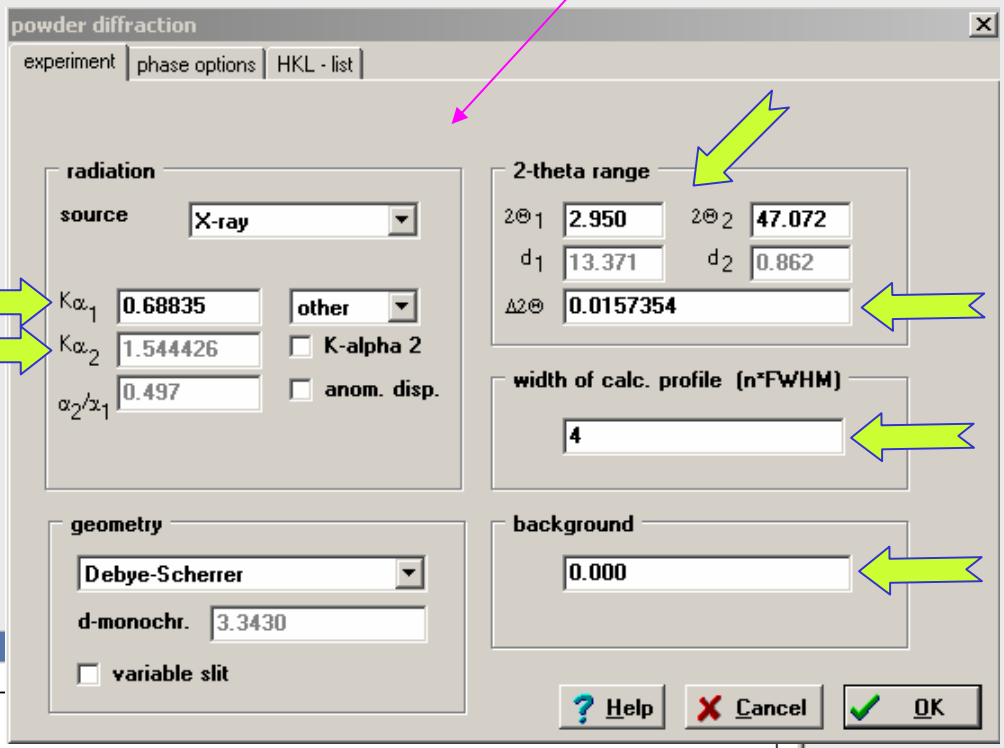
Modify/Create the unit cell

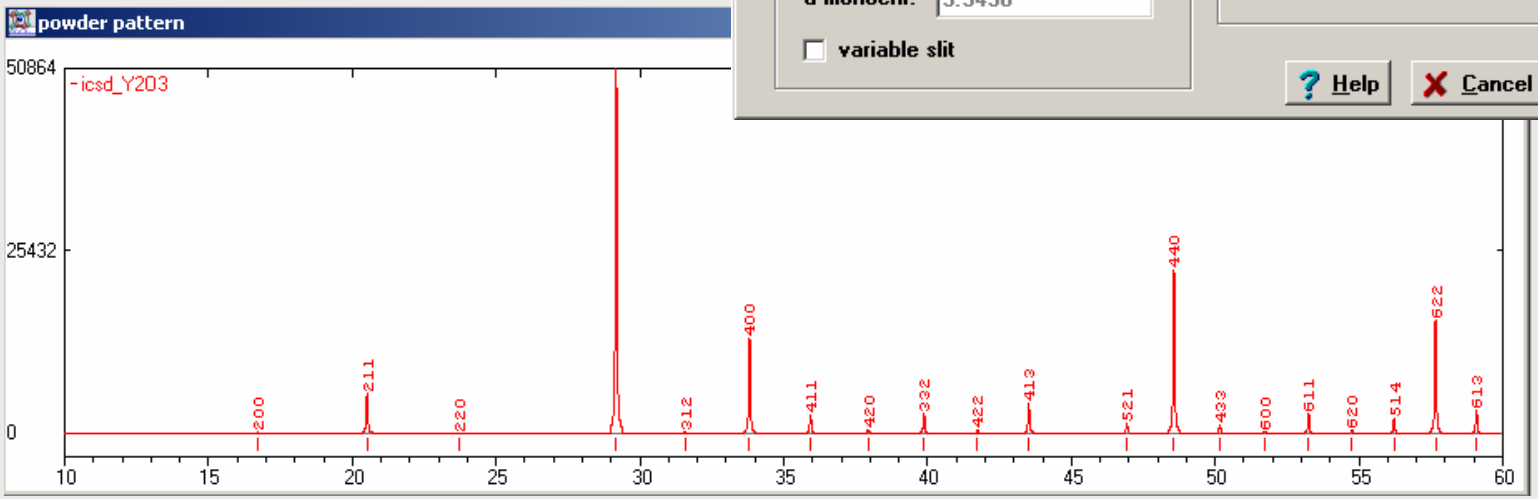
Load structure file (.cel)

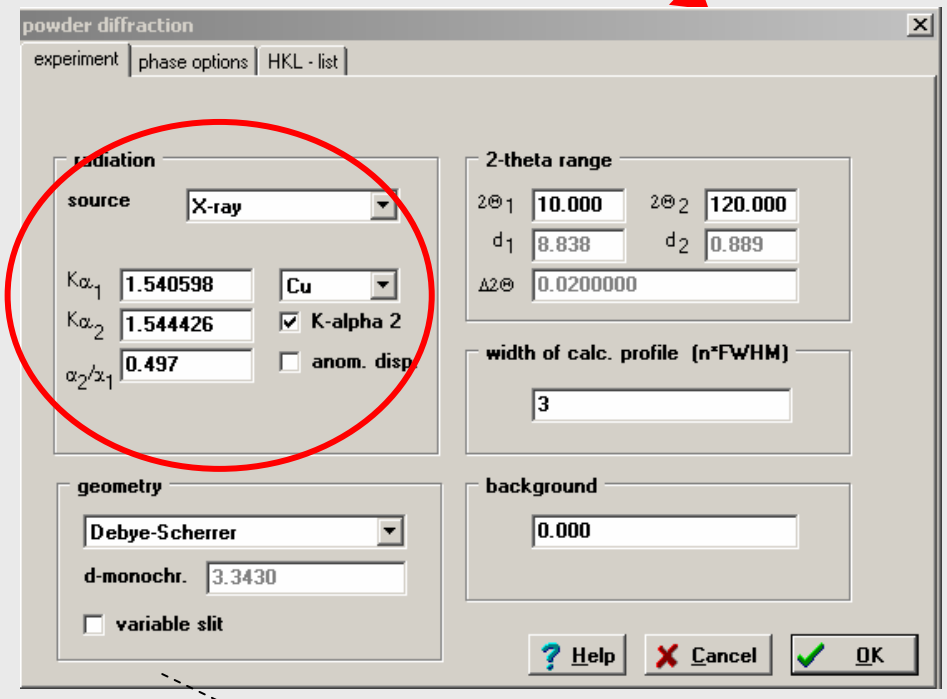
Load structure file

icsd_86815.cel





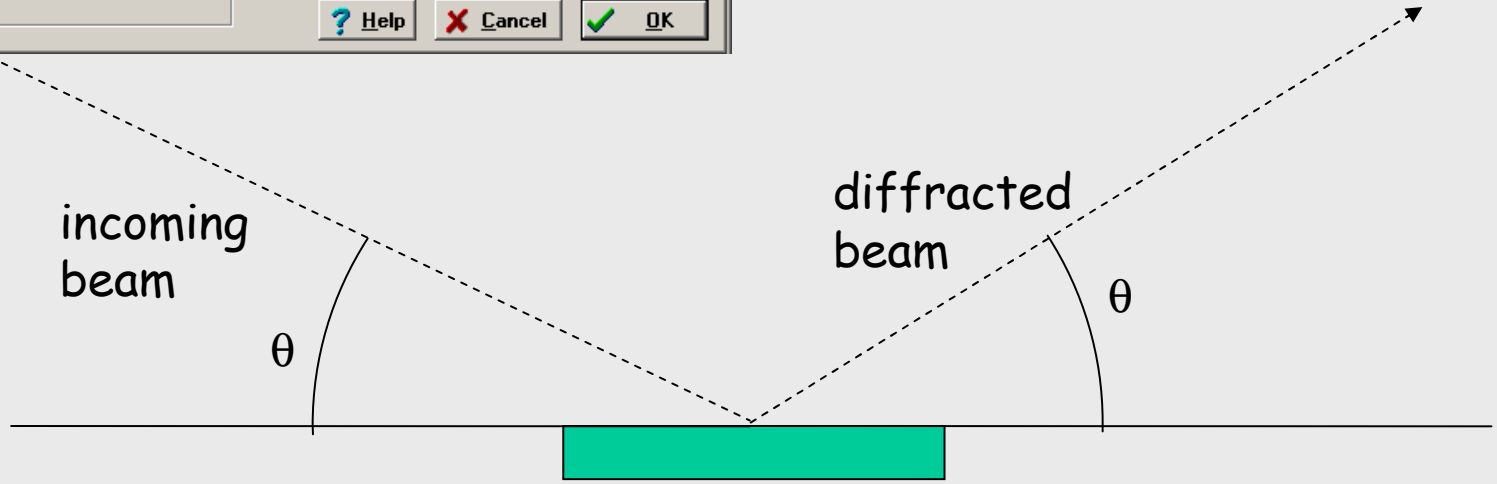


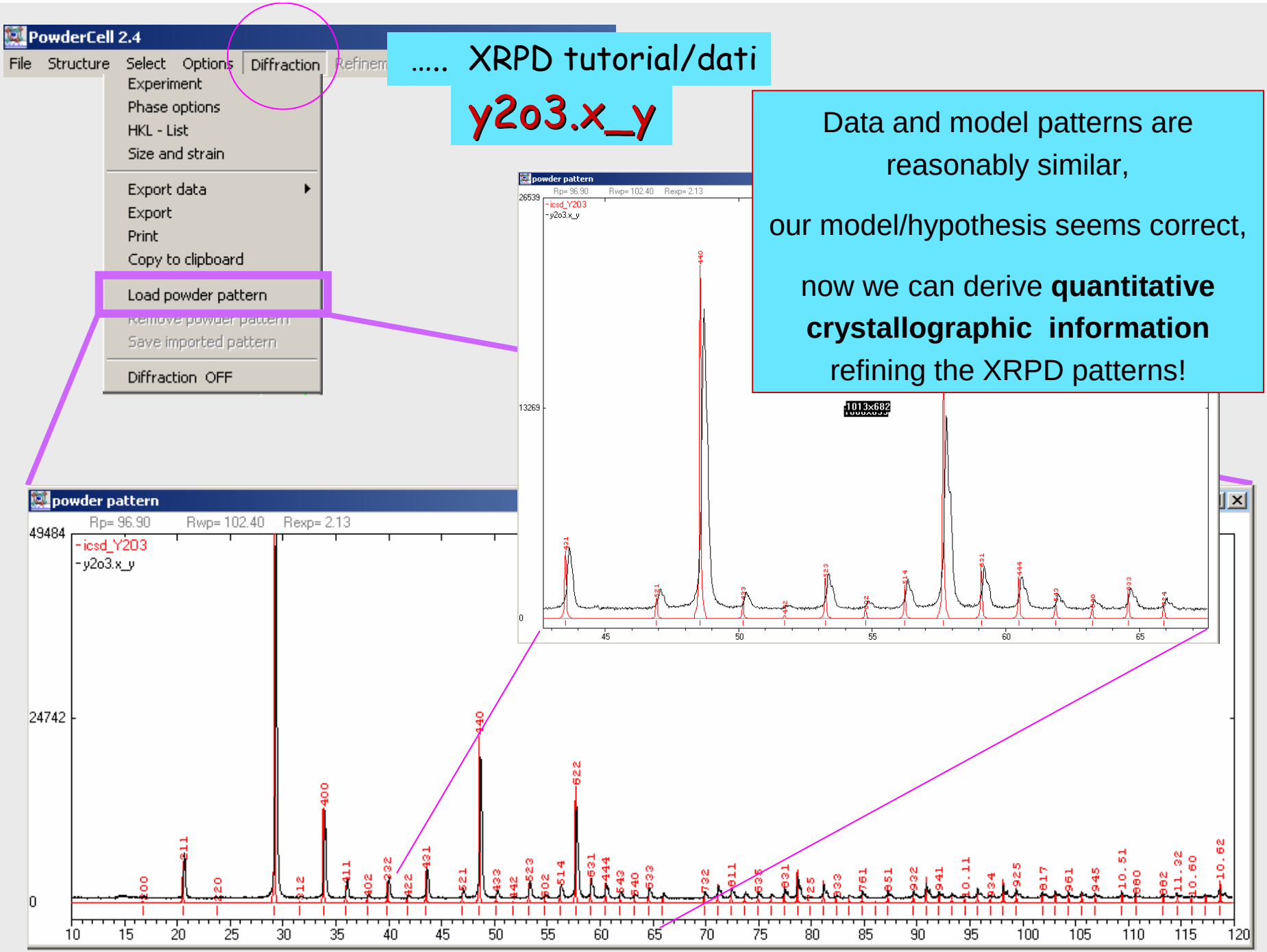


Provide here the information about the experiment, mainly:

Wavelength

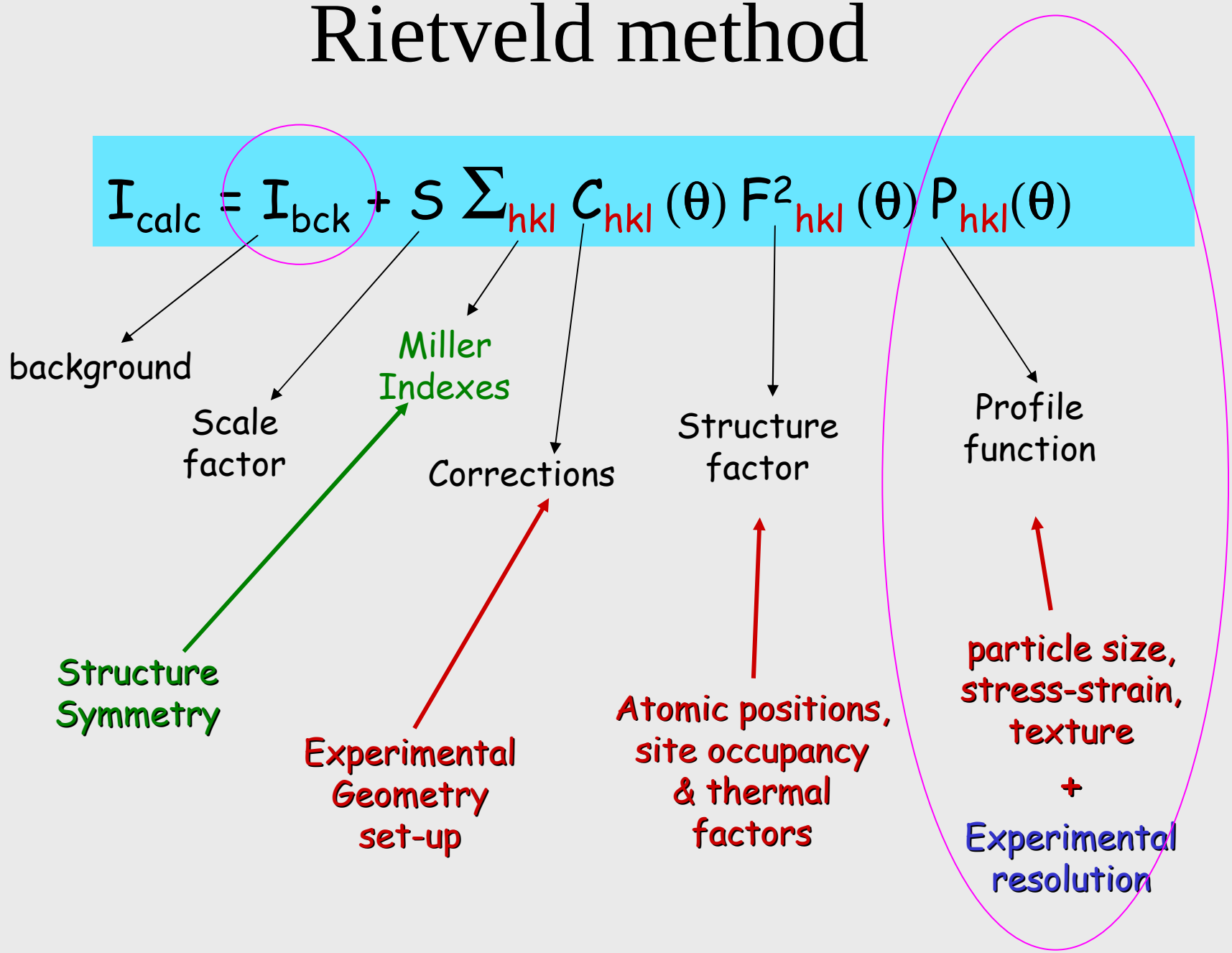
experimental geometry



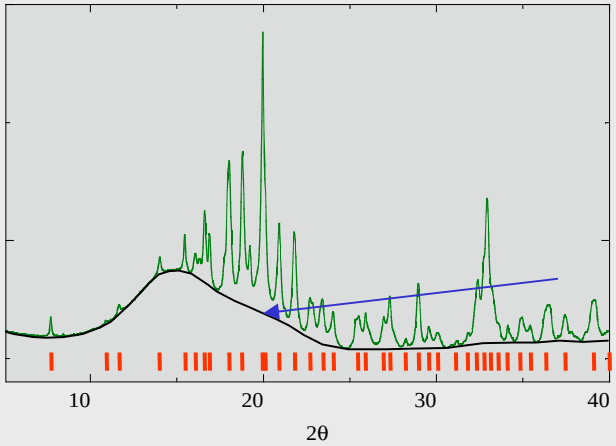

PowderCell 2.4
File Structure Select Options Diffraction Refinem
Experiment
Phase options
HKL - List
Size and strain
Export data
Export
Print
Copy to clipboard
Load powder pattern
Remove powder pattern
Save imported pattern
Diffraction OFF
..... XRPD tutorial/dati
y2o3.x_y
Data and model patterns are reasonably similar,
our model/hypothesis seems correct,
now we can derive **quantitative crystallographic information** refining the XRPD patterns!
powder pattern
Rp= 96.90 Rwp= 102.40 Rexp= 2.13
-icsd_Y203
-y2o3.x_y
49484
24742
0
10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120
211 220 312 400 411 402 232 422 431 440 521 433 442 523 602 514 622 631 444 543 640 633 732 611 635 631 725 633 761 651 932 941 10.11 934 925 617 961 945 10.51 980 992 11.32 10.60 10.62
powder pattern
Rp= 96.90 Rwp= 102.40 Rexp= 2.13
-icsd_Y203
-y2o3.x_y
26539
13269
0
45 50 55 60 65
1013x682

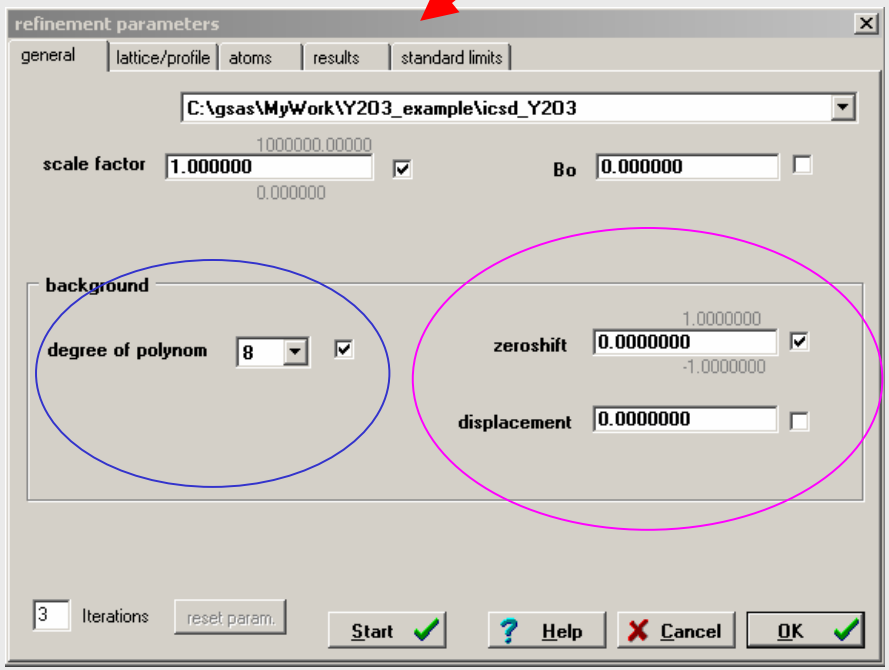
Rietveld method

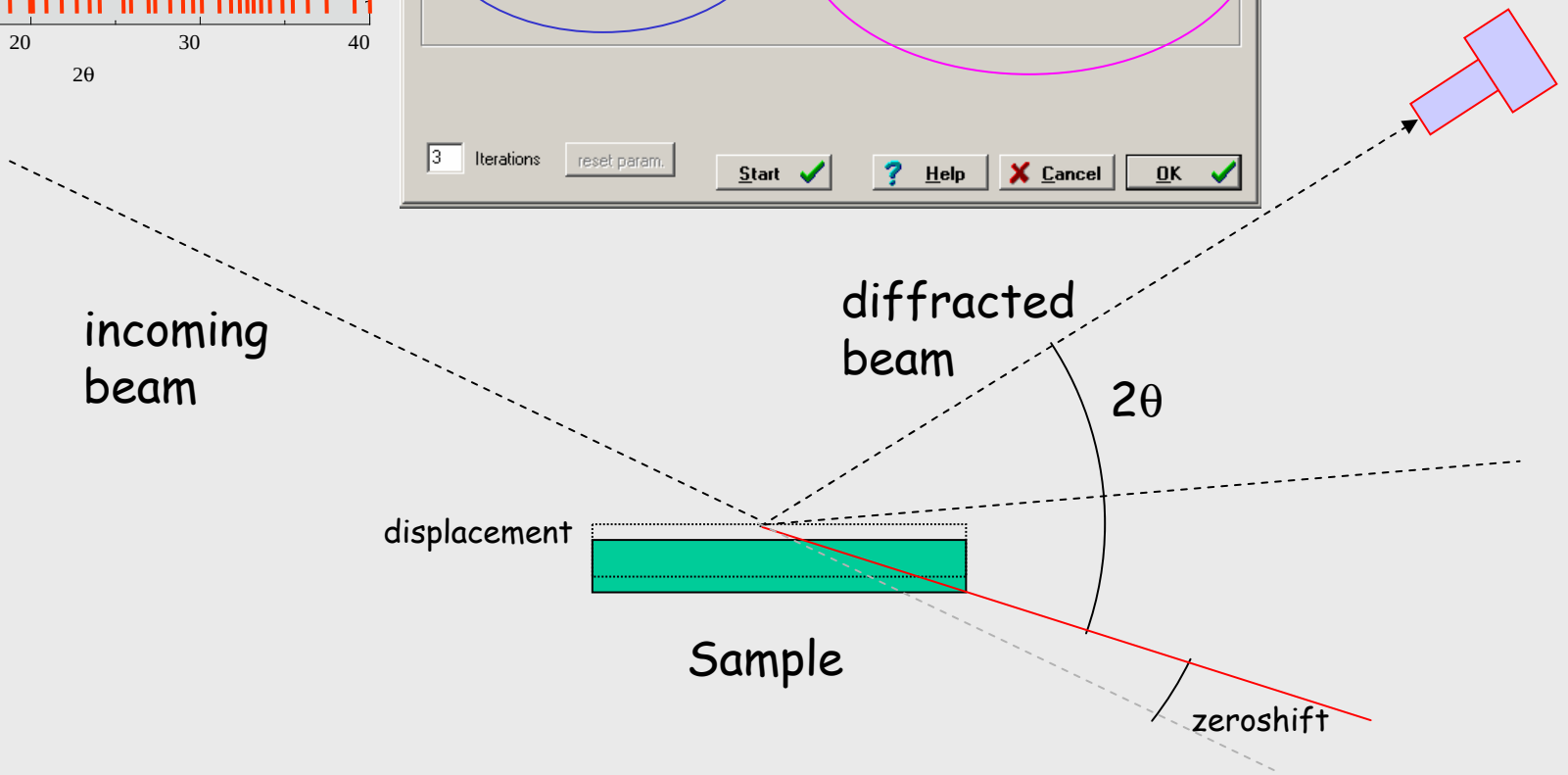
$$I_{calc} = I_{bck} + S \sum_{hkl} C_{hkl}(\theta) F^2_{hkl}(\theta) P_{hkl}(\theta)$$



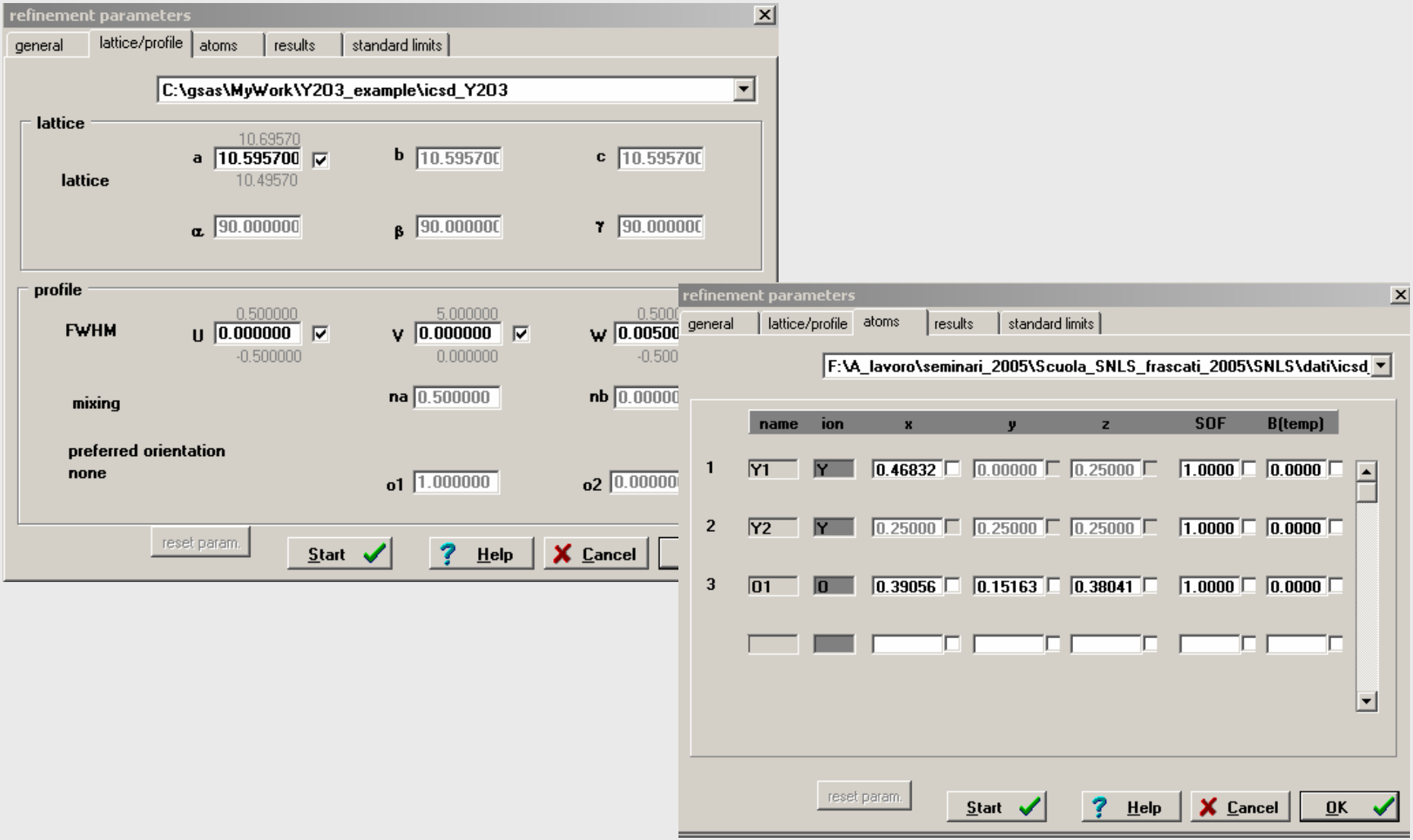

PowderCell 2.4
File Structure Select Options Diffraction Refinement Windows Special Help


10
20
30
40
2θ


refinement parameters
general
lattice/profile
atoms
results
standard limits
C:\gsas\MyWork\Y2O3_example\icsd_Y2O3
scale factor
1.000000
1000000.00000
0.000000
Bo
0.000000
background
degree of polynom
8
zeroshift
0.0000000
1.0000000
-1.0000000
displacement
0.0000000
3
Iterations
reset param.
Start
Help
Cancel
OK


incoming beam
diffracted beam
2θ
displacement
Sample
zeroshift

$$I_{calc} = I_{bck} + S \sum_{hkl} C_{hkl}(\theta) F^2_{hkl}(\theta) P_{hkl}(\theta)$$



PowderCell 2.4

File Structure Select Options Diffraction Refinement Windows Special Help

refinement parameters

general | lattice/profile | atoms | results | standard limits

F:\A_lavoro\seminari_2005\Scuola_SNLS_frascati_2005\SNLS\dati\icsd

parameter		old	new
icsd_86815			
scaling	:	1.0000	1.2000
lattice	a :	10.5957	10.5968
profile	U :	0.0000	-0.1870
PsVoigt2	V :	0.0000	0.0017
	W :	0.0050	0.1051
overall B	:	0.0000	-

global parameters

reset param. Start Help Cancel OK

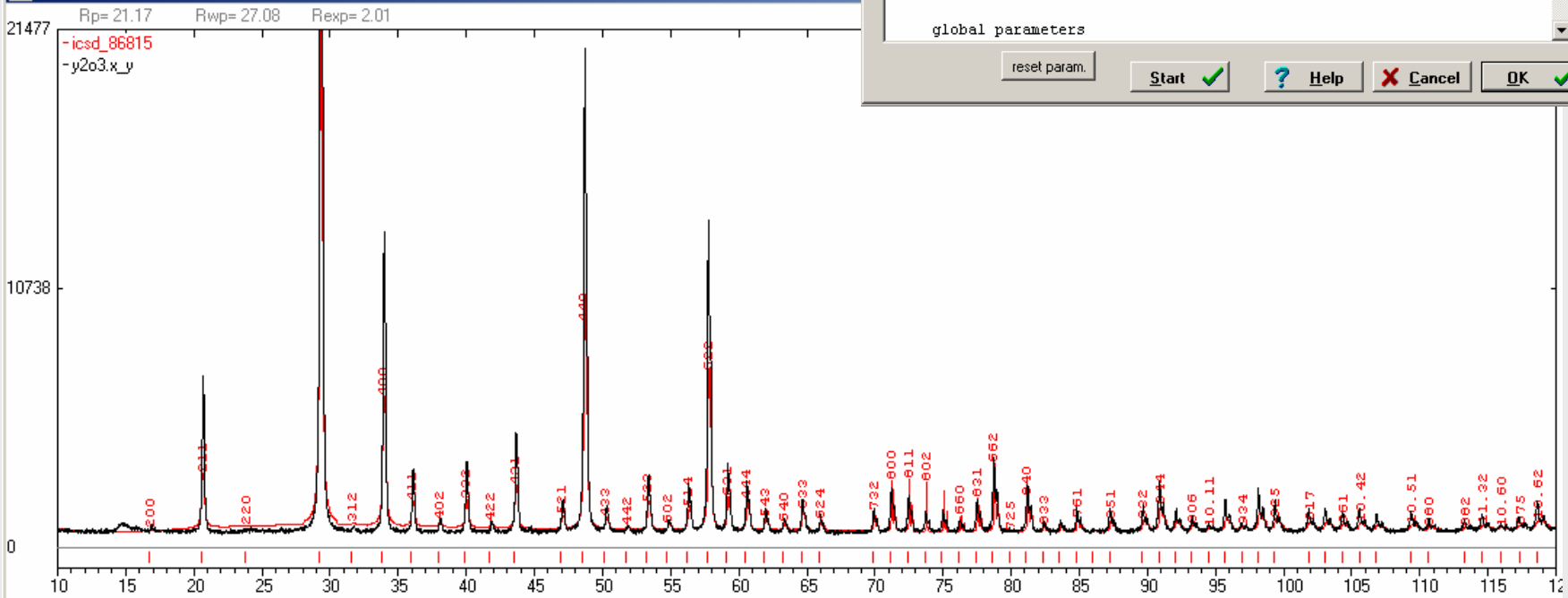
powder pattern

Rp= 21.17 Rwp= 27.08 Rexp= 2.01

21477 10738 0

-icsd_86815

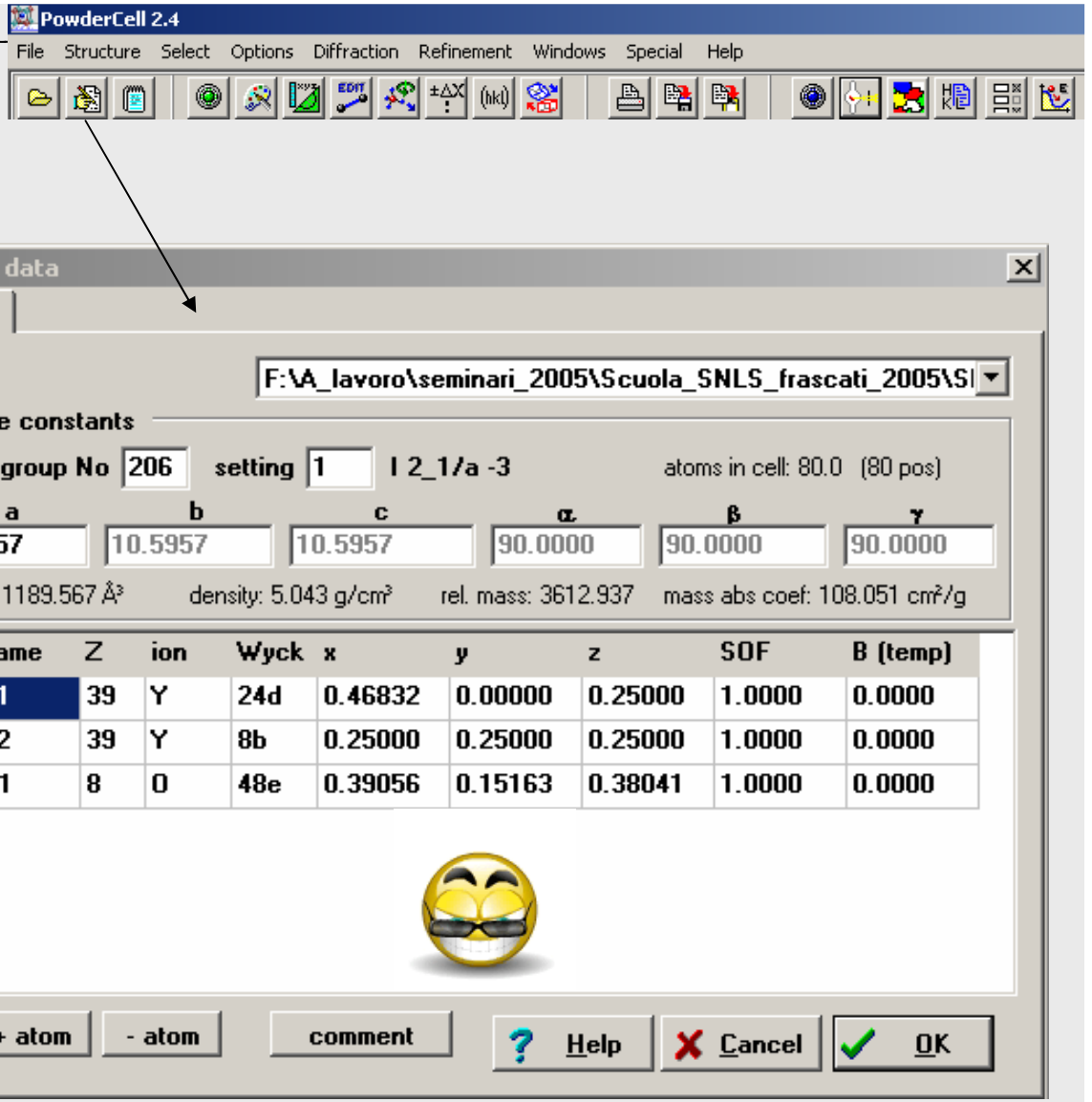
-y2o3.x_y



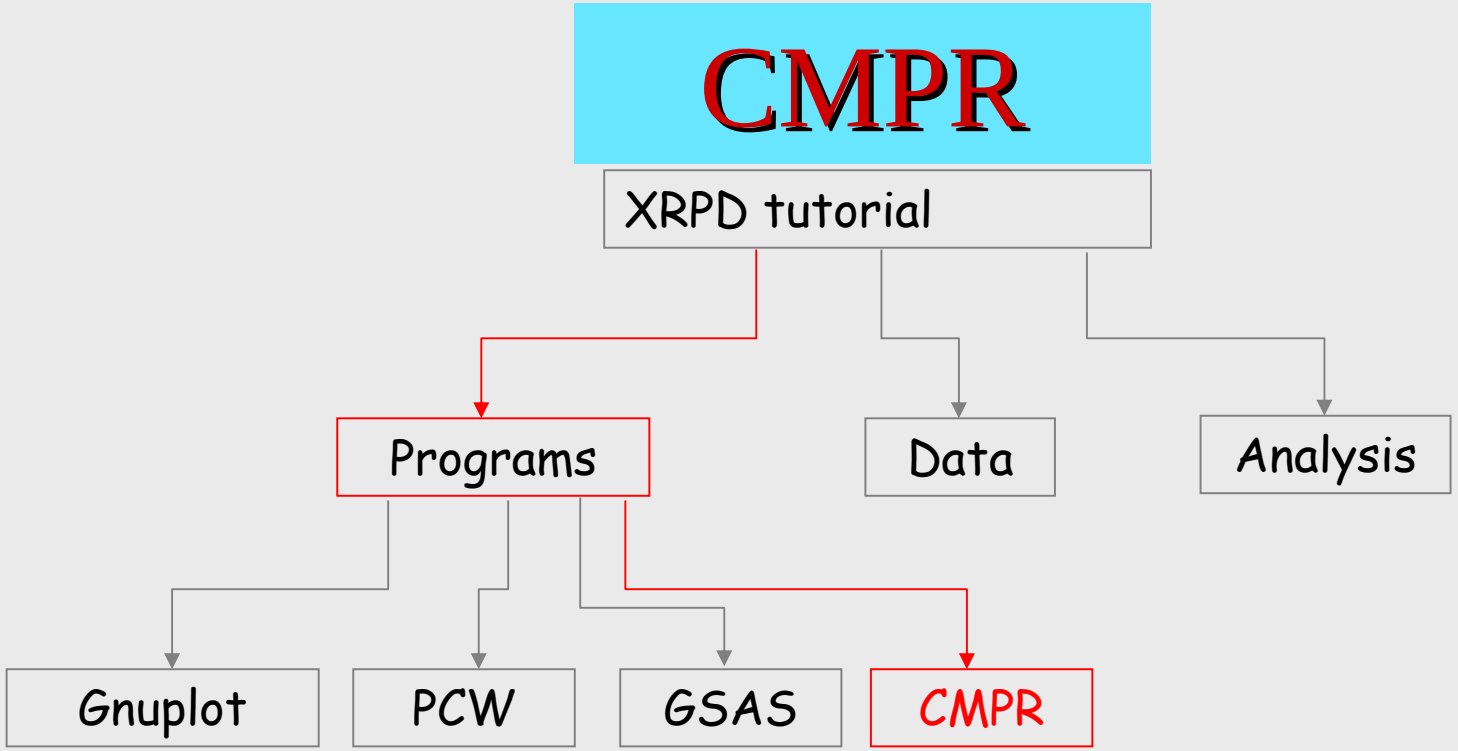
Refinement of y2o3.x_y 12/10/2005 12.58.11

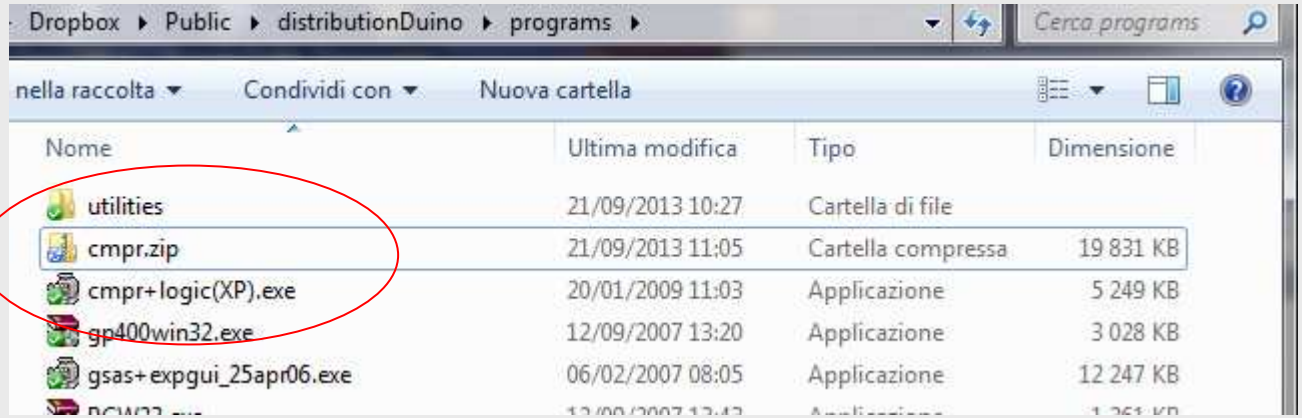
R-values Rp=18.08 Rwp=24.85 Rexp=2.01
2 iterations of 6

parameter		old	new
icsd_86815			
scaling	:	1.2000	1.2000
lattice	a :	10.5968	10.6090
profile	U :	-0.1870	-0.4973
PsVoigt2	V :	0.0020	0.7986
	W :	0.1050	-0.2679
overall B	:	0.0000	-
global parameters			
zero shift	:	-0.1475	-0.1997
displacement	:	0.0000	-
backgr. polynom	:	13	13
coeff.	a0 :	4515.8630	6590.1070
	a1 :	-841.4	-1060
	a2 :	68.74	75.86
	a3 :	-2.669	-2.745
	a4 :	0.05247	0.0522
	a5 :	-0.0004457	-0.0004386
	a6 :	-6.29E-7	-5.966E-7
	a7 :	3.161E-8	3.121E-8
	a8 :	-3.797E-12	-8.06E-12
	a9 :	-1.953E-12	-1.947E-12
	a10 :	2.97E-16	6.361E-1
	a11 :	1.251E-16	1.261E-1
	a12 :	-6.315E-19	-6.571E-1
	a13 :	8.173E-22	8.861E-2



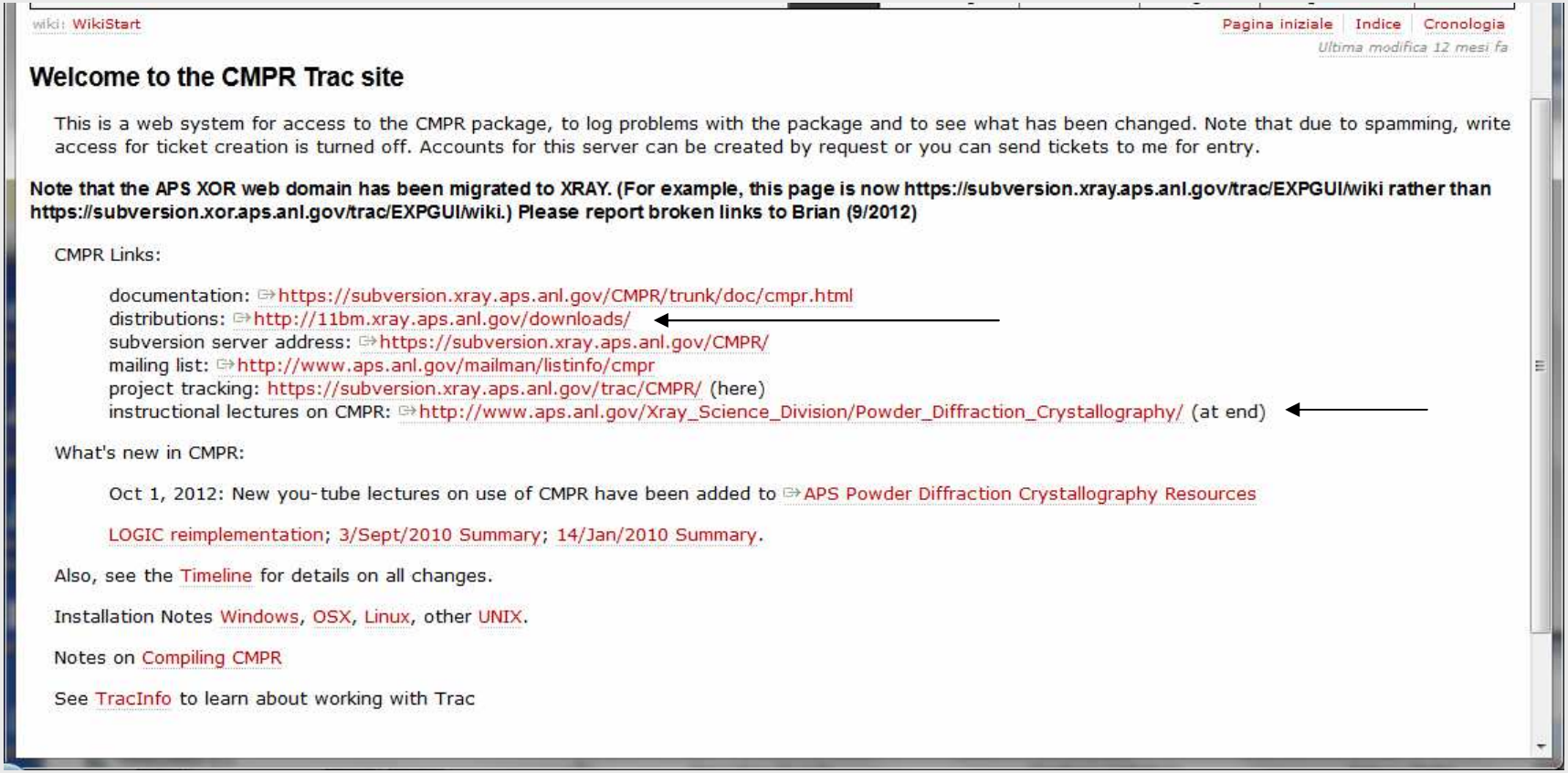
Getting some other information from your data





Informations and tutorials for CMPR

Repository: https://subversion.xor.aps.anl.gov/trac/CMPR



wiki: WikiStart

Pagina iniziale | Indice | Cronologia

Ultima modifica 12 mesi fa

Welcome to the CMPR Trac site

This is a web system for access to the CMPR package, to log problems with the package and to see what has been changed. Note that due to spamming, write access for ticket creation is turned off. Accounts for this server can be created by request or you can send tickets to me for entry.

Note that the APS XOR web domain has been migrated to XRAY. (For example, this page is now https://subversion.xray.aps.anl.gov/trac/EXPGUI/wiki rather than https://subversion.xor.aps.anl.gov/trac/EXPGUI/wiki.) Please report broken links to Brian (9/2012)

CMPR Links:

documentation: https://subversion.xray.aps.anl.gov/CMPR/trunk/doc/cmpr.html
distributions: http://11bm.xray.aps.anl.gov/downloads/
subversion server address: https://subversion.xray.aps.anl.gov/CMPR/
mailing list: http://www.aps.anl.gov/mailman/listinfo/cmpr
project tracking: https://subversion.xray.aps.anl.gov/trac/CMPR/ (here)
instructional lectures on CMPR: http://www.aps.anl.gov/Xray_Science_Division/Powder_Diffraction_Crystallography/ (at end)

What's new in CMPR:

Oct 1, 2012: New you-tube lectures on use of CMPR have been added to APS Powder Diffraction Crystallography Resources

LOGIC reimplementation; 3/Sept/2010 Summary; 14/Jan/2010 Summary.

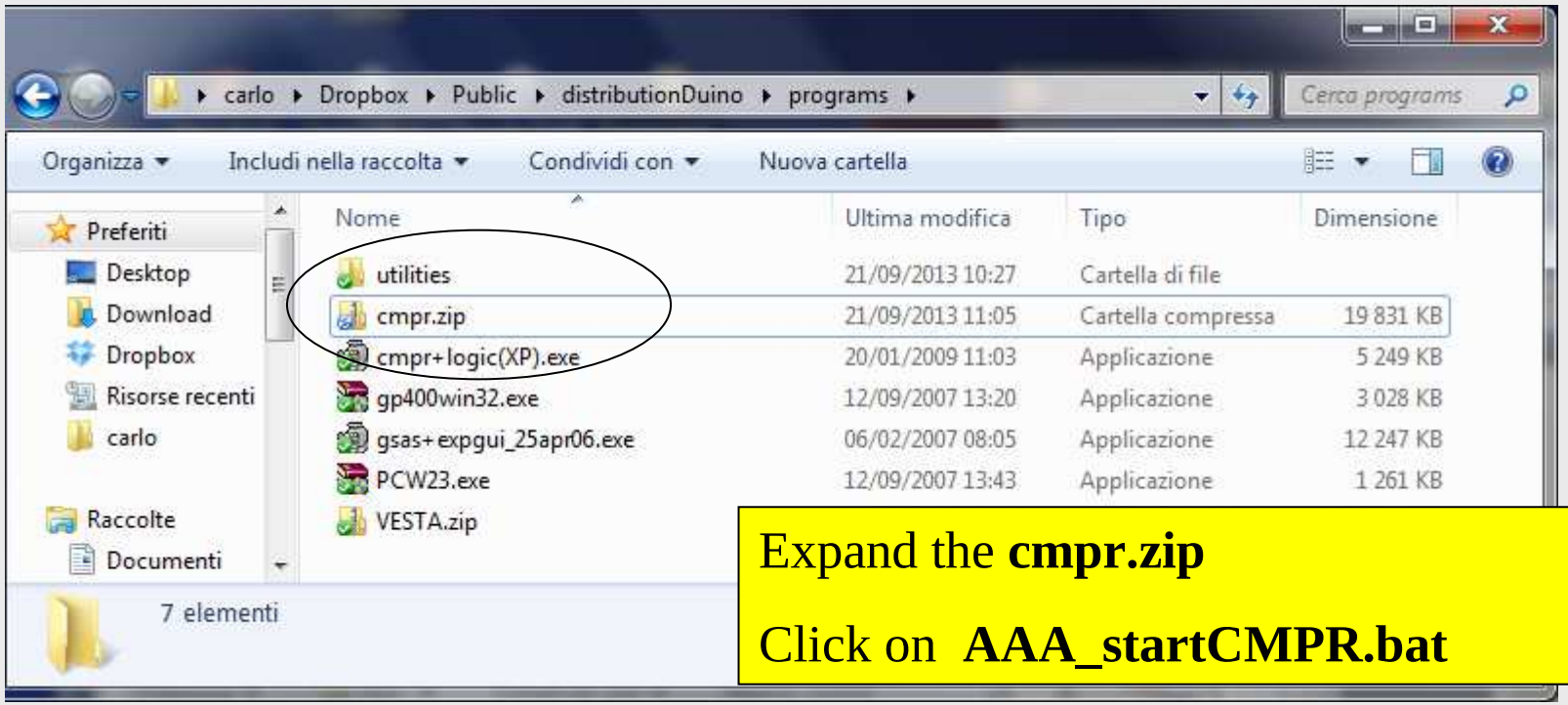
Also, see the Timeline for details on all changes.

Installation Notes Windows, OSX, Linux, other UNIX.

Notes on Compiling CMPR

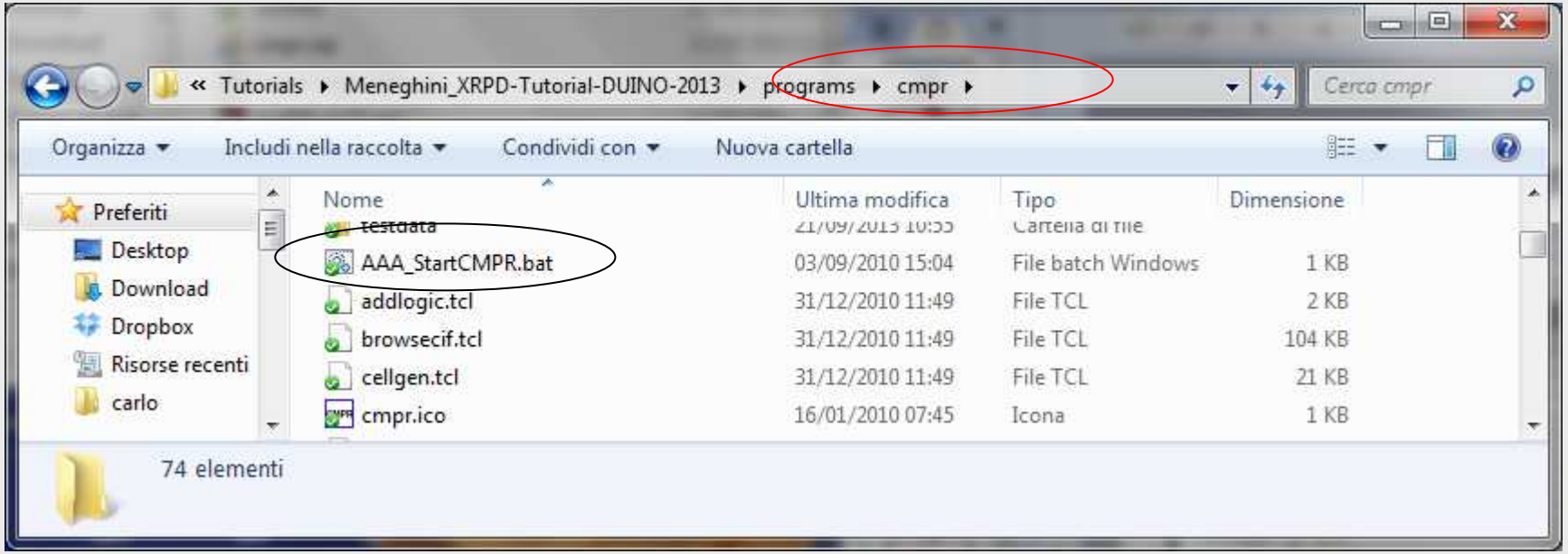
See TracInfo to learn about working with Trac

Installing CMP on W7 maybe difficult, use the cmpr.zip file for start

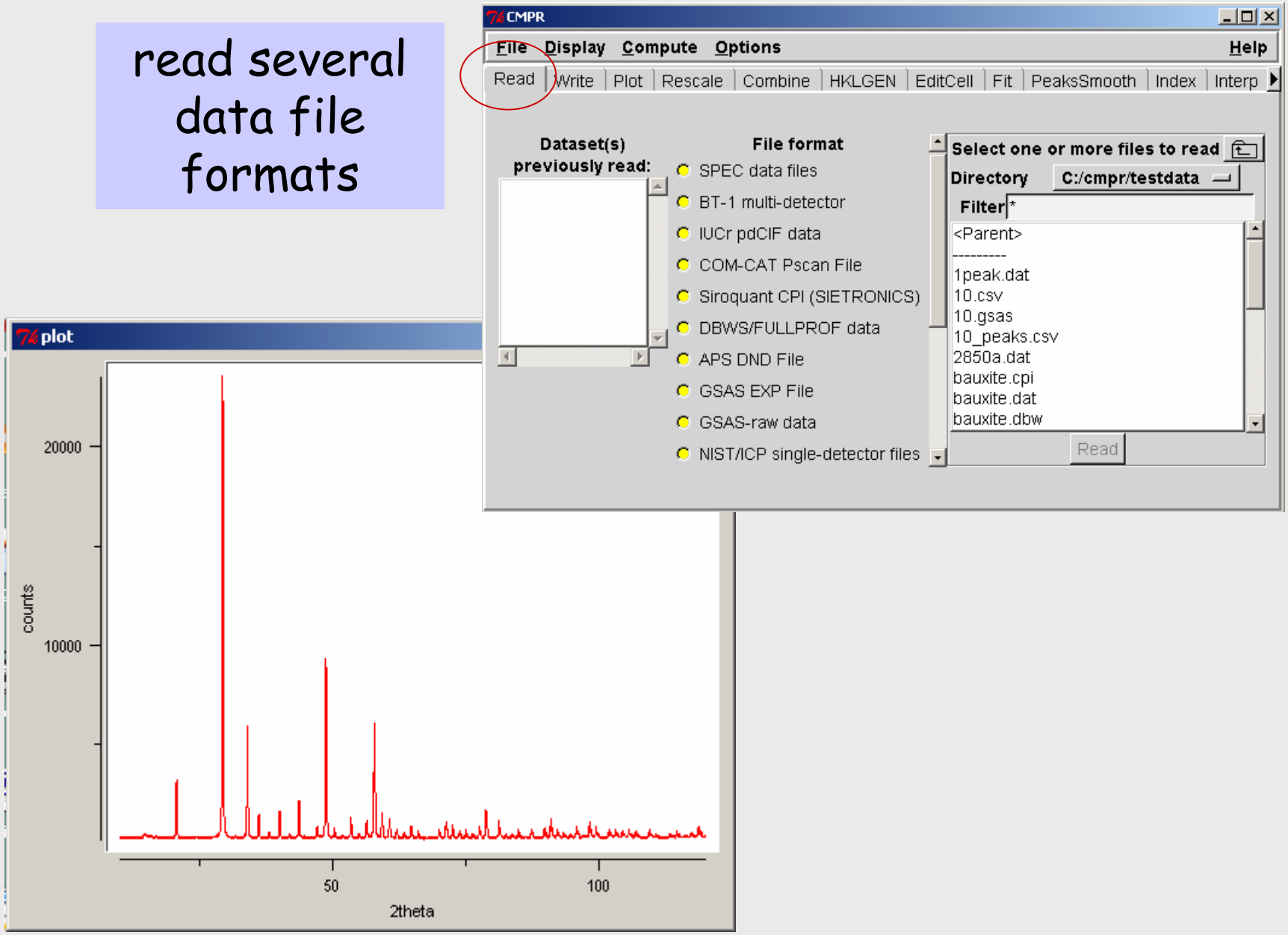


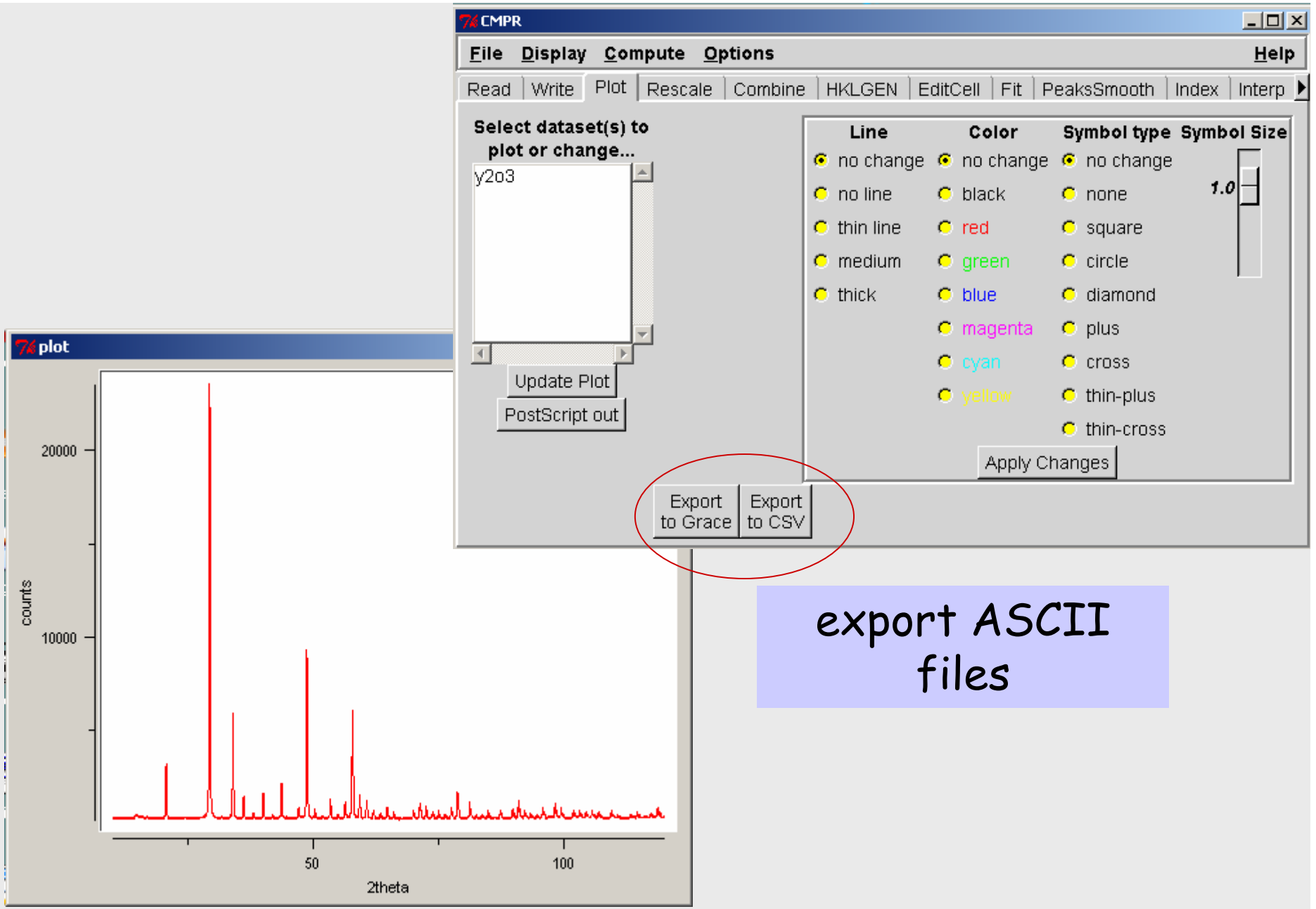
Expand the **cmpr.zip**

Click on **AAA_startCMPR.bat**



read several data file formats





export ASCII files

select the data file you want to playing with

CMPR

File Display Compute Options Help

Read | Write | Plot | Rescale | Combine | HKLGEN | EditCell | Fit | PeaksSmooth | Index | Interp

Select dataset(s) to rescale

y2o3

X-axis units

- no change or reset
- d-space
- Q
- 2Theta @ A

Y-axis units

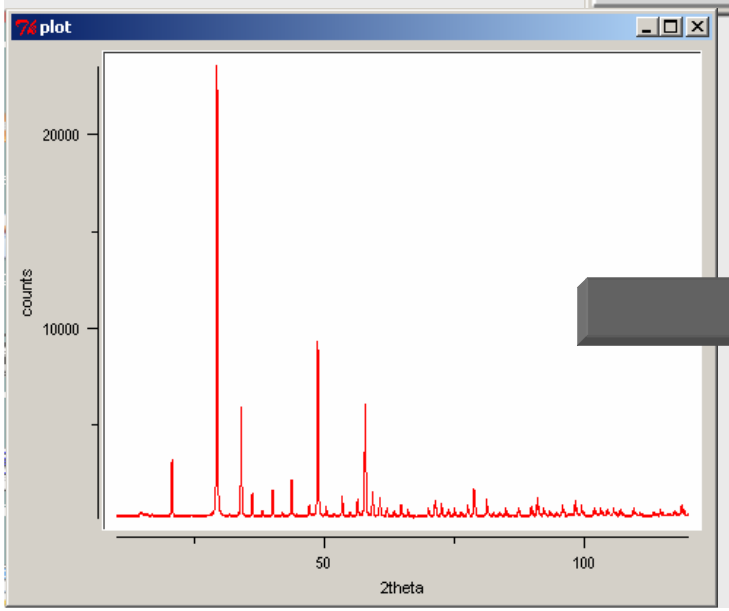
- Linear or reset
- Sqrt(I)
- Log(I)
- I/sig(I)
- I2/sig(I)2

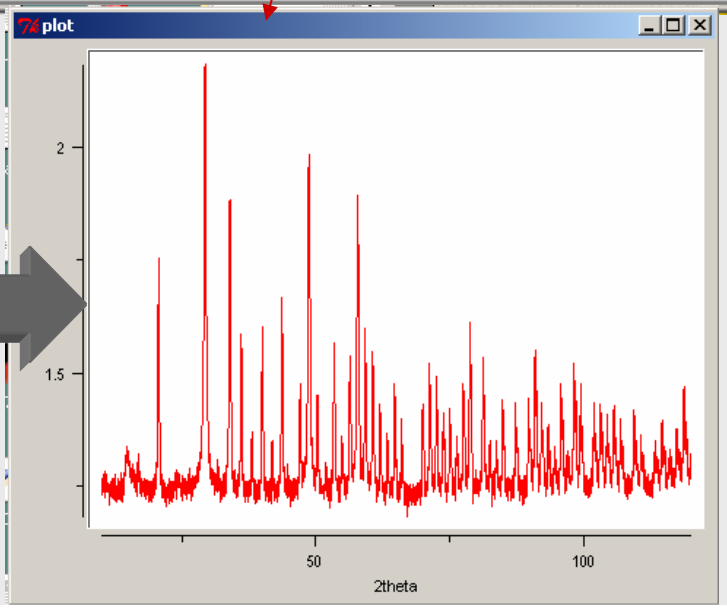
Scaling

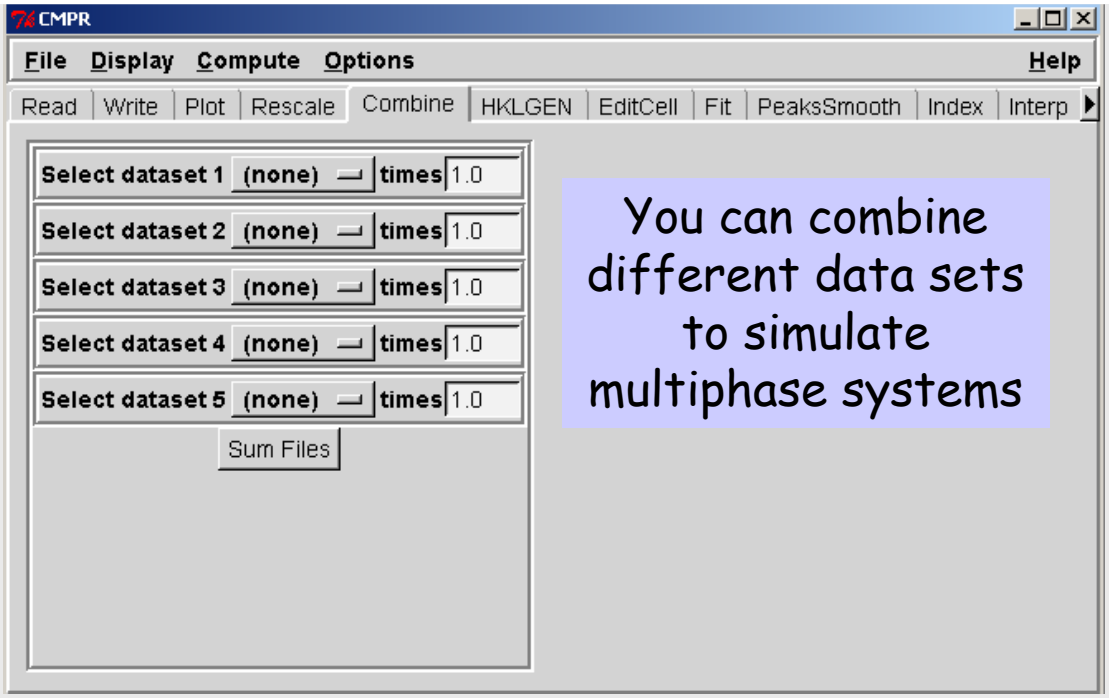
X offset	0
X multiplier	1
Y offset	0
Y multiplier	1

Reset previously applied scaling

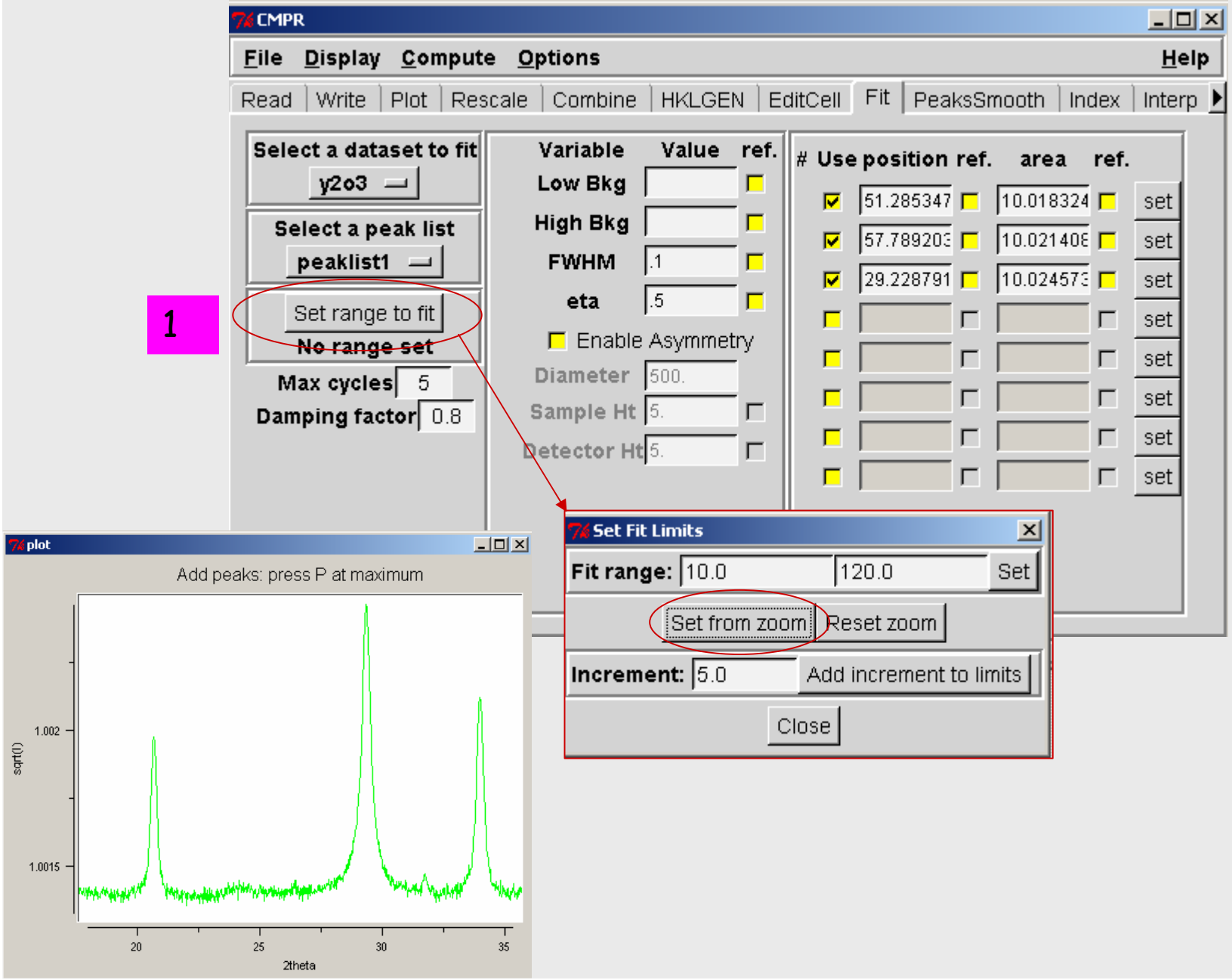
Rescale

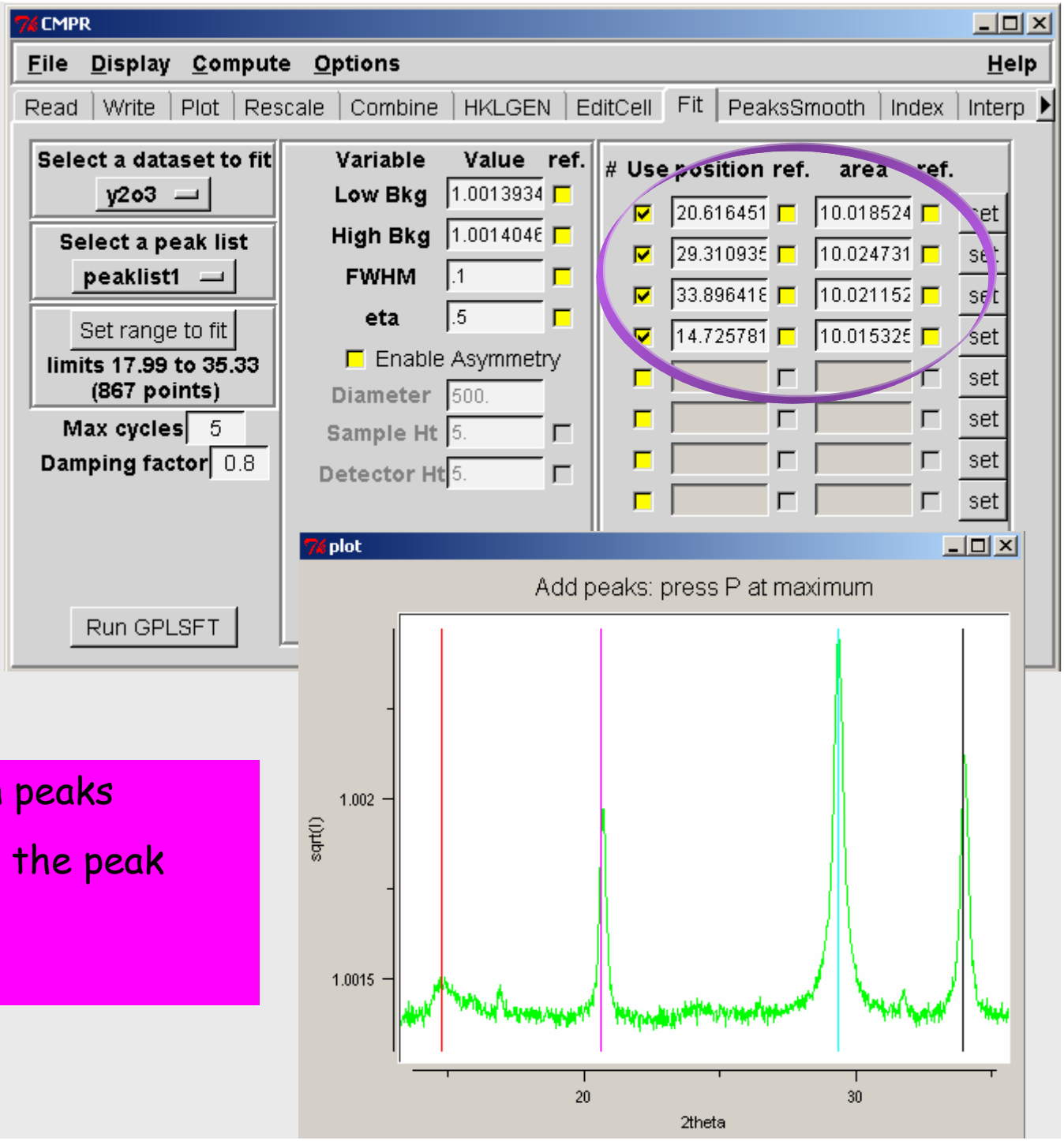





CMPR
File Display Compute Options Help
Read Write Plot Rescale Combine HKLGEN EditCell Fit PeaksSmooth Index Interp
Select dataset 1 (none) times 1.0
Select dataset 2 (none) times 1.0
Select dataset 3 (none) times 1.0
Select dataset 4 (none) times 1.0
Select dataset 5 (none) times 1.0
Sum Files
You can combine different data sets to simulate multiphase systems

(multi-) peak fitting routines

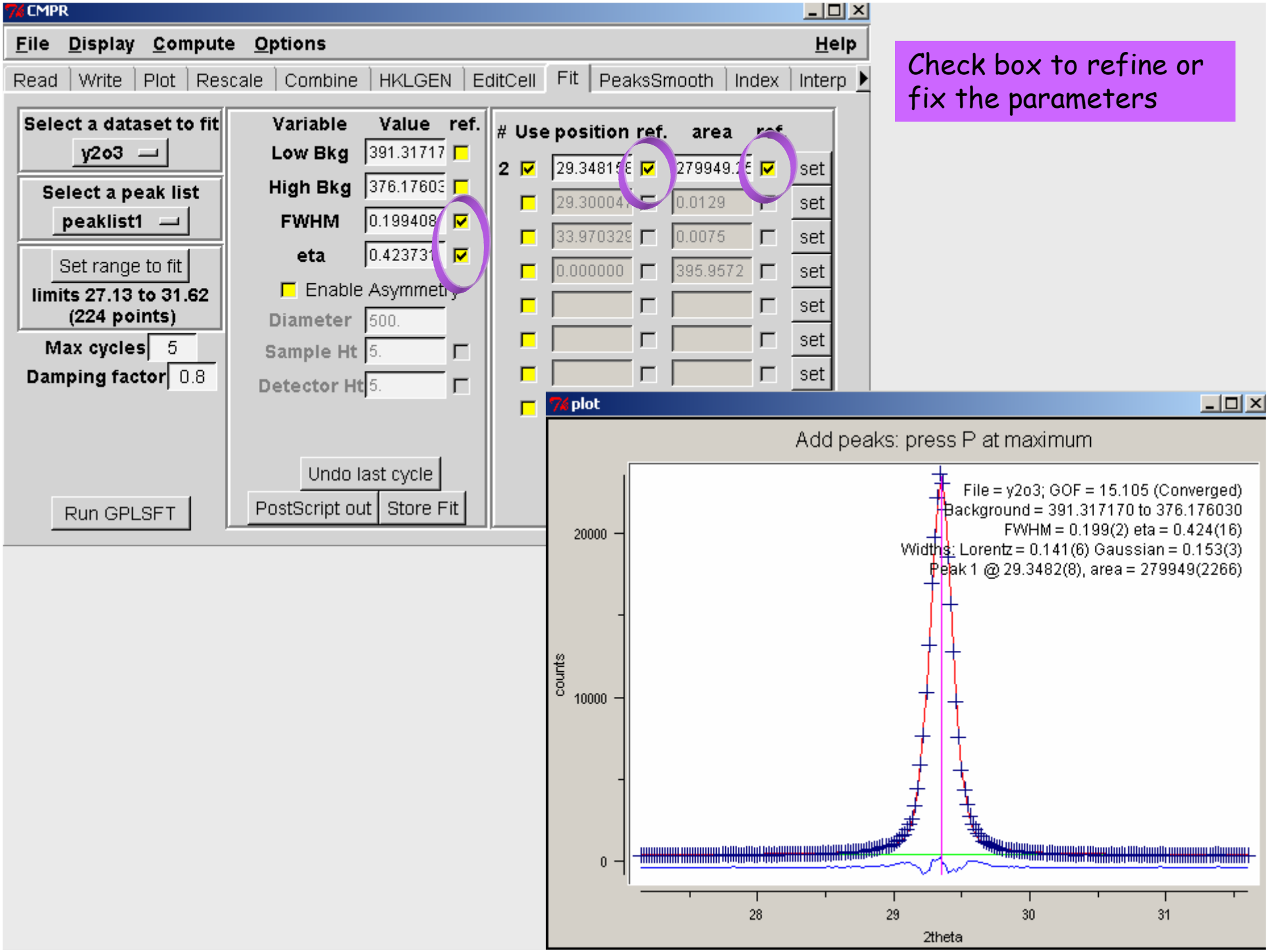


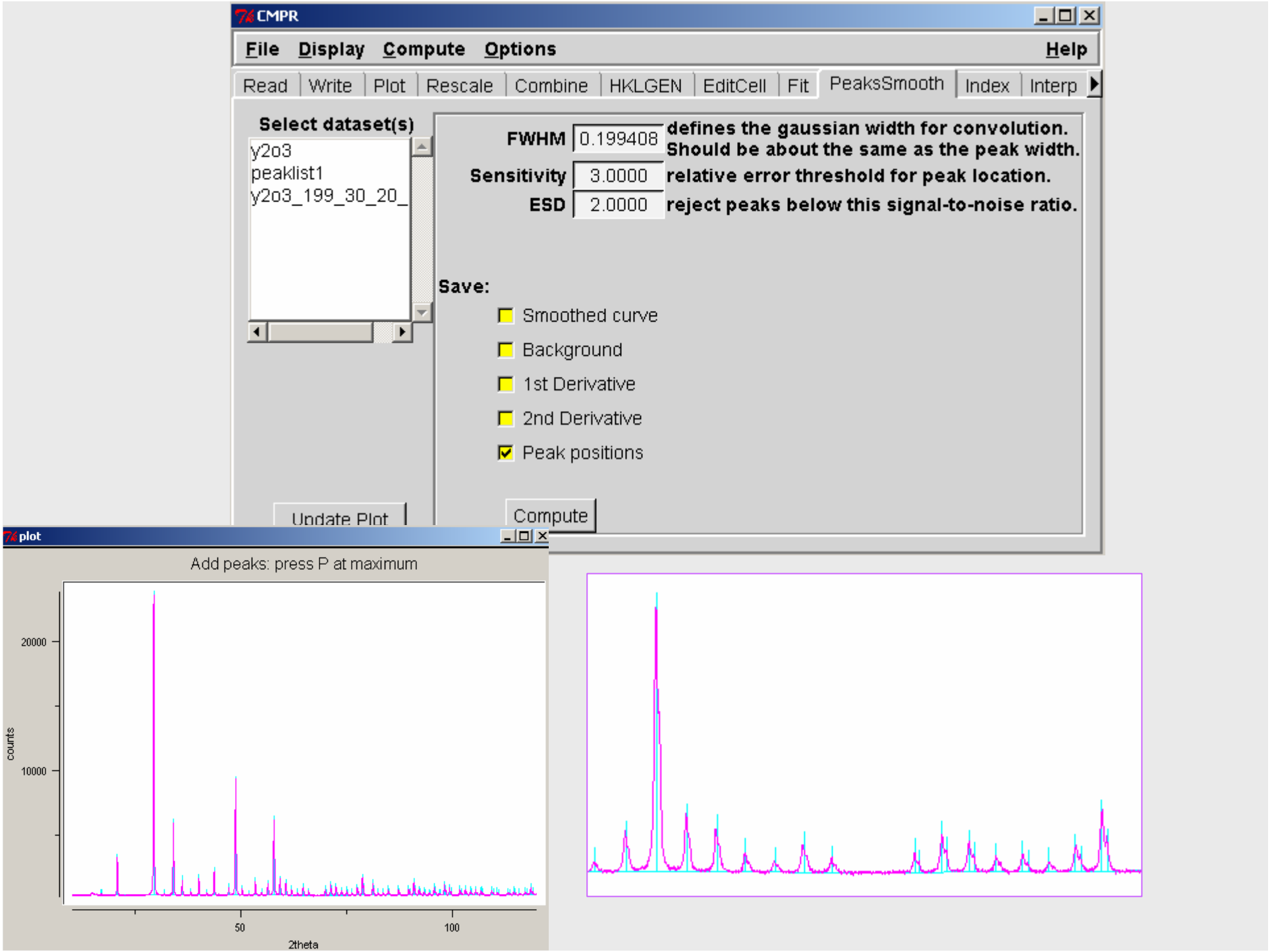


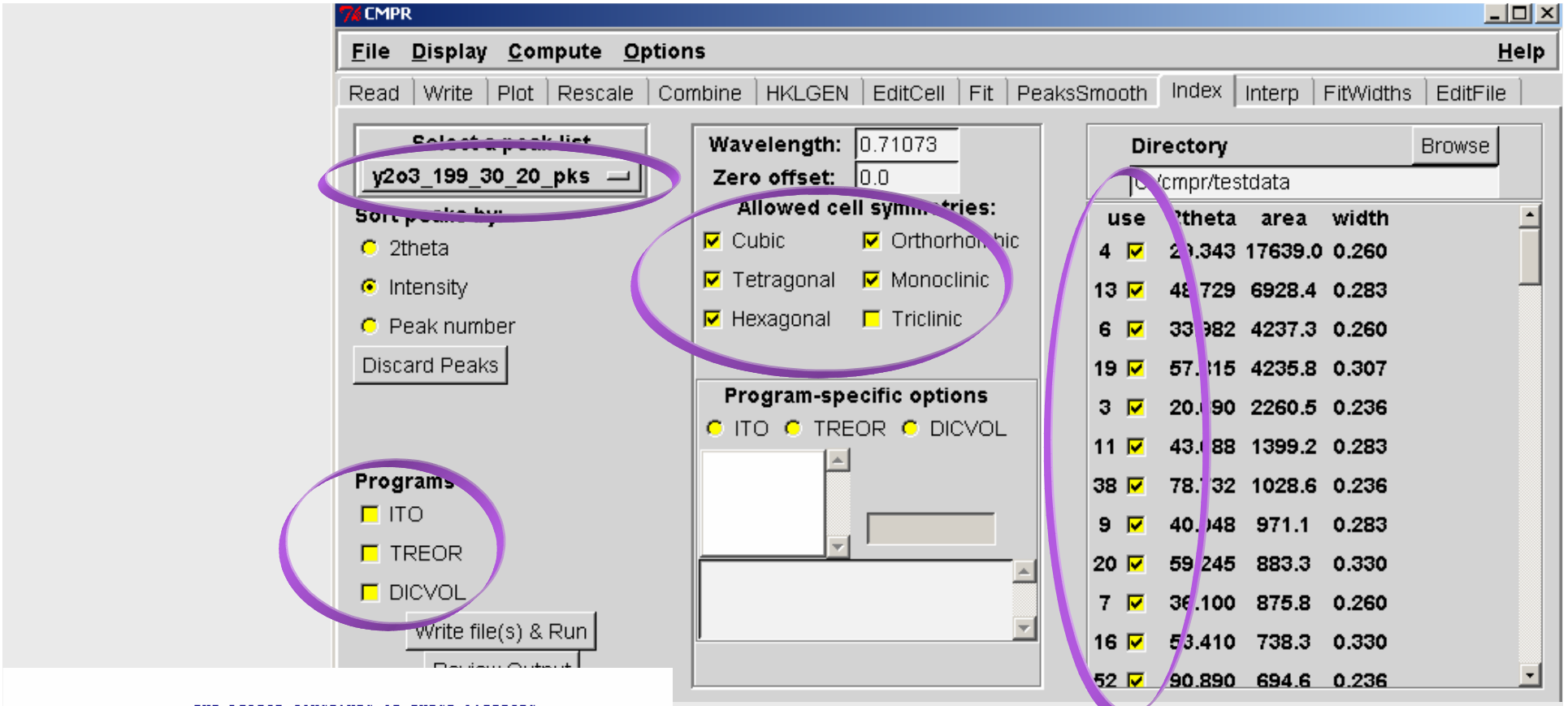
To Select diffraction peaks

1) Move the mouse on the peak maximum and

2) pres the p key


CMPR
File Display Compute Options Help
Read Write Plot Rescale Combine HKLGEN EditCell Fit PeaksSmooth Index Interp
Select a dataset to fit
y2o3
Select a peak list
peaklist1
Set range to fit
limits 27.13 to 31.62
(224 points)
Max cycles 5
Damping factor 0.8
Run GPLSFT
Variable Value ref.
Low Bkg 391.31717
High Bkg 376.17603
FWHM 0.199408
eta 0.423731
Enable Asymmetry
Diameter 500.
Sample Ht 5.
Detector Ht 5.
Undo last cycle
PostScript out Store Fit
Use position ref. area ref.
2 29.34819 279949.1
29.30004 0.0129
33.970329 0.0075
0.000000 395.9572
set
Check box to refine or fix the parameters
plot
Add peaks: press P at maximum
File = y2o3; GOF = 15.105 (Converged)
Background = 391.317170 to 376.176030
FWHM = 0.199(2) eta = 0.424(16)
Widths: Lorentz = 0.141(6) Gaussian = 0.153(3)
Peak 1 @ 29.3482(8), area = 279949(2266)
counts
20000
10000
0
28
29
30
31
2theta


CMPR
File Display Compute Options Help
Read Write Plot Rescale Combine HKLGEN EditCell Fit PeaksSmooth Index Interp
Select dataset(s)
y2o3
peaklist1
y2o3_199_30_20_
FWHM 0.199408 defines the gaussian width for convolution. Should be about the same as the peak width.
Sensitivity 3.0000 relative error threshold for peak location.
ESD 2.0000 reject peaks below this signal-to-noise ratio.
Save:
Smoothed curve
Background
1st Derivative
2nd Derivative
Peak positions
Update Plot
Compute
plot
Add peaks: press P at maximum
20000
10000
counts
50
100
2theta



THE DIRECT CONSTANTS OF THESE LATTICES

A	B	C	ALFA	BETA	GAMMA	VOLUME
7.3428	10.3106	5.1124	98.8696	94.1817	86.1676	380.80

ITO: y2o3_199_30_20_pks

STUDY ALSO THE RESULTS IN THE OTHER OUTPUT FILES!!

```
**************************************************************************
*                                                                        *
* THIS PROGRAM GIVES A NUMBER OF SUGGESTIONS THAT MAY HELP YOU TO        *
* FIND THE UNIT CELL THAT EXPLAINS YOUR EXPERIMENTAL DATA. HOWEVER       *
*                                                                        *
*      WWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWW          *
*      W                                                     W          *
*      W  IT IS ENTIRELY THE USERS RESPONSIBILITY TO DECIDE  W          *
*      W  WHETHER ANY OF THE SUGGESTED UNIT CELLS IS THE     W          *
*      W   CORRECT CELL.                                     W          *
*      W                                                     W          *
*      WWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWWW          *
*                                                                        *
**************************************************************************

END OF INPUT,  NORMAL END OF PROGRAM
```

For advanced users:

Search for the possible symmetry using ITO, TREOR or DICVOL algorithms

GSAS

XRPD tutorial

Programs

Data

Analysis

Gnuplot

PCW

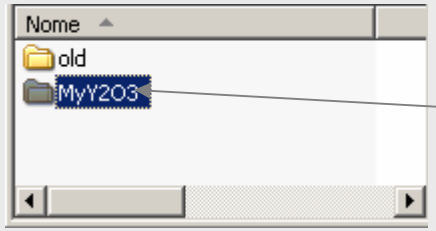
GSAS

UTIL

(XP)gsas+expgui.exe

Create a new directory in the path:

C:\gsas\MyWork



Copy into the new folder

Y2O3_PCW

Y2O3_GSAS

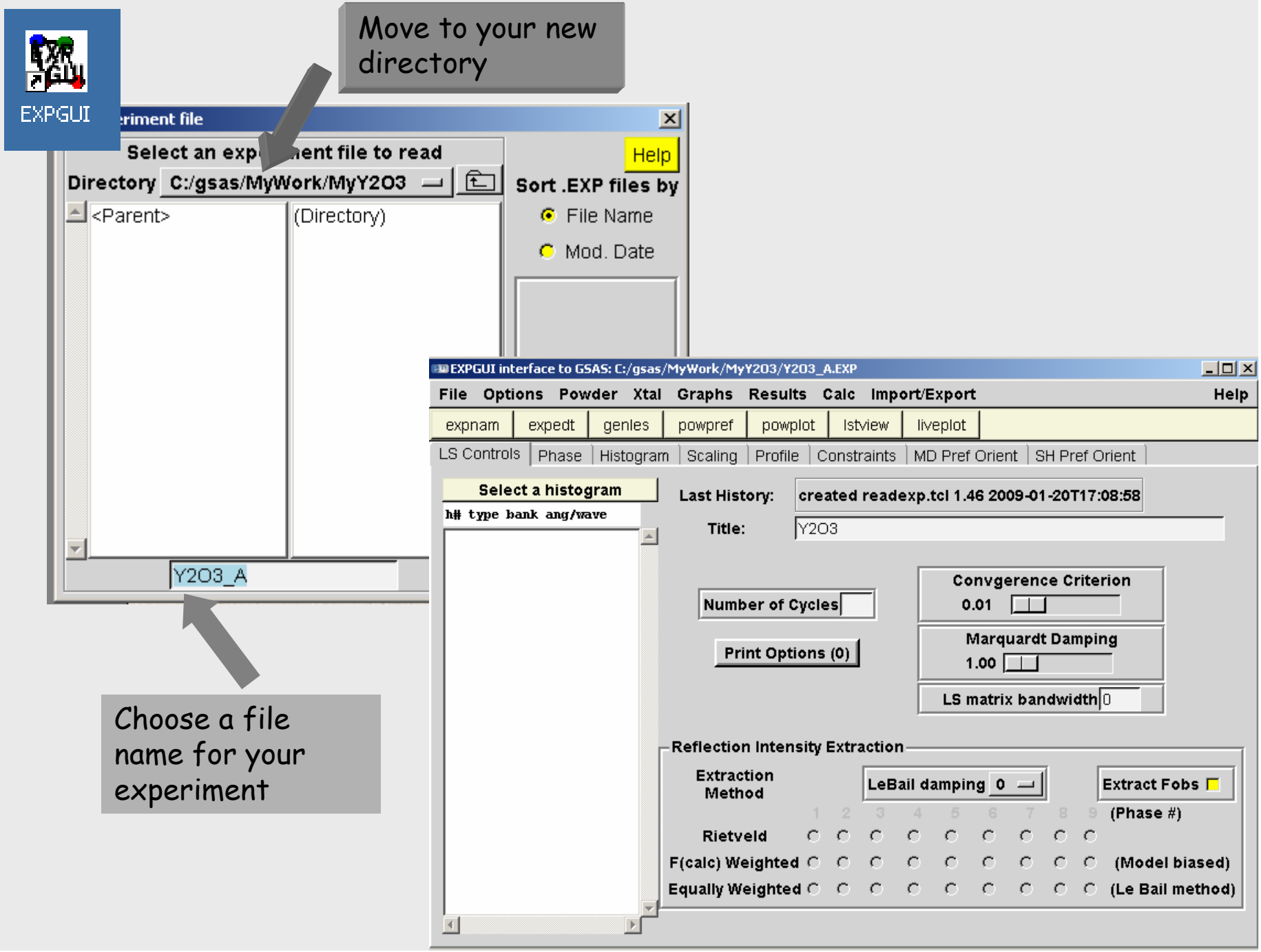
Other_XRD

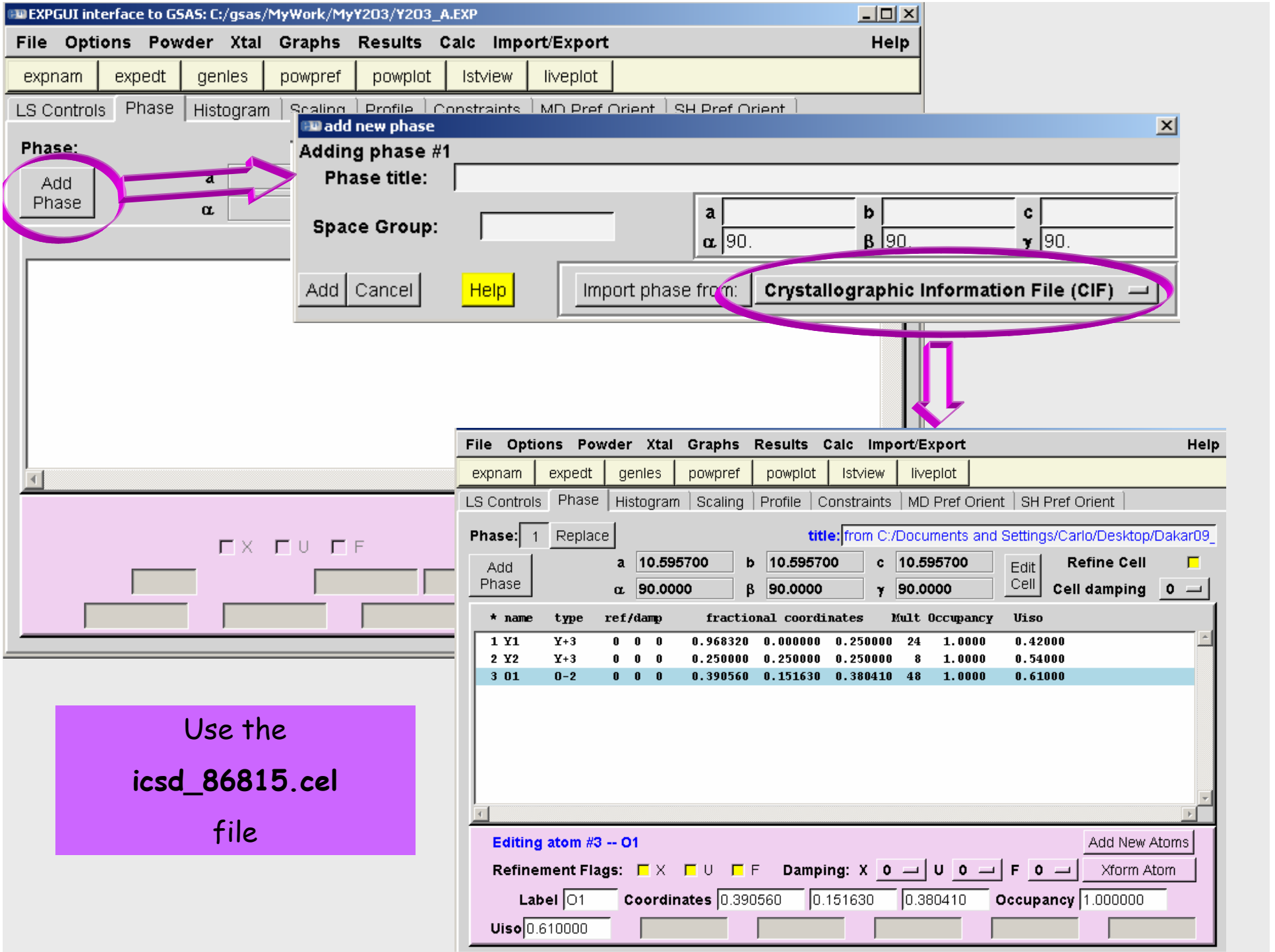
Au_GSAS

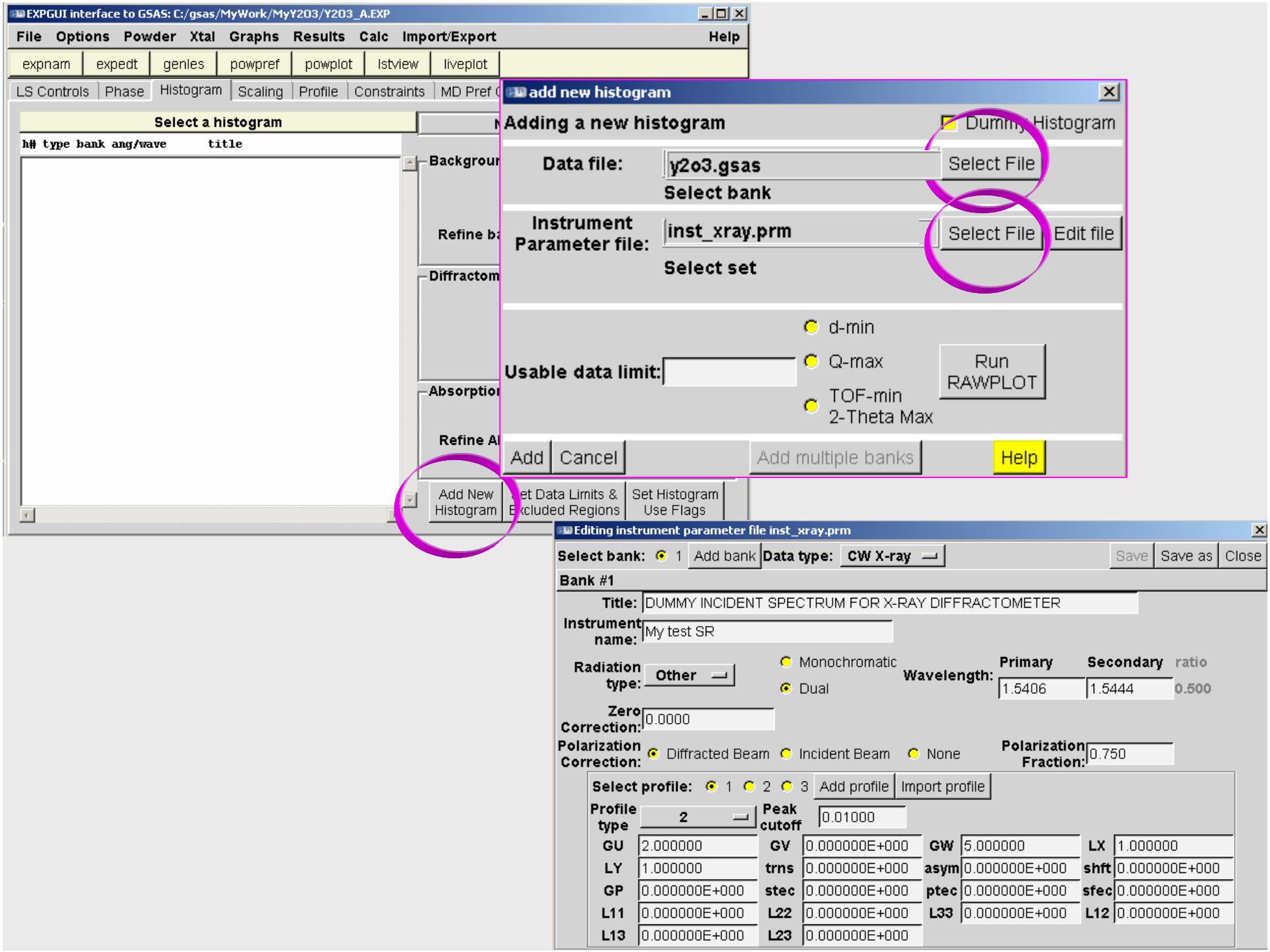
y2o3.gs,
inst_xry.prm

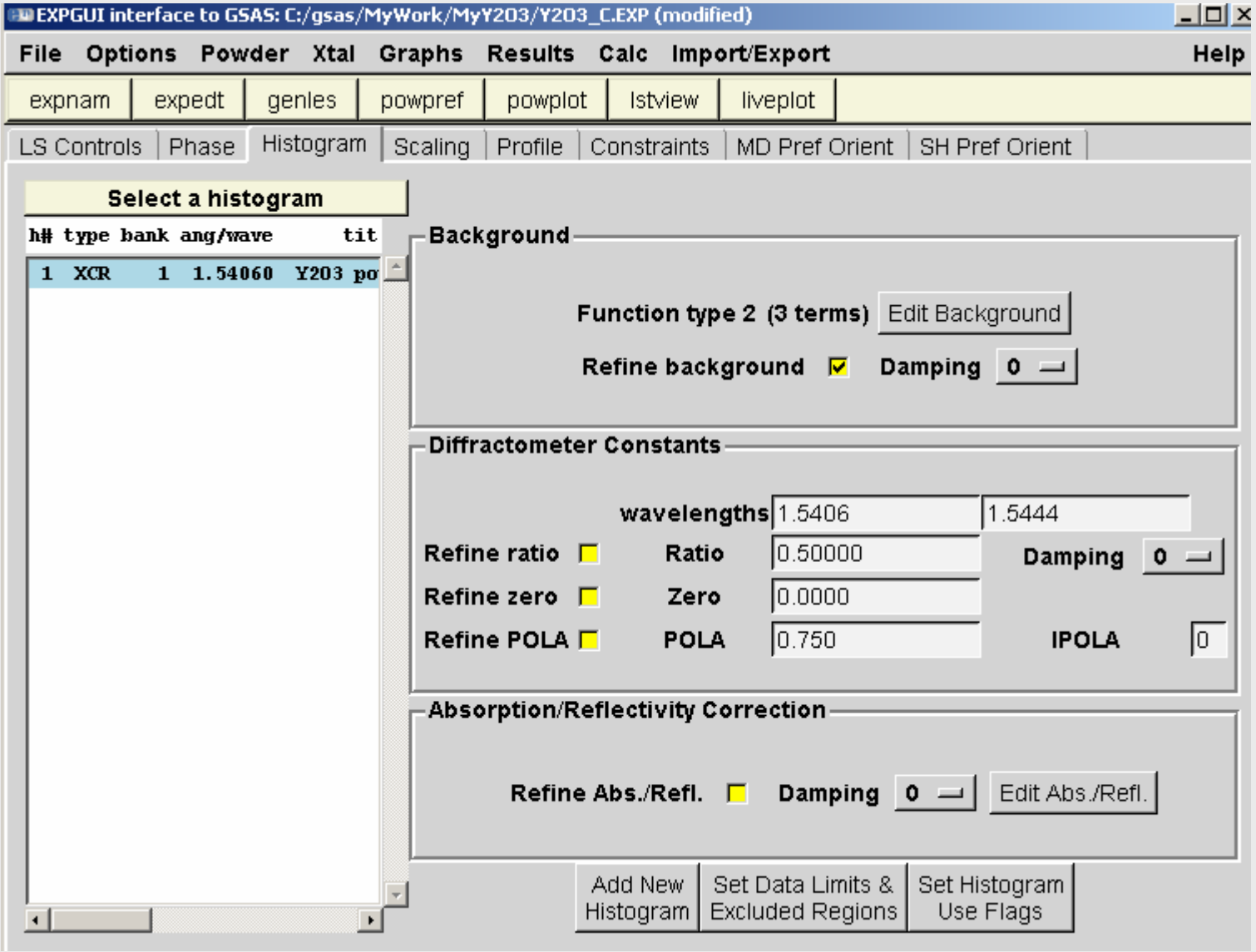
Obtaining GSAS

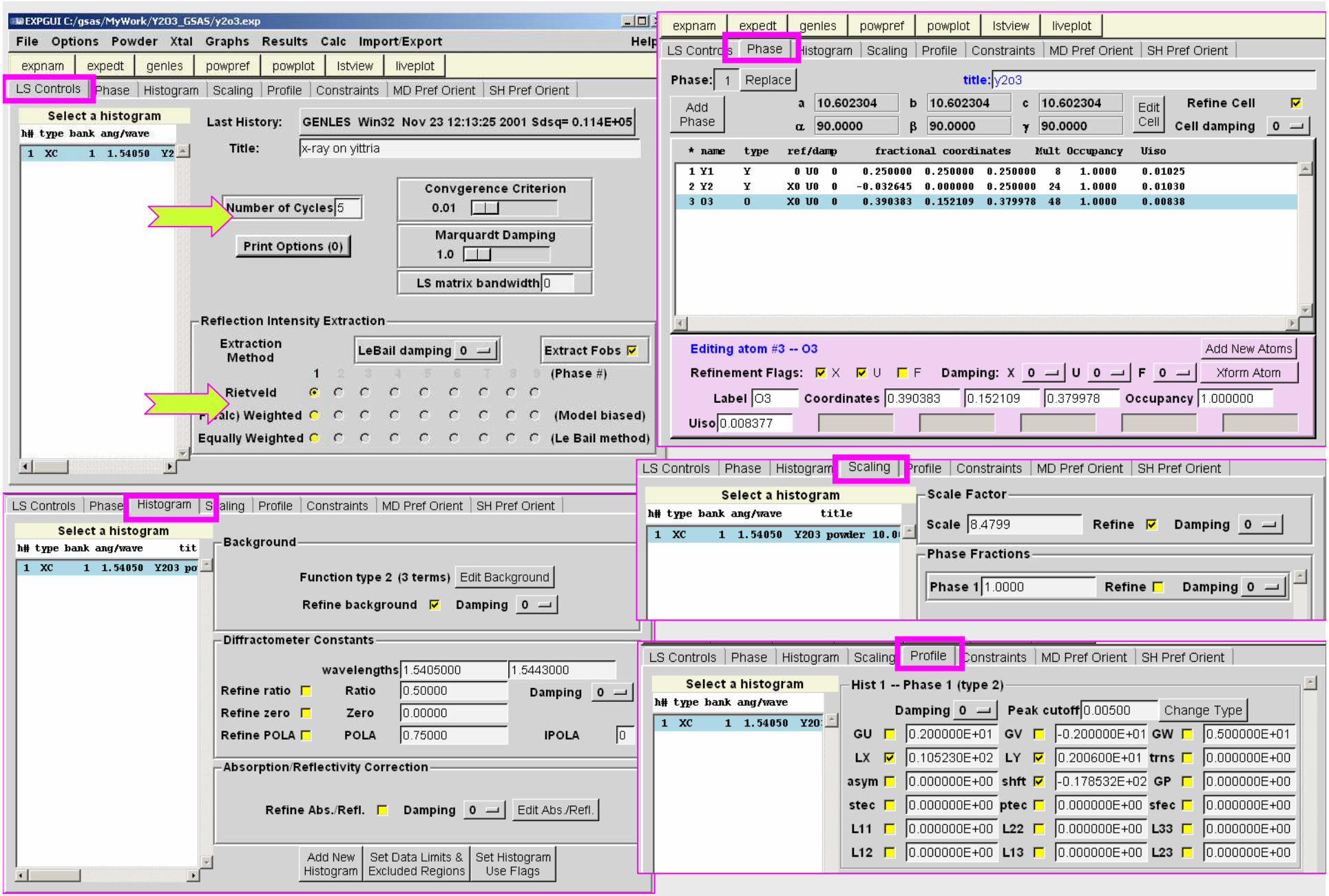
http://www.ccp14.ac.uk/solution/gsas/gsas_with_expgui_install.html


EXPGUI
Move to your new directory
Select an exp ent file to read
Directory C:/gsas/MyWork/MyY2O3
Help
Sort .EXP files by
File Name
Mod. Date
<Parent>
(Directory)
Y2O3_A
Choose a file name for your experiment
EXPGUI interface to GSAS: C:/gsas/MyWork/MyY2O3/Y2O3_A.EXP
File Options Powder Xtal Graphs Results Calc Import/Export Help
expnam expedt genles powpref powplot lstview liveplot
LS Controls Phase Histogram Scaling Profile Constraints MD Pref Orient SH Pref Orient
Select a histogram
h# type bank ang/wave
Last History: created readexp.tcl 1.46 2009-01-20T17:08:58
Title: Y2O3
Number of Cycles
Convgerence Criterion
0.01
Print Options (0)
Marquardt Damping
1.00
LS matrix bandwidth 0
Reflection Intensity Extraction
Extraction Method
LeBail damping 0
Extract Fobs
(Phase #)
Rietveld
F(calc) Weighted
(Model biased)
Equally Weighted
(Le Bail method)

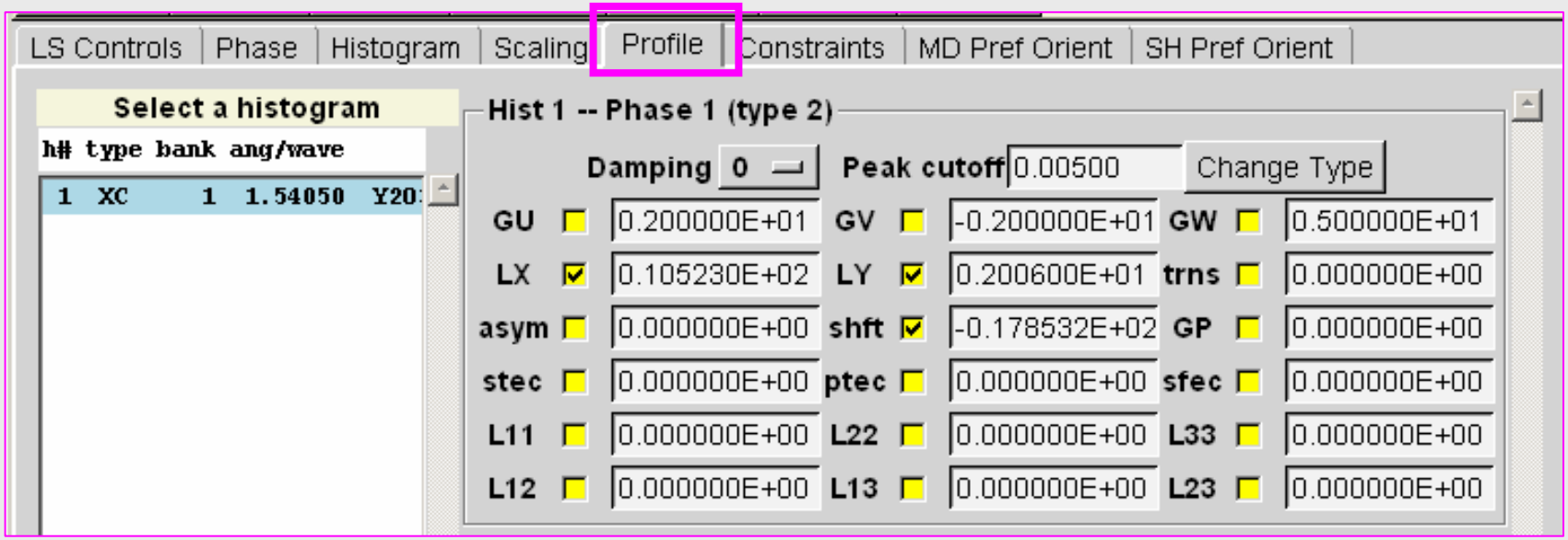

EXPGUI interface to GSAS: C:/gsas/MyWork/MyY2O3/Y2O3_A.EXP
File Options Powder Xtal Graphs Results Calc Import/Export Help
expnam expedt genles powpref powplot lstview liveplot
LS Controls Phase Histogram Scaling Profile Constraints MD Pref Orient SH Pref Orient
Phase:
Add Phase
add new phase
Adding phase #1
Phase title:
Space Group:
Add Cancel Help
Import phase from: Crystallographic Information File (CIF)
Use the icsd_86815.cel file
Phase: 1 Replace
title: from C:/Documents and Settings/Carlo/Desktop/Dakar09_
Refine Cell
Cell damping
Edit Cell
* name type ref/damp fractional coordinates Mult Occupancy Uiso
1 Y1 Y+3 0 0 0 0.968320 0.000000 0.250000 24 1.0000 0.42000
2 Y2 Y+3 0 0 0 0.250000 0.250000 0.250000 8 1.0000 0.54000
3 O1 O-2 0 0 0 0.390560 0.151630 0.380410 48 1.0000 0.61000
Editing atom #3 -- O1
Add New Atoms
Refinement Flags: X U F Damping: X U F
Xform Atom
Label O1 Coordinates 0.390560 0.151630 0.380410 Occupancy 1.000000
Uiso 0.610000


EXPGUI interface to GSAS: C:/gsas/MyWork/MyY2O3/Y2O3_A.EXP
File Options Powder Xtal Graphs Results Calc Import/Export Help
expnam expedt genles powpref powplot lstview liveplot
LS Controls Phase Histogram Scaling Profile Constraints MD Pref
Select a histogram
h# type bank ang/wave title
Add New Histogram
Set Histogram Use Flags
add new histogram
Adding a new histogram
Dummy Histogram
Data file: y2o3.gsas
Select File
Select bank
Instrument Parameter file: inst_xray.prm
Select File
Edit file
Select set
Usable data limit:
d-min
Q-max
TOF-min
2-Theta Max
Run RAWPLOT
Add
Cancel
Add multiple banks
Help
Editing instrument parameter file inst_xray.prm
Select bank: 1 Add bank
Data type: CW X-ray
Save
Save as
Close
Bank #1
Title: DUMMY INCIDENT SPECTRUM FOR X-RAY DIFFRACTOMETER
Instrument name: My test SR
Radiation type: Other
Monochromatic
Dual
Wavelength: Primary 1.5406 Secondary 1.5444 ratio 0.500
Zero Correction: 0.0000
Polarization Correction: Diffracted Beam Incident Beam None
Polarization Fraction: 0.750
Select profile: 1 2 3 Add profile Import profile
Profile type 2
Peak cutoff 0.01000
GU 2.000000 GV 0.000000E+000 GW 5.000000 LX 1.000000
LY 1.000000 trns 0.000000E+000 asym 0.000000E+000 shft 0.000000E+000
GP 0.000000E+000 stec 0.000000E+000 ptec 0.000000E+000 sfec 0.000000E+000
L11 0.000000E+000 L22 0.000000E+000 L33 0.000000E+000 L12 0.000000E+000
L13 0.000000E+000 L23 0.000000E+000


EXPGUI interface to GSAS: C:/gsas/MyWork/MyY203/Y203_C.EXP (modified)
File Options Powder Xtal Graphs Results Calc Import/Export Help
expnam expedt genles powpref powplot lstview liveplot
LS Controls Phase Histogram Scaling Profile Constraints MD Pref Orient SH Pref Orient
Select a histogram
h# type bank ang/wave tit
1 XCR 1 1.54060 Y203 po
Background
Function type 2 (3 terms) Edit Background
Refine background Damping 0
Diffractometer Constants
wavelengths 1.5406 1.5444
Refine ratio Ratio 0.50000 Damping 0
Refine zero Zero 0.0000
Refine POLA POLA 0.750 IPOLA 0
Absorption/Reflectivity Correction
Refine Abs./Refl. Damping 0 Edit Abs./Refl.
Add New Histogram
Set Data Limits & Excluded Regions
Set Histogram Use Flags



$$I_{calc} = I_{bck} + S \sum_{hkl} C_{hkl}(\theta) F^2_{hkl}(\theta) P_{hkl}(\theta)$$



peak breadth Gaussian: $\sigma^2 = GU \tan^2\theta + GV \tan\theta + GW + GP/\cos^2\theta$

Gaussian Sherrer broadening

sample shift: $s = -\pi R\, shft / 3600$

sample absorption: $\mu_{eff} = -9000 / (\pi R\, Asym)$

peak breadth Lorentzian : $\gamma = (LX - ptec \cos\phi)/\cos\theta + (LY - stec \cos\phi) \tan\theta$

Lorentzian Sherrer broadening (particle size)

Anisotropy

Lorentzian strain broadening

Anisotropy (stacking faults)

Gaussian Breadth: $\sigma^2 = GU \tan^2\theta + GV \tan\theta + GW + GP/\cos^2\theta$

Lorentzian Breadth: $\gamma = (LX - ptec \cos\phi)/\cos\theta + (LY - stec \cos\phi) \tan\theta$

Strain: **S** = $\Delta d/d$

Gaussian contrib. $S = \text{sqrt}[8 \ln 2 \, (GU - U_i)] \, (\pi/18000) \cdot 100\%$

Instrumental contribution

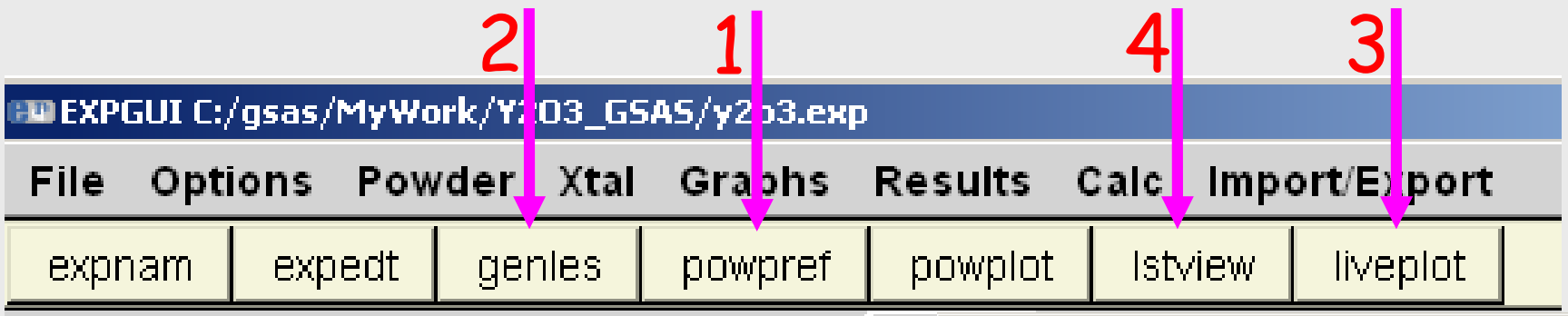
Lorentzian contrib. $S = (LY - Y_i) \, (\pi/18000) \cdot 100\%$

Instrumental contribution

Particle size: **P**

$P = (18000/\pi) \, K \lambda / LX$

Scherrer constant

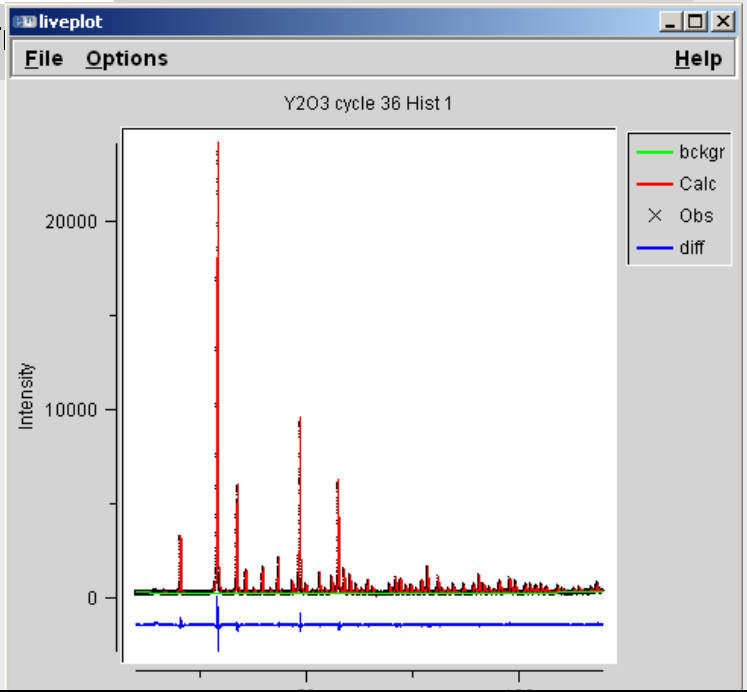


$$Mp = \Sigma\, w\,(I_{exp}-I_{calc})^2$$

$$Rp = \Sigma\,(I_{exp}-I_{calc}) \,/\, \Sigma\, I_{exp}$$

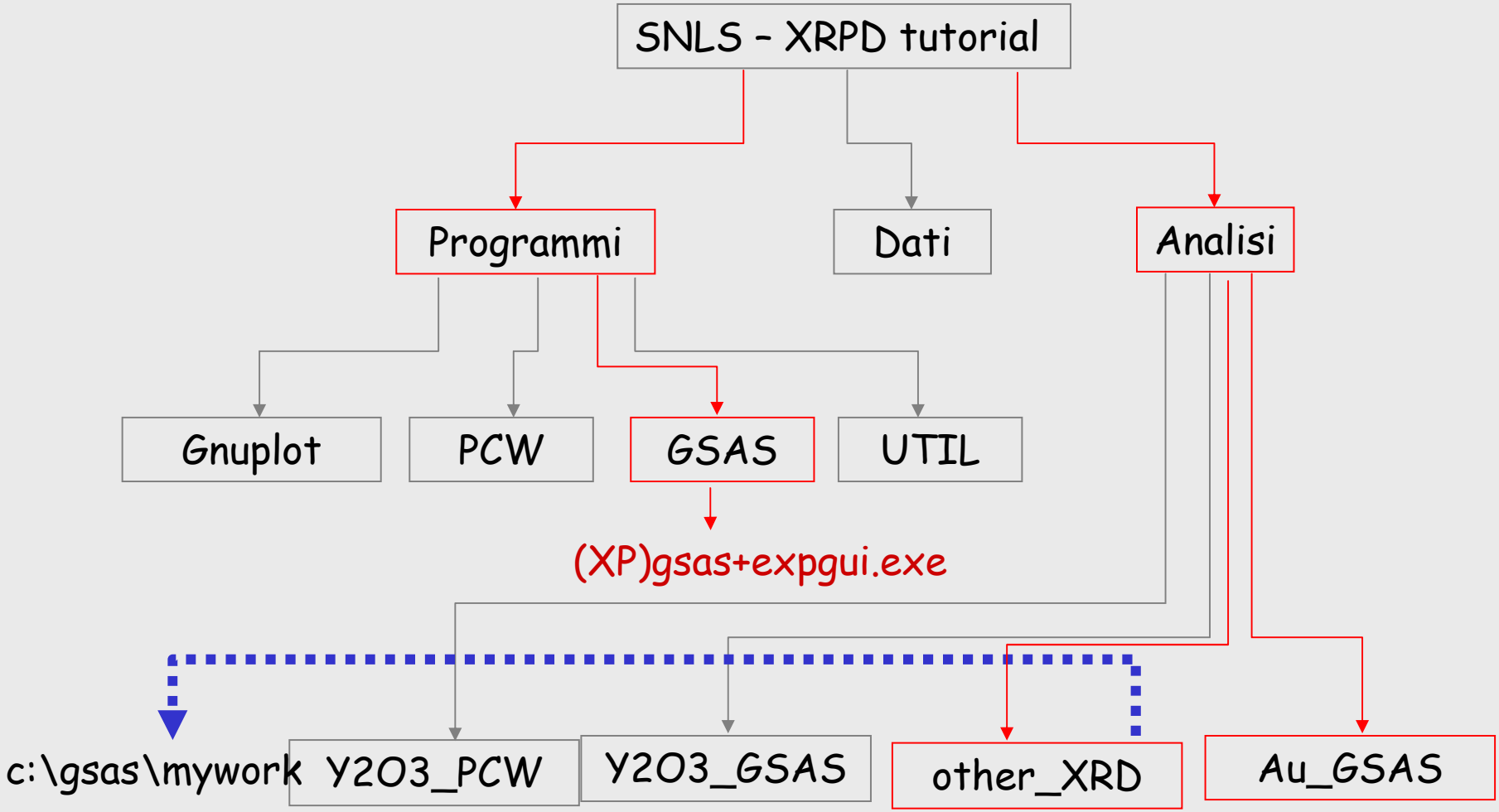
$$wRp = sqrt[\, Mp \,/\, \Sigma\, I^2_{exp}\,]$$

$$\chi^2 = Mp \,/\, (N_{obs} - N_{var})$$




```
Powder data statistics                    Fitted          -Bknd                  Average
            Bank Ndata Sum(w*d**2)   wRp      Rp     wRp      Rp      DWd   Integral

Hstgm  1 PXC   1  5500   11430.     0.0646 0.0486 0.0575 0.0458   1.013    0.938
Powder totals     5500   11430.     0.0646 0.0486 0.0575 0.0458   1.013
Cycle  37 There were   5500 observations.
Total before-cycle CHI**2 (offset/sig) = 1.1430E+04 ( 5.6760E+01)

Reduced CHI**2 =  2.084     for    15 variables
Histogram  1  Type PXC   Nobs=  306 R(F**2) =  0.0361
```

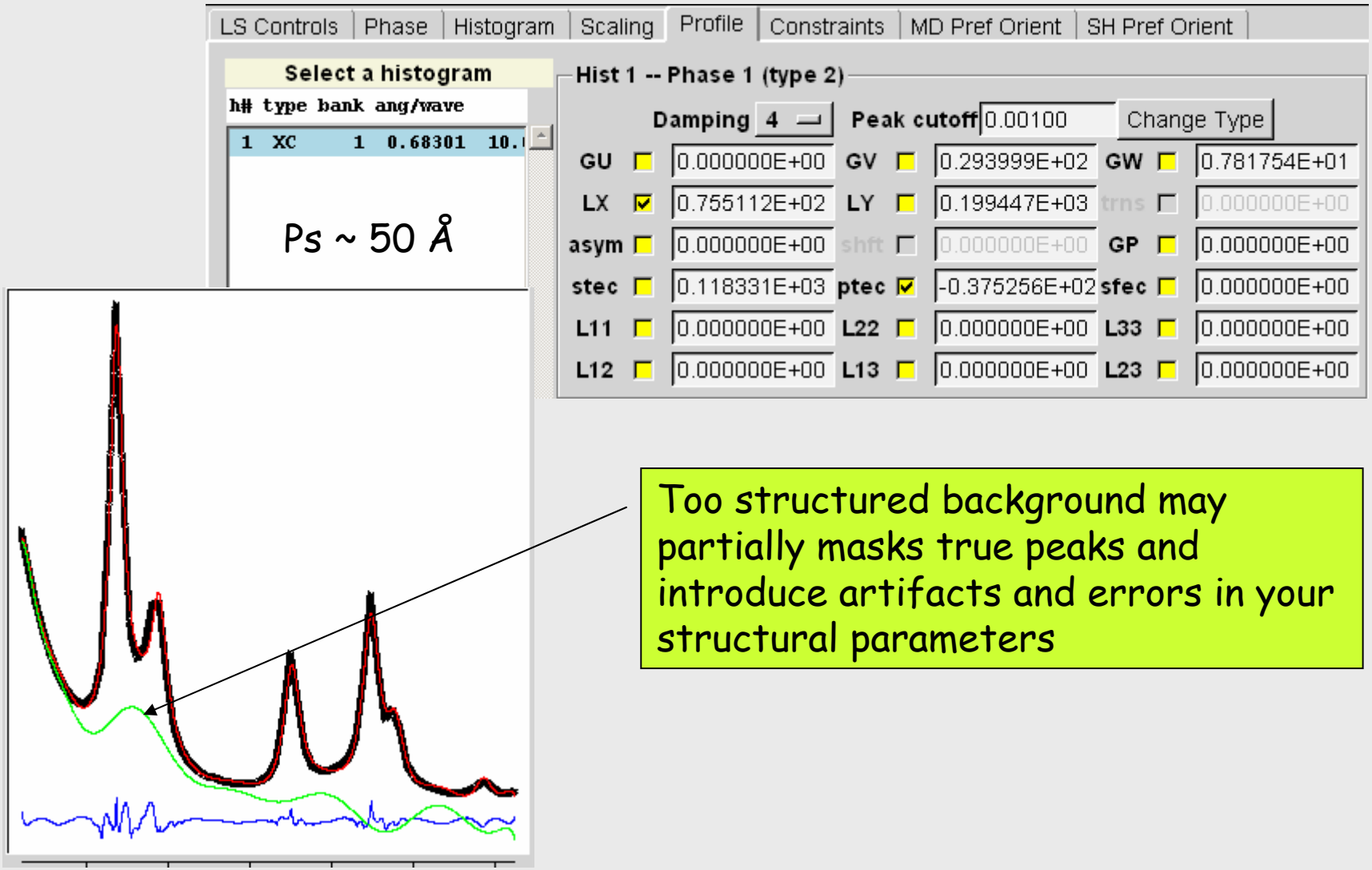
GSAS


SNLS – XRPD tutorial
Programmi
Dati
Analisi
Gnuplot
PCW
GSAS
UTIL
(XP)gsas+expgui.exe
c:\gsas\mywork
Y2O3_PCW
Y2O3_GSAS
other_XRD
Au_GSAS

GOLDsF m3m λ=0.688011
Au= 0. 0. 0.
a = 4.0782

Au nanosized particles supported on wax

wide broad peaks on intense structured background!



Too structured background may partially masks true peaks and introduce artifacts and errors in your structural parameters

Now... you can (must) try!

Use files in XRD_DATA directory

For comments, suggestions, support request etc...

contacts:

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