Lecture notes

Rietveld 方法原理

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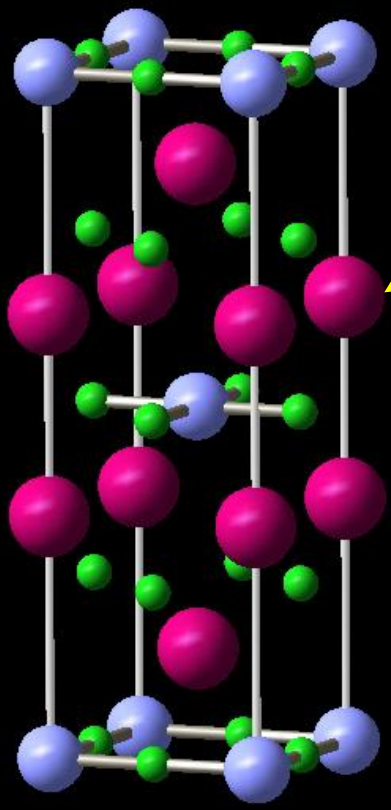
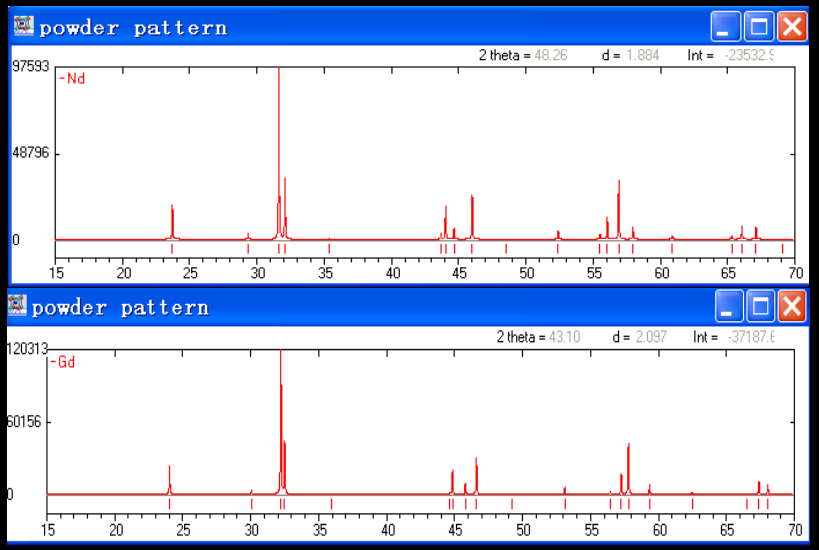
Isostructural Compounds

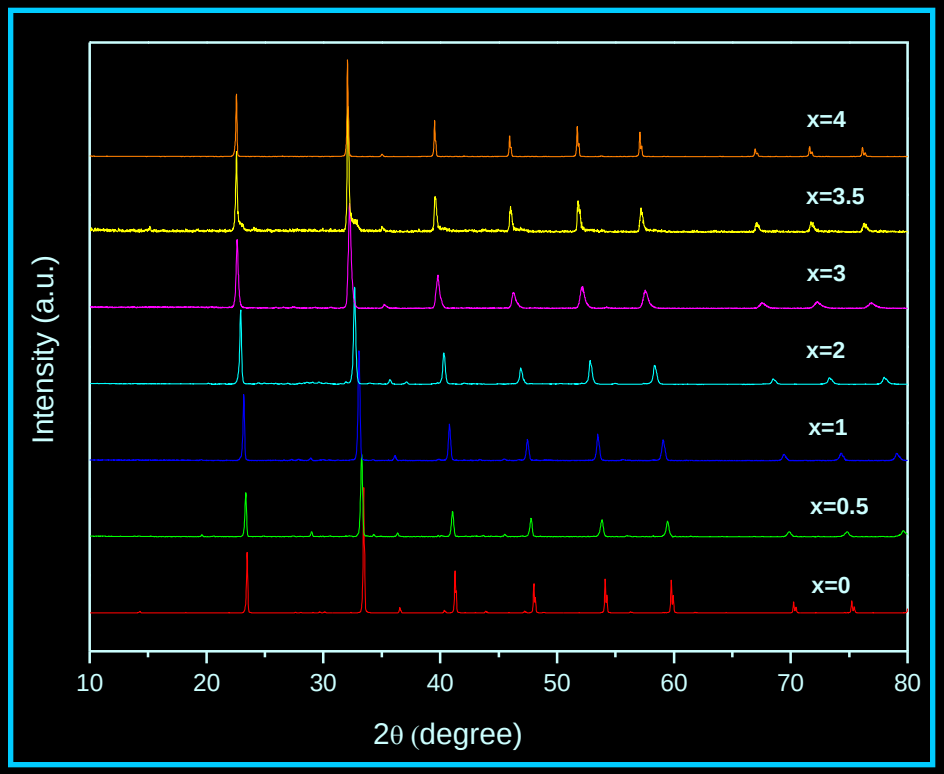


Nd(Gd)

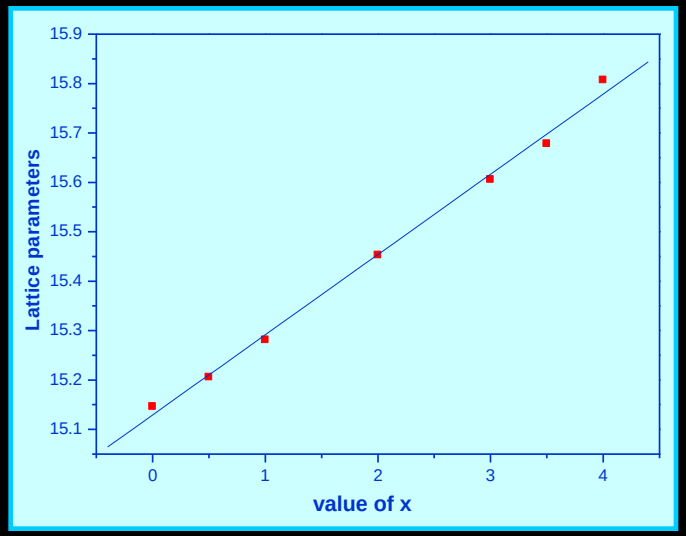


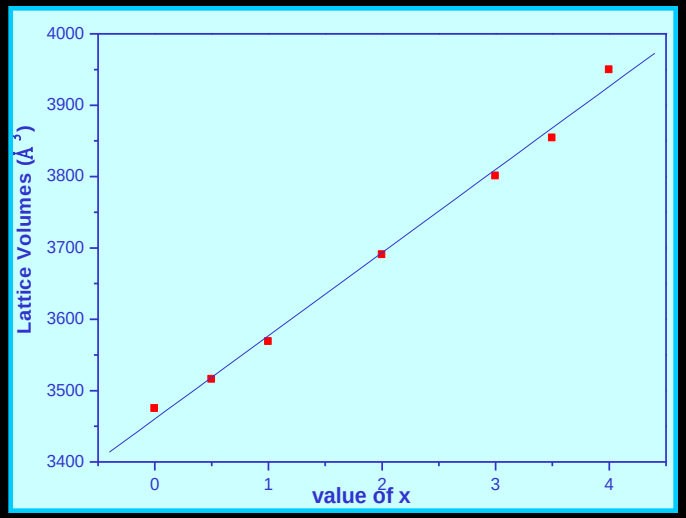
Nd_2CuO_4	Gd_2CuO_4
a=0.39419nm	a=3.8938nm
c=1.21627nm	c=11.8810nm
Z_{Nd}=0.353	Z_{Gd}=0.349

XRD patterns of Solid solution $NaSr_{4-x}Ba_xB_3O_9$ ($0 \le x \le 4$) :First cubic borate only with BO_3



Cubic borates are estimated below 1%.





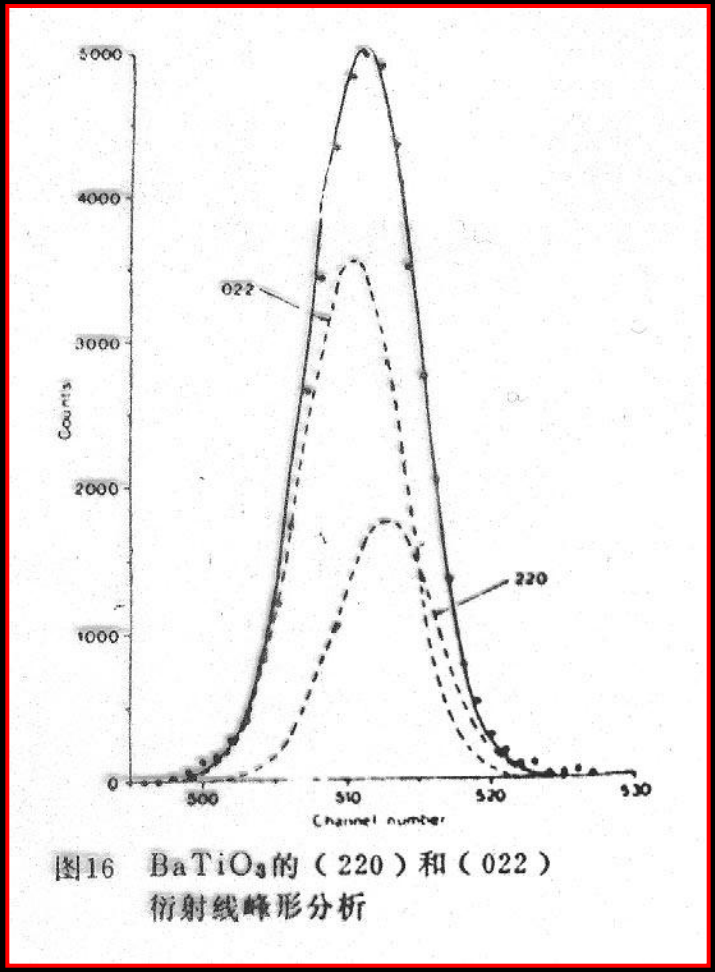
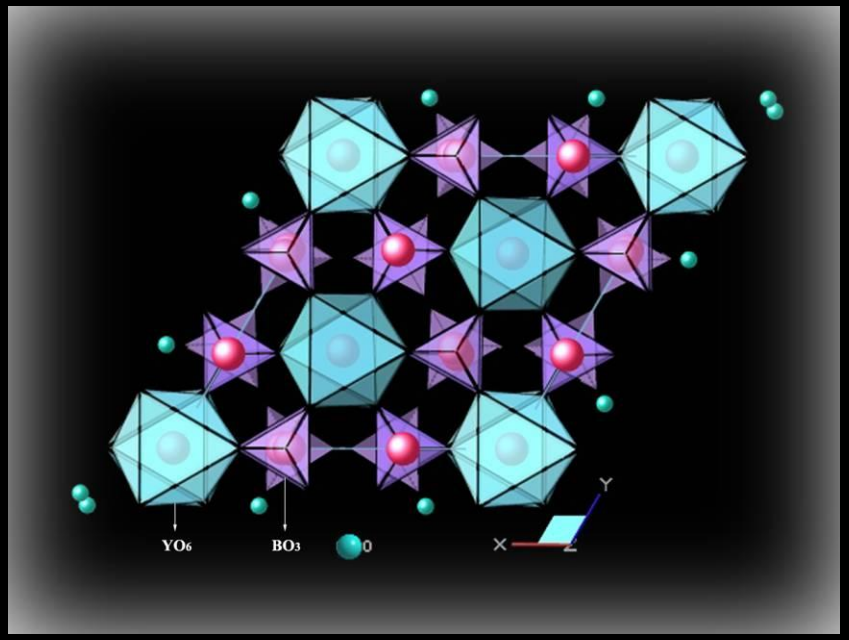
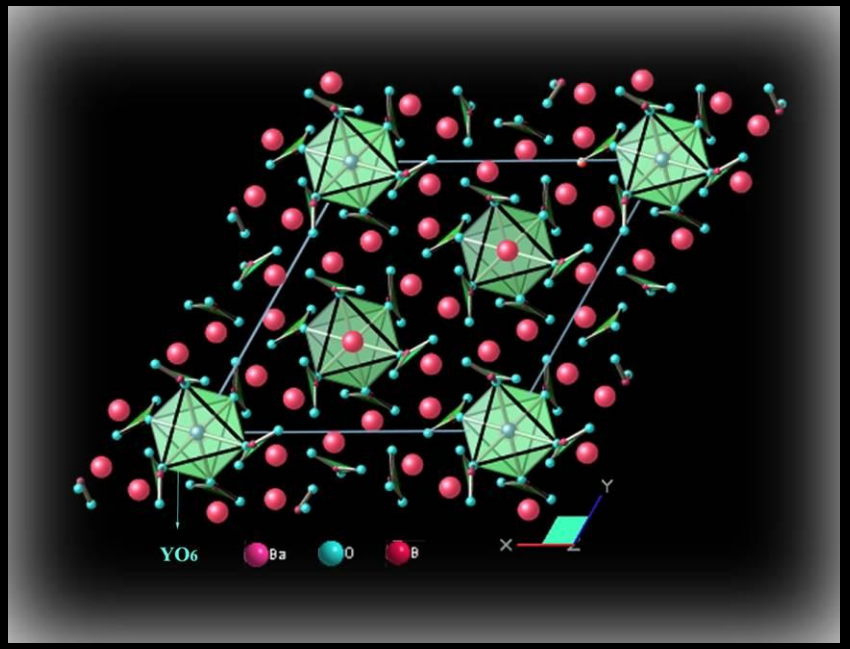


图16 $BaTiO_3$的（220）和（022）衍射线峰形分析

Phase transition of $BaTiO_3$ from tetragonal to cubic at about 132˚C

$YBa_3B_3O_9$: Phase transition and structure determination

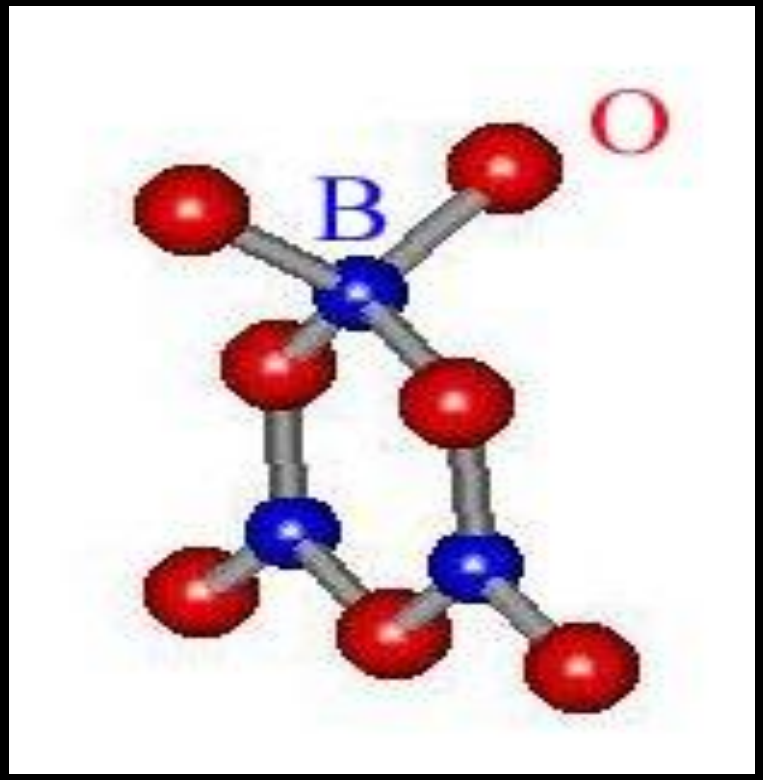


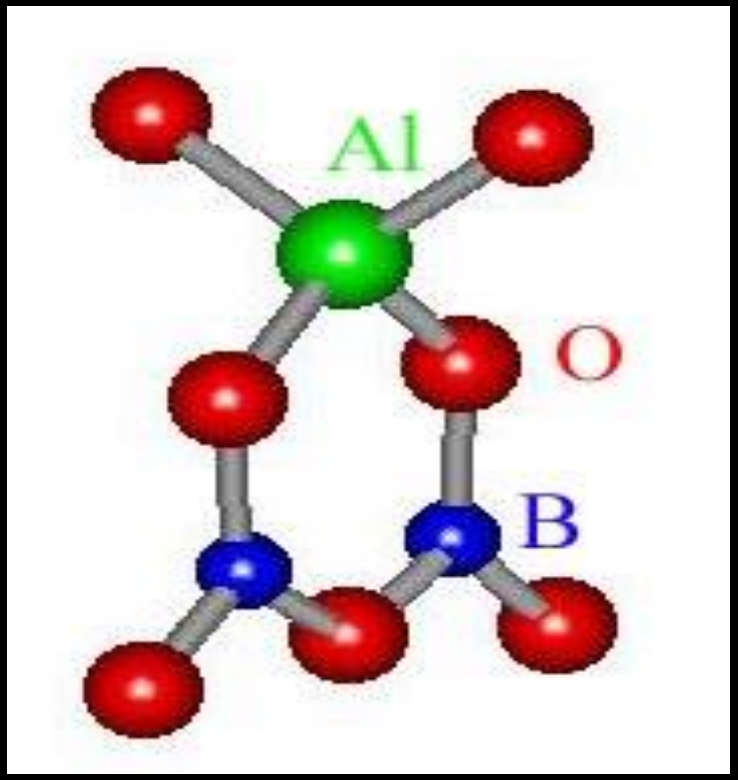


S. G.: *$P6_3cm$* (No. 185)
a=9.4235(4)Å, c=17.602(1) Å
1100°C

S. G.: *R-3* (No. 148)
a=13.0441(1)Å, c=9.5291(1) Å
1140°C

$LiAlB_2O_5$: Search for new SHG materials



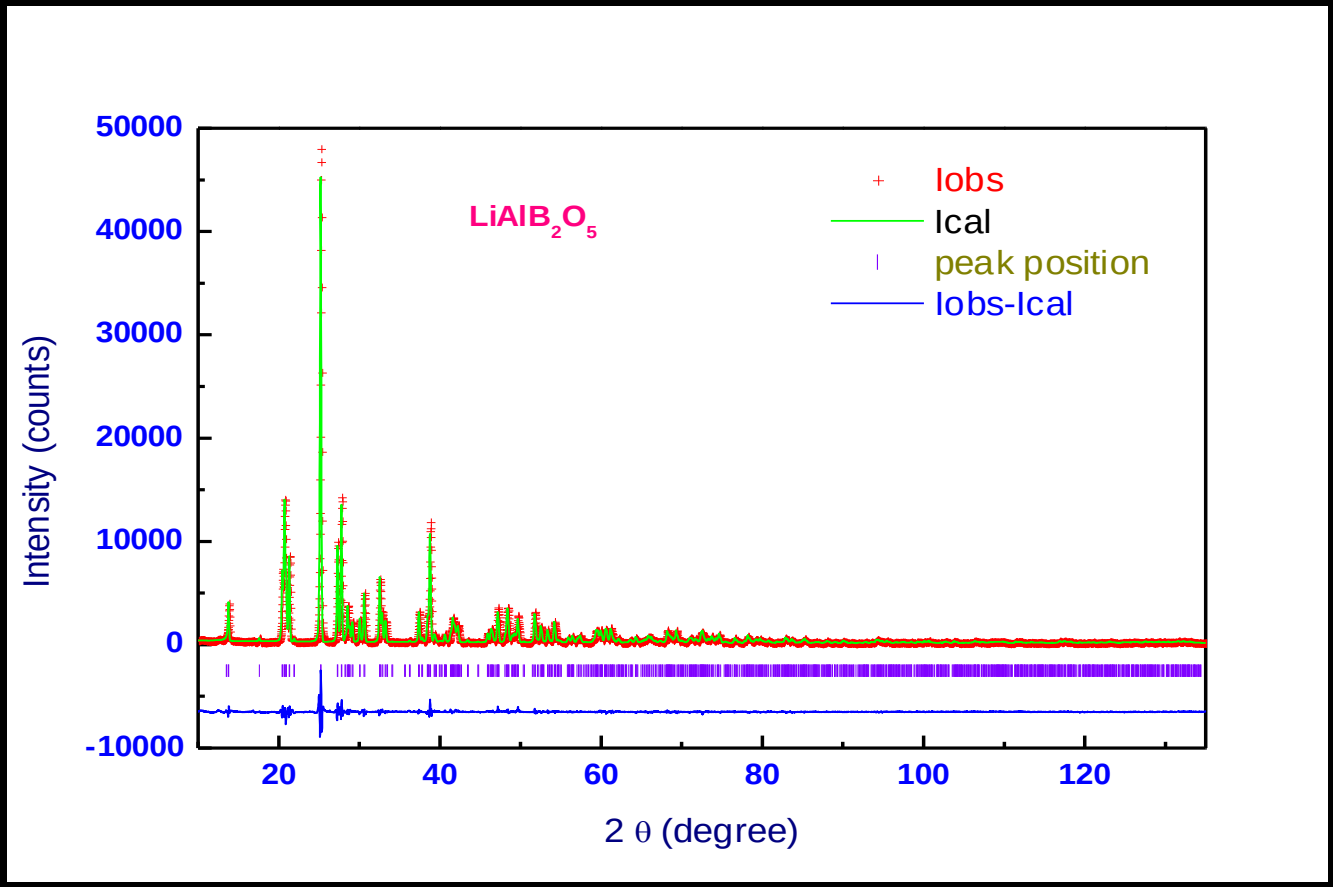


$[B_3O_7]^{5-}$ & $[AlB_2O_7]^{5-}$

Structural Data for $LiAlB_2O_5$

atoms	site	x	y	z	B(Å^2)
Li (1)	4e	0.0000	0.378 (1)	0.2500	2.6(3)
Li (2)	4e	0.0000	0.159 (2)	-0.2500	3.2(3)
Al	8f	0.1948(2)	0.1523(2)	0.1510(2)	1.18(4)
B (1)	8f	-0.0662(7)	0.3266(7)	-0.0308(8)	1.5(1)
B (2)	8f	0.2341(8)	-0.0050(6)	0.4071(8)	1.4(1)
O(1)	8f	0.0582(4)	0.2754(3)	0.1104(4)	1.63(8)
O(2)	8f	-0.1232(3)	0.2944(3)	-0.1897(3)	0.98(8)
O(3)	8f	-0.1386(4)	0.4271(3)	0.0238(4)	1.76(8)
O (4)	8f	0.1581(4)	0.0184(3)	0.2428(4)	1.64(8)
O (5)	8f	0.1846 (3)	0.1071(3)	-0.0311(4)	1.19(8)

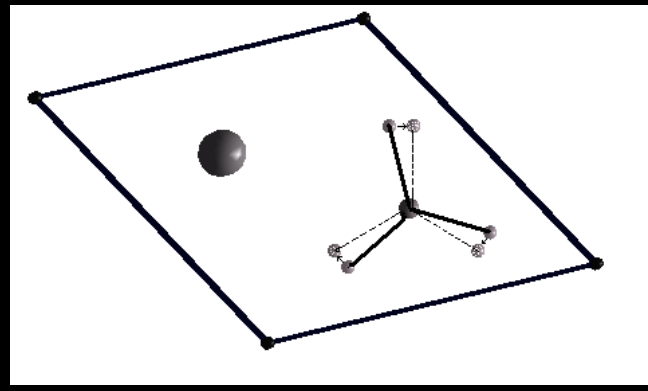
Final Refinement of New compound of $LiAlB_2O_5$



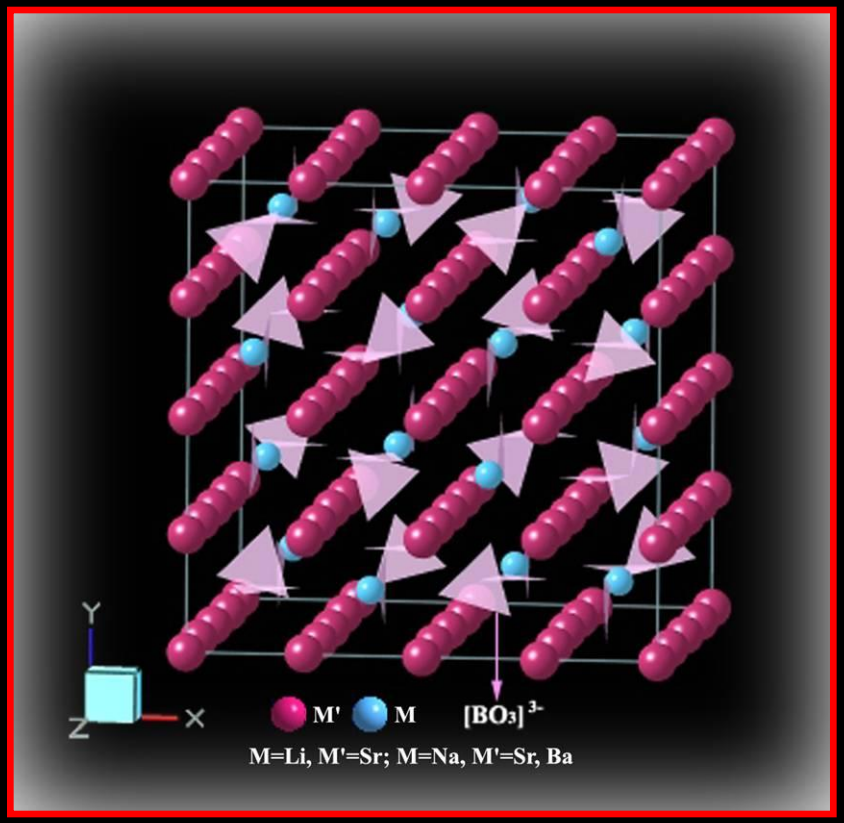
Structure vs Temperature:KCaCO3F

atoms	site	x	y	z	temperature (K)
K	1a	0	0	0	295-673
C	1f	0.66667	0.33333	0.5	295-673
F	1c	0.33333	0.66667	0	295-673
Ca	1d	0.33333	0.66667	0.5	295-673
		0.81094(7)	0.18906(-7)	0.5	295
		0.81044(8)	0.18957(-8)	0.5	373
O	3k	0.80974(9)	0.19026(9)	0.5	473
		0.81060(10)	0.19182(-10)	0.5	573
		0.80948(12)	0.19052(-12)	0.5	673

0.8120(3) 0.1880(3)
0.5 by x-ray data



$LiSr_4B_3O_9$: A comparison between structure determination from single-crystal and powder X-ray diffraction



Single-crystal: R_{int}=0.0745

R_1(all data)=0.0695

wR_2(all data)=0.1887

with weighting scheme:

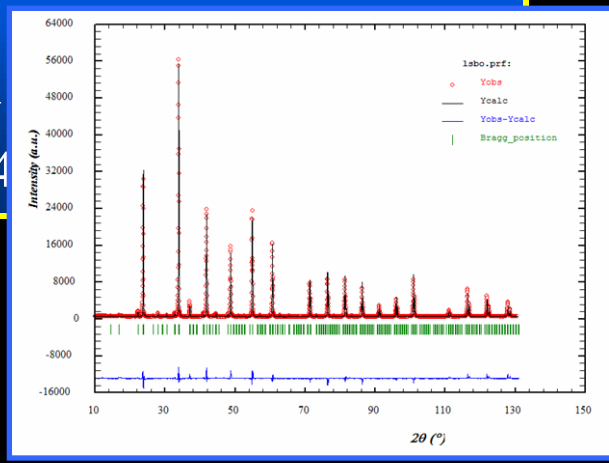
$W=1/[\sigma^2(Fo^2)+(0.0000P)^2+359.71P]$
where $P=(Fo^2+2Fc^2)/3$

SDPD: R_B=0.07

R_p=0.0609

R_{wp}=0.0811

R_{exp}=0.0314



a= 14.9470 Å S.G: Ia-3d
$(Z_B/Z_{Sr})^2=(3/38)^2 \approx 0.6\%$

What is a Rietveld Refinement?

-a standard treatment of powder diffraction data to make the final structural model achieve the accepted criterion;

-a best known method that fully makes use of the step-mode scanned data to dig out a lot of structural and other information;

- a procedure for structural solution in nature.

What can we get to perform a Rietveld refinement?

Lattice Parameters

Atomic Positions

Atomic Occupancy

Debye Temperatures

Crystallinity

Magnetic structures

Quantitative phase Analysis

Grain size

Incommensurate Structure

Structure factors

Phase transitions

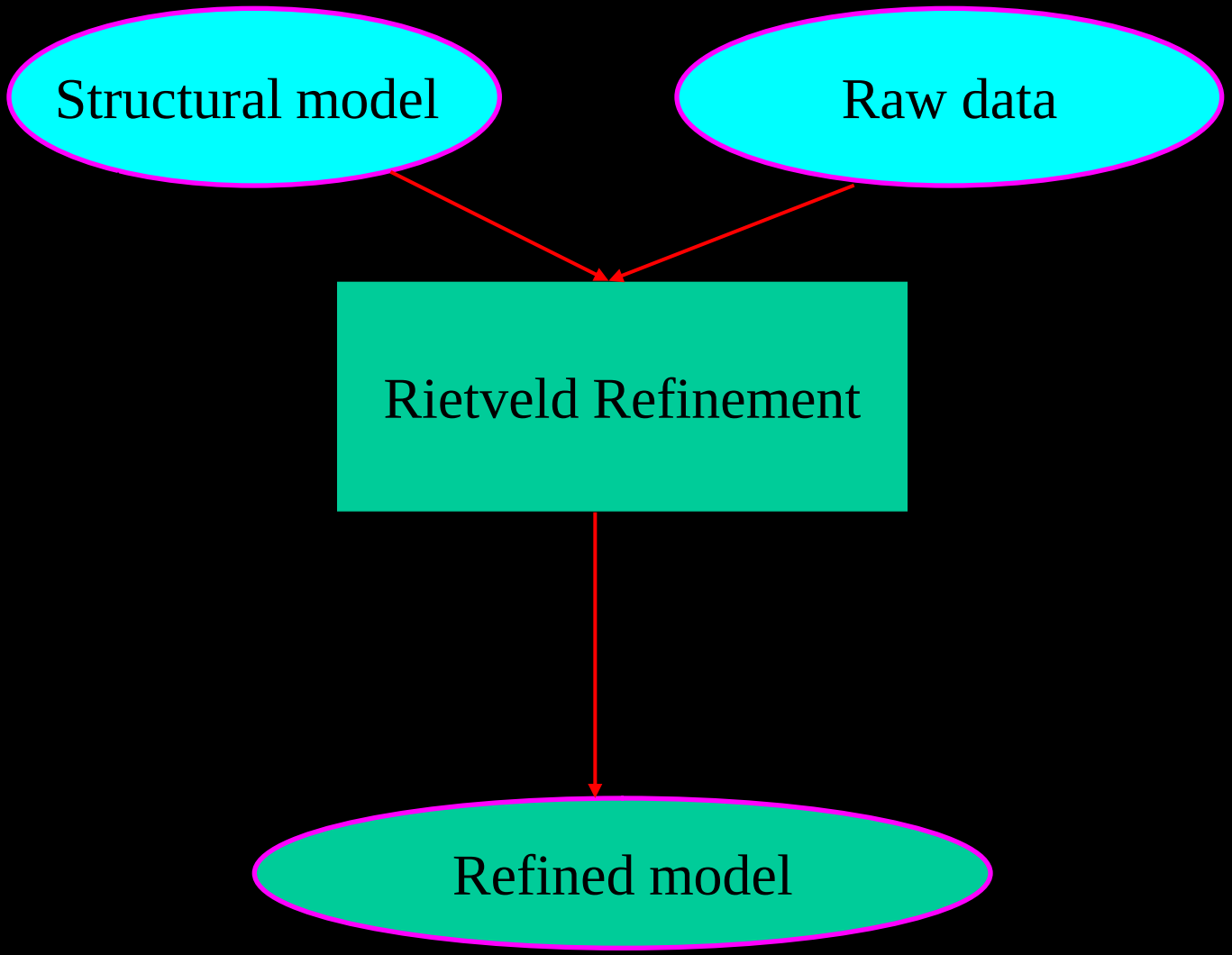
……

History Review

- Rietveld originally introduced the Profile Refinement method (Using step-scanned data rather than integrated Powder peak intensity) (1966,1967)
- Rietveld developed first computer Program for the analysis of neutron data for Fixed-wavelength diffractometers (1969)
- Malmos & Thomas first applied the Rietveld refinement method (RR) for analysis of x-ray powder data collected on a Ginier Hagg focusing Camera (1977)
- Khattack & Cox first applied the RR to x-ray powder data collected on a diffractometer (1977)
- Conference on Diffraction Profile Anlysis Sponsored by IUCr in Poland, suggested the term “Rietveld Method”(1978)
- Wiles and Yang developed a general computer program (D.B.W) for both x-ray & neutron diffraction data (fixed wavelength)(1981)
- Von Dreele, Jorgensen and Windsor extended to the program to the neutron diffraction data (1982)
- Fitch et al, 193 refined parameters,UO2 DAs.4D2O (1982)

Aminoff Prize, Stockholm,1995

- **H.M. Rietveld *Acta crystallogr*., 22, 151 (1967).**
- **H.M.Riveted, *J. Appl. Crystallogr*., 2, 65 (1969).**


Structural model
Raw data
Rietveld Refinement
Refined model

How RM works?

The RM refines a structure by minimizing a quantity through the Newton-Raphson algorithm

$$\chi^2 = \sum_{i=1}^{n} w_i \left\{ y_i - y_{c,i}(\boldsymbol{\alpha}) \right\}^2$$

where, y_i is the observed intensity at a certain 2θ,

$y_{c,i}$ is the calculated intensity at the same angle,

w_i is a weight, we usually take $w_i = 1/y_i$

$i = 1,2,\ldots n$

$\alpha = (\alpha_1\ \alpha_2\ \ldots \alpha_p)$, the parameters to be refined.

$$\left(\frac{\partial \chi^2}{\partial \alpha}\right)_{\alpha=\alpha_{opt}} = 0$$

Given a solution $\alpha=\alpha_{opt}(\alpha_1, \alpha_2 \ldots \alpha_p)$ that approximately satisfy the above equation. To find a better solution, we begin an iterative process by expanding $\left(\frac{\partial \chi^2}{\partial \alpha}\right)$ into a Taylor series

$$\left.\frac{\partial \chi^2}{\partial \alpha}\right|_{\alpha=\alpha_1} = \left.\frac{\partial \chi^2}{\partial \alpha}\right|_{\alpha=\alpha_{opt}} + \sum_{i=1}^{p} \frac{\partial \chi^2}{\partial \alpha} \cdot \frac{\partial \chi^2}{\partial \alpha_i} \delta_i$$

$$A\delta_{\alpha 1}=b$$

$$A_{kl} = \sum_i w_i \frac{\partial y_{c,i}(\alpha_0)}{\partial \alpha_k} \frac{\partial y_{c,i}(\alpha_0)}{\partial \alpha_l}$$

$$b_k = \sum_i w_i (y_i - y_{c,i}) \frac{\partial y_{c,i}(\alpha_0)}{\partial \alpha_k}$$

$$\alpha_1 = \alpha_0 + \delta_{\alpha 1}$$

$$y_{c,i} = \sum_{\phi} S_{\phi} \sum_{\mathrm{h}} I_{\phi,\mathbf{h}} \, \Omega(T_i - T_{\phi,\mathbf{h}}) + b_i$$

$$I_{\phi,\mathbf{h}} = \left\{ L \, A \, P \, C \, F^2 \right\}_{\phi,\mathbf{h}}$$

S_{ϕ} is the scale factor of the phase ϕ
L_{h} contains the Lorentz, polarisation and multiplicity factors.
F_{h} is the structure factor
A_{h} is the absorption correction
P_{h} is the preferred orientation function
Ω is the reflection profile function that models both instrumental and sample effects

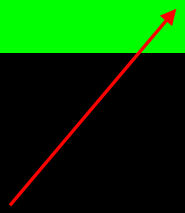
$$F_{hkl} = \sum_{i}^{n} f_i e^{2\pi(hx_i + ky_i + lz_i)}$$

where, f_i atomic scattering factor for ith atom

x_i, y_i and z_i the fractional coordinates for ith atom

$$f_i = f_{i0} e^{-M}$$

$$M = 8\pi^2 \overline{u^2} \left(\frac{\sin\theta}{\lambda}\right)^2 = B\left(\frac{\sin\theta}{\lambda}\right)^2$$



The mean square displacement of the atom in a direction normal to the reflecting planes

$$\alpha_i^{n+1} = \alpha_i^n + \delta_i \cdot RELAX \cdot CC$$

Where RELAX is relaxtion factors that are used to control the shifts to avoid divergence;

and CC is a multiplier.

What we need to perform a RR?

A set of step-mode scanned data,

usually 2θ=10-120˚ or more, step $\Delta 2\theta$=0.02˚

collecting time is instrument dependent from 1-20s

for laboratory diffractometer;

An initial structural model having roughly accurate lattice constants, correct space group and approximate atomic positions

How we obtain an initial structural model?

- solid solutions usually adopt same structure types of their parent compounds;

  $NaSr_{4-x}Ba_xB_3O_9$ ($0 \leq x \leq 4$)

- Compounds with same chemical formula

  $YBa_2Cu_3O_7$ and $NdBa_2Cu_3O_7$

  but always alert that exceptions are not uncommon

  La_2CuO_4 and Nd_2CuO_4

- Try and error

- Ab inito structure determination

Is the compound known? Crystallographic Structure Databases

- ICSD (Minerals and Inorganics)
  - http://www.fiz-karlsruhe.de/
  - **Minerals and Inorganic**
  - Over 60000 entries
- Cambridge Structure Data Bank）
  - http://www.ccdc.cam.ac.uk
  - Organics & Organometallics
  - Over 250000 entries

- ICDD diffraction data
  - http:http://www.icdd.com/
  - Inorganic & Organic
  - Over 140000 entries
- NIST Crystal Data
  - http://www.nist.gov/srd/nist3.htm
  - Inorganic & Organic
- Over 230000 entries

A new structural database(2003):

aimed at freely retrieving data

18000 Patterns already!

Parameters in PCR file

The parameters in PCR file can be divided into three categories

-relating only to samples, refinable
such as atomic positions, temperature factors
-relating both to samples and instruments
such as scale factors, FWHM (Full width at half maximum)
-user-specified parameters
such as BKPOS, Nba

```
COMM   KFCaCO3 Aug.15,2003
! Current global Chi2 (Bragg contrib.) =      17.11
! Files => DAT-file: kca1,  PCR-file: kca1
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
   0   5   1   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0   0
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
   0   0   1   0   2   0   0   0   0   1   0   1   0   1   2   0   0
!
! lambda1 Lambda2    Ratio     Bkpos     Wdt     Cthm     muR    AsyLim   Rpolarz ->Patt# 1
 1.540560 1.544390  0.5000    90.000 12.0000  0.8009  0.0000    80.00    0.0000
!
!NCY  Eps  R_at  R_an  R_pr  R_gl     Thmin       Step       Thmax    PSD    Sent0
 10  0.01  0.50  0.50  0.50  0.50     10.0000   0.020000   135.0000   0.000   0.000
!
! Excluded regions (LowT  HighT) for Pattern#  1
        0.02        9.98
      135.02      180.00
!
!
      24    !Number of refined parameters
!
!  Zero     Code    SyCos     Code   SySin     Code  Lambda     Code MORE ->Patt# 1
  0.00000  11.00  0.00000    0.00  0.00000 240.01 0.000000    0.00   0
!   Background coefficients/codes  for Pattern#  1
   0.000       0.000        0.00        0.00        0.00     -  00.0
      31.000      41.000      51.000     211.000     221.000     231.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     9.43
!-------------------------------------------------------------------------------
```

Codewords(I)

codewords are used to control parameters when to be refined, when to be fixed and when to be constrained and etc. A codeword is formed as

$$C\alpha=S(10P+CC)$$

Where S stands for the sign mark, P is an ordinal number set by users from 1 to p, the maximum number of parameters

Codeword(II)

- For example, an atom Ca position is (0,0,z) with z to be refined from its initial value 0.1. The codeword in your PCR file looks like the following

……

Ca1 Ca+2 0.0 0.0 0.1 …

0.0 0.0 120.5 …

……

Here, S=1, P=12, and CC=0.5. That means that z of Ca1 is the 12nd parameter to be refined in the iterative process, and x and y of Ca1 occupy special positions not needed to be refined.

Codeword(III)

- Another example: the lattice constants of a tetragonal compound are to be refined. The codeword in your PCR file looks as follows

  3.891 3.891 11.732

  51.0 51.0 61.0

  In this case, constraint is put on *a* and *b* by using the same codeword since *a*=*b* always holds in tetragonal compounds

Codeword(IV)

One more example: the occupancies of two kinds of atoms at one site are to be refined. Solid solutions are the most common among this kind of refinements. The codewords in your PCR file are set as

Y	Y+3	……	0.8
		……	10.3
Yb	Yb+3	……	0.2
		……	-10.3

Only in this way are the occupancies guaranteed to satisfy

Occ(Y)+Occ(Yb)=1

Codeword(V)

- each parameter usually controlled by one codeword. Be alert that one codeword should be given to two or more parameters that are irrelevant;

- there is no limit to choose ordinal numbers.

  But we usually set the first ordinal numbers to global parameters such as zero point, background parameters and the etc.

Modeling backgrounds

- The background intensity bi at the ith step may be obtained by any of the following three method.

- a specified background function, usually a polynomial;
- linear interpolation between user-selected points in the pattern
- A user-supplied function

Control flags

The choice of background type is indicated by a control flag

- Comment line(4) Job Npr Nph Nba Nex…

  0 5 1 0 2

Nba:

=0 Refine background with a polynomial

=1 Read background from file COFHIL.bac

=2,3,…,N linear interpolation between N given points

…

$$b_i = \sum_{m}^{11} B_m \left(\frac{2\theta}{BKPOS} - 1\right)^m$$

Where Bm are parameter to be refined

BKPOS is a user-specified parameter, origin of polynomial function, non-refinable.

If 2θ=BKPOS, we see $b_i = B_0$

Users can look into their data files to set the values of BKPOS

Profile functions (I)

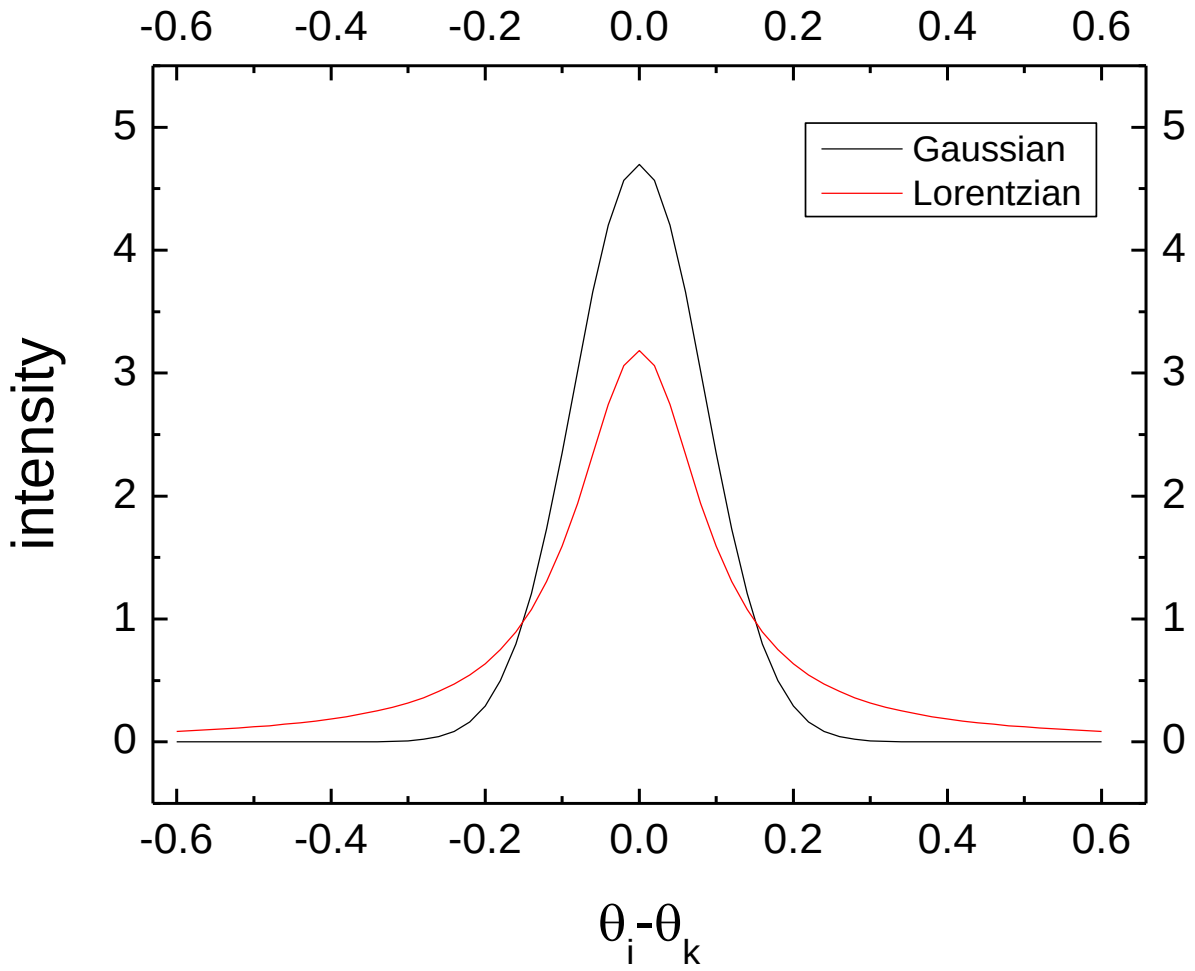
Npr=0

$$\frac{C_0^{0.5}}{H_K \pi^{0.5}} \exp(-C_0 (2\theta_i - 2\theta_k)^2 / H_K^2)$$

Lorentzian (L)
Npr=1

$$\frac{C_1^{0.5}}{\pi H_K} \times \frac{1}{[1 + C_1 \frac{(2\theta_i - 2\theta_k)^2}{H_K^2}]}$$

Parameter to be refined: H_k, Full Width at Half Maximum (FWHM)


-0.6
-0.4
-0.2
0.0
0.2
0.4
0.6
5
4
3
2
1
0
intensity
Gaussian
Lorentzian
θi-θk

H_k=0.2

Profile functions (II)

Mod.I Lorentzian

Npr=2

$$\frac{2C_2^{05}}{\pi H_K} \times \frac{1}{[1+C_2 \frac{(2\theta_i - 2\theta_k)^2}{H_K^2}]^2}$$

Mod.I Lorentzian

Npr=3

$$\frac{C_3^{05}}{2\pi H_K} \times \frac{1}{[1+C_3 \frac{(2\theta_i - 2\theta_k)^2}{H_K^2}]^{3/2}}$$

Parameter to be refined: H_k,

Profile functions (III)

Psudo-Voigt

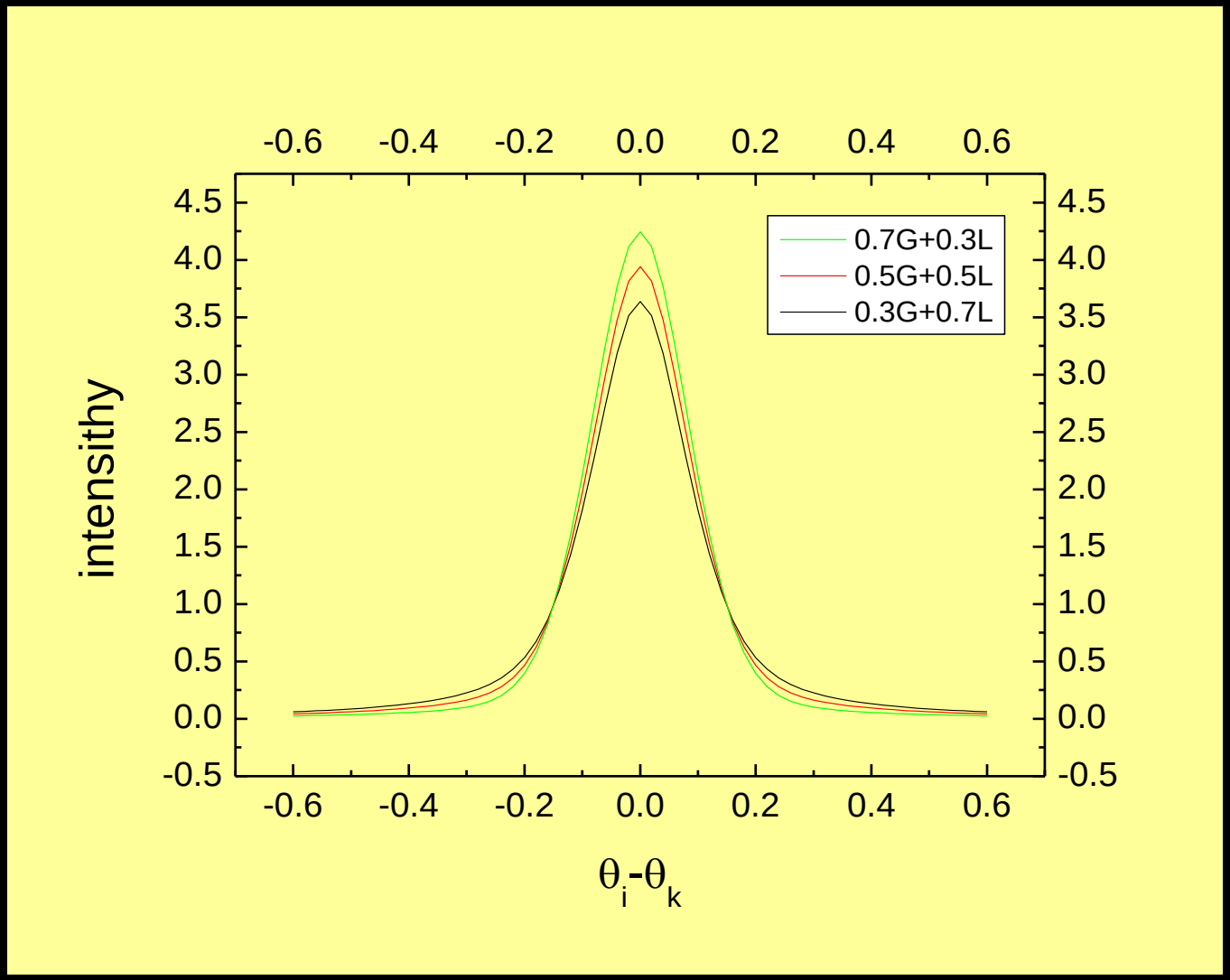
Npr=5

$$\eta L + (1-\eta)G$$

$$\eta = \eta_0 + X * (2\theta)$$

Parameters to be refined: H_k, η_0, X

η_0 = shape



Pseudo-Voigt functions

H_k=0.2

Profile functions (III)

Pearson VII

$$\frac{C_4}{H_K}\left[1+4*(2^{1/m}-1)\frac{(2\theta_i-2\theta_k)^2}{H_K^2}\right]^{-m}$$

$$m = m_0 + 100\frac{X}{2\theta} + 1000\frac{Y}{(2\theta)^2}$$

Parameters to be refined: H_k, m_0, X, Y

Profile functions (IV)

(Mod-TCHZ pV) $$L(x) \otimes G(x) = \int_{-\infty}^{+\infty} L(x-u)G(u)du$$

L(x) and G(x) have different FWHMs H_L and H_G

$$\eta = 1.36603\frac{H_L}{H} - 0.47719(\frac{H_L}{H})^2 + 0.1116(\frac{H_L}{H})^3$$

$$H = \left(H_G^5 + AH_G^4H_L + BH_G^3H_L^2 + CH_G^2H_L^3 + DH_GH_L^4 + H_L^5\right)^{0.2}$$

Parameters to be refined: H_G and H_L

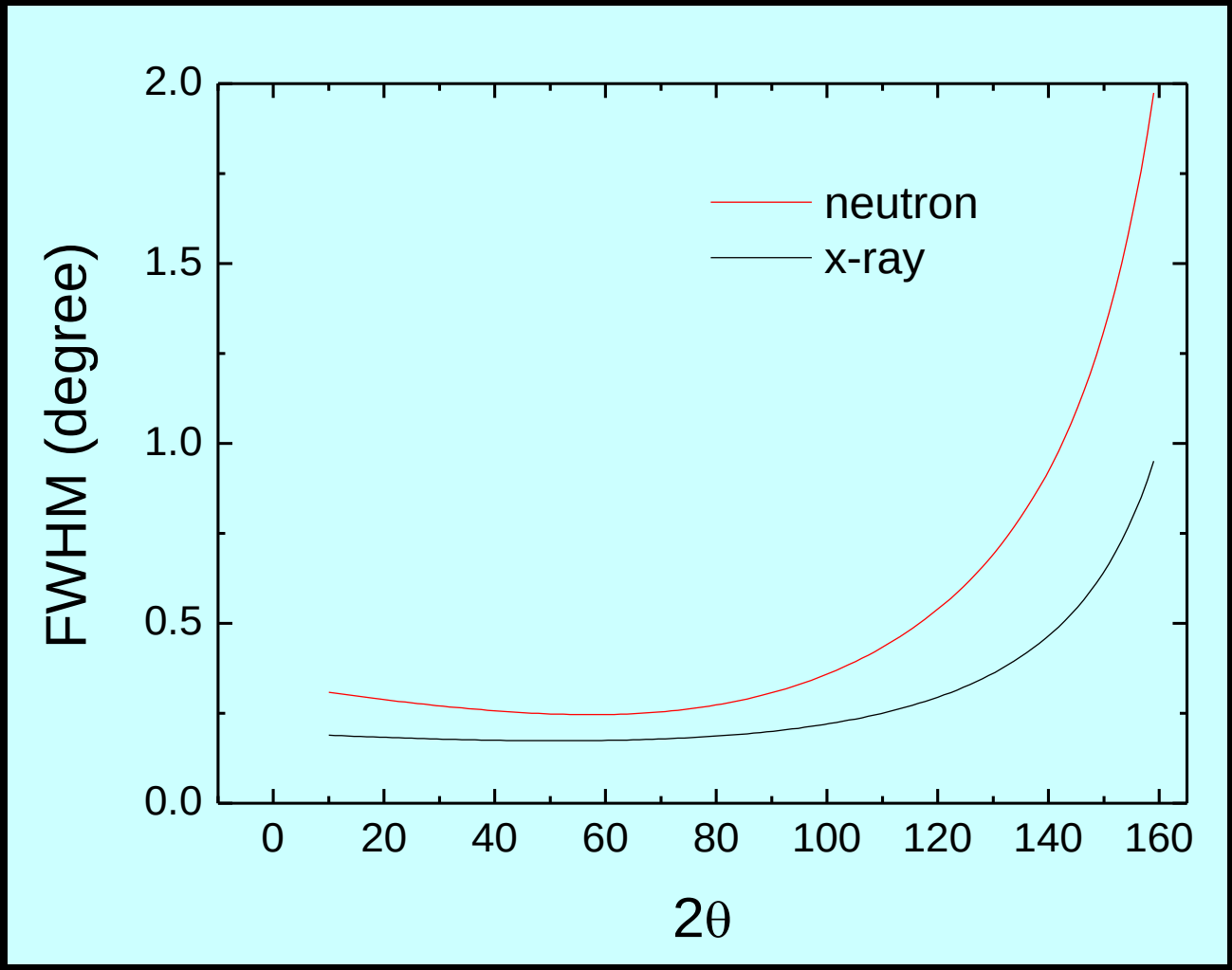
Full width at half Maximum (FWHM)

$$H_G = U\tan^2\theta + V\tan\theta + W + \frac{I_g}{\cos^2\theta}$$

For Npr=0…6, $H_k=H_G$

$$H_L = X\tan\theta + \frac{[Y + F(S_z)]}{\cos\theta}$$

For Npr=7, H_L is required apart from H_G



Typical variations of FWHM vs 2θ

Summary for the parameters to be refined with different profiles

Npr=0, Gaussian: U, V, W, I_g	3
Npr=5, pv: U,V,W,I_g, η_0(Shape), X	5
NPr=6, Pearson VII: U,V,W,I_g, η_0(Shape), X,Y	6
NPr=7, TCHZpv: U,V,W,I_g, X,Y,S_z	6

```
COMM   KFCaCO3 Aug.15,2003
! Current global Chi2 (Bragg contrib.) =      17.11
! Files => DAT-file: kca1,  PCR-file: kca1
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
   0   5   1   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0   0
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
   0   0   1   0   2   0   0   0   0   1   0   1   0   1   2   0   0
!
! lambda1 Lambda2     Ratio     Bkpos     Wdt     Cthm      muR    AsyLim    Rpolarz ->Patt# 1
 1.540560 1.544390  0.5000    90.000 12.0000  0.8009   0.0000    80.00     0.0000
K+1  C+4  CA+2
O-2  F-1
!
P -6 m 2                  <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
K    K+1      0.00000   0.00000   0.00000  0.0        0.08333   0   0   0    1
                 0.00      0.00      0.00   161.00      0.00
C1   C        0.66667   0.33333   0.50000  0.0        0.08333   0   0   0    2
                 0.00      0.00      0.00   171.00      0.00
Ca   Ca+2     0.33333   0.66667   0.50000  0.0        0.08333   0   0   0    3
                 0.00      0.00      0.00   201.00      0.00
O1   O-1      0.80000   0.20000   0.50000  0.0        0.25000   0   0   0    4
                151.00   -151.00      0.00   181.00      0.00
F    F-1      0.33333   0.66667   0.00000  0.0        0.08333   0   0   0    5
                 0.00      0.00      0.00   191.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 1.00000E-01    0.10000    0.00000    0.00000    0.00000    0.00000         0
   21.00000     61.000       0.000      0.000      0.000      0.000
!       U          V          W          X          Y        GauSiz     LorSiz Size-Model
   0.100000  -0.100000   0.200000   0.000000   0.000000   0.000000   0.000000     0
     71.000     81.000     91.000      0.000      0.000      0.000      0.000
!      a          b          c        alpha      beta       gamma      #Cell Info
   5.100000   5.10000    4.500000  90.000000  90.000000 120.000000
```

Preferred orientations (I)

Nor=0,

Rietveld-Toraya Model

$$P_H = G_2 + (1 - G_2)\exp(G_1 \alpha_H^2)$$

G_1 and G_2 are refinable parameters

α_H is the acute angle between d^*_H and the normal to the crystallites (platy habit)

Note: preferred orientation vector Pr1,Pr2 and Pr3 is needed to specify *a priori* by users

Preferred orientations (II)

Nor=1

$$P_H = G_2 + (1 - G_2)\left[(G_1 \cos\alpha_H)^2 + \frac{\sin^2\alpha_H}{G_1}\right]^{-\frac{3}{2}}$$

G_1 and G_2 are refinable parameters

G_1<1, platy habit,

G_1=1, no preferred orientation

G1>1 Needle-like habit

```
! Current global Chi2 (Bragg contrib.) =      17.11
! Files => DAT-file: kca1    PCR-file: kca1
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
   0   5   1   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
   0   0   1   0   2   0   0   0   0   1   0   1   0   1   2   0   0
!


!-------------------------------------------
 K/Ca/F/O/C
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0        158.170    0   5   1
!
!Jvi Jdi Hel Sol Mom Ter  Brind    RMua     RMub     RMuc    Jtyp  Nsp_Ref Ph_Shift
   0   3   0   0   0   0  1.0000  0.0000   0.0000   0.0000     0       0       0
!
! Max_dst(dist) (angles)  Bond-Valence Calc.
      3.5000       0.0000        BVS
!  N_cations    N_anions      Tolerance(%) / Name or cations/ and Anions
       3            2              200.00

!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 1.00000E-01    0.10000    0.00000    0.00000    0.00000    0.00000        0
    21.00000     61.000      0.000      0.000      0.000      0.000
!       U           V           W           X           Y         GauSiz    LorSiz Size-Model
   0.100000   -0.100000    0.200000    0.000000    0.000000    0.000000    0.000000     0
     71.000      81.000      91.000       0.000       0.000       0.000       0.000
!      a           b           c         alpha       beta        gamma       #Cell Info
   5.100000    5.10000     4.500000   90.000000   90.000000  120.000000
  101.00000   101.00000   111.00000     0.00000     0.00000   101.00000
!  Pref1     Pref2       Asy1       Asy2       Asy3       Asy4
  0.00000   0.00000    0.00000    0.00000    0.00000    0.00000
    121.00      0.00     131.00     141.00       0.00       0.00
```

Systematic line-shift

Bragg-Brentano Geometry

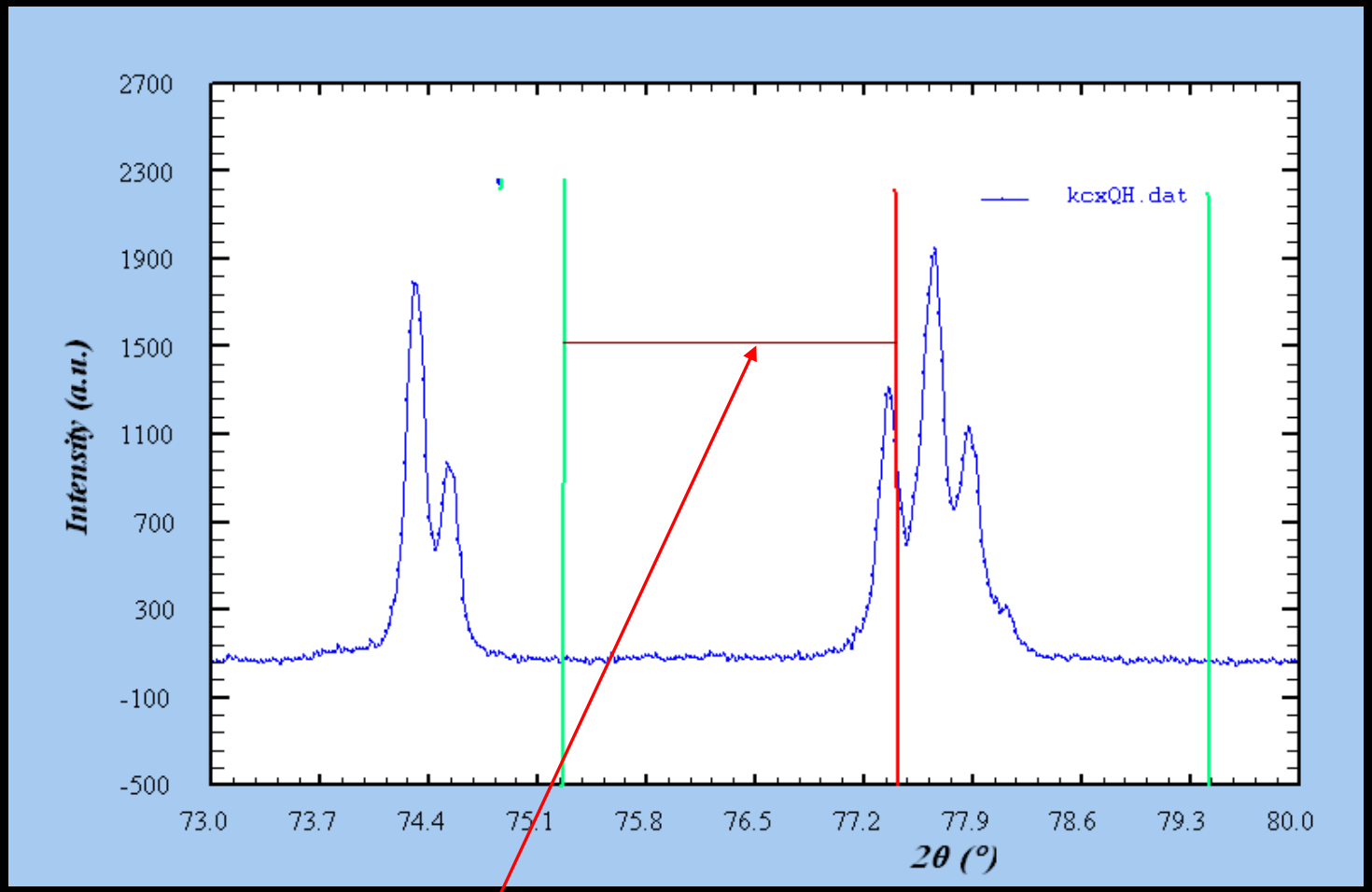
- Specimen displacement

$$\Delta 2\theta = \frac{-2s}{R}\cos\theta$$

- Specimen Transparency

$$\Delta 2\theta = \frac{1}{2\mu R}\sin 2\theta$$

μ: the linear absorption coefficient of the sample



WDT FWHM

WDT>5,
preferably ≥10

Monochromator polarization correct

$$LP = \frac{1 + \cos^2 2\alpha \cos^2 2\theta}{\sin^2 \theta \cos \theta}$$

α Incident angle to a monochromator

CTHM=$\cos^2 2\alpha$=0.8009 for a graphite monochromator, CuKα

Asymmetry correction for profiles

$$A_s(z) = 1 + \frac{P_1 F_a(z) + P_2 F_b(z)}{\tanh \theta_H} + \frac{P_3 F_a(z) + P_4 F_b(z)}{\tanh 2\theta_H}$$

$$z = \frac{2\theta_i - 2\theta_H - S_{shf}}{FWHM}$$

P_1, P_2, P_3, and P_4 are parameters to be refined

AsymLim: peaks below this 2θ angle limit are corrected for asymmetry

```
! Files => DAT-file: kca1,  PCR-file: kca1
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
  0   5   1   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
  0   0   1   0   2   0   0   0   0   1   0   1   0   1   2   0   0
!
! lambda1 Lambda2    Ratio     Bkpos     Wdt     Cthm      muR    AsyLim   Rpolarz ->Patt# 1
1.540560 1.544390  0.5000    90.000 12.0000  0.8009  0.0000    80.00    0.0000
!
!NCY  Eps  R_at  R_an  R_pr  R_gl     Thmin       Step        Thmax     PSD    Sent0
10  0.01  0.50  0.50  0.50  0.50     10.0000   0.020000    135.0000   0.000   0.000
!
! Excluded regions (LowT  HighT) for Pattern#  1
       0.02        9.98
     135.02      180.00
!
!
     24    !Number of refined parameters
!
!  Zero     Code    SyCos     Code   SySin     Code  Lambda     Code MORE ->Patt# 1
 0.00000  11.00  0.00000    0.00  0.00000 240.01 0.000000    0.00   0
!   Background coefficients/codes  for Pattern#  1
  0.000       0.000        0.00        0.00        0.00      -  00.0
 0.000     0.000
!       U         V         W         X         Y        GauSiz   LorSiz Size-Model
  0.100000  -0.100000  0.200000  0.000000  0.000000  0.000000  0.000000    0
    71.000    81.000    91.000     0.000     0.000     0.000     0.000
!      a         b         c       alpha      beta       gamma      #Cell Info
  5.100000   5.10000   4.500000 90.000000 90.000000 120.000000
 101.00000 101.00000 111.00000   0.00000   0.00000 101.00000
!  Pref1    Pref2      Asy1      Asy2      Asy3      Asy4
 0.00000  0.00000  0.00000  0.00000  0.00000  0.00000
  121.00     0.00   131.00   141.00     0.00     0.00
```

Occupancy

$$Occ = chemOcc \cdot \frac{m}{M}$$

m is the site multiplicity, M is the multiplicity of the general site for a given space group.

For example, KCaFCO3, P-6m2(187)

K+1 occupies 1(a) site; O-2 3(k) site; general site 12(o), Occ(K+)=1/12=0.08333, Occ(O-2)=0.25, both atoms' chemical occupancy=1.0

Agreement Factors (I)

$$R_p = 100 \frac{\sum_i^n \left| y_i - y_{ci} \right|}{\sum_i^n y_i}$$

Profile Factor

$$R_{wp} = 100 \left[\frac{\sum_i^n w_i \left| y_i - y_{ci} \right|^2}{\sum_i^n w_i y_i} \right]^{\frac{1}{2}}$$

Weighted Profile Factor

$$R_{\exp} = 100 \left[\frac{n-p}{\sum_i^n w_i y_i^2} \right]^{\frac{1}{2}}$$

Expected
Weighted
Profile Factor

Agreement Factors (II)

$$S = \frac{R_{wp}}{R_{\exp}}$$

Goodness of fit indicator

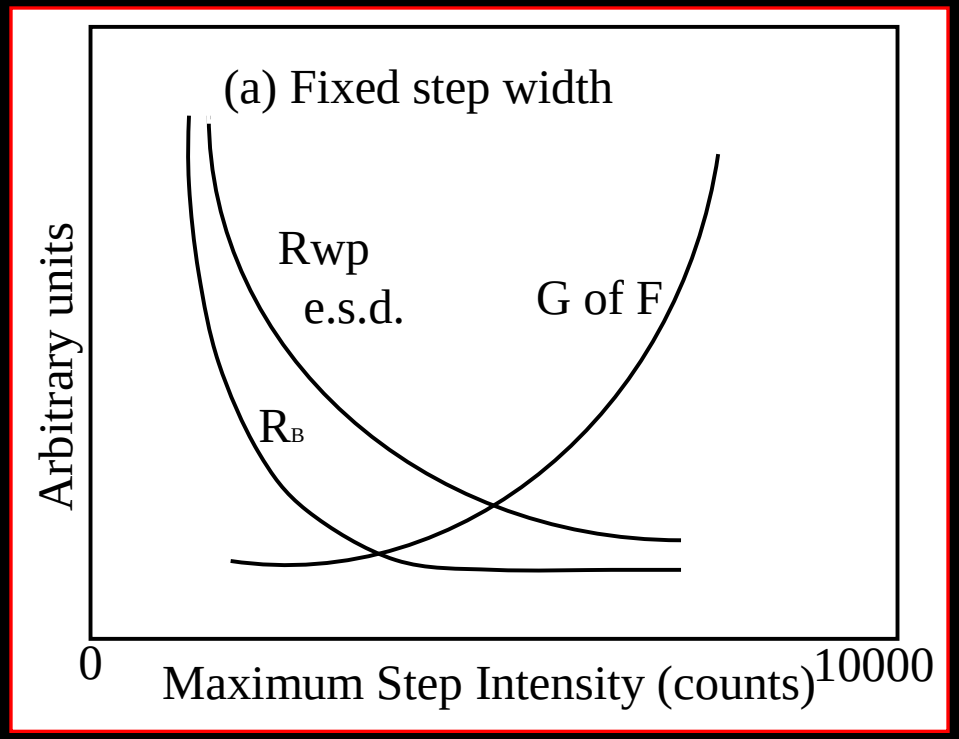
$$R_B = 100 \frac{\sum_h \left| {}'I_{obs,h}{}' - I_{calc,h} \right|}{\sum_h \left| {}'I_{obs,h}{}' \right|}$$

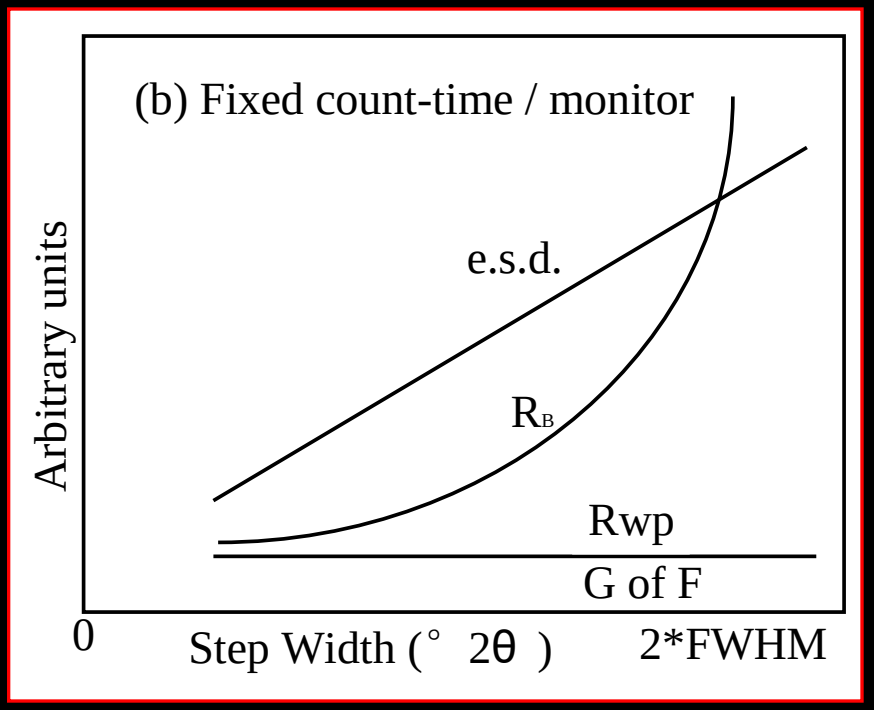
Bragg Factor

$$R_F = 100 \frac{\sum_h \left| {}'F_{obs,h}{}' - F_{calc,h} \right|}{\sum_h \left| {}'F_{obs,h}{}' \right|}$$

Crystallographic R_F factor

Variations of agreement factors and esd.





Hill & Madsen, Powder Diffraction(1987)

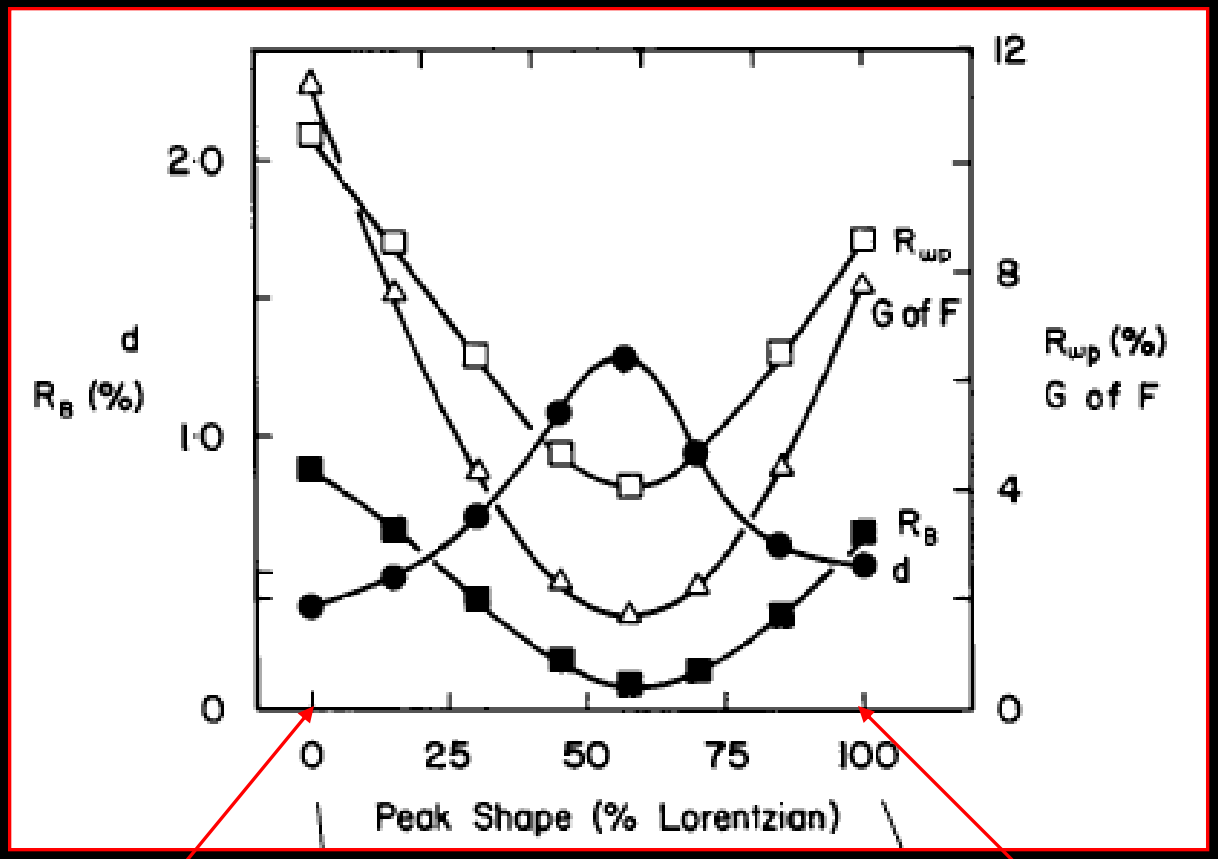
An estimation of S for an ideal refinement

take $\sigma_i = \sqrt{y_i}$

$$(y_i - y_{ci})^2 = \left[y_i - (y_i \pm \sqrt{y_i})\right]^2 = y_i$$

$$S = \left[\frac{\sum_i^N w_i (y_i - y_{ci})^2}{N - P}\right]^{\frac{1}{2}} = \left[\frac{\sum_i^N w_i y_i}{N - P}\right]^{\frac{1}{2}} = \left[\frac{N}{N - P}\right]^{\frac{1}{2}}$$

S≈1 since N>>P



Gaussian

Lorentzian

R.J. Hill and H.D. Flack, J.Appl. Cryst. 20 (1987) 356-361

Durbin-Watson statistic parameters

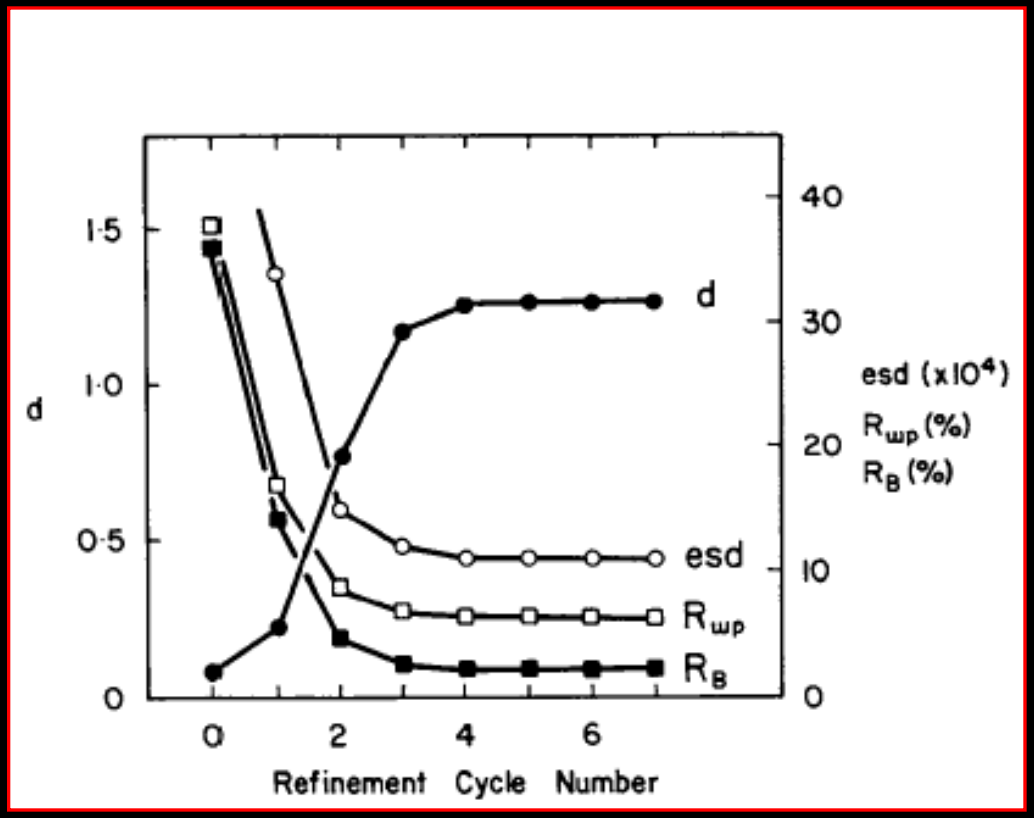
$$d = \frac{\sum_{2}^{N} [w_i (y_i - y_{ci}) - w_{i-1}(y_{i-1} - y_{ci-1})]^2}{\sum_{i}^{N} [w_i (y_i - y_{ci})]^2}$$

$$Q_D = 2\left[\frac{N-1}{N-P} - \frac{3.0901}{\sqrt{N+2}}\right]$$

$d < Q_D$: positive serial correlation

$Q_D < d < 4-Q_D$: no serial correlation

$d > 4-Q_D$: negative serial correlation



Variations of d, eds, R_{wp} and R_B vs cycles

R.J. Hill and H.D. Flack, J.Appl. Cryst. 20 (1987) 356-361

Comments on agreement factors

- R_F and R_B are more indicative of structural model fits
- R_p and R_{exp} are more indicative of overall profile fits

  R_B , R_p and R_{exp} are not good indices for the refinements of different patterns
- S should be as close as 1

  is a more sensitive index over R_F ,R_B , R_p and R_{exp}
- At least Rp, Rwp and Rexp should be given when submitting a paper to a journal

Quantitative Phase Analysis

$$W_j = \frac{S_j Z_j M_j V_j / t_j}{\sum_i^N S_i Z_i M_i V_i / t_i}$$

where, W_j is the weight fraction for the jth phase;

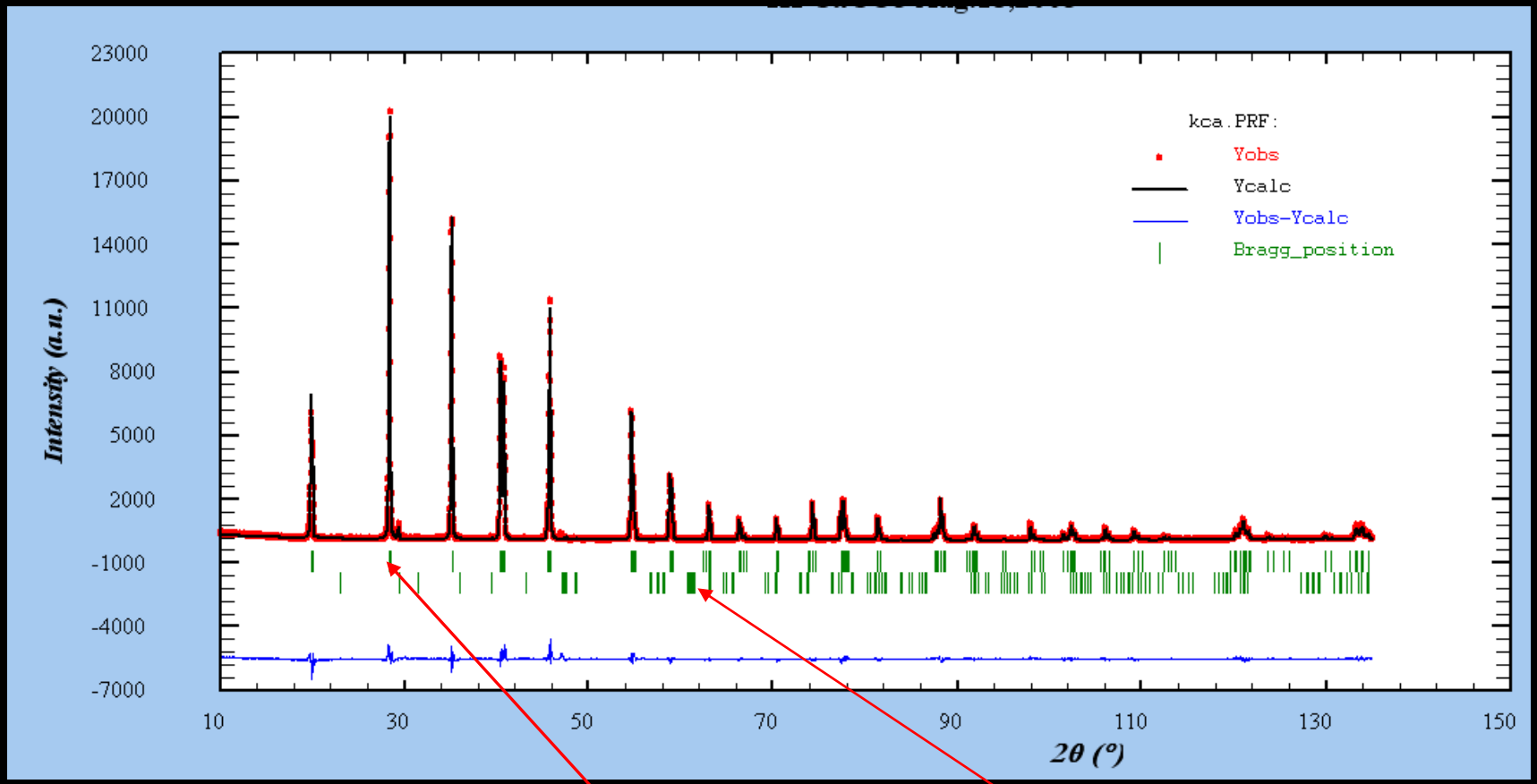
S_j is scale factor for the jth the phase;

Z_j is the number formula units per cell for the jth phase;

M_j is the mass of the formula unit;

V_j is the unit cell volume;

t_j Brindley coefficient that comes into effect when the linear absorption coefficients of phases in powder differ a lot to each other.



$KCaFCO_3$

$CaCO_3$

Multiphase Rietveld Analysis

```
COMM    KFCaCO3 Aug.15,2003
! Current global Chi2 (Bragg contrib.) =       4.520
! Files => DAT-file: kca,  PCR-file: kca
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
   0   5   2   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0   0
...
!   Background coefficients/codes  for Pattern#  1
  56.357      -52.995       282.29       567.97      -802.96      -1594.7
       0.000        0.000        0.000        0.000        0.000        0.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:    1  ==> Current R_Bragg for Pattern#  1:      5.10
!-------------------------------------------------------------------------------
 K/Ca/F/O/C
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0        158.170    0   5   0
!
P -6 m 2                  <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
K     K+1     0.00000   0.00000   0.00000   1.01101   0.08333    0   0   0     0
                0.00      0.00      0.00    161.00      0.00
C1    C       0.66667   0.33333   0.50000   1.41570   0.08333    0   0   0     0
                0.00      0.00      0.00    171.00      0.00
...
  0.99619   0.00000   0.11323   0.06140   0.00000   0.00000
  121.00      0.00    131.00    141.00      0.00      0.00
!-------------------------------------------------------------------------------
!  Data for PHASE number:    2  ==> Current R_Bragg for Pattern#  1:     35.91
!-------------------------------------------------------------------------------
Ca/O/C
C1    C       0.00000   0.00000   0.25000   0.57456   0.16667    0   0   0     0
                0.00      0.00      0.00      0.00      0.00
O1    O-1     0.24530   0.00000   0.25000   0.89400   0.50000    0   0   0     0
                0.00      0.00      0.00      0.00      0.00
```

Result in *.out file

```
   65    1       2    1   16     12   0.840773 -0.105816      119.199          1.4          1.1        0.359
   66    1       1    1   18     12   0.867166 -0.105816      120.696          2.0          2.2        0.144
   67    1       1    3   13     12   0.872609 -0.105816      120.997          0.0          0.0        0.000
   68    1       5    0    2      6   1.000124 -0.105816      127.295          0.1          0.0        0.129
   69    1       3    2   10     12   1.015829 -0.105816      127.984          1.1          0.0        0.665
   70    1       1    2   17     12   1.027419 -0.105816      128.482          0.4          0.0        0.158
   71    1       3    1   14     12   1.032087 -0.105816      128.680          0.5          0.1        0.198
   72    1       0    5    4      6   1.087037 -0.105816      130.905          1.1          1.3        0.344
   73    1       1    4    9     12   1.107829 -0.105816      131.699          0.0          0.0        0.001
   74    1       4    1    9     12   1.107829 -0.105816      131.699          0.0          0.0        0.001
   75    1       2    2   15     12   1.137859 -0.105816      132.805          0.0          0.0        0.004
   76    1       0    1   20      6   1.167770 -0.105816      133.859          2.6          2.8        0.173
   77    1       2    3   11     12   1.186786 -0.105816      134.506          0.2          0.2        0.020

     ------------------------------------------------------
     BRAGG R-Factors and weight fractions for Pattern #  1
     ------------------------------------------------------

=> Phase:  1
=> Bragg R-factor:  5.10        Vol:  100.395( 0.000)  Fract(%):   98.60( 0.05)
=> Rf-factor= 3.83              ATZ:         158.170   Brindley:  1.0000

=> Phase:  2
=> Bragg R-factor:  35.9        Vol:  367.819( 0.145)  Fract(%):    1.40( 0.05)
=> Rf-factor= 35.3              ATZ:         600.550   Brindley:  1.0000

     ------------------------------------------------------------------
     SYMBOLIC NAMES AND FINAL VALUES AND SIGMA OF REFINED PARAMETERS:
     ------------------------------------------------------------------

      ->  Parameter number    1 :       Scale_ph2_pat1    0.36961414E-04( +/-    0.14040444E-05
      ->  Parameter number    2 :       EtaPV_ph2_pat1   -0.10581600     ( +/-    0.91128334E-01
      ->  Parameter number    3 :      Cell_A_ph2_pat1     4.9883246     ( +/-    0.24863641E-04
      ->  Parameter number    4 :      Cell_C_ph2_pat1     17.068441     ( +/-    0.15078003E-05
```

No absorption correction is applied

To obtain a satisfactory quantitative phase analysis based on the Rietveld method, we should be cautioned:

- Sample should be carefully prepared: powder is homogeneous in compositions and have a sufficient number of grains with random orientations;
- Structures of phases are well known;
- Absorption correction is applied whenever the phases differ a lot in their linear absorption coefficients. The Brindley coefficients can be consulted in the Fullprof Manual.

Calculation of bond length, angle and bond valence sum

```
      26     !Number of refined parameters
!
!  Zero     Code     Sycos     Code    Sysin     Code  Lambda      Code MORE ->Patt# 1
 -0.01390  11.00  0.00000    0.00  0.00000    0.00 0.000000     0.00    0
!    Background coefficients/codes  for Pattern#  1
  56.185      -52.498       281.17       575.63      -805.86      -1612.2
      31.000       41.000       51.000      211.000      221.000      231.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:      5.74
!-------------------------------------------------------------------------------
 K/Ca/F/O/C
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 1.0 0.0 0.0   0   0   0   0   0        158.170   0   5    1
!
!Jvi Jdi Jel Sol Mom Ter   Brind    RMua     RMub     RMuc    Jtyp  Nsp_Ref
   0   3   0   0   0   0  1.0000  0.0000   0.0000   0.0000    0    0
!
 Max_dst(dist) (angles)  Bond-Valence Calc.
      3.5000    130.0000         BVS
!  N_cations   N_anions      Tolerance(%) / Name or cations/ and Anions
       3            2               200.00
K+1  C+4  CA+2
O-2  F-1
!
P -6 m 2                    <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
K    K+1      0.00000   0.00000   0.00000   0.99419   0.08333    0   0   0     1
```

```
Orig. extr. p.equiv.                                          tx   ty   tz        XYZ of Ligand Atom


   2   3   1   (C1  )-(Ca  ):    2.9450  (   0)             0   -1    0       0.33333 -0.33333  0.50000
   2   3   1   (C1  )-(Ca  ):    2.9450  (   0)             0    0    0       0.33333  0.66667  0.50000
   2   3   1   (C1  )-(Ca  ):    2.9450  (   0)             1    0    0       1.33333  0.66667  0.50000
   2   4   1   (C1  )-(O1  ):    1.2840  (  14)             0    0    0       0.81200  0.18800  0.50000
   2   4   2   (C1  )-(O1  ):    1.2840  (  12)             1    0    0       0.81200  0.62400  0.50000
   2   4   3   (C1  )-(O1  ):    1.2840  (  14)             1    1    0       0.37600  0.18800  0.50000


   Angles around atom: C1

   (O1  )-(C1  )-(O1  ): 120.000  (  162)     d12= 1.284( 1)  d23= 1.284( 1)  d13= 2.224( 2)
   (C1  )-(O1  )-(O1  ):  30.000  (   79)
   (C1  )-(O1  )-(O1  ):  30.000  (   85)
        (O1  ) :  0.8120  0.1880  0.5000  (O1  ) :  0.8120  0.6240  0.5000
   (O1  )-(C1  )-(O1  ): 120.000  (  189)     d12= 1.284( 1)  d23= 1.284( 1)  d13= 2.224( 2)
   (C1  )-(O1  )-(O1  ):  30.000  (   93)
   (C1  )-(O1  )-(O1  ):  30.000  (   93)
        (O1  ) :  0.8120  0.1880  0.5000  (O1  ) :  0.3760  0.1880  0.5000
   (O1  )-(C1  )-(O1  ): 120.000  (  162)     d12= 1.284( 1)  d23= 1.284( 1)  d13= 2.224( 2)
   (C1  )-(O1  )-(O1  ):  30.000  (   85)
   (C1  )-(O1  )-(O1  ):  30.000  (   79)
        (O1  ) :  0.8120  0.6240  0.5000  (O1  ) :  0.3760  0.1880  0.5000
```

ESDs

```
   ------------------------------------------------------------------
=> Bond-valence and coordination of atom: C1    occupancy: 1.000(  0)
   ------------------------------------------------------------------

(C1  )-(O1  ) :  1.2840(  14)    1.332(  5)
(C1  )-(O1  ) :  1.2840(  12)    1.332(  4)
(C1  )-(O1  ) :  1.2840(  14)    1.332(  5)

Coordination number:     3       Eff.Coor. number:  3.00  for atom: C1
Average distance  :  1.2840(   8)  Distortion:    -0.001 xE-04
Predicted distance:  1.2836        Single bond-valence S= 1.333
                                     Valence:    4.000
                                        Sums:    3.995(  8)
Deviation from the Valence Sum Rule (r1,%dev):  -0.005   0.121
(r1=Sumj(sij)-Vi, %dev=100abs(r1)/Vi)
Deviation from the Equal Valence Rule     (r2):   0.000
(r2=<sij-<sij>>rms)


   ------------------------------------------------------------------
=> Bond-valence and coordination of atom: Ca    occupancy: 1.000(  0)
   ------------------------------------------------------------------
```

Bond valence sum is a good indicator of the structural validity. For details, see

I.D.Brown, Acta Crystallogr. B48, 141(1992)

Forced termination when shifts<EPS*esd

Recommended EPS=0.1

$$\alpha_i^{n+1} = \alpha_i^n + \delta_i \cdot RELAX \cdot CC$$

R_at: Atomic parameters, including atomic coordinates and etc.

R_an: anisotropic parameters

R_pr: profile parameters, lattice parameters, preferred orientations and the etc;

R_gl: global parameters, such as zero shift, backgrounds and the etc.

```
COMM   KFCaCO3 Aug.31,2003
! Current global Chi2 (Bragg contrib.) =      6.114
! Files => DAT-file: kca1,  PCR-file: kca1
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
   0   5   1   0   2   0   1   1   0   0   1   0   0   0   0   0   0   0
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
   1   0   1   0   1   0   0   0   0   1   0   1   0   1   2   0   0
!
! lambda1 Lambda2     Ratio     Bkpos     Wdt     Cthm      muR    AsyLim    Rpolarz ->Patt# 1
 1.540560 1.544390  0.5000    90.000 15.0000  0.8009  0.0000    80.00     0.0000
!
!NCY  Eps  R_at  R_an  R_pr  R_gl     Thmin        Step        Thmax     PSD     Sent0
 10  0.01  0.50  0.50  0.50  0.50     10.0000    0.020000    135.0000    0.000    0.000
!
! Excluded regions (LowT  HighT) for Pattern#  1
        0.02         9.98
      135.02       180.00
!
!
      26     !Number of refined parameters
!
!  Zero     Code     Sycos     Code    Sysin     Code  Lambda      Code MORE ->Patt# 1
-0.01390  11.00  0.00000    0.00  0.00000    0.00 0.000000     0.00    0
!   Background coefficients/codes  for Pattern#  1
  56.185      -52.498       281.17       575.63      -805.86      -1612.2
      31.000       41.000       51.000      211.000      221.000      231.000
!-------------------------------------------------------------------------------
```

2 Excluded 2θ regions

Suggested turn-on sequences for the parameters

1. Scale factors
2. Zero shift
3. Background
4. FWHM
5. Shape1, X, Y,…
6. Lattice parameters (if accurate to some extent otherwise do it before refining FWHM)
7. Atomic coordinates
8. Temperature factors, occupancies
9. Preferred orientation, GauSiz, LorSiz…

Some factors affecting refinement results

- Low instrument's resolution. RS=0.1-0.2mm
- Too low counts. Strongest counts >10000
- Too less sample. Sample should fully cover the sample holder window;
- Overflow in low angle region;
- Too less angle region $2\theta>120^{\circ}$;
- Too large EPS that leads to false minimum;
- Improper profile function;
- Too less WDT values .

Error messages (I)

-‘Hole in Matrix’ : the number of parameters to be refined NPR larger than the number of codewords

For example, you set NPR= 12, while one codeword 80.5 is missing or 101.0 is mistyped as 11.0

- ‘Negative FWHM’: $H_G^2 < 0$, meaningless! Increasing the negative U,V, or W while set smaller Relax values in the ensuing the refinements

$$H_G^{\ 2} = U\tan^2\theta + V\tan\theta + W + \frac{I_g}{\cos^2\theta}$$

‘No scattering factor’ : atom identifier ‘TYP’ is not recognizable by Fullprof.

For example, Ca+2 is accepted while Ca2+ is not accepted.

-‘Too many reflections’: For a given point, there are too many reflections contributing to the intensity that are beyond the software’s capacity. Usually this results from the false FWHM

- ‘Invalid integer’ or ‘Invalid real’: Examine the format of parameters

Note: The software does not always give the correct lines where errors occur. Look into the nearby lines!

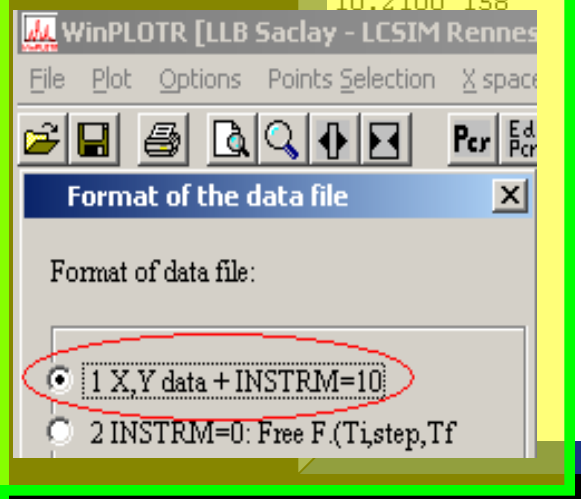
MAC MXP18A-HF

```
numbr     1
offst      1024
nexdt     57024
files     1
lastp ?
nextp
mdate 92.10. 9 14
gonio        1
atach     100
x_ray Cu
wavel    154050
voltg      50
curnt     200
opert
sampl CAZB01-1
comnt
mode1     1
mode2     1
mode3     3
axisf     1
start    100100
stopa    800000
speed     80000
stepw     100
. . . . . .
100200   180
100300   176
100400   170
100500   161
100600   149
100700   136
100800   130
100900   131
101000   137
101100   137
101200   136
```

数据


```
10.00 0.01 80.00 起始角度，步长，终止角度
10.0100 181
10.0200 180
10.0300 176
10.0400 170
10.0500 161
10.0600 149
10.0700 136
10.0800 130
10.0900 131
10.1000 137
10.1100 137
10.1200 136
10.1300 138
10.1400 141
10.1500 142
10.1600 147
10.1700 14
10.1800 152
10.1 00 154
10.2000 15
10.2100 158
```

数据



理学 DMAX 2000

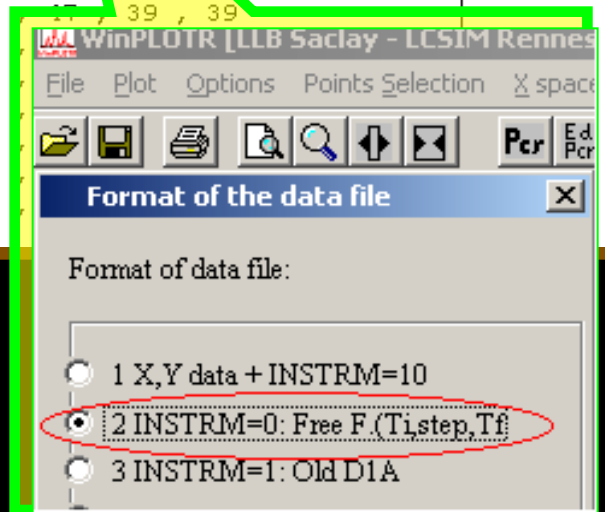
```
*TYPE           =  Raw
*CLASS          =  Standard measurement
*SAMPLE         =  CA2
*COMMENT        =
*FNAME          =
*DATE           =  2005-1-18  8:44:40

*GROUP_COUNT    =  1
*GONIO          =  RINT2000 Wide angle goniometer, 185
*ATTACHMENT     =  Standard Sample Holder
*ASC            =  1, 0, 1, 0
*FILTER         =  Not installed
*C_MONOCHRO     =  Automatic monochromator, 3.3539
*SLIT_NAME      =  0, Divergence slit
*SLIT_NAME      =  1, Scattering slit
*SLIT_NAME      =  2, Receiving slit
*COUNTER        =  Scintillation counter, 0
*POS_FORMAT     =  0
*SCAN_AXIS      =  2 Theta / Theta
*MEAS_MODE      =  CONTINUOUS
*TARGET         =  29
*XRAY_CHAR      =  K-ALPHA1
*WAVE_LENGTH1   =  1.540562
*WAVE_LENGTH2   =
. . . . .
*START          =  10
*STOP           =  50
*STEP           =  0.02
. . . . .
27 , 54 , 51 , 60
41 , 45 , 51 , 45
46 , 52 , 47 , 41
45 , 45 , 49 , 63
48 , 50 , 53 , 35
54 , 37 , 44 , 55
54 , 63 , 69 , 61
```

样品信息及测试条件

数据

```
10.00 0.02 50.00   起始角度，步长，终止角度
27 , 54 , 51 , 60
41 , 45 , 51 , 45
46 , 52 , 47 , 41
45 , 45 , 49 , 63
48 , 50 , 53 , 35
54 , 37 , 44 , 55
54 , 63 , 69 , 61
72 , 71 , 90 , 93
137 , 117 , 87 , 88
75 , 78 , 79 , 82
98 , 110 , 120 , 131
102 , 122 , 142 , 136
169 , 162 , 182 , 214
256 , 264 , 270 , 272
332 , 503 , 551 , 703
759 , 801 , 1025 , 1161
1436 , 1659 , 2010 , 2090
1924 , 1537 , 983 , 488
212 , 124 , 81 , 58
60 , 50 , 54 , 68
56 , 46 , 58 , 43
35 , 49 , 49 , 54
53 , 44 , 53 , 35
46 , 37 , 50
39 , 47 , 46 , 40
46 , 49 , 42 ,
50 , 39 , 39
50
42
49
42
45
42
```

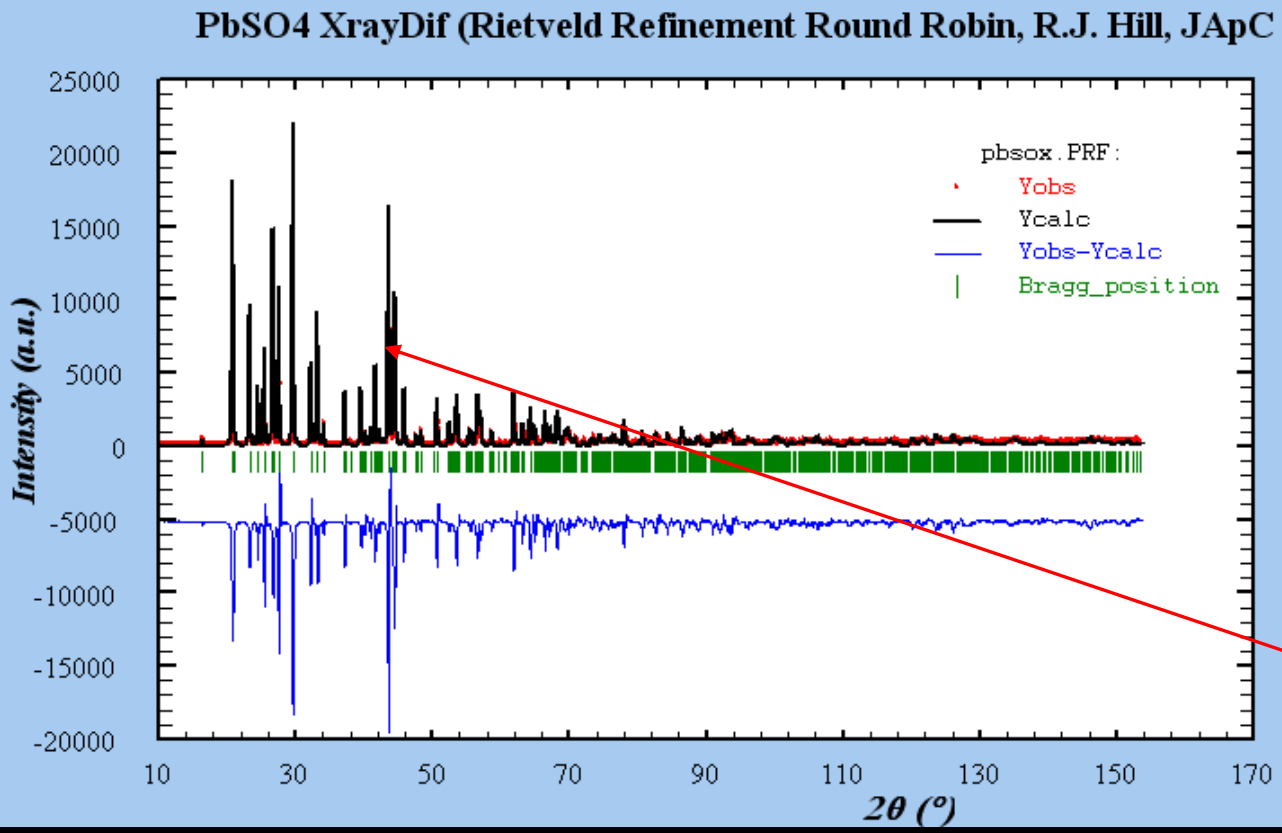
数据

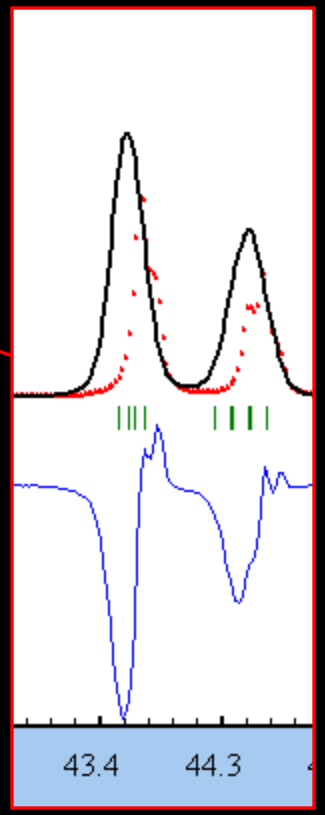


```
  4    0.10   1.00   1.00   1.00   1.00      10.0000    0.025000    160.0000    0.000    0.000
!
! Excluded regions (LowT  HighT) for Pattern#  1
        0.00        10.00
      154.00       180.00
!
!
      1     !Number of refined parameters
!
!  Zero     Code     SyCos     Code    SySin     Code  Lambda      Code MORE ->Patt# 1
  0.00   21.00  0.00000    0.00  0.00000    0.00 0.000000     0.00    0
!   Background coefficients/codes  for Pattern#  1
   00.00        0.000        0.000        0.000       00.00         0.000
      31.000       41.000       51.000       61.000       71.000       81.000
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030    0   5    0
!
P n m a                  <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    PB       0.19007   0.25000   0.17000   1.00000   0.50000    0   0   0     0  #conn S O O 2
                171.00      0.00    181.00    281.00      0.00
S     S        0.06000   0.25000   0.70000   1.00000   0.50000    0   0   0     0
                191.00      0.00    201.00    291.00      0.00
O1    O        0.90000   0.25000   0.60000   0.50000   0.50000    0   0   0     0
                211.00      0.00    221.00    301.00      0.00
O2    O        0.20000   0.25000   0.50000   0.50000   0.50000    0   0   0     0
                231.00      0.00    241.00    311.00      0.00
O3    O        0.08000   0.03000   0.80000   0.50000   1.00000    0   0   0     0
                251.00    261.00    271.00    321.00      0.00
!------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov       Str1       Str2       Str3   Strain-Model
 0.500000-02    0.00000    0.00000    0.00000    0.00000    0.00000        0
   11.00000       0.000      0.000      0.000      0.000      0.000
!       U          V          W          X          Y        GauSiz   LorSiz Size-Model
```

Refine scale factor S

File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tool
Help

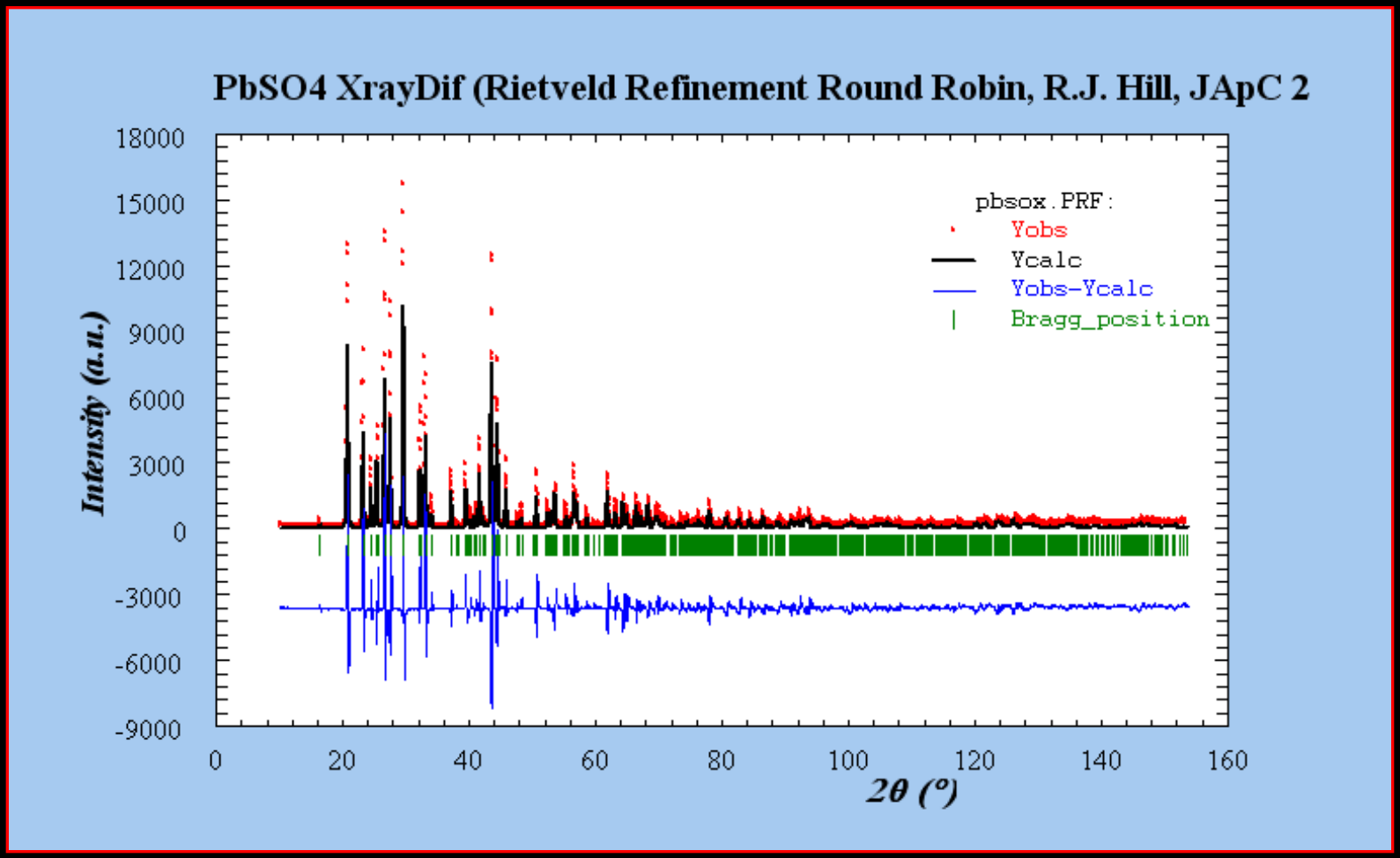




R_{wp}=962 → 138, S=0.5E-2 → 0.64E-3

```
      2     !Number of refined parameters
!
!  Zero     Code    SyCos     Code   SySin     Code  Lambda     Code MORE ->Patt# 1
  0.00  21.00  0.00000    0.00  0.00000    0.00 0.000000    0.00    0
!    Background coefficients/codes  for Pattern#  1
   00.00       0.000       0.000       0.000      00.00       0.000
      31.000      41.000      51.000      61.000      71.000      81.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     4.48
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030   0   5   0
!
P n m a                  <--Space group symbol
!Atom Typ           X           Y           Z         Biso       Occ      In Fin N_t Spc /Codes
Pb    PB       0.19007   0.25000   0.17000   1.00000   0.50000   0   0   0    0  #conn S O 0 2
                171.00      0.00    181.00    281.00      0.00
S     S        0.06000   0.25000   0.70000   1.00000   0.50000   0   0   0    0
                191.00      0.00    201.00    291.00      0.00
O1    O        0.90000   0.25000   0.60000   0.50000   0.50000   0   0   0    0
                211.00      0.00    221.00    301.00      0.00
O2    O        0.20000   0.25000   0.50000   0.50000   0.50000   0   0   0    0
                231.00      0.00    241.00    311.00      0.00
O3    O        0.08000   0.03000   0.80000   0.50000   1.00000   0   0   0    0
                251.00    251.00    271.00    321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.50000E-02     0.50000    0.00000    0.00000    0.00000    0.00000        0
    11.00000       0.000      0.000      0.000      0.000      0.000
!       U           V           W           X           Y        GauSiz    LorSiz Size-Model
   0.050004   -0.040000   0.050000   0.000000   0.000000   0.000000   0.000000     0
```

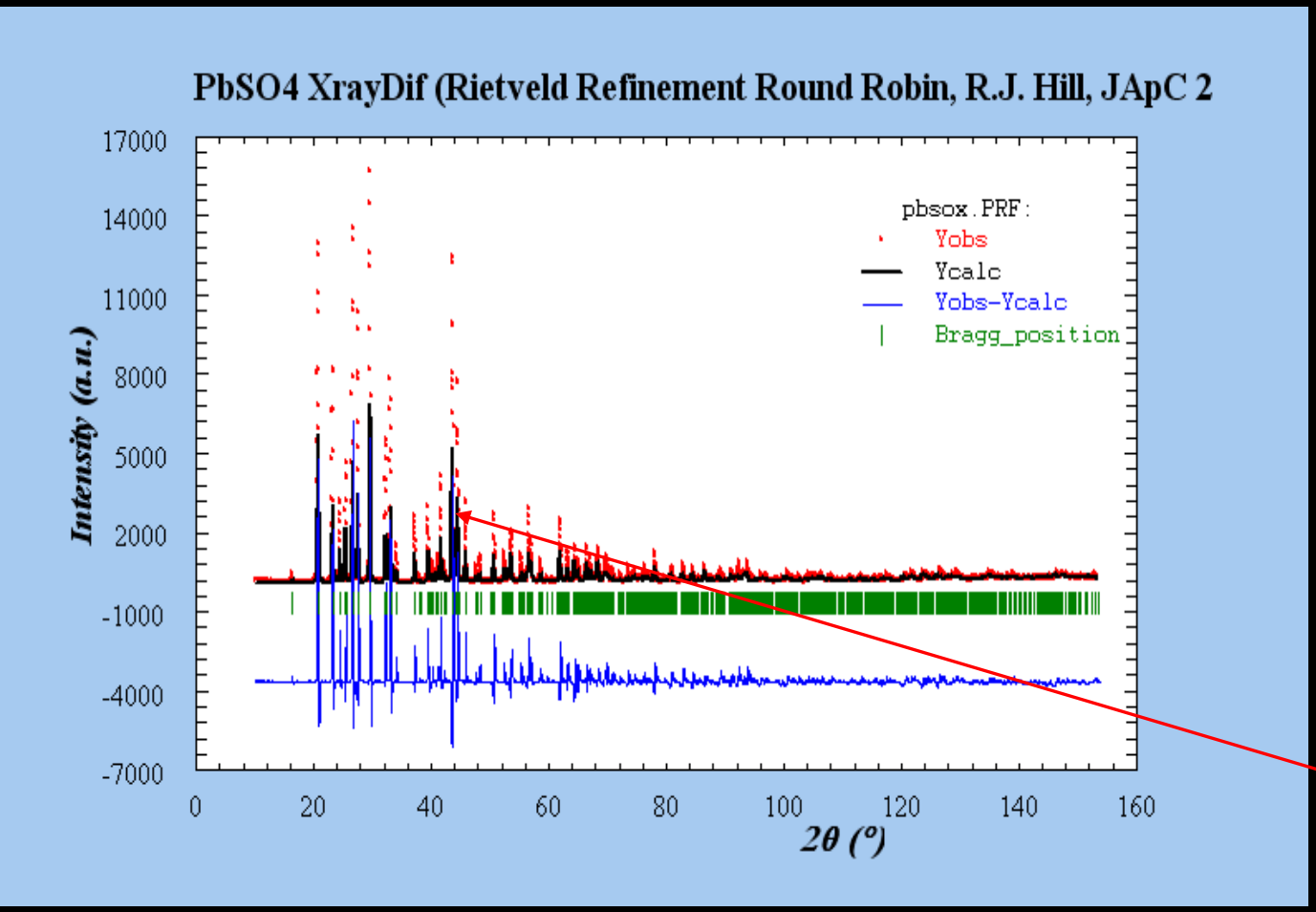
Refine zero point along with S

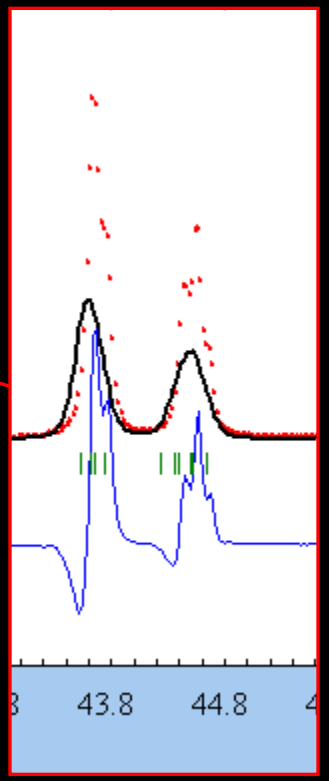


Rwp=62.8 ZP≈0.05

```
      154.00      180.00
!
!
       8     !Number of refined parameters
!
!  Zero     Code     SyCos     Code    SySin     Code  Lambda      Code MORE ->Patt# 1
  0.05101  21.00  0.00000     0.00  0.00000    0.00 0.000000     0.00    0
!  Background coefficients/codes  for Pattern#  1
 0.0000       0.0000       0.0000       0.0000       0.0000       0.0000
      31.000       41.000       51.000       61.000       71.000       81.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:    37.36
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030    0   5   0
!
P n m a                  <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    PB       0.19007   0.25000   0.17000  1.00000   0.50000    0   0   0     0  #conn S O 0 2
                171.00      0.00    181.00   281.00      0.00
S     S        0.06000   0.25000   0.70000  1.00000   0.50000    0   0   0     0
                191.00      0.00    201.00   291.00      0.00
O1    O        0.90000   0.25000   0.60000  0.50000   0.50000    0   0   0     0
                211.00      0.00    221.00   301.00      0.00
O2    O        0.20000   0.25000   0.50000  0.50000   0.50000    0   0   0     0
                231.00      0.00    241.00   311.00      0.00
O3    O        0.08000   0.03000   0.80000  0.50000   1.00000    0   0   0     0
                251.00    261.00    271.00   321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.41623E-03    0.50000    0.00000    0.00000    0.00000    0.00000         0
```

Refine background along with S and ZP

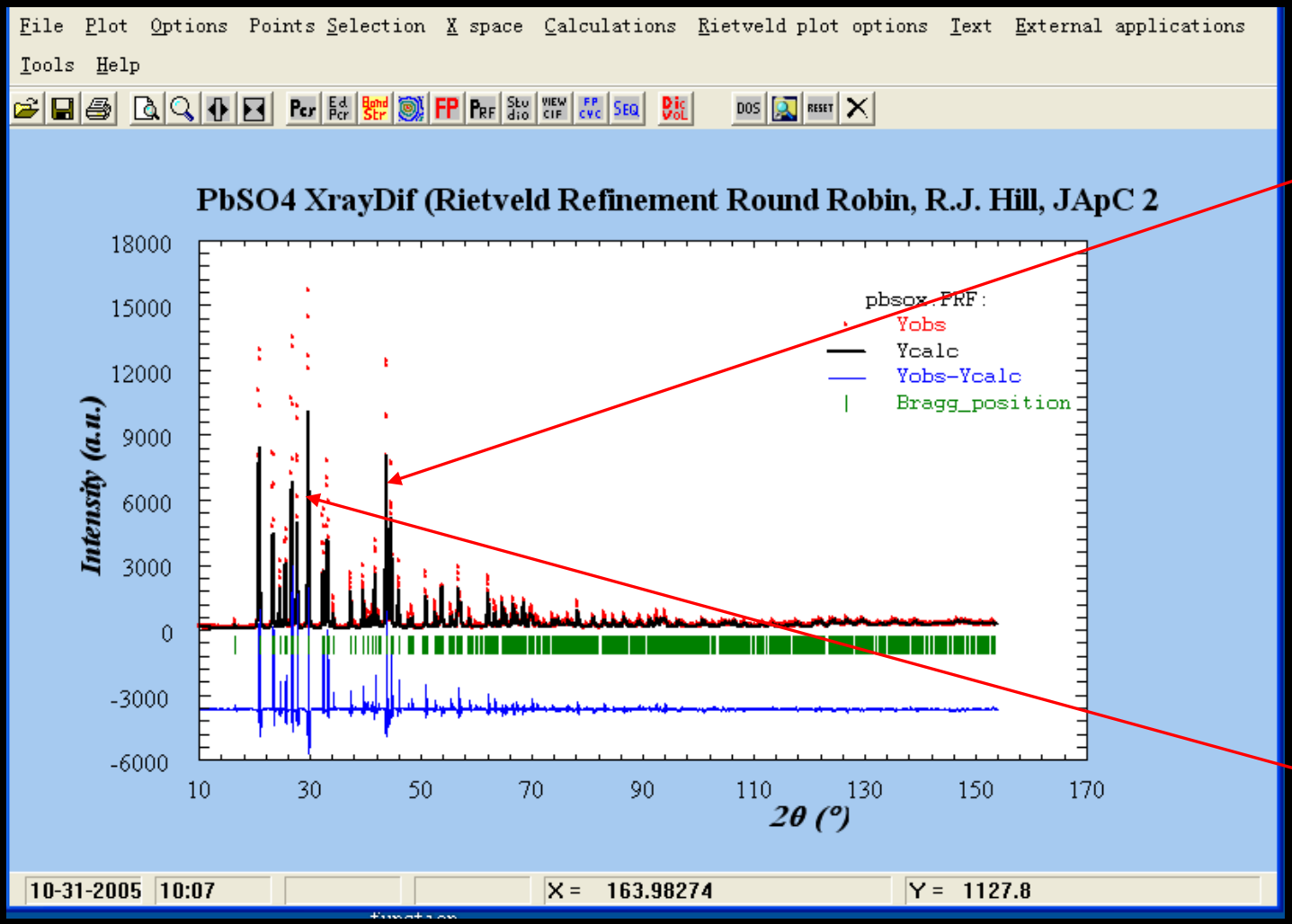

PbSO4 XrayDif (Rietveld Refinement Round Robin, R.J. Hill, JApC 2
pbsox.PRF:
Yobs
Ycalc
Yobs-Ycalc
Bragg_position
Intensity (a.u.)
17000
14000
11000
8000
5000
2000
-1000
-4000
-7000
0
20
40
60
80
100
120
140
160
2θ (°)

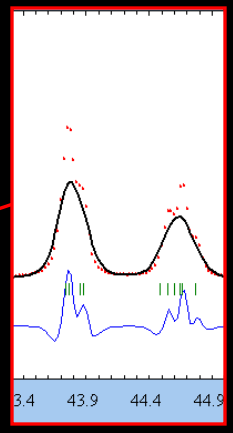

43.8
44.8

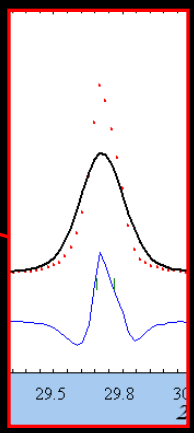
Rwp=32.9%

```
     11      !Number of refined parameters
!
!  Zero     Code     SyCos     Code    SySin     Code  Lambda      Code MORE ->Patt# 1
  0.05449  21.00  0.00000    0.00  0.00000    0.00 0.000000     0.00    0
!   Background coefficients/codes  for Pattern#  1
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030    0   5   0
!
P n m a                  <--Space group symbol
!Atom Typ       X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    PB      0.19007   0.25000   0.17000   1.00000   0.50000   0   0   0    0  #conn S O O 2
               171.00      0.00    181.00    281.00      0.00
S     S       0.06000   0.25000   0.70000   1.00000   0.50000   0   0   0    0
               191.00      0.00    201.00    291.00      0.00
O1    O       0.90000   0.25000   0.60000   0.50000   0.50000   0   0   0    0
               211.00      0.00    221.00    301.00      0.00
O2    O       0.20000   0.25000   0.50000   0.50000   0.50000   0   0   0    0
               231.00      0.00    241.00    311.00      0.00
O3    O       0.08000   0.03000   0.80000   0.50000   1.00000   0   0   0    0
               251.00    261.00    271.00    321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale        Shape1       Bov       Str1       Str2       Str3    Strain-Model
 0.28895E-03    0.50000   0.00000   0.00000    0.00000    0.00000        0
    11.00000      0.000     0.000     0.000      0.000      0.000
!       U          V          W          X          Y        GauSiz    LorSiz Size-Model
   0.050004  -0.040000   0.050000   0.000000   0.000000   0.000000   0.000000     0
    121.000    131.000    141.000    151.000      0.000      0.000      0.000
!       a          b          c        alpha      beta       gamma      #Cell Info
   8.500000   5.400000   7.000000   90.000000  90.000000  90.000000   # box -0.25 1.25 -0.1
   91.00000  101.00000  111.00000    0.00000    0.00000    0.00000
!  Pref1      Pref2      Asy1       Asy2       Asy3       Asy4
  0.00000   0.00000   0.00000   0.00000   0.00004   0.00000
     0.00      0.00    161.00    331.00    341.00      0.00
```

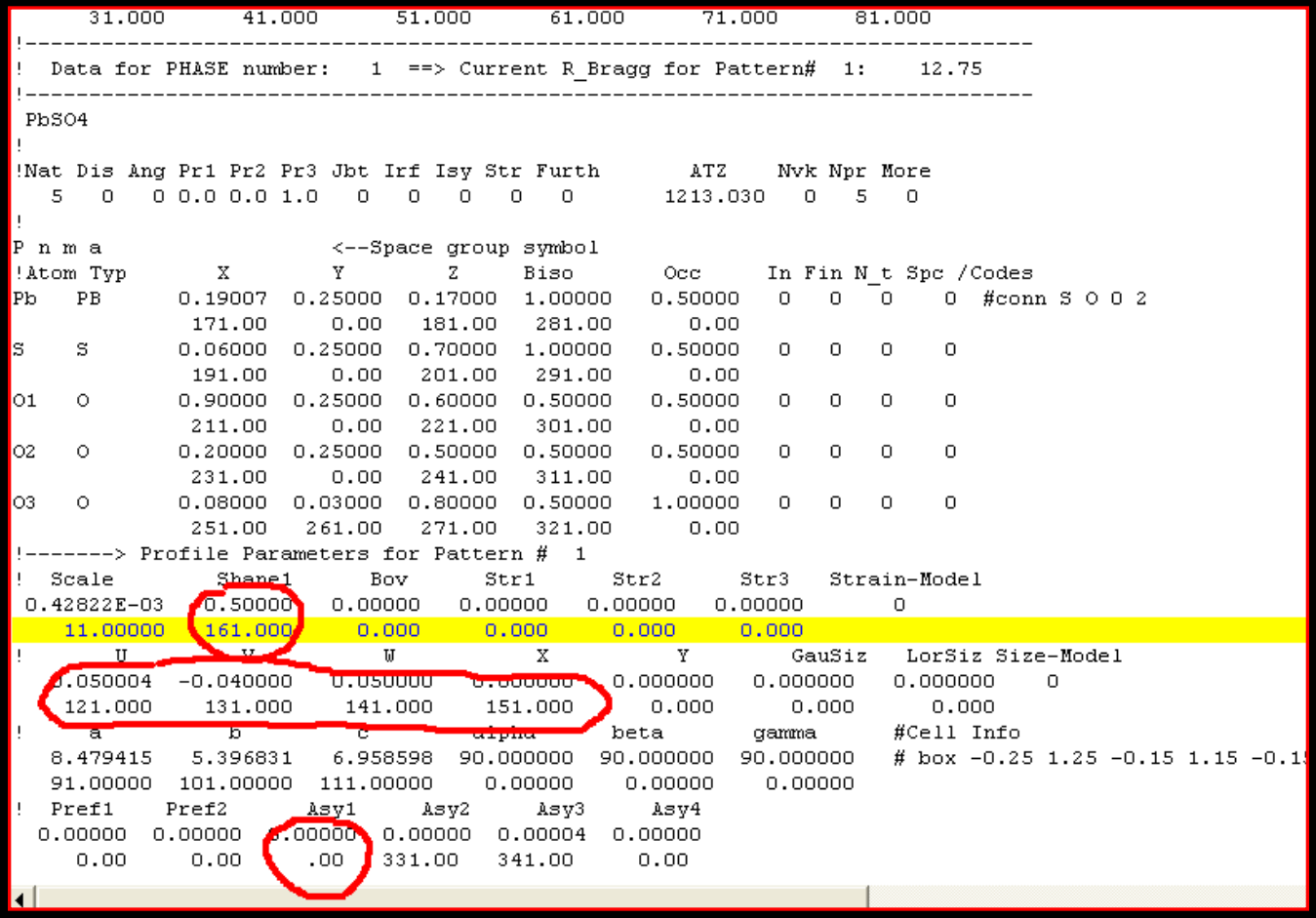
Refine lattice parameters along with others


File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications
Tools Help
PbSO4 XrayDif (Rietveld Refinement Round Robin, R.J. Hill, JApC 2
pbsox.PRF:
Yobs
Ycalc
Yobs-Ycalc
Bragg_position
Intensity (a.u.)
2θ (°)
10-31-2005 10:07
X = 163.98274
Y = 1127.8



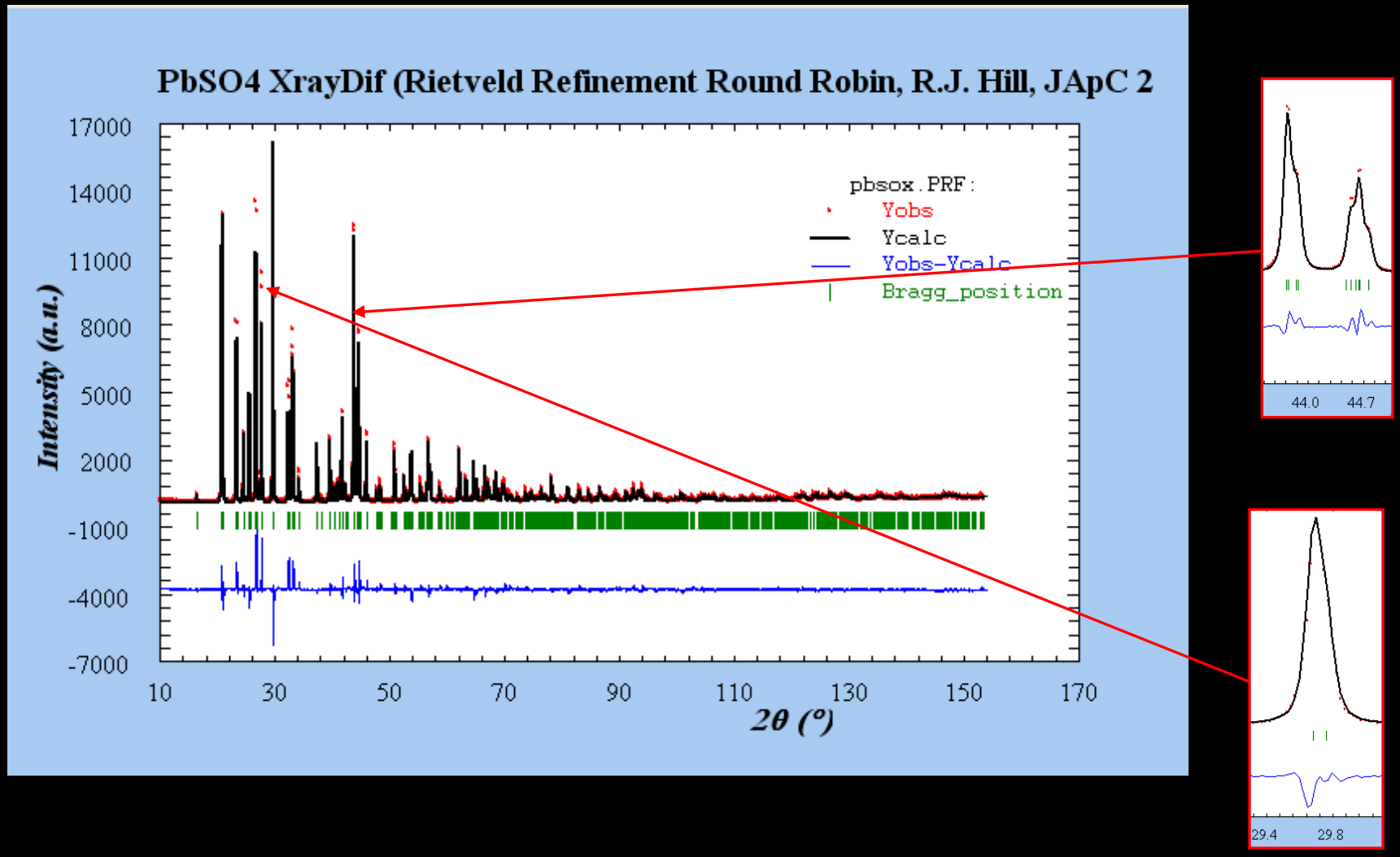


Rwp=28.9%



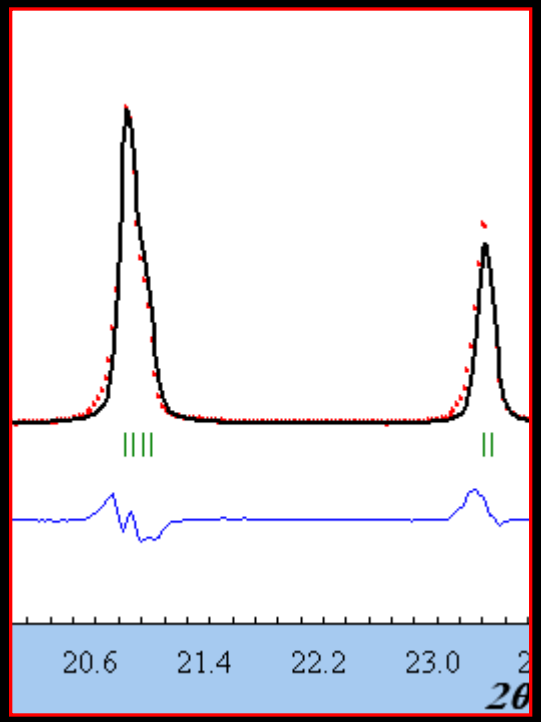
```
      31.000       41.000       51.000       61.000       71.000       81.000
!------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:    12.75
!------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ    Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030   0   5   0
!
P n m a                  <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    PB       0.19007   0.25000   0.17000  1.00000   0.50000   0   0   0    0  #conn S O O 2
                171.00      0.00    181.00   281.00      0.00
S     S        0.06000   0.25000   0.70000  1.00000   0.50000   0   0   0    0
                191.00      0.00    201.00   291.00      0.00
O1    O        0.90000   0.25000   0.60000  0.50000   0.50000   0   0   0    0
                211.00      0.00    221.00   301.00      0.00
O2    O        0.20000   0.25000   0.50000  0.50000   0.50000   0   0   0    0
                231.00      0.00    241.00   311.00      0.00
O3    O        0.08000   0.03000   0.80000  0.50000   1.00000   0   0   0    0
                251.00    261.00    271.00   321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale         Shape1        Bov          Str1         Str2         Str3    Strain-Model
 0.42822E-03    0.50000     0.00000      0.00000      0.00000      0.00000         0
    11.00000    161.000       0.000        0.000        0.000        0.000
!       U           V           W            X            Y          GauSiz     LorSiz Size-Model
   0.050004   -0.040000    0.050000     0.000000     0.000000     0.000000     0.000000     0
   121.000     131.000     141.000      151.000        0.000        0.000        0.000
!       a           b           c          alpha        beta         gamma       #Cell Info
   8.479415    5.396831    6.958598   90.000000   90.000000   90.000000    # box -0.25 1.25 -0.15 1.15 -0.1
   91.00000   101.00000   111.00000    0.00000      0.00000     0.00000
!   Pref1       Pref2        Asy1         Asy2         Asy3         Asy4
  0.00000     0.00000     0.00000      0.00000      0.00004      0.00000
     0.00        0.00         .00       331.00       341.00         0.00
```

Refine peak profile along with other parameters

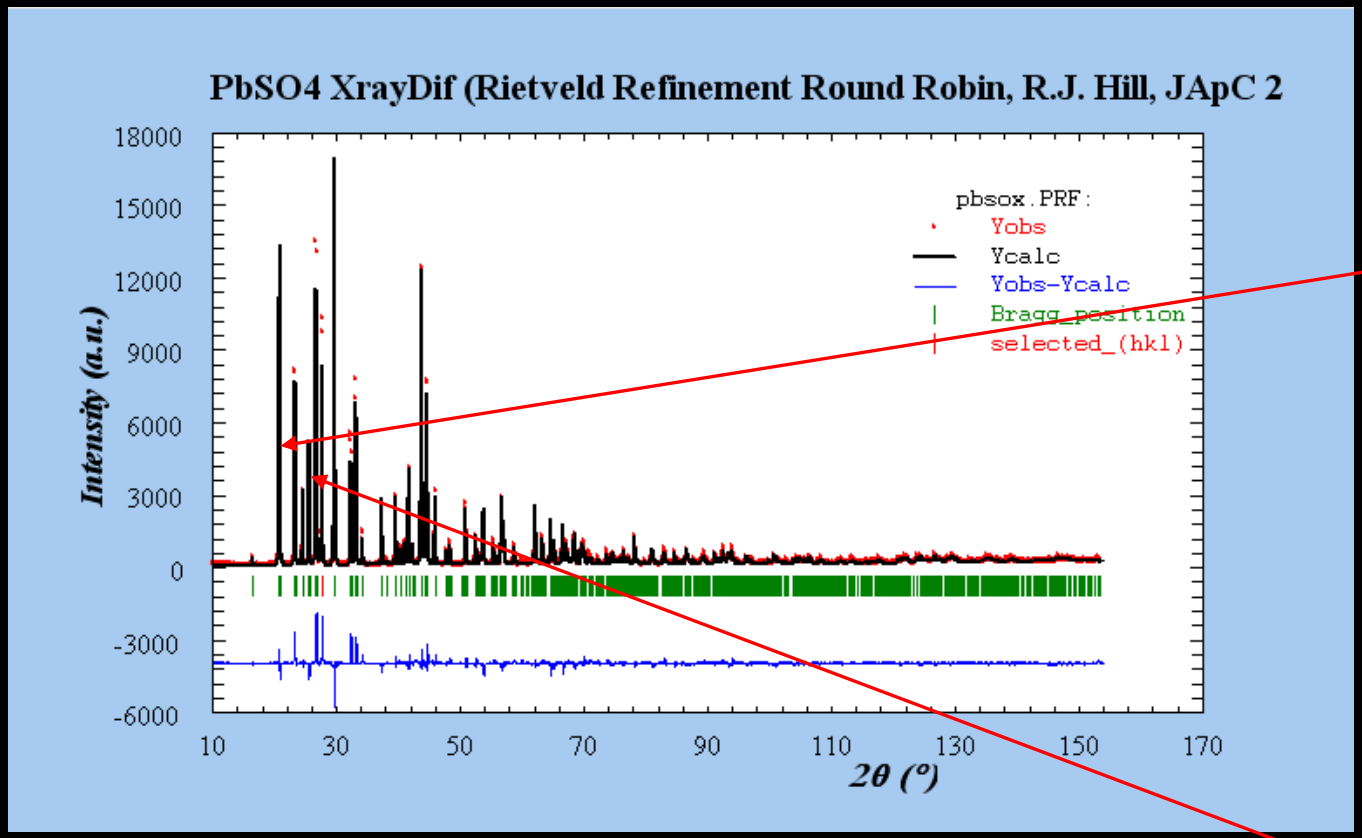

PbSO4 XrayDif (Rietveld Refinement Round Robin, R.J. Hill, JApC 2
Intensity (a.u.)
17000
14000
11000
8000
5000
2000
-1000
-4000
-7000
10
30
50
70
90
110
130
150
170
2θ (°)
pbsox.PRF:
Yobs
Ycalc
Yobs-Ycalc
Bragg_position
44.0
44.7
29.4
29.8

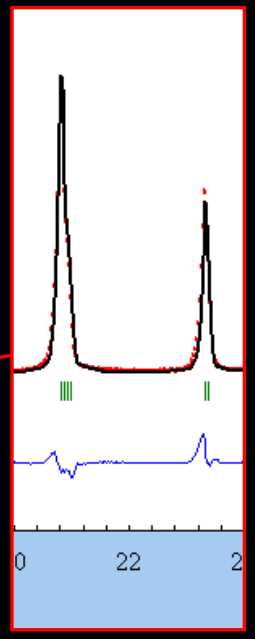
Rwp=18.9%

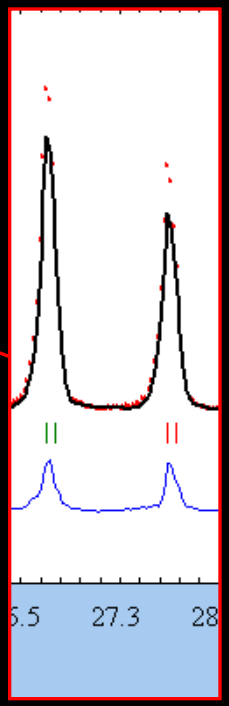
```
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:    11.58
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth        ATZ    Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1213.030   0   5   0
!
P n m a                   <--Space group symbol
!Atom Typ        X         Y         Z        Biso       Occ      In Fin N_t Spc /Codes
Pb    PB       0.19007   0.25000   0.17000   1.00000   0.50000   0   0   0    0  #conn S O O 2
                 .00       0.00      .00      281.00      0.00
S     S        0.06880   0.25000   0.70000   1.00000   0.50000   0   0   0    0
                191.00      0.00    201.00    291.00      0.00
O1    O        0.90000   0.25000   0.60000   0.50000   0.50000   0   0   0    0
                211.00      0.00    221.00    301.00      0.00
O2    O        0.20000   0.25000   0.50000   0.50000   0.50000   0   0   0    0
                231.00      0.00    241.00    311.00      0.00
O3    O        0.08000   0.03000   0.80000   0.50000   1.00000   0   0   0    0
                251.00    261.00    271.00    321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.45011E-03    0.45326    0.00000    0.00000    0.00000    0.00000        0
    11.00000    161.000      0.000      0.000      0.000      0.000
!       U          V          W          X          Y        GauSiz    LorSiz Size-Model
   0.028650  -0.019695   0.015045   0.006195   0.000000   0.000000   0.000000     0
    121.000    131.000    141.000    151.000      0.000      0.000      0.000
!      a          b          c        alpha       beta       gamma      #Cell Info
   8.479496   5.397915   6.959387  90.000000  90.000000  90.000000   # box -0.25 1.25 -0.15 1.15
   91.00000   101.00000  111.00000    0.00000    0.00000    0.00000
!  Pref1      Pref2      Asy1       Asy2       Asy3       Asy4
  0.00000    0.00000    0.00000    0.00000    0.00004    0.00000
     0.00       0.00     171.000    181.00     341.00       0.00
```



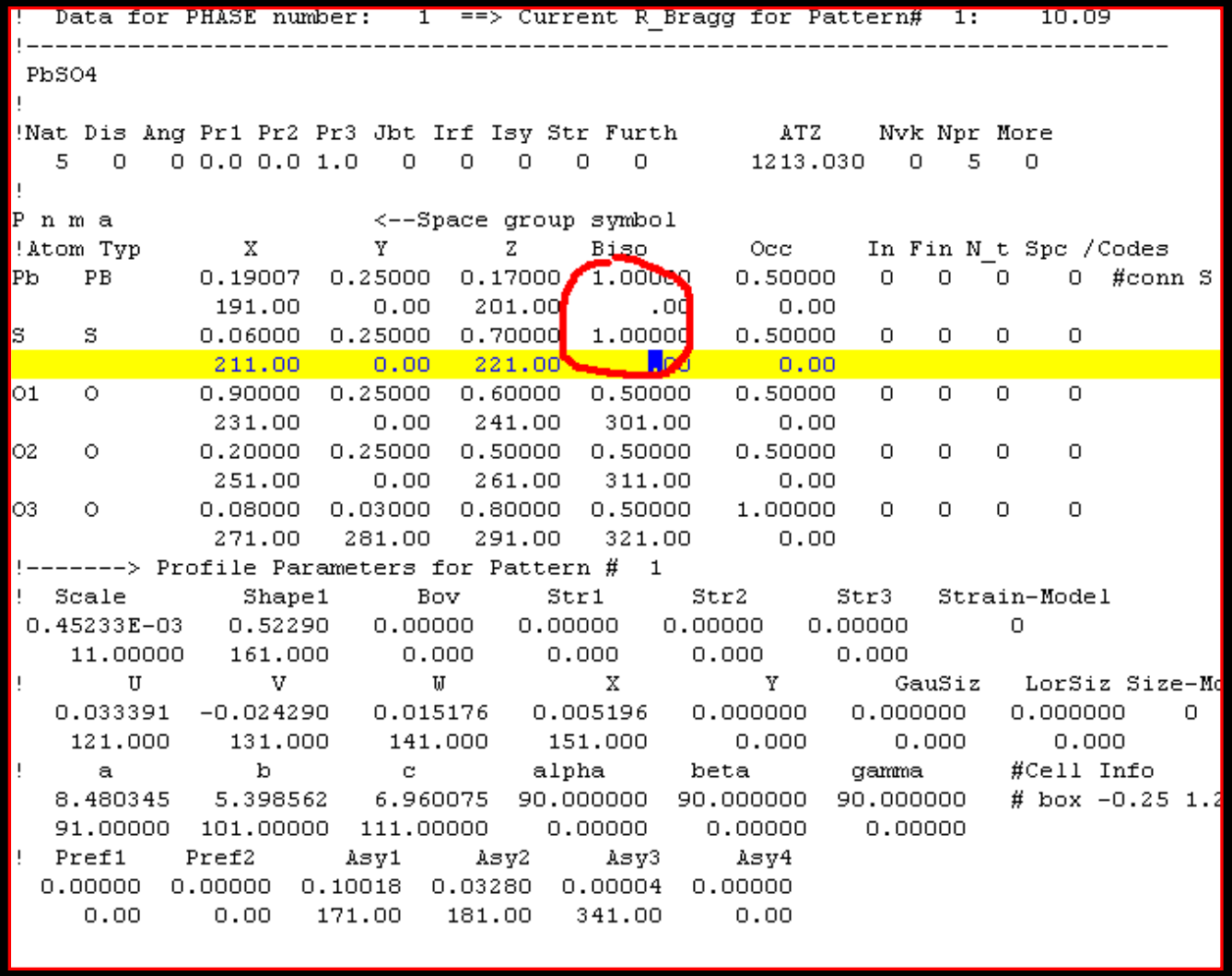
Refine asymmetry


PbSO4 XrayDif (Rietveld Refinement Round Robin, R.J. Hill, JApC 2
Intensity (a.u.)
18000
15000
12000
9000
6000
3000
0
-3000
-6000
10
30
50
70
90
110
130
150
170
2θ (°)
pbsox.PRF:
Yobs
Ycalc
Yobs-Ycalc
Bragg_position
selected_(hkl)


0
22
2


5.5
27.3
28

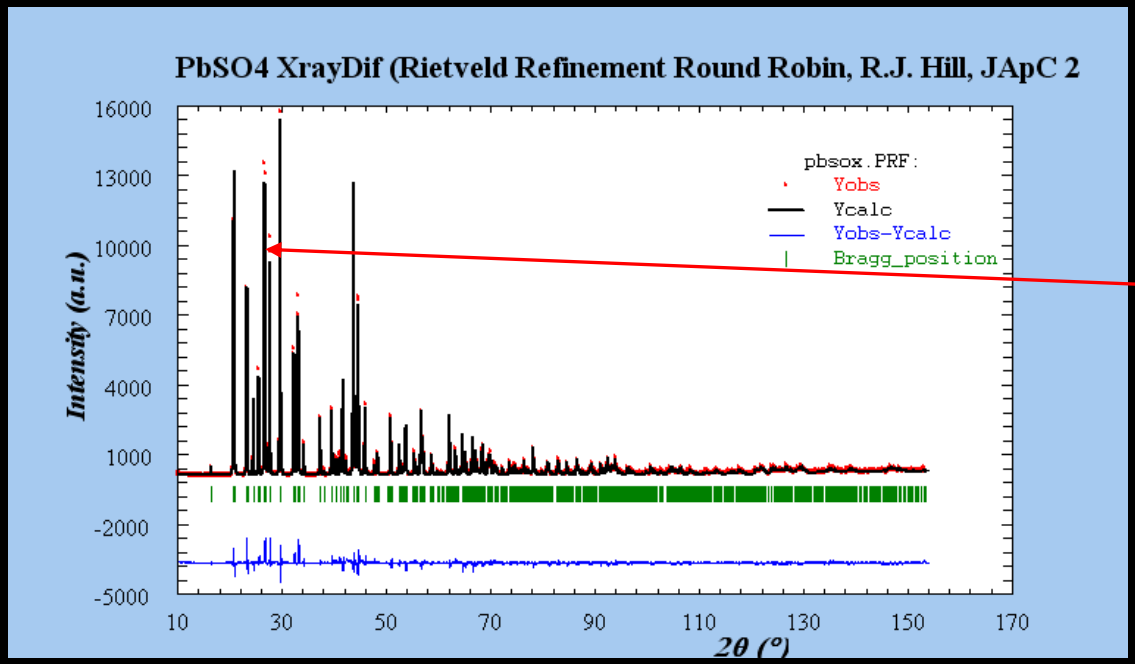
Rwp=16.8%

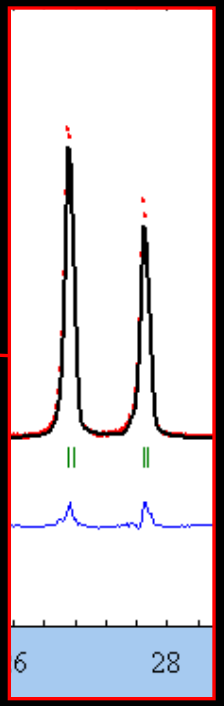


```
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:    10.09
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0      1213.030   0   5   0
!
P n m a                    <--Space group symbol
!Atom Typ       X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    PB      0.19007   0.25000   0.17000  1.00000   0.50000   0   0   0     0  #conn S
              191.00      0.00    201.00      .00      0.00
S     S       0.06000   0.25000   0.70000  1.00000   0.50000   0   0   0     0
              211.00      0.00    221.00      .00      0.00
O1    O       0.90000   0.25000   0.60000  0.50000   0.50000   0   0   0     0
              231.00      0.00    241.00   301.00      0.00
O2    O       0.20000   0.25000   0.50000  0.50000   0.50000   0   0   0     0
              251.00      0.00    261.00   311.00      0.00
O3    O       0.08000   0.03000   0.80000  0.50000   1.00000   0   0   0     0
              271.00    281.00    291.00   321.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.45233E-03     0.52290    0.00000    0.00000    0.00000    0.00000         0
    11.00000     161.000      0.000      0.000      0.000      0.000
!       U           V           W           X           Y         GauSiz    LorSiz Size-Mo
   0.033391   -0.024290    0.015176    0.005196    0.000000    0.000000   0.000000     0
    121.000     131.000     141.000     151.000       0.000       0.000      0.000
!      a           b           c         alpha       beta        gamma       #Cell Info
   8.480345    5.398562    6.960075   90.000000   90.000000   90.000000   # box -0.25 1.2
   91.00000   101.00000   111.00000     0.00000     0.00000     0.00000
!  Pref1      Pref2       Asy1        Asy2        Asy3        Asy4
  0.00000    0.00000    0.10018     0.03280     0.00004     0.00000
     0.00       0.00     171.00      181.00      341.00        0.00
```

Refine atomic coordinates: first two atoms Pb and S

the number of parameters to be refined:22

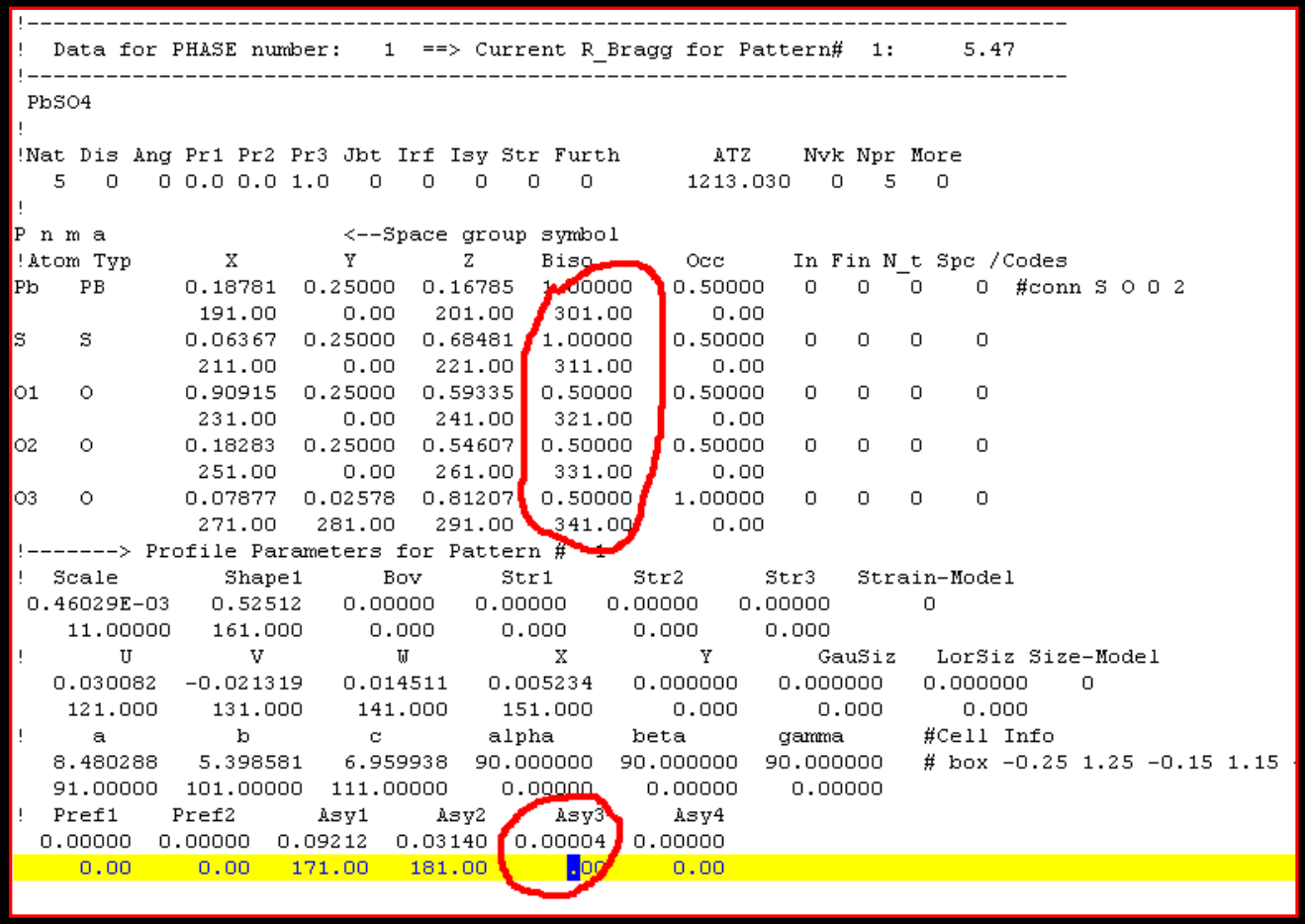




Rwp=14.0%

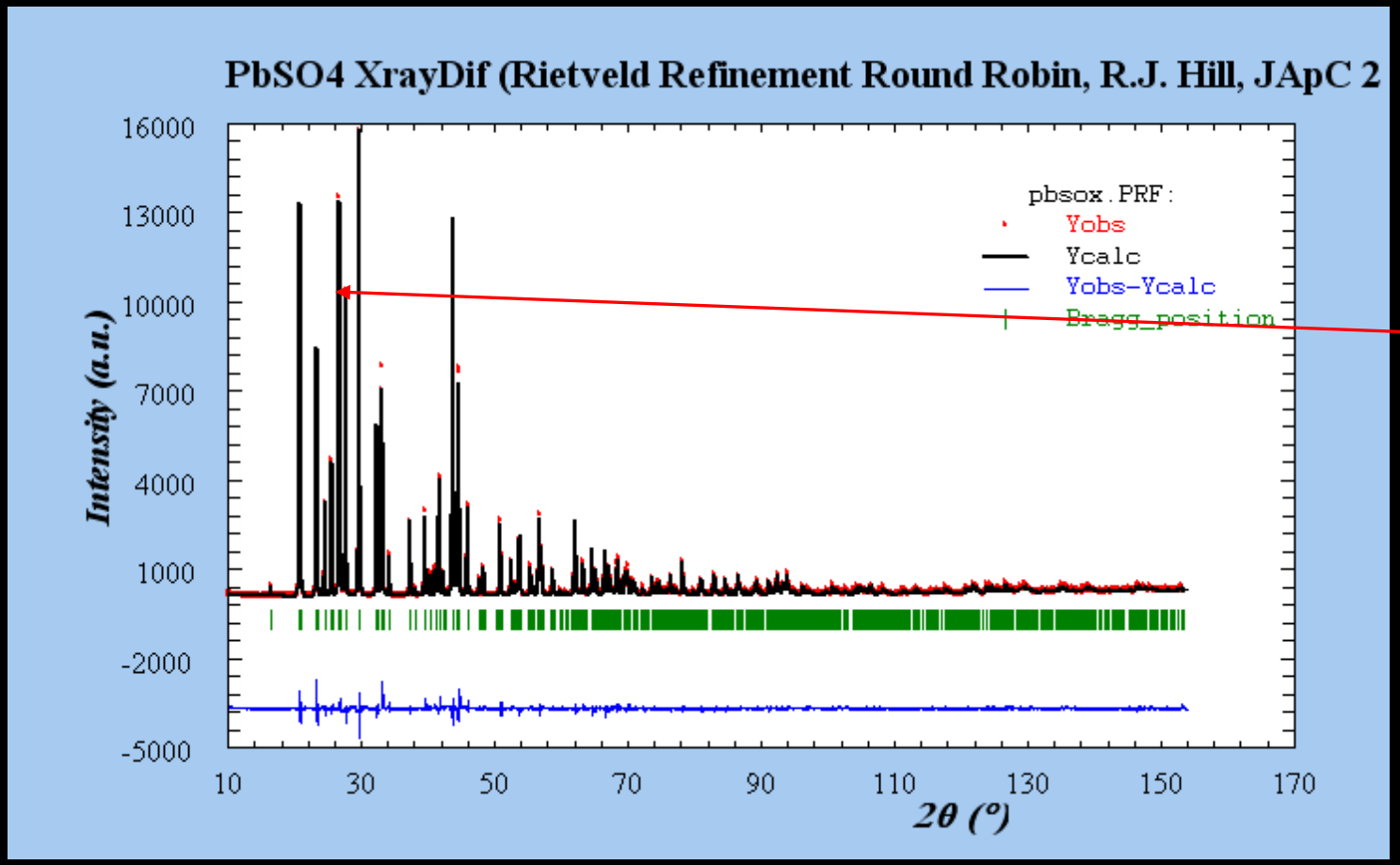
Further refine atomic coordinates of 3 O atoms

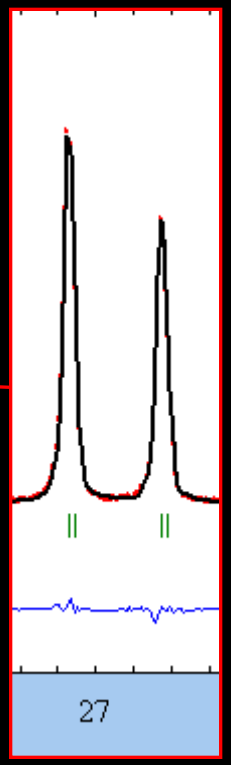
Rwp=13.1%



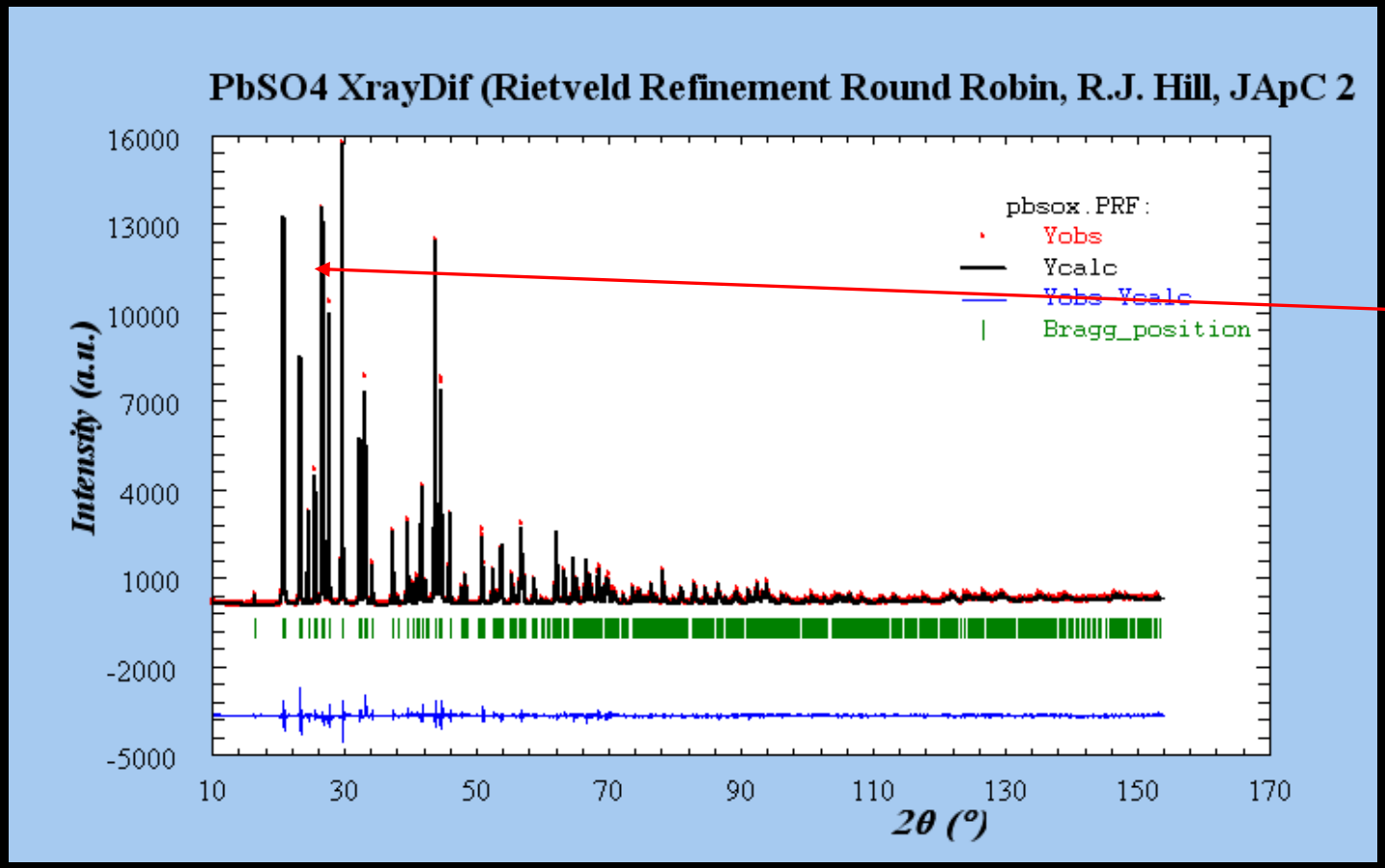
```
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     5.47
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ    Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0      1213.030   0   5   0
!
P n m a                    <--Space group symbol
!Atom Typ       X         Y         Z        Biso       Occ     In Fin N_t Spc /Codes
Pb    PB      0.18781   0.25000   0.16785   1.00000   0.50000   0   0   0    0  #conn S O O 2
               191.00      0.00    201.00    301.00      0.00
S     S       0.06367   0.25000   0.68481   1.00000   0.50000   0   0   0    0
               211.00      0.00    221.00    311.00      0.00
O1    O       0.90915   0.25000   0.59335   0.50000   0.50000   0   0   0    0
               231.00      0.00    241.00    321.00      0.00
O2    O       0.18283   0.25000   0.54607   0.50000   0.50000   0   0   0    0
               251.00      0.00    261.00    331.00      0.00
O3    O       0.07877   0.02578   0.81207   0.50000   1.00000   0   0   0    0
               271.00    281.00    291.00    341.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3    Strain-Model
 0.46029E-03     0.52512    0.00000    0.00000    0.00000    0.00000        0
    11.00000    161.000      0.000      0.000      0.000      0.000
!       U          V          W          X          Y        GauSiz    LorSiz Size-Model
   0.030082  -0.021319   0.014511   0.005234   0.000000   0.000000   0.000000     0
    121.000    131.000    141.000    151.000      0.000      0.000      0.000
!      a          b          c       alpha       beta       gamma      #Cell Info
   8.480288   5.398581   6.959938  90.000000  90.000000  90.000000   # box -0.25 1.25 -0.15 1.15
   91.00000  101.00000  111.00000    0.00000    0.00000    0.00000
!  Pref1    Pref2      Asy1       Asy2       Asy3       Asy4
  0.00000  0.00000   0.09212    0.03140    0.00004    0.00000
     0.00     0.00    171.00     181.00          00       0.00
```

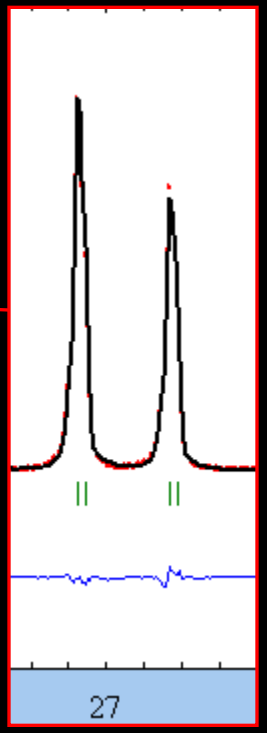
Refine temperature factors along other parameters


PbSO4 XrayDif (Rietveld Refinement Round Robin, R.J. Hill, JApC 2
pbsox.PRF:
Yobs
Ycalc
Yobs-Ycalc
Bragg_position
Intensity (a.u.)
16000
13000
10000
7000
4000
1000
-2000
-5000
10
30
50
70
90
110
130
150
170
2θ (°)


27

Rwp=12.5%





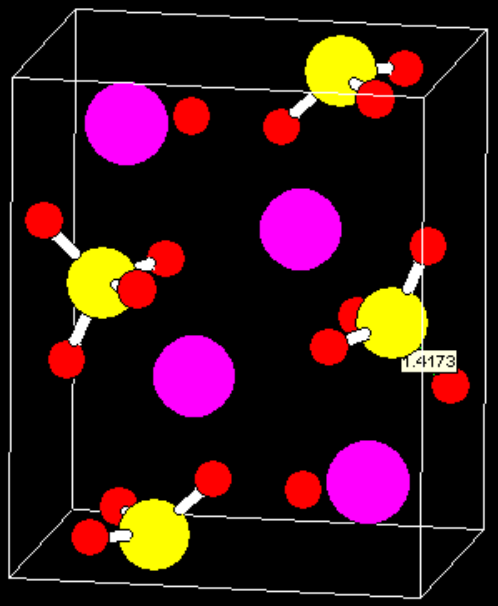
R_p=8.92%, R_{wp}=10.8%, R_{exp}=6.57%

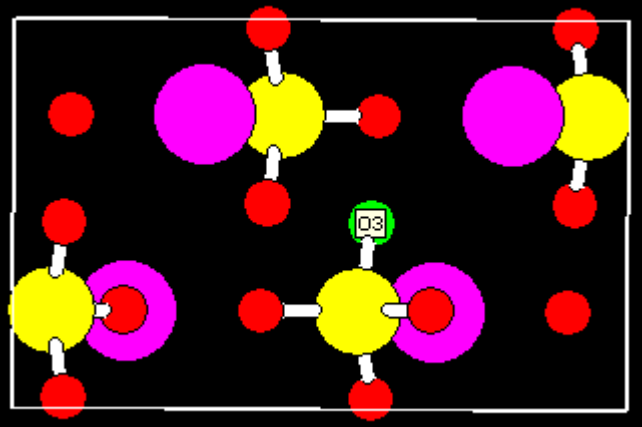
```
!
! Excluded regions (LowT  HighT) for Pattern#  1
       0.00       10.00
     154.00      180.00
!
!
     38    !Number of refined parameters
!
!  Zero     Code     SyCos     Code    SySin     Code  Lambda      Code MORE ->Patt# 1
-0.02668  21.00  0.00000    0.00  0.00000    0.00 0.000000     0.00    0
!   Background coefficients/codes  for Pattern#  1
  129.00       40.163      -144.54       40.135       221.74      -118.53
      31.000       41.000       51.000       61.000       71.000       81.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     3.72
!-------------------------------------------------------------------------------
 PbSO4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ     Nvk Npr More
   5   0   0 1.0 0.0 2.0   0   0   0   0   0       1213.030   0   5    1
!
!Jvi Jdi Hel Sol Mom Ter   Brind    RMua     RMub     RMuc    Jtyp  Nsp_Ref Ph_Shift
   0   3   0   0   0   0  1.0000  0.0000   0.0000   0.0000     0       0       0
!
! Max_dst(dist) (angles)  Bond-Valence Calc.
      4.5000       0.0000         BVS
!  N_cations   N_anions      Tolerance(%) / Name or cations/ and Anions
       2            3               300.00
Pb+2 S+6
O-2  O-2  O-2
!
P n m a                    <--Space group symbol
!Atom Typ        X         Y         Z      Biso       Occ      In Fin N_t Spc /Codes
Pb    Pb      0.18785   0.25000   0.16742   1.59384   0.50000    0   0   0     1  #conn S O O 2
             191.00      0.00    201.00    301.00      0.00
```

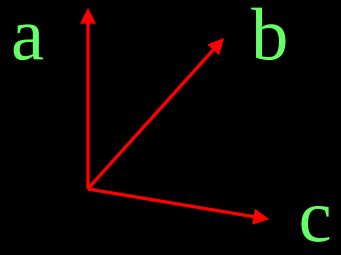
Calculate the bond lengths and bond valences

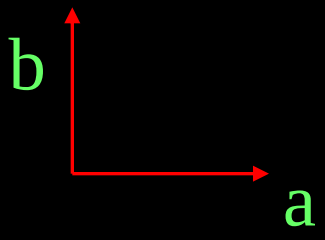
And the results are stored in *.dis

$PbSO_4$

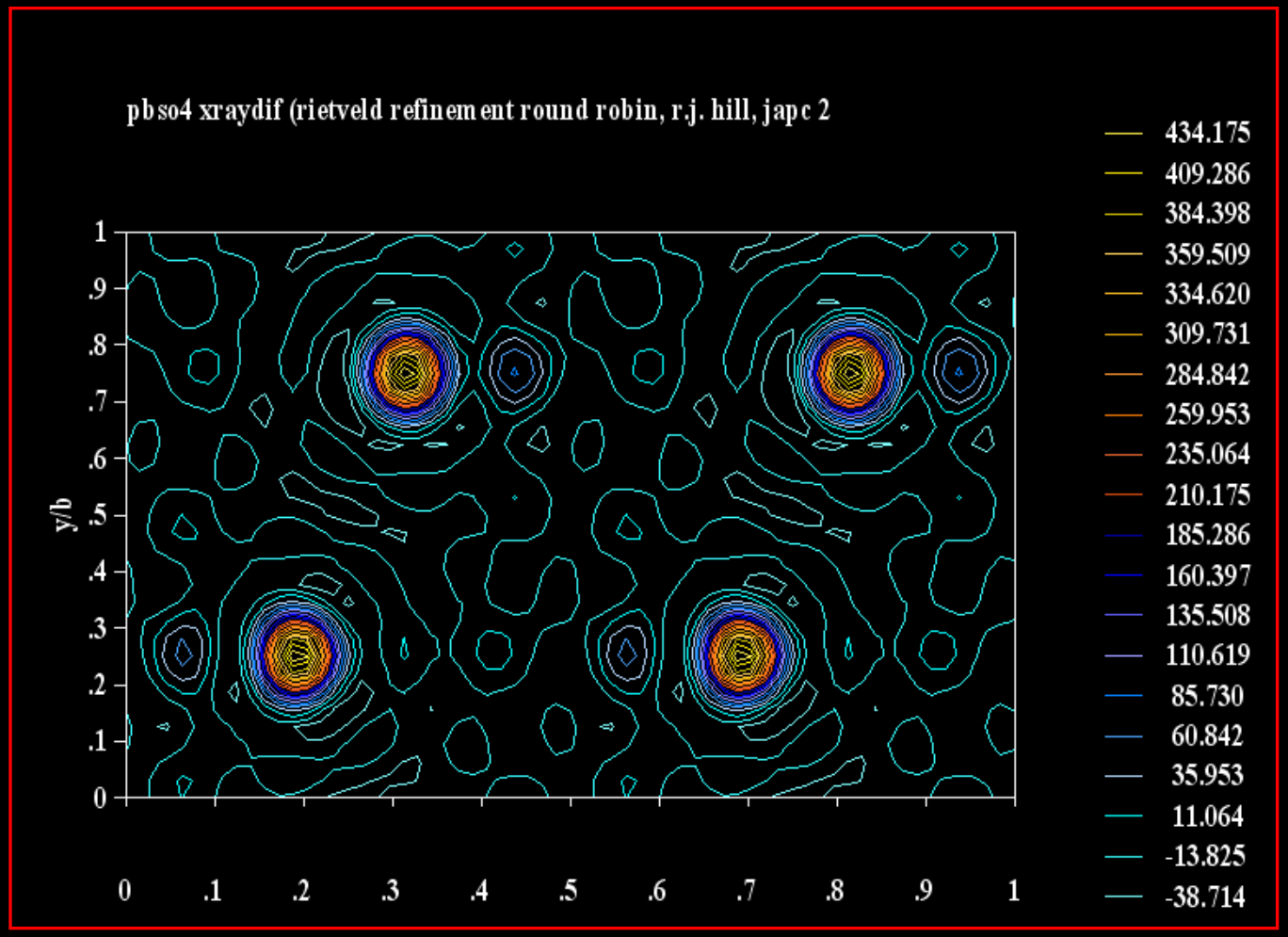








$PbSO_4$



Set Fou=4 in your Pcr file

Fourier synthesis

Compare with the Rietveld Refinement Round Robin

R_p=7.3%-16.6%	5.82%	8.91%
R_{wp}=8.2-20.0%	7.83%	10.8%
R_{exp}=1.5%-7.0%	4.83%	6.71%
GodF=1.3-7.4	1.6	1.6

Total 23 respondents

Background excluded

Compare with the Rietveld Refinement Round Robin

	Range	mean	single crystal	this work
a(Å)	8.4764-8.4859	8.4804(4)	8.482(2)	8.4818(1)
b(Å)	5.3962-5.4024	5.3989(3)	5.398(2)	5.3997(1)
c(Å)	6.9568-6.9650	6.9605(4)	6.959(2)	6.9614(1)

R.J.Hill, J. Appl.Cryst. 25, 589(1992)

Compare with the Rietveld Refinement Round Robin

		Range	mean	single crystal	this work
Pb	x	0.1875-0.1883	0.18783(4)	0.1879(1)	0.18785(7)
	z	0.1669-0.1683	0.16752(9)	0.1667(1)	0.16742(10)
S	x	0.0621-0.0673	0.0642(2)	0.0633(6)	0.0638(4)
	z	0.6799-0.6860	0.6838(4)	0.6842(7)	0.6834(6)
O1	x	0.902-0.924	0.9083(13)	0.908(2)	0.9069(11)
	z	0.585-0.601	0.5945(7)	0.596(3)	0.5929(14)

Compare with the Rietveld Refinement Round Robin

		Range	mean	single crystal	this work
O2	x	0.177-0.200	0.1850(11)	0.194(2)	0.1894(11)
	z	0.523-0.548	0.5398(13)	0.543(2)	0.5423(14)
O3	x	0.071-0.080	0.0778(5)	0.082(1)	0.0789(6)
	y	0.018-0.041	0.026(13)	0.026(2)	0.0214(9)
	z	0.806-0.819	0.8139(7)	0.809(2)	0.8130(9)

Compare with the Rietveld Refinement Round Robin

	Range	mean	single crystal	this work
B Pb(Å^2)	0.90-2.39	1.42(11)	1.48	1.59(2)
S (Å^2)	0.29-1.37	0.98(8)	0.74	1.21(8)
O1(Å^2)	0.50-4.2	1.24(10)	1.87	1.32(21)
O2(Å^2)	0.1-5.8	1.31(13)	1.76	2.02(21)
O3(Å^2)	0.8-4.6	1.27(11)	1.34	1.05(13)

Output file: Data.dis

Bond length, Angel, Bond Valence

(Pb)-(O1): 2.595(8)

(Pb)-(O2): 3.022(4)

(Pb)-(O3): 2.909(6)

(S)-(O1) : 1.4721(84)

(S)-(O2) : 1.4486(102)

(S)-(O3) : 1.5347(59)

(S)-(O3)-(S): 56.10(13)

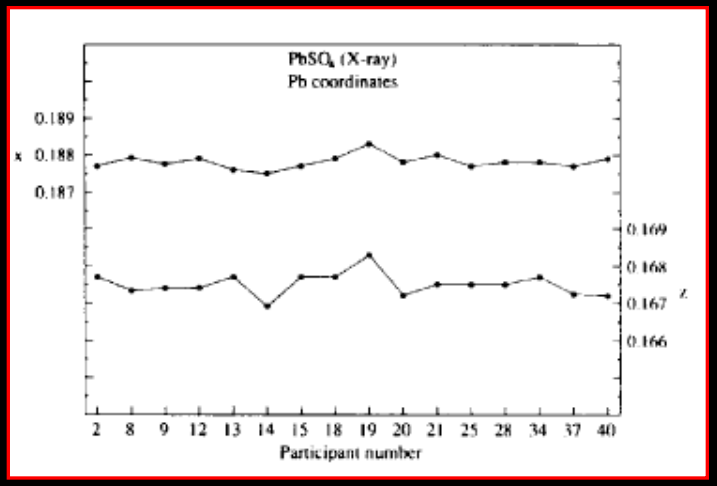
Pb : 2.273(9)

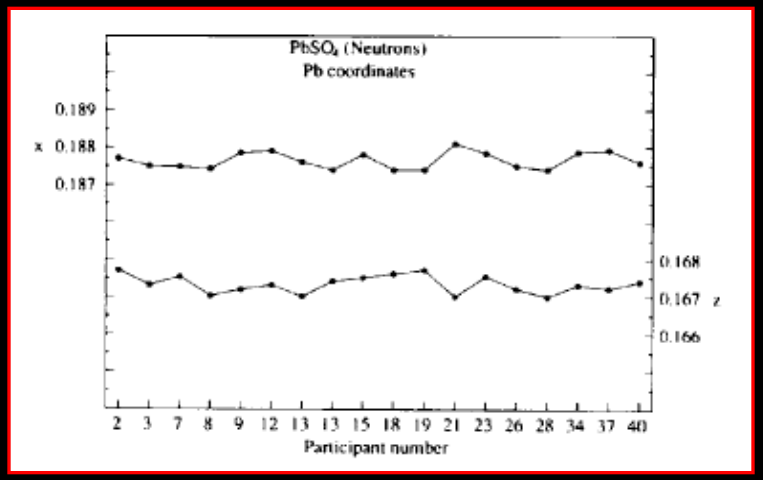
S: 5.714(63)

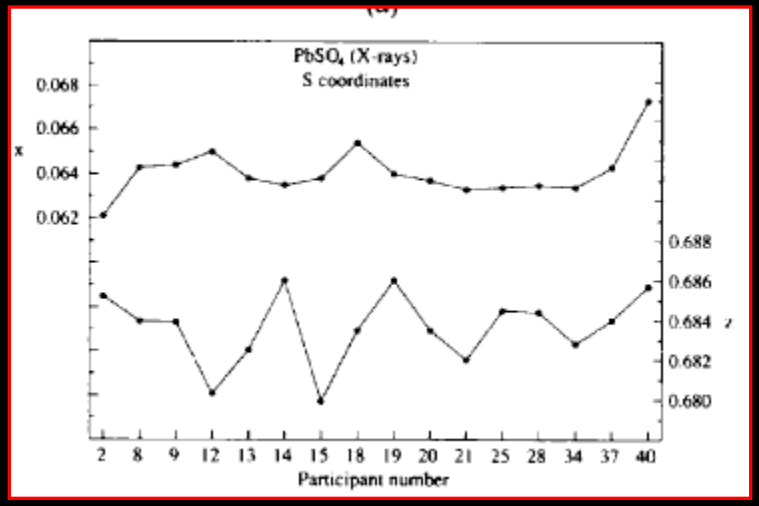
O1: 1.994(35)

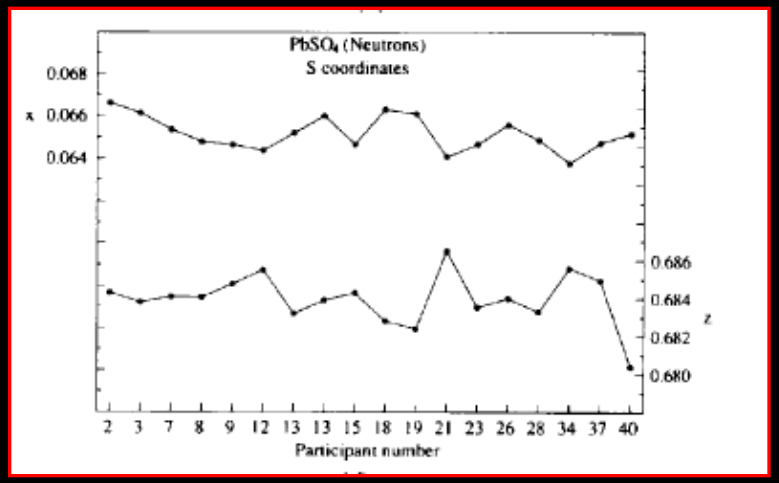
O2: 2.143(45)

O3: 1.908(21)



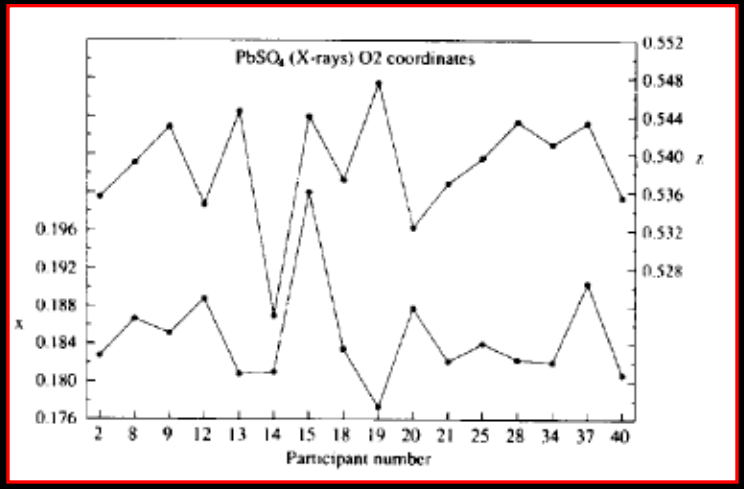


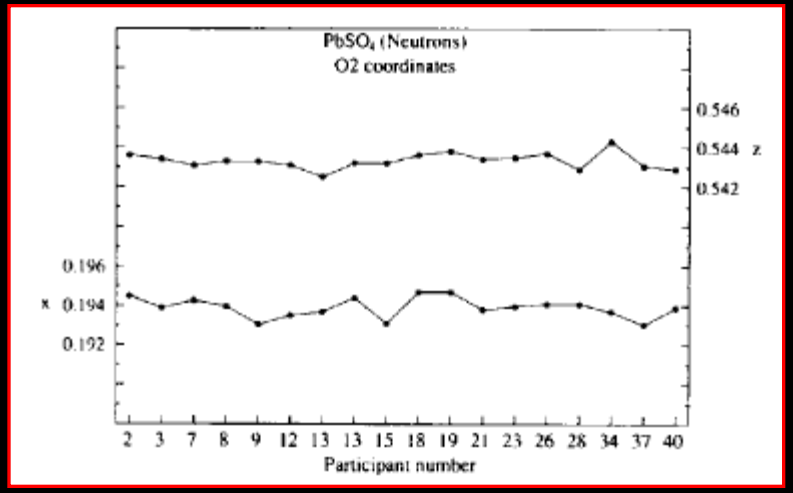


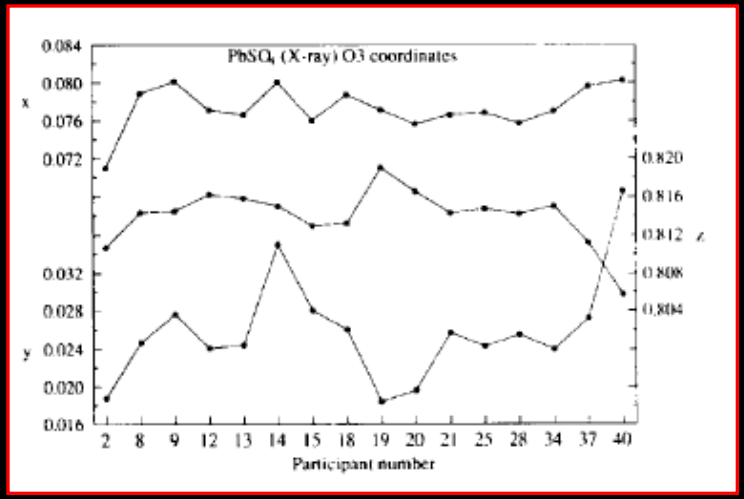


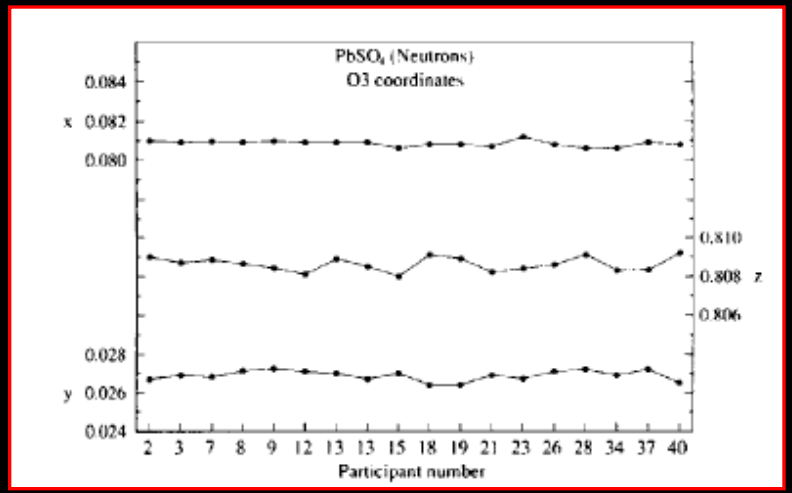
X-ray

neutron





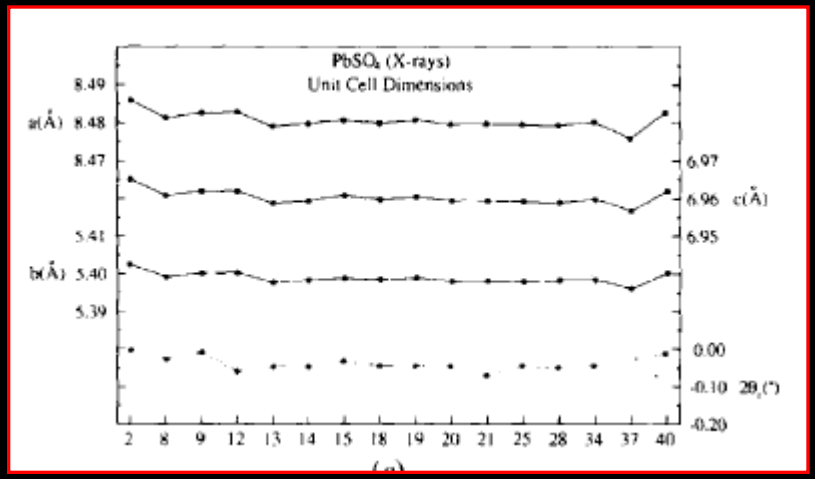




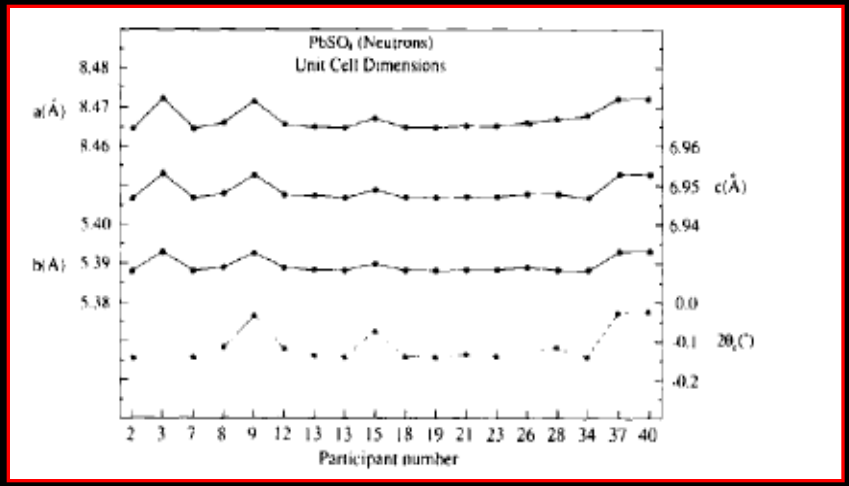
X-ray

neutrons

Lattice parameters

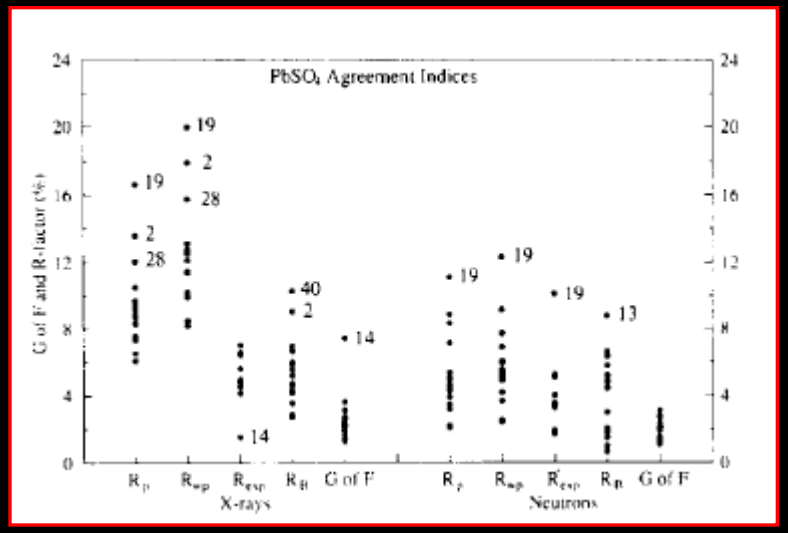


X-ray

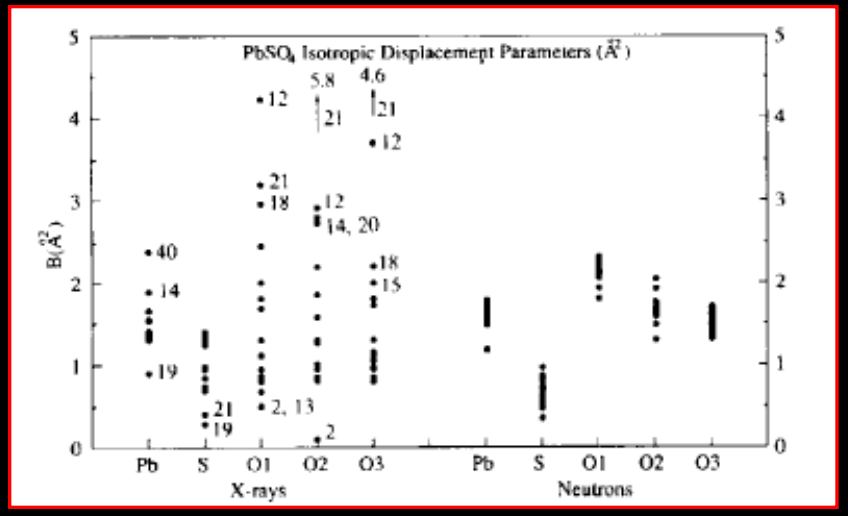


neutron

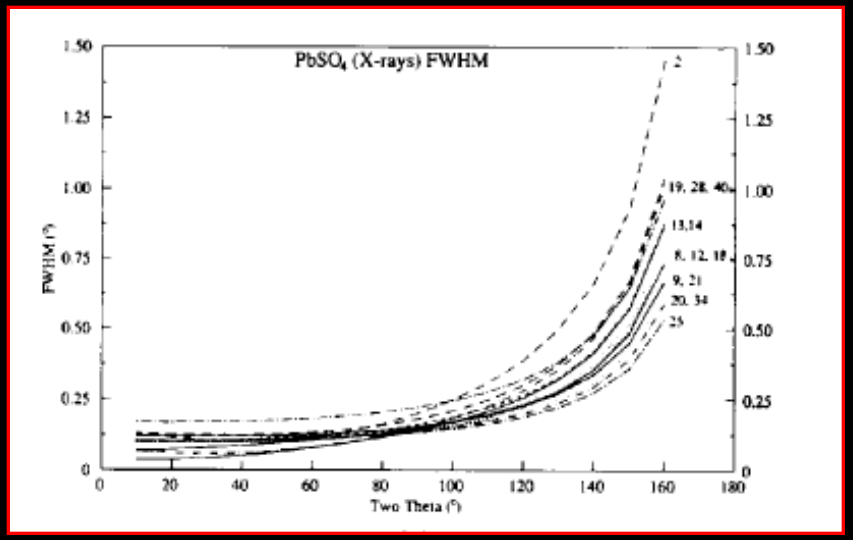
Agreement indices



Temperature factors



FWHM



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J.Rodriguez-Carvajal

Program Fullprof.2k, version 3.30, Laboratoire Leon Brillouin, France, June 2005.


谢谢！