Strategy for Rietveld refinement

Outline

I) Experiment

a) Sample

b) Data Acquisition

1) Choice of Instrument

2) Measuring time

3) Visible problems

II) Refinement

a) Literature Search

b) Starting model

c) "Strategy"

1) Zeroshift, wavelength, background (by hand)

2) Zeroshift, scalefactor, lattice parameters, 1rst background parameter

3) Atomic positions, isotropic temperature factor, background parameters

4) Peak shape parameters, asymmetry parameters

5) Individual B factors, atom occupancies

6) Zero displacements

7) Anisotropic B factors

8) Preferred Orientation, microstructural parameters

} Depends on the Data quality and the individual problem

III) Some Selected Specific Problems

1) Peak Intensities: Fourier Difference Map

2) Peak Shape: Phase Separation, Microstrain, Size effects

IV) Constraints and Restraints

1) Symmetry Constraints

2) Constraints due to direct correlation

3) Linear Constraints

4) Strategic Constraints

5) Restraints

6) Soft Distance (or Angle) Constraints

Strategy for Rietveld refinement

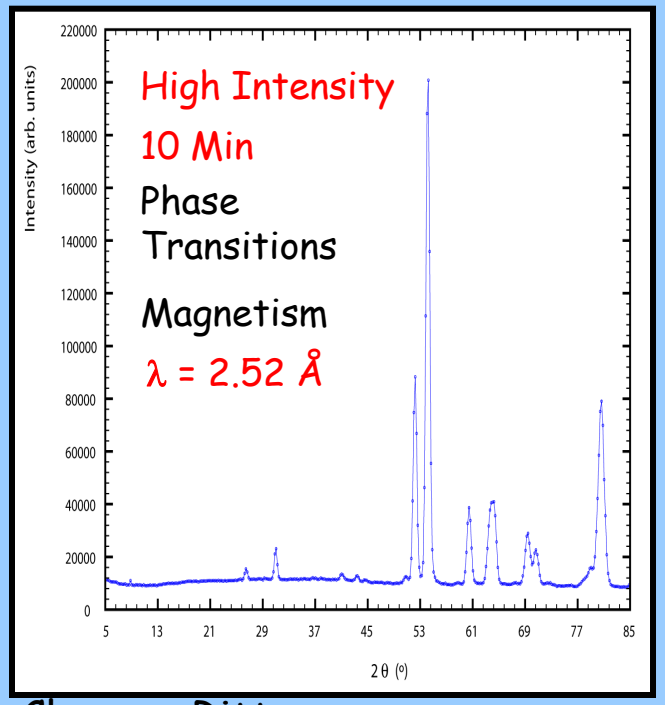
I) Experiment:

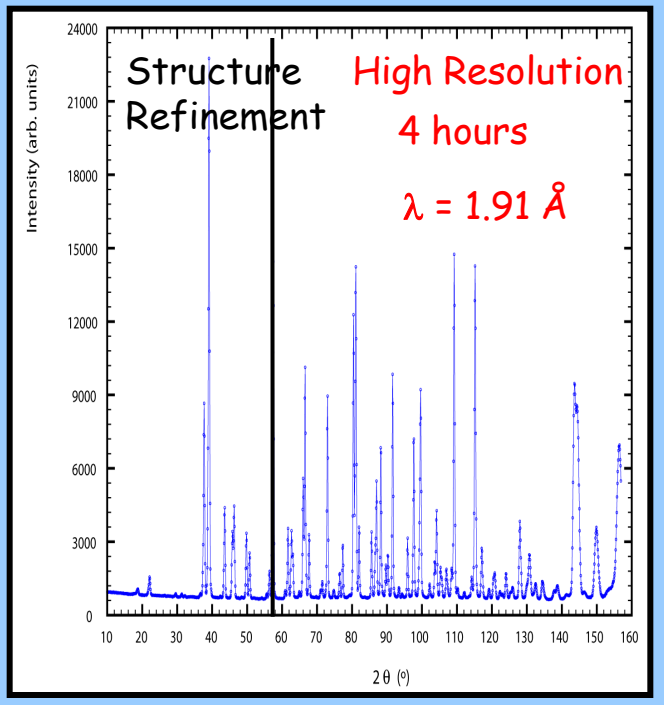
a) Sample: should be impurity free, should be enough (depending on Instrument and aim), should be well crystallized, avoid the presence of hydrogen (absorbed water)

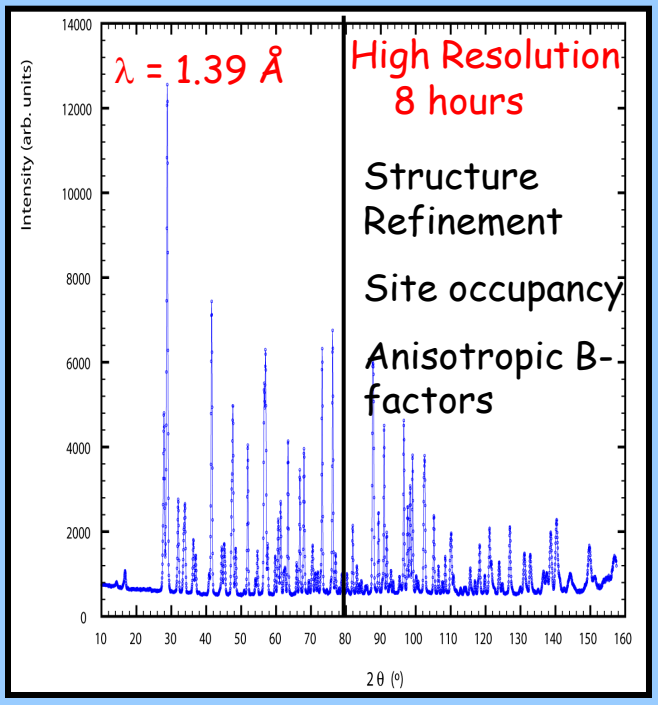
b) Data acquisition:

1) Choice of instrument: What do I want to find out? Crystallographic structure, phase transition, magnetic structure … High resolution, high intensity, q-range

Run a standard on the chosen instrument: Determine the zeroshift and the resolution function







Strategy for Rietveld refinement

At an early stage of the measurement : **Always look at the raw data**

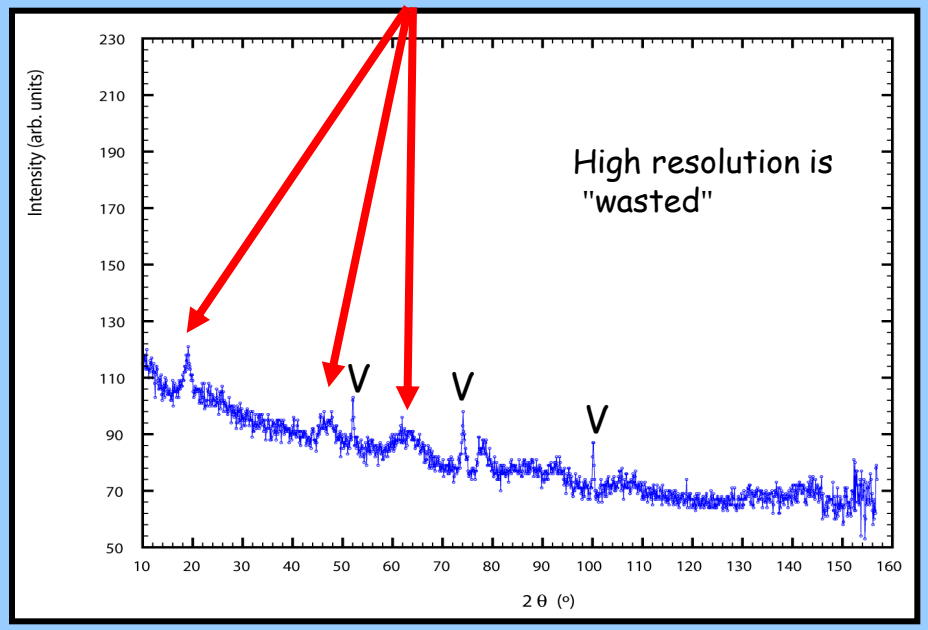
2) Measuring time: Do you see peaks?, Do they have enough intensity?, How do the peaks look like?

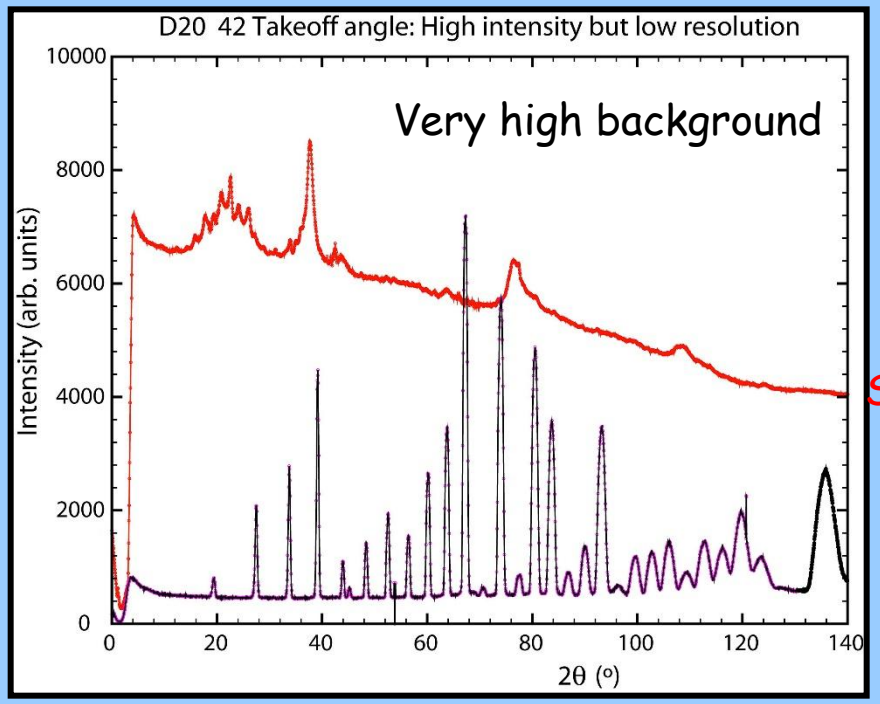
3) Visible problems:

Are the peaks only very broad? No good crystallisation, amorphous system? Go to HI machine!

How looks the background? Hydrogen? → Deuteration

"Peaks" from Sample





Strategy for Rietveld refinement

3) Visible problems:

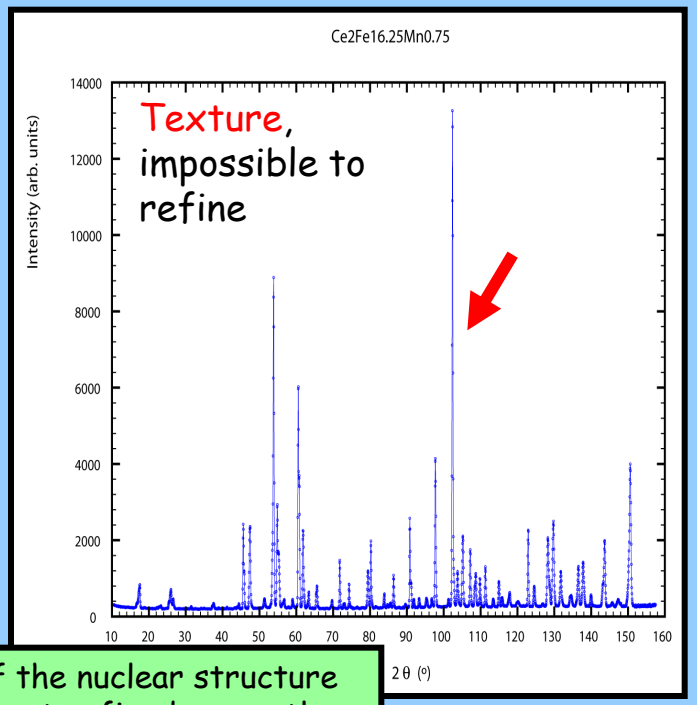
Do the spectra look different between two measurements taking the powder out?

Preferred orientation? (crystallites with layer or needle shape) : Can be corrected for,

but try to keep it constant within one measuring cycle (T-dependence).

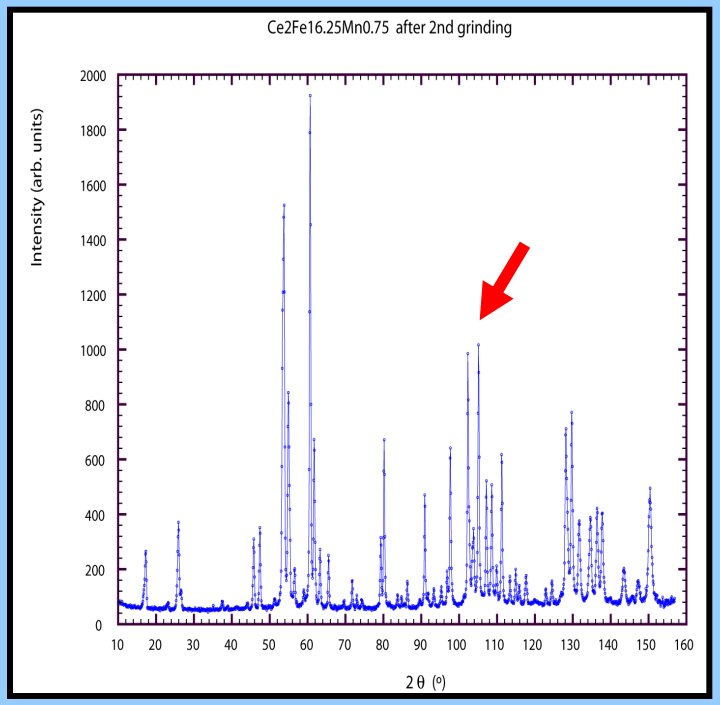
Are there peaks which are much stronger and sharper than the rest?

Single crystal!, no powder average = grind further. ➔ Try to refine as early as possible during the experiment



Same sample after grinding twice

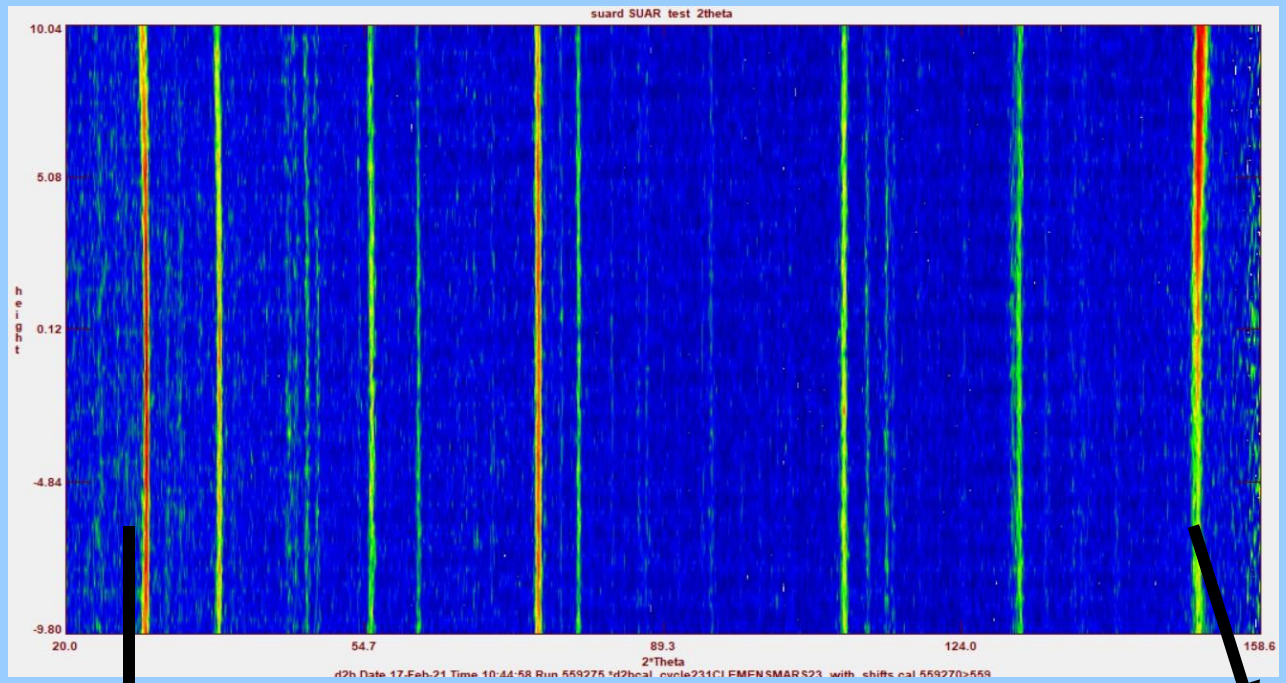
Absolute and relative intensities have totally changed!



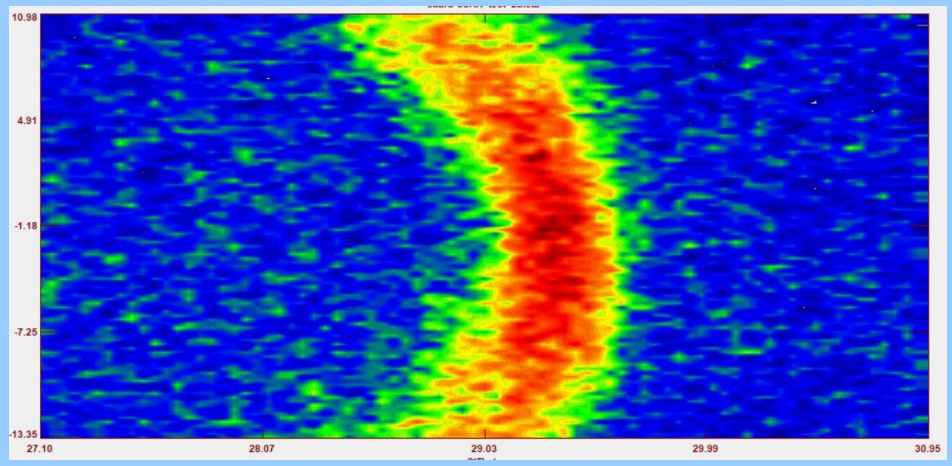
If the nuclear structure is not refined correctly you can't get anything for the magnetic structure!

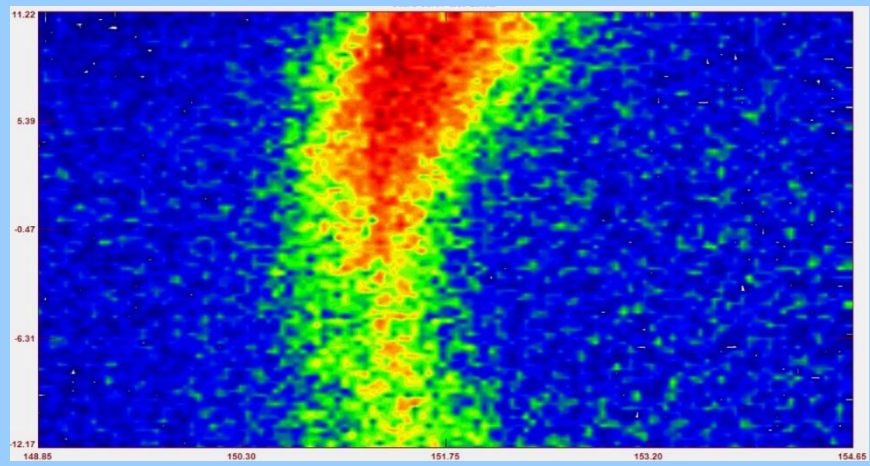
Strategy is hopeless if the data are not good!

2D plot of powder data from D2B



Texture from the Al of the used cryostat





Strategy for Rietveld refinement

NEUTRONS FOR SOCIETY

II) Refinement

a) Literature Search:

Recuperate all the information you can get from literature about the system studied.

X-ray results of the same compound, Results for similar compounds

b) Starting Model:

In order to start refinement you need a model with lattice constants (LeBail fit), spacegroup (CheckGroup), and atom positions

Recuperate old pcr file! (one where you know that it works) and introduce your model

Peakshape parameters with starting values from instrument (Standard sample).

c) Strategy:

Run FULLPROFF with zero parameters: Where is the background? Are the peak positions correct?

If not: Are the shifts between calculated and observed peak positions similar: zeroshift wrong

Are the shifts q-dependant: lattice constants wrong or wavelength wrong

In order to refine the lattice constants the cal/obs peak positions must at least partially overlap,

Start with small 2 Theta range at great d-spacings first.

Background can be put at suited starting value by hand.

Strategy for Rietveld refinement

Refine the zeroshift, the scalefactor and the lattice constants.

Always keep a "reserve" pcr file in case the program diverges.

(Put the parameter "pcr" = 2, this creates a *.new file conserving the *.pcr file)

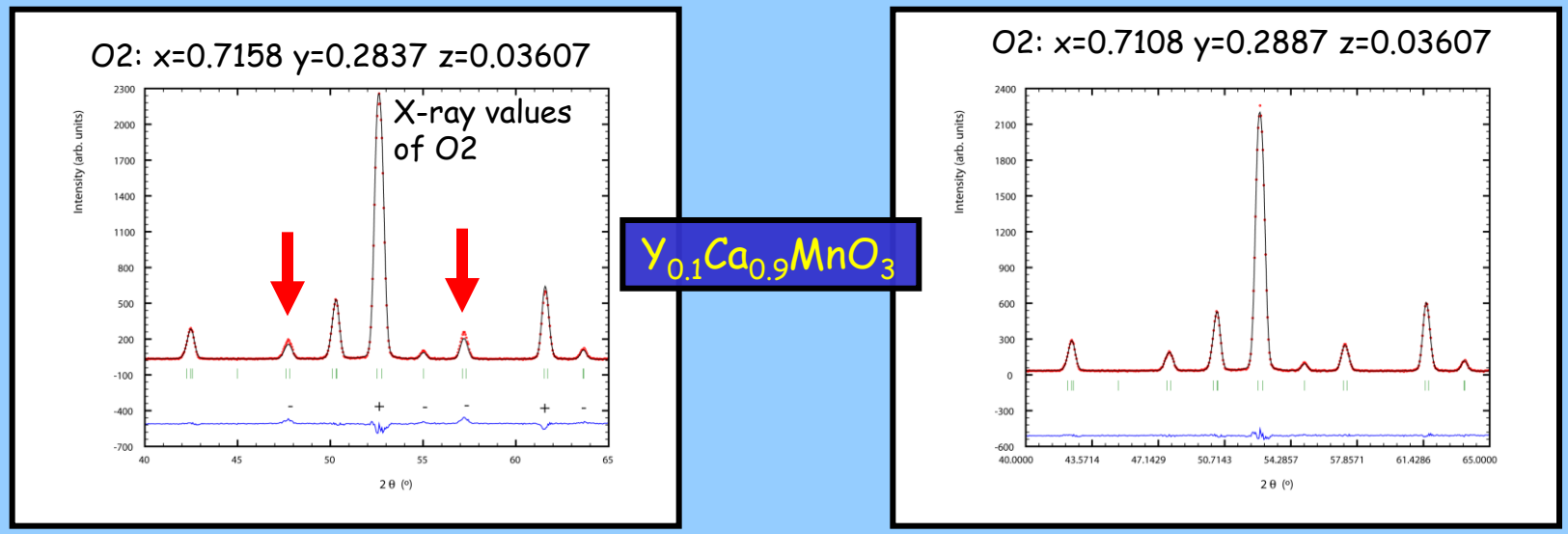
As long as the program did not "lock in", no sense to continue!

Always follow the progress of the refinement by looking at the resulting fit!

Refine the atomic positions (which are free to move), an isotropic temperature factor and the background

Wrong atom position: Difference curve shows "ups" and "downs",

very sensitive to Oxygen position ➔ be careful with from x-ray data determined structures containing Oxygen

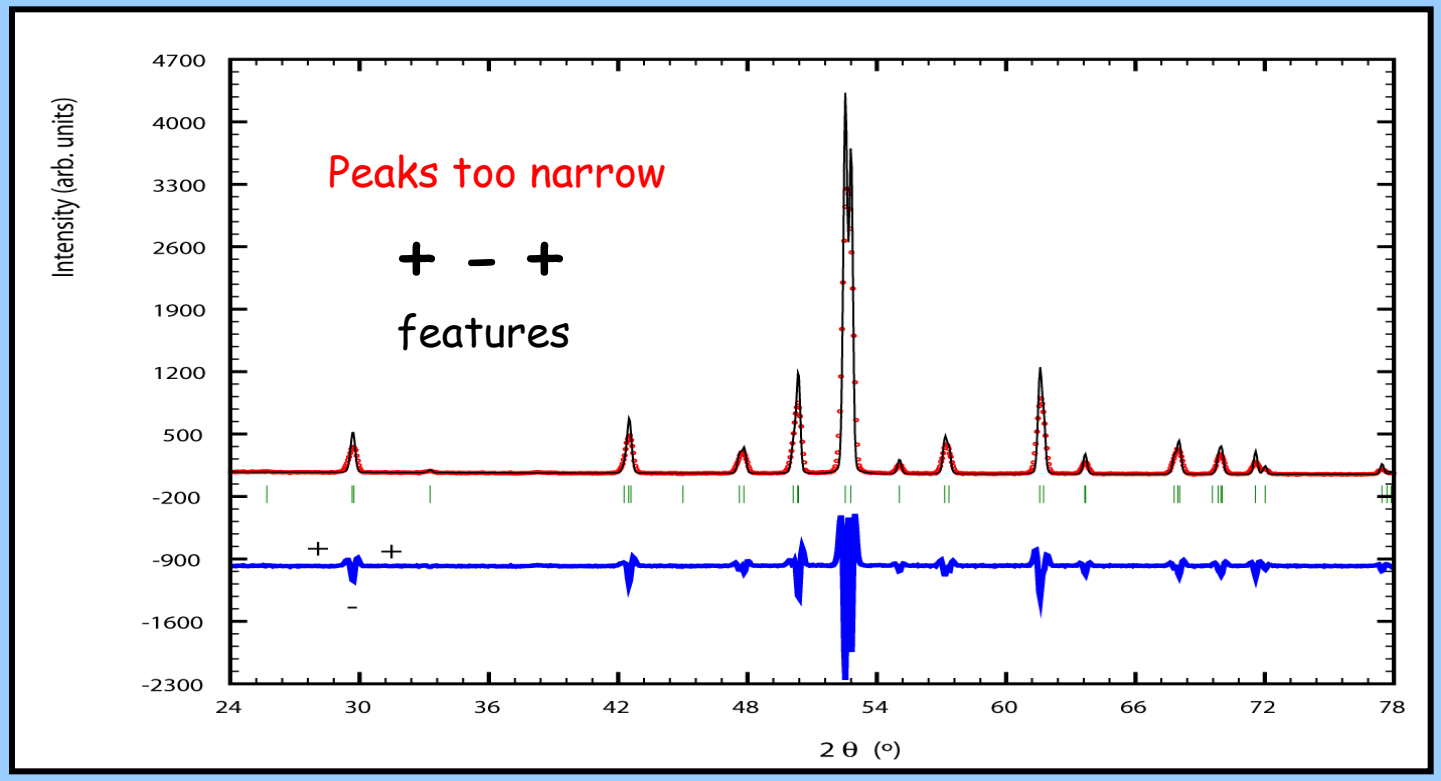


Strategy for Rietveld refinement

Refine Peakshape parameters and asymmetry parameters

Wrong lineshape parameters: Calculated peakshape too narrow

Always better to start with a too narrow peakshape! A too braod peakshape hides the real problems!

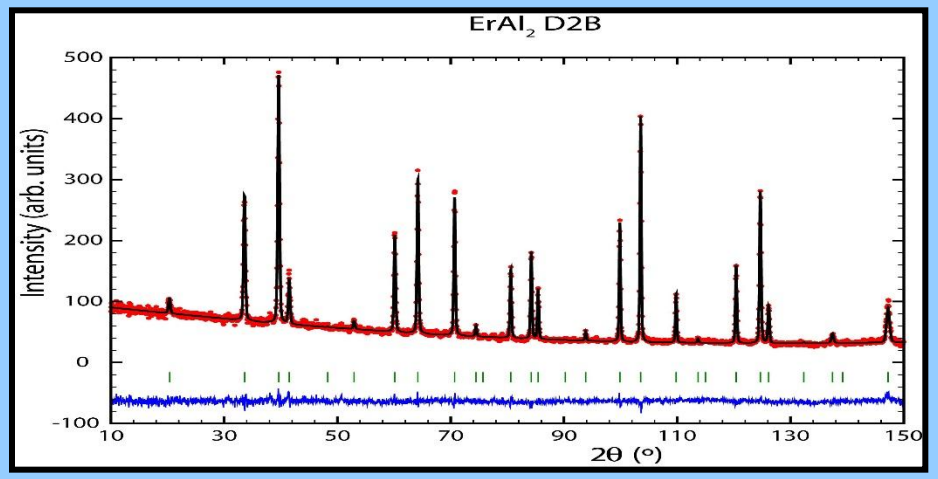


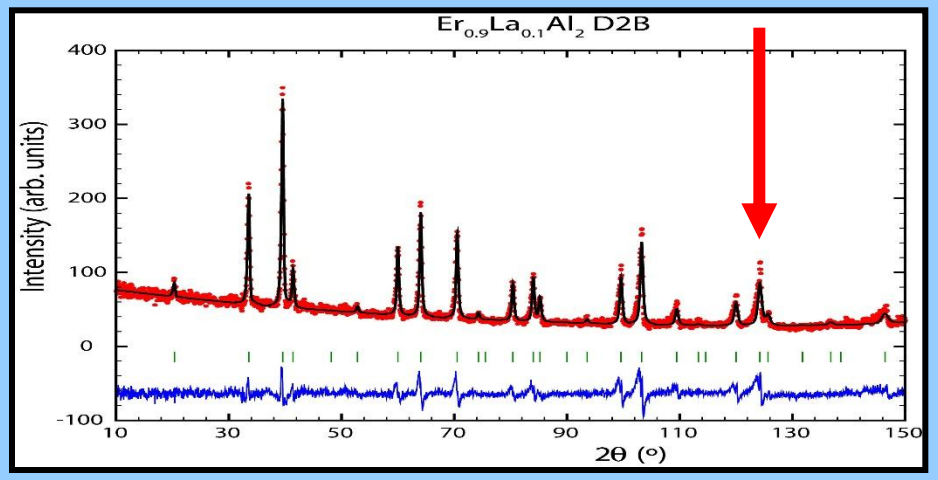
Strategy for Rietveld refinement

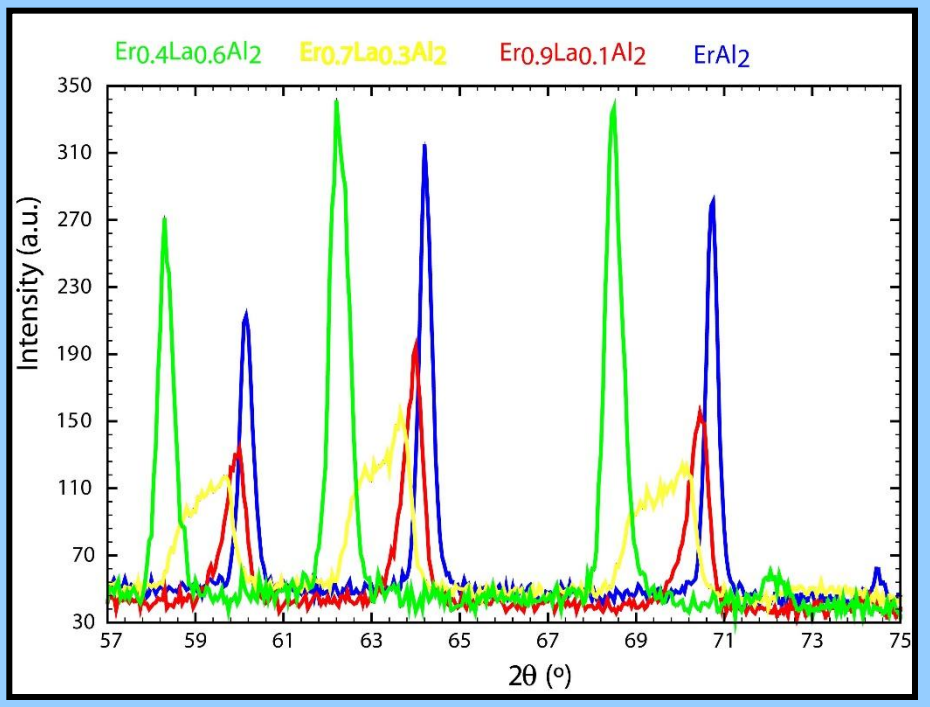
Wrong lineshape parameters: Calculated peakshape too large

R-factor = 4! But: Always look at the plot of the refinements !

The braod lineshape of the calculated pattern accounts partially for the wrong model!







Inhomogeneous system: $Er_{1-x}La_xAl_2$

No symmetry reduction but a distribution of cubic unit cell parameters

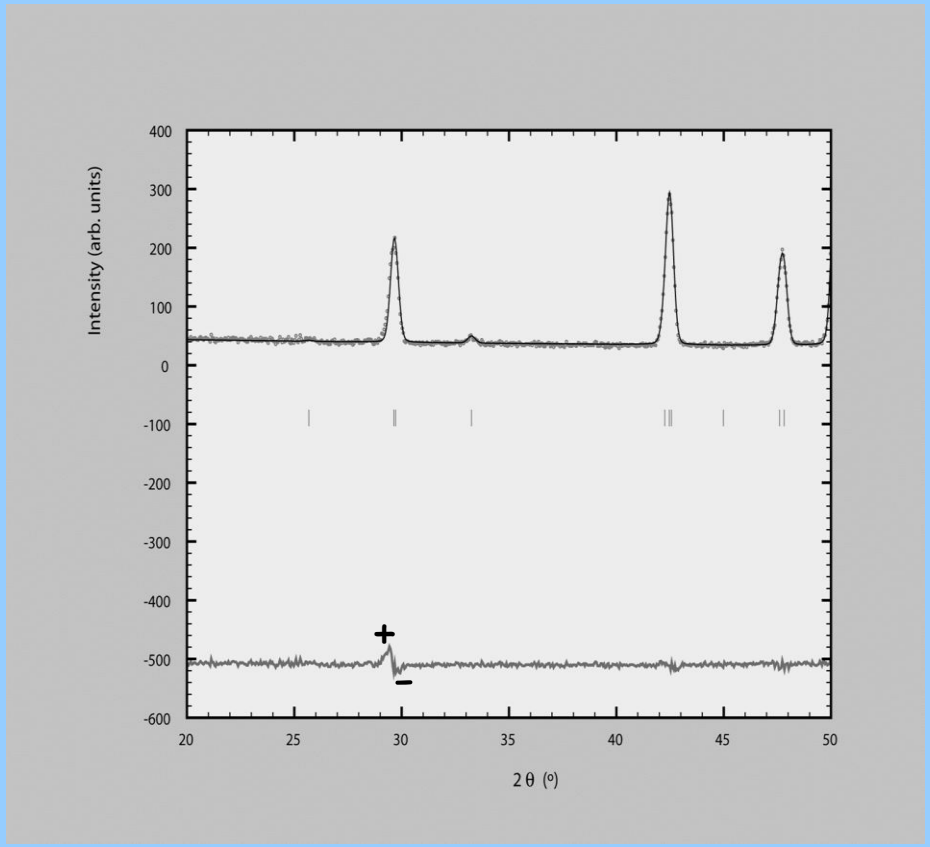
as system is not behaving as a solid solution, regions with more or less La replacing Er.

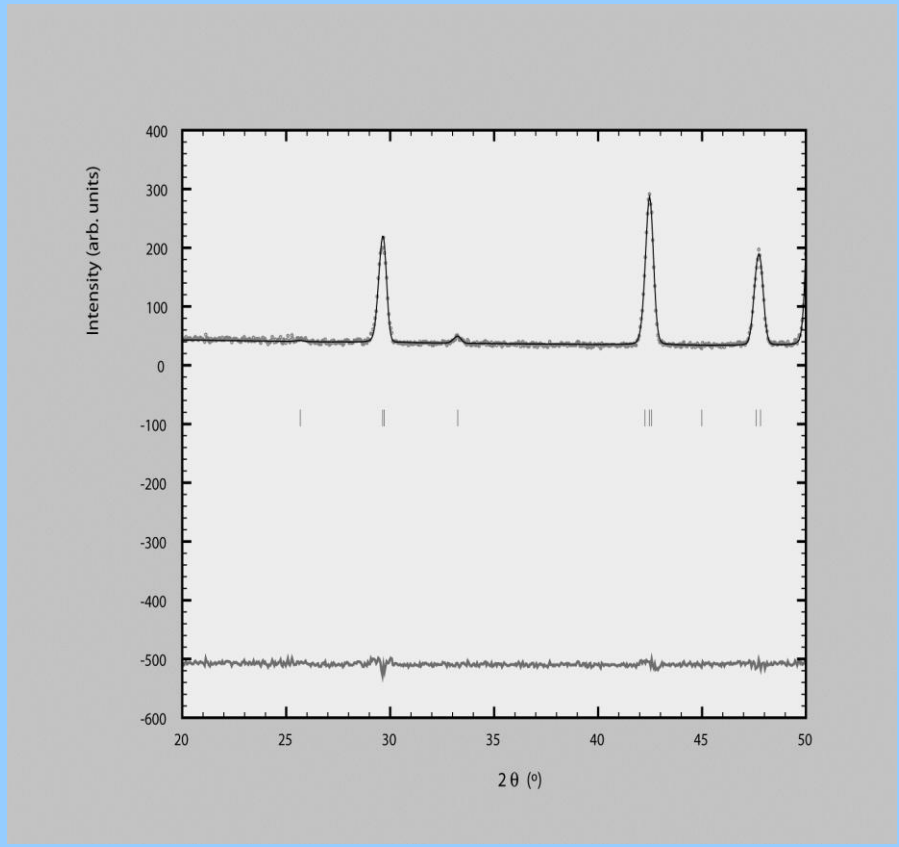
Strategy for Rietveld refinement

Influence of Asymmetry parameters

Mainly important at low angles

Typical + − pattern in the difference curve





Strategy for Rietveld refinement

Practical Example: $YFe_3(BO_3)_4$, neutron data at 295 K (1.91Å) and 520 K (1.39Å)

Already known: $TbFe_3(BO_3)_4$, recuperate the pcr file

..\..\FullProf_Suite\winplotr.exe

II Refinement

a) Literature search:

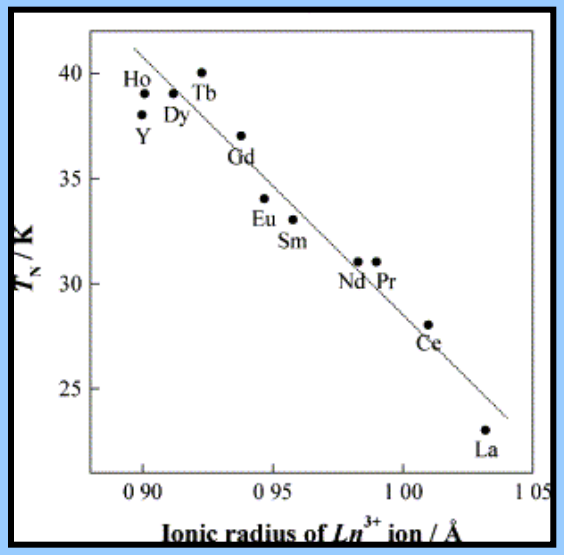
1) J.A. Campa et al., Chem. Mat. 1997, 237: Wyckoff positions are given

$RFe_3(BO_3)_4$ series: Trigonal Huntite type structure, R32,

2) Y. Hinatsu et al. J. Sol. St. Chem. **172** (2003) 438:

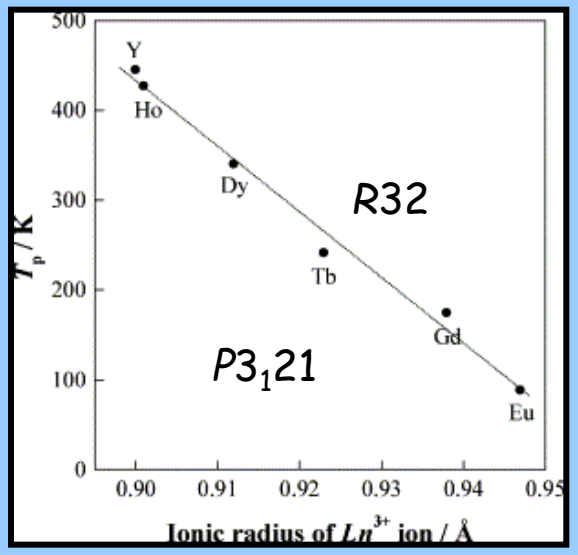
Magnetic transition temperature:

Y-compound non magnetic at RT



Crystallographic phase transition:

Y-compound at RT in same phase as Tb-compound below 240 K



3) S.A. Klimin et al., Acta Cryst. **B61** (2005) 481: Low temperature structure of $GdFe_3(BO_3)_4$ is $P3_121$

Strategy for Rietveld refinement

b) Starting Model:

In order to start refinement you need a model with lattice constants (LeBail fit), spacegroup (CheckGroup), and atom positions

Recuperate old pcr file! (one where you know that it works) and introduce your model:

Already known: $TbFe_3(BO_3)_4$, recuperate the pcr file Fp

Peakshape parameters with starting values from instrument (Standard sample).

c) "Strategy "

1) Zeroshift, wavelength, background (by hand)
2) Zeroshift, scalefactor, lattice parameters, 1rst background parameter
3) Atomic positions, isotropic temperature factor, background parameters
4) Peak shape parameters, asymmetry parameters

5) Individual B factors, atom occupancies
6) Zero displacements
7) Anisotropic B factors
8) Preferred Orientation, microstructural parameters

Strategy for Rietveld refinement

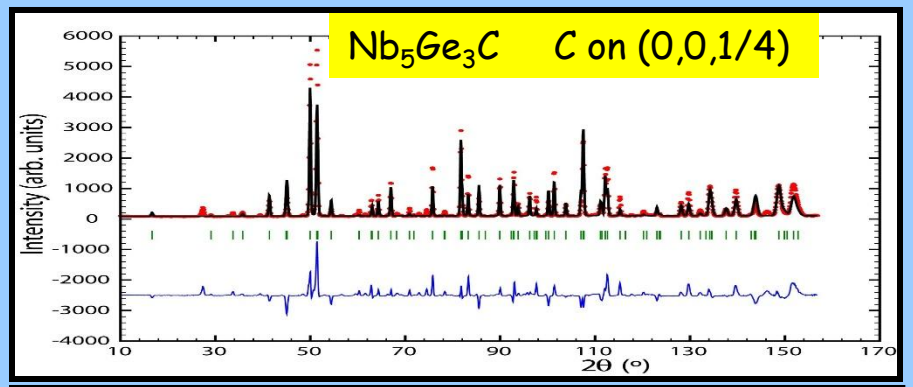
III) Some Selected Specific Problems:

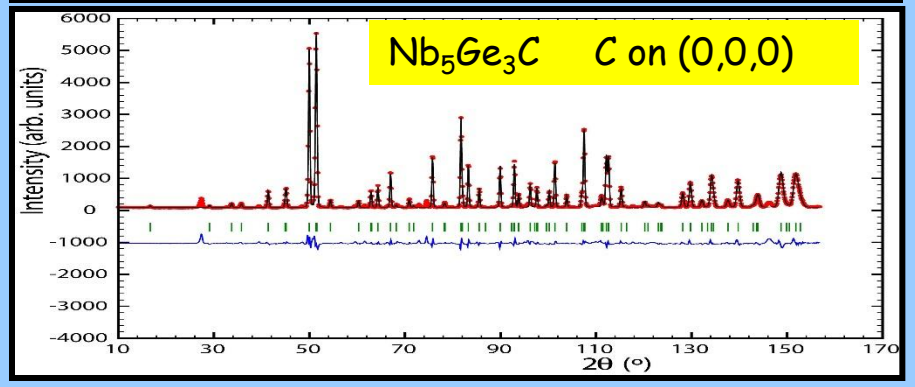
1) Wrong peak intensities, but all atom positions refined ➔ Ferromagnetic? (Fe above RT!)

```
---------------------------------------------------------------------------
 => Phase No.  1 NUCLEAR                          P 6_3/mcm
---------------------------------------------------------------------------
 =>  No. of reflections for pattern#:   1:    70
 ==> ATOM PARAMETERS:
  Name     x         sx        y         sy        z         sz       B      sB      occ.  socc.
 Nb1   0.33333 (   0)  0.66667(   0)  0.00000(   0) -0.164(103)  0.167 (  0)
 Nb2   0.23783 (  69)  0.00000(   0)  0.25000(   0) -0.164(103)  0.250 (  0)
 Ge1   0.59901 (  74)  0.00000(   0)  0.25000(   0)  0.303(126)  0.250 (  0)
 C1    0.00000 (   0)  0.00000(   0)  0.25000(   0)  0.544(  0)   0.006 (  2) should be about 0.075
```

Don't refine now anisotropic B-factors!! Will always give better R-values …

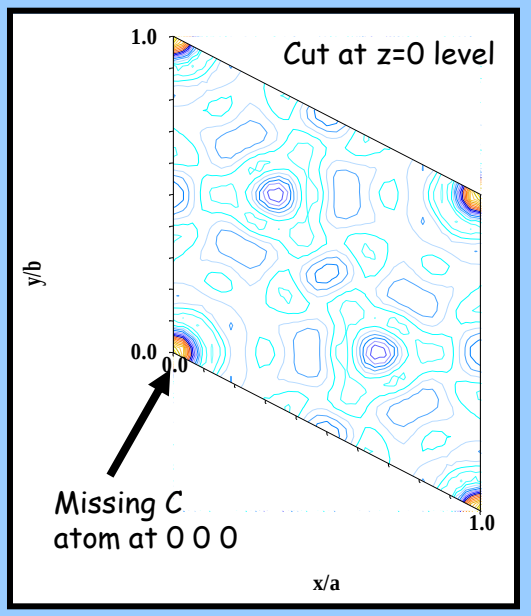
➔ Fourier Difference Map (FOU=4 in pcr file)





Pcr file with C on (0 0 ¼)

Fp



FP Toolbar

Creates file *.inp

1) Open *.inp in GFourier

2) Edit and choose Fourier Procedure: (Fo-Fc) Difference

3) Save

4) Calculations: Fourier Program

Strategy for Rietveld refinement

III) Some Selected Specific Problems:

NEUTRONS FOR SOCIETY

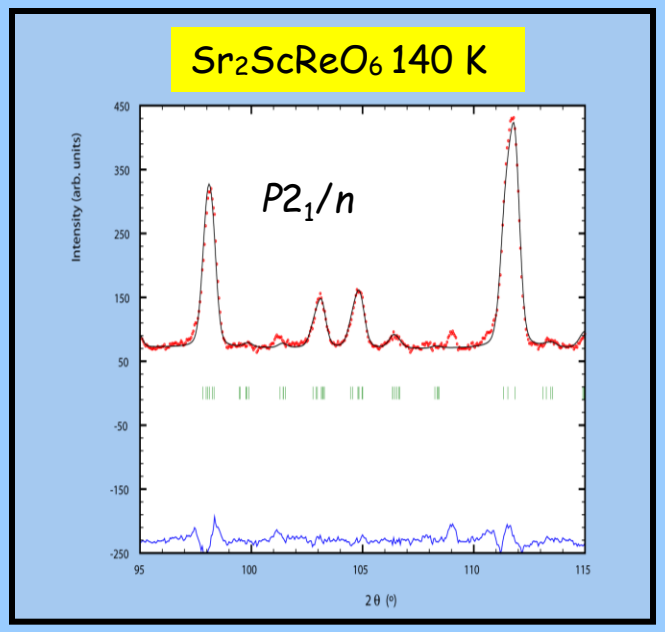
2) Peak Shape:

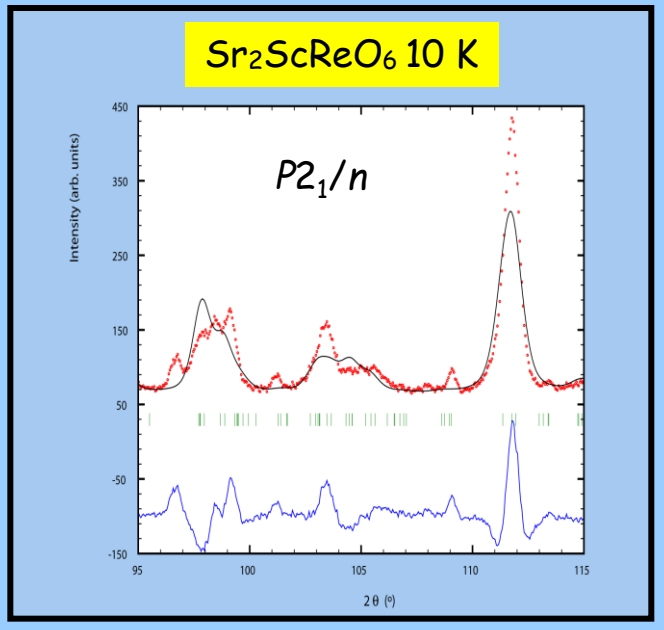
Peak Broadening, Change the model or use more parameters?

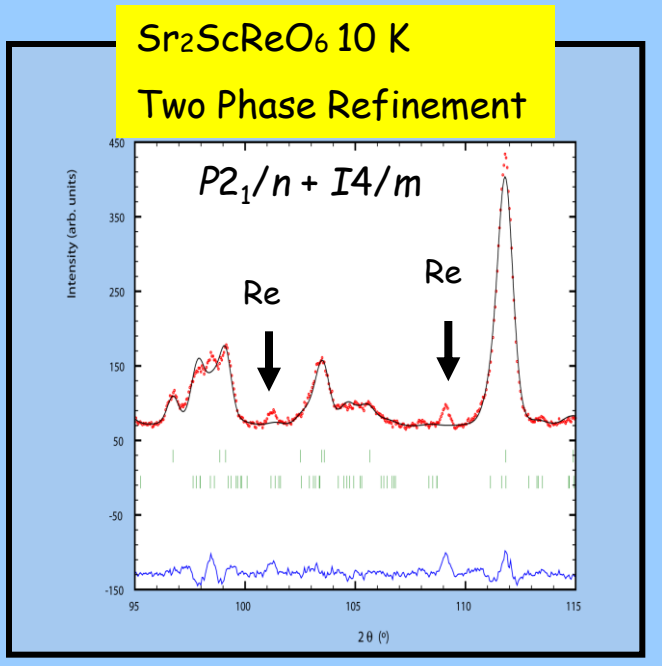
Symmetry reduction (classic), phase separation, microstrain, size effects

Phase separation: no chance of indexing, look at the evolution with temperature, peak broadening even for peaks with multiplicity 2!

Fp







Sr_2ScReO_6: Reentrant phase transition: High temperature $I4/m \rightarrow P2_1/n \rightarrow$ Low temperature $P2_1/n + I4/m$

Strategy for Rietveld refinement

III) Some Selected Specific Problems:

2) Peak Shape:

Peak Broadening, Change the model or use more parameters?

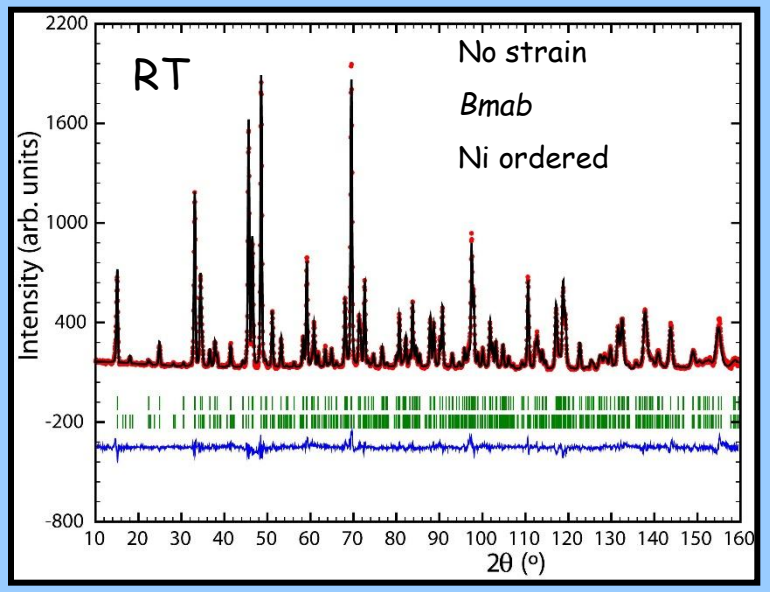
Symmetry reduction (classic), phase separation, microstrain, size effects

Microstrain, size effects, how are they visible?

Peak broadening already at low 2Θ: size effect, at high 2Θ: strain,

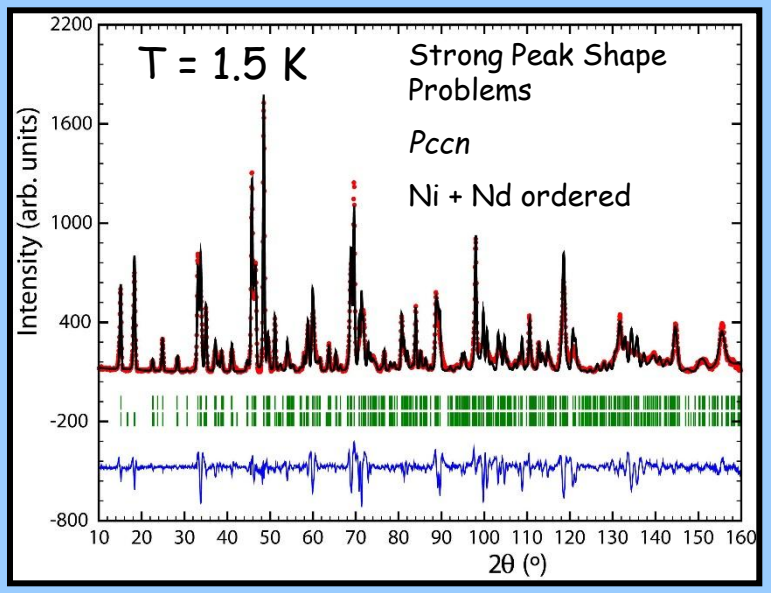
Refinement: Use the instrument resolution "irf" file (Res = 1 in pcr file) and

"U" and "X" for isotropic strain and "GauSiz" and "Y" for isotropic size



Nd_2NiO_4

Structural transition from *Bmab* at high temperature to *Pccn* at low temperature



Strategy for Rietveld refinement

III) Some Selected Specific Problems:

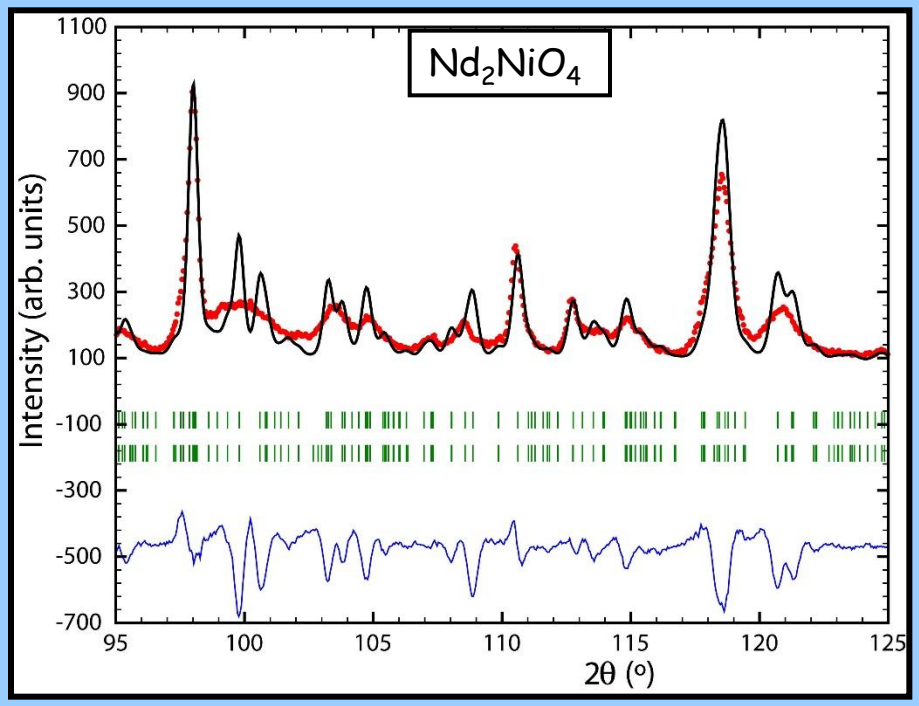
2) Peak Shape:

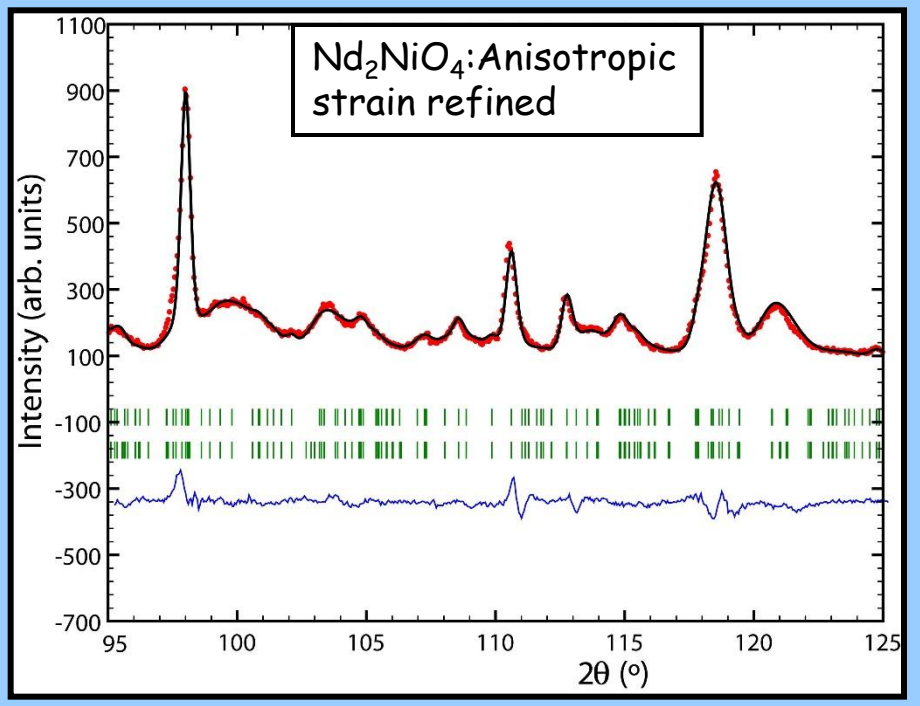
Nd_2NiO_4:

Problem linked to anisotropic Microstrain:

Some peaks are calculated to broad, others to narrow and some nearly perfectly well!

In order to refine anisotropic microstrains (or anisotropic size effects) one has to choose the correct model according to the symmetry onto which the strain is acting.





Strategy for Rietveld refinement

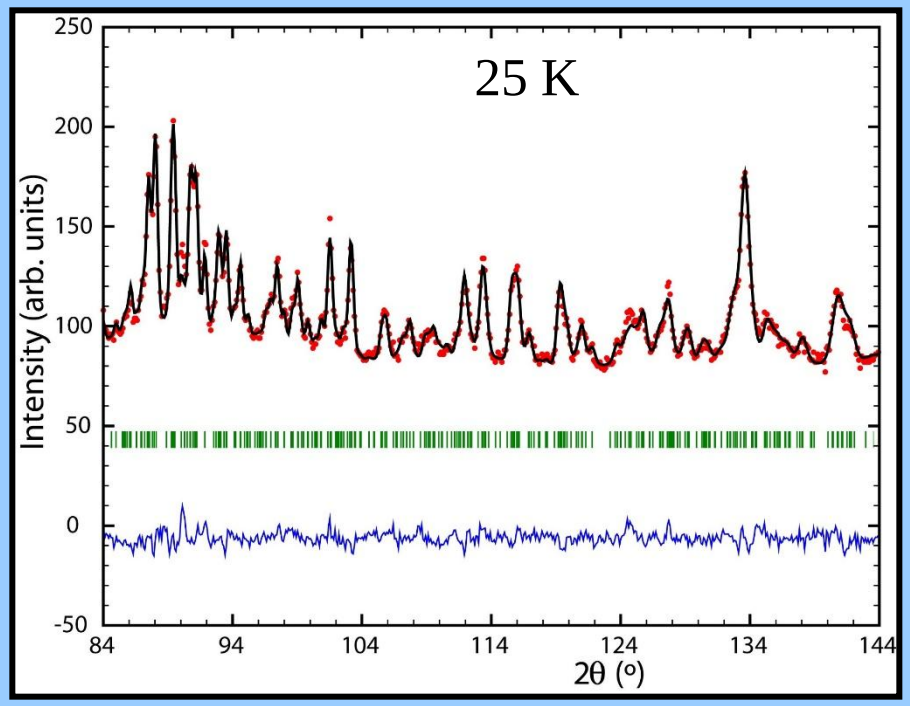
III) Some Selected Specific Problems:

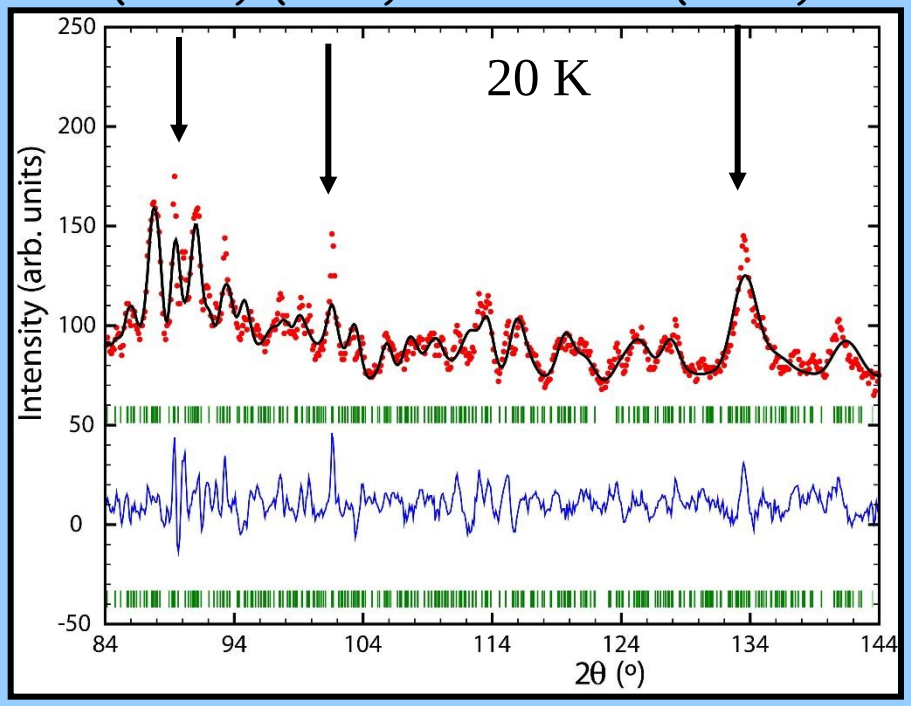
2) Peak Shape:

Ho_5Ge_4, magnetocaloric compound showing magnetostriction, orthorhombic structure:

Strong anisotropic microstrain appears as the Ho becomes magnetic

hkl dependence of peakwidth: peaks with large k are narrower





Ho_5Ge_4

Use of the Stevens notation for the description of anisotropic strain

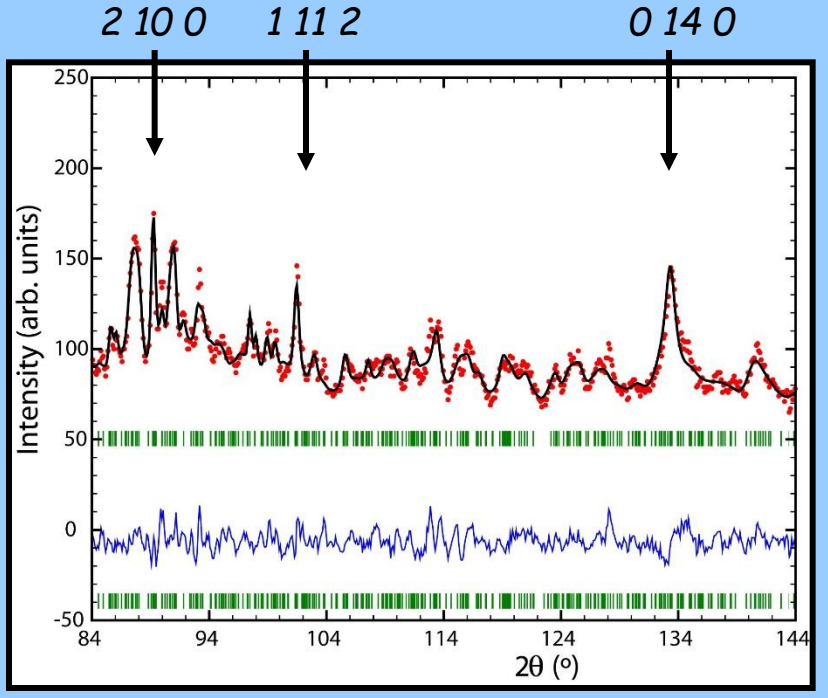
Needs the instrument resolution file as input!

```
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
  1   7   2   34   1   0   0   0   0   0   0   1   0   0   0   0   0   0   0
!
!  Resolution file for Pattern#   1
New_D1A_191.irf
.
.

!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth      ATZ    Nvk Npr More
  6   0   0  0.0 1.0 0.0   0   0   0   1   0    17501.770   0   7    0

.

.
!-------> Profile Parameters for Pattern #  1
!  Scale          Shape1       Bov        Str1       Str2       Str3      Strain-Model
 0.50723E-01   0.00000   0.00000   0.00000   0.00000   0.00000      3
   11.00000     0.000     0.000     0.000     0.000     0.000
!  U          V          W          X          Y        GauSiz     LorSiz   Size-Model
 0.000000  0.000000  0.000000  0.000000  0.000000  0.000000  0.000000    0
     0.000     0.000     0.000     0.000     0.000     0.000     0.000
!    a        b        c       alpha      beta       gamma     #Cell Info
  7.565165 14.560355  7.620977 90.000000 90.000000 90.000000
  21.00000  31.00000  41.00000   0.00000   0.00000   0.00000
! Pref1     Pref2     Asy1      Asy2      Asy3      Asy4      S_L       D_L
  0.00000  0.00000  0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
    0.00      0.00      0.00      0.00      0.00      0.00      0.00      0.00
!    S_400      S_040      S_004      S_220      S_202      S_022
    0.180832   0.003863   0.043940   0.179977  20.769495   0.178759
     141.00     161.00     121.00     131.00     111.00     311.00
! Lorentzian strain coeff.+ code
     0.17642   151.00000
```

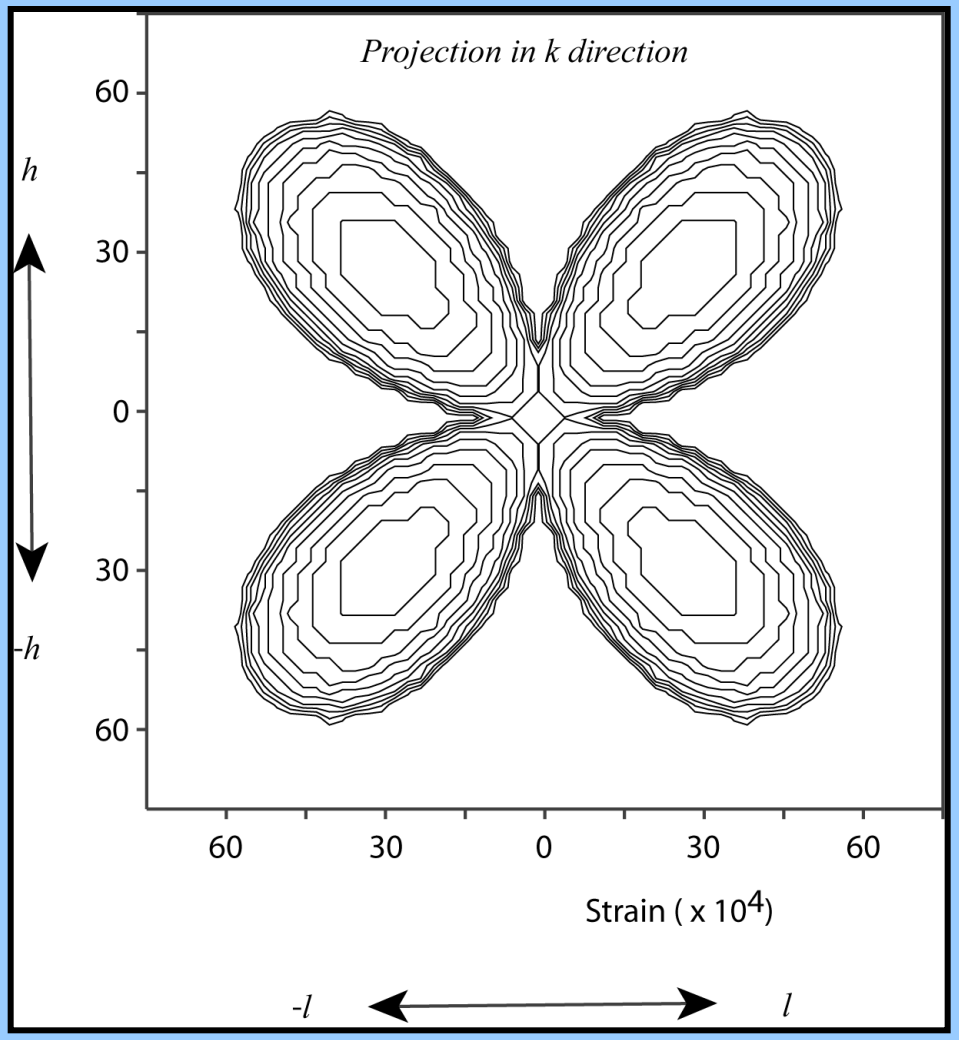
Laue Class mmm

Including anisotropic monoclinic strain:

Refinement a lot better but still not perfect



The patterns of microstrains can be visualized putting Jvi=5 in the PCR file (More=1) and reading the binary file with GFOURIER. Use projection mode.



Ho_5Ge_4

S_202 is very large: strong tendency of the compound to switch to a monoclinic structure

Synchrotron data revealed later the real spacegroup: $P2_1/m$!

Microstructural parameters (see folder "Documents"):

$$H_{hG}^2 = (U_f + (1-\xi_f)^2 D_{fST}^2(\boldsymbol{\alpha}_D))\tan^2\theta + \frac{I_{fG}}{\cos^2\theta} + H_{gG}^2$$

$$H_{hL} = (X_f + \xi_f D_{fST}(\boldsymbol{\alpha}_D))\tan\theta + \frac{[Y_f + F_f(\boldsymbol{\alpha}_S)]}{\cos\theta} + H_{gL}$$

PCR Notation:

U_f = "U" = Gaussian component of isotropic strain
X_f = "X" = Lorentzian component of isotropic strain
I_{fg} = "GauSiz" = Gaussian component of isotropic size
Y_f = "Y" = Lorentzian component of isotropic size

D_{fST}, $F_f(\alpha s)$ depend on the particular model chosen to describe anisotropic strain and size
D_{fST} in the Stevens Notation of anisotropic strain, up to 15 independent variables S_{hkl} with (h+k+l)=4
ξ = Mixing coefficient for Lorentzian contribution to anisotropic strain = "Lorentzian strain coeff."
Lorentzian component to anisotropic size, depends on "Size-Model" = "LorSiz "

Strategy for Rietveld refinement

NEUTRONS FOR SOCIETY

IV) Constraints and restraints:

Applied in order to couple parameters so that they undergo the same linear or proportional shifts or to restrain parameters to stay within in a given range.

1) Symmetry Constraints:

e.g.: Lattice parameters in a tetragonal system, code e.g.: 71.00 71.00 81.00

Atomic coordinates linked by symmetry e.g.: $x, -x, z$, code e.g.: 101.00 -101.00 111.00

or: $x, 2x, z$, code e.g.: 100.50 101.00 111.00

Scale factors of nuclear and magnetic phases having different unit cells,

with e.g.: a b c (nuc) and 2a 2b c (mag): code 11.0000 (nuc) and 10.0625 (mag)

Refining a low symmetric structure with limited data (forcing e.g. part of a framework of atoms to keep a higher symmetry, e.g.: charge ordered structures with Mn^{3+} and Mn^{4+} in $Tb_{0.5}Ca_{0.5}MnO_3$:

Pnma with *a b c* has 7 positional parameters,

$P2_1/m$ with *2a b c* has 31 positional parameters. Nowadays: Use Amplimodes to refine distortions

Spin direction in highly symmetric cases, e.g.: hexagonal system: refine only *Mx* and *Mz* as powder data don't allow the determination of the moment direction within the basal plane.

Strategy for Rietveld refinement

NEUTRONS FOR SOCIETY

IV) Constraints and restraints:

2) Constraints due to direct correlation, e.g.:

Wavelength or lattice parameters (refine one or the other)

Scale factor or magnetic moment value in a purely magnetic phase (using a difference data set)

Scale factor and site occupancies (has to be fixed for one site)

General Remark 1:

Put Ana = 1 in pcr file for analysis of Refinement:

Pay attention to message" Correlation of special kind" indicates coupling of strongly correlated = wrongly coupled parameters.

General Remark 2:

Some correlations are very strong, e.g.: zero shift and sample displacement.

This will increase your standard deviations. If interested in relative behaviour of parameters as function of e.g. T keep some fixed.

General Remark 3:

When you do a sequential refinement using data from a stationary multidetector (e.g. D20, D1B) you refine only once the zeroshift using one data set and then you fix it.

Strategy for Rietveld refinement

IV) Constraints and restraints:

3) Linear Restraints:

E.g.: Partial site occupancies (total must be fixed, not negative, with several sites …)

```
                x         y         z         B        Occ
CR  CR    0.00000   0.00000   0.00000   0.00000   0.01563  0  0  0  2
          0.00      0.00      0.00      0.00      211.00
RE  RE    0.00000   0.00000   0.00000   0.00000   0.00190  0  0  0  3
          0.00      0.00      0.00      0.00      221.00
FE  FE    0.00000   0.00000   0.00000   0.00000   0.00331  0  0  0  3
          0.00      0.00      0.00      0.00      271.00
RE  RE    0.50000   0.50000   0.50000   0.00000   0.01372  0  0  0  3
          0.00      0.00      0.00      0.00      231.00
FE  FE    0.50000   0.50000   0.50000   0.00000   0.00190  0  0  0  3
          0.00      0.00      0.00      0.00      281.00
CR  CR    0.50000   0.50000   0.50000   0.00000   0.00520  0  0  0  2
          0.00      0.00      0.00      0.00      241.00
```

Occupation of site =
Site Multiplicity/ Multiplicity of the general site
Fm3m: 4/192 = 0.02083 if full

Site 1

Site 2

$Sr_2CrFe_{0.25}Re_{0.75}O_6$ with Cr,Fe and Re distributed over 2 cation sites (4*a*: 0 0 0 and 4*b*: $\frac{1}{2}\ \frac{1}{2}\ \frac{1}{2}$):

Introduce linear restraints, NLI in pcr file, NLI = 5 (second line of pcr-file)

```
!  Set of  5 linear restraints:
!  Identifier, number of coeff, value, sigma / List of coeff & Parameters

Site_a   3      0.020830      0.0000001
 1.0000 21   1.0000 22   1.0000 27          (Limits the occupation on site 0 0 0 to 0.02083 = FULL)
Site_b   3      0.020830      0.0000001
 1.0000 23   1.0000 24   1.0000 28          (Limits the occupation on site ½ ½ ½ to 0.02083 = FULL)
Chemcomp  2      0.020830      0.0000001
 1.0000 21   1.0000 24                      (Limits the total content of Cr on both sites to 0.02083 = 1)
Chemcomp  2      0.015620      0.0000001
 1.0000 22   1.0000 23                      (Limits the total content of Re on both sites to 0.01562 = 0.75)
Chemcomp  2      0.005210      0.0000001
 1.0000 27   1.0000 28                      (Limits the total content of Fe on both sites to 0.00521 = 0.25)
```

Constraint pcr

Fp

Strategy for Rietveld refinement

NEUTRONS FOR SOCIETY

IV) Constraints and restraints:

4) 'Strategic' Constraints: Used in order to assure a successful refinement
(can be lifted as the refinement proceeds)

In theory not needed if the data would be perfect in intensity, resolution and q-range.

In powder always limitations: Same q-value for different reflections (5 5 3, 7 1 1 in cubic system)
Multiplicity of reflections (311, 131, 113…)

Typical 'strategic' constraints:

One overall temperature factor for all atoms
The same temperature factor for all atoms of the same type

Magnetic moment values of different magnetic sites kept alike

Limit the possible Spin directions (basis vectors)

Refining a magnetic structure using high intensity/low resolution data:

Keep all atom coordinates fixed if the crystallographic structure had been determined before from high resolution data.

Again: Keep the zeroshift fixed in a sequential refinement!!

Strategy for Rietveld refinement

IV) Constraints and restraints:

5) Restraints: Helps the refinement program not to get trapped in false minima
Very important for e.g. simulated annealing

```
P 21/n                    <--Space group symbol
!Atom Typ      X        Y        Z     Biso      Occ     In Fin N_t Spc /Codes
Sr   SR     0.87930  0.41798  0.73560  1.55853  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
C1   C      0.63200  0.23920  0.57800  1.48511  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
C2   C      0.58300  0.51640  0.09300  1.48511  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
C3   C      0.64800  0.27780  0.34700  1.48511  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O1   O      0.62200  0.22870  0.15700  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O2   O      0.67300  0.28130  0.75600  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
Ow3  O      0.85600  0.56610  0.57600  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O4   O      0.77900  0.50850  0.07000  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O5   O      0.57500  0.16760  0.56900  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O6   O      0.52400  0.55480  0.26500  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
O7   O      0.69400  0.34880  0.32900  1.24468  1.00000   0   0   0   0
            0.00     0.00     0.00     0.00     0.00
H1   D      0.77449  0.06512  0.01765  2.00000  1.00000   0   0   0   0
           11.00    21.00    31.00     0.00     0.00
H2   D      0.81474  0.75317  0.49374  2.00000  1.00000   0   0   0   0
           41.00    51.00    61.00     0.00     0.00
H3   D      0.64942  0.10371  0.81023  2.00000  1.00000   0   0   0   0
           71.00    81.00    91.00     0.00     0.00
```

Hydrated acid strontium oxalate $Sr(HC_2O_4)\cdot\frac{1}{2}(C_2O_4)\cdot H_2O$, deuterated $SrC_3O_7D_3$

Where is the Hydrogen?

Keep the known framework fixed

Coordinates of 3 H atoms free

NRE in pcr file = number of restraints

Put NRE = 9 (first line of pcr-file)

Nine Restraints to be put into the pcr-file:

! Limits for selected parameters (+ steps & BoundCond for SA):

```
1     0.0000    1.0000    0.0152   1    X_H1
2     0.0000    1.0000    0.0073   1    Y_H1
3     0.0000    1.0000    0.0264   1    Z_H1
4     0.0000    1.0000    0.0279   1    X_H2
5     0.0000    1.0000    0.0080   1    Y_H2
6     0.0000    1.0000    0.0323   1    Z_H2
7     0.0000    1.0000    0.0334   1    X_H3
8     0.0000    1.0000    0.0087   1    Y_H3
9     0.0000    1.0000    0.0346   1    Z_H3
```

Strategy for Rietveld refinement

IV) Constraints and restraints:

6) Soft distance (or angles) constraints

Running Fullprof with the option of calculating distances and angles (Jdi = 3, needs More=1 in line starting with Nat) will not only produce a file called *.dis with all the interatomic distances and angles, but as well a file called CFML_Restraints.tpcr.

pcr file

```
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth      ATZ    Nvk Npr More
  7   0   0  0.0 0.0 1.0   0   0   0   0    0      967.370  0   5    1
!Jvi Jdi Hel Sol Mom Ter  Brind   RMua    RMub    RMuc   Jtyp  Nsp_Ref Ph_Shift N_Domains
  0   3   0   0    0    0 1.0000  0.0000  0.0000  0.0000  1       0        0         0
! Max_dst(dist) (angles)  Bond-Valence Calc.
     3.6000     2.6000       BVS
!  N_cations   N_anions     Tolerance(%) / Name or cations/ and Anions
       3           1            0.00
PR+3 FE+3 B3+
O-2
```

Fp

CFML_Restraints.tpcr

YFe3BO34disconstraints.pcr

YFe3BO34disconstraints.dis

The file CFML_Restraints.tpcr contains in the appropiate format distance or angle restraints which can be pasted into the pcr file. This is useful if you want to restrain a certain type of interatomic distances (or angles) to be the same

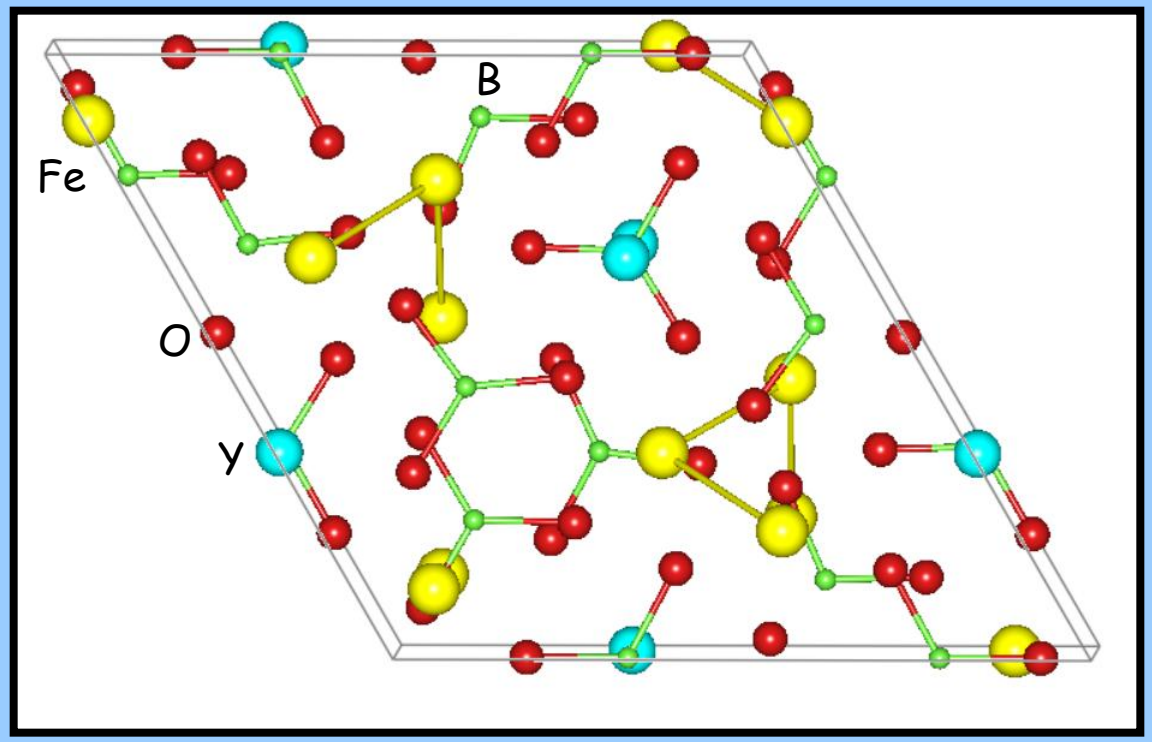
```
 => Help for possible angle restraints around atom FE2
FE2   O2   O2   32  33   0.0000 -1.0000  0.6667  -1.0000 -1.0000  0.3333  46.36   0.18
FE2   O2   O4   32  25   0.0000 -1.0000  0.6667   0.0000  0.0000  0.0000   5.53   0.18
FE2   O2   O5   32  26   0.0000 -1.0000  0.6667   0.0000 -1.0000  0.3333  41.58   0.24
FE2   O2   O5   32  27   0.0000 -1.0000  0.6667   0.0000  0.0000  0.6667  51.04   0.24

At1  At2  ITnum    T1       T2       T3          DIST   SIGMA
FE2  FE2   26  -1.00000  -1.00000  0.33333     3.1853 0.0049
FE2  FE2   27   0.00000  -1.00000 -0.33333     3.1853 0.0044
FE2  FE2   33   0.00000   0.00000  0.33333     4.4180 0.0053
FE2  Y     25   0.00000   0.00000  0.00000     3.8107 0.0073
```

In the pcr file you have to change as well the parameters DIS or MOM to the number of restraints you introduced.

Strategy for Rietveld refinement

IV) Constraints and restraints:

6) Soft distance (or angles) constraints



Vesta to non-constrained

Vesta to constrained

Example of $YFe_3(BO_3)_4$:

Groups of BO_3

R_{Bragg} = 3.7	unconstrained
R_{Bragg} = 3.9	B-O distances constrained to be equal

NEUTRONS FOR SOCIETY

Thank you for your attention!