Crystallography course SCHEDULE

		Date		Topic
	1.	Wed	28.02.	Lec-1: Introduction
	2.	Mon	04.03.	Lec-2: Crystal Chemistry & Tolerance parameter
	3.	Mon	04.03.	EXERCISE 1
	4.	Wed	06.03.	Lec-3: Crystal Chemistry & BVS
	5.	Fri	08.03.	Lec-4: Symmetry & Point Groups
	6.	Mon	11.03.	EXERCISE 2
	7.	Wed	20.03.	Lec-5: Crystallography & Space Groups (Linda) Ke3
	8.	Fri	22.03.	Lec-6: XRD & Reciprocal lattice (Linda) Ke4
	9.	Mon	25.03.	EXERCISE 3 (Linda) Ke4
TODAY	10.	Thu	04.04.	Lec-7: Rietveld (Linda) 12:15-14, Ke3
	11.	Fri	05.04.	EXERCISE 4: Rietveld (Linda)
		Mon	08.04.	EXERCISE 4: Rietveld (Linda)
	12.	Thu	11.04.	Lec-8: ND & GI-XRD 12:15-14, Ke3
	13.	Fri	12.04.	Lec-9: XRR (Topias)
	14.	Mon	15.04.	EXERCISE 5: XRR (Topias)
		Wed	17.04.	EXERCISE 5: XRR (Topias)
	15.	Mon	22.04.	Lec-10: Synchrotron radiation & XAS & EXAFS
	16.	Thu	25.04.	Mössbauer 12:15-14, Ke3
	17.	Fri	26.04.	EXERCISE 6
	18.	Mon	29.04.	Seminars:
	19.	Fri	03.05.	Seminars:
	20.	Mon	06.05.	ADDITIONAL DISCUSSION/QUESTION POSSIBILITY

LINDA’S LECTURES & EXERCISES

- 20.03. WEDNESDAY: 14.15 – 16 (Ke3)
- 22.03. FRIDAY: 12:15 – 14 (Ke3)
- 25.03. MONDAY: 14.15 – 16 (Ke4) *Exercise session*
    - *Exercise deadline 27.3 at 13:00*

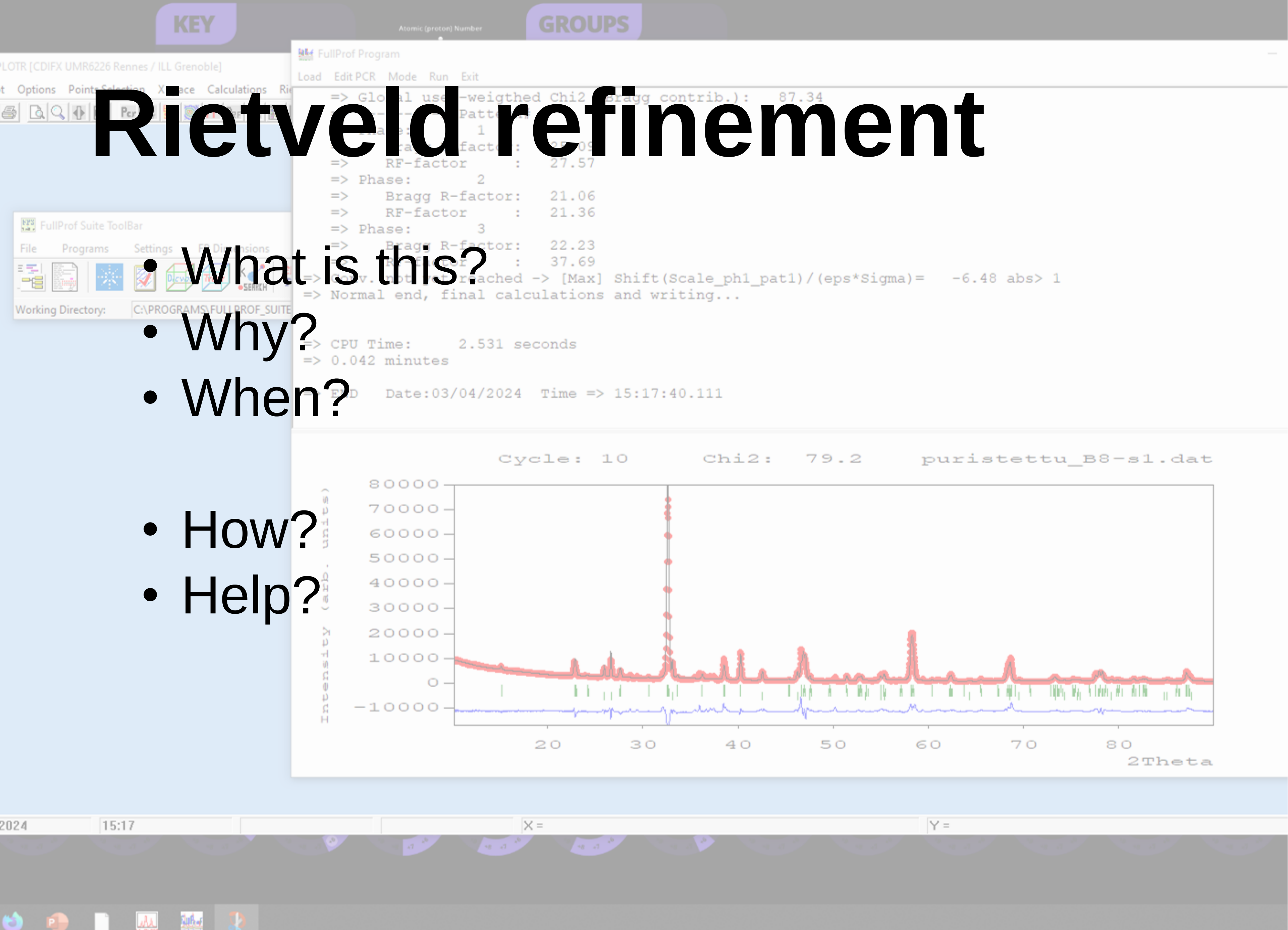
Easter holidays break! ☺

- 04.04. THURSDAY: 12:15 – 14 (Ke3)
- 05.04. FRIDAY: 12:15 – 14 (Ke4) *Rietveld exercise 1*
    - *Getting started on FullProf*
- 08.04. MONDAY: 14:30 – 16 (Ke4) *Rietveld exercise 2*
    - *Help with finishing the exercise*
    - *Exercise deadline on 10.4. at 13:00*

Rietveld refinement

- What is this?
- Why?
- When?

- How?
- Help?



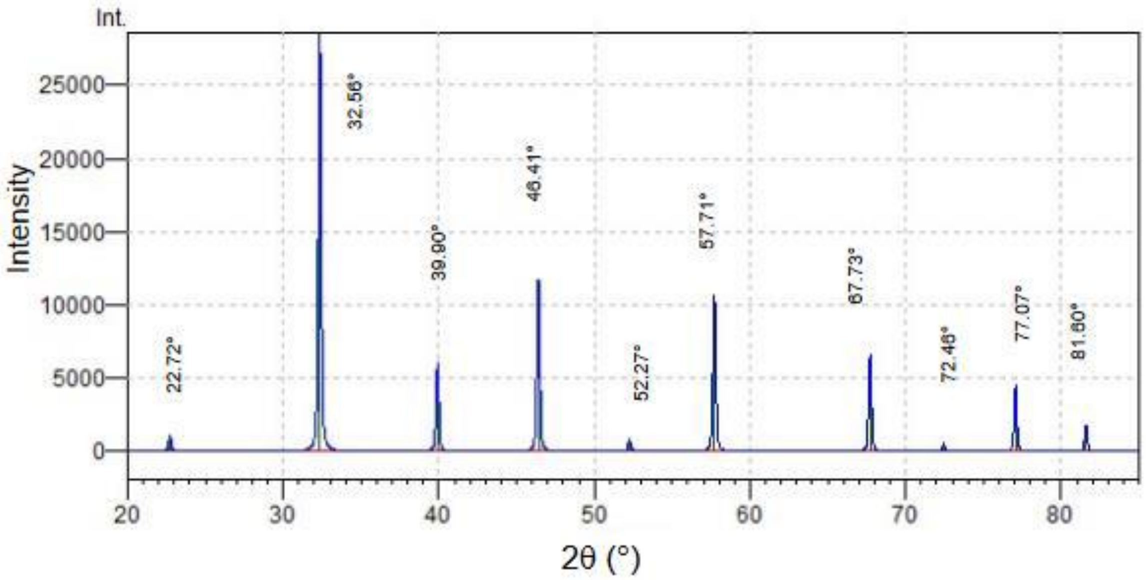
```
=> Global user-weigthed Chi2 (Bragg contrib.):   87.34
=>                     Patte
=> Phase:       1
=>    Bragg R-factor:   25.09
=>    RF-factor      :   27.57
=> Phase:       2
=>    Bragg R-factor:   21.06
=>    RF-factor      :   21.36
=> Phase:       3
=>    Bragg R-factor:   22.23
=>    RF-factor      :   37.69
=> Conv. not yet reached -> [Max] Shift(Scale_ph1_pat1)/(eps*Sigma)=   -6.48 abs> 1
=> Normal end, final calculations and writing...

=> CPU Time:     2.531 seconds
=> 0.042 minutes

=> END  Date:03/04/2024  Time => 15:17:40.111
```

Understanding XRD data

- We did peak indexing
  - ... of structures with known lattice types

EXERCISE 3.7.

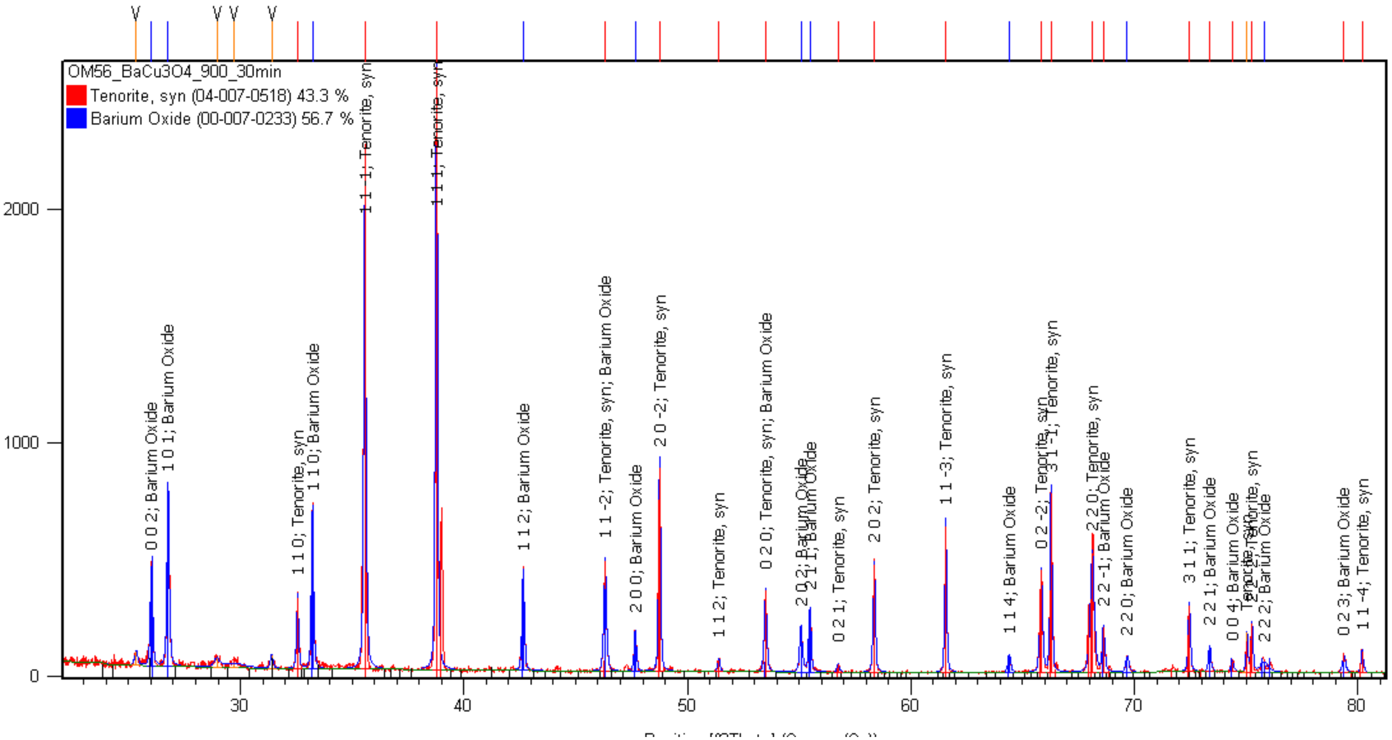
Please index the diffraction pattern below; the unit cell is cubic. What are the lattice parameters and what is the centering? λ=1.5406 Å (Cu Kα)

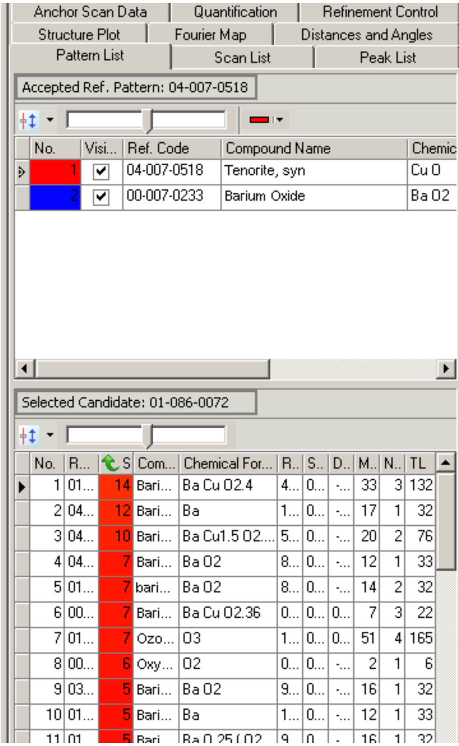


Understanding XRD data

- Database matching for phase identification

  ... when the structures are characterized before





Accepted Ref. Pattern: 04-007-0518

No.	Visi...	Ref. Code	Compound Name	Chemic
🟥 1	☑	04-007-0518	Tenorite, syn	Cu O
🟦 2	☑	00-007-0233	Barium Oxide	Ba O2

Selected Candidate: 01-086-0072

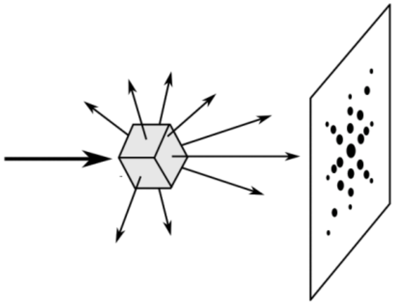
No.	R...	S	Com...	Chemical For...	R..	S..	D..	M..	N..	TL
1	01...	14	Bari...	Ba Cu O2.4	4...	0...	-...	33	3	132
2	04...	12	Bari...	Ba	1...	0...	-...	17	1	32
3	04...	10	Bari...	Ba Cu1.5 O2....	5...	0...	-...	20	2	76
4	04...	7	Bari...	Ba O2	8...	0...	-...	12	1	33
5	01...	7	bari...	Ba O2	8...	0...	-...	14	2	32
6	00...	7	Bari...	Ba Cu O2.36	0...	0...	0...	7	3	22
7	01...	7	Ozo...	O3	1...	0...	0...	51	4	165
8	00...	6	Oxy...	O2	0...	0...	-...	2	1	6
9	03...	5	Bari...	Ba O2	9...	0...	-...	16	1	32
10	01...	5	Bari...	Ba	1...	0...	-...	12	1	33
11	01	5	Bari	Ba O.25 (O2	9	0	-	16	1	32

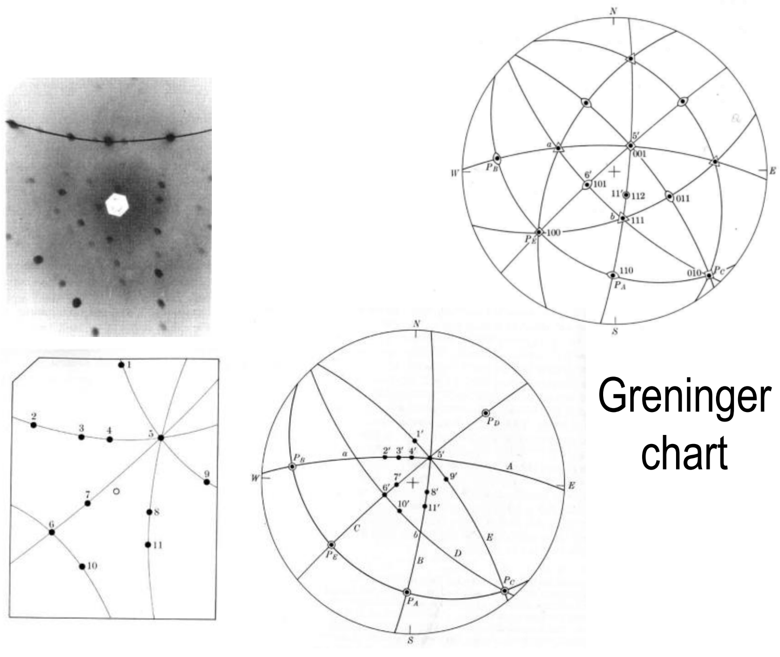
When the structure is unknown...?

- The hard but precise method: single-crystal XRD
- Can identify the crystal system and symmetry functions:

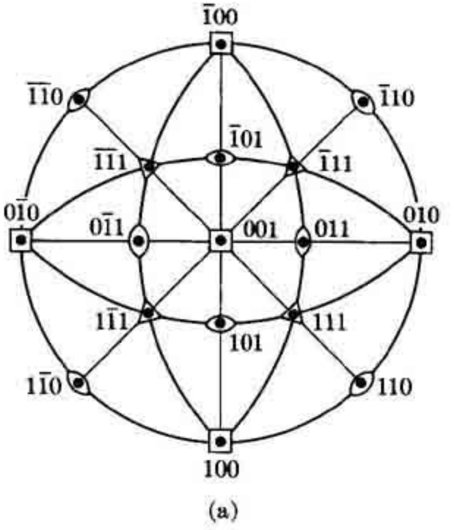
stereographic projection of the symmetry operations map: Laue diagram

- Very time consuming





Greninger chart



Stereographic projection of SG #225 Fm3m

Hugo M Rietveld

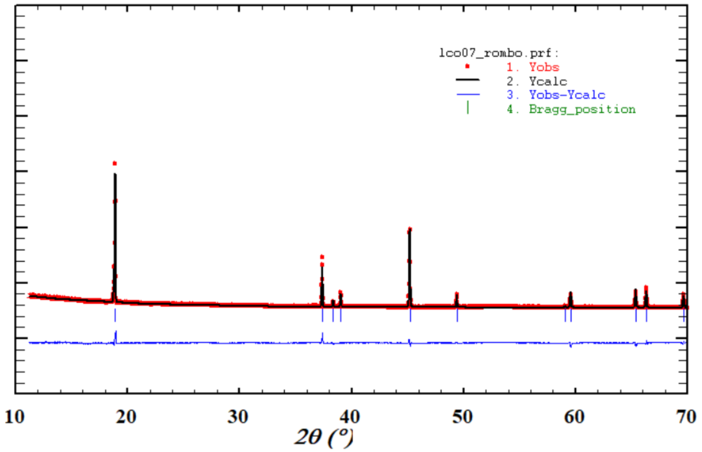
- Developed the Rietveld method in the 60’s
- Structural refinement from POWDER diffraction
- Most used method to analyze or determine the crystal structure of inorganic solids!
- Originally developed for neutron diffraction data, but modified it is applicable to (lab) X-rays too
- Is performed mainly with sharewares, produced in research centers like ILL, Los Alamos, ISIS, Spring8…also commercial software available

To “refine” diffraction data is a method used for single crystal data whereas Rietveld method is for powder diffraction data, they are very different. Both X-rays and neutrons can be used.

What is Rietveld refinement?

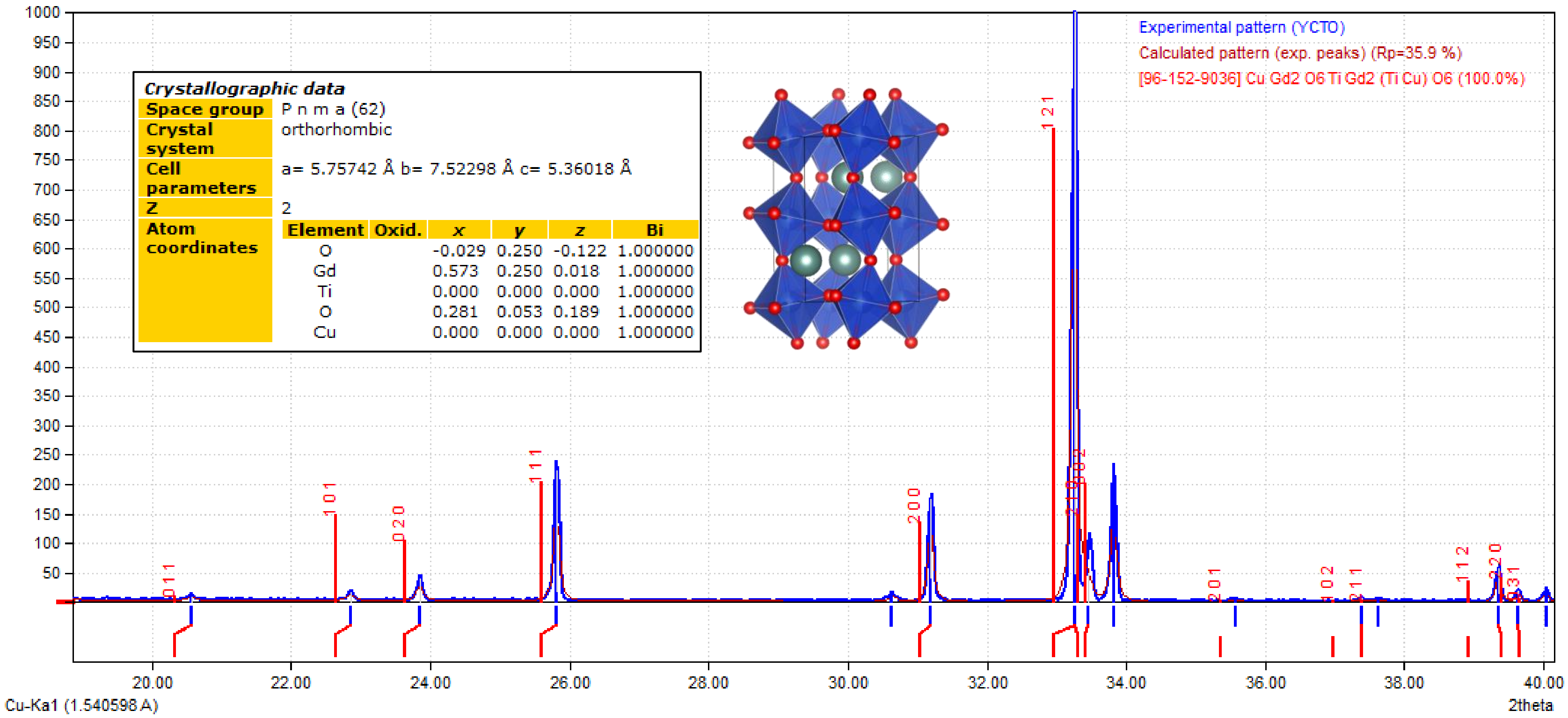
- Methodical trial-and-error calculation to find the structure
- Based on experimental XRD data of your sample
- Needs a complete structural model as a starting point
- Steps:

1. Compare calculated intensity from the model to observed intensity in the collected data
2. Optimize parameters in the model (= refine) until the calculated pattern fits the observed pattern (or it's obvious that the model is wrong)
3. Try different models until you find one that fits and makes chemical sense (atom positions, bonding, coordination)



Example: Y_2CuTiO_6

- The perovskite phase Y_2CuTiO_6 was never made before
- Chemically related phase found: Gd_2CuTiO_6
- Use this structure as starting point, adjust to match my data



Crystallographic data

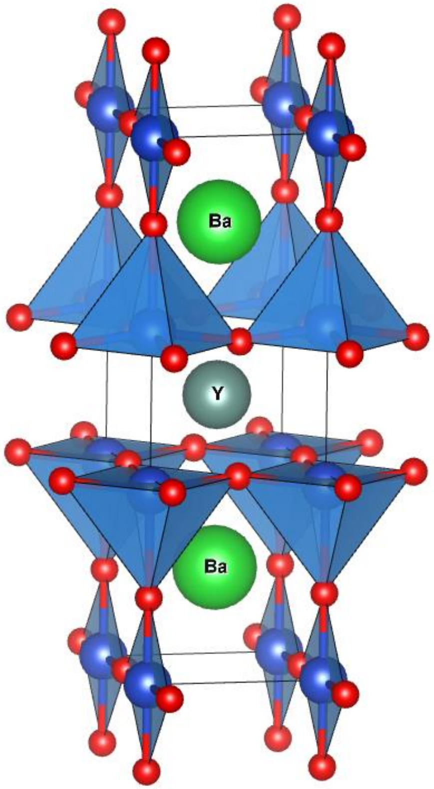
Space group	P n m a (62)					
Crystal system	orthorhombic					
Cell parameters	a= 5.75742 Å b= 7.52298 Å c= 5.36018 Å					
Z	2					
Atom coordinates	**Element**	**Oxid.**	***x***	***y***	***z***	**Bi**
	O		-0.029	0.250	-0.122	1.000000
	Gd		0.573	0.250	0.018	1.000000
	Ti		0.000	0.000	0.000	1.000000
	O		0.281	0.053	0.189	1.000000
	Cu		0.000	0.000	0.000	1.000000

L Sederholm, T Tiittanen, M Karppinen. High-pressure stabilisation of R = Y member of R2CuTiO6 double perovskite series. 2023. JSSC 317 part A, 123646. https://doi.org/10.1016/j.jssc.2022.123646

Why is Rietveld important?

- Allows us to study a precise structure of a material
  - Crystal structure is the key to material properties!
  - Properties known => applications designed
- Before the method, it was practically compulsory to grow a single crystal (SC) to solve its crystal structure
  - SCs are often very hard to prepare
- $YBa_2Cu_3O_7$ discovery (late 1980's) brought Rietveld alive: its crystal structure was determined earlier from powder than SC

  => for first time **powder beats crystals**!
- However, solving crystal structure using the method is complicated, mainly used to analyze samples



Powder diffraction challenge

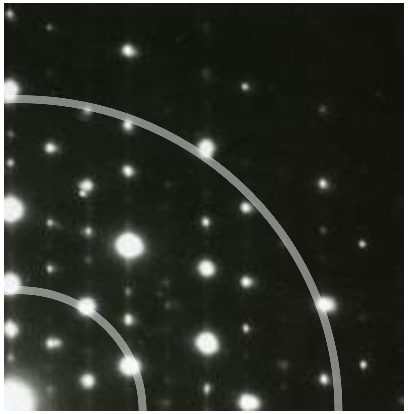
- Powder XRD is a 1D representation of a 3D system:
  - Describes scattering from all the atomic planes in the unit cell

  = exactly the information we want!

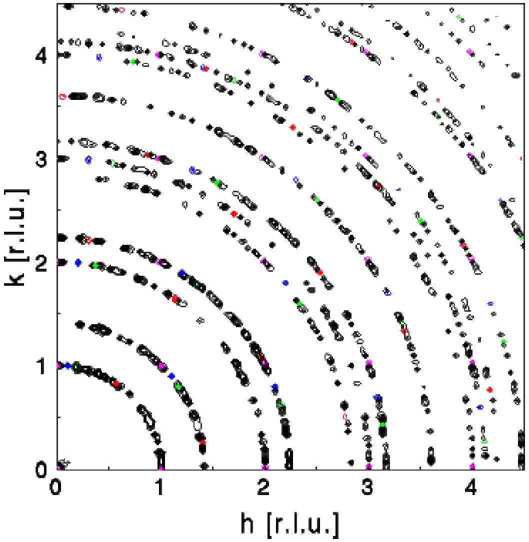
but…

- Only $\mathbf{F}_{hkl}^2$ ($|\mathbf{F}_{hkl}|$) is known, phase angle information is lost
- **overlapping reflections**; multiple *hkl* can produce the same *d* value

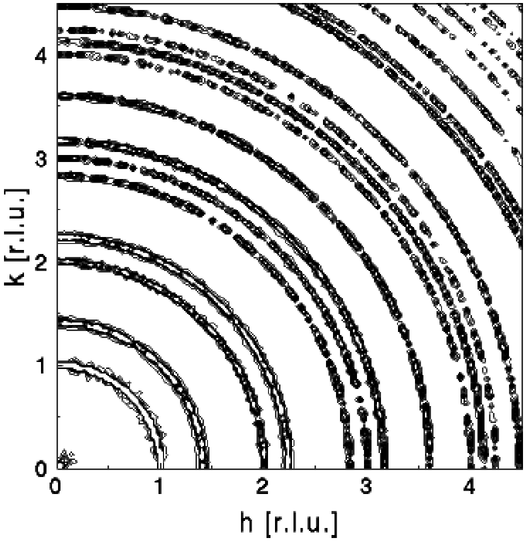
  => Structure determination from powder requires some pre-information



Single crystal



40 single crystals



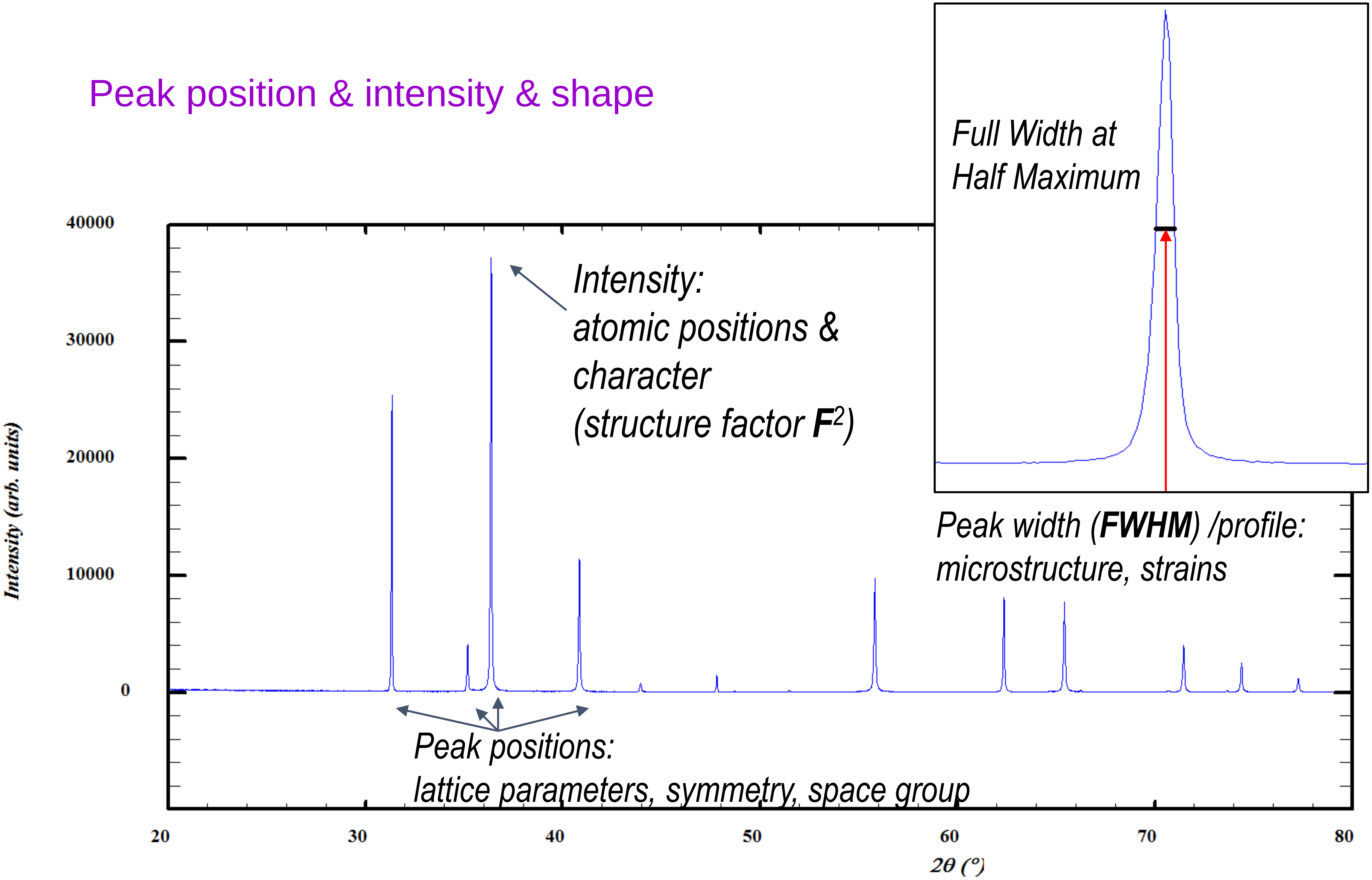
200 single crystals

*each "ring" corresponds to a particular reciprocal lattice-vector **G** in the sample crystal(s)*

Information in XRD data

Information from an XRD pattern

Peak position & intensity & shape



Intensity:
atomic positions &
character
*(structure factor **F**^{2})*

Full Width at
Half Maximum

*Peak width (**FWHM**) /profile:*
microstructure, strains

Peak positions:
lattice parameters, symmetry, space group

How Rietveld refinement calculates

The model is refined against data by using the Least Squares method to minimize the sum:

$$S_y = \sum_i w_i (y_i - y_{ci})^2$$

y_i = observed intensity at 2θ i
y_{ci} = calculated intensity at 2θ i
$w_i = 1/y_i$ at 2θ i

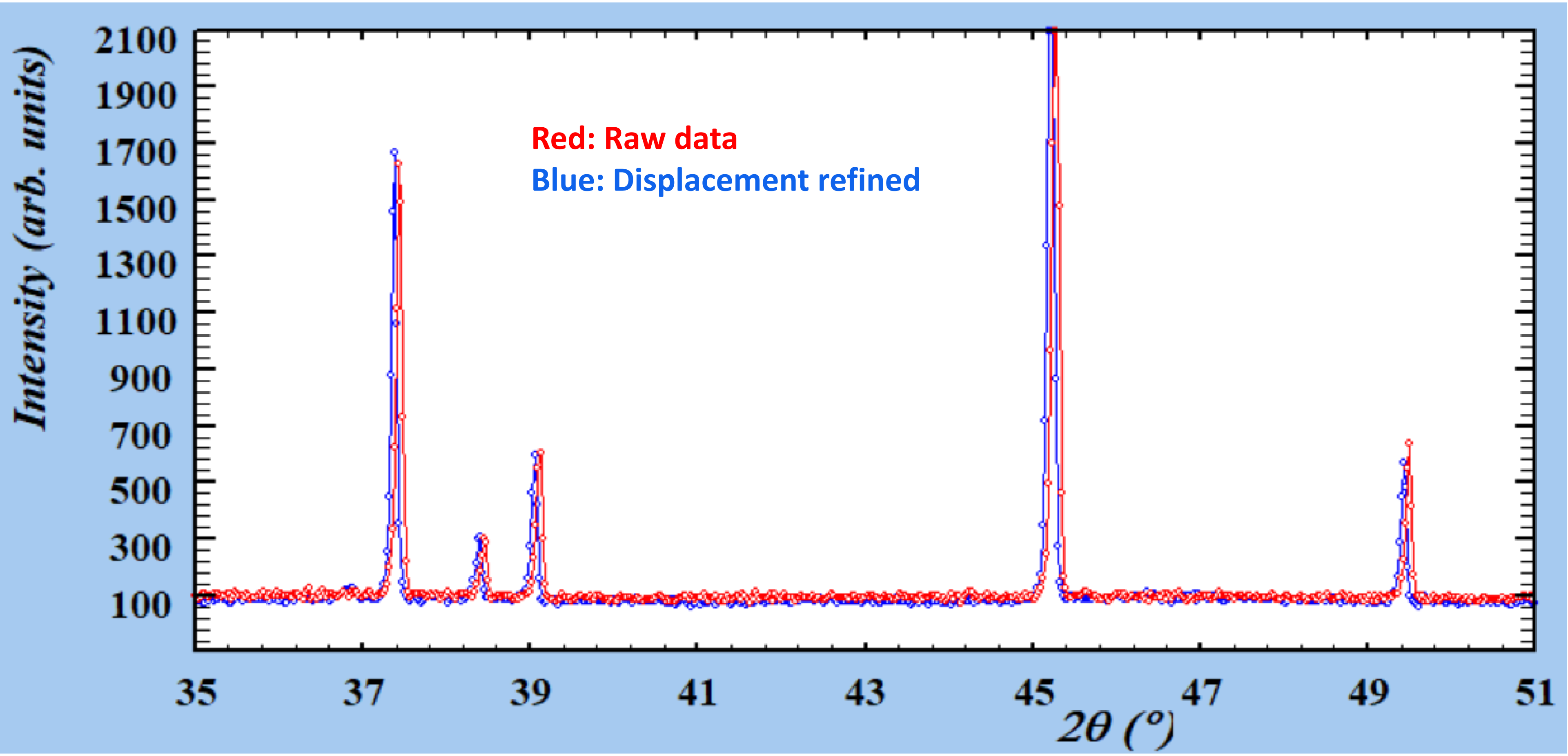
$$\boxed{y_{ci} = s\sum_{hkl} L_{hkl}\left|F_{hkl}\right|^2 \phi(2\theta_i - 2\theta_{hkl}) P_{hkl} A + y_{bi}}$$

hkl = Miller indices of reflection
s = scale factor
F_{hkl} = structure factor
ϕ = profile function for peak

P = preferred orientation function
A = absorption term
y_{bi} = background intensity at i

Method developed for neutron data → works rather well with X-rays too

Peak position: Sample displacement error



Red: Raw data

Blue: Displacement refined

Peak position: Sample displacement error

- When the sample is not in the calibrated focus circle, the beams converge at incorrect position

→ observed peak position is wrong

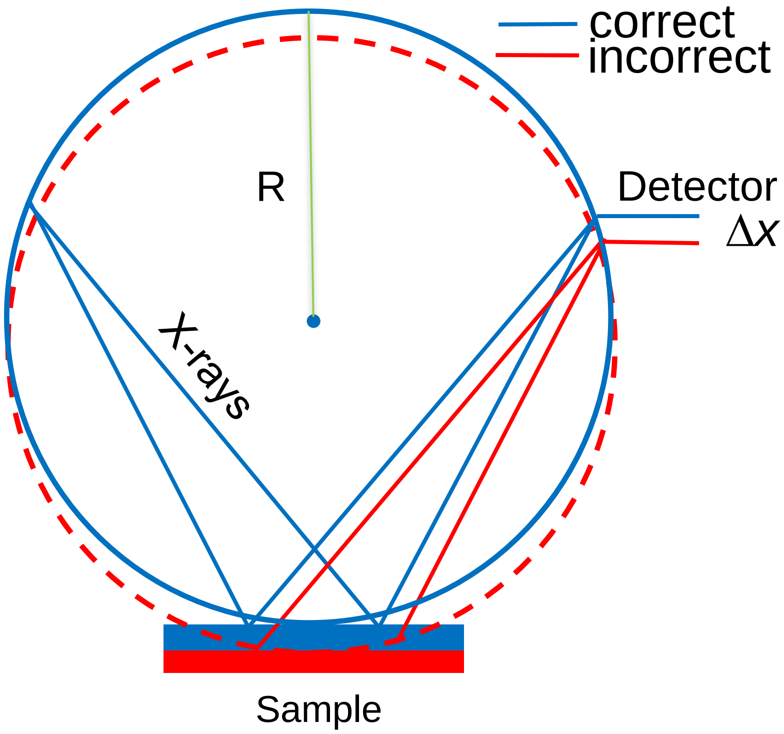
- Most common source of error in lab X-ray measurements
- Systematic, cos-dependent:

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

We will refine these

s = shift, R = goniometer radius

(at 2θ=28.4°, s = 0.006 rad => 0.08°peak shift)



- Integrated in the Lorentz factor L* in the Rietveld refinement

$$y_{ci} = s\sum_{hkl} \boxed{L_{hkl}^{*}} |F_{hkl}|^{2} \phi(2\theta_i - 2\theta_{hkl}) P_{hkl} A + y_{bi}$$

Peak intensity

$$\mathbf{I}_{hkl} = sLp\mathbf{F}_{hkl}^2$$

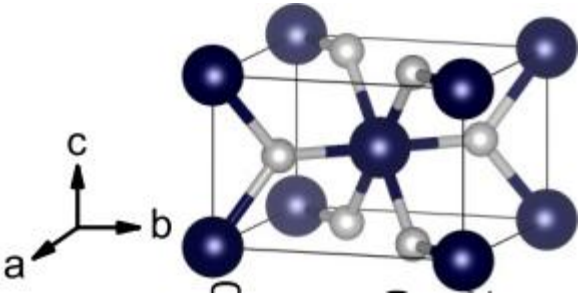
$\mathbf{I}_{hkl}$ = intensity of a Bragg reflection (”peak”) from plane *hkl*
s = scale factor
L = Lorentz factor *(corrects for geometry of experiment, also sample disp.)*
p = Polarisation
$\mathbf{F}_{hkl}$ = The structure factor for given reflection plane

sLp are *instrument based*, thus $\mathbf{I}\ \boldsymbol{\alpha}\ \mathbf{F}^2$

→ Normalize the data and sLp can be ignored

Peak intensity: Structure factor

$$\mathbf{F}_{hkl} = \sum_j \mathbf{f}_j \mathbf{N}_j e^{2\pi i(\mathbf{hx}_j + \mathbf{ky}_j + \mathbf{lz}_j)} e^{-M_j}$$



Where

f_j = *atomic scattering factor*

N_j = *occupancy factor of the atom on the site*

x_j, y_j, z_j = *fractional coordinates of atom "j"*

$M_j = 8\pi^2 U_{iso}^2 \sin^2\theta / \lambda^2$

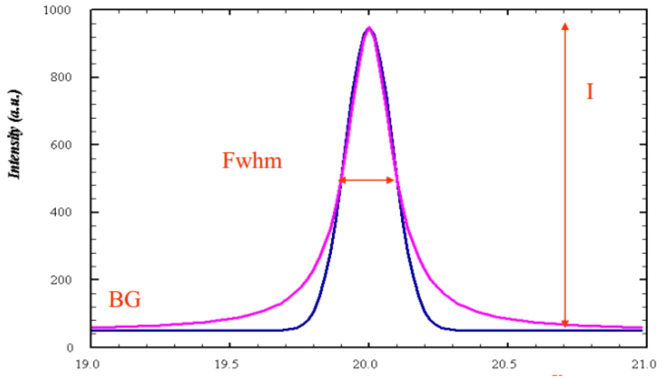
U_{iso} = *square root of the mean thermal parameter*

Describes scattering from *all the atoms in the unit cell*
= Exactly the information that we want in analyses of crystal materials

But not explicitly solvable for powder measurements

Peak shape: width

Sample properties and instrument itself cause the broadening of a diffraction line



- Both effects are superimposed

  → FWHM from raw data is never solely from the sample.
- Rietveld method takes this into account; effect unique for each equipment and sample
- The value of FWHM is sinθ dependent
- Belongs to profile function ϕ

Full
Width at
Half
Maximum

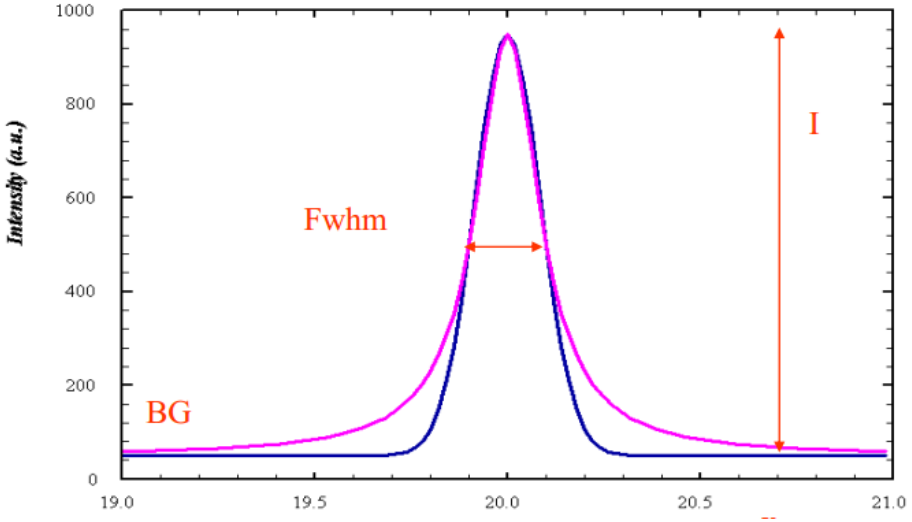
$$y_{ci} = s\sum_{hkl} L_{hkl}\left|F_{hkl}\right|^2 \overset{*}{\boxed{\phi}}(2\theta_i - 2\theta_{hkl})P_{hkl}A + y_{bi}$$

Peak shape: profile functions

- Most commonly used model is a linear combination of Gaussian and Lorentzian functions (named the *pseudo-Voigt* profile function)
- Roughly FWHM(x) = $\alpha H_G(x) + (1-\alpha)H_L(x)$, where

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

$$H_L = X\tan\theta + \frac{Y}{\cos\theta}$$



and where

G = Gaussian, L = Lorenzian, I = intensity, θ = scattering angle.

U,V,W,X,Y = refinable parameters

Peak shape: profile parameters (ϕ*)

Profile parameters U, V, W, I_G, X, Y describe FWHM of the Gaussian and Lorentzian components

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

$$H_L = \enclose{circle}{X}\tan\theta + \frac{Y}{\cos\theta}$$

U = instrument + strain (value: positive)

V = instrument only (always negative)

W = instrument only (positive)

X = instrument + strain (positive)

Y = instrument + size (positive)

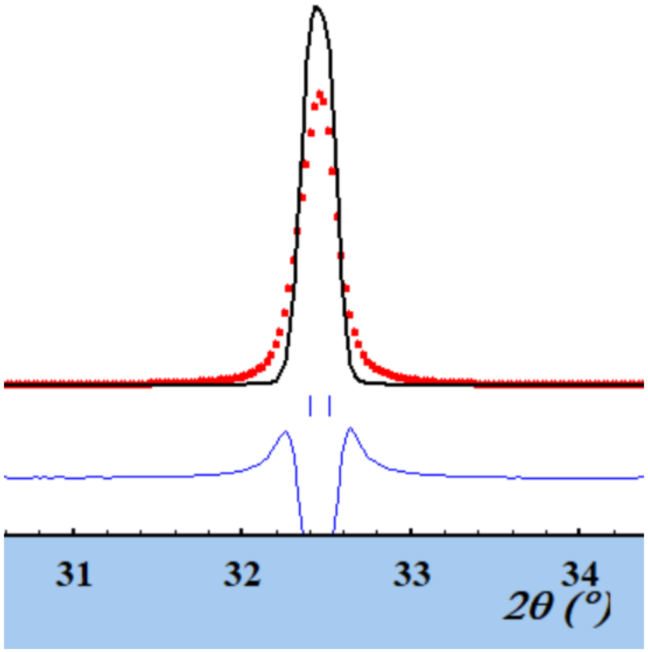
$$y_{ci} = s\sum_{hkl} L_{hkl}\left|F_{hkl}\right|^2 \overset{*}{\boxed{\phi}}(2\theta_i - 2\theta_{hkl})P_{hkl}A + y_{bi}$$

Problem: the profile functions and coefficient values for PXRD instruments are not well-documented and depend on the software used (&%# § !)

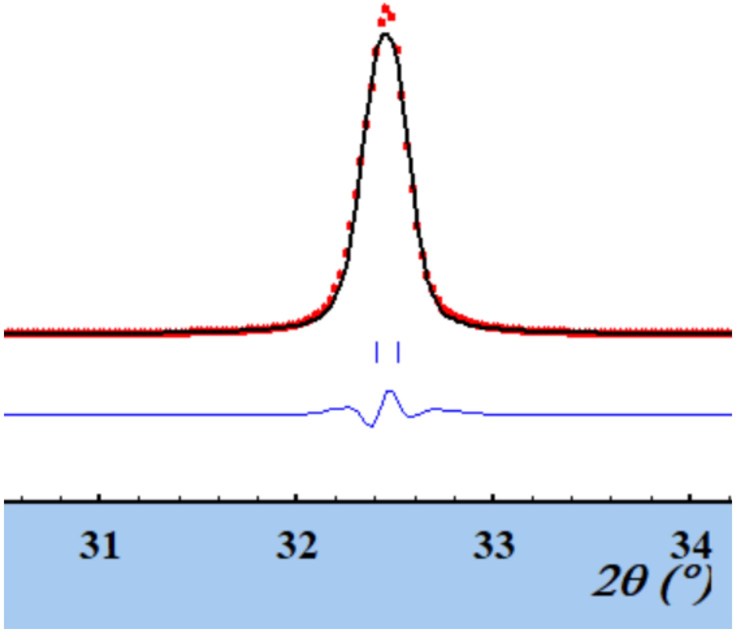
Peak shape: profile parameters (ϕ*)

Example of effects on profile

U, X with starting values

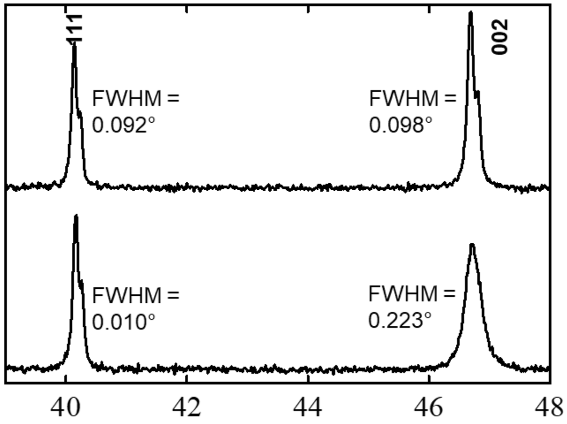


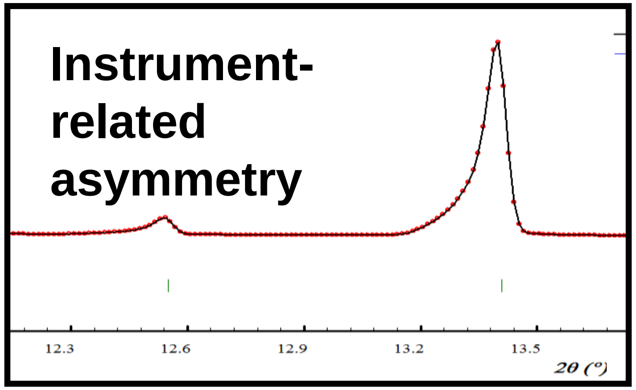
U, X refined



Peak shape: informative features

- Line (“peak”) broadening analysis (FWHM value) gives information on **sample microstructure** (crystallite size, strain) and **texture**
  - The sharper the line, the larger the crystallites
- Wrong shape distorts e.g. lattice parameter values
- At low 2θ angles, **instrument-related asymmetry** is observed, typically vanishes around 2θ ~ 30-40° (slits, axial divergence,…)
- Left-side asymmetry after that is commonly sample-related: secondary phase, strong strains etc.
- Anisotropic (below) broadening is challenging to analyze

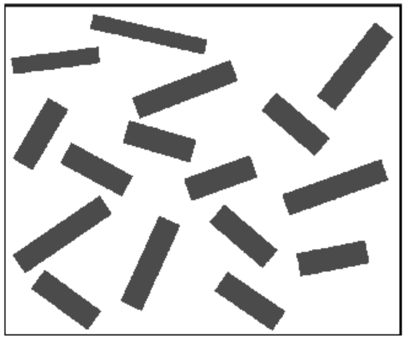




Peak shape: preferred orientation

Rietveld method assumes there are a very large number of crystallites (full powder average) that are **randomly oriented**

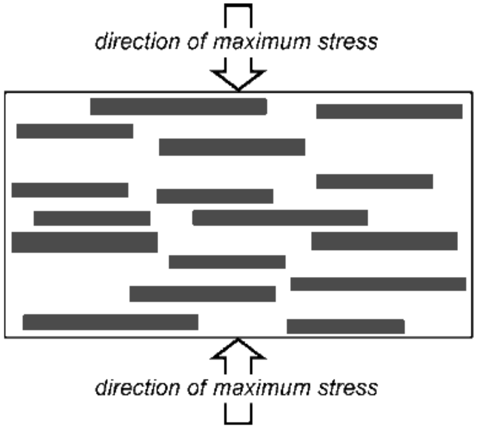
randomly oriented minerals



Sample shape can cause preferred orientation

→ *hkl*-dependent, systematic intensity change

preferentially oriented minerals



Most typical model: Modified March’s function with refinable *parameter* G:

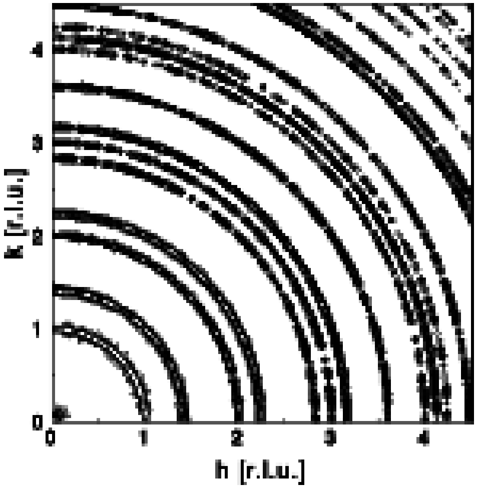
G = 1 => no orientation

G < 1 => plate-like crystallites

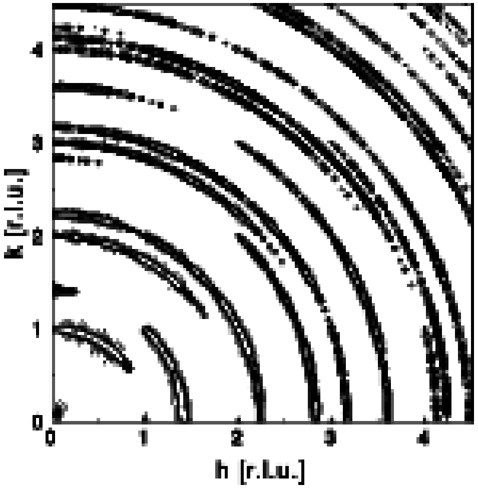
G > 1 => needle-like crystallites

$$y_{ci} = s\sum_{hkl} L_{hkl}\left|F_{hkl}\right|^2 \phi(2\theta_i - 2\theta_{hkl})\boxed{\overset{*}{P}_{hkl}}A + y_{bi}$$

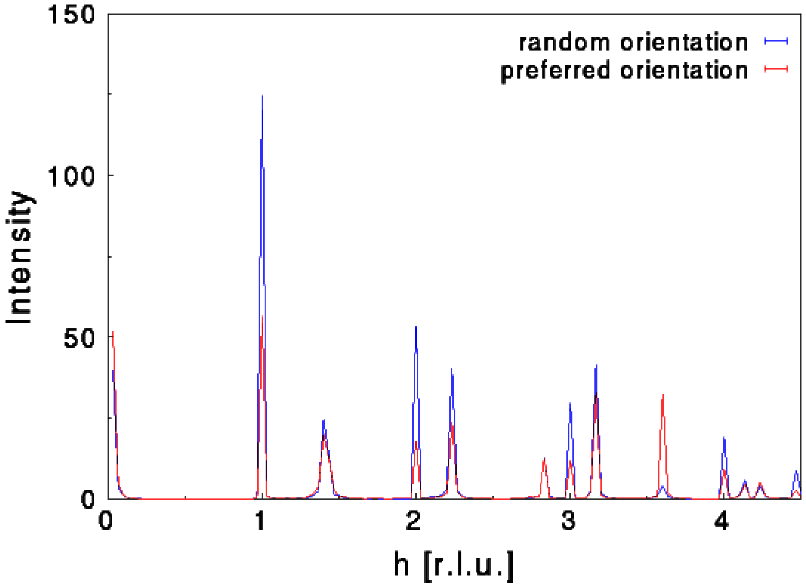
Peak shape: preferred orientation



random



orientation

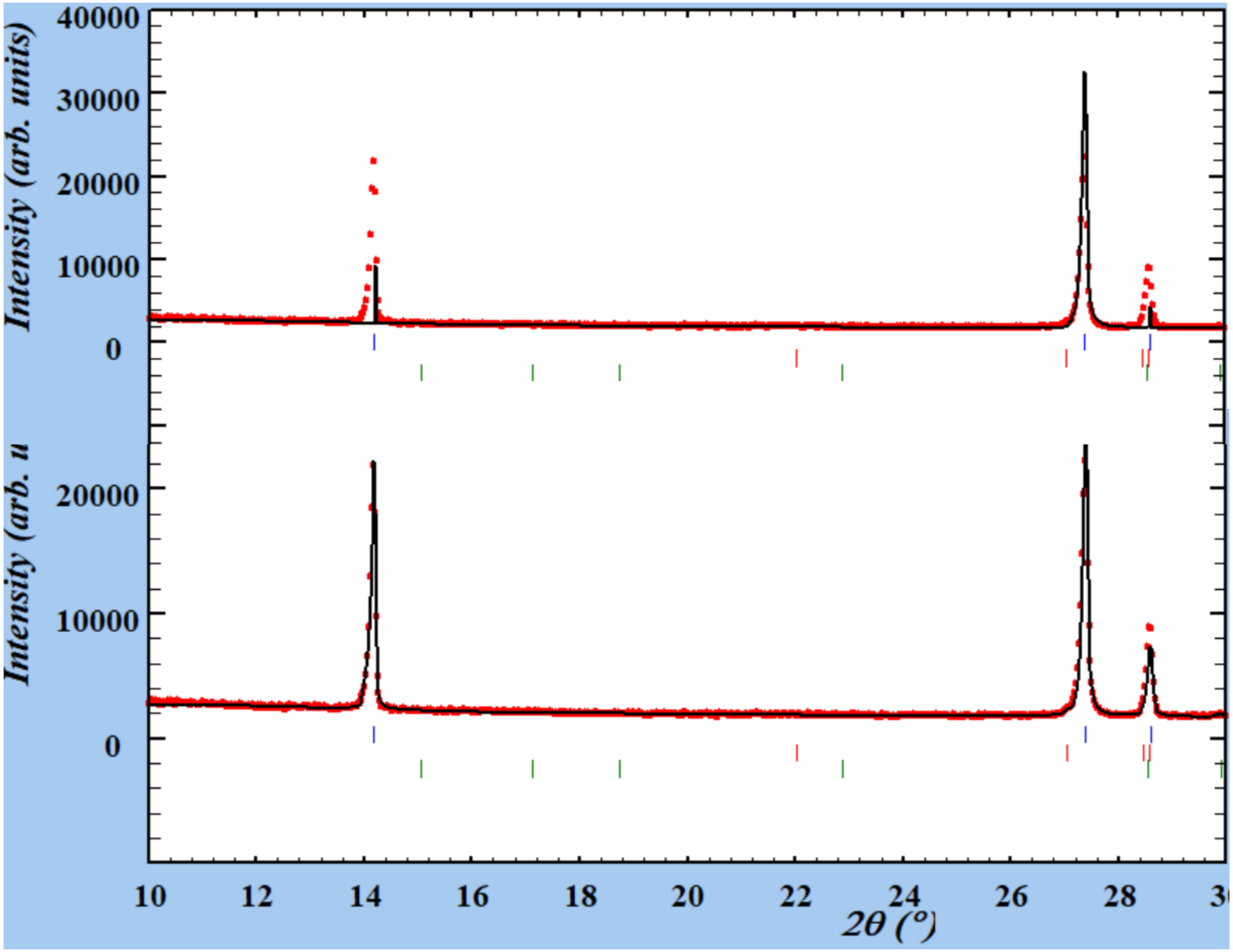


- Proper treatment of sample helps but will never take the problem away
- Crystallite shape preferences depend on Miller plane atomic density and chemical properties: orientation preference can be theoretically predicted via surface energy calculations, but this is rarely done

Peak shape: preferred orientation

Orientation effect, not refined

Taken into account



Peak intensity: quantity estimation

Scale factors s can be linked to mass percentages in the sample

$$W_\alpha = \frac{\boxed{S_\alpha}(ZMV)_\alpha}{\sum_{k=1}^{n} S_k (ZMV)_k}$$

W_α= weight fraction of **phase α**

S = Rietveld scale factor

ZMV = mass and volume of unit cell

We will refine this: scale factor

- Used e.g. in impurity level detection and polymorph analysis* (even in amorphous vs. crystalline phase determination and quantification)
- Rather accurate (as compared to intensity comparison most manufacturers' software use for the same purpose)
- Structure of all phases in the sample must be known *and used*

**XRD is the only method that can distinguish structures from chemical composition*

Rietveld in practice

- There are several different software packages for Rietveld refinement
- We are going to use FullProf
- More details in the exercise session (& exercise material)

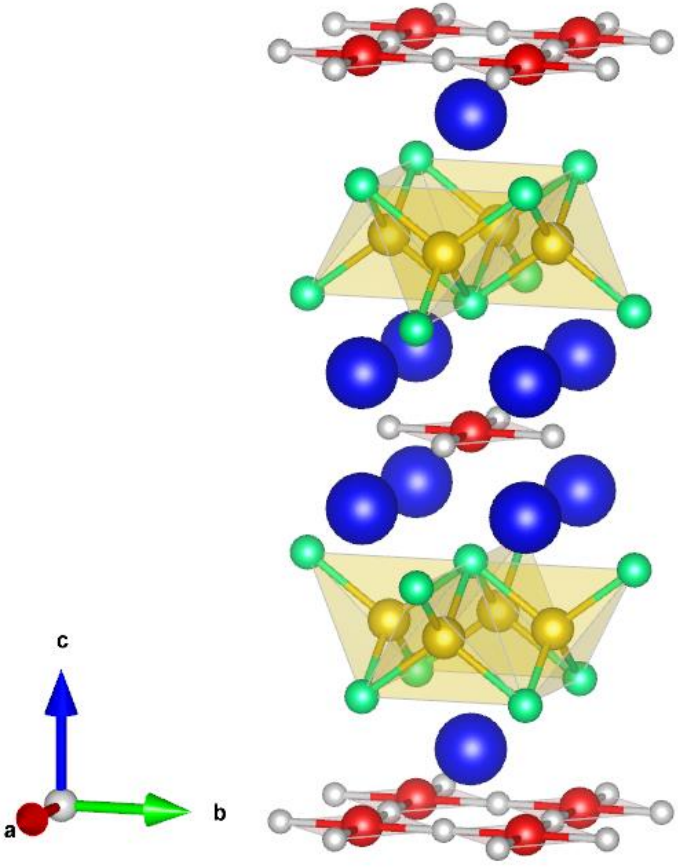
What goes in, what comes out?

IN:

- The XRD measurement data
- The PCR document: refinement settings
- Some guess at the structure

OUT:

- The lattice parameters and atomic coordinates & occupancies
- Phase fractions (if sample is a mixture)
- Values of the parameters that describe the peak shape
    - Crystallite size, shape estimation



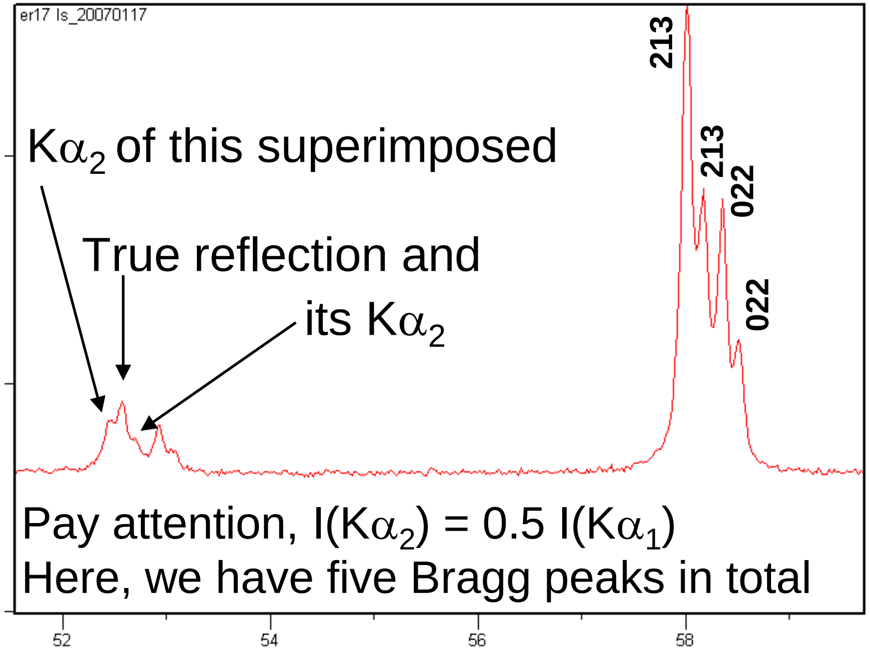
Layered structure of $Sr_2CoO_2Cu_2Se_2$

But with PXRD we are never sure! We only learn if our model is able to produce a similar pattern as our measurement → supportive methods will increase the certainty

What the expert (= you) **needs to know**

- What is a realistic guess of structure?
- What are the measurement parameters?
- When is the calculation going in the wrong direction?

Kα or Kα1?



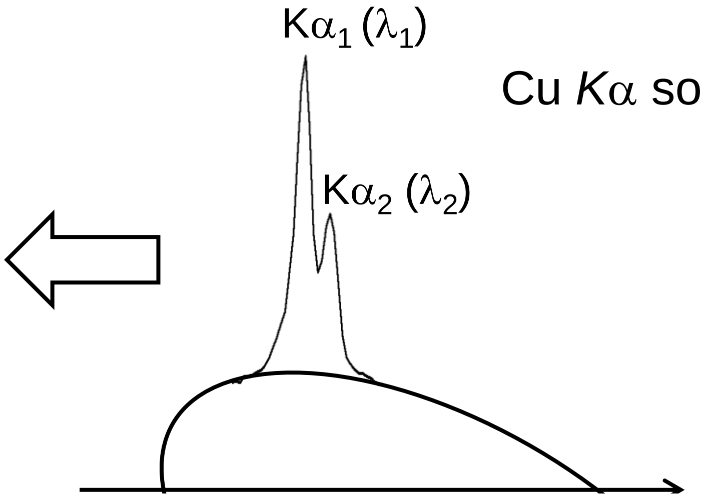
Kα_2 of this superimposed

True reflection and

its Kα_2

Pay attention, I(Kα_2) = 0.5 I(Kα_1)

Here, we have five Bragg peaks in total

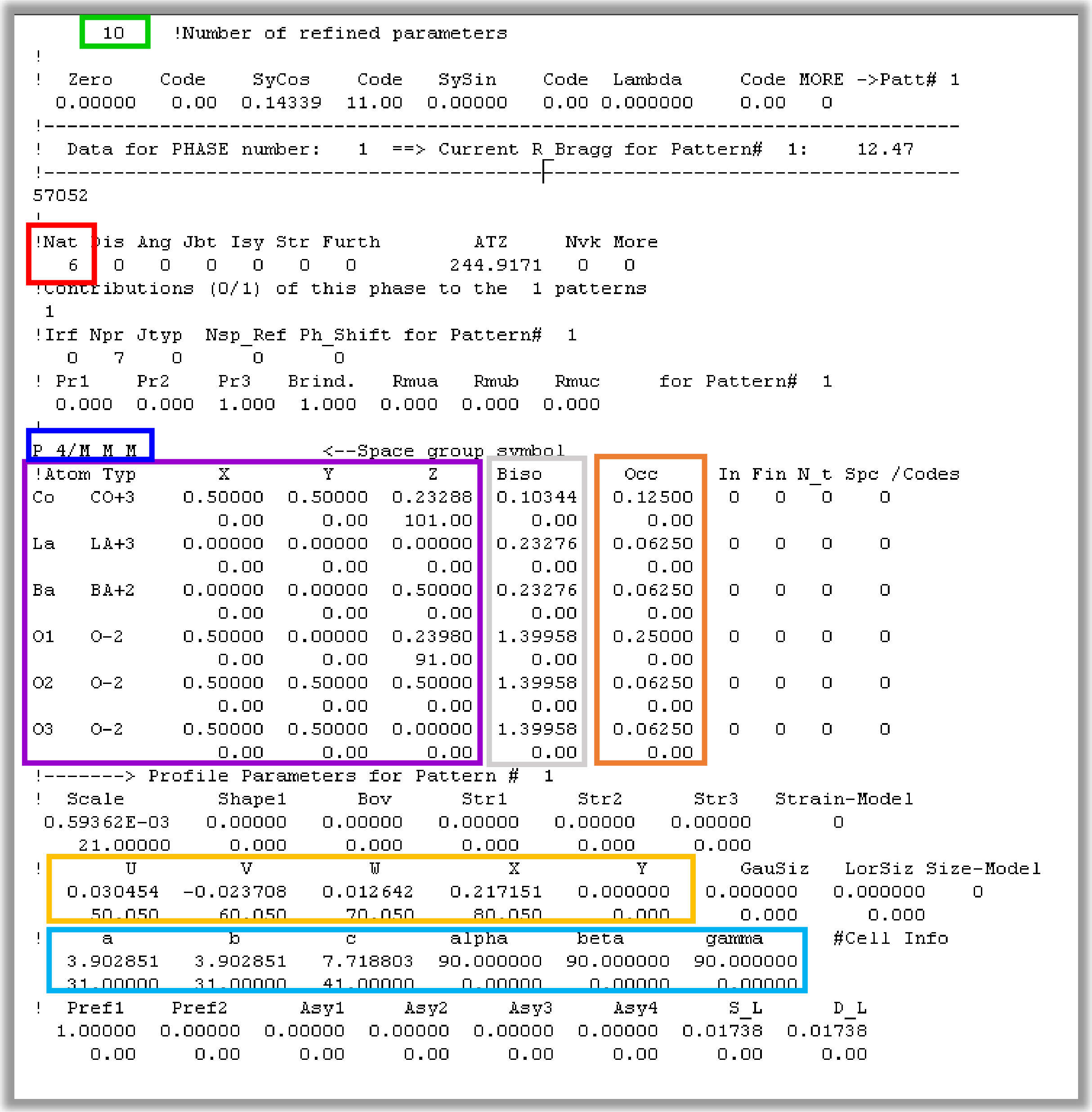


Cu *K*α source

wavelenght λ

(characteristic X-ray excitation spectrum of Cu anode)

The PCR document (for FullProf)



```
     10      !Number of refined parameters
!
!  Zero     Code     SyCos     Code    SySin     Code  Lambda      Code  MORE ->Patt# 1
  0.00000   0.00  0.14339  11.00  0.00000    0.00 0.000000     0.00    0
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:     12.47
!-------------------------------------------------------------------------------
57052
!
!Nat Dis Ang Jbt Isy Str Furth          ATZ      Nvk More
   6   0   0   0   0   0   0         244.9171    0   0
!Contributions (0/1) of this phase to the  1 patterns
 1
!Irf Npr Jtyp  Nsp_Ref Ph_Shift for Pattern#  1
   0   7    0       0        0
! Pr1     Pr2     Pr3    Brind.    Rmua    Rmub    Rmuc      for Pattern#  1
  0.000   0.000   1.000   1.000   0.000   0.000   0.000
!
P 4/M M M                  <--Space group symbol
!Atom Typ       X         Y         Z      Biso       Occ     In Fin N_t Spc /Codes
Co    CO+3    0.50000   0.50000   0.23288  0.10344   0.12500   0   0   0    0
                 0.00      0.00    101.00     0.00      0.00
La    LA+3    0.00000   0.00000   0.00000  0.23276   0.06250   0   0   0    0
                 0.00      0.00      0.00     0.00      0.00
Ba    BA+2    0.00000   0.00000   0.50000  0.23276   0.06250   0   0   0    0
                 0.00      0.00      0.00     0.00      0.00
O1    O-2     0.50000   0.00000   0.23980  1.39958   0.25000   0   0   0    0
                 0.00      0.00     91.00     0.00      0.00
O2    O-2     0.50000   0.50000   0.50000  1.39958   0.06250   0   0   0    0
                 0.00      0.00      0.00     0.00      0.00
O3    O-2     0.50000   0.50000   0.00000  1.39958   0.06250   0   0   0    0
                 0.00      0.00      0.00     0.00      0.00
!-------> Profile Parameters for Pattern #  1
!  Scale        Shape1      Bov      Str1      Str2      Str3   Strain-Model
 0.59362E-03   0.00000   0.00000   0.00000   0.00000   0.00000       0
    21.00000     0.000     0.000     0.000     0.000     0.000
!       U          V          W          X          Y        GauSiz   LorSiz Size-Model
   0.030454  -0.023708   0.012642   0.217151   0.000000   0.000000   0.000000    0
     50.050     60.050     70.050     80.050      0.000      0.000      0.000
!       a          b          c        alpha      beta       gamma      #Cell Info
   3.902851   3.902851   7.718803  90.000000  90.000000  90.000000
   31.00000   31.00000   41.00000    0.00000    0.00000    0.00000
!  Pref1    Pref2      Asy1      Asy2      Asy3      Asy4      S_L       D_L
  1.00000  0.00000   0.00000   0.00000   0.00000   0.00000   0.01738   0.01738
     0.00     0.00      0.00      0.00      0.00      0.00      0.00      0.00
```

Line 13-14: NCY = ’number of calculation cycles’

How many parameters are open to refinement? (auto-updated)

How many atoms are in the basis? MUST BE RIGHT

What is the symmetry (space group) of the unit cell?

List the atoms of the basis, their valence, and their coordinates

Occupancy of each atomic site: check carefully that it is right!

Thermal vibration parameter – works better for neutron diffraction

Peak profile parameters

Unit cell parameters

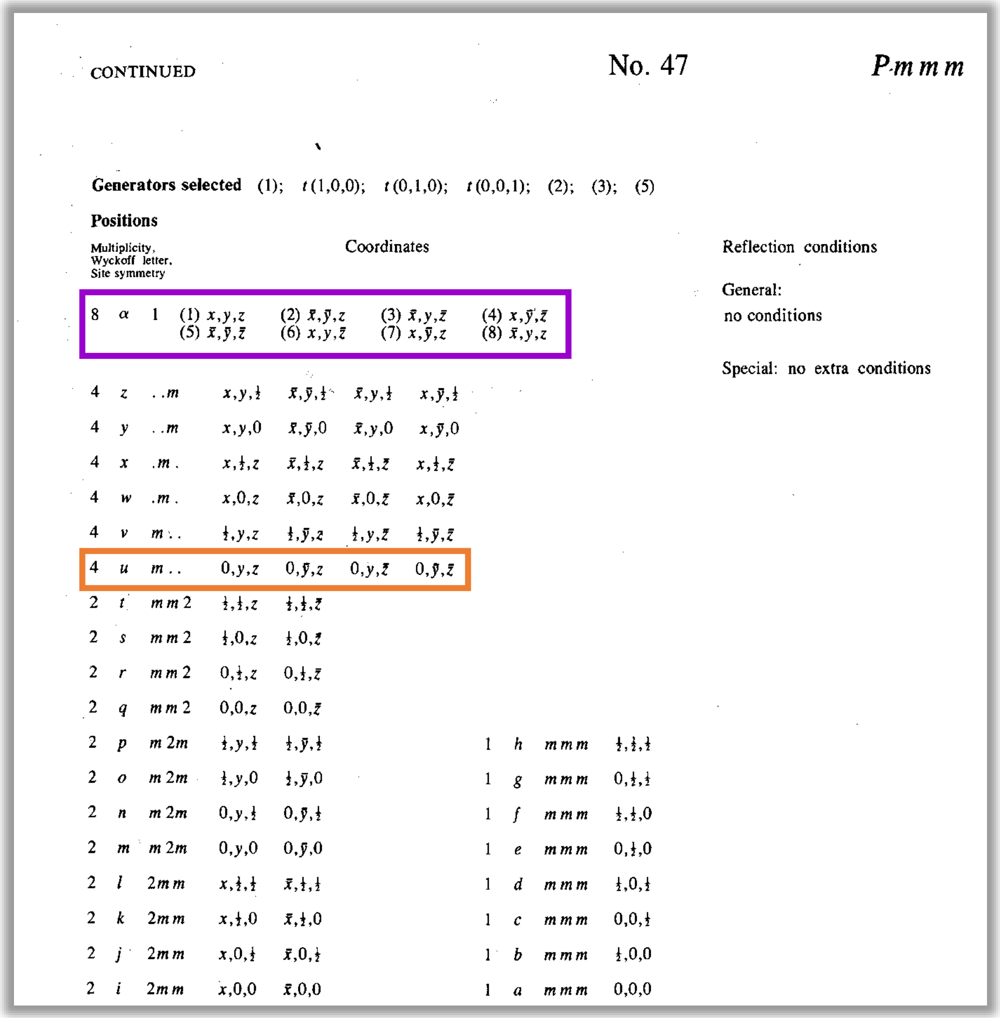
Site occupancy calculation

- In real structures, atoms are often missing from some % of the atomic site → influences the structure factor and peak intensity
- Site occupancy factor abbreviations: ”Occ”, ”g”, ”S” or ”s.o.f”

$$Occ = \frac{the\ atom's\ Wyckoff\ site\ multiplicity}{the\ space\ group's\ general\ Wyckoff\ multiplicity} \cdot site\ occupancy\ fraction$$

- Good idea to double-check in VESTA that the *Occ* are sensible!

Site occupancy calculation: example



CONTINUED No. 47 *Pmmm*

Generators selected (1); $t(1,0,0)$; $t(0,1,0)$; $t(0,0,1)$; (2); (3); (5)

Positions

Multiplicity, Wyckoff letter, Site symmetry			Coordinates							
8	α	1	(1) x,y,z (5) $\bar{x},\bar{y},\bar{z}$	(2) $\bar{x},\bar{y},z$ (6) $x,y,\bar{z}$	(3) $\bar{x},y,\bar{z}$ (7) $x,\bar{y},z$	(4) $x,\bar{y},\bar{z}$ (8) $\bar{x},y,z$				
4	z	$..m$	$x,y,\frac{1}{2}$	$\bar{x},\bar{y},\frac{1}{2}$	$\bar{x},y,\frac{1}{2}$	$x,\bar{y},\frac{1}{2}$				
4	y	$..m$	$x,y,0$	$\bar{x},\bar{y},0$	$\bar{x},y,0$	$x,\bar{y},0$				
4	x	$.m.$	$x,\frac{1}{2},z$	$\bar{x},\frac{1}{2},z$	$\bar{x},\frac{1}{2},\bar{z}$	$x,\frac{1}{2},\bar{z}$				
4	w	$.m.$	$x,0,z$	$\bar{x},0,z$	$\bar{x},0,\bar{z}$	$x,0,\bar{z}$				
4	v	$m..$	$\frac{1}{2},y,z$	$\frac{1}{2},\bar{y},z$	$\frac{1}{2},y,\bar{z}$	$\frac{1}{2},\bar{y},\bar{z}$				
4	u	$m..$	$0,y,z$	$0,\bar{y},z$	$0,y,\bar{z}$	$0,\bar{y},\bar{z}$				
2	t	$mm2$	$\frac{1}{2},\frac{1}{2},z$	$\frac{1}{2},\frac{1}{2},\bar{z}$						
2	s	$mm2$	$\frac{1}{2},0,z$	$\frac{1}{2},0,\bar{z}$						
2	r	$mm2$	$0,\frac{1}{2},z$	$0,\frac{1}{2},\bar{z}$						
2	q	$mm2$	$0,0,z$	$0,0,\bar{z}$						
2	p	$m2m$	$\frac{1}{2},y,\frac{1}{2}$	$\frac{1}{2},\bar{y},\frac{1}{2}$			1	h	mmm	$\frac{1}{2},\frac{1}{2},\frac{1}{2}$
2	o	$m2m$	$\frac{1}{2},y,0$	$\frac{1}{2},\bar{y},0$			1	g	mmm	$0,\frac{1}{2},\frac{1}{2}$
2	n	$m2m$	$0,y,\frac{1}{2}$	$0,\bar{y},\frac{1}{2}$			1	f	mmm	$\frac{1}{2},\frac{1}{2},0$
2	m	$m2m$	$0,y,0$	$0,\bar{y},0$			1	e	mmm	$0,\frac{1}{2},0$
2	l	$2mm$	$x,\frac{1}{2},\frac{1}{2}$	$\bar{x},\frac{1}{2},\frac{1}{2}$			1	d	mmm	$\frac{1}{2},0,\frac{1}{2}$
2	k	$2mm$	$x,\frac{1}{2},0$	$\bar{x},\frac{1}{2},0$			1	c	mmm	$0,0,\frac{1}{2}$
2	j	$2mm$	$x,0,\frac{1}{2}$	$\bar{x},0,\frac{1}{2}$			1	b	mmm	$\frac{1}{2},0,0$
2	i	$2mm$	$x,0,0$	$\bar{x},0,0$			1	a	mmm	$0,0,0$

Reflection conditions

General:
no conditions

Special: no extra conditions

- Space group of structure is *Pmmm*
  - → Space group has **multiplicity 8**
- Atom X is in (0; 0.233; 0.167)
  - → Atom X has W-pos **4u**
  - → **Multiplicity is 4**
- Atom X is missing from 12% of its positions in the crystal
  - → sof = 100%-12%= 0.88
- Occupancy of atom X:

$$Occ = \frac{4}{8} \cdot 0.88 = 0.44$$

The exercise on Rietveld refinement

- The exercise will be published on MyCourses today
- *Before* the exercise session, please:
  - Download FullProf on your own computer (instructions in exercise)
  - Look at the exercise questions and other material
  - Calculate occupancy factors in advance!
- *During* the exercise session, we will:
  - Discuss the most important refinement parameters
  - Finalize the documents for refinement
  - Run the refinement together
- *After* the exercise, please:
  - Submit the answers to the exercise questions in MyCourses