

# Tutorial on Rietveld Refinement using the program FullProf

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## INTRODUCTION

The aim of this tutorial is to give a feeling of what information can be extracted from x-ray or neutron powder diffraction (NPD) patterns and to show how this can be done in the real world. Most of the work will be carried on a neutron powder diffraction pattern taken at I.L.L. with the instrument D1A on a sample of the compound  $\text{YBa}_2\text{Cu}_{3-x}\text{CO}_x\text{O}_{7+y}$  with  $x = 0.33$ . All calculations will be performed with the FullProf software and the GUI WinPLOTR.

## THE SCIENTIFIC PROBLEM

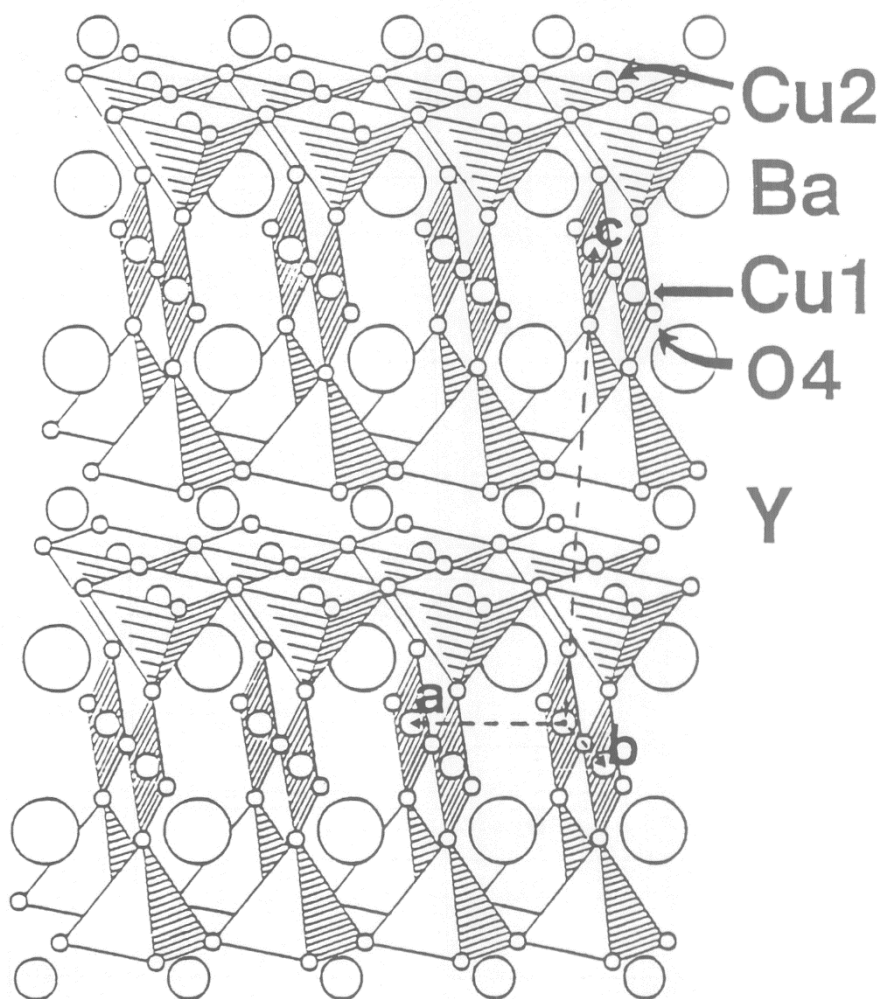
The compound  $\text{YBa}_2\text{Cu}_3\text{O}_7$  is a (too) well-known high  $T_c$  superconductor with  $T_c=90$  K. Its space group is  $Pmmm$  (orthorhombic symmetry) with cell parameters  $a = 3.206(7)$  Å,  $b=3.885(1)$  Å and  $c=11.6757(4)$  Å (figure 1). It can be described based on the perovskite structure with copper on the B sites, barium and yttrium on the A sites. Barium and yttrium are ordered along the  $c$  axis following the Y-Ba-Ba-Y... sequence, leading to the tripling of the unit cell along  $c$ , with respect to the perovskite cell (cubic,  $a=b=c=3.8$  Å). All the oxygen atoms on the yttrium planes are absent. One half of the oxygen atoms of the barium planes are absent so that chains of  $\text{CuO}_4$  squares running parallel to the  $b$  axis are formed. (See figure)

Oxygen can be reversibly removed from the above compound down to the stoichiometry  $\text{YBa}_2\text{Cu}_3\text{O}_6$ . This is accompanied by the disappearance of the superconducting properties, the compound with  $\text{O}_6$  stoichiometry being an antiferromagnetic insulator (figure 2). The removed oxygen atoms are those forming the chains (called  $\text{O}_4$ ). Since no difference between the  $a$  and  $b$  directions exists in the  $\text{O}_6$  compound, its symmetry is tetragonal, space group  $P4/mmm$ ,  $a=b=3.872(1)$  Å,  $c=11.738(2)$  Å. (see figure)

When substituting copper by a transition metal element in  $\text{YBa}_2\text{Cu}_3\text{O}_7$ , the superconducting properties disappear after only  $\approx 10\%$  substitution. The symmetry changes from orthorhombic to tetragonal after only a few % substitution, and the presence of oxygen excess with respect of the  $\text{O}_7$  stoichiometry is observed. These preliminary results have been obtained by susceptibility measurements, inspection of the positions of Bragg peaks on laboratory x-ray diffraction powder patterns, and thermogravimetric measurements, respectively.

The big question is "why does superconductivity disappear when a transition metal element substitutes for copper". Of course, everybody hopes that answering this question could help answering an even bigger one: "why superconductivity does exist when no transition metal element substitutes for copper".... However, let us be reasonable and restrict ourselves to the first question. To look for an answer, more details about the structure of the substituted compounds must be obtained. Among these are the following:

- How much of the substituting element really enters the structure (if it does)?
- Which copper sites are substituted (there are two non-equivalent ones in the structure)?
- How much excess oxygen is present in the compound and where is it localised?
- How is the structure distorted by the substitution?



Structure du composé  $\text{YBa}_2\text{Cu}_3\text{O}_7$

*Paramètres structuraux pour le composé  $\text{YBa}_2\text{Cu}_3\text{O}_7$*

Atom	Pos.	x	y	z	B
Y	1h	1/2	1/2	1/2	0.58(10)
Ba	2t	1/2	1/2	0.1841(3)	0.59(10)
Cu1	1a	0	0	0	0.38(11)
Cu2	2q	0	0	0.3549	0.51(7)
O1	2q	0	0	0.1581(4)	0.93(12)
O2	2s	1/2	0	0.3779(4)	0.11(9)
O3	2r	0	1/2	0.3777(5)	0.31(9)
O4	1e	0	1/2	0	*

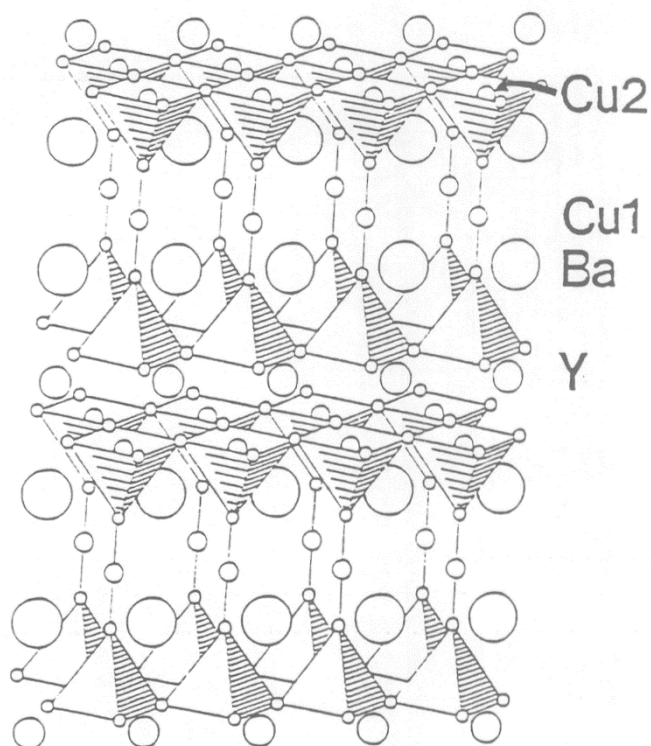
$a = 3.8206(1)\text{\AA}$ ,  $b = 3.8851(1)\text{\AA}$ ,  $c = 11.6757(4)\text{\AA}$

\* anisotropic thermal parameters for O4 are :  
 $B_{11} = 3.2(5)$ ,  $B_{22} = 0.6(4)$ ,  $B_{33} = 3.5(6)$

*Distances interatomiques pour le composé  $\text{YBa}_2\text{Cu}_3\text{O}_7$*

Ba-O1 (x4) : 2.741(1)	Y-O2 (x4) : 2.409(3)
Ba-O2 (x2) : 2.982(5)	Y-O3 (x4) : 2.385(3)
Ba-O3 (x2) : 2.959(6)	mean Y-O : 2.397
Ba-O4 (x2) : 2.876(4)	Cu2-O1 (x1) : 2.298(6)
mean Ba-O : 2.860	Cu2-O2 (x2) : 1.929(1)
Cu1-O1 (x2) : 1.846(5)	Cu2-O3 (x2) : 1.961(1)
Cu1-O4 (x2) : 1.942(0)	mean Cu2-O : 2.015
mean Cu1-O : 1.894	

***Figure 1:*** *The structure of  $YBa_2Cu_3O_7$*



Structure du composé  $\text{YBa}_2\text{Cu}_3\text{O}_6$

Paramètres structuraux pour le composé  $\text{YBa}_2\text{Cu}_3\text{O}_6$ .

	Wyckoff symbol	x	y	z	$U_{11}$	$U_{22}$	$U_{33}$
Ba	2h	1/2	1/2	0.1921(1)	0.0105(2)	$= U_{11}$	0.0148(4)
Y*	1d	1/2	1/2	1/2	0.0051(5)	$= U_{11}$	0.0111(9)
Cu1	1a	0	0	0	0.0193(8)	$= U_{11}$	0.017(2)
Cu2	2g	0	0	0.3605(2)	0.0029(3)	$= U_{11}$	0.0129(8)
O1	2g	0	0	0.154(1)	0.018(2)	$= U_{11}$	0.010(5)
O2	4l	1/2	0	0.3794(7)	0.003(2)	0.009(2)	0.015(3)

\*  $\text{Y} = 0.89(4)\text{Y} + 0.11(4)\text{Ba}$

Distances interatomiques pour le composé  $\text{YBa}_2\text{Cu}_3\text{O}_6$ .

Ba-O1*	2.775(3)	x4	Ba-Ba	3.8715(6)
Ba-O2	2.929(7)	x4	Ba-Y	3.614(1)
Average	2.852		Ba-Cu1	3.5468(7)
			Ba-Cu2	3.377(1)
Cu2-O1	2.428(16)	x1	Y-Cu2	3.190(1)
Cu2-O2	1.948(1)	x4	Cu1-Cu2	4.232(2)
Average	2.044		Cu2-Cu2	3.274(1)
Cu1-O1	1.804(16)		O1-O2	3.281(15)
			O2-O2	2.738(6)
Y-O2	2.399(6)	x8		

Figure 2: The structure of  $\text{YBa}_2\text{Cu}_3\text{O}_6$

## WHY CHOOSE NEUTRON POWDER DIFFRACTION?

### Why neutrons?

- because the copper contribution can be easily separated from that of the transition metal elements, which would be difficult with x-rays because of the small difference in atomic numbers. The Fermi lengths used below are:  $b(\text{Y})=0.775$ ,  $b(\text{Ba})=0.525$ ,  $b(\text{Cu})=0.7718$ ,  $b(\text{Co})=0.25$ ,  $b(\text{O})=0.5805$  ( $\times 10^{-12}\text{cm}$ )
- because oxygen can be detected with a higher accuracy than with x-rays for which its diffracting power is low with respect to Y, Ba, because of its low atomic number.

### Why powders?

- Because..., they can be prepared in large quantities needed for neutron diffraction.
- Because... the substitution ratio can be controlled in a better way for powders than for single crystals.
- Because... it works well, is easy to handle, needs less neutron beam time and therefore is more accessible.

## THE EXAMPLE

### The Sample

The sample used for this study was a 10g powder sample of  $\text{YBa}_2\text{Cu}_{3-x}\text{Co}_x\text{O}_{7+y}$  with nominal  $x=0.33$ . It looked pure by x-ray powder diffraction and was not superconducting. The indexation of the x-ray powder diffraction pattern yielded tetragonal symmetry with  $a=3.869\text{\AA}$  and  $c=11.66\text{\AA}$ . A table of indexed reflexions based on this cell and the wavelength used for the neutron diffraction experiment is given. The excess oxygen value  $y$  is unknown.

### The experiment

The NPD pattern was collected at I.L.L. on the D1A powder diffractometer.

Wavelength:  $1.9095\text{\AA}$

Start  $2\theta$  :  $0.069^\circ$

Step  $2\theta$ :  $0.05^\circ$

End  $2\theta$ :  $150.019^\circ$

Number of points: 3000

### The data

The file containing the data is called `Cocuc.dat`. You can see how it looks like on the list below (figure 3). The counting values for each step are preceded by the number of detectors over which these values have been averaged (since D1A is a 1D-detector diffractometer). The raw diffraction pattern can be seen on figure 4.

0.069	0.05	150.0	S151 (Co)	RT	D1A	1.91A	MON=5000				
1	21 1	19 1	23 1	17 1	15 1	15 1	13 1	9 1	7 1	8	
1	7 1	8 1	8 1	11 1	11 1	14 1	20 1	22 1	20 1	22	
1	22 1	21 1	24 1	22 1	20 1	26 1	19 1	14 1	14 1	10	
1	10 1	8 1	11 1	14 1	13 1	8 1	8 1	8 1	19 1	22	
1	21 1	17 1	11 1	8 1	10 1	21 1	21 1	14 1	13 1	12	
1	17 1	21 1	34 1	30 1	30 1	30 1	33 1	37 1	43 1	43	
1	41 1	46 1	58 1	55 1	61 1	62 1	67 1	67 1	61 1	62	
1	54 1	51 1	54 1	59 1	50 1	41 1	51 1	56 1	55 1	49	
1	45 1	59 1	57 1	51 1	54 1	62 1	59 1	55 1	53 1	58	
1	59 1	55 1	69 1	68 1	57 1	60 1	75 1	67 1	62 1	58	
1	58 1	67 1	75 1	62 1	62 1	71 1	67 1	68 1	83 1	78	
1	68 1	75 1	78 1	75 1	82 1	86 1	86 1	81 1	75 2	85	
2	84 2	79 2	80 2	79 2	75 2	74 2	80 2	87 2	91 2	82	
2	90 2	92 2	93 2	98 2	88 2	82 2	87 2	92 2	100 2	96	
2	97 2	97 2	101 2	106 2	102 2	98 2	109 2	105 2	108 2	108	
2	112 2	122 2	125 2	119 2	125 2	133 2	136 2	136 2	132 2	129	
2	145 2	159 2	164 2	142 2	149 2	151 2	166 2	178 2	192 2	196	
2	202 2	200 2	215 2	229 2	244 2	268 2	280 2	290 2	326 2	341	
2	362 2	378 2	400 2	443 2	494 2	495 2	477 2	450 2	427 2	400	
2	358 2	300 2	239 2	192 2	160 2	128 2	100 2	90 2	84 2	81	
2	70 2	60 2	64 2	72 2	63 2	61 2	64 2	74 2	72 2	70	

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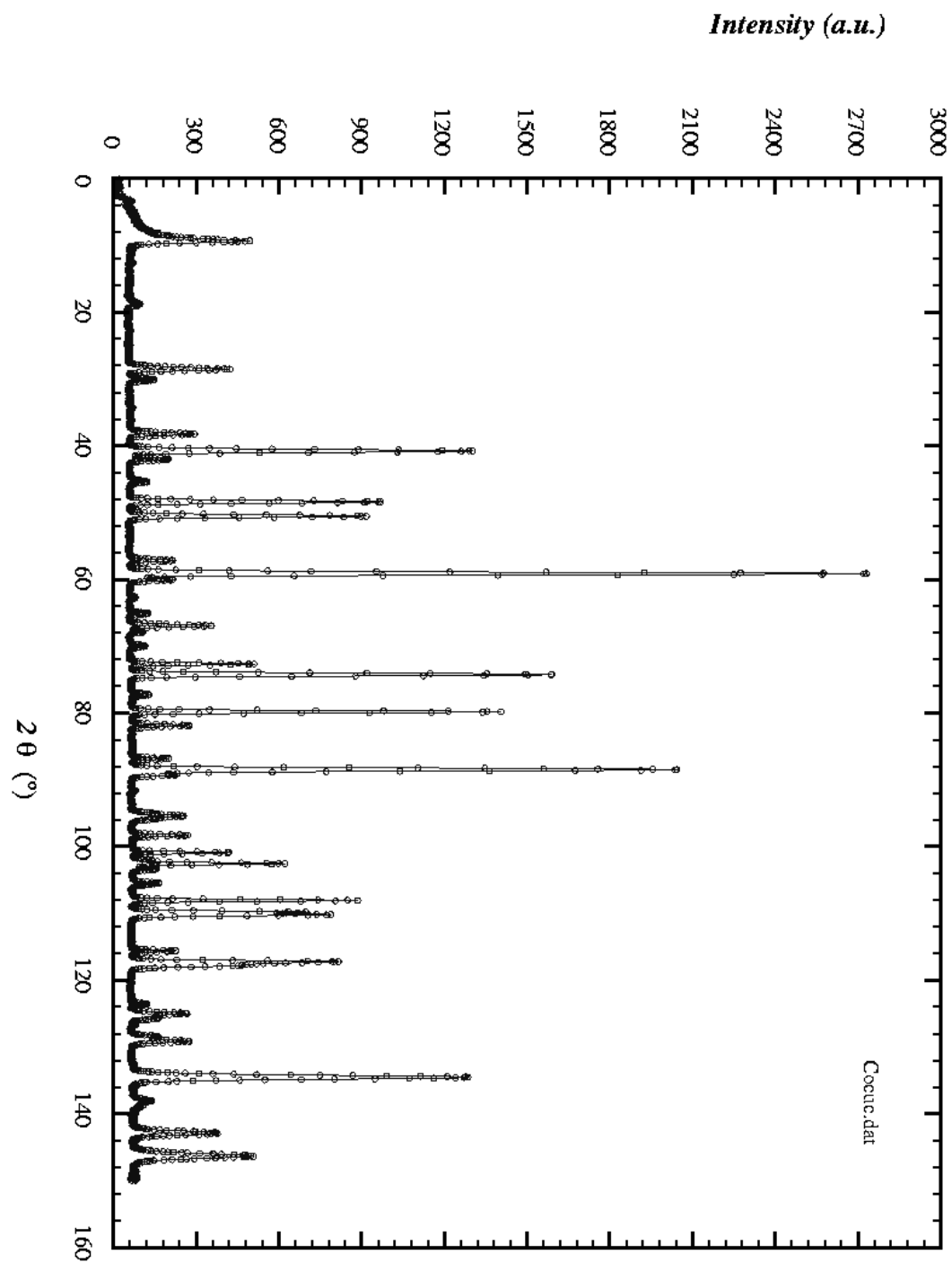
10	191 10	212 10	215 10	206 10	189 10	172 10	146 10	117 10	100 10	86
10	75 10	67 10	63 10	62 10	60 10	63 10	61 10	61 10	58 10	62
10	60 10	60 10	62 10	67 10	71 10	73 10	86 10	102 10	125 10	163
10	226 10	310 10	419 10	559 10	717 10	954 10	1220 10	1569 10	1926 10	2274

-----

3	87 3	77 3	77 3	76 3	79 3	70 3	72 3	76 3	70 3	78
3	76 3	76 3	73 2	74 2	73 2	74 2	78 2	71 2	75 2	72
2	73 2	70 2	83 2	81 2	77 2	74 2	76 2	68 2	76 2	81
2	73 2	72 2	77 2	82 2	73 2	79 2	84 2	65 2	74 2	86
2	70 2	64 2	70 2	62 2	68 2	74 2	78 2	78 2	65 2	80
2	69 2	88 2	73 2	80 2	72 2	72 2	76 2	74 2	82 2	83
2	84 2	78 2	87 2	74 2	92 2	75 2	81 2	97 2	82 2	72
2	84 2	86 2	76 2	86 2	83 2	78 2	82 2	81 2	83 2	80
2	75 2	69 2	70 2	79 2	67 2	85 2	75 2	68 2	74 2	77
2	72 2	68 2	71 2	70 2	65 2	71 2	63 2	75 2	77 2	67
2	76 2	76 2	68 2	70 2	70 2	63 2	73 2	79 2	63 2	74
2	68 2	69 2	70 2	85 2	80 2	76 2	88 2	79 2	74 2	73
2	73 2	77 2	71 2	71 2	73 2	68 2	68 2	84 2	79 2	72
2	70 1	72 1	69 1	78 1	87 1	72 1	77 1	86 1	72 1	72
1	86 1	66 1	84 1	78 1	73 1	78 1	75 1	69 1	73 1	80
1	79 1	75 1	63 1	78 1	75 1	73 1	91 1	93 1	85 1	72
1	73 1	86 1	90 1	92 1	109 1	74 1	85 1	86 1	86 1	108
1	94 1	119 1	102 1	104 1	110 1	127 1	119 1	117 1	123 1	113
1	124 1	133 1	109 1	136 1	127 1	143 1	127 1	109 1	115 1	118
1	113 1	108 1	108 1	116 1	97 1	100 1	89 1	106 1	85 1	91
1	79 1	92 1	72 1	82 1	97 1	89 1	80 1	80 1	91 1	66
1	64 1	63 1	87 1	71 1	80 1	80 1	72 1	91 1	62 1	82
1	92 1	80 1	87 1	99 1	74 1	99 1	82 1	85 1	104 1	85
1	94 1	94 1	107 1	109 1	106 1	100 1	85 1	123 1	105 1	124
1	135 1	179 1	141 1	166 1	190 1	192 1	193 1	206 1	223 0	0

-1000  
-10000

**Figure 3:** The Cocuc.dat raw data file



*Figure 4: Plot of the raw data collected on D1A*

## Extracting structural information from the diffraction pattern

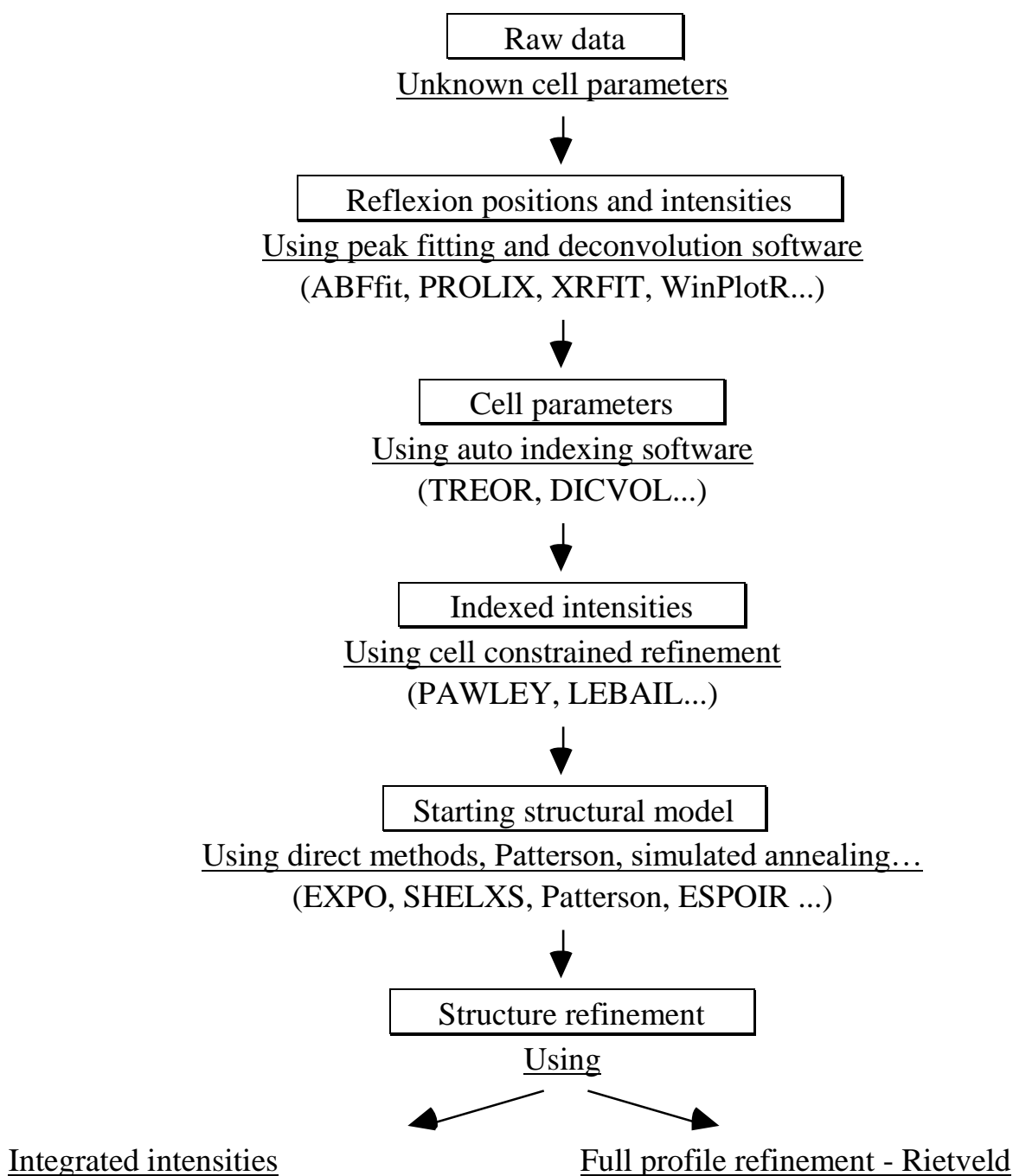
A block diagram is presented below to summarise the various paths that one can choose in order to retrieve the structure of a compound from its powder diffraction pattern (it does not matter here whether neutron or x-ray diffraction was used).

A guide for:

### **"ab initio" Structure Determination**

Using:

#### **Powder diffraction patterns**





(MXD, SHELXL...)

(FullProf, GSAS, Jana...)

The first question to ask oneself is "do I have a reasonable structural model to start with?". This includes, cell parameters, space group and atomic positions.

If the answer is "yes", one can directly try a full profile refinement procedure (so called "structure constrained refinement" or "Rietveld refinement") which should lead to the complete structure solution. Another possibility would be to obtain the integrated intensities of each peak by fitting separately each part of the data containing one or a few peaks. The refinement can then be carried out as if single crystal data had been measured. It is worth saying that, from a theoretical point of view; both procedures are equivalent and should lead to the same results. However, in case of strong overlapping of peaks (large cell, low symmetry) the separate peak integration can yield inaccurate intensity values.

Suppose the answer is "no". Then you must obtain a starting model from the data themselves. To do this, several procedures can be tried: the Patterson transform, the Direct Methods, or the direct space techniques (grid search, simulated annealing...). However, all of them must be fed with a list of integrated intensities, e.g. a list of ( $h\ k\ l$ , I, sig(I)). If you do not even know the unit cell, the only way is to integrate separately the Bragg peaks. Then, from the peak positions, the unit cell and systematic extinctions can be obtained (although it may be difficult, sometimes), using for example auto indexing programs (TREOR, DICVOL, ITO...). Once you know the unit cell, then you can use the "cell constrained refinement" (also called Pawley, or LeBail type refinement) which extracts integrated intensities by cell constrained full profile fitting. When the ( $h\ k\ l$ , I, sig(I)) list has been obtained, a starting model could be found by the above mentioned procedures and the refinement can be carried out, either by full profile (Rietveld) fitting, or by least-squares refinement of integrated intensities.

Now, let us try...

### **Fitting overlapping reflections with WinPLOTTR**

Basically with any program of this kind, you must define the angular range of the pattern you want to work with, define the background and give for each peak the position of the maximum, the full width at half maximum (FWHM), and the type of analytical function supposed to best fit the peak shape. The output will be the integrated intensity, the position and the FWHM of each peak plus their standard deviations, and the refined values of the background parameters (but you won't care about them, generally). WinPLOTTR, the FullProf GUI, has many functions to visualise, inspect and carry out various types of pre-treatment with your data: background subtraction, peak searching, peak fitting, interface to auto indexing programs...

Start WinPLOTTR from the icon on the desktop, and open the data file COCUS.DAT, using instr=1 (defines type of data format used for D1A data). You can easily zoom, un-zoom and move around in the data by using the mouse. As an exercise, it is interesting to try to integrate the reflexions in two intervals: 58°-62°, and 116°-120° (you can then try others if you like). The 2 $\theta$  range can be defined by clicking on the magnifying glass icon. You then need to define the starting model for the fit. Click on the fit icon, and define the left and right background, the top and FWHM of each peak with left button clicks, then finish with a right button click (everything you should do is written at the bottom left of the window). At the right button click, a window will pop up where you can define the parameters to be refined. Closing this window starts the refinement. Two new windows appear,

one showing the progress of convergence, the other the fit results, also graphically shown in the main window. Try to look critically to the results...

When the fit is finished, you can reload the full pattern in the "calculation → reload data file" menu. Eventually, it may be interesting to compare these results to those of the automatic peak search procedure, which you can make with the "point selection" menu...

### **Full profile refinements using FullProf**

FullProf is a program allowing both the cell and the structure-constrained refinements by the whole pattern fitting technique. It is one of the numerous sons of the original program written by Rietveld (himself!). The input of the program consists in two files. One (the `xxxx.dat` file) contains the experimental data. The other one (the `xxxx.pcr`) is a control file that contains all the information needed by the program to run. A printout of this file is shown below. It can be accessed and modified directly from the GUI by clicking on the PCR icon. Its content will be explained during the tutorial. FullProf can be started from the GUI by clicking on the FP icon and choosing the `xxxx.pcr` file (if an `xxxx.dat` file exists, it will be used, otherwise, the program will ask for a data file).

The first step will be to carry out a cell constrained refinement (or Le Bail type refinement). It is assumed that a unit cell has been found from the knowledge or individual peak positions. Even if a structural model is already known, it is wise to start by a Le Bail type refinement. What you will obtain is:

- A file containing *hkl* I for each reflexion, to be used for structure solution
- Refined values for the cell, overall parameters (zero shift, background...) profile parameters, etc, so you know them accurately before starting the Rietveld refinement
- The best  $X^2$  and Rwp factors that you can expect from subsequent Rietveld refinement.

The control file to use for the cell-constrained refinement is called `Cocuc_LeBail.pcr`. The important thing to remember here is that there is always a strategy to be used for least-squares refinement: not all parameters can be refined in a single shot, and some should be refined before others...

The second step will be to carry out the Rietveld, structure constrained refinement. The control file to use is called `Cocus_start-excl.pcr`. It is, basically, the same as the previous one, where the atomic positions have been added. This allows using in a simple way the results from the Le Bail refinement. Here again, a reasonable refinement strategy has to be applied. The first thing to do is obviously to refine the scale factor. You can then free all the parameters you had previously refined in the cell-constrained refinement. This should not change much the results. Then refine the positional parameters and then isotropic thermal parameters. Normally, one should do both operations in decreasing order of atomic scattering power. Then the obtained thermal parameters should be critically scrutinised, and if required, some occupancies should be refined. If physically justified, some anisotropic thermal displacement parameters could be refined too.

```

COMM YBA2CU2COO7 D1A LeBail Fit
! Current global Chi2 (Bragg contrib.) = 26.69
! Files => DAT-file: Cocuc.dat, PCR-file: Cocuc_LeBail
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
1 5 1 0 1 0 1 1 0 0 1 0 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 1 1 0 4 0 0 1 1 0 0 0 0 0 0 0
!
! Lambda1 Lambda2 Ratio Bkpos Wdt Cthm muR AsyLim Rpolarz 2nd-muR -> Patt# 1
1.909500 1.909500 1.00000 30.000 6.0000 0.0000 0.0000 45.00 0.0000 0.0000
!
!NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
9 0.10 1.00 1.00 1.00 1.00 0.0690 0.050000 150.0000 0.000 0.000
!
! Excluded regions (LowT HighT) for Pattern# 1
0.00 15.00
!
!
0 !Number of refined parameters
!
! Zero Code SyCos Code SySin Code Lambda Code MORE ->Patt# 1
0.00000 11.0 0.00000 0.0 0.00000 0.0 0.000000 0.00 0
! Background coefficients/codes for Pattern# 1 (Polynomial of 6th degree)
50.000 0.000 0.000 0.000 0.000 0.000
41.00 51.00 101.00 0.00 0.00 0.00
!-----
! Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 0.41
!-----
YBaCuCoO
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
0 0 0 1.0 1.0 1.0 2 0 0 0 0 0.000 0 5 0
!
P 4/m m m <--Space group symbol
!-----> Profile Parameters for Pattern # 1
! Scale Shapel Bov Str1 Str2 Str3 Strain-Model
1.0000 0.00001 0.00000 0.00000 0.00000 0.00000 0
0.00000 0.000 0.000 0.000 0.000 0.000
!
! U V W X Y GauSiz LorSiz Size-Model
0.086000 -0.229000 0.254000 0.000000 0.000000 0.000000 0.000000 0
81.000 71.000 61.000 0.000 0.000 0.000 0.000
!
! a b c alpha beta gamma #Cell Info
3.869000 3.869000 11.660001 90.000000 90.000000 90.000000
21.00000 21.00000 31.00000 0.00000 0.00000 0.00000
! Pref1 Pref2 Asy1 Asy2 Asy3 Asy4
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00 0.00
! 2Th1/TOF1 2Th2/TOF2 Pattern to plot
15.000 150.000 1

```

**Figure 5:** The Cocuc\_LeBail.pcr control file for cell-constrained refinement

```

COMM YBA2CU2COO7 D1A
! Current global Chi2 (Bragg contrib.) = 10.15
! Files => DAT-file: Cocuc.dat, PCR-file: Cocus_start
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
1 5 1 0 1 0 1 0 0 0 0 0 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 1 1 0 4 0 0 1 1 -1 0 0 4 0 0
!
! Lambda1 Lambda2 Ratio Bkpos Wdt Cthm muR AsyLim Rpolarz 2nd-muR -> Patt# 1
1.909500 1.909500 1.00000 30.000 6.0000 0.0000 0.0000 45.00 0.0000 0.0000
!
!NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
9 0.10 1.00 1.00 1.00 1.00 0.0690 0.050000 150.0000 0.000 0.000
!
! Excluded regions (LowT HighT) for Pattern# 1
0.00 15.00
!
!
1 !Number of refined parameters
!
! Zero Code SyCos Code SySin Code Lambda Code MORE ->Patt# 1
-0.00610 41.0 0.00000 0.0 0.00000 0.0 0.000000 0.00 0
! Background coefficients/codes for Pattern# 1 (Polynomial of 6th degree)
56.868 0.405 -5.878 9.084 -3.647 0.452
91.00 101.00 111.00 121.00 131.00 141.00
!-----
! Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 12.92
!-----
YBaCuCoO
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
9 0 0 1.0 1.0 1.0 0 0 0 0 0 666.238 0 5 0
!
P 4/M M M <---Space group symbol
!Atom Typ X Y Z Basis Occ In Fin N_t Spc /Codes
Y Y 0.50000 0.50000 0.50000 0.70000 0.06250 0 0 0 0
0.00 0.00 0.00 191.00 0.00
BA BA 0.50000 0.50000 0.19200 0.70000 0.12500 0 0 0 0
0.00 0.00 151.00 201.00 0.00
CU1 CU 0.00000 0.00000 0.00000 0.70000 0.06250 0 0 0 0
0.00 0.00 0.00 211.00 261.00
CO1 CO 0.00000 0.00000 0.00000 0.70000 0.00001 0 0 0 0
0.00 0.00 0.00 211.00 -261.00
CU2 CU 0.00000 0.00000 0.36050 0.70000 0.12500 0 0 0 0
0.00 0.00 161.00 221.00 271.00
CO2 CO 0.00000 0.00000 0.36050 0.70000 0.00001 0 0 0 0
0.00 0.00 161.00 221.00 -271.00
O1 O 0.00000 0.00000 0.15400 1.00000 0.12500 0 0 0 0
0.00 0.00 171.00 231.00 0.00
O2 O 0.50000 0.00000 0.37940 1.00000 0.25000 0 0 0 0
0.00 0.00 181.00 241.00 0.00
O4 O 0.00000 0.50000 0.00000 1.00000 0.06250 0 0 0 0
0.00 0.00 0.00 251.00 281.00
!-----> Profile Parameters for Pattern # 1
! Scale Shape1 Bov Str1 Str2 Str3 Strain-Model
2.0731 0.11920 0.00000 0.00000 0.00000 0.00000 0
11.00000 81.000 0.000 0.000 0.000 0.000
! U V W X Y GauSiz LorSiz Size-Model
0.145660 -0.366010 0.368960 0.000000 0.000000 0.000000 0.000000 0
71.000 61.000 51.000 0.000 0.000 0.000 0.000
! a b c alpha beta gamma #Cell Info
3.868937 3.868937 11.659280 90.000000 90.000000 90.000000
21.00000 21.00000 31.00000 0.00000 0.00000 0.00000
! Pref1 Pref2 Asy1 Asy2 Asy3 Asy4
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00 0.00
! 2Th1/TOF1 2Th2/TOF2 Pattern to plot
15.000 150.000 1

```

**Figure 6:** The starting Cocus\_start-excl.pcr control file for structure constrained Rietveld refinement. Notice that the first strong peak affected by asymmetry is excluded.

The output of the program consists essentially in four files (but other files may exist too)

- `cocuxxx.pcr`: updated input file.
- `cocuxxx.out`: exhaustive result file
- `cocuxxx.sum`: summary of the refinement
- `cocuxxx.prf`: input file for plot program, which is automatically shown on the graph window at the end of refinement.

**As a final remark, do not forget that the work of the crystallographer is to understand the crystal structure. When the refinement ends, the real interesting work begins...**