

Plot of refined parameters after cyclic FullProf refinements

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Experimental requirements

- diffraction experiments versus external parameter (T, P ...)
- single format of data files
- particular data files name
- one starting PCR file (input parameters for FullProf)

All the collected patterns will be analysed with the same refinements conditions (background, physical model, number of phases, number of refined parameters...).

The evolution of the refined parameters has to be continuous.

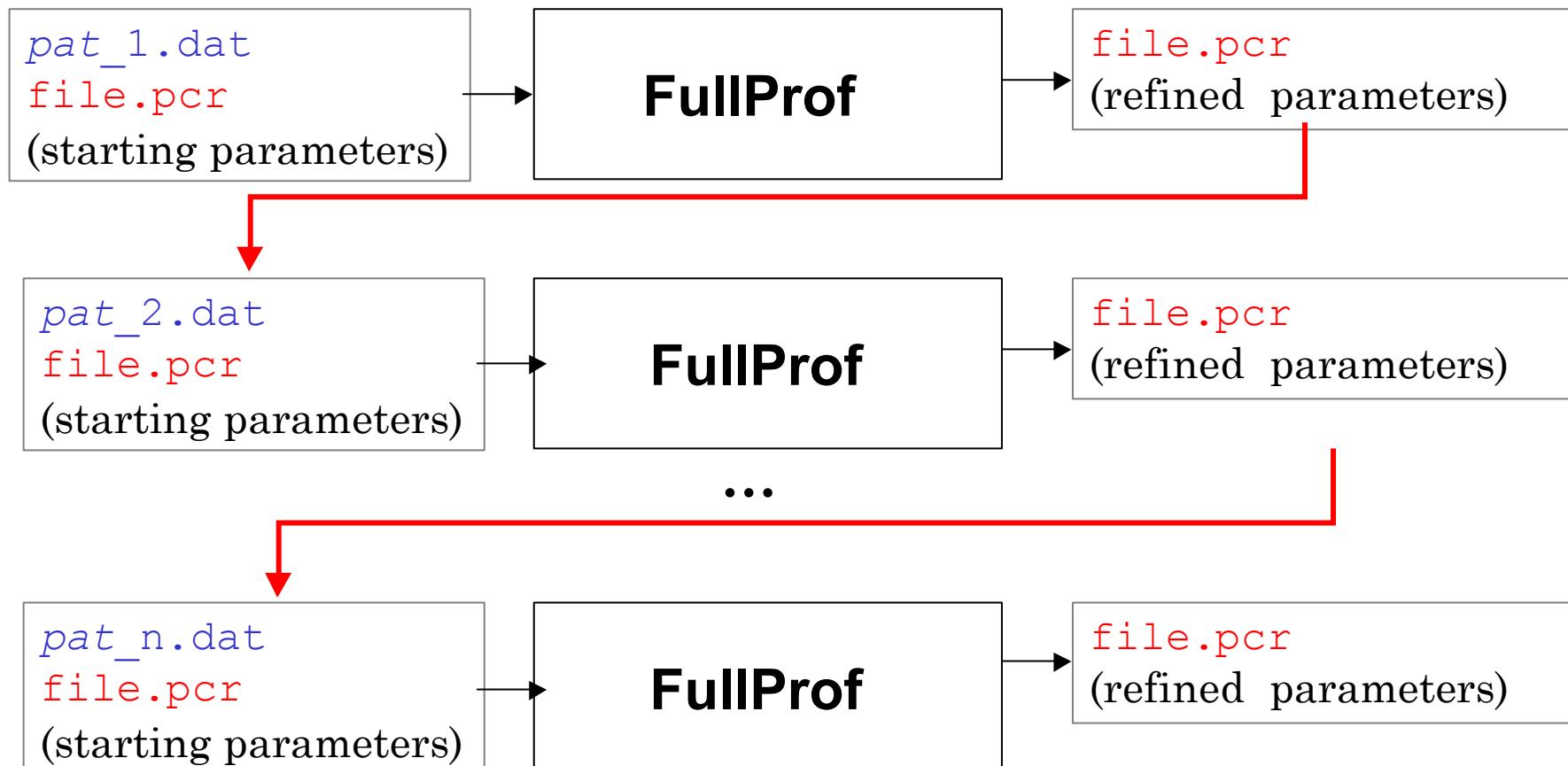
FullProf in cyclic mode

Diffraction data:

pat_1.dat, pat_2.dat ... pat_n.dat

FullProf input file:

file.pcr (starting parameters) **PCR=1**



FullProf output files

- .SEQ (if RPA=-2) refined parameters
- .datcodeFPCYC.hkl hkl features (intensities, positions, FWHM)

Launching FullProf in cyclic mode

- Version of FullProf: `wfp2k.exe` (Windows) or `fp2k.exe` (console version)
- Run through command line, using arguments list:

```
> (w) fp2k arg(1:9)
```

with:

<code>arg_1:</code>	<code>cyc</code>
<code>arg_2:</code>	pcr file
<code>arg_3:</code>	data file name
<code>arg_4:</code>	numor of starting file
<code>arg_5:</code>	numor of final file
<code>arg_6:</code>	save .PRF files (y/n)
<code>arg_7:</code>	save .MIC files (y/n)
<code>arg_8:</code>	create .hkl files (y/n) (only for profile matching refinement)
<code>arg_9:</code>	create .dis files (y/n) (only for Rietveld refinement)

Launching FullProf in cyclic mode

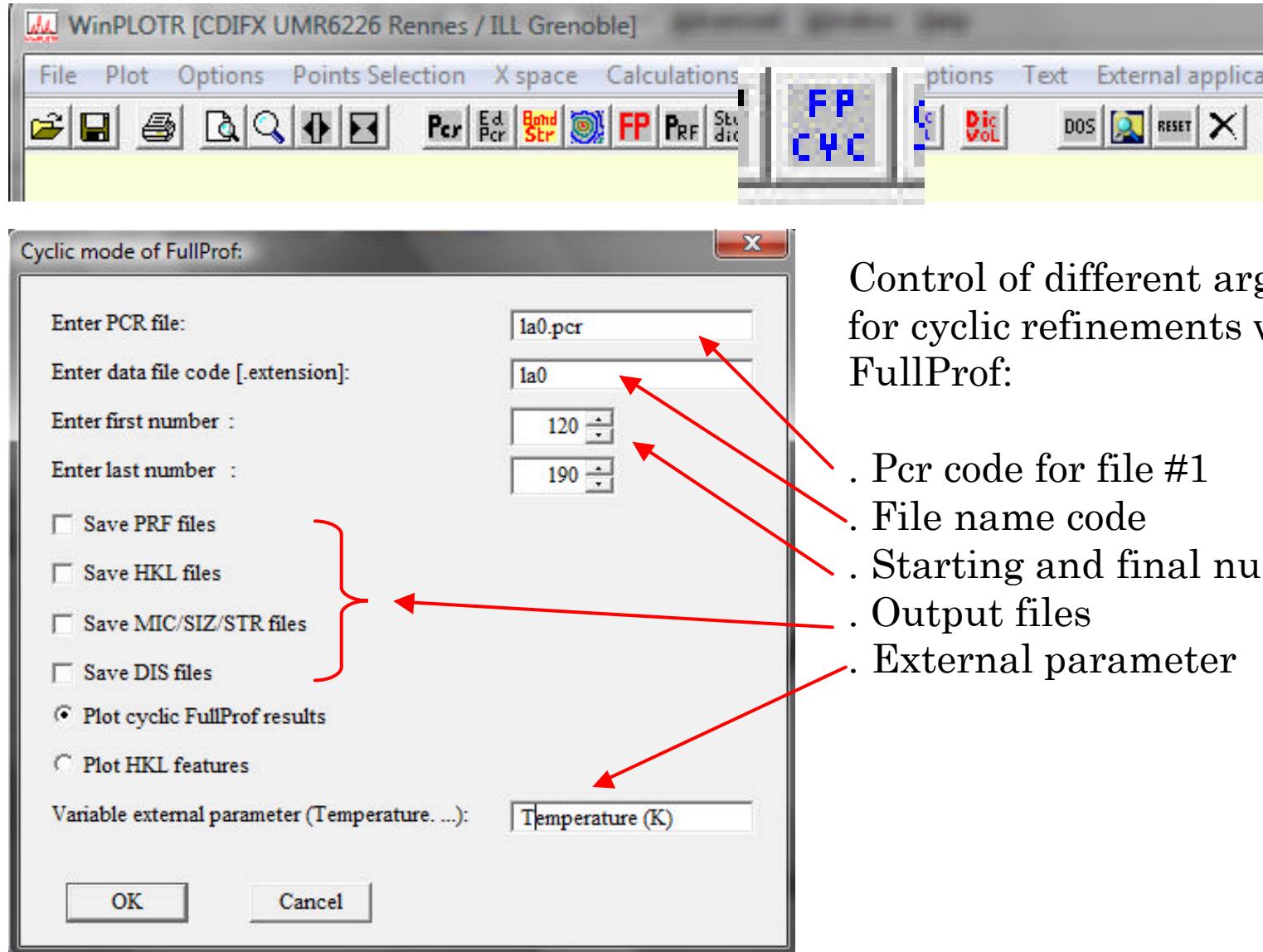
- Example:

```
> wfp2k cyc my_pcr datcode.uxd 23 268 y n y n
```

Starting PCR file: my_pcr

Pattern data: .UXD extension
files names: datcode23.uxd
 datcode24.uxd
 ...
 datcode268.uxd

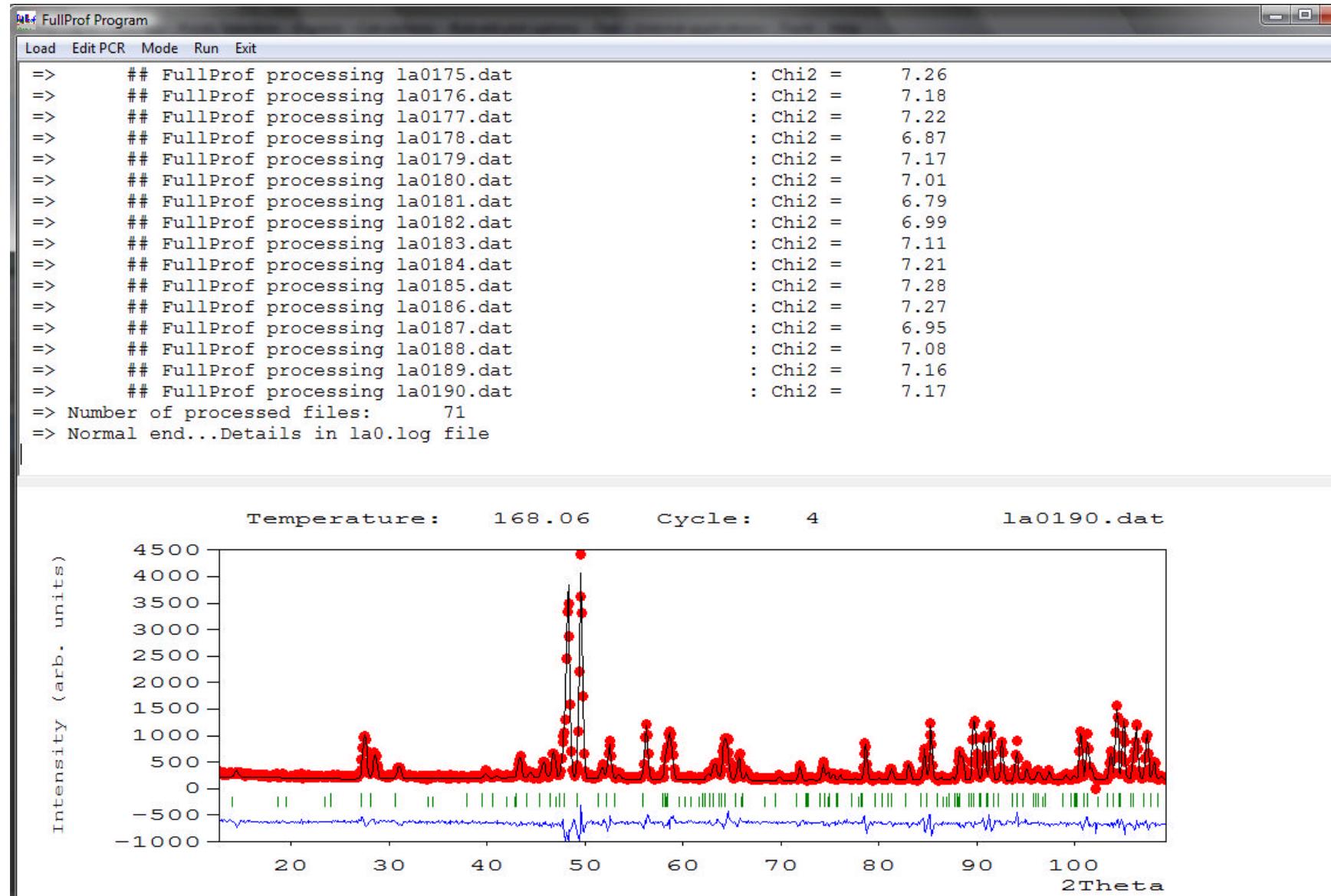
FullProf cyclic refinements using **WinPLOTR** GUI



Control of different arguments for cyclic refinements with FullProf:

- . Pcr code for file #1
- . File name code
- . Starting and final numbers
- . Output files
- . External parameter

FullProf cyclic refinements



Rem:55 sec. to refine 70 diffraction patterns (Rietveld mode / 21 parameters / 1535 points) !

1. After Rietveld type refinement

Output file created by FullProf: filename.SEQ (if RPA=-2)

- NPAT: number of refined data files
- NPAR: number of refined parameters for each file

filename.SEQ file contains, in append mode (from the pattern #1 to pattern #NPAT):

- global refinement parameters: Chi^2 , $R_B(\text{PAT})$, $R_F(\text{PAT})$
- structural refined parameters:
 - . Atomic positions
 - . ADP
 - . Occupancy (ordering, composition)
 - . Magnetic components

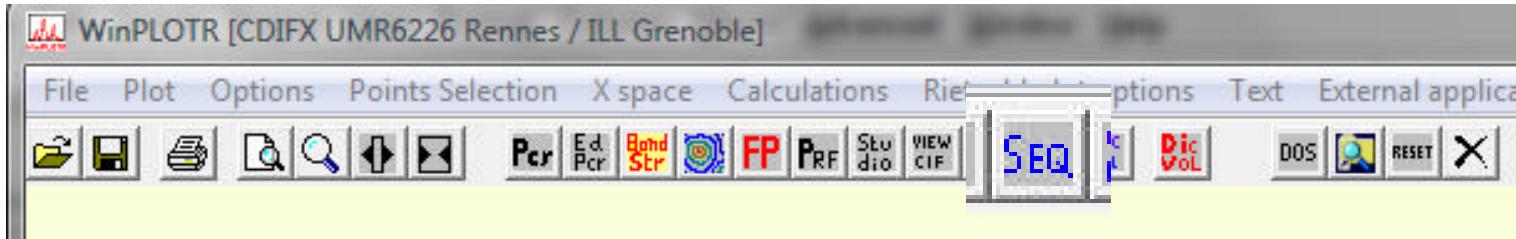
Contain of the filename.SEQ file

- profile related parameters:
 - . Cell parameters
 - . Zero shift
 - . U,V,W,X,Y,eta...
 - . Broadening line parameters (microstructure)

- background parameters

NEW_REFINEMENT		la0120
NUMPAR	21	
N_PATT	1	
NPHASE	2	
PARVAL	0	68.8610 73430
GLOBAL_CHI_2		4.96969
1	R_Bragg(%):	8.5521326 6.0470099
1	Unit_Cell_Volume:	245.05150 0.72612939E-02
1	Weight_Fraction:	100.00000 1.8796988
2	R_Bragg(%):	8.6017923 8.5862312
2	Unit_Cell_Volume:	245.05150 0.72612939E-02
2	Weight_Fraction:	0.0000000 0.0000000
1	Scale_ph1_pat1	1.2691045 0.16868232E-01
2	Zero_pat1	0.40351850 0.35467718E-02
3	Cell_A_ph1_pat1	5.5425119 0.95329837E-04
4	Cell_B_ph1_pat1	5.7574425 0.96214215E-04
5	Cell_C_ph1_pat1	7.6792908 0.13365930E-03
6	U_ph1_pat1	0.17840168 0.69734403E-02
7	V_ph1_pat1	-0.39761341 0.14373429E-01
8	W_ph1_pat1	0.30380064 0.69085192E-02
9	Ry_Mn1_ph2	3.5205884 0.41629475E-01
10	X_La_ph1	-0.81843417E-02 0.60215301E-03
11	X_O_ph1	0.78588009E-01 0.72646193E-03
12	Y_La_ph1	0.49782123E-01 0.57684432E-03
13	X_O_ph1	0.72687072 0.61505073E-03
14	Y_O_ph1	0.30875507 0.61562215E-03
15	Z_O_ph1	0.39268631E-01 0.39140851E-03
16	Biso_Mn_ph1	-0.33981135 0.12610333
17	Biso_La_ph1	0.0000000 1.0000000
18	Biso_O_ph1	0.0000000 1.0000000
19	Biso_O_ph1	0.77939883E-01 0.66306442E-01
20	Asym1_ph1_pat1	0.75355873E-01 0.12604496E-01
21	Asym2_ph1_pat1	0.33547625E-01 0.52135768E-02

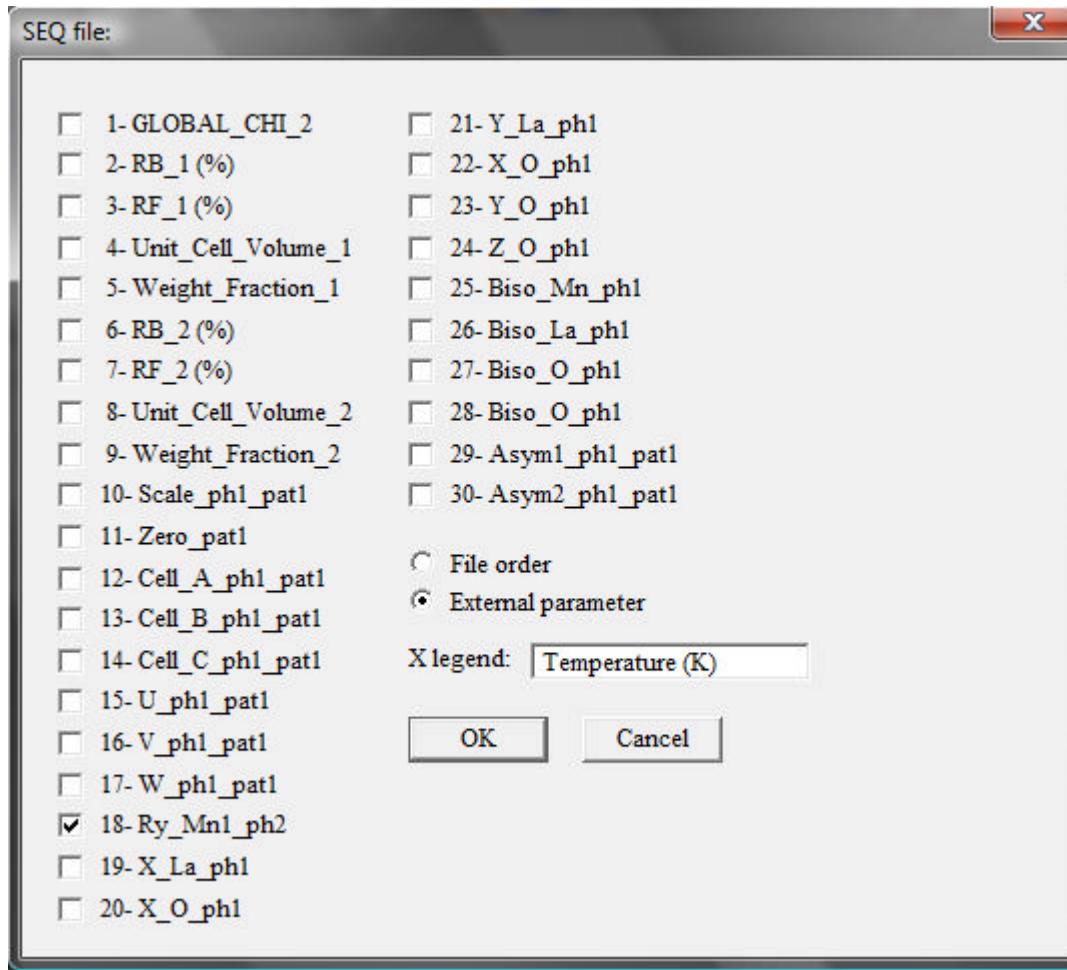
Use of **WinPLOTR** (blue? version) to plot the evolution of refined parameters during cyclic FullProf



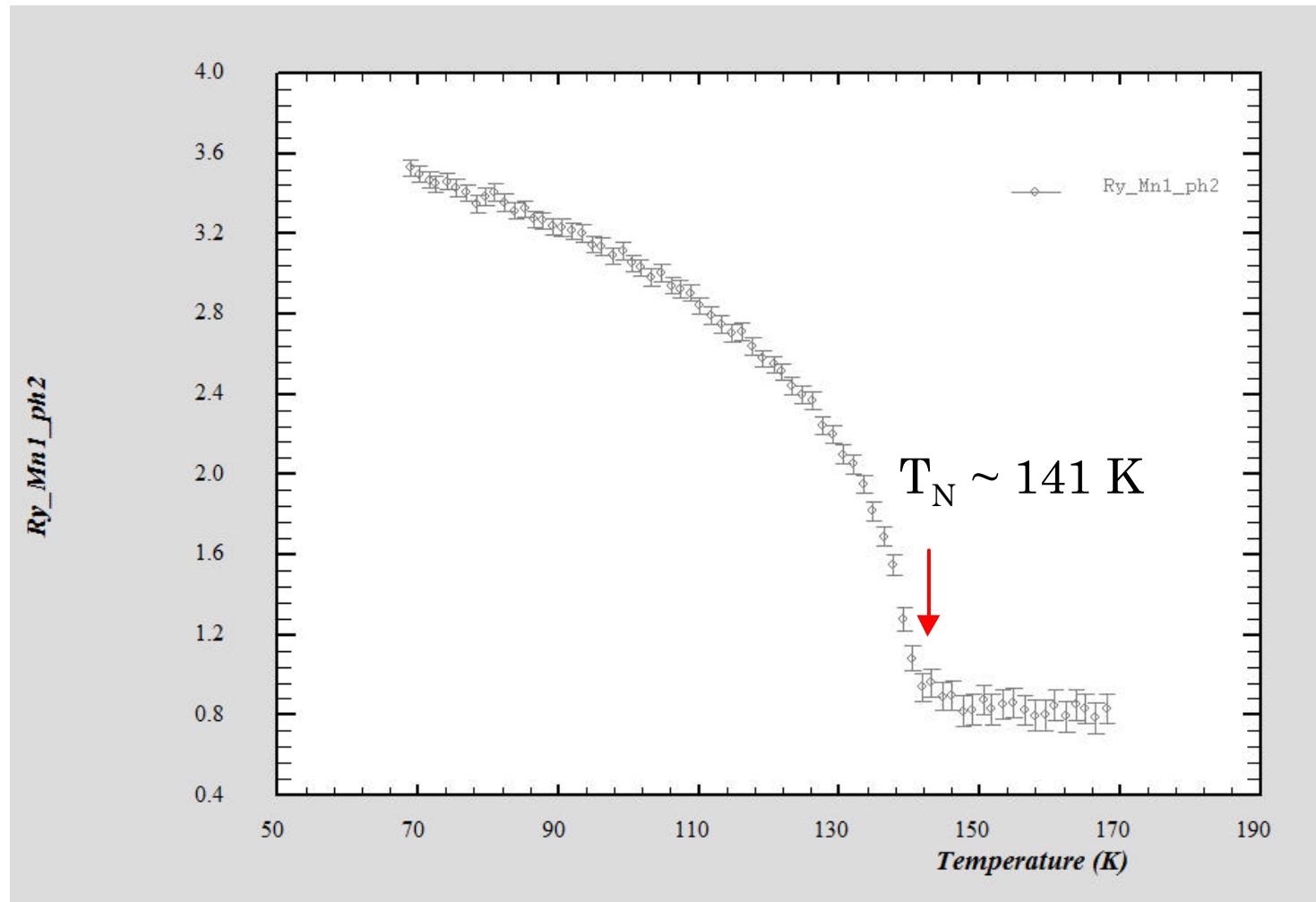
- SEQ** button:
- . select a .SEQ file
 - . Open a dialog box with refined parameters list
 - . Select parameter(s) to plot
- . Several .SEQ files can be opened simultaneously to plot evolution of parameters coming from different FullProf cyclic refinements (i.e. different temperature ranges)
- . Errors bars are automatically display (use **Option/error bars** to remove them)

RESET button to change « sample job »

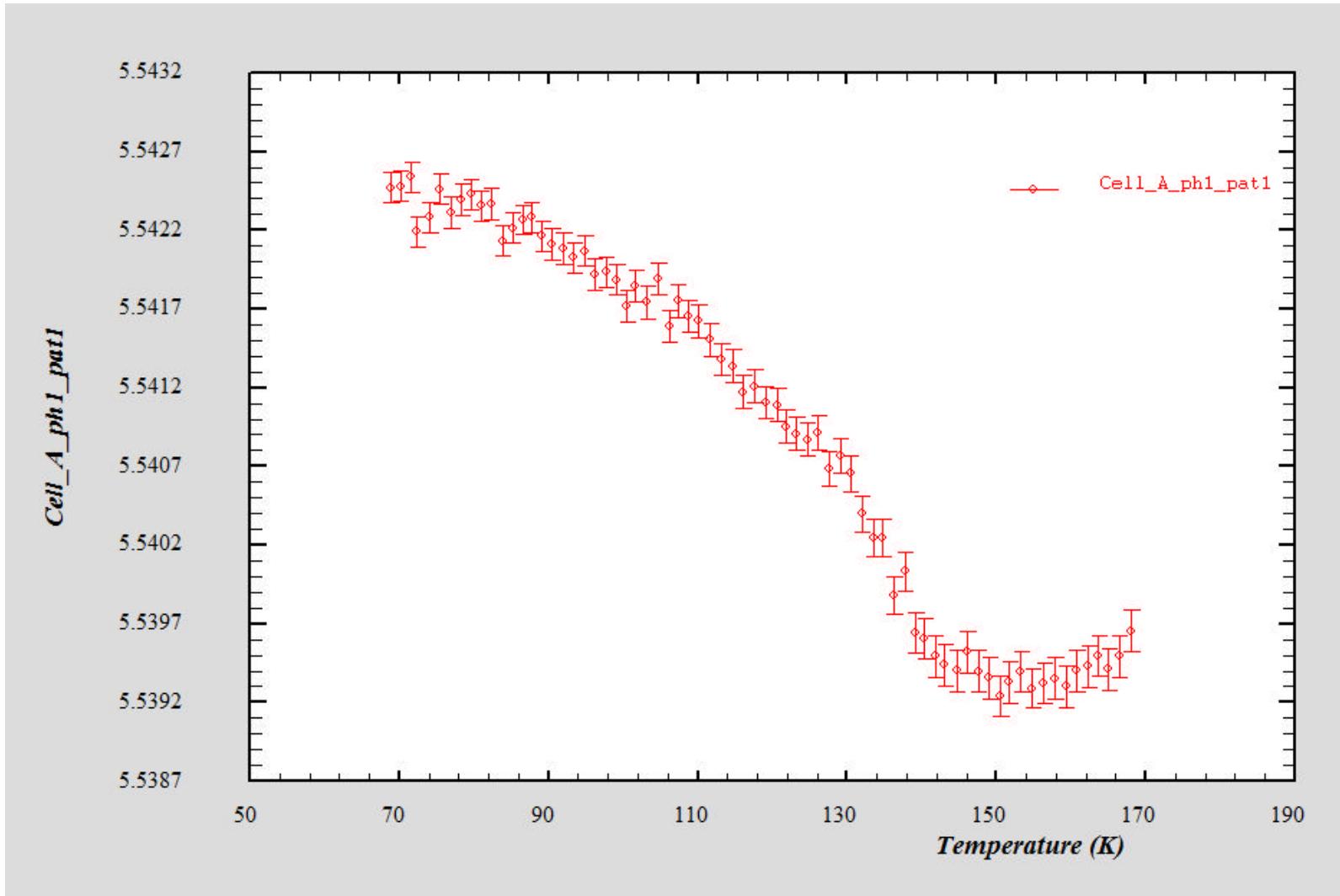
Use of WinPLOTR (« blue » version) to plot the evolution of refined parameters during cyclic FullProf



Use of **WinPLOTR** (« blue » version) to plot the evolution of refined parameters during cyclic FullProf

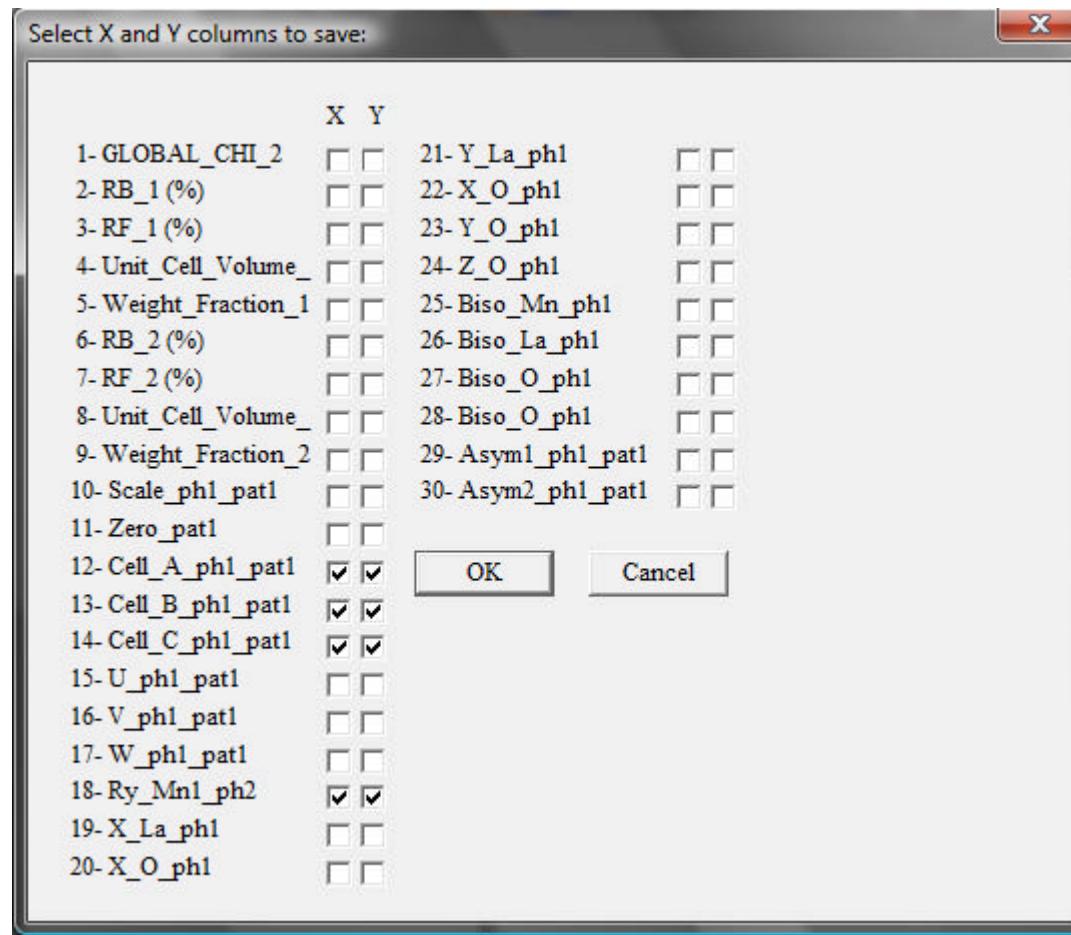


Use of **WinPLOTR** (« blue » version) to plot the evolution of refined parameters during cyclic FullProf



WinPLOTR creates output files containing evolution of refined parameters

File/save data / save data as multicolumns file (.XYN)

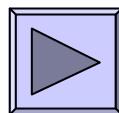


Format of the **.XYN** multicolumns file:

Number of columns = 3^*NPAR (X, Y, sig)_{NPART}

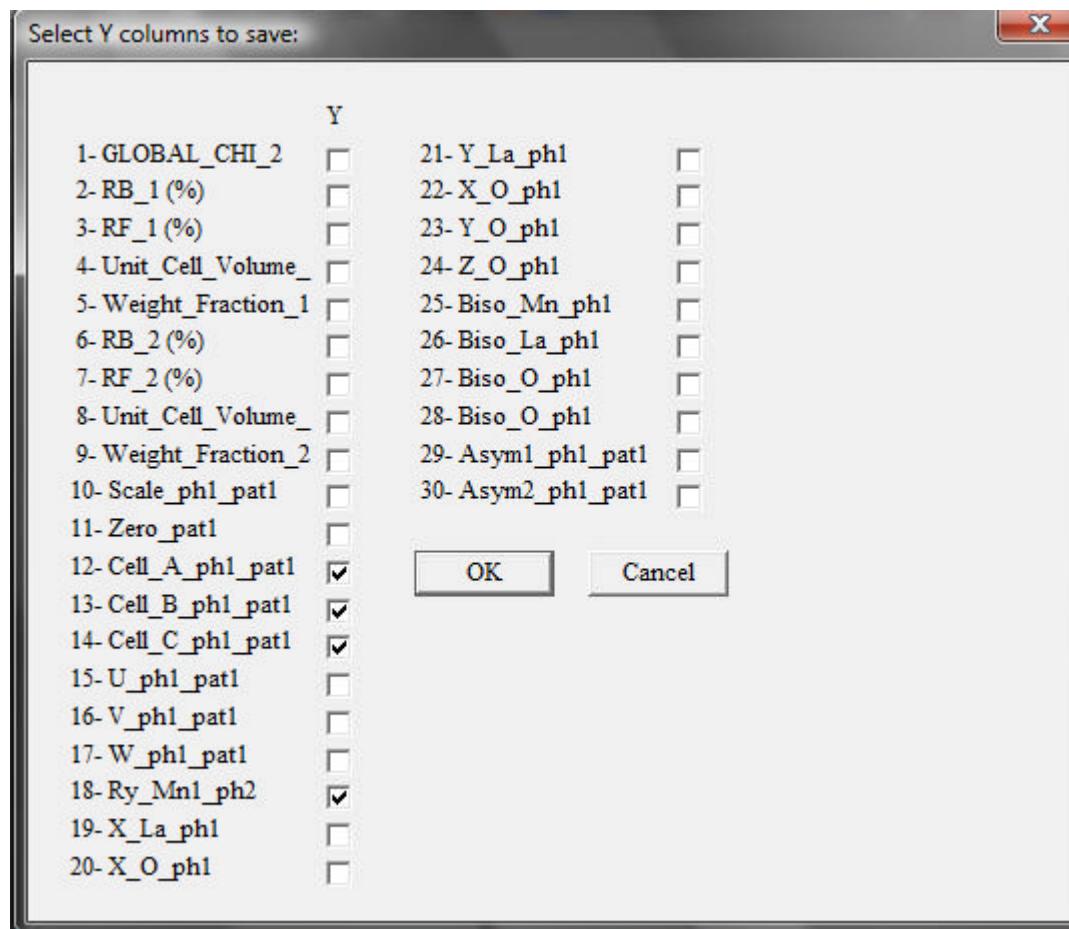
Number of lines = NPAT

Rectangular format for ORIGIN, SigmaPLOT, EXCEL



WinPLOTR creates output files containing evolution of refined parameters

File/save data as/save as .XYY

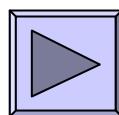


Format of the **.XYY** multicolumns file:

Number of columns = 1 (X) + 2*NPAR (Y, sig)_{NPAR}

Number of lines = NPAT**2

Rectangular format with « holes » for **KaleidaGRAPH**, **ORIGIN**, **EXCEL**,
WinPLOTR (Open/.XYY)



2. After profile matching type refinement

Output file created by FullProf: filename_fpcyc.hkl

- NPAT: number of refined data files
- NREF: number of reflexions

Filename_fpcyc.SEQ file contains, in append mode (from the pattern #1 to pattern #NPAT):

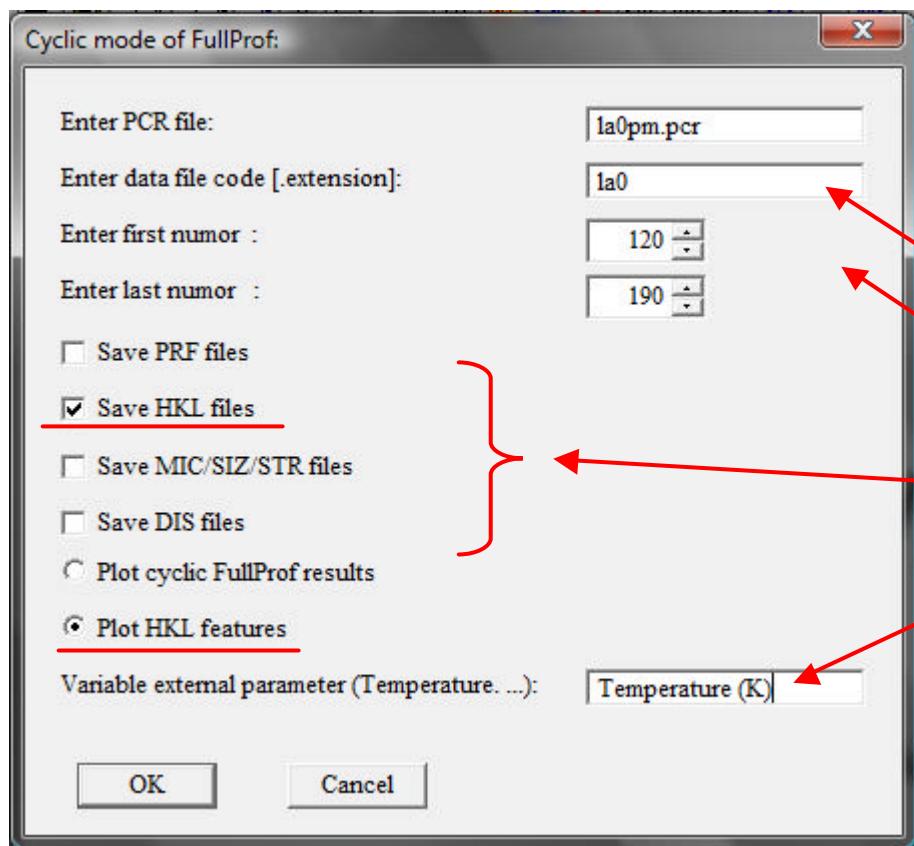
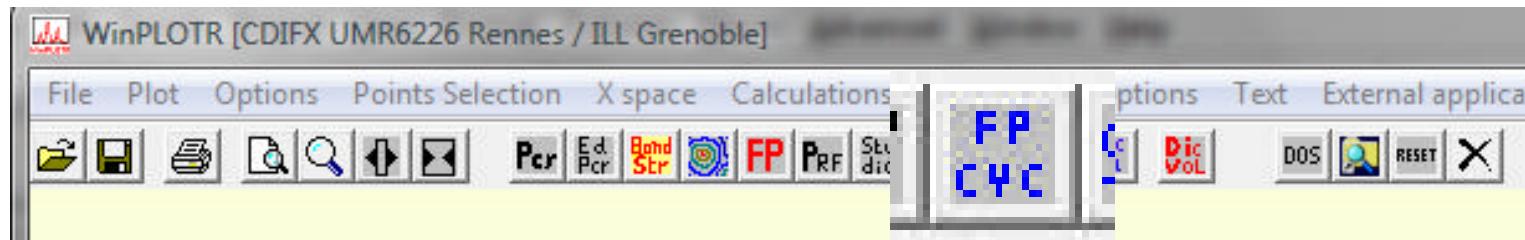
- for every reflexion H:

h k l nvk m I sig(I) 2THETA ($^{\circ}$) FWHM ($^{\circ}$)

Format of filename_fpcyc.hkl file

```
# Cyclic FullProf:  
# Number of data files:    71  
#  
#   PCR file: la0pm.pcr  
#   DAT file: la0120.dat  
#   HKL file: la0120_1.hkl  
Pattern# 1 Phase No: 1 LaMnO3 x=0.0 Nuclear Lambda: 1.870000 CELL:      5.5426  
193    73430    68.86  SPGr: P m m m  <-- The number of effective reflections may be lower  
      0    0    1    2      1137.692      13.839      13.9855      0.5001  
      0    1    0    2       4.515       6.154      18.6924      0.4861  
      1    0    0    2       0.001       0.002      19.4236      0.4840  
      0    1    1    4       0.001       0.002      23.4209      0.4721  
      1    0    1    4       4.110       5.982      24.0145      0.4703  
      1    1    0    4      370.325      9.284      27.0842      0.4613  
      0    0    2    2      199.992      8.163      28.1849      0.4580  
      1    1    1    8      517.822     10.513      30.6052      0.4508  
      0    1    2    4       0.001       0.002      34.0365      0.4406  
...  
...
```

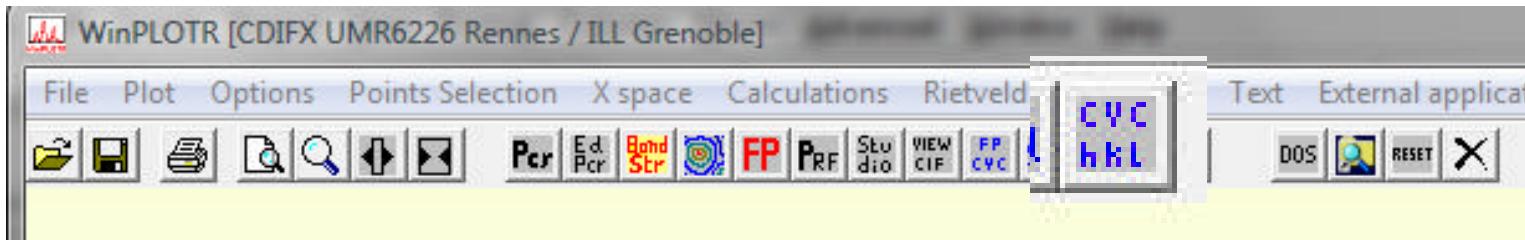
Profile matching sequential FullProf refinements using WinPLOTR GUI



Control of different arguments for cyclic refinements with FullProf:

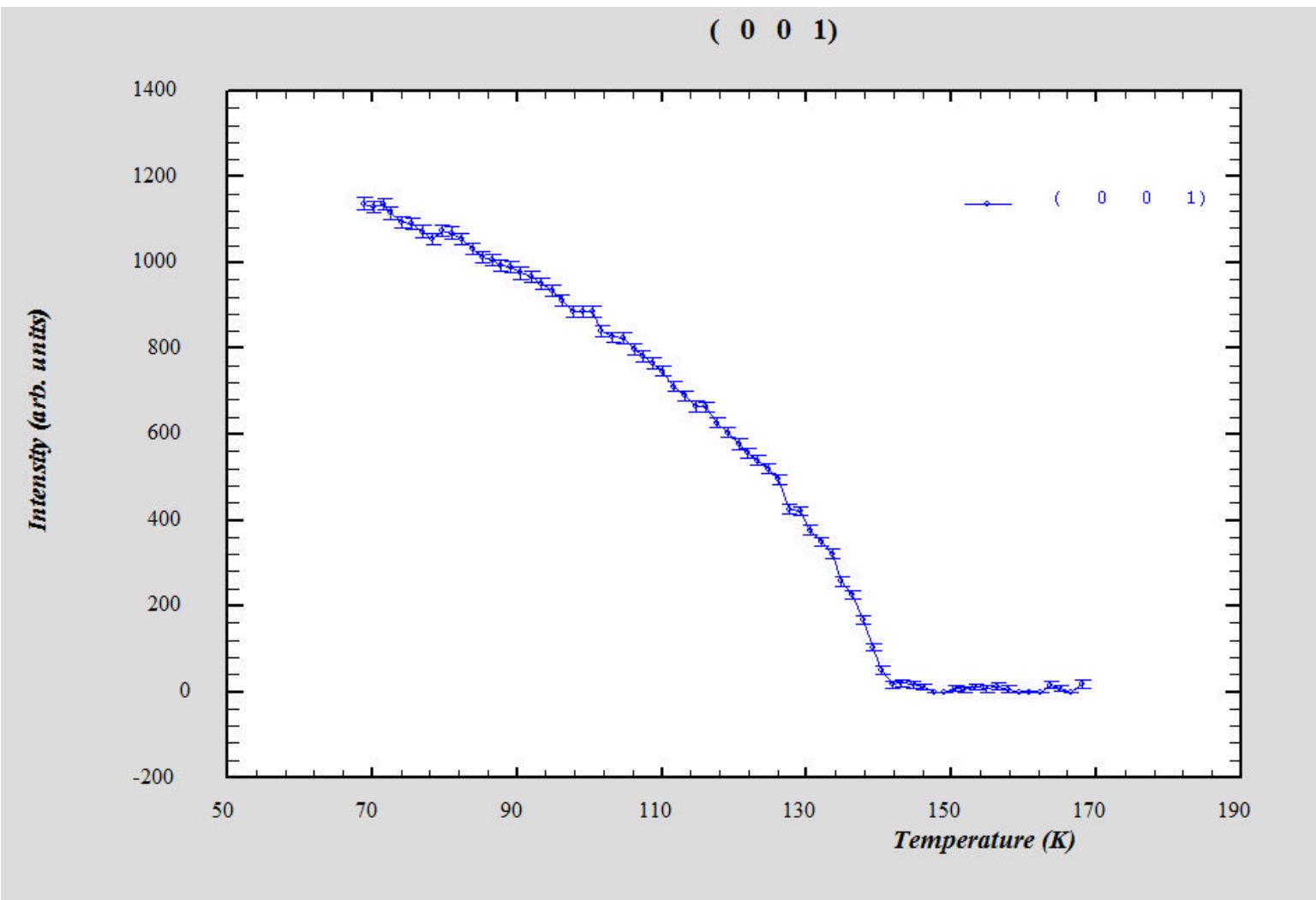
- . Pcr code for file #1
- . File name code
- . Starting and final numors
- . Output files
- . External parameter

Use of **WinPLOTR** (« blue » version) to plot the evolution of reflexions related parameters during cyclic FullProf



- CYChkl** button:
- . select a filename_fpcyc.hkl file
 - . Open a dialog box with refined parameters list
 - . Select (hkl) reflexion
 - . Select reflexion parameter (intensity/position/FWHM)
 - . Several .SEQ files can be opened simultaneously to plot evolution of reflexions coming from different FullProf cyclic refinements (i.e. different temperature ranges)

RESET button to change « sample job »



Buttons are then activated, allowing to plot every reflexions

