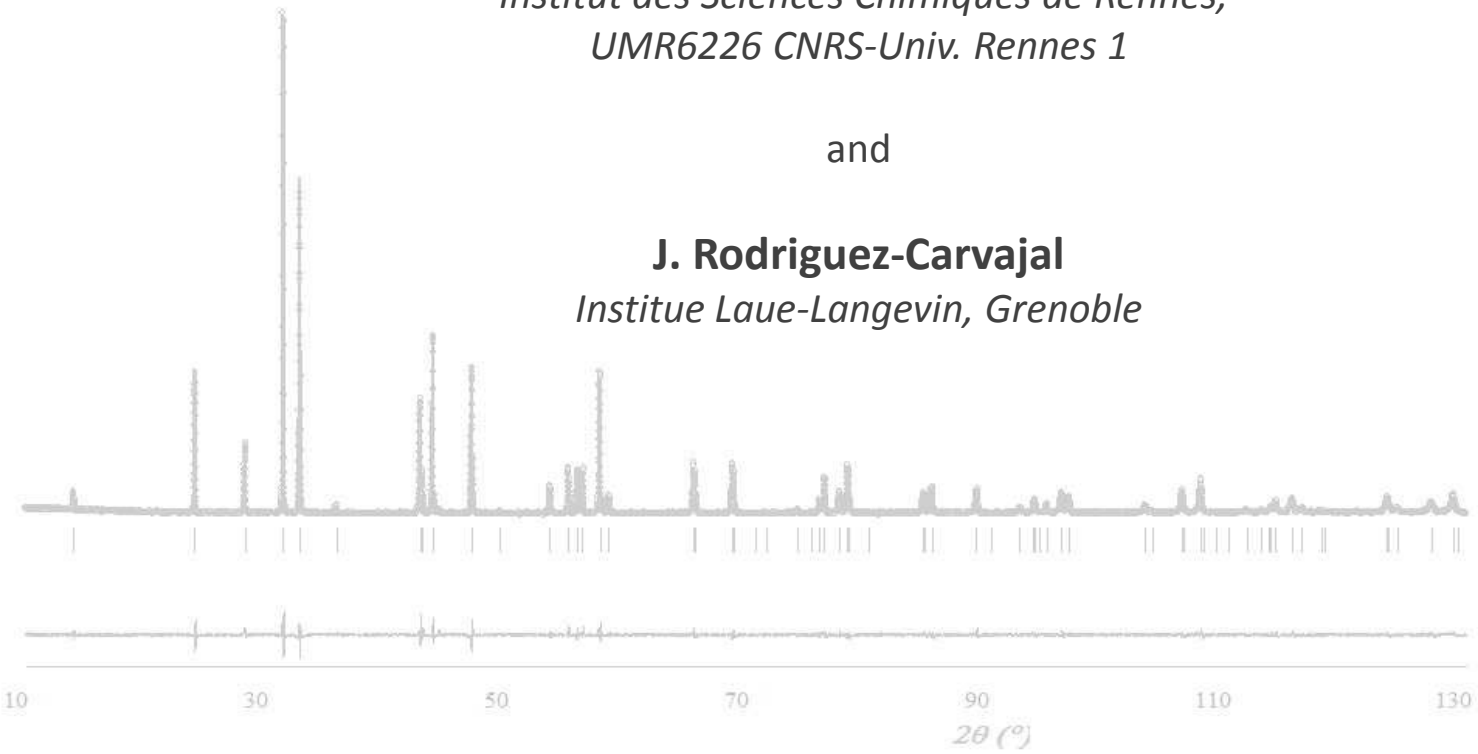
WinPLOTR :

a graphical tool for powder diffraction data analysis

T. Roisnel
Centre de Diffractométrie X (CDIFX)
Institut des Sciences Chimiques de Rennes,
UMR6226 CNRS-Univ. Rennes 1

and

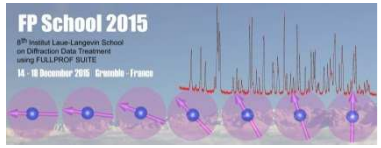
J. Rodriguez-Carvajal
Institue Laue-Langevin, Grenoble



http://www.cdifx.univ-rennes1.fr/winplotr/winplotr.htm

(Tutorials and demos, download, what's new ?, links …)

What can be done with WinPLOTR ?

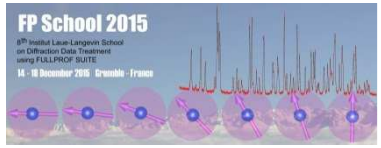


WinPLOTR has been designed following two concepts :

- **WinPLOTR** is a program to plot and analyse powder diffraction patterns. It can be used to plot raw or normalized data files coming from different kinds of diffractometers (neutron and X-ray, conventional or synchrotron radiation) as well as profile refinement files (**.PRF**) created by the FullProf program.

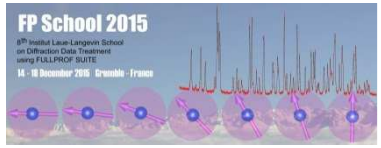
- **WinPLOTR** can also be used as a Graphical User Interface for programs used frequently in powder diffraction data analysis (ex: FullProf, DicVol ...) or other external programs defined by the user.

1. Plot and process powder diffraction data



Powder diffraction data features

- **Raw data, normalized ou refined data**
  - neutrons, X-Rays (conventional or synchrotron)
  - constant wavelength, dispersive energy, time of flight
- **Large number of data file formats (only ASCII) coming from several kinds of diffractometers** (commercial and home-made)
- **File conversion** : save data as **`INSTRM0`** FP format, multicolumns
- **Scattering space**
  - scattering variable : 2θ(°)/time of flight (µsec.)/energy (KeV)
  - reciprocal space : Q ($Å^{-1}$) , 1/d ($Å^{-1}$), sinθ/λ ($Å^{-1}$)
  - direct space : d (Å)
- **Different Y scales** : linear, log, √, $I.Q^2$



Graphical features

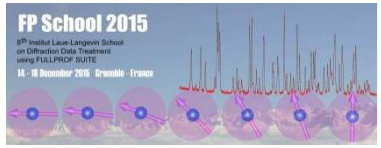
- **Graphics options (mouse handling)**
  - Cursor informations : X (including 2θ/d values simultaneously) and Y values
  - Zoom, focus
  - Select and save points (countings, background)

- **Plot options**
  - Change colors, markers type and size, styles …
  - Shift and offset the data (in X and/or Y) [pseudo 3D plots]
  - Hidden parts management
  - Error barrs

- **Graphics output files**
  - Bitmap
  - Postscript
  - WinPLOTR output file (**`.wpl`**, **`.pgf`**)

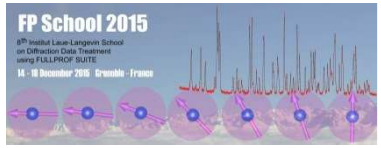
…

1. Plot and process powder diffraction data



Calculations features

- **Automatic search procedures**: background points, Bragg reflections
- **Background substraction**
- **Calculations on diffraction patterns** :
  - patterns summation
  - pattern difference
  - smoothing
  - multiply X and Y
  - multiscan data normalisation
- **Profile fitting procedure** (interactif / automatic) (T.C.H. profile function)
- **Microstructural analysis** (Williamson Hall plots)



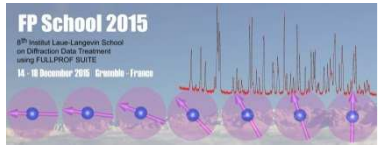
Calculations tools

- **FWHM calculation**
  - Cagliotti formula
  - T.C.H. formula

- **Crystallographic calculations** :
  - space groups
  - *hkl* list generation
  - unit cell volume
  - …

…

2. GUI for external programs



- **FullProf** *(JRC): profile refinement (Rietveld, whole pattern fitting)

- **.PCR** input file selection and edition

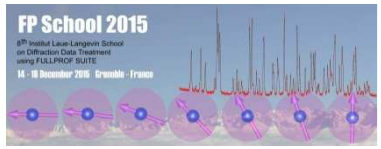
- FullProf launching : selection of input files (**.pcr** and diffraction data file)

- **.PRF** file plotting (Rietveld plot) : Yobs, Ycalc, Yobs-Ycalc, Bragg positions
  - (hkl) information (Bragg peak position, indexation …)
  - display (hkl) features

- Sequential FullProf refinement (ex: data=f(T)) and plot refined parameters

*included in the FPSuite package

2. GUI for external programs



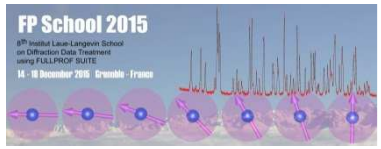
- Automatic indexation : **DICVOL** * (D. Louër), **TREOR** * (P.E. Werner), …:
  - Peak search
  - Save Bragg peak positions as input files for indexation programs (dialog box)
  - **WinDICVOL/WinTREOR90** launching
  - If successfull indexation : automatic creation of a **PCR** file for **FullProf** (« pattern matching » mode)

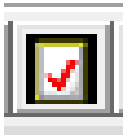
- **SuperCELL*** (JRC) : determination of a super unit cell

- **K-search*** (JRC) : search propagation wave vector components of a magnetic or structural modulated structure
  - Save Bragg peaks on difference pattern of a **PRF** file

*included in the FPSuite package

2. GUI for external programs





Check_Group* (JRC) : search of a space group from a list of integrated intensities

BOND_STR* (JRC) : distances, angles and bond valence calculations

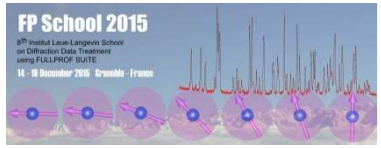
EdPCR* (JGP-JRC): . editing and modifying **PCR** input file

. Files conversion: **CIF** to **PCR**, **INS** to **PCR**

- Any program defined by the user (**winplotr.set** setting file)

*included in the FPSuite package

winplotr.set setting file

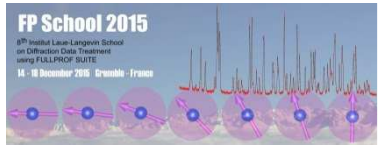


- Located in the folder associated to the **WINPLOTR** environment variable
  (ex: **c:\>fullprof_suite**)

- Definitions of :
  - arrays dimensions
  - external programs to execute through **WinPLOTR**
  - system applications : browser, editor …
  - graphical plot options : colours, markers …
  - instrumental parameters : λ, IRF
  - data files extensions
  - …

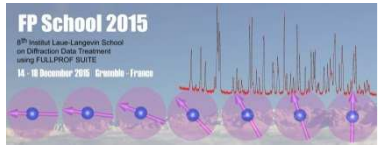
- Accessible via the « **External applications / Edit a file / winplotr.set file** » menu option

winplotr.set setting file



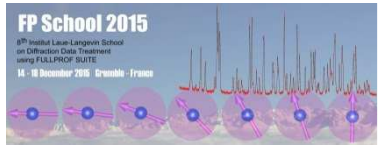

```
*********************************************
*                                           *
*     W i n P L O T R   s e t t i n g s     *
*                                           *
*********************************************

[MAIN WINDOW POSITION AND SIZE]
      0.100  0.100  0.800  0.800
.
.
.
[DIMENSIONS OF ARRAYS]
 Max_Patterns =       20    ! max. number of patterns
 Max_Points   =    60000    ! max. number of data points
 Max_Refl     =    20000    ! max. number of Bragg reflections
.
.
.
[RUN PROGRAMS]
     FullProf = wfp2k                                  ! FullProf program    !  WINDOWS VERSION
         Edit = C:\Program Files\Keditw\KEDITW32.EXE   ! My favorite editor  !  WINDOWS VERSION
       Dicvol = wdicvol06                              ! Dicvol program      !  WINDOWS VERSION
    SuperCell = SuperCell                              ! SuperCell program   !  WINDOWS VERSION
        Treor = wtreor90                               ! TREOR90 program     !  WINDOWS VERSION
          ITO = wito15                                 ! ITO15 program       !  WINDOWS VERSION
       MENDEL = mendel                                 ! MY PERIODIC TABLE   !  WINDOWS VERSION
     GFOURIER = gfourier                               ! FOURIER PROGRAM     !  WINDOWS VERSION
    @CALC_PDF = calc_pdf                               ! PDF calculation     !  WINDOWS VERSION
    @CRYSCALC = d:\progs\cryscalc                      ! Crystallographic calculator  !  WINDOWS VERSION
@WINPLOTR2006 = D:\FullProf_Suite\WinPLOTR-2006.exe    ! WinPLOTR_2006       ! Windows version
    @K-search = k_search.exe                           ! K_search            ! DOS
       @VESTA = D:\progs\VESTA-win64\vesta.exe         ! VESTA               ! W
        @EXPO = D:\progs\EXPO2004\expo2004.exe         ! EXPO                ! W
.
.
.
[SYSTEM APPLICATIONS]
Browser          = "C:\PROGRAM FILES (X86)\MOZILLA FIREFOX\FIREFOX.EXE"
PostScriptViewer = "C:\Program Files\gs\gs9.14\bin\gswin64.exe"
PDFreader        = "C:\Program Files (x86)\Adobe\Reader 11.0\Reader\ACRORD32.EXE"
BMPviewer        = "C:\WINDOWS\SYSTEM32\MSPAINT.EXE"
CIFviewer        = "D:\PROGS\CCDC\MERCURY 3.1\MERCURY.EXE"
```

winplotr.set setting file



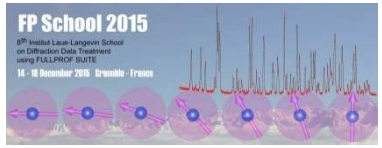

```
[AFTER FULLPROF RUN]
  plot_prf  edit_pcr  no_draw_fst
.
.
.
[MY WAVELENGTHS (A)]
    1.54060  1.54060  1.00000  0.00000

[MY RESOLUTION PARAMETERS (U,V,W,X,Y,Z)]
! HG**2= U.tan**2(theta) + V.tan(theta) + W
! HL    = X.tan(theta) + Y/cos(theta) + Z
    0.0100 -0.0100  0.0050  0.0000  0.0000  0.0000
.
.
.
[DATA FILES EXTENSIONS]
! format_number   extension                              type
       1     *.XY;*.DAT;*.BGR;*.EPF;*.XYN;*.XYS  ! 1. X,Y data + INSTRM=10
       2     *.DAT;*.SUB;*.SIM;*.BAC             ! 2. INSTRM=0: Free F.(Ti,step,Tf)
       3     *.DAT                               ! 3. INSTRM=1: Old D1A
       4     *.DAT;*.D1B                         ! 4. INSTRM=3: D1B (ILL)
       5     *.DAT                               ! 5. INSTRM=4: Brookhaven(Synchr.)
       6     *.DAT                               ! 6. INSTRM=5: G4.1
       7     *.DAT                               ! 7. INSTRM=6: D2B/3T2/G4.2
       8     *.DAT                               ! 8. INSTRM=8: HRPT/DMC (PSI)
       9     *.UXD                               ! 9. INSTRM=9: .UXD (D8 CSM)
      10     *.DAT                               ! 10. INSTRM=11: Variable Time step
      11     *.DAT;*.GSS;*.GSA                   ! 11. GSAS data
      12     *.CPI                               ! 12. CPI (Xrays)
      13     *.UDF;*.XRDML;*.JCP;*.CSV           ! 13. PANalytical formats
      14     *.DAT;*.GSS;*.GSA                   ! 14. INSTRM=14: ISIS normalized data
      15     *.TXT                               ! 15. Rigaku RINT
      16     *.SPC                               ! 16. DIFFAX (.spc)
.
.
.
[MY DEFAULT FORMATS (pattern, raw, Rietveld/profile)]
     1  56 101

[MY COMMAND LINE DEFAULT FORMATS (.dat, .uxd, .prf)]
    2   56 101
```

winplotr.set setting file



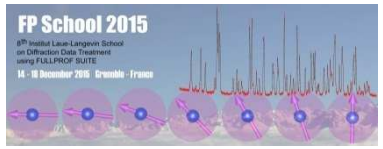

```
[BACKGROUND SCREEN]
  background screen color: RGB(220,220,220)
  background text color:   RGB( 60, 60, 60)
  background plot color:   RGB(255,255,255)
  plot frame color:        RGB(  0,  0,  0)


[EXCLUDED REGION COLOR]
 RGB(192,192,192)
.
.
.
[PATTERNS PLOT OPTIONS]
! n     color             marker    size    style    pen_width
  1 RGB(  0,  0,255)        4       1.0       1           1
  2 RGB(255,  0,  0)        4       1.0       1           1
  3 RGB(  0,128,  0)        4       1.0       1           1
  4 RGB(255,  0,255)        4       1.0       1           1
  5 RGB(128,  0,  0)        4       1.5       1           1

[PRF PLOT OPTIONS]
! n     color             marker    size    style   pen_width
  1 RGB(255,  0,  0)        4       1.0       0           2
  2 RGB(  0,  0,  0)        4       0.0       1           2
  3 RGB(  0,  0,255)        4       0.0       1           1
  4 RGB(  0,128,  0)        8       3.0       0           1
write_Y_negative_graduations=0
write_PRF_filename=0
exclude_CELL_from_title=1
colored_tics=0
.
.
.
[TEXT FONTS]    Typeface              point     italic  underline  strikeout       bold
 main title   :  Arial                 18          F          F          F           F
 X legend     :  Times New Roman       18          T          F          F           T
 Y legend     :  Courier New           18          T          F          F           T
 X graduations:  Courier New           16          F          F          F           T
 Y graduations:  Courier New           16          F          F          F           T
 file name    :  Courier               10          F          F          F           F
 (hkl) indices:  Times New Roman       10          F          F          F           F
 dialog boxes :  Times New Roman       10          F          F          F           F
```

WinPLOTR demos



- Visualisation of (pseudo) 3D plots
- Run **FP** in sequential mode and plot refined parameters
- Profile fitting procedure
- Background points selection
- WinPLOTR tools
- Run WinPLOTR in command line

Visualisation of (pseudo) *3D* plots



1. Powder diffraction data

- Create a buffer file containing the list of data files (common format) to plot, by using the

  Tools / create buffer file menu option

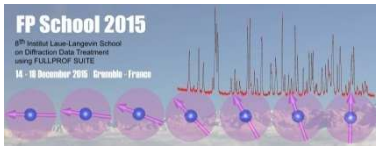
  Or use the DOS command :

```
d:\data_3D>dir *.dat >> bufferfilename.buf /b
```

- Open a buffer file
- Select the common data file format

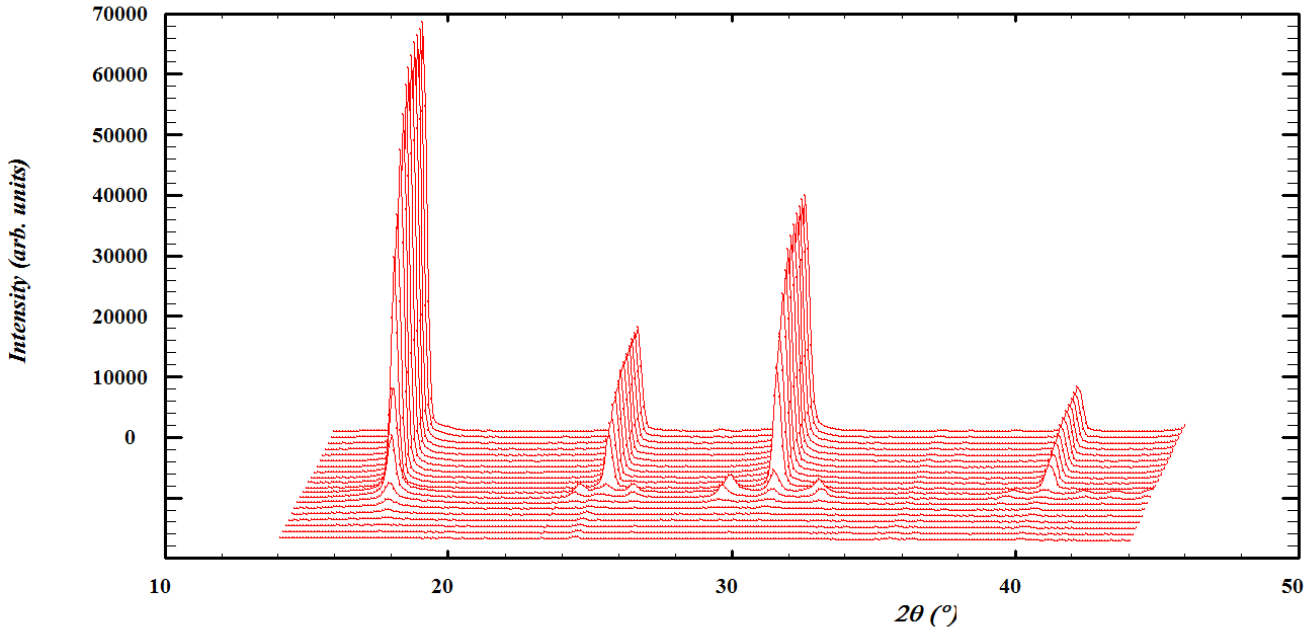
WinPLOTR

Visualisation of (pseudo) *3D* plots



2. Graphical options in WinPLOTR :

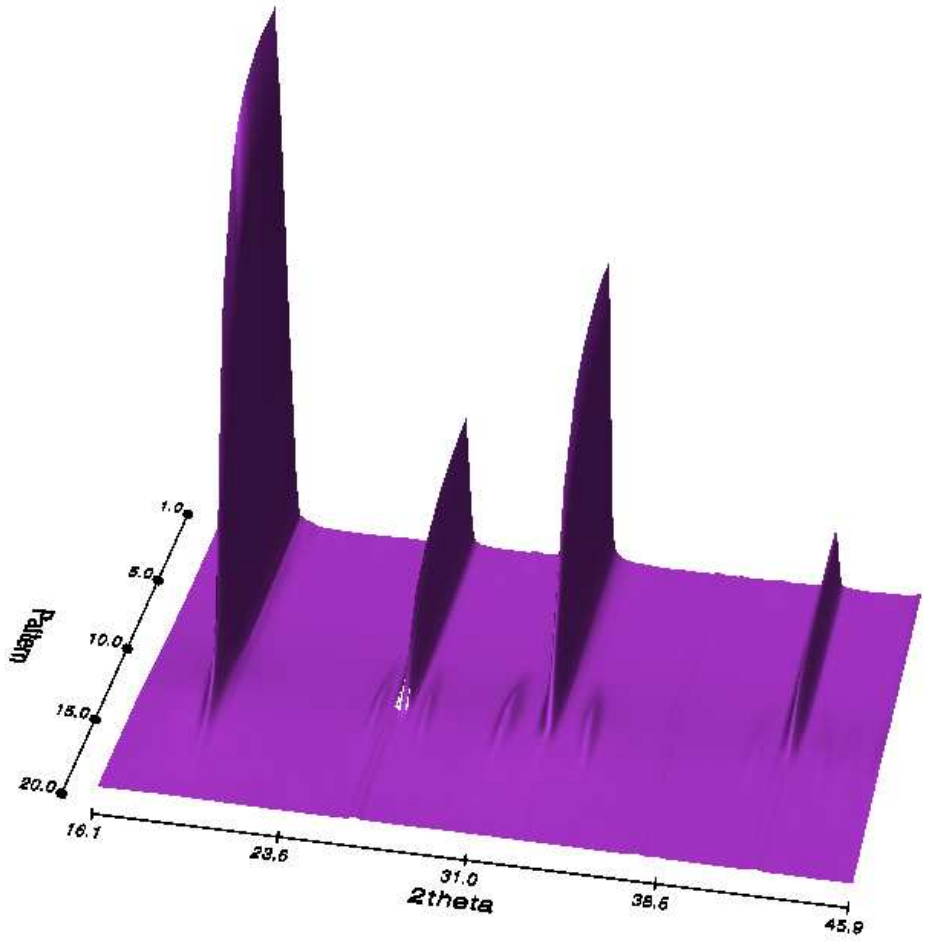
- X and Y shifts
- Change colors, markers, style
- Hidden part



WinPLOTR

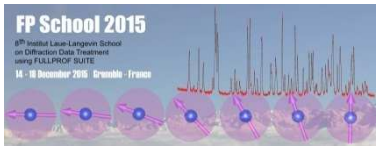
Or : *3D* plots with WinPLOTR2006

2. OpenGL 3D model



WinPLOTR-2006.

Run FullProf in sequential mode : plot of refined parameters



- Experimental requirements:

. Diffraction experiment versus external parameter (T, P, time, …)
. Common format for all data files
. Specific naming for data files

- FullProf specificities:

. Only 1 starting PCR file (refine first temperature separately)
. Profile matching, Rietveld, multiphases, microstructure …
. Distances calculations …

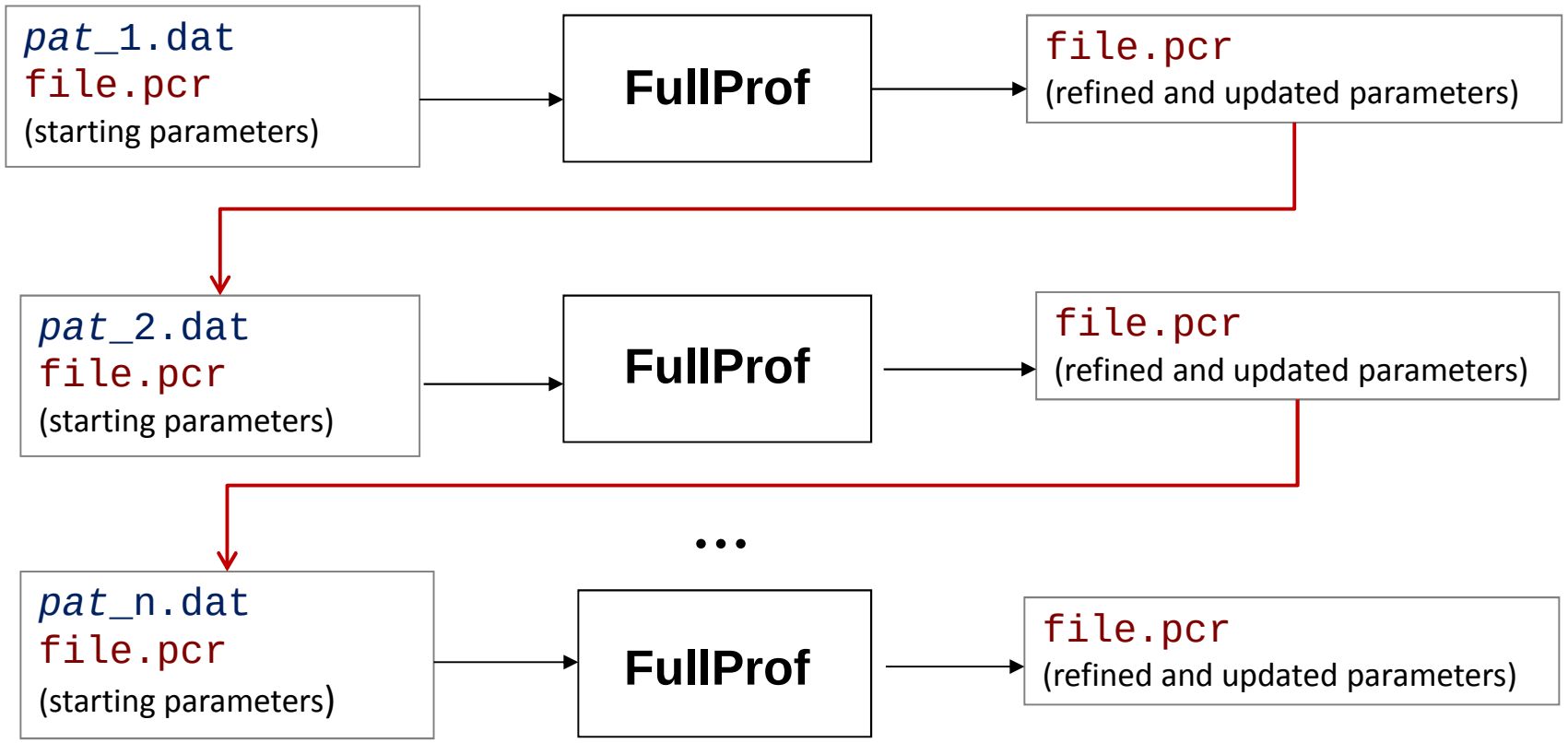
All the patterns will be analysed in the same refinement conditions (background, physical model, number of phases, number of refined parameters…).

The evolution of the refined parameters has to be continuous ! Split temperature range if necessary.

FullProf in sequential mode

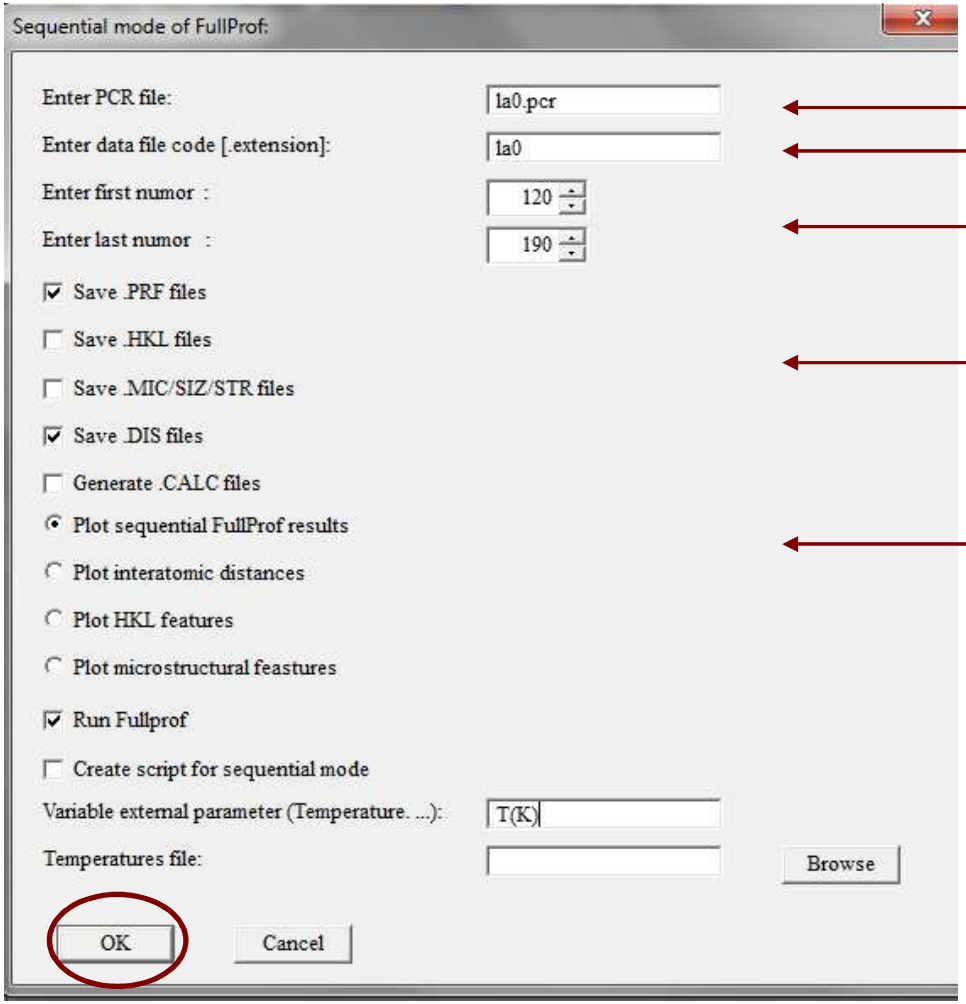
N diffraction data: *pat*_1.dat, *pat*_2.dat … *pat*_n.dat

Only 1 pcr input file: file.pcr (starting parameters) **PCR=1**



All refined parameters during sequential run are stored in a **.SEQ** file

Prepare sequential FullProf launching setup



PCR file

File name code

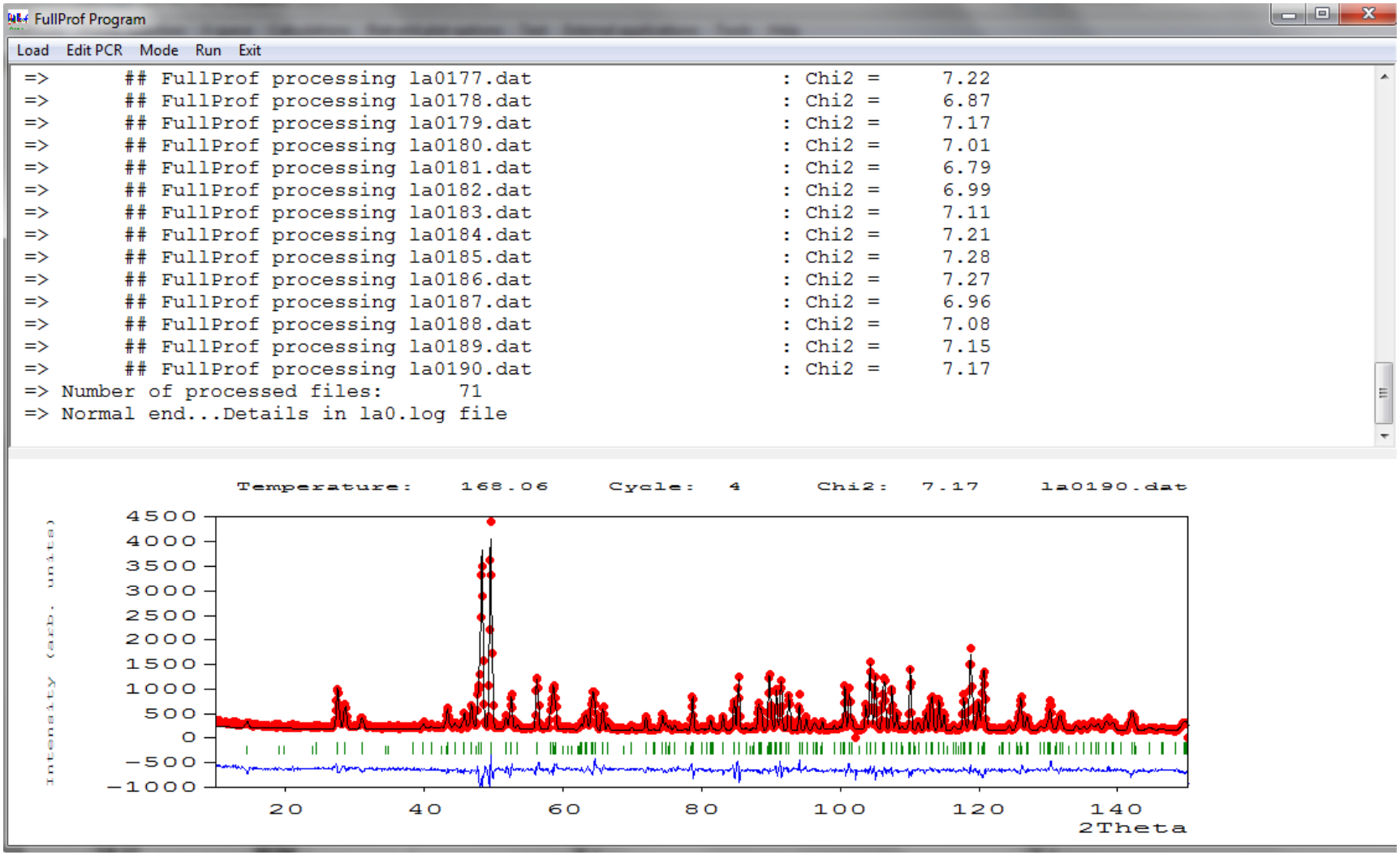
First and last numor

Output files

Results to plot

WinPLOTR

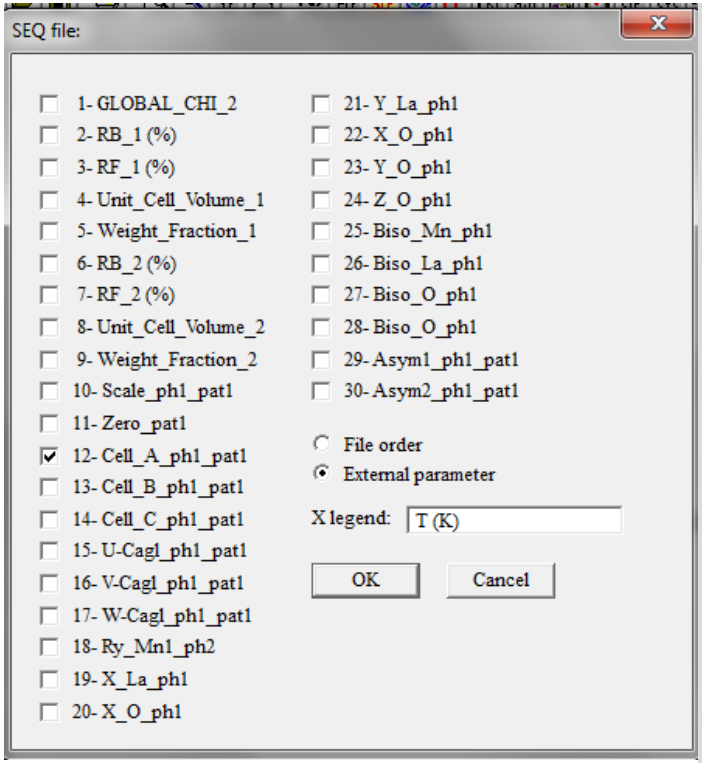
Running sequential FullProf

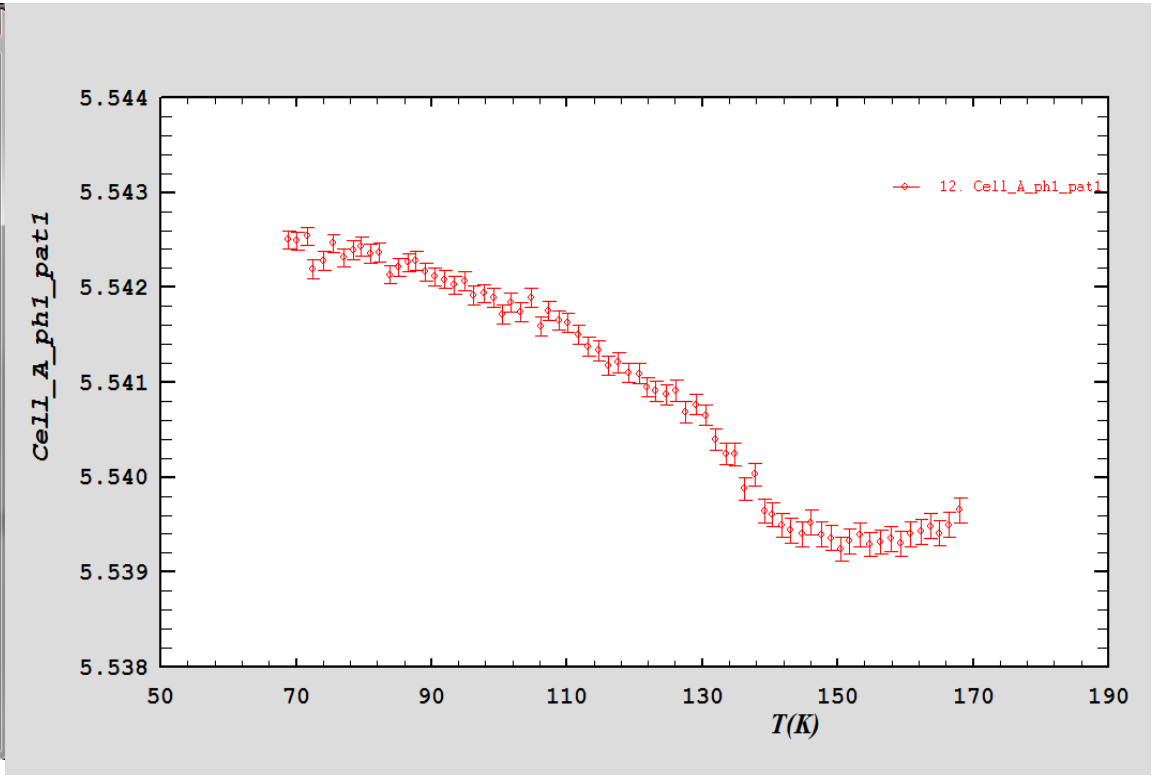


71 patterns (1535 points) / 21 refined parameters : 26 sec. of cpu time on my labtop !

WinPLOTR

Plotting sequential FP results

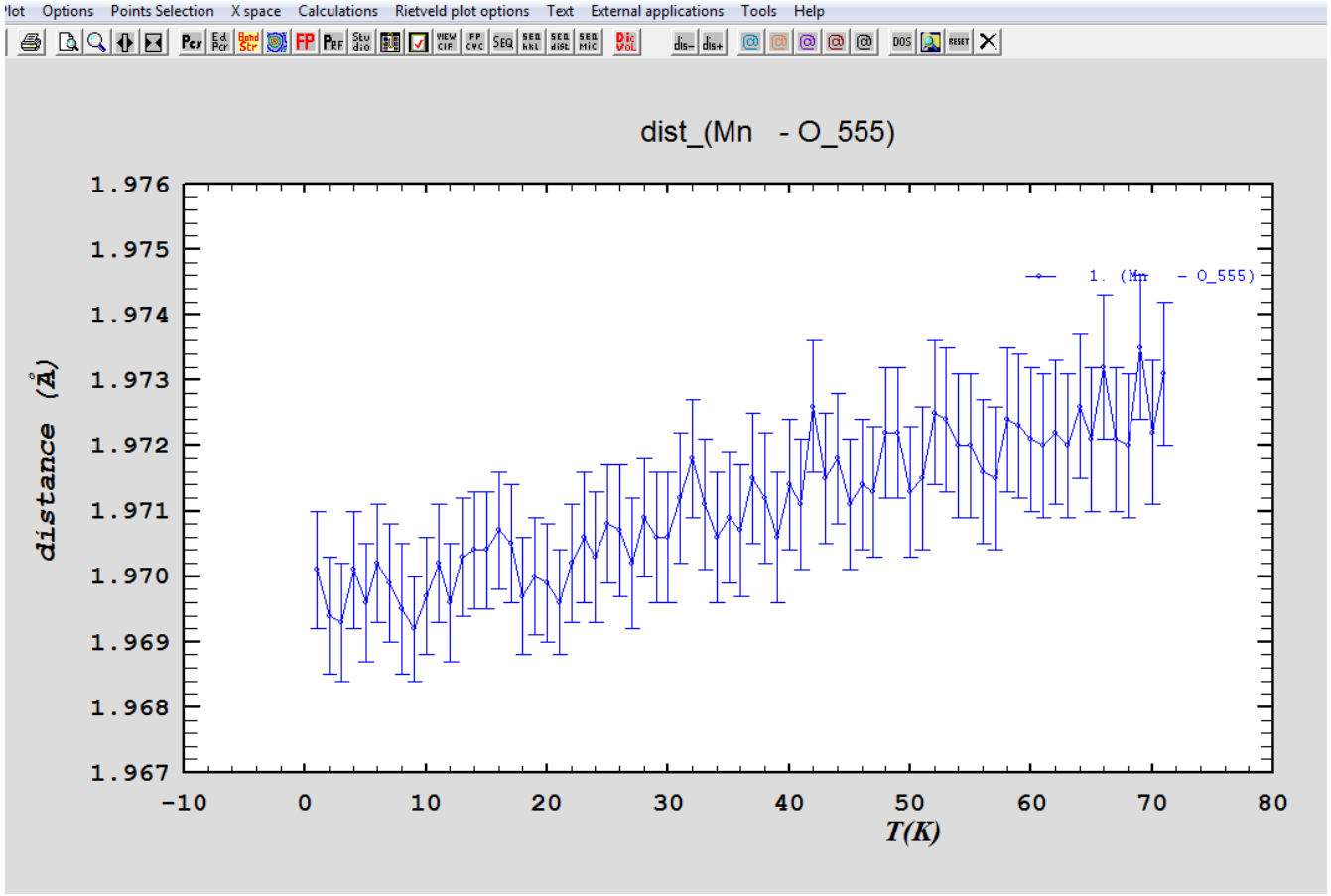




WinPLOTR @ FPSchool, ILL, 14-18 dec. 2015

WinPLOTR

Plotting sequential FP results



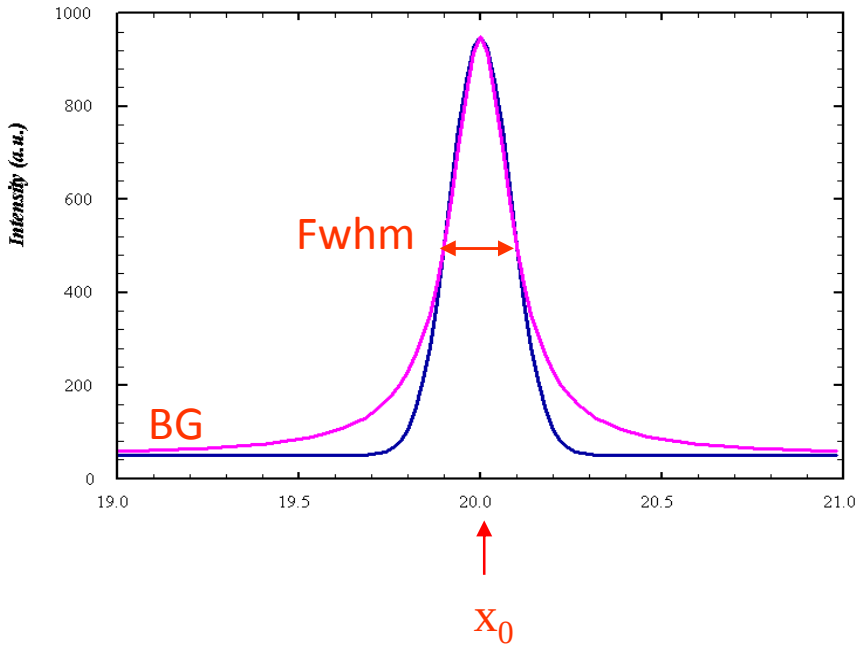
WinPLOTR

Profile fitting procedure in **WinPLOTR**

- Profile function : pseudo-Voigt $PV(x) = \eta.L(x) + (1-\eta).G(x)$

  T.C.H. formulation (idem **NPROF=7**) to mimic Voigt function $(L \otimes G)$

- Least-squares refinement of parameters :
  - linear background values
  - reflections parameters : intensity, position, FWHM, η



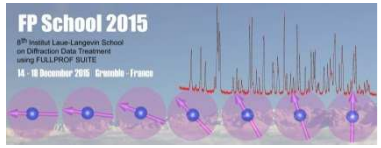
Profile fitting procedure

- Starting parameters of L.S. refinements ?

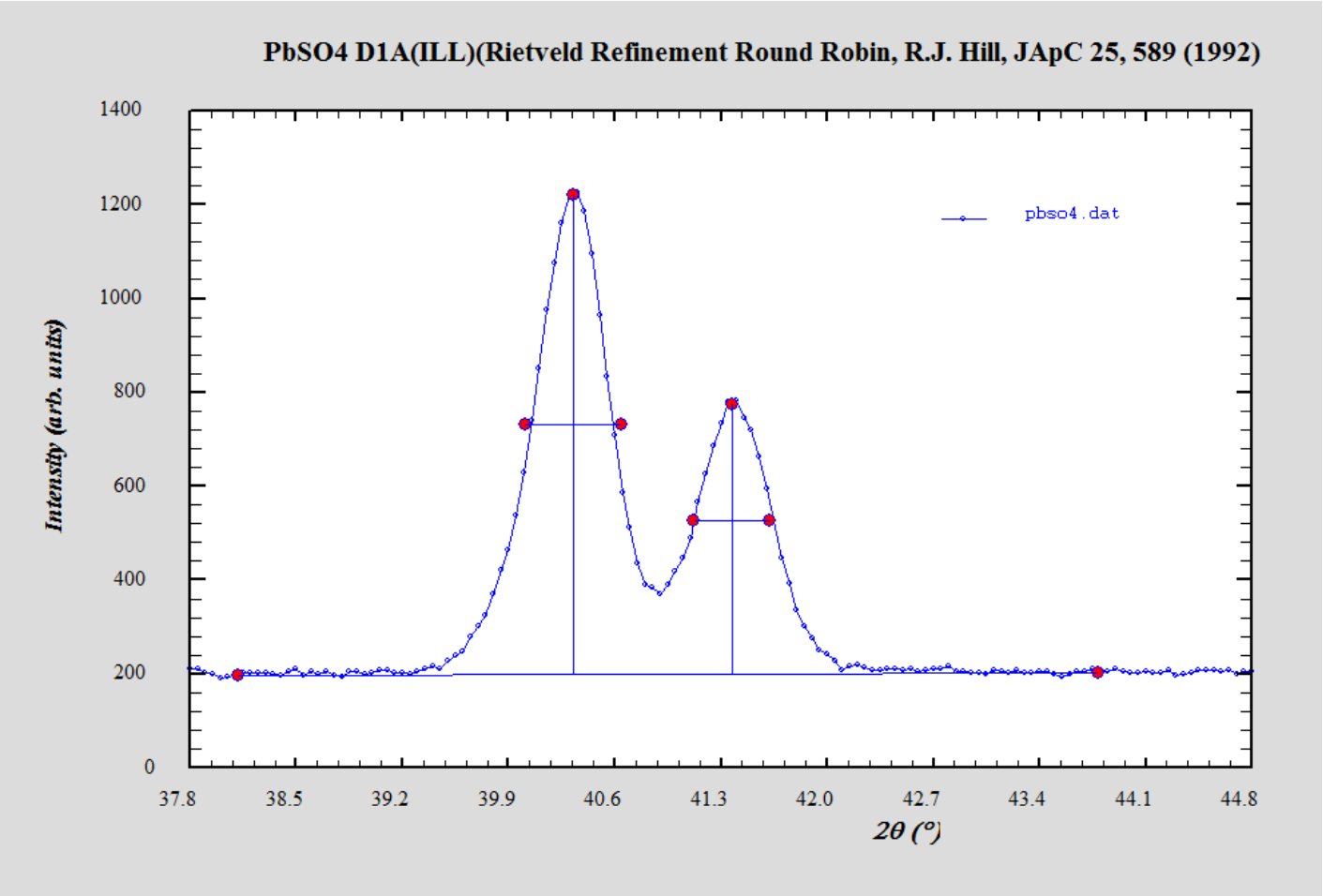
1. Select parameters with the mouse
2. Automatic search
3. From a .**PIK** input file
4. Single peak / doublet

WinPLOTR

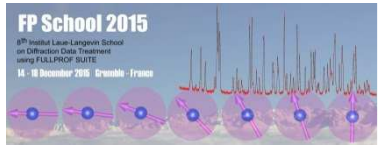
1. Profile fitting procedure : handling mode

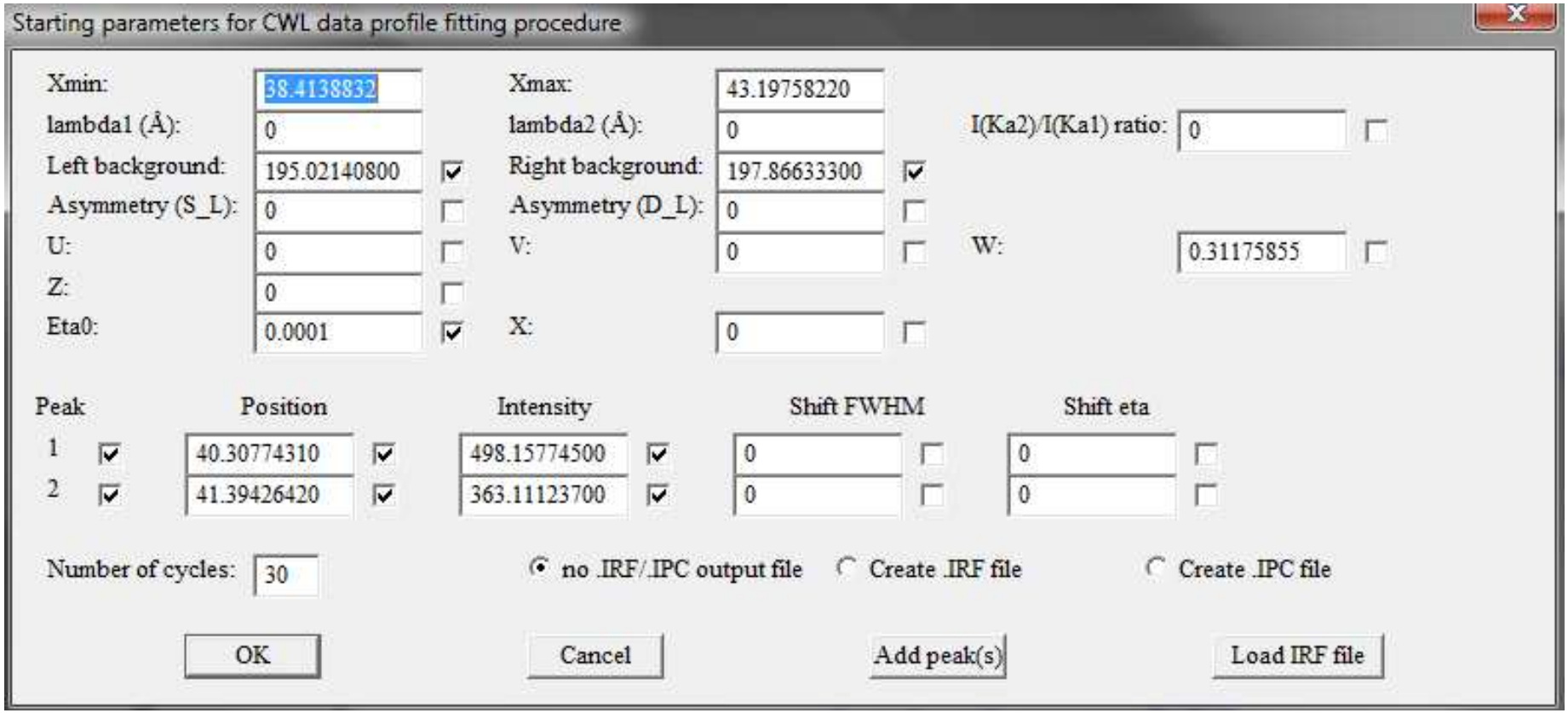


- selection of starting parameters values



1. Profile fitting procedure : handling mode



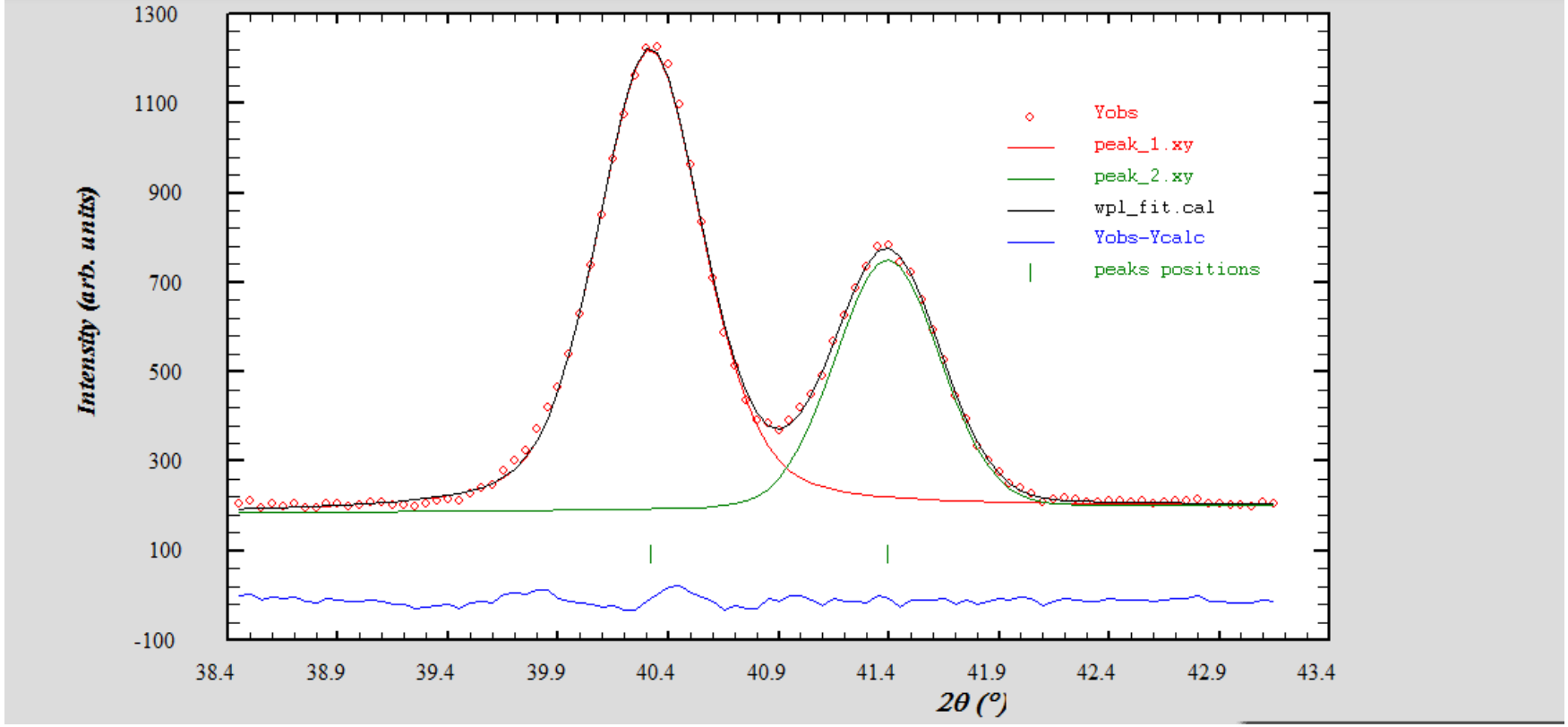


C:\Window

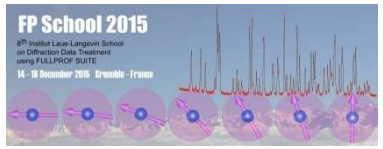
Fichier Edition Affichage Image Couleurs Masque Sélection Capture Fenêtre Aide

Constant wavelength data profile refinement:

```
=> Parameter  X            :      0.000000      0.000000
  Scatt. Variable       Background            Sigma
          38.4500          184.1887           2.1392
          43.1500          200.8363           2.1189

   Position      Sigma      Intensity       Sigma     Shf_FWHM       Sigma     Shf_Eta       Sigma        FWHM       Sigma          eta
  40.315678   0.001408         737.35        8.67     0.006986    0.004230    0.488205    0.028404    0.565339    0.004230    0.488305     0.0
  41.393456   0.002117         342.73        8.13    -0.000860    0.005647    0.114697    0.062580    0.557493    0.005647    0.114797     0.0
     Rp(%)     Rwp(%)      Rexp(%)        Chi2
       1.8        6.1       4.6516     1.73565

Output files:
   . pbso4_PF.new:  input file with refined parameters
   . pbso4_PF.out:  summary of the fitting procedure
```



Profile fitting procedure

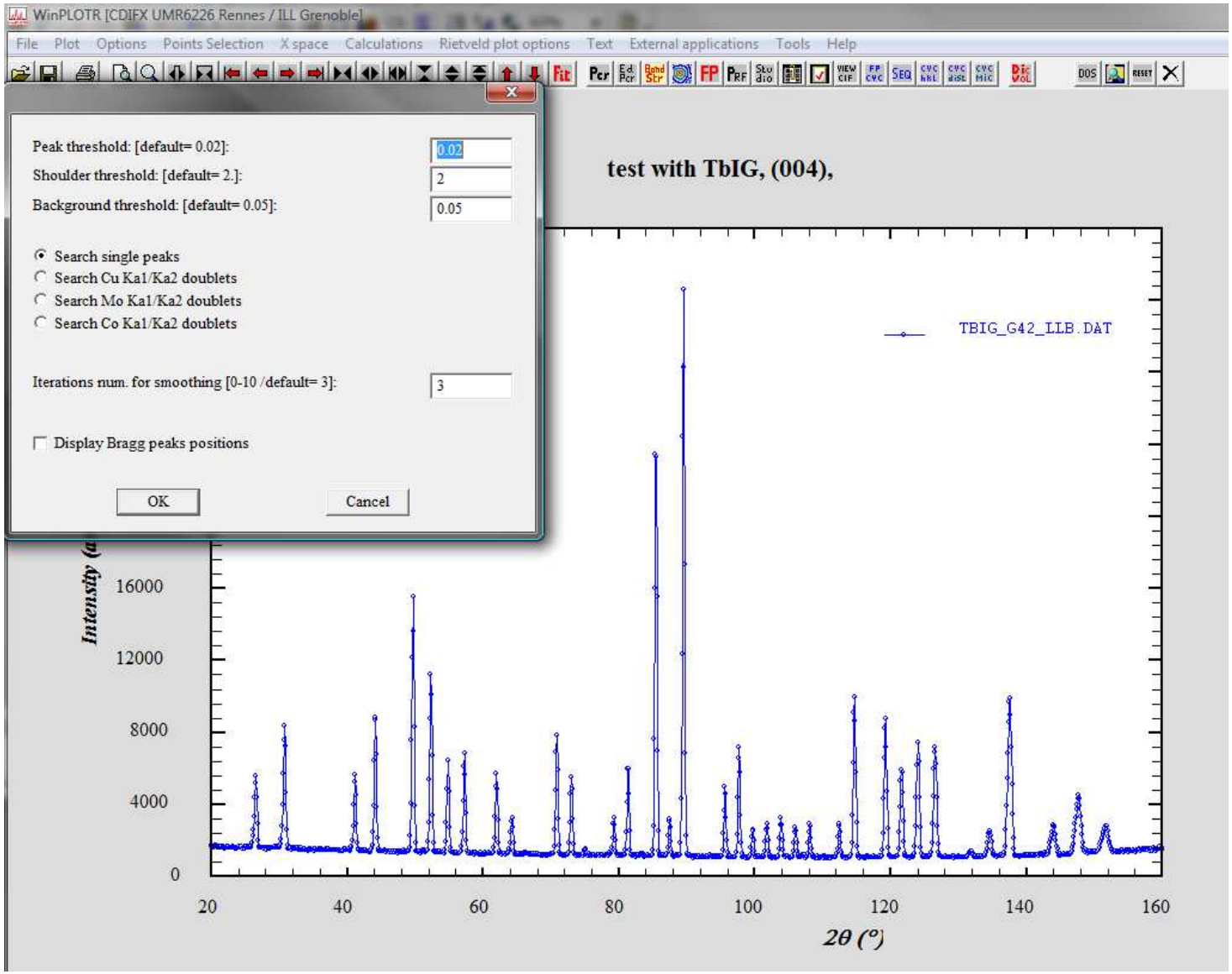


- Starting parameters of L.S. refinements ?

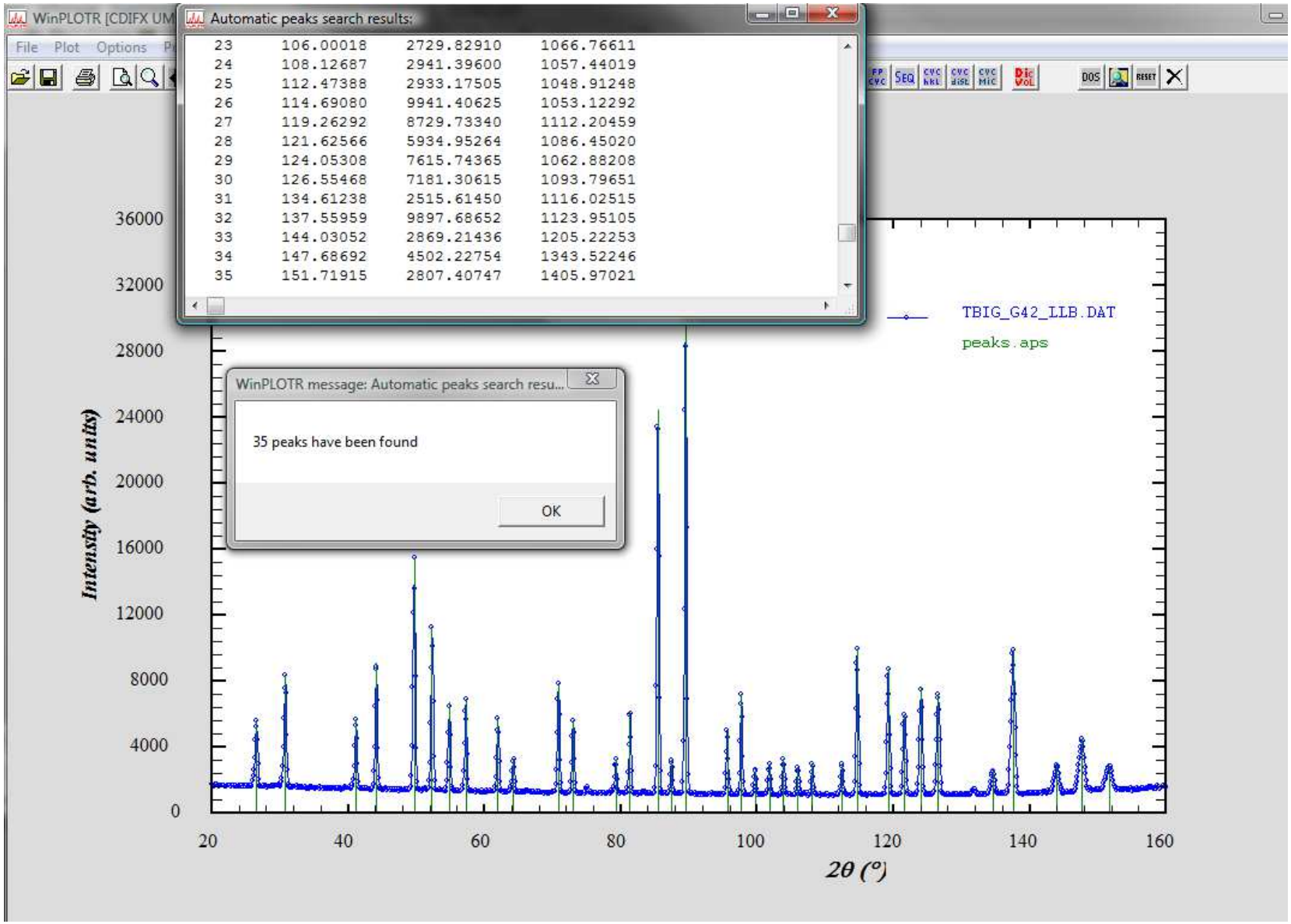
1. Select parameters with the mouse
2. Automatic search
3. From a .**PIK** input file
4. Single peak / doublet

WinPLOTR

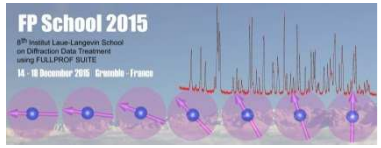
2. Fitting procedure : automatic peak search

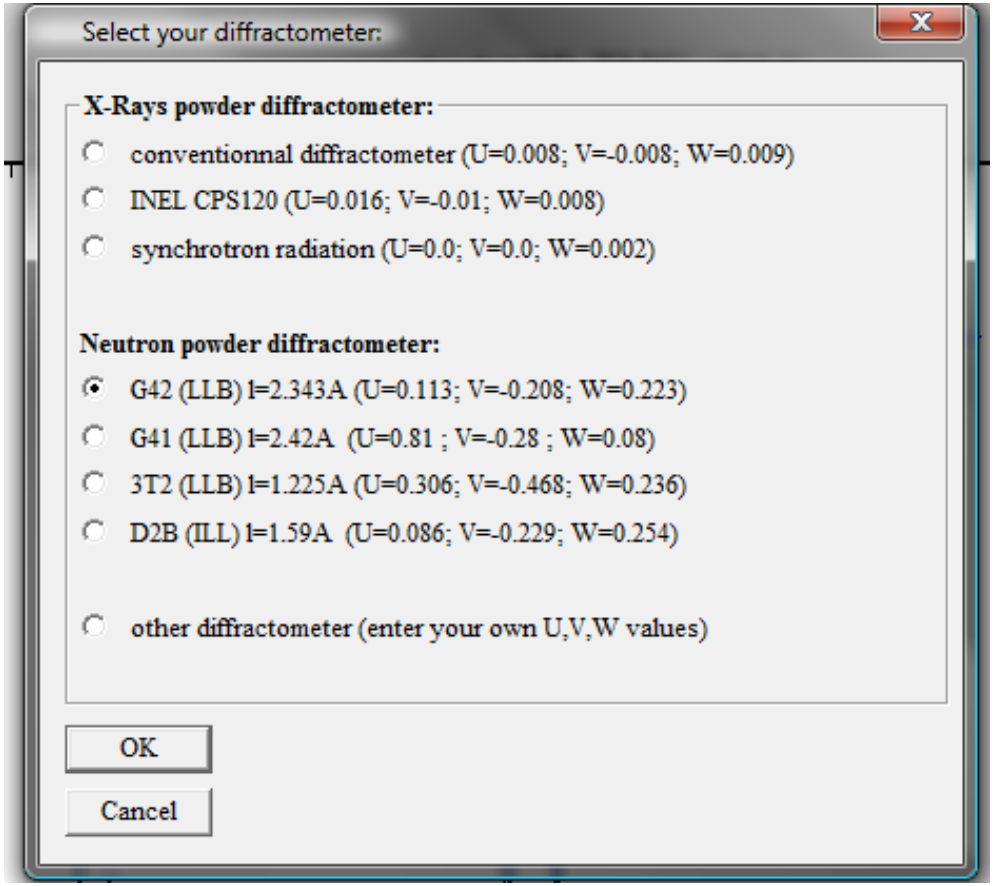


2. Fitting procedure : automatic peak search

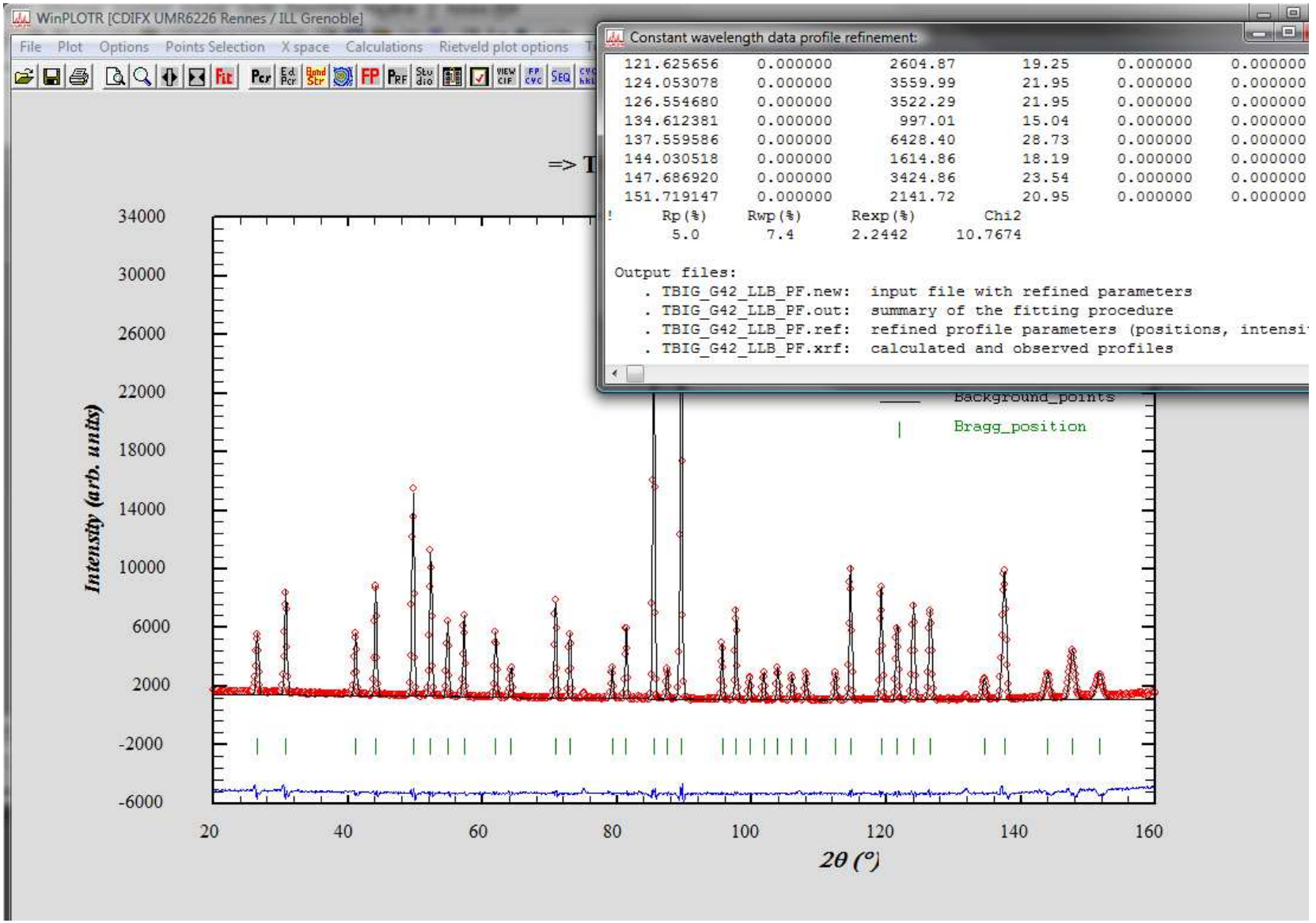


2. Fitting procedure : automatic peak search

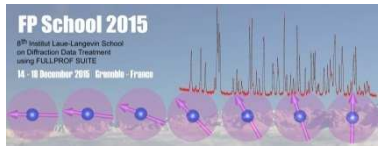




2. Fitting procedure : automatic peak search



Profile fitting procedure

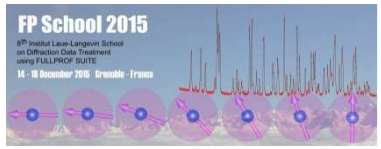


- Starting parameters of L.S. refinements ?

1. Select parameters with the mouse
2. Automatic search
3. From a .**PIK** input file
4. Single peak / doublet

WinPLOTR

3. Fitting procedure : from **.PIK** input file



KEDIT - [D:\WinPLOTR\examples\pbso4_n.new]

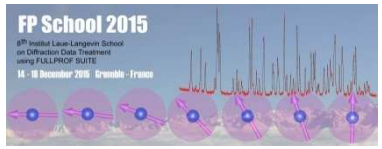
File Edit Actions Options Window Help

continue

```
====>
* * * Top of File * * *
 => TITLE: pbso4_n.dat
! Ang_init    Ang_fin   Nbac Npeak  Ncyc    Inte   Inst   Jobt   Cont Weight Corr Constr
   44.0000    62.0000      2    11    10       0      6      2      0      0    0.      0
    1.540560    1.544330  <=  Lambda1 & Lambda2
!  Global Profile Parameters:
         0.0000      0      <= Kalph2/Kalph1 ratio  &  Flag
         0.0000      0      <= Asymmetry parameter1 &     "
         0.0000      0      <= Asymmetry parameter2 &     "
         0.0860      1      <= Parameter  U         &     "
        -0.2290      1      <= Parameter  V         &     "
         0.2540      1      <= Parameter  W         &     "
         0.0000      0      <= Parameter  Z         &     "
         0.1000      0      <= Parameter Eta0       &     "
         0.0000      0      <= Parameter  X         &     "
!  Background Parameters:
!    2Theta/TOF/Energy   Background    Flag
       44.0000       200.0000      1
       62.0000       215.3333      1
!  Reflection Parameters:
!  2Theta/TOF/Energy     Intensity     Shift-FWHM     Shift-Eta      Flags
       46.5785       450.5725        0.0000         0.0000   1   1   1   0
       47.7482       129.3865        0.0000         0.0000   1   1   1   0
       49.5211       342.3236        0.0000         0.0000   1   1   1   0
       50.4567       248.8786        0.0000         0.0000   1   1   1   0
       51.5505       548.4024        0.0000         0.0000   1   1   1   0
       52.2982       848.1331        0.0000         0.0000   1   1   1   0
       53.1669       320.1445        0.0000         0.0000   1   1   1   0
       54.9535      1134.3667        0.0000         0.0000   1   1   1   0
       56.0510       929.1931        0.0000         0.0000   1   1   1   0
       57.8286       370.5429        0.0000         0.0000   1   1   1   0
       60.1728        73.4670        0.0000         0.0000   1   1   1   0
!  Chi2 =    149.170
* * * End of File * * *
```

Line=18 Col=35 Alt=6,6;7 Size=32 Files=1 Windows=1 OVR R/W 12:17

Profile fitting procedure

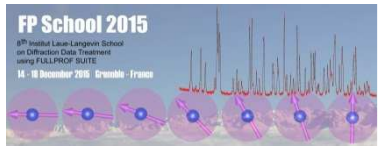


- Starting parameters of L.S. refinements ?

1. Select parameters with the mouse
2. Automatic search
3. From a .**PIK** input file
4. Single peak / doublet

WinPLOTR

4. Profile fitting procedure : single peak



. Starting parameters are detected automatically :

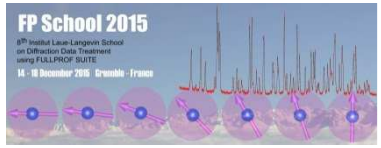
. background points

. single peak / doublet parameters : position, FWHM

Example: NdSrNi0.8Cu0.2O4 / RX-D500

WinPLOTR

Background points selection



. Automatic mode (**Points selection / Automatic background**):

. Created from the pattern file

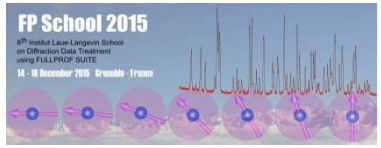
. Number of points ~ SQRT(n), with a quasi uniform repartition on 2θ

. Modify background (**Points selection / Add /remove background points**)

. Save background points to paste into **.PCR** file for **FullProf**

>> Raw background for raw refinements (not recommanded for advanced analysis)

Background points selection



. Manual mode (**Points selection / Select background points**):

. Created from the data file or preferentially from the **.PRF** file

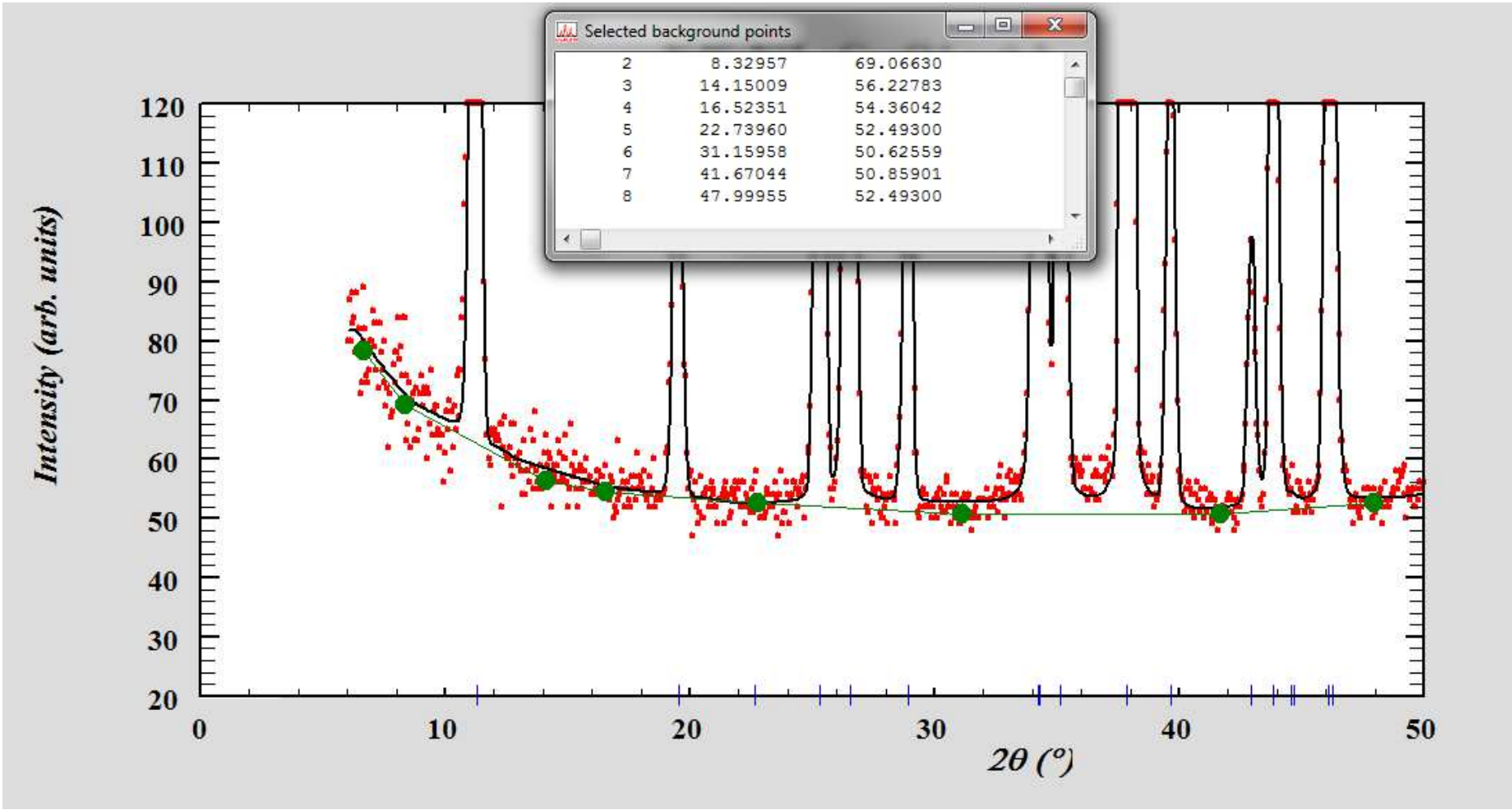
. Number of points = depends on peaks overlap, background modulation, …

. Modify backgrounds (**Points selection / Add / background points**)

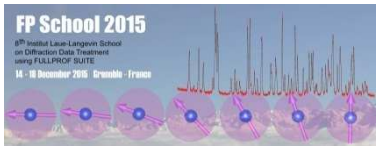
. Save background points to include into **.PCR** file for **FullProf**

>> Good starting points for **FullProf** refinements (Rietveld / profile matching (strongly recommended !)

Background points selection



WinPLOTR tools (**Tools** menu option)



- **Space group informations :**
  - . features
  - . symmetry operators
  - . Wyckoff positions
  - . extinctions

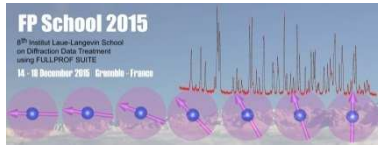
- **(hkl) list generation in a given 2θ range, from :**
  - . space group
  - . unit cell parameters
  - . wavelength

- **unit cell volume calculation**

- **molecular weight calculation**

WinPLOTR

Advanced use of WinPLOTR



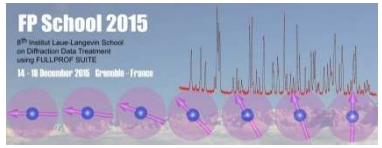
for repeatable actions :

. Files convertion
. Multi-scans **UXD** or **XRDML** file normalization
. Change *X* space
. Automatic single peaks/doublet profile fitting
. Save graphics as bitmap or PostScript files

Use a command file, containing keywords and arguments:

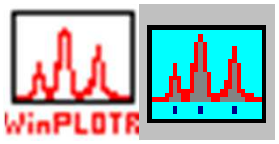
```
. FILE file%name file%format
. SAVE_AS_XY, SAVE_AS_INSTRM_0
. UXD_NORMA/XRDML_NORMA
. SHIFT_X/SHIFT_Y shift_x/shift_y
. OFFSET_X/OFFSET_Y offset_x/offset_y
. MULTIPLY_X/MULTIPLY_Y mult_x/mult_y
```

Advanced use of WinPLOTR

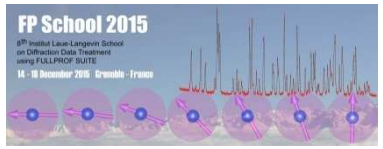


Use a command file, containing keywords and arguments:

- **SUBSTRACT** file_1 file_2 format
- **WAVE**
- **TRANSF_X1_TO_X2** (2THETA / Q / STL / D / S)
- **FIT_SINGLE_PEAK** xmin xmax
- **FIT_DOUBLET_CU/MO/CO** xmin xmax
- **BITMAP / EPS**



Launching **WinPLOTR** from the command line, using a command input file

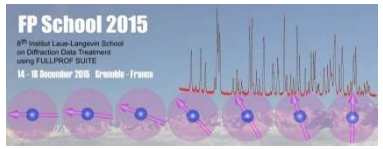



```
d:\data>winplotr command_file.cmd
```

Example 1 : conversion of files

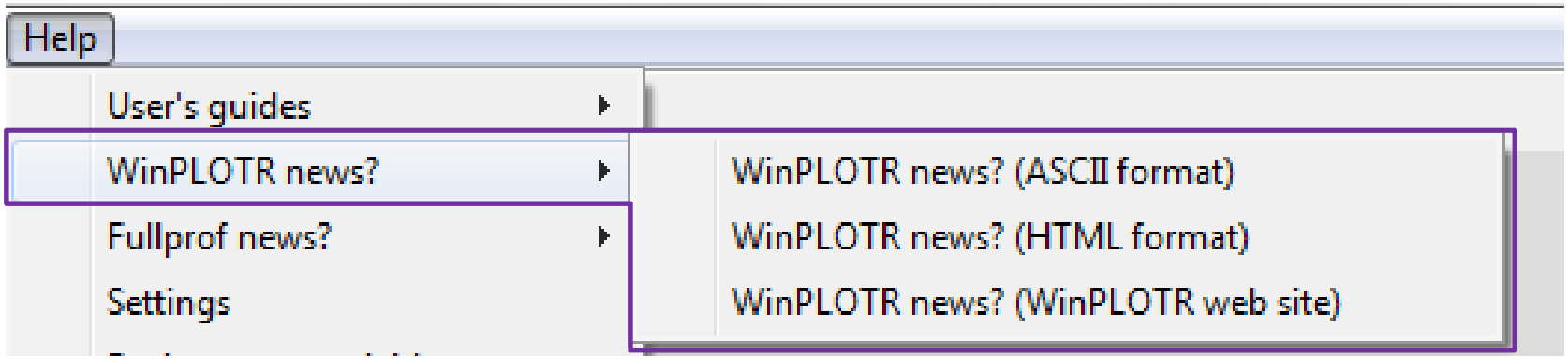
```
FILE data_file_001.uxd 9
SAVE_AS_XY
FILE data_file_002.uxd 9
SAVE_AS_XY
…
FILE data_file_455.uxd 9
SAVE_AS_XY
```

Launching **WinPLOTR** from the command line, using a command input file



Example 2 : profile fitting

```
FILE ndsrni_rx.dat 1
FIT_SINGLE_PEAK 24. 25.
FILE ndsrni_rx_pf.xrf 100
BITMAP
PLOT
```

WinPLOTR

WinPLOTR : what's new ?