*Win*PLOTR: a graphic tool for powder diffraction pattern analysis

T. Roisnel[1,2] and J. Rodriguez-Carvajal[1]

1- Laboratoire Léon Brillouin (CEA-CNRS), C.E. Saclay
91191 Gif sur Yvette Cedex (France)

2- Laboratoire de Chimie du Solide et Inorganique Moléculaire
(UMR CNRS 6511), Institut de Chimie de Rennes,
Université de Rennes 1
35042 Rennes Cedex (France)

plotr@llb.saclay.cea.fr

http://www-llb.cea.fr/fullweb/winplotr/winplotr.htm

*Win*PLOTR

☞ *computer system:*

. Windows 9x (x=5,8),2k ,NT

. Graphic Colour Screen (1024 x 768)

☞ *language:*

Fortran 95 (www.lahey.com)

RealWin (www.indowsway.com) *as an interface to the Windows API.*

==> windows, menus, dialog boxes, mouse ...

*Win*PLOTR

☞ *installation:*

- *winplotr.zip* (http://www-llb.cea.fr/fullweb/winplotr/winplotr.htm)

*Win*PLOTR		T.R. – J.R.C.
MENDEL	Neutrons periodic table	T.R.
DICVOL	Unit cell determination	D. Louër (Rennes)
TREOR	Unit cell determination	P.E. Werner (Stockholm)
SUPERCELL	Supercell determination / incommensurate propagation vector components	J.R.C.
FullPROF	'Single and multi-patterns' Windows version Examples User's guide	J.R.C.

- install.exe: . automatic installation of WinPLOTR, FullProf and the accompanying programs

- WinPLOTR and FullProf environment variables

*Win*PLOTR

☞ *Plotting powder diffraction patterns* :

- raw data, normalised or refined data:
  . neutrons, X-Rays (conventional or synchrotron)
  . constant wavelength, energy dispersive , time of flight
- a large number of data file formats
- scattering space: - 2 θ(°)/TOF (msec.)/Energy (KeV)
  - Q (Å^{-1}) , 1/d (Å^{-1}), sin θ/λ (Å^{-1})
  - d (Å)
- automatic search procedures: background, reflections
- background substraction
- fit procedure (interactive / automatic)
- graphical options: zoom, shift/offset, pseudo-3D, error bars
- PostScript files

*Win*PLOTR

☞ *Graphical User Interface for powder diffraction analysis programs*:

- FullProf (JRC): profile refinement (Rietveld, whole pattern fitting)
  - PCR file editing
  - FullProf running
  - PRF file plotting
- automatic indexing of powder diffraction patterns: DICVOL (D.Louër), TREOR (P.E. Werner):
  . Peak search
  . Input file creating (help of a dialog box)
  . WinDICVOL/WinTREOR90 launching
  . Automatic creation of a PCR file for FullProf (« profile matching » mode)
- SuperCELL (JRC): determination of a super unit cell or the components of an incommensurate propagation vector
- user's defined program (*winplotr.set*)

*Win*PLOTR: ' winplotr.set ' settings file

☞ *adapting WinPLOTR capabilities to the user needs:*

- arrays dimensions:
  - maximum of data files to plot simultaneously
  - maximum of points in the data files
- associated software:
  - FullProf, DICVOL, TREOR, editor
  - user's defined software (DOS / Windows)
- graphical options:
  - colours, markers, grid
  - main window size and position in the screen
  - ...
- miscellaneous:
  - data file extensions
  - resolution parameters (U,V,W)
  - ...

*Win*PLOTR: ' winplotr.set ' settings file

*Win*PLOTR

- ☞ 'pseudo-3D ' mode plot
- ☞ automatic search procedures:
  - background points
  - reflections
- ☞ reflections fitting
  - handling mode
  - automatic mode
  - through a parameters file
- ☞ *GUI* for external applications:
  - FullProf (JRC): Rietveld refinement
  - DICVOL91, TREOR90: indexing powder patterns
  - user's defined software: ex. = INEL.bat
  - the cimetidine example

*Win*PLOTR

☞ *Plotting in 'pseudo-3D' mode*

*Win*PLOTR: plotting in 'peudo-3D' mode

1- *data*
- create a buffer file, containing the names of the data files to plot
- open the buffer file
- select the common data file format

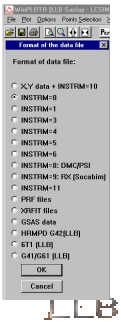
2- *graphical options*
- X and Y shifts
- hidden part
- change colours / markers

Ex: neutron powder diffraction study of the antiferromagnetic behaviour of Tb_2Pd_2Sn at low temperature

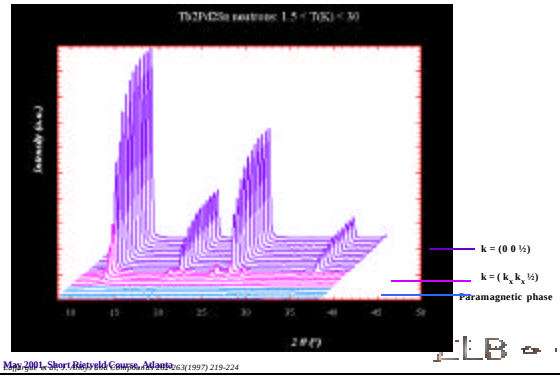
1- Edit a buffer file

2- Open the buffer file

3- Select the format



Typical diffraction patterns of magnetic ordering set-up (G4.1)



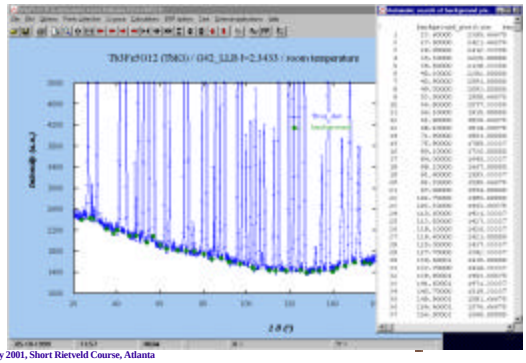
WinPLOTR

☞ *Automatic search procedures*

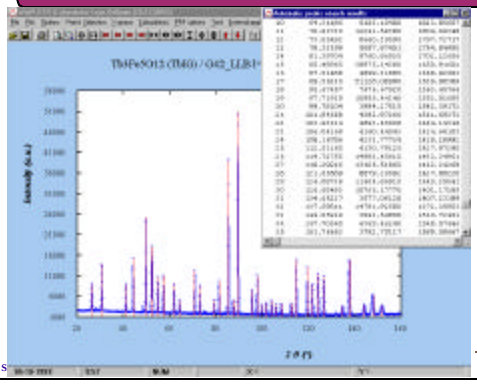
. Background points

. Reflections positions

WinPLOTR: determination of background points



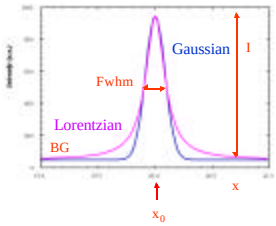
WinPLOTR: determination of reflection positions



WinPLOTR: fitting reflections

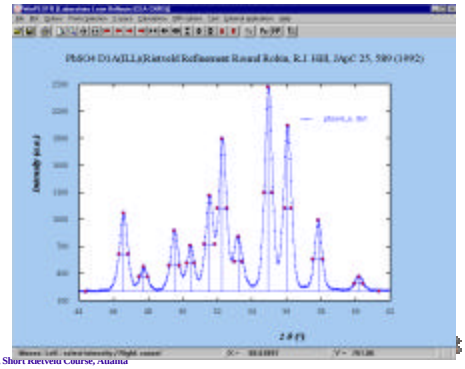
. Linear background

. Pseudo-Voigt function (PV = ηL + (1- η).G)



Profile fitting procedure: 1-handling mode

☞ select reflection parameters

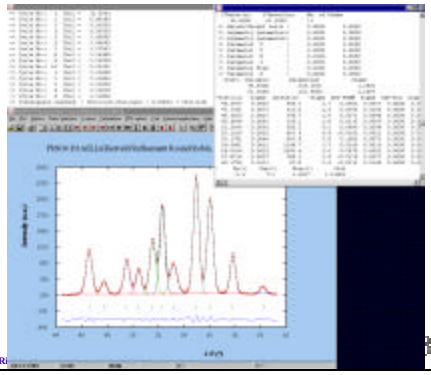


Profile fitting procedure: 1-handling mode

☞ enter fitting parameters

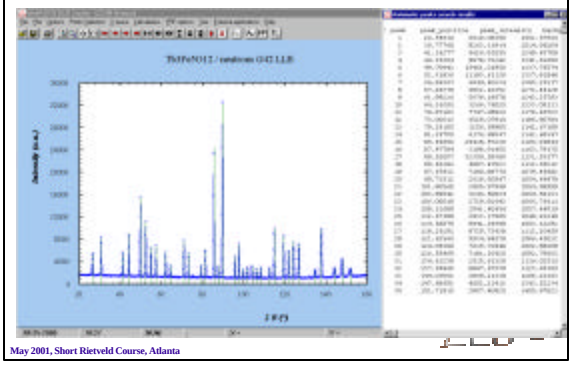
wpllp.ico

Profile fitting procedure:
☛ run and display results



wpllp.ico

Profile fitting procedure: 2-automatic mode
☛ automatic peak search

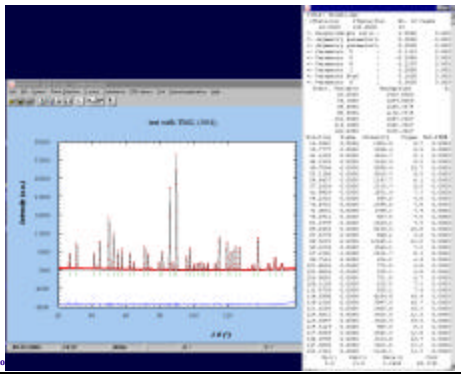


wpllp.ico

Profile fitting procedure: 2- automatic mode
☛ enter resolution parameters

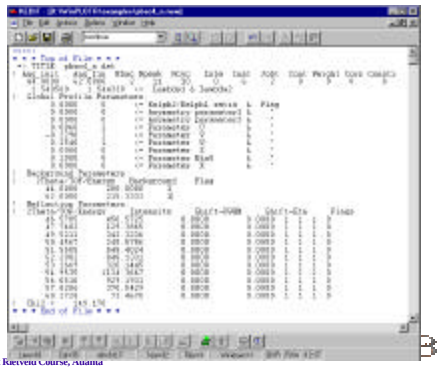
wpllp.ico

Profile fitting procedure:
☛ run and display results



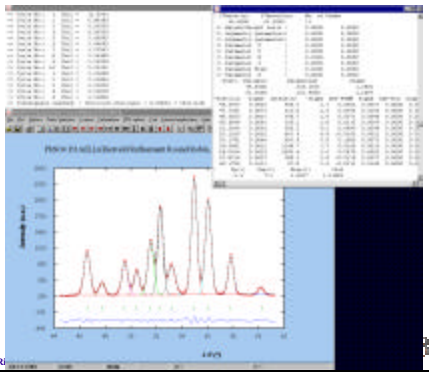
wpllp.ico

Profile fitting procedure:
3- edit a parameter file (.pik)



wpllp.ico

Profile fitting procedure:
☛ run and display results



*Win*PLOTR : using a user's defined program

Example: calibration of INEL data files

Winplotr.set:

```
! RUN PROGRAMS:
INEL = inel  ! INEL data files conversion       ! Dos version (2)
```

With:

```
inel.bat:    inat %1                  ! .DAT (channel) => . RAW (2theta)
             calib %1/E/G %1 %2       ! .RAW (2theta) => .RAW
(2theta_ calib)
             xch                      ! .RAW (binary) => .UXD (ASCII)
```

- **Select raw data file (.dat) and calibration data file (.cal)**
- **Automatic plot of the calibrated data file (.UXD)**

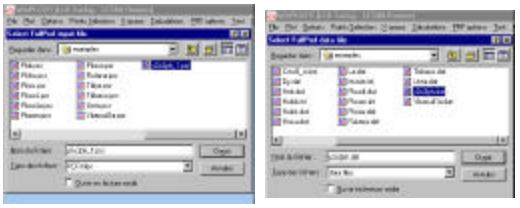
*Win*PLOTR

☞*GUI for FullProf*

- ***Edit PCR file (input parameters)***
- ***Select PCR and DAT files to launch FullProf***
- ***Plotting the PRF file (Y_{obs}, Y_{calc}, Y_{obs}-Y_{calc}, Bragg positions)***

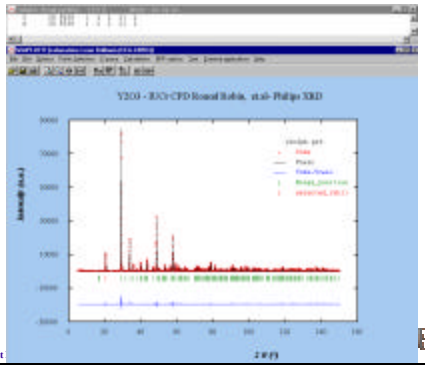
GUI for FullProf: edit the PCR file

GUI for FullProf: selecting the PCR and data files



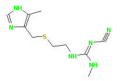
GUI for FullProf : running FullProf

GUI pour FullProf: automatic visualisation of the PRF file



*Win*PLOTR: GUI for various crystallographic software

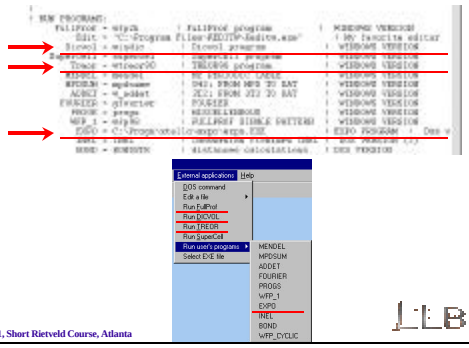
Ex: Cimetidine $S_4C_{40}N_{24}H_{64}$
(*J. Appl. Cryst. (1991), 24, 222-226*)



Synchrotron powder diffraction data (λ=1.52904Å, Daresbury, UK):

1- Unit cell determination ☛ DICVOL (D. Louër), TREOR (P.E. Werner)
2- Integrated intensities ☛ FullProf in pattern matching mode (JRC)
3- Structure determination ☛ EXPO (C. Giacovazzo)
4- Structure refinement ☛ FullProf in Rietveld mode (JRC)

*Win*PLOTR setting file (winplotr.set)

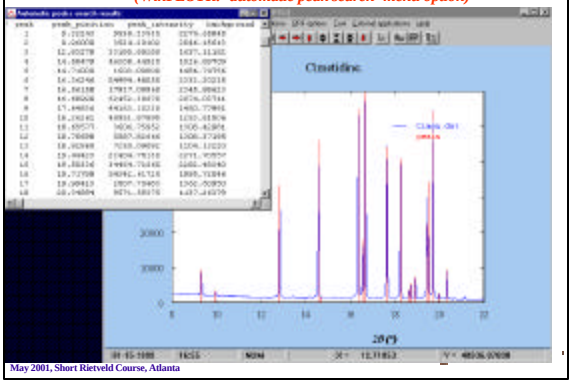


*Win*PLOTR

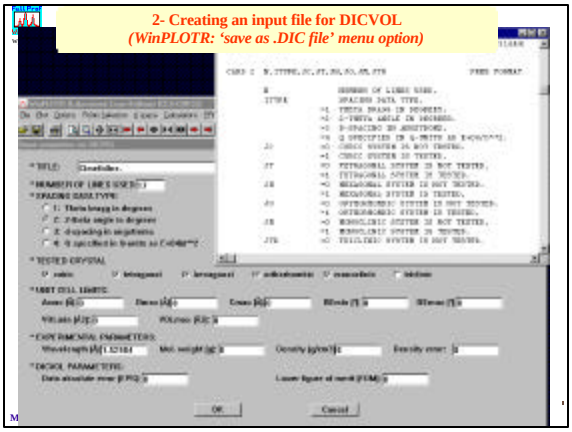
☞*Unit cell determination:*

1- peak positions (at low 2θ angles)
2- automatic indexing

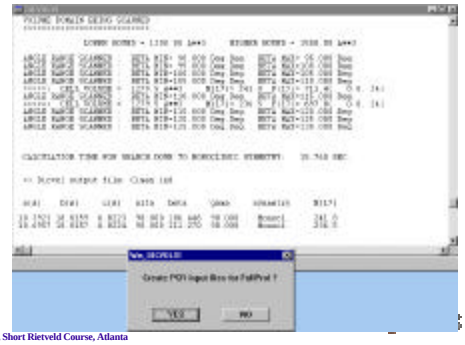
1- reflection positions automatic determination
(WinPLOTR: 'automatic peak search' menu option)



2- Creating an input file for DICVOL
(WinPLOTR: 'save as .DIC file' menu option)



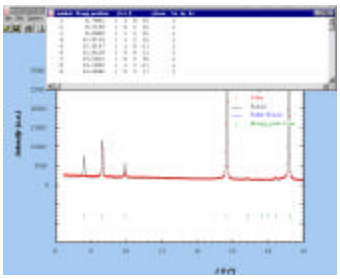
3- Launching WinDIC
(WinPLOTR: 'Run DICVOL' menu option)



4- Creating an input PCR file for FullProf (« profile matching » mode)

. Instrumental parameters: (dialog box)
- . Wavelength
- . Resolution parameters (U, V, W)
- . Peaks profile

. Structural parameters: (through WinDICVOL)
- . Unit cell parameters
- . Space group (point group)



Extinction analysis:

100 non observed : h00 h=2n
001 non observed : 00l l=2n
030 non observed : 0k0 k=2n
20-1 non observed : h0l h+l=2n
101 observed : h0l h+l=2n

☛ *P21/n*

*Win*PLOTR

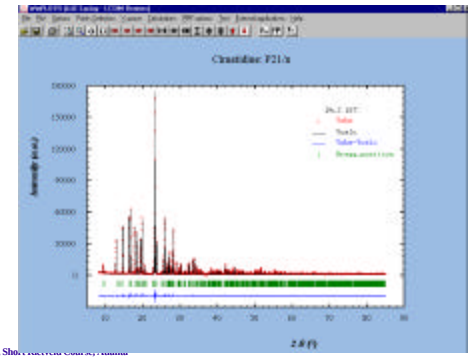
☞ *Integrated intensities extraction:*

FullProf in « pattern matching » mode

*Win*PLOTR: edit the PCR file (input parameters for FullProf)

- Extend the 2θ range to the whole diffraction pattern
- Refine:
  - cell parameters, zero shift
  - peaks profile (shape, FWHM, asymmetry)
  - background
  - JHKL=-2 (create output HKL file for EXPO)

4- FullProf profile refinement («pattern matching » mode) => Extracting integrated intensities



Structure determination: EXPO

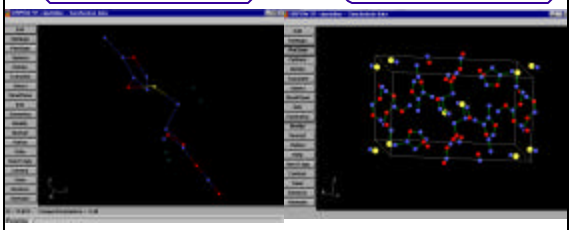
(http://www.ba.cnr.it/IRMEC/SirWare_main.html)

. *EXPO* input file

```
%struct cime
%job cimetidine -- Synchrotron data
%init
%data
cont s 4 c 40 n 24 h 64
wave 1.52904
cell    10.6986  18.8181 6.8246     90.000   111.284   90.000
space p 21/n
ref2   pm_2.hkl
%continue
```

Structure determination: EXPO

Raw results | After re-labelling

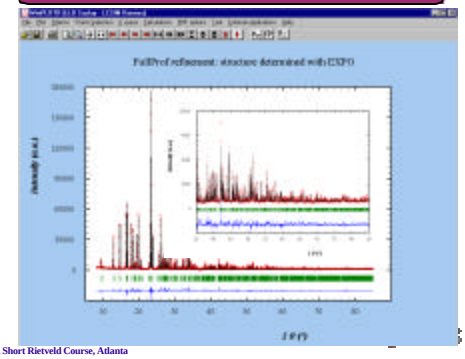


*Win*PLOTR: edit the PCR file (input parameters for FullProf)

Rietveld refinement

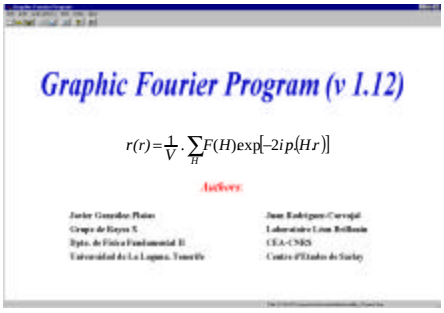
- **Structural parameters:**
  - **. Atomic positions (previously determined by EXPO)**
  - **. Isotropic thermal parameters**
  - **. Cell parameters, zero shift**
- **Profile parameters:**
  - **- peaks profile (shape, FWHM, asymmetry)**
  - **- background**

Rietveld structure refinement with FullProf

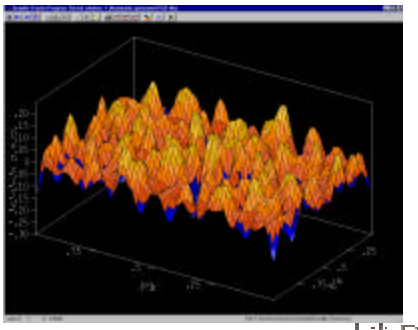


Gfourier: scattering density calculation inside

(JFOU=4: creating an input file for Gfourier)



Locating Hydrogen atoms ?



Hydrogen contribution to an X-ray powder diffraction pattern