DIFFRAC.SUITE

User Manual

TOPAS 5 User Manual

Innovation with Integrity

XRD

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1 INTRODUCTION

1.1 TOPAS overview

TOPAS is a graphics based profile analysis program built around a general non-linear least squares fitting system. TOPAS integrates various types of X-ray and neutron diffraction analyses by supporting all profile fit methods and related applications currently employed in powder diffractometry:

- Single line fitting up to Whole Powder Pattern Fitting
- Indexing
  - LSI method (Coelho, 2003)
  - LP-Search method (Coelho & Kern, 2005)
- Whole Powder Pattern Decomposition
  - Pawley fitting (Pawley, 1981)
  - Le Bail fitting (Le Bail, 1988)
- Ab-initio structure determination
  - Monte-Carlo
  - Simulated annealing (Coelho, 2000)
  - Charge Flipping (Oszlányi & Süto, 2004; as modified by Coelho, 2007)
  - Difference Fourier analysis
- Structure refinement
  - Rietveld method (Rietveld, 1967, 1969)
  - Two-Step method (Will, 1979)
- Quantitative phase analysis
  - Rietveld method (Hill & Howard, 1987)
  - Internal standard method
  - External standard method (O'Connor & Raven, 1988)
  - PONKCS method (Scarlett & Madsen, 2006)
  - Pattern scaling method (Chipera & Bish, 2002)
  - Degree of crystallinity method

1.2 TOPAS features

1.2.1 GUI and Launch Mode

TOPAS supports two modes of operation:
1. A Graphical User Interface mode for parameter input (*GUI Mode*)
2. Direct editing of an input file (*Launch Mode*)

In *GUI Mode* refinements are controlled using a Graphical User Interface for parameter input.

Operation in *Launch Mode* gives access to the full functionality of TOPAS including structure determination (see section 1.2.2). In *Launch Mode* input to the kernel is through an input file (*.INP). Advantageous is the possibility to include user-defined parameters and models into the refinement. Knowledge of the simple but extremely powerful TOPAS macro and equation language is required; for details refer to the Technical Reference manual.

Fig. 1.1 provides a schematic representation of the GUI - Launch - Kernel architecture of TOPAS. The *Scan Window* with its zooming and panning functionality is available in both modes of operation. The same is true for the *Fit Window* with its kernel output. Note the possibility to exchange data between *GUI Mode* and *Launch Mode*. An input file exported from the GUI is fully compatible to *Launch Mode*. This is not true vice versa due to the extended functionality available in *Launch Mode*. It is up to the user to import only input files into the GUI, which contain valid statements. Otherwise TOPAS will throw an exception.

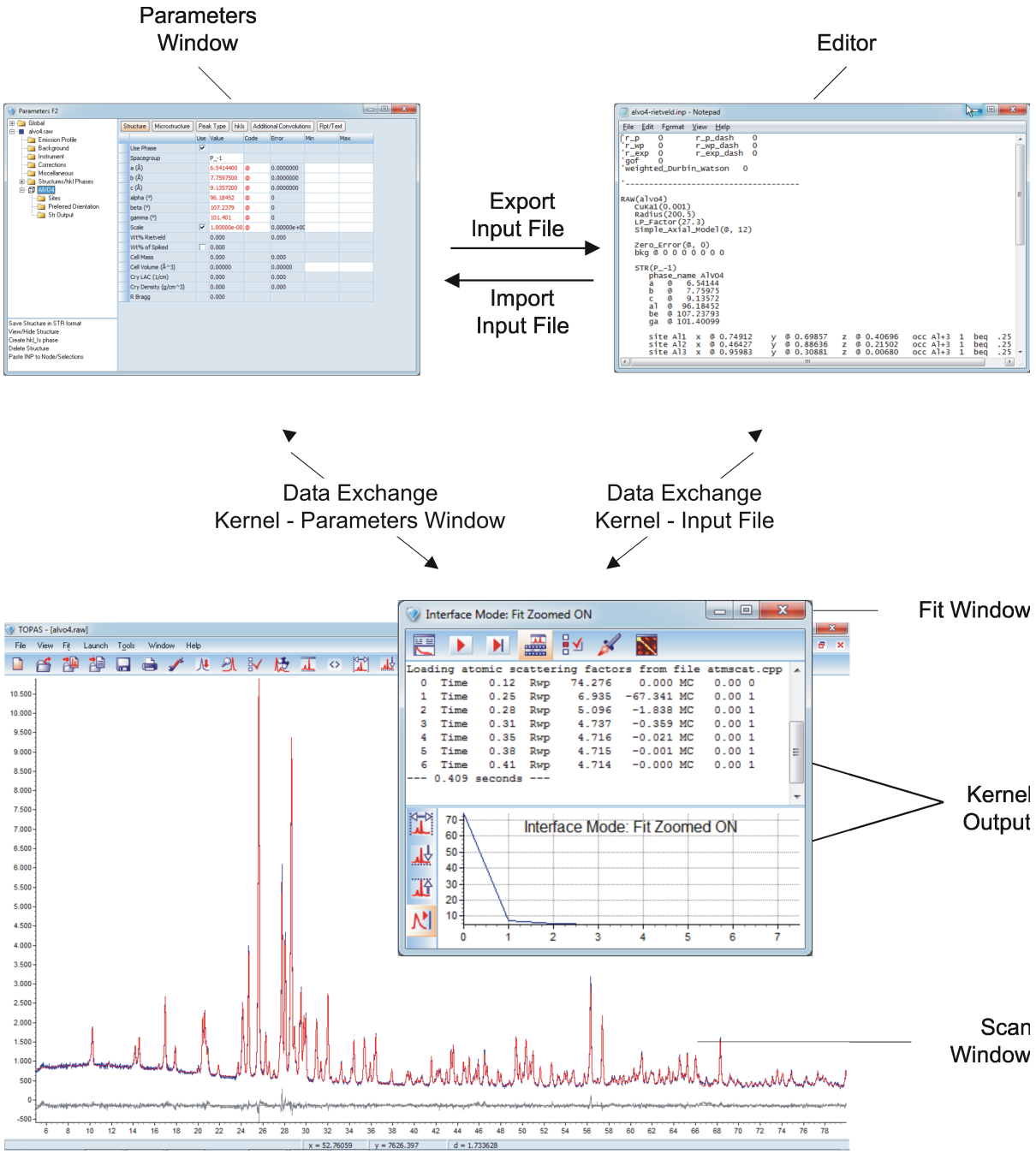


Fig. 1.1: Schematic representation of the GUI - Launch - Kernel architecture of TOPAS.

1.2.2 Features available in GUI and Launch Mode

An overview about the functionality of TOPAS in GUI and Launch Mode is given in Table 1.1. For more details refer to the Technical Reference manual.

Table 1.1: Functionality of TOPAS. Features marked with "GUI" are available in GUI Mode.

Features	
Profile Fitting Methods and Related Applications	
Single line up to Whole Powder Pattern Fitting	GUI
Indexing (LSI-Index, LP-Search)	GUI
Whole Powder Pattern Decomposition (Pawley method, Le Bail method)	GUI
Rietveld structure refinement	GUI
Quantitative Rietveld analysis	GUI
Structure determination	
Monte-Carlo	GUI
Simulated annealing	
Charge Flipping	GUI
Fourier analysis	
Measurement Data and Refinement Parameters	
Laboratory and synchrotron X-ray data, constant wavelength and TOF neutron data	GUI
Single crystal data	
EDXRD data	
Magnetic structure refinement	
Combined refinement of X-ray and neutron powder data	GUI
Combined refinement of powder and single crystal data	
Variable Counting Time (VCT)	GUI
Non dependence on X-ray data (all kinds of XY data can be fitted)	GUI
Support of non-equidistant x-axis steps	GUI
Support of negative x-axis values	GUI
Refines simultaneously on any number of diffraction patterns with any number of peaks, any number of datapoints, and any number of parameters	GUI
Refines on any number of structures per diffraction pattern with any number of sites per structure and atoms per site	GUI
All parameters can be fixed, refined, constrained and restrained	GUI
Quantitative Phase Analysis	
Rietveld method	GUI
Quantification of phases with partial or no known crystal structures (PONKCS method)	GUI
Internal standard method	GUI
External standard method	GUI
PONKCS method	GUI
Pattern scaling method	
Degree of crystallinity analysis	GUI
Brindley microabsorption correction	GUI
Constrained phase composition	
Constrained elemental composition	
Automatic Removal of Phases	

Convolution based profile fitting

Profile functions	
Pearson VII, Split-Pearson VII, Gaussian, Lorentzian	GUI
Voigt, Pseudo-Voigt, Modified Pseudo-Voigt, TCHZ-type Pseudo-Voigt	GUI
Hats, hyperbolas, circles, exponentials	GUI
Simple and Full Axial Models (Cheary & Coelho, 1998a, b)	GUI
Finger asymmetry correction (Finger et al., 1994)	GUI
User-defined profile functions	
User-defined convolutions, Fourier transforms	
Measured instrument functions	GUI
Calculated instrument functions - Fundamental Parameters Approach (FPA)	GUI
Standardless size-strain analysis	GUI
Refinable instrument parameters	GUI
Determination of mean sample absorption coefficient	GUI
Tube tails modelling	GUI
Absorption edge modelling	GUI

Size-strain analysis

Classic Scherrer method	GUI
Integral breadth approach equivalent to the "Double-Voigt Approch" (Balzar, 1999)	GUI
Isotropic size-strain broadening	GUI
Anisotropic size-strain broadening	
WPPM	
User-defined	

Background Models

Chebychev polynomial of n'th order	GUI
Single peaks	GUI
1/X-type background	GUI
Modulated background	
User-defined	

Preferred Orientation Models

March-Dollase	GUI
Spherical Harmonics	GUI

Anisotropic Refinement Models

Microstructure (size-strain-disorder)
Peak shifts
Preferred orientation
Temperature factors

Constraints and Restraints

All refinement parameters can be constrained / restrained	GUI
Linear and non-linear constraints	GUI
Rigid and soft bodies with all parameters refineable (bond angles,	
Support of fractional, Cartesian, and internal coordinates (Z-matrix notation)	
Penalty functions. Can be applied to all refineable parameters	
Anti-Bump, Parabola, lattice energy minimization, user-defined	
Rigid Body Editor for graphical creation of rigid bodies	

Minimization Procedures

Marquardt	GUI
BFGS method	GUI
Line minimisation	GUI
Extrapolation	GUI
Sparse matrix method	GUI

Miscallenous

Extensive macro-language /
support of user-defined refinement parameters and refinement models
Fully automated operation possible

1.3 TOPAS references

For publication of results obtained with TOPAS the following references can be used:

- For general use of TOPAS:
  Bruker AXS (2008): TOPAS V4: *General profile and structure analysis software for powder diffraction data.* - User's Manual, Bruker AXS, Karlsruhe, Germany.

- Convolution based profile fitting / fundamental parameters approach:
  Cheary, R.W. & Coelho, A.A. (1992): *A fundamental parameters approach to X ray line-profile fitting.* - J. Appl. Cryst., **25**, 109-121.
  Cheary, R.W., Coelho, A.A. & Cline, J.P. (2004): *Fundamental Parameters Line Profile Fitting in Laboratory Diffractometers.* - J. Res. Natl. Inst. Stand. Technol., **109**, 1-25.
  Kern, A., Coelho, A.A. & Cheary, R.W. (2004): *Convolution based profile fitting.* - Diffraction Analysis of the Microstructure of Materials, edited by Mittemeijer, E.J. & Scardi, P. Materials Science, Springer, ISBN 3-540-40510-4, 17 - 50.

  Kern, A. (2008): *Convolution Based Profile Fitting.* - Principles and Applications of Powder Diffraction. Editors: Clearfield, A., Bhuvanesh N. & Reibenspies J. Blackwell Publishers, 400 pages.

- Indexing
  Coelho, A.A. (2003): *Indexing of powder diffraction patterns by iterative use of singular value decomposition.* - J. Appl. Cryst., **36**, 86–95.
  Coelho, A.A. & Kern, A. (2005): *Discussion of the indexing algorithms within TOPAS.* - CPD Newsletter, **32**, 43-45.

- Structure determination:
  Coelho, A.A. (2000): *Whole Profile Structure Solution from Powder Diffraction Data using Simulated Annealing.* - J. Appl. Cryst., **33**, 899-908.
  Coelho, A.A. (2007): *A charge-flipping algorithm incorporating the tangent formula for solving difficult structures.* - Acta Cryst., **A36**, 400–406.

2 ELEMENTS OF THE USER INTERFACE

The TOPAS graphical user interface is intended for working in both GUI and Launch Mode. Refinements in GUI Mode require input to a *Parameters Window*, refinements in Launch Mode are controlled by an INP format file. Consequently, several windows and dialogs used in GUI mode will be not available or have no effect in Launch Mode.

The TOPAS screen (Fig. 2.1) consists of the following elements:

- **Menubar**
  Contains the names of submenus, which provide lists of all commands available together with their shortcuts respectively toolbar icons, if existing.
- **Toolbar**
  Displays the most important commands in form of icons for fast access.
- **Working Area**
  Contains all elements needed for profile fitting such as observed and calculated data, fit parameters, and fit results, which are displayed in separated views.
- **Status Bar**
  Displays some context sensitive help information and the x- and y-coordinates of the data cursor in the active *Scan Window*.

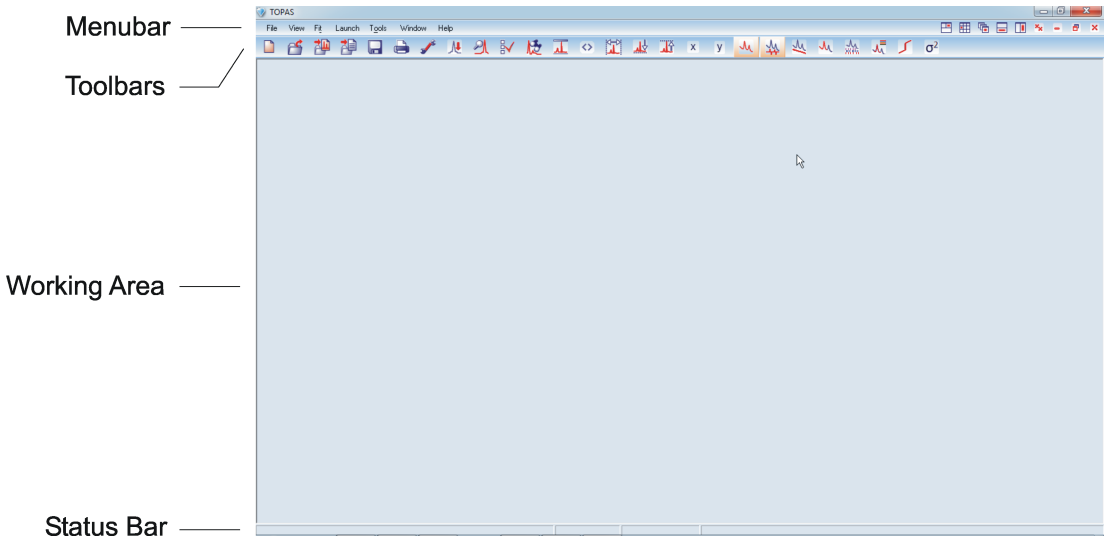


Fig. 2.1: TOPAS screen.

The *Working Area* serves as a container for the following views:

- ***Scan Window***
  The *Scan Window* (Fig. 2.2) is the actual field of operation, and is available in both the GUI and the Launch Mode. The following views are related to it:
  - ***Quick Zoom Window***
  - ***Weight Percent Pie Chart Window***
  - ***Chart Options Dialog***
  - ***Peak Search Dialog*** (GUI mode only)
  - ***Peak Details Dialog*** (GUI mode only)
  - ***Options Dialog*** (most options available in GUI mode only)
  - ***Fit Window*** with its ***Refinement Options Dialog***

  The *Scan Window* is described in section 3.
- ***Parameters Window*** (GUI mode only)
  The *Parameters Window* represents all refinement parameters available in GUI Mode by a hierarchically organized tree view (Fig. 2.3). It can be docked at the left, right, top or bottom of the *Working Area* and is described in section 4. The *Parameters Window* has no effect in Launch Mode.
- ***Structure Viewer*** / ***Rigid Body Editor Window***
  The *Structure Viewer* / *Rigid Body Editor Window* allows to view crystal structures and 3D electron densities, and provides for creation and editing of rigid bodies. It can be docked at the left, right, top or bottom of the *Working Area* and is described in section 5.
  - ***Normals Plot Viewer*** (Launch mode only)
    The *Normals Plot Viewer* is integrated into the *Structure Viewer* and allows plotting of lattice plane normals to e.g. visualize three-dimensional crystallite size and strain distributions.

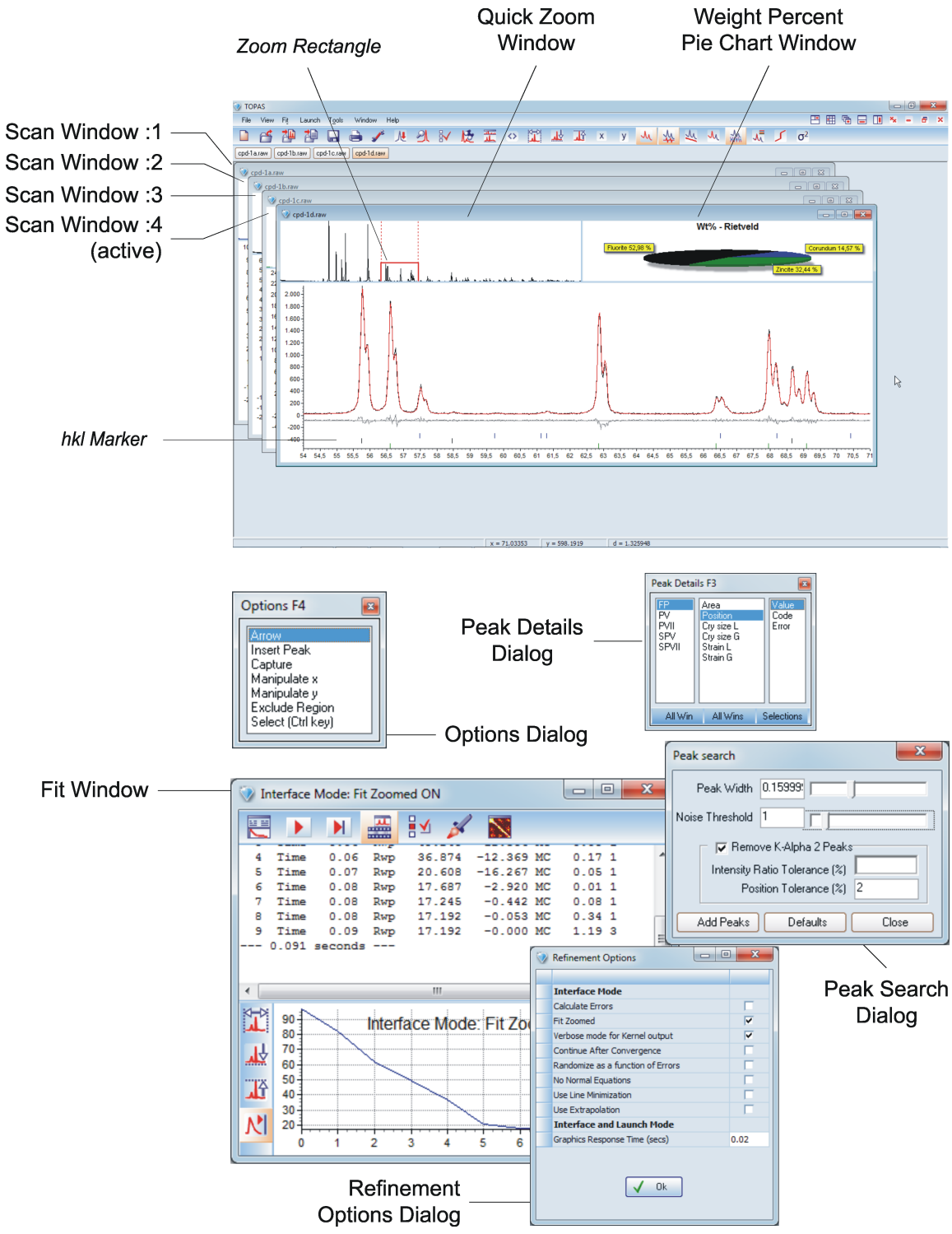


Fig. 2.2: Scan Windows with related views.

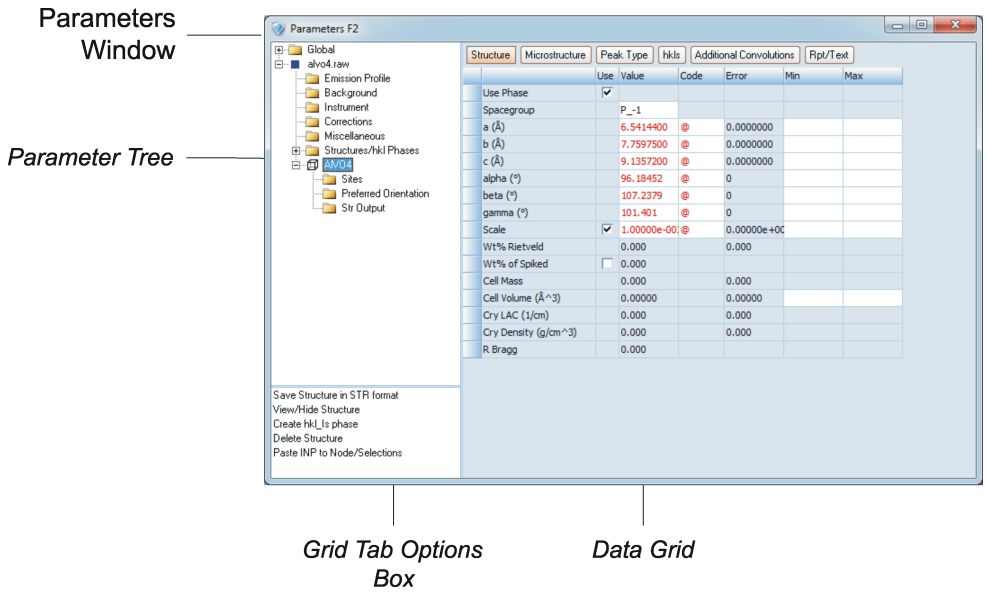


Fig. 2.3: *Parameters Window*.

The *Toolbar* offers the following menu commands and toolbar icons to display or hide the various windows and dialogs:

Menu:	Icon:	Shortcut:	Result:
View - Quick Zoom			Displays / hides *Quick Zoom Window*
View - Pie Chart	n.a.		Displays / hides *Pie Chart Windows*
View - Parameters Window		F2	Displays / hides the *Parameters Window*
View - Peak Details Window		F3	Displays / hides the *Peak Details Dialog*
View - Options Window		F4	Displays / hides the *Options Dialog*
Fit - Fit Window		F5	Displays / hides the *Fit Window*
View - Search Peaks...			Displays / hides the *Peak Search Dialog*
Tools - New Rigid Body Editor Window	n.a.		Displays / hides the *Rigid Body Editor*

3 THE SCAN WINDOW

3.1 Common features in GUI Mode and in Launch Mode

3.1.1 Display

In TOPAS an unlimited number of data sets can be loaded and refined simultaneously. The data can be displayed in one single *Scan Window* (default) or in individual *Scan Windows*, which can be tiled horizontally and vertically, cascaded and closed within the *Working Area* using the appropriate *Window* menu commands.

If there are no *Scan Windows* then a *Scan Window* is created when a scan in loaded. Closing a *Scan Window* does not unload its data! The data can be redisplayed by selecting *Window - One Scan per Window*.

The following features concern the data displayed in the *Scan Window*:

- Axes scales comprise x (= 2θ), Q (=2π/d), and d for the x-axis and y (= linear), Sqrt(y) (= square root) and Ln(y) (= logarithmic) for the y-axis.
- Display of observed, calculated, difference, and background data
- Display of calculated intensities for individual single peaks or phases
- Animated display of the profile fitting process

For Whole Powder Pattern Decomposition (Pawley and Le Bail method) and Rietveld refinement the following additional items are displayed:

- hkl markers at the bottom
- The phase name plus relative phase amounts in the upper right part of the window

Available menu commands and toolbar icons are:

Menu:	Icon:	Result:
View - X-Axis Scale - *Linear* *Q* *d-spacing*	 X Q d	Sets the x-axis scale to x Q d
View - Y-Axis Scale - *Linear* *Sqrt(y)* *Ln(y)*	 Y √y lny	Sets the y-axis scale to y Sqrt(y) Ln(y)
View - Calculated, Difference, etc... - Calculated		Displays / hides calculated curves
View - Calculated, Difference, etc... - Background		Displays / hides background curves
View - Calculated, Difference, etc... - Difference		Displays / hides difference curves
View - Calculated, Difference, etc... - Single Peaks		Displays / hides single peaks
View - Calculated, Difference, etc... - Cumulative Chi²		Displays / hides cumulative Chi²
View - Calculated, Difference, etc... - Normalized SigmaYobs²		Displays / hides normalized SigmaYobs²
View - hkl Ticks		Displays / hides hkl ticks
View - Phase names		Displays / hides phase names and abundance
n.a.		View previous / next *Scan Window* using LMB / RMB

Further features for Whole Powder Pattern Decomposition (Pawley and LeBail method) and Rietveld refinement are (*View - Show hkl Ticks* must be on):

- When moving the mouse onto the phase name, the intensity distribution of this particular phase will be shown using a bold line. Additionally the hkl markers for this phase are highlighted using small triangles at the bottom of the markers.
- When moving the mouse onto the hkl markers, the cursor will always lock-on to the nearest hkl marker, which makes it easier to select closely spaced hkl's. After lock-on the marker will be highlighted using a small triangle at the top of the marker and some hkl information will be displayed. For systematic overlaps of non-symmetry equivalent reflections all possible hkl's will be shown.

3.1.2 Y-axis limits, zooming, panning and selecting

- **Y-axis limits**
  Y-axis minimum (Y1) and maximum limits (Y2) can be fixed or unfixed using the *Fix Y1* and *Fix Y2* toolbar icons. Fixing options include fixing of Y1 to zero as well as to the minimum count value, and Y2 to the maximum count value. Fixing Y1 and / or Y2 affects both the zooming and panning behaviour as described below.

- **Zooming**
  Can can be performed in both the *Scan Window* and the *Quick Zoom Window* (section 3.3.1). Zooming is performed by pressing the LMB at the upper left position of the targeted zoom area and dragging the mouse to define the zoom area. By doing the same backwards, the axies are reset to the full X and Y scale. Zooming and unzooming is also possible by pressing and scrolling the mouse wheel. For zooming out the X-scale can be compressed using the *Compress* toolbar icon. The previously zoomed region can be reset using the shortcut menu command *Reset Axies to Previous* (section 3.1.3).
  Alternatively the rectangle in the *Quick Zoom Window* allows a modification of the zoomed area by grabbing one of the red lines with the mouse and moving it. A double click on the *Quick Zoom Window* resets the *Scan Window* to full scale.
  Note: If Y1 or Y2 have been fixed to the minimum or maximum count value respectively, the y-axis limits will be automatically adjusted to the lowest and highest count value within the zoomed x-region.
  X and Y limits can also be set directly to discrete values using either the shortcut menu or the chart options dialog, both described in section 3.1.3.

- **Panning**
  Possible in both the *Scan Window* and the *Quick Zoom Window*. By pressing the RMB and moving the mouse, the zoomed area is moved over the data range horizontally and vertically (note: vertical panning requires Y1 and Y2 to be unfixed).
  Horizontal panning is also possible by scrolling the mouse wheel or by using the horizontal scroll bar, which is displayed if *View - Horizontal Scroll Bar* has been selected.
  Note: When panning horizontally with fixed Y1 and Y2, the y-axis limits will be automatically adjusted while moving the zoomed region along the x-axis.

- **Selecting**
  Performed similar to zooming with the CTRL key pressed simultaneously. During movement the mouse cursor changes into a selection cursor. All peaks in the selection area are marked by a dashed line. Selected regions are marked with dashed patterns. Selected peaks and excluded regions can be deleted with the DEL key or using the shortcut menu (see section 3.1.3). In addition selected peaks can be manipulated using the *Peak Details Window*.

Note: Zooming and panning also work while fitting!

Available menu commands and toolbar icons are:

Menu:	Icon:	Result:
View -		
Dont fix Y1		Does not fix Y1
Fix Y1 to zero		Fixes Y1 to zero
Fix Y1 to min value		Fixes Y1 to the minimum count value
View -		
Dont fix Y2		Does not fix Y2
Fix Y2 to max value		Fixes Y2 to the maximum count value
View - Horizontal Scroll Bar	n.a.	Displays / hides the horizontal scroll bar
View - Unzoom	n.a.	Resets to the full X and Y scale
n.a.		Compresses the x-axis left / right using the left / right mouse buttons

3.1.3 Shortcut menu options

the RMB anywhere in the *Scan Window* will open a shortcut menu offering access to the following features:

- **Set X1 / X2 / Y1 / Y2 to Mouse Position**
  Sets the x- and y-axis limits to the actual mouse position. Note: Fixed Y1 or Y2 axis limits will be automatically unfixed to allow setting of Y1 and Y2.
- **Reset Axies to Previous**
  Resets to the previously zoomed region
- **Unzoom**
  Resets axies to the full X and Y scale
- **Edit / Print / Chart Options**
  Opens the *Chart Options Dialog* (section 3.3.3) for defining various chart properties such as labels, ticks and fonts, which can be saved as default. Charts can be printed directly or copied to the clipboard.
- **Delete Selection**
  Deletes selected peaks and excluded regions

- **Delete Nearest Peak F9**
  Deletes the peak nearest to the actual mouse position

Note: As it is not possible in Launch Mode to graphically insert peak and excluded regions, the *Delete Selection* and *Delete Nearest Peak F9* will have no effect in Launch Mode (see also section 3.2).

3.2 Features available in GUI Mode only

The following features are available in GUI Mode only, as in Launch Mode the refinement is completely controlled by the input file:

- Graphical peak treatment such as manual peak insertion, automatic peak search, and the editing of peak properties
- Display of peak markers and profile parameters for single peaks
- Highlighting the intensity distribution of a peak with a bold line as the mouse is moved over its peak marker
- Graphical insertion of excluded regions
- Selection of peaks and excluded regions with the mouse (pressing the CTRL key allows multi-selection)
- Deletion of the selection with the DEL key

With respect to the fitting process it is important to understand the following features of the *Scan Window* in GUI Mode:

- In general all datasets in visible *Scan Windows* are fitted simultaneously.
- If several datasets are loaded in several *Scan Windows*, the data of minimized as well as closed windows will not be fitted.
- If several datasets are loaded in several *Scan Windows*, and one of these windows is maximized, then only the data within the maximized window are fitted.
- If the menu switch *Fit - Fit Zoomed* is selected only the data points displayed in the *Scan Window* are used for fitting. All data outside the zoomed area are ignored!

3.3 Views related to the Scan Window

3.3.1 Quick Zoom Window

Always displays the complete scan, which is particularly useful if the *Scan Window* only displays a zoomed area. Inside of the *Quick Zoom Window* a zoom rectangle represents the actually displayed data range within the active *Scan Window*. It allows a modification of the zoomed area by grabbing one of the red lines with the mouse and moving it. A double click on the *Quick Zoom Window* resets the *Scan Window* to full scale.

3.3.2 Weight Percent Pie Chart Window

Displays a weight percent pie chart providing quantitative analysis results.

3.3.3 Chart Options Dialog

This dialog (Fig. 3.1) is linked to the shortcut menu of the *Scan Window* (RMB) and provides two pages allowing to copy and print the chart as well as to define properties of the chart title and axes. Settings can be saved in STARTUP.DEF (see the Technical Reference manual) with the exception of the x- and y-axis limits, which cannot be kept as defaults. Note: Definition of Y1 or Y2 axis limits requires them to be unfixed.

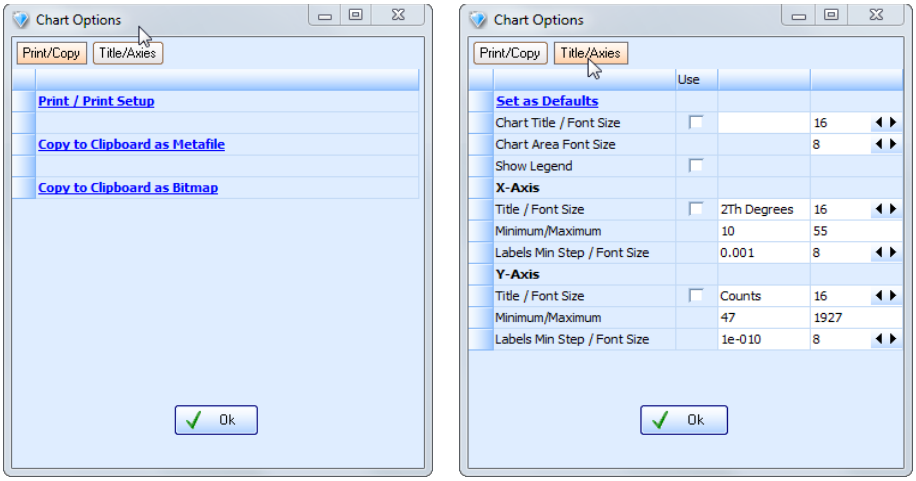


Fig. 3.1: *Chart Options Dialog.*

3.3.4 Peak Search Dialog

Note: GUI mode only

Offers an automatic peak search according to Savitzky & Golay, 1964 (Fig. 3.2). $K\alpha_2$ peaks can be removed automatically.

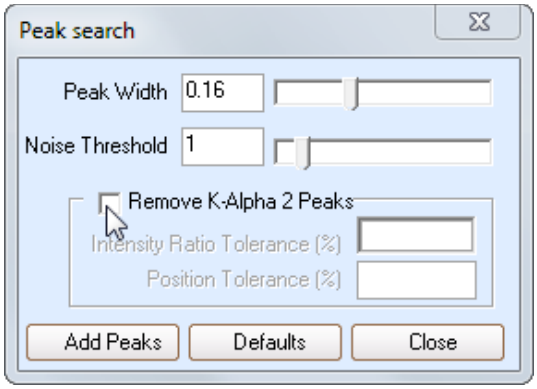


Fig. 3.2: *Peak Search Dialog.*

- **Peak Width**
  Peak search parameter. Should correspond approximately to the peak halfwidth
- **Noise Threshold**
  Peak search parameter. Defines the minimum intensity of a peak. A noise threshold of one corresponds to a noise level of two sigma.
- **Remove K-Alpha 2 Peaks**
  Removes $K\alpha_2$ peaks automatically. *Intensity Ratio Tolerance* and *Position Tolerance* define a window to recognize $K\alpha_2$ peaks.

3.3.5 Peak Details Dialog

Note: GUI mode only

Allows the insertion of different peak types as well as the direct editing of peak parameter values and refinement codes in the *Scan Window* (Fig. 3.3).

Peaks inserted into the *Scan Window* will be of the peak type selected in the left column. It is possible to insert different peak types into the same powder pattern. For each peak inserted a peak marker is displayed. In addition near to the top of the peak marker some peak properties such as profile parameter values or refinement codes for the associated peak can be shown depending on the selections in the second and third column of the *Peak Details Dialog*.

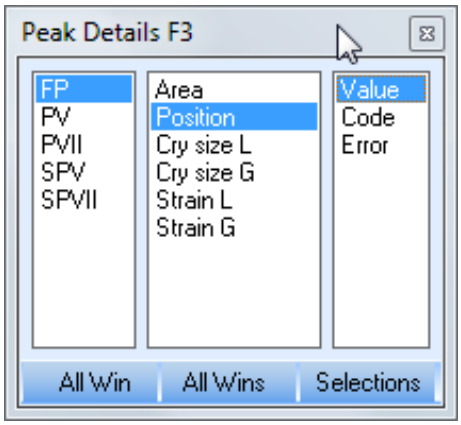


Fig. 3.3: *Peak Details Dialog.*

When opening the *Peak Details Dialog* the cursor will change to peak insertion mode. The form of the cursor unambiguously reflects the selected peak type. A mouse click using the left mouse button in the *Scan Window* will insert a peak, when the *Peak Details Dialog* is open. Peaks can also be inserted anytime even when the *Peak Details Window* is closed by pressing the CTRL key when clicking the left mouse button.

Peak insertion is assisted by the "Bouncing Ball" feature. The color of the ball follows the scan that it is on. When more that one range is displayed then the scan with the

bouncing ball is selected for peak insertion; the Bouncing Ball feature automatically selects the scan closest to the cursor. If the scan with the Bouncing Ball has more than one peaks phase then the user is prompted to select the a peaks phase from the tree view.

An important feature of the *Peak Details Dialog* is the direct editing capability of peak parameter values and refinement codes in the *Scan Window*. A mouse click (LMB) on the text displayed nearby the peak using the left mouse button will open an edit field, which allows to change parameter values or refinement codes. Any changes have to be confirmed using the Enter key.

For changing the values of a peak group there are three buttons on the bottom of the Peak Details Window:

- **All Win**
  Overwrites the values of all peaks in the active Scan Window with the value in the edit field.
  Note: A change of the peak position moves all peaks to the same position!
- **All Wins**
  Same as All Win, but changes will be applied to all peaks in all Scan Windows.
- **Selections**
  Changes are applied to all selected peaks in the active Scan Window.

3.3.6 Options Dialog

Provides several mouse controlled modes (Fig. 3.4). Of these only *Capture* will have an effect in Launch mode, all other options are intended for use in GUI mode only.

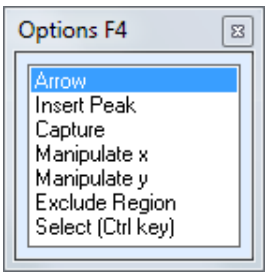


Fig. 3.4: *Options Dialog.*

- **Arrow**
  Normal mouse operation for zooming and panning.
- **Insert Peak**
  Shows the *Peak Details Dialog* and switches the mouse to peak insertion mode.
- **Capture**
  Any observed or calculated data including difference plots in a *Scan Window* can be captured by the mouse. After clicking on the diagram line a new *Scan Window* is opened which contains the captured data. This feature allows for the export of any calculated data in a separate data file.

- **Manipulate X**
  Allows the movement of a selected scan in the positive or negative x-direction. This feature is useful for comparing different data sets or for visualisation of 2θ errors.
  Note: The data set in memory which constitute loaded data is directly manipulated; the original data file remains unchanged. Subsequent calculations are performed on the modified data.
- **Manipulate Y**
  Similar to *Manipulate X* but in the y-direction.
  Note: The data set in memory which constitute loaded data is directly manipulated; the original data file remains unchanged. Subsequent calculations are performed on the modified data.
- **Exclude Region**
  Allows the exclusion of selected data ranges from calculations. Excluded regions are defined with the mouse by clicking and dragging. An unlimited number of excluded regions are allowed including the case of overlap.
- **Select**
  Allows the selection of peaks and excluded regions in the *Scan Window*.

3.3.7 Fit Window

This is the central place for controlling the refinement process (Fig. 3.5). It basically consists of a text field and a plot window, and is a normal top level window by default which can be docked at the left, right, top or bottom of the *Working Area*. Note: If the *Fit Window* is docked then it cannot be maximised or minimised.

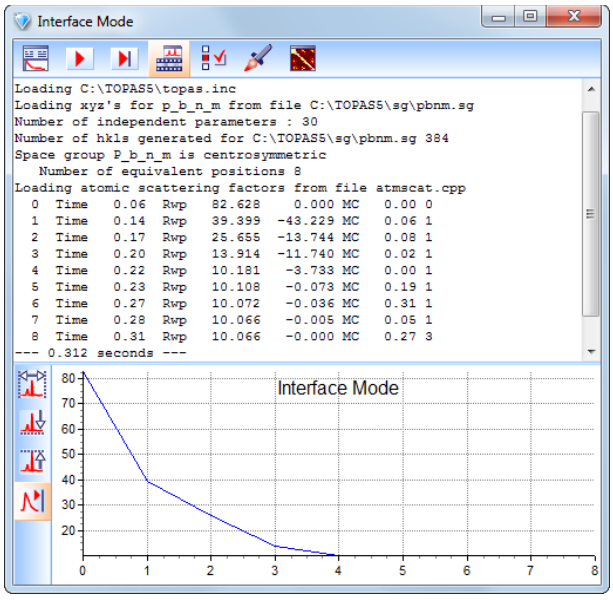


Fig. 3.5: *Fit Window.*

The text field provides information about the refinement process such as some space group information, number of independent parameters, and details about the refinement iterations including iteration number, elapsed time, R_{WP} as well as R_{WP} difference, and the number of Marquardt cycles (MC).

The plot window shows an R_{WP} plot versus refinement cycle, which is updated after each iteration. This window offers the same zooming and panning possibilities as the *Scan Window*; additionally, similar toolbar icons as well as a shortcut menu (RMB) are available. For these options refer to sections 3.1.2 and 3.1.3. When zooming into the R_{WP} plot, updating stops in order to allow examination of the zoomed region. Updating can be started again using the *Keep X2 at max X* toolbar icon, an unzoom operation will show the whole R_{WP} plot again.

Icon:	Shortcut:	Result:
	n.a.	Update the R_{WP} plot after each iteration and keep X2 at the maximum x-axis limit

When working in GUI mode, the *Fit Window* caption will change to “Interface Mode” and additionally display the "Fit Zoomed" status, which is either ON or OFF (and can be set in the *Refinement Options Dialog*, see below). In Launch Mode the *Fit Window* caption will change to “Launch Mode” and additionally display the input file name. The R_{WP} plot window copies the content of the *Fit Window* caption.

At the end of a refinement, due to convergence or by user intervention, the Start, Step, Stop, and Break Cycle buttons are hidden. The "Refinement Converged" dialog at the end of refinement is modeless and allows inspection of the refinement results before accepting any changes.

The horizontal toolbar offers the following options:

Icon:	Shortcut:	Result:
	n.a.	Displays or hides the text and / or the R_{WP} plot
	F6	Starts the refinement
	F7	Performs one refinement iteration
	Shift + F8	Stops the refinement
	n.a.	Cancels the current refinement cycle.
	n.a.	Animation of the graphics in the *Scan Window* on/off
	n.a.	Displays the *Refinement Options Dialog*
	n.a.	Switches between GUI Mode and Launch Mode
	n.a.	Displays the Correlation Matrix Window

The Correlation Matrix Window operates in both Launch and GUI mode, example output is as shown in Fig. 3.6. Providing meaningful parameter names eases reading of the correlation matrix. MouseMove over the correlation matrix displays a hint comprising the corresponding parameter names, values and errors.

The display of the matrix can be zoomed using Ctrl-MouseWheel. Pressing the LMB and dragging the mouse translates the matrix.

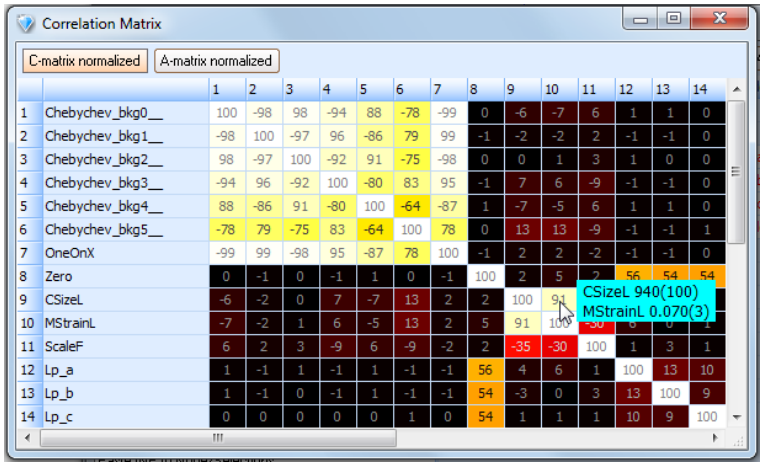


Fig. 3.6: *Correlation Matrix Window*.

The *Refinement Options Dialog* (Fig. 3.7) offers several options to control the refinement mainly in GUI mode.

Note: All options provided in the "Interface Mode" section of the *Refinement Options Dialog* will have no effect in Launch Mode, as the refinement will be completely controlled by the input file.

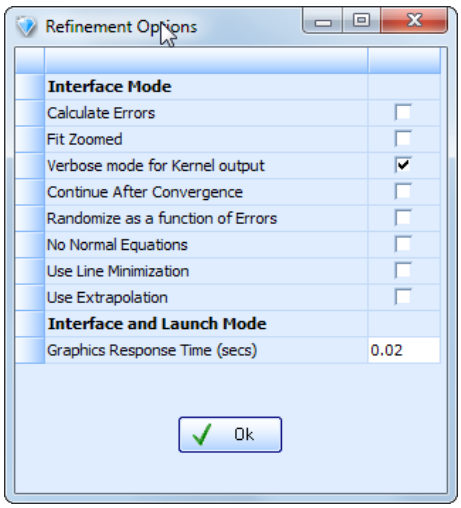


Fig. 3.7: *Refinement Options Dialog*.

Interface Mode

- **Calculate Errors**
  Calculates errors, if checked
- **Fit Zoomed**
  If checked, only the zoomed region will be fitted
- **Verbose mode for Kernel output**
  Toggles kernel output in the text field between verbose and brief
- **Continue After Convergence**
  Refinement is continued after convergence
- **Randomize as a function of Errors**
  Useful if *Continue After Convergence* is used. Performs a random parameter change based on its error and then continues the refinement.
- **No Normal Equations**
  Prevents the use of normal equations in the minimization routine; useful if only effects of line minimization are sought
- **Use Line Minimization**
  Invokes the use of line minimization. Faster convergence and often to a lower minima is observed if the refinement is far from the global minimum.
- **Use Extrapolation**
  Often increases the convergence rate if used with *Line Minimization*

Interface and Launch Mode

- **Graphics Response Time**
  Defines the update frequency of the *Scan Window* and the R_{WP} plot window. Reducing the update frequency will leave more CPU time for numerical calculations.

4 THE PARAMETERS WINDOW

4.1 Elements of the Parameters Window

The *Parameters Window* (Fig. 2.3) provides access to all refinement parameters available in GUI Mode. It consists of a hierarchal tree view of refinement models and a data grid containing the associated refinement parameters and parameter attributes.

For details about any refinement parameters and parameter attributes please refer to the Technical Reference manual.

The command *Window - Clone Parameters Window* clones the *Parameters Window* which is useful for viewing different parameter types simultaneously. For example, parameter values and their codes can be viewed simultaneously with two *Parameter Windows* in cases where one *Parameter Window* displays only one type of these types at a time. The number of cloned *Parameters Windows* is not restricted.

4.1.1 The tree view

Displays a dynamic tree essentially representing all contributions to scan data coming from the source, the instrument and the sample, which may consist of one or more (crystallographic) phases. After starting TOPAS the tree only contains the *Global* item, which is a permanent first level item and allows defining of global refinement settings such as the convergence criterion. For each scan (*Range*) loaded, additional first level items (*Range* items) are created.

For single line up to Whole Powder Pattern Fitting, Whole Powder Pattern Decomposition, and Rietveld analysis so-called "Peak Phases", "hkl Phases", and "Structures", respectively, are added as range dependent items (second level items) to a selected *Range* item[1]. Using the checkbox displayed next to the tree node, phases can be set to use or no use for the refinement.

The *Global* item is most useful when more than one scan is loaded. It allows the simultaneous viewing and manipulation of refinement parameters common to ranges. Some of the possibilities include the defining of parameter values and constraints across ranges as well as the loading of a common source emission profile or structural data into all or selected ranges.

Most tree items have shortcut menus via the RMB. The contents of these menus are also mirrored in the *Grid Tab Options Box* (Fig. 2.3) which can be used alternatively.

1 Peak Phases, hkl Phases and Structures all represent phases and are related to the keywords *x_o* / *d_o*, *hkl*, and *str* respectively; see the Technical Reference manual.

4.1.2 The data grid

The data grid contains refinement parameters and parameter attributes, as well as range and phase dependent display properties such as colors, data point size and line width. The contents of the data grid are related to the tree item selected; it consists of a varying number of pages dependent on the type as well as amount of information to be displayed. In the following some general information about the data grid is provided, for a detailed description of all data grid pages refer to section 4.2.

In the data grid, for each tree item, there is at least one grid page containing refinement parameters and codes, and the *Rpt/Text* page, which, when selected, expands to the *Report Format*, *Text*, and *Grid* pages. Selecting the *Grid* page switches back to the last used grid page.

Elements of the grid page

Each grid page allows the selection of parameters for refinement and to define their parameter attributes including "Use", "Value", "Code", "Error", "Min/Max":

- **Use**
  Boolean switch to indicate the use of the parameter for refinement
- **Value**
  The parameters value. If the parameter is to be refined, the parameter value will be updated with the refined value after each refinement cycle.
- **Code**
  Can be either:
  - a switch indicating independent refinement of the parameter ("Refine" or "@")
  - a switch indicating that the parameter is not to be refined ("Fix" or "!")
  - a user defined name given to the parameter
  - an equation, also in terms of other parameters
- **Error**
  The parameters error (esd)
- **Min / Max**
  User defined limits for the parameter value

A double click on the code field will switch the code from "Refine" to "Fix" and vice versa. If a user defined parameter name has been defined, the "!" character will be placed in front of the parameter name or removed, respectively.

The colors used in the grid have the following meaning:

- Parameter values with a grey background are read only
- Parameter values with a white background can be modified by the user. The text color is dependent on its code:
  - Black text: The parameter is fixed
  - Red text: The parameter is refined
  - Blue text: The parameter is calculated from an equation

Selection in the grid

Rows or columns can be selected by clicking the row or column heading. Adjacent rows or columns can be selected by either dragging across the row or column headings, or by selecting the first row or column and then holding down SHIFT and selecting the last row or column. Nonadjacent rows or columns can be selected by selecting the first row or column, and then holding down CTRL and selecting the other rows or columns. The complete grid can be selected by clicking the *Select All* button of the grid at the top left of the grid.

Export of grid data

The grid page contents can be copied to the clipboard or transferred to the TopasEditor or Microsoft Word (if available) using the shortcut menu. This allows for the easy creation of files with particular refinement results such as various peak versus intensity lists, xyz lists of atomic coordinates, and much more. A detailed description of the shortcut menu options is provided in section 4.2.3.

Import of grid data

The GUI supports general pasting of information in INP format from the clipboard into tree nodes or selected grid items. This provides for extremely convenient and fast duplication / exchange of any refinement data.

The Report Format and Text pages

Contains the content of the grid pages in the form of differently formatted text. Note: As for the grid, the contents of the *Report Format* and *Text* pages is dependent on the selected tree item.

The *Text* page represents the grid content as plain text in INP format and is useful for learning the INP format of TOPAS. It also faciliates the setup of INP files via copying & pasting of selected text. The *Report Format* page displays the grid page contents in the form of a formatted report.

The contents of both the *Report Format* and *Text* page can be copied to the clipboard, saved to a TXT file, printed directly, or transferred to the TopasEditor or Microsoft Word (if available) using the shortcut menu, see section 4.2.3.

4.2 Tree items and their associated data grid pages

4.2.1 Global item

The *Global* item (Fig. 4.1) allows viewing and manipulating of both global refinement settings as well as refinement parameters and codes across all loaded ranges.

Global refinement settings are provided in the *Global* items data grid including calculated step size for XY type measurement data, convolution steps, the global convergence criterion, and the maximum number of iterations to be performed.

With more than one scan data file loaded, the *Global* item represents a transposition of the *Range* item contents, where all refinement parameters common to all ranges

can be viewed and manipulated individually as well as simultaneously. Selected ranges can be turned on/off at once.

Second level items of the *Global* item are:

- *Background* (section 4.2.2.2)
- *Instrument* (section 4.2.2.3)
- *Corrections - Convolutions* (section 4.2.2.4)
- *Miscellaneous* (section 4.2.2.5)
- *Display*

Additionally the following items may be displayed as well:

- *All Peaks* (section 4.2.2.6)
- *All Structures / hkl Phases* (section 4.2.2.9)

The *Display* item provides for customization of display properties including colors, data point size and line width for each range. Note: The *Global Defaults* page allows for the definition of program defaults for the display properties with the exception of the observed data color, which cannot be kept as default to prevent ambiguity.

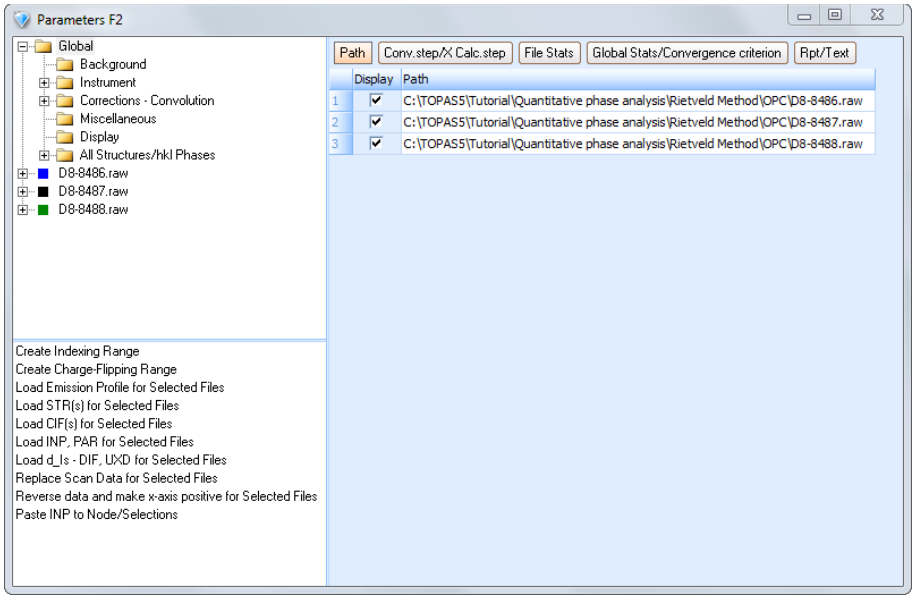


Fig. 4.1: *Global* item with its associated data grid.

The shortcut menu of the Global item offers the following options, for file types and formats refer to the Technical Reference manual:

- **Create Indexing Range**
  Creates a range for indexing, see section 4.2.3.
- **Create Charge-Flipping Range**
  Creates a range for Charge Flipping, see section 4.2.4.
- **Load Emission Profile for Selected Files**
  Loads a source emission profile from a LAM file for all selected files

- **Load STR(s) for Selected Files**
  Loads structure information from STR file(s) for all selected files
- **Load CIF(s) for Selected Files**
  Loads structure information from CIF file(s) for all selected files
- **Load INP, PAR for Selected Files**
  Loads information from INP and PAR file(s) for all selected files; while PAR files are intended for instrument parameters only, with INP files any keywords and macros supported by the GUI can be loaded
- **Load d_Is – DIF, UXD for Selected Files**
  Loads peak information from DIF or UXD files for all selected files, providing a link to the ICDD PDF via DIFFRACplus EVA and SEARCH
- **Replace Scan Data for Selected Files**
  Allows to exchange scan data for selected files to be refined using the same refinement model
- **Reverse data and make X-axis positive**
  Allows the use of data obtained from scans in negative x-axis regions
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2 Range item(s)

For each range loaded, an additional *Range* item is created and labelled using the file name of the measurement data file (Fig. 4.2). For multi-range RAW files an additional range number is appended to the range label. Ranges can be enabled or disabled using the checkbox next to the range node.

Range dependent second level items of the Range item are:

- *Emission Profile* (section 4.2.2.1)
- *Background* (section 4.2.2.2)
- *Instrument* (section 4.2.2.3)
- *Corrections* (section 4.2.2.4)
- *Miscellaneous* (section 4.2.2.5)

Additionally the following items may be displayed as well:

- *Peak Phase* (section 4.2.2.6)
- *hkl Phase* (section 4.2.2.7)
- *Structure* (section 4.2.2.8)
- *Structures / hkl Phases* (section 4.2.2.9)

Indexing and *Charge Flipping* ranges are specialized 1st level *Range* items and are described in sections 4.2.3 and 4.2.4, respectively.

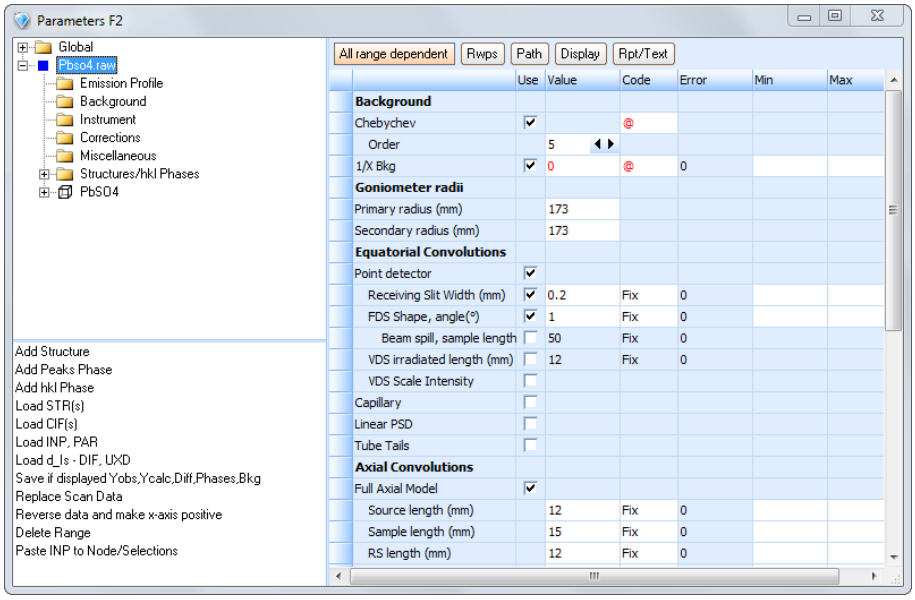


Fig. 4.2: *Range* item with its associated data grid showing the *All Range Dependent* page. All range dependent parameter values and codes can be viewed and manipulated in this data grid as well as in the respective second level items.

The shortcut menu of the *Range* item offers the following options, for file types and formats refer to the Technical Reference manual:

- **Add Structure**
  Adds an empty structure; default values are predefined in the file STR.DEF, see the Technical Reference manual.
- **Add Peaks Phase**
  Adds an empty peaks phase; default peak type is fundamental parameters
- **Add hkl Phase**
  Adds an empty hkl phase; default values are predefined in the file HKLI.DEF, see the Technical Reference manual.
- **Load STR(s)**
  Loads structure information from STR file(s)
- **Load CIF(s)**
  Loads structure information from CIF file(s)
- **Load INP, PAR**
  Loads information from INP and PAR file(s); while PAR files are intended for instrument parameters only, with INP files any keywords and macros supported by the GUI can be loaded
- **Load d_Is – DIF, UXD**
  Loads peak information from DIF or UXD files, providing a link to the ICDD PDF via DIFFRACplus EVA and SEARCH
- **Save if displayed Yobs, Ycalc, Diff, Phases, Bkg**
  Output of plot data. Allows to save observed, calculated, difference, background and individual phase intensity data in a comma separated text file for plot

generation using 3rd party software. Data is saved according to the y-axis scaling and the x-axis is changed to the selected 2θ, d, Q.

- **Replace Scan Data**
  Allows to exchange scan data for selected files to be refined using the same refinement model
- **Reverse Data and make X axis positive**
  Allows the use of data obtained from scans in negative x-axis regions
- **Delete Range**
  Deletes the selected range
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.1 Emission profile

Gives the ability to add and delete emission lines (Fig. 4.3) which form the emission profile of the X-ray source.

The *Options* page contains the following options:

Option:	Remarks:
Ymin on Ymax	Determines the x-axis extent to which peak tails are calculated (cut off)
No Th dependence	Defines an emission profile that is 2θ independent. Allows the use of non-X-ray data or fitting to negative 2θ values.
For LAM cursor	Switches the mouse cursor to a multi-line cursor representing the different emission lines of the current emission profile (Fig. 4.4)
Lam for Bragg angle	Refer to the Technical Reference manual
Calculate Lam	Refer to the Technical Reference manual

The shortcut menu of the Emission Profile item offers the following options:

- **Load Emission Profile**
- **Save Emission Profile**
- **Add Emission Line**
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

Selected emission rows can be deleted using the DEL key.

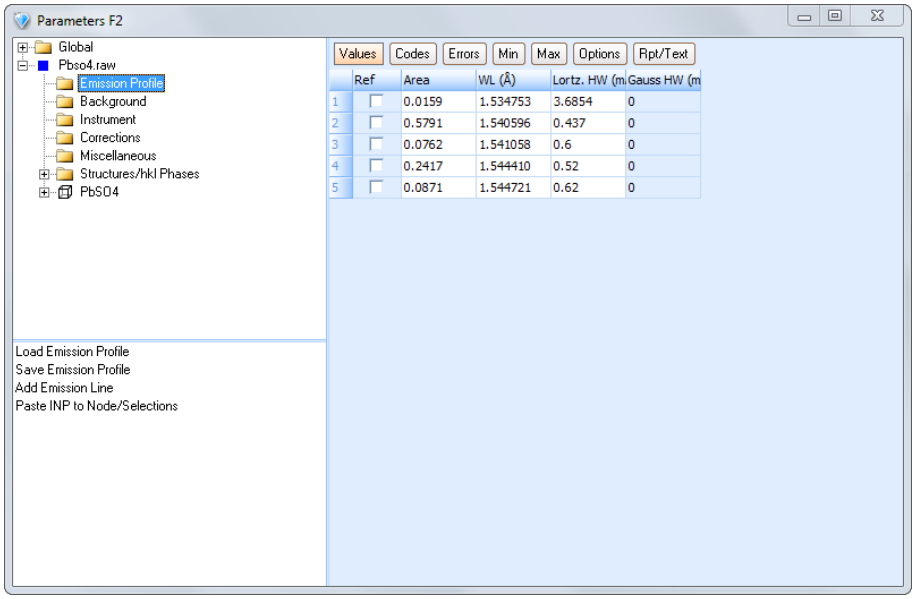


Fig. 4.3: *Emission Profile* item with its associated data grid showing a list of emission lines of the current emission profile (here CuKa5).

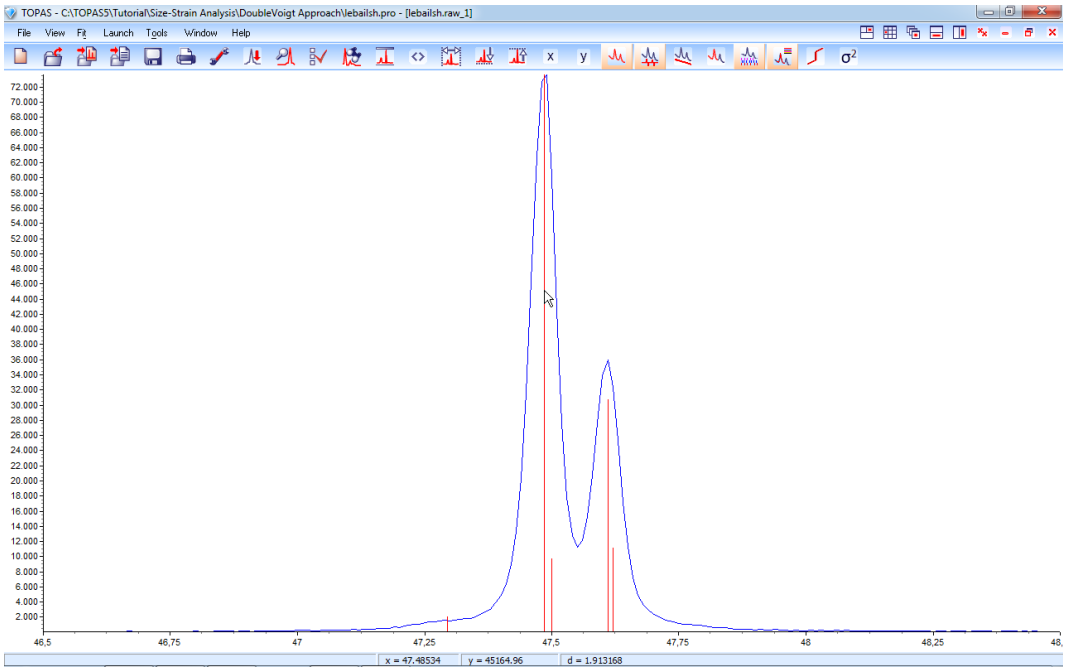


Fig. 4.4: With *For Lam cursor* switched on, a multi-line cursor representing the different emission lines of the current emission profile (here CuKa5) will be shown

For laboratory diffractometer systems a selection of predefined emission profiles is available in the LAM directory covering the most common anode target materials.

For other target materials as well as for synchrotron and neutron sources it is necessary to define a suitable emission profile. For accurate work it is necessary to refine on the emission profile shape using e.g. the NIST SRM 660a (LaB6) standard.

4.2.2.2 Background

Two background functions are provided (Fig. 4.5), a Chebychev polynomial of any order and a 1/X function.

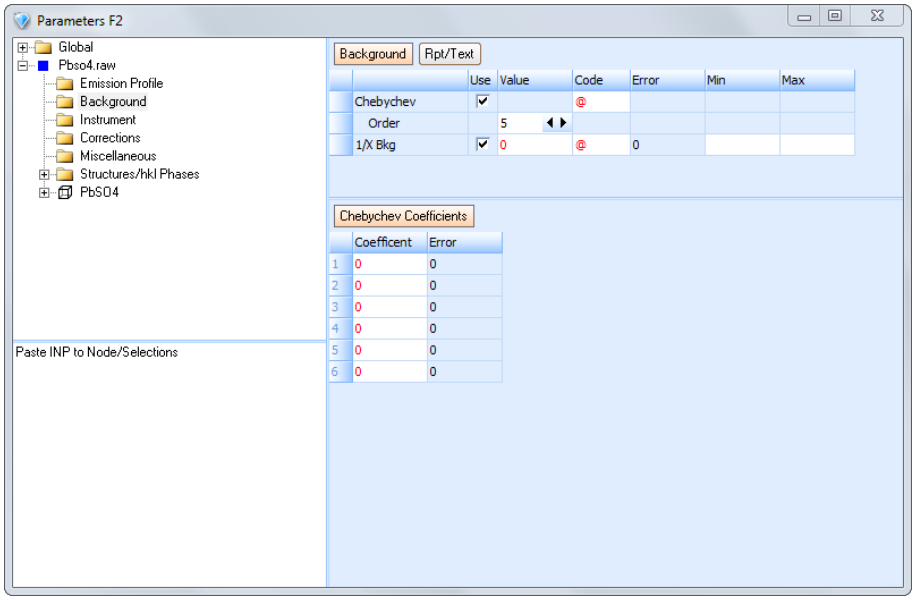


Fig. 4.5: *Background* item with its associated data grid.

The shortcut menu of the *Background* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.3 Instrument

Provides for the definition of the instrument function. For instruments operating in divergent beam geometry the Fundamental Parameters Approach is available in the *Divergent beam* page (Fig. 4.6); other instrument geometries can be empirically modelled, some more functions are offered in the *Additional Convolutions* page (Fig. 4.7). Note, that both the *Divergent beam* and the *Additional Convolutions* page provide different shortcut menus.

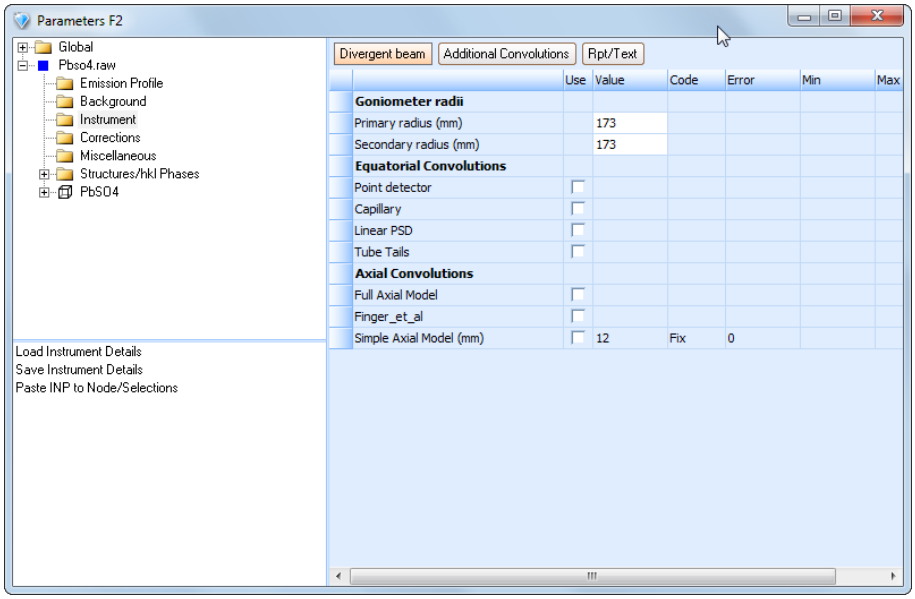


Fig. 4.6: *Instrument* item with its associated data grid showing the *Divergent beam* page. Note: *Point detector*, *Capillary*, *Linear PSD*, *Tube Tails*, *Full Axial Model*, and *Finger_et_al* are "Use" dependent expand/collapse grid items.

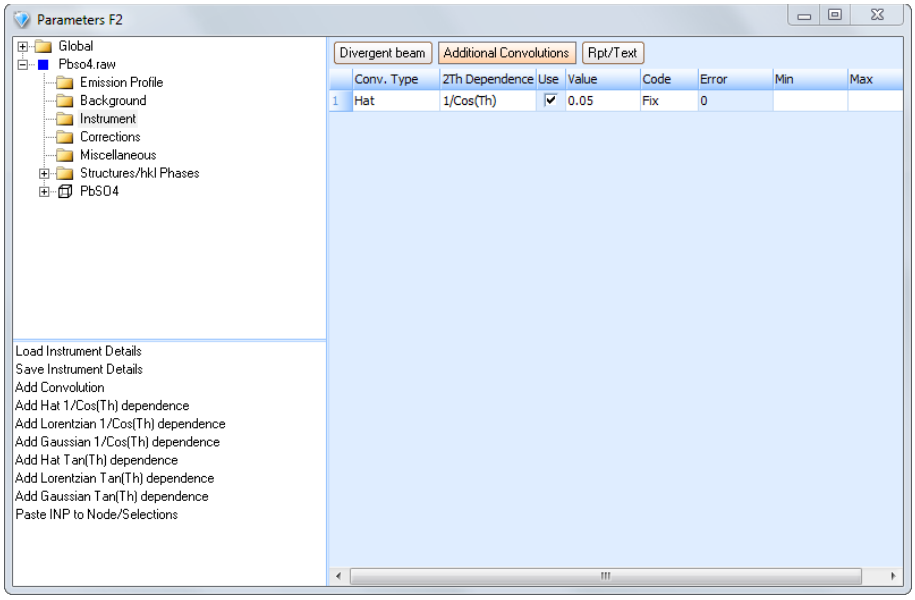


Fig. 4.7: Instrument item with its associated data grid showing the *Additional Convolutions* page with one example convolution added.

The Divergent beam page:

Contains all instrument details necessary to model the instrument function using the Fundamental Parameters Approach. This includes the following refinement models:

Parameter name:	Remarks:
Goniometer radii	
Primary Radius	Primary goniometer circle radius [mm]
Secondary Radius	Secondary goniometer circle radius [mm]
Equatorial Convolutions	
Point detector	
Receiving Slit Width	Receiving Slit (= detector slit) width [mm]
FDS Shape, Angle	Fixed Divergence Slit angle [°]
Beam spill, sample length	Sample length [mm] for beam overflow related profile shape correction
Correct Intensity	Beam overflow related intensity correction
VDS Irradiated Length	Irradiated sample length in beam direction for Variable Divergence Slits [mm]
VDS Scale Intensity	1/sin(θ) intensity correction for Variable Divergence Slits
Capillary	
Diameter	Diameter of the capillary
LAC	Linear Absorption Coefficient [1/cm]
Linear PSD	
2Th angular range	Angular range (window size) in [°]
FDS Shape, Angle	Fixed Divergence Slit angle [°]
Beam spill, sample length	Sample length [mm] for beam overflow related profile shape correction
Correct Intensity	Beam overflow related intensity correction
Tube Tails	Tube tails correction (Bergmann, 2000)
Source Width	Width of the tube filament [mm]
Z1	Length of the left tail (negative) [mm]
Z2	Length of the right tail [mm]
Fraction	Fractional height of the tube tails relative to the main beam
Axial Convolutions	
Full Axial Model	Accurate model for describing peak asymmetry
Source Length	Length of the tube focus [mm]
Sample Length	Length of the irradiated sample [mm]
RS Length	Receiving Slit length [mm]
Prim. Soller	Primary Soller slit angle [°]
Sec. Soller	Secondary Soller slit angle [°]
N Beta	Refer to the Technical Reference manual
Finger_et_al	Simplified model for describing peak asymmetry
Simple Axial Model	Simplified model for describing peak asymmetry

Shortcut menu options of the *Divergent beam* page are:

- **Load Instrument Details**
  Loads instrument details from a PAR file
- **Save Instrument Details**
  Saves instrument details to a PAR file
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Additional Convolutions page:

Provides for empirical modelling of instrument functions. Available convolutions include the Hat, Lorentzian, Gaussian, Circles, Exponential, and One_on_X convolutions to be selected from the *Conv. Type* combo box. Predefined angular dependencies are Constant, 1/Cos(Th), Tan(Th), and Sin(2 Th) to be selected from the *2Th Dependence* combo box; alternatively, user-defined angular dependencies can be provided in the form of equations.

Shortcut menu options of the *Additional Convolutions* page are:

- **Load Instrument Details**
  Loads instrument details from a PAR file
- **Save Instrument Details**
  Saves instrument details to a PAR file
- **Add Convolution**
  Adds an additional convolution. Convolution type and angular dependence can be defined using the *Conv. Type* and *2Th Dependence* combo boxes.

Additionally it offers quick access to the following important convolutions:

- **Add Hat 1/cos(Th) dependence**
- **Add Lorentzian 1/cos(Th) dependence**
- **Add Gaussian 1/cos(Th) dependence**
- **Add Hat Tan(Th) dependence**
- **Add Lorentzian Tan(Th) dependence**
- **Add Gaussian Tan(Th) dependence**

Selected additional convolution rows can be deleted using the DEL key.

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.4 Corrections

The Corrections page:

The *Corrections* page (Fig. 4.8) provides the following correction functions.

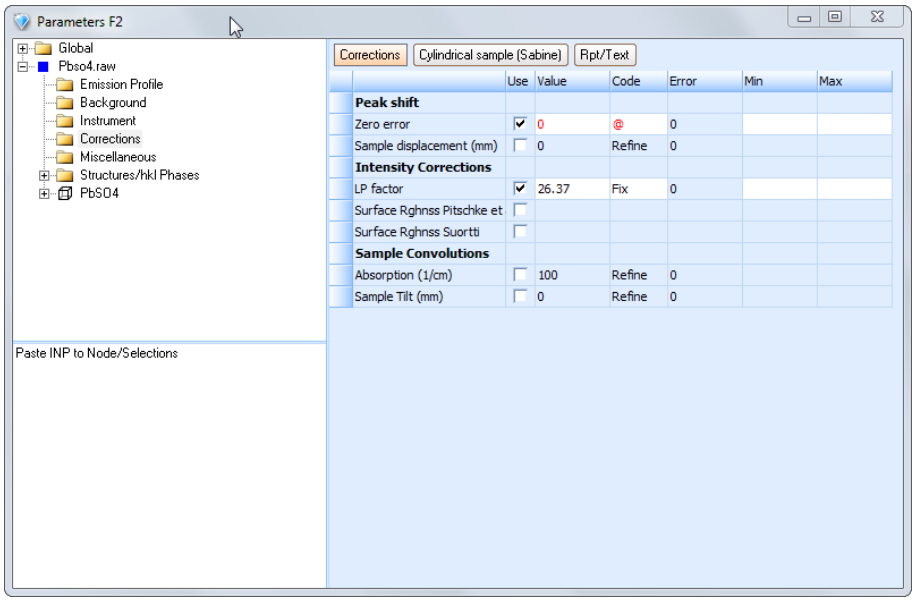


Fig. 4.8: *Corrections* item with its associated data grid showing the *Corrections* page. Note: *Absorption* is a "Use" dependent expand/collapse grid item.

Parameter name:	**Remarks:**
Zero Error	Zero point error in [° 2θ].
Sample Disp.	Sample displacement in [mm].
LP Factor	Lorentz-Polarisation factor using the monochromator angle in [°2θ].
Surface Rghnss Pitschke	Allows surface roughness intensity corrections according to Pitschke et al. (1993)
Surface Rghnss Suortti	Allows surface roughness intensity corrections according to Suortti (1972)
Absorption	Linear absorption coefficient used for adjusting the peak shape [cm^{-1}].
Sample Thickness	Sample thickness in [mm] in the direction of the scattering vector
Scale Intensity	Peak intensity correction for absorption effects
Sample Tilt	Sample tilt in [mm].

The Lorentz-Polarisation factor for unpolarized radiation is 90 (e.g. X-ray diffractometers without any monochromator) and 0 for fully polarized radiation (e.g. synchrotron radiation). Values for most common monochromators (Cu radiation) are:

- Ge : 27.3

- Graphite : 26.4
- Quartz : 26.6

There is no polarization factor for neutron data and thus the angle for Lorentz Polarization should be set to 90; this gives the Lorentz only part.

The shortcut menu of the *Corrections* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Cylindrical Sample (Sabine) page:

The *Cylindrical Sample (Sabine)* page (Fig. 4.9) provides two correction functions related to capillaries.

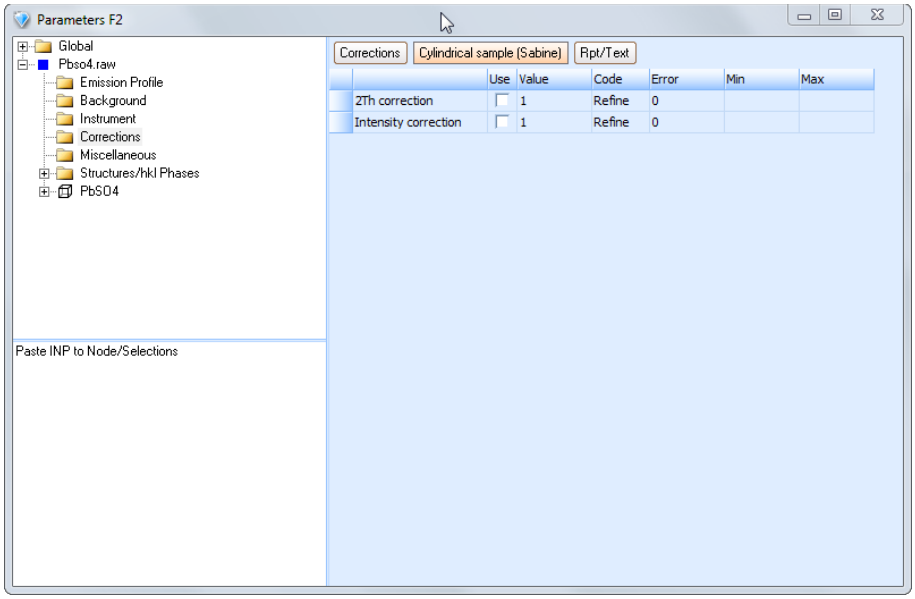


Fig. 4.9: *Corrections* item with its associated data grid showing the *Cylindrical sample (Sabine)* page.

Parameter name:	Remarks:
2Th correction	Allows a 2θ correction for cylindrical samples
Intensity correction	Allows an intensity correction for cylindrical samples

Note: Both corrections should not be used in combination with the Capillary convolution (instrument page, see section 4.2.2.3).

The shortcut menu of the *Corrections* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.5 Miscellaneous

Provides the following options (Fig. 4.10):

Parameter name:	Remarks:
Convolution Steps	An integer corresponding to the number of calculated data points per measured data point. It may be useful to increase this number when numerical instabilities are introduced. This can happen when a particular convolution has a small effect on the profile shape or when the measurement step is large.
Start X, Finish X	Used to limit the refined X range independent on zooming
Fixed WL Neutron	Signals the use of neutron atomic scattering lengths

The *Excl. Regions* page allows for a definition of an unlimited number of excluded regions, which may overlap. Options of the shortcut menu are:

- **Add Excluded Region**
  Adds an excluded region.
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

Selected excluded region rows can be deleted using the DEL key.

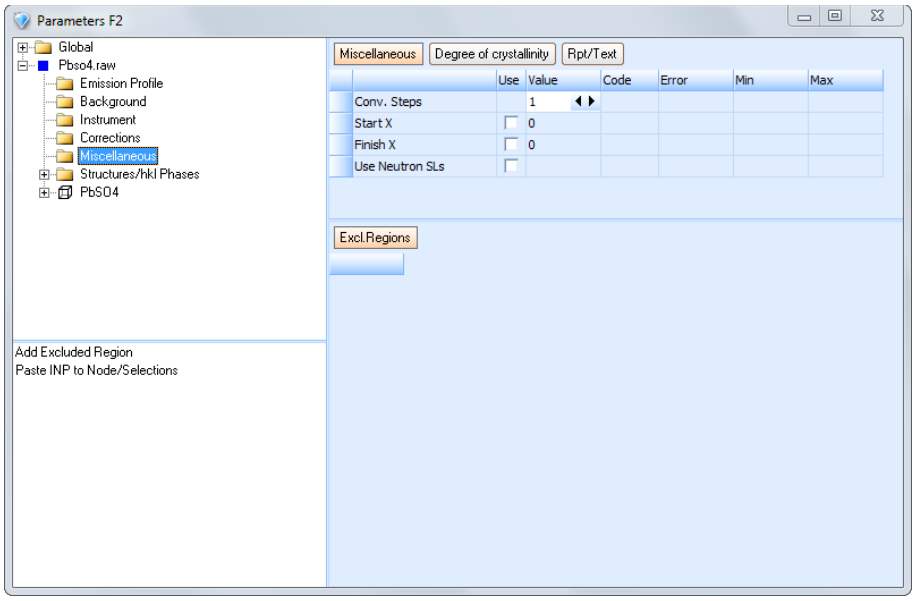


Fig. 4.10: *Miscellaneous* item with its associated data grid.

The *Degree of crystallinity* page allows degree of crystallinity calculations, see section 4.2.2.9.

4.2.2.6 Peak Phase

Peak Phase items provide all parameters required for single line up to Whole Powder Pattern Fitting (Fig. 4.11). The first *Peak Phase* item will be created automatically, if a peak has been inserted manually or if a peak search has been performed. *Peak Phase* items can also be created manually using the shortcut menu, see below. With more than one *Peak Phase* item present, additional peaks, either inserted manually or found by peak search, will be moved to the selected *Peak Phase* item. A new *Peak Phase* item is always created when importing a peak list (d-I values) from a DIF or a UXD file.

Individual peaks of a *Peak Phase* can be of the type FP, PV, PVII, SPV, and SPVII. Furthermore there are several grid pages related to microstructure parameters as well as another *Additional Convolutions* page. For information about the microstructure parameters refer to the Technical Reference manual. The *Additional Convolutions* page provides for empirical modelling of the observed line profile shapes; its options are identical to the *Additional Convolutions* page of the *Instrument* item, see section 4.2.2.3.

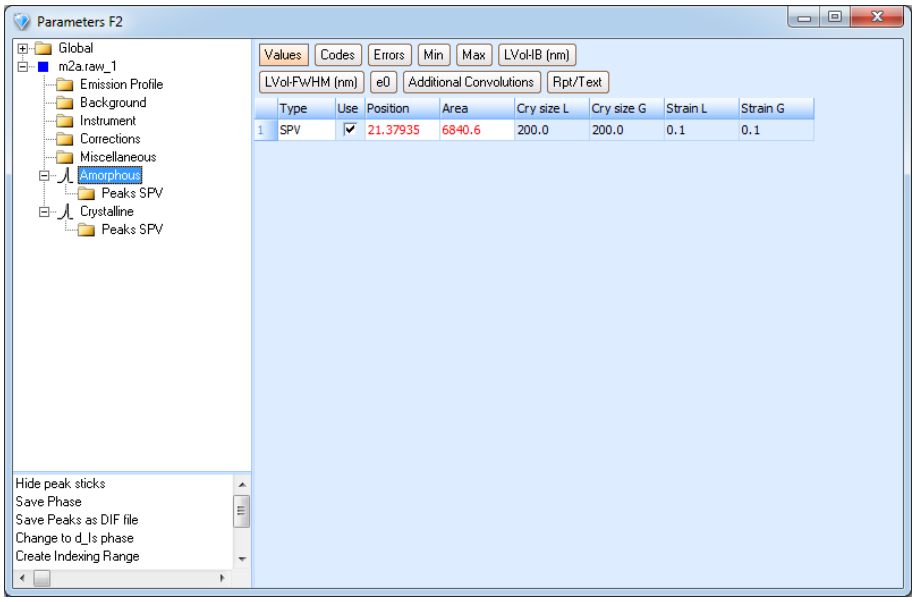


Fig. 4.11: *Peak Phase* items. The associated data grid only shows the profile function specific refinement parameters, which are different for the peak types *FP*, *PV*, *PVII*, *SPV*, and *SPVII*. The peak type can be changed for each peak at any time using the *Type* combo box (refined profile parameters will be lost). The "Use" checkbox enables/disables individual peaks.

A single mouse click (LMB) on a selected *Peak Phase* item allows direct editing of its label.

Shortcut menu options of the *Peak Phase* item are:

- **Hide peak sticks / Show peak sticks**
  Hides or shows peak sticks for individual peak phases
- **Save Phase**
  Saves the selected *Peak Phase* item in INP format

- **Save Peaks as DIF file**
  Saves all peaks of the selected *Peak Phase* item as a d-I list in DIF format
- **Change to d_Is phase / Change to xo_Is phase**
  Switches between display of 2θ and d-values
- **Create Indexing Range**
  Creates a new range for indexing, see section 4.2.3. If peaks / wavelengths are present then they are placed into the indexing range.
- **Create Pawley Range**
  Creates a Pawley range adopting the refinement model as used for profile fitting
- **Delete Peaks Phase**
  Deletes the selected *Peak Phase* item
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.7 hkl Phase

hkl Phase items (Fig. 4.12) provide all parameters required for Whole Powder Pattern Decomposition including both the Pawley and the Le Bail method. The data grid comprises the following pages: *Phase Details*, *Microstructure*, *Peak Type*, *hkl_Is*, and *Additional Convolutions*.

The 3rd level item *Indexing Details* provides for multiple Pawley or Le Bail refinements. It is specific for LSI Indexing and described in section 4.2.3.

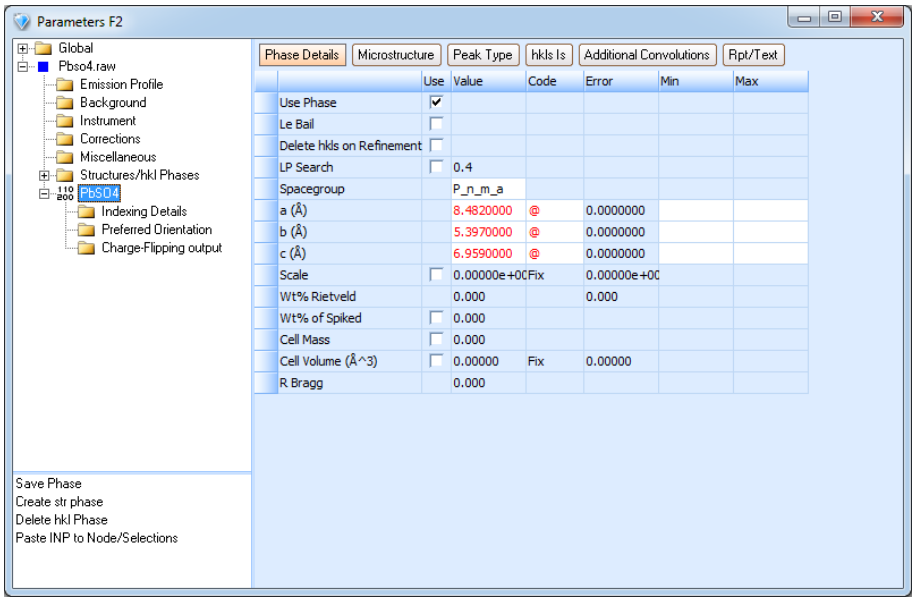


Fig. 4.12: *hkl Phase* item with its associated data grid showing the *Phase Details* page.

The *Phase Details* page offers the following refinement parameters and options:

Parameter name:	Remarks:
Use Phase	Includes or excludes the phase from the refinement
Le Bail	If checked, the Le Bail method will be used for refinement of peak intensities
Delete hkls on Refinement	If checked (default), the current hkls will be replaced by new hkls calculated for the actual spacegroup and data range, everytime a new refinement is started (refined intensities will be lost). This option should be used, if the spacegroup or the data range has been changed.
LP Search	If checked LP-Search indexing will search the correct lattice parameters starting from dummy values. LP-Search will test a single crystal system, which is defined by the space group provided. Typically the space group will correspond to one that is of lowest symmetry with the particular crystal system tested, i.e. for triclinic put space group number "1", for monoclinic put "3", and so forth.
Spacegroup	Space group symbol or space group number. When selecting the field, a drop down button opens a dialog for space group selection.
a, b, c, alpha, beta, gamma	Lattice parameters. Spacegroup depend symmetry constraints are automatically applied including removal of redundant lattice parameters from the grid.
Scale	Scale factor
Wt % Rietveld	Relative phase amount
Wt % of Spiked	Weighed phase amount, if phase is used as a spike, see also section 4.2.2.9
Wt % in Spiked sample	Absolute phase amount in the spiked sample after considering amorphous phase amounts
Wt % in Original sample	Absolute phase amount in the original sample after considering amorphous phase amounts
Cell Mass	Cell mass
Cell Vol	Cell volume
R Bragg	R-Bragg value

The *Microstructure* page (Fig. 4.13) offers the following models for microstructure analysis:

Parameter name:	Remarks:
Cry Size Cry Size L Cry Size G LVol-IB LVol-FWHM	Microstructure parameters related to crystallite size, refer to the Technical Reference manual
Strain Strain L Strain G e0	Microstructure parameters related to strain, refer to the Technical Reference manual
Stephens model	Phenomenological model of anisotropic peak broadening in powder diffraction (Stephens, 1999), refer to the Technical Reference manual

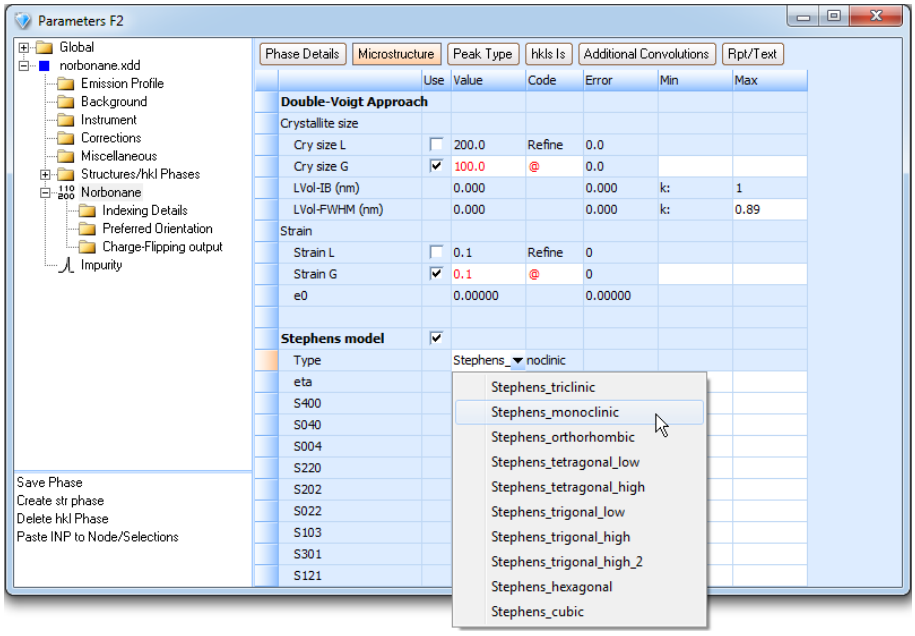


Fig. 4.13: *Microstructure* page.

The *Peak Type* page allows selection of the profile shape function, available functions are *FP*, *PV_Mod*, *PV_TCHZ*, and *PVII*. For information about these profile shape functions and their parameters refer to the Technical Reference manual.

A list of all generated peaks is displayed in the *hkl_Is* page including the following parameters: h, k, l, m (multiplicity), d, 2θ and intensity I. Individual hkls can be excluded from refinement using their "Use" checkbox.

The *Additional Convolutions* page provides for empirical modelling of the observed line profile shapes; its options are identical to the *Additional Convolutions* page of the Instrument item, see section 4.2.2.3.

Note: A single mouse click (LMB) on a selected *hkl Phase* item allows direct editing of its label.

Shortcut menu options of the *hkl Phase* item are:

- **Save Phase**
  Saves the selected hkl Phase item in INP format
- **Create str Phase**
  Creates a Structure item adopting the crystal lattice and microstructure model as used for Pawley / Le Bail fitting
- **Delete hkl Phase**
  Deletes the selected hkl Phase item
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Preferred Orientation item

The *Preferred Orientation* item (Fig. 4.14) offers two models for preferred orientation correction.

1. March-Dollase (March, 1932) preferred orientation correction for up to two directions. hkls provided in the hkl field needs to be separated with spaces as shown in Fig. 4.14.
2. A correction for preferred orientation effects using a spherical harmonics series, its coefficients can be viewed and manipulated in the *Coefficients* page. The Order parameter corresponds to the order of the spherical harmonics series, which must be an even integer ranging from 2 to 8.

The shortcut menu of the *Preferred Orientation* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

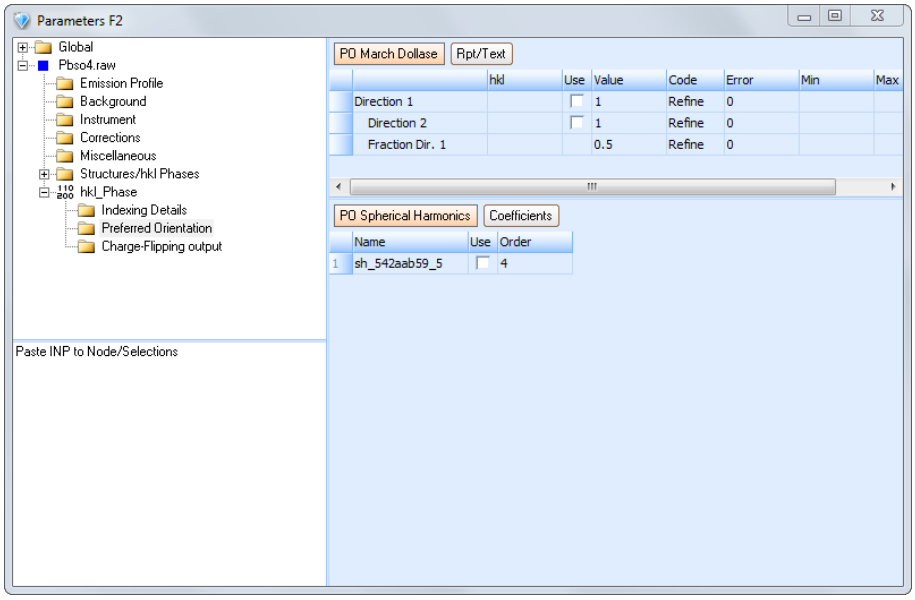


Fig. 4.14: *Preferred Orientation* item

The Charge-Flipping output item

The *Charge-Flipping output* item (Fig. 4.15) allows to create *A matrix files* or *hkl files* (".hkl") for structure determination using Charge Flipping.

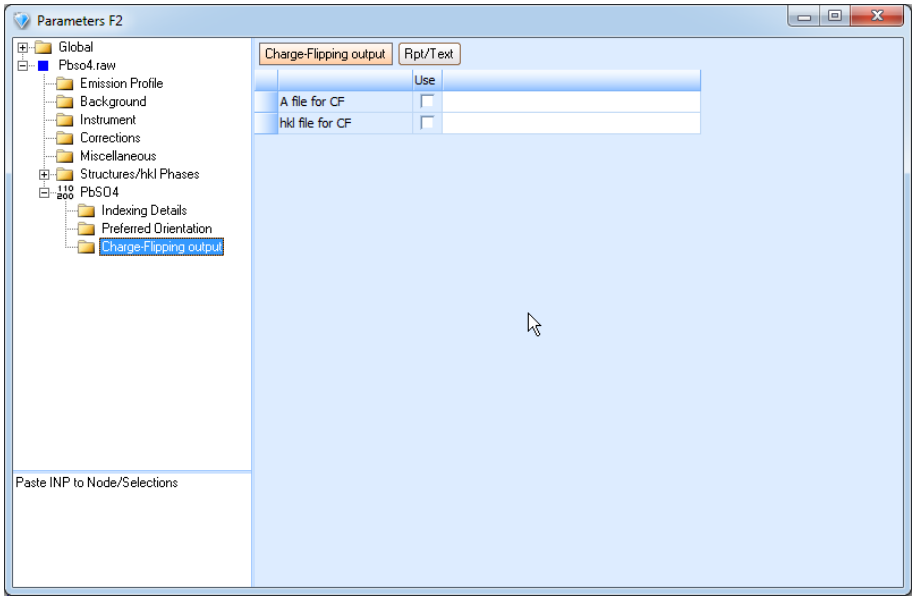


Fig. 4.15: *Charge-Flipping output* item.

4.2.2.8 Structure

Structure items (Fig. 4.17) provide all parameters required for Rietveld structure refinement as well as for quantitative Rietveld analysis. The *Structure* dependent items *Sites*, *PO March-Dollase*, *PO-Spherical Harmonics*, and *Str Output* are described below.

The *Structure* item data grid comprises the following pages: *Structure*, *Microstructure*, *Peak Type*, *hkl_Is*, and *Additional Convolutions*.

The *Structure* page contains the following structure information:

Parameter name:	Remarks:
Use Phase	Includes or excludes the phase from the refinement
Spacegroup	Space group symbol or space group number. When selecting the field, a drop down button opens a dialog for space group selection.
a, b, c, alpha, beta, gamma	Lattice parameters. Spacegroup depend symmetry constraints are automatically applied including removal of redundant lattice parameters from the grid.
Scale	Scale factor
Wt % Rietveld	Relative phase amount
Wt % of Spiked	Weighed phase amount, if phase is used as a spike, see also section 4.2.2.9
Wt % in Spiked sample	Absolute phase amount in the spiked sample after considering amorphous phase amounts
Wt % in Original sample	Absolute phase amount in the original sample after considering amorphous phase amounts
Cell Mass	Cell mass
Cell Vol	Cell volume
Cry Linear Absorption Coeff	Phase linear absorption coefficient (for a packing density of 1)
Cry Density	Phase X-ray density (for a packing density of 1)
R Bragg	R-Bragg value

The *Microstructure* page (Fig. 4.16) offers the following models for microstructure analysis:

Parameter name:	Remarks:
Cry Size Cry Size L Cry Size G LVol-IB LVol-FWHM	Microstructure parameters related to crystallite size, refer to the Technical Reference manual
Strain Strain L Strain G e0	Microstructure parameters related to strain, refer to the Technical Reference manual
Stephens model	Phenomenological model of anisotropic peak broadening in powder diffraction (Stephens, 1999), refer to the Technical Reference manual

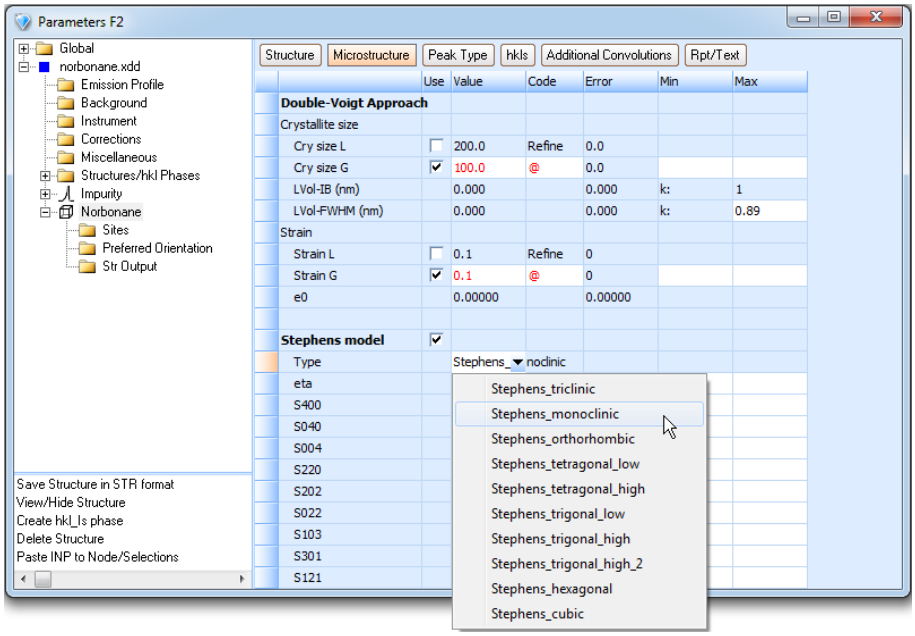


Fig. 4.16: *Microstructure* page.

The *Peak Type* page allows selection of the profile shape function, available functions are *FP*, *PV_Mod*, *PV_TCHZ*, and *PVII*. For information about these profile shape functions and their parameters refer to the Technical Reference manual.

A list of all generated peaks is displayed in the *hkl_Is* page including the following parameters: h, k, l, m (multiplicity), d, 2θ and F2 (structure factor).

The *Additional Convolutions* page provides for empirical modelling of the observed line profile shapes; its options are identical to the *Additional Convolutions* page of the *Instrument* item, see section 4.2.2.3.

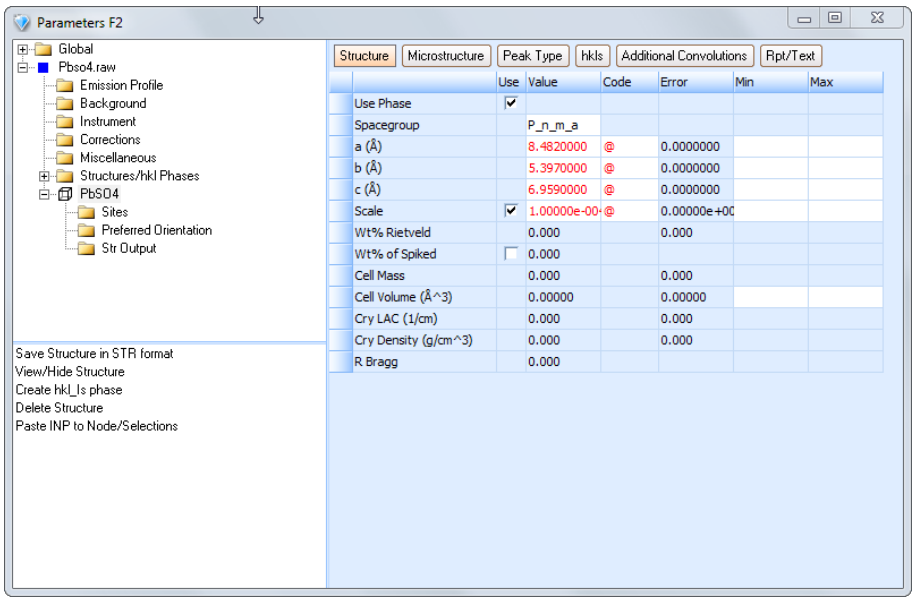


Fig. 4.17: *Structure* item with its associated data grid showing the *Structure* page.

Note: A single mouse click (LMB) on a selected *Structure* item allows direct editing of its label.

Shortcut menu options of the *Structure* item are:

- **Save Structure in STR Format**
  Saves the selected *Structure* item in INP format (*.STR)
- **View/Hide Structure**
  Displays the structure in the *Structure Viewer*, see section 5
- **Create hkl_Is phase**
  Creates a hkl_Is phase adopting the refinement model as used for Rietveld refinement
- **Delete Structure**
  Deletes the selected *Structure* item
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Sites item

The *Sites* item contains the following information concerning atomic sites:

Parameter name:	Remarks:
Site	Customizable site name
x, y, z	Fractional atomic coordinates
Atom	X-ray data: Chemical species (atom/ion) determining the scattering factor Neutron data: Chemical symbol / isotope to determine the scattering length Both can be selected from a drop down list
Occ.	Site occupancy factor; 0 <= Occ. <= 1
Beq.	Isotropic temperature factor

Note: A comparison of atomic positions is performed in the generation of the unique positions with a tolerance in fractional coordinates of 10^{-15}. When entering a fractional coordinate for a special position, such as 1/3, 1/6, etc., it is mandatory to enter a fraction in the form of an equation such as

= 1/3, =1/6, etc. in the *Codes* page,

instead of entering a value with re-occuring digits such as

0.3333..., 0.1666..., etc. in the *Values* page,

as shown in Fig. 4.18. The correct parameter value will be calculated automatically from the equation and displayed in blue color.

Not adhering to this convention may lead to severely wrong refinement results!

Values | Codes | Errors | Min | Max | Rpt/Text

	Site	Np	x	y	z	Atom	Occ.	Beq.
1	Zn	0	0.33333	0.66667	0.00000	Zn+2	1	0.25
2	O	0	0.33333	0.66667	0.38260	O-2	1	0.5

Values | Codes | Errors | Min | Max

	Site	Np	x	y	z	Atom	Occ.	Beq.
1	Zn	0	=1/3	=2/3	@	Zn+2	Fix	@
2	O	0	=1/3	=2/3	@	O-2	Fix	@

Fig. 4.18: *Sites* item data grids showing the *Values* (top) and *Codes* page (bottom) for a structure example requiring atomic coordinates provided in form of equations.

Note: The *Fit Window* drops a warning, if a coordinate is closer than 0.001Å to particular special positions as shown in Fig. 4.19. With increasing structure size there is an increasing probability that a general position may be close to a special position by chance; nevertheless a cross-check is always strongly recommended.

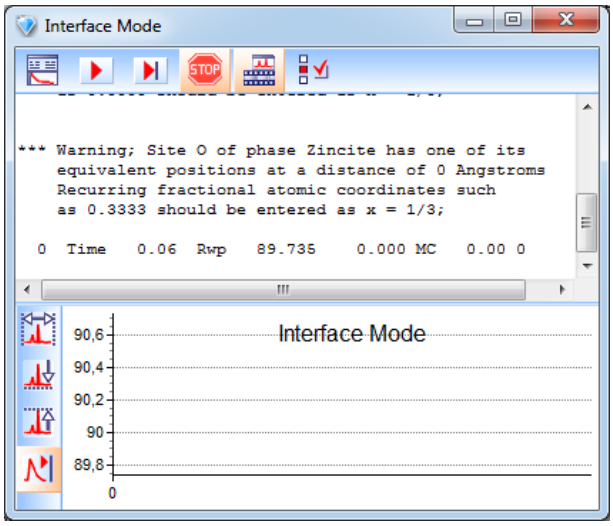


Fig. 4.19: *Fit Window* showing a warning for a coordinate closer than 0.001Å to a 1/3 special position.

Shortcut menu options of the *Sites* item are:

- **Add Site Before Current Site**
  Adds a new site before the current site
- **Add Site at Bottom**
  Adds a new site at the bottom of the list
- **Add Atom at Current Site**
  Adds a new atom at the current site
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Preferred Orientation item

The *Preferred Orientation* item (Fig. 4.20) offers two models for preferred orientation correction.

1. March-Dollase (March, 1932) preferred orientation correction for up to two directions. hkls provided in the hkl field needs to be separated with spaces as shown in Fig. 4.20.
2. A correction for preferred orientation effects using a spherical harmonics series, its coefficients can be viewed and manipulated in the *Coefficients* page. The Order parameter corresponds to the order of the spherical harmonics series, which must be an even integer ranging from 2 to 8.

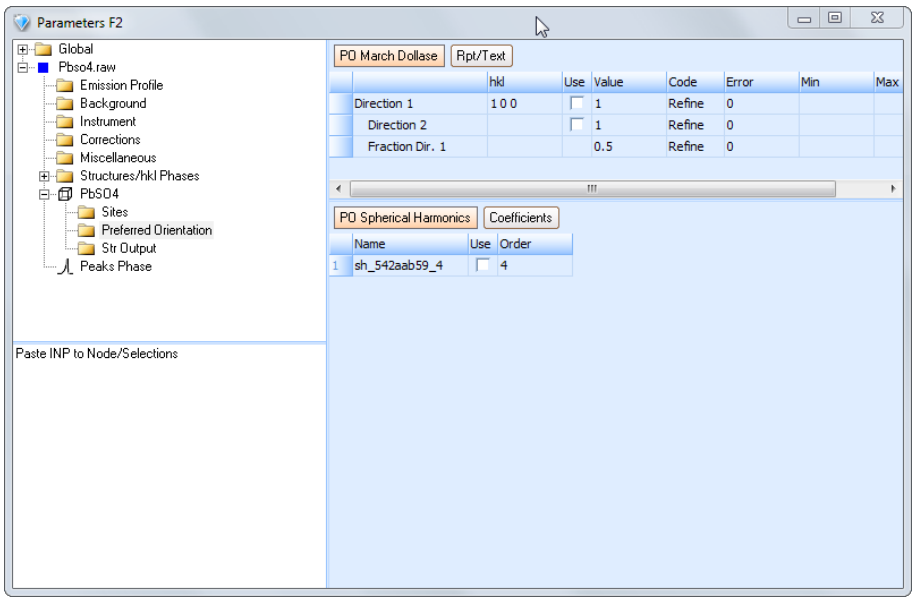


Fig. 4.20: *Preferred Orientation* item

The shortcut menu of the *Preferred Orientation* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The Str Output item

Allows for the output of the following structure details (Fig. 4.21):

- **Generate Bondlengths/Errors**
  If checked, bondlengths and bondangles will be calculated. Additionally errors for bondlengths and bondangles will be provided, if *Calculate Errors* has been turned on in the *Fit* menu or in the *Refinement Options Dialog* (section 3.3.7). The results will be displayed in the *Bondlengths* page.
- **Consider Lattice Parameters in Errors**
  If checked, lattice parameter errors will be considered for calculation of bondlength and bondangle errors

- **Generate CIF Output for Structure**
  If checked, crystal structure details will be generated in CIF format and displayed in the *CIF Structure Output* page
- **Generate FCF Output**
  If checked, structure factor details will be generated in FCF format and displayed in the *FCF Output* page

An example bond lengths output is as follows:

```
Y1:0     O1:0     2.23143
         O2:0     2.23143     88.083
         O3:0     2.28045     109.799     99.928
```

The first line gives the distance between the sites Y1 and O2. The first number in the second line gives the distance between sites Y1 and O2. The third number of 88.083 gives the angle between the vectors Y1 to O1 and Y1 to O2. The first number on the third line contains the distance between sites Y1 and O3. The second number in the third line contains the angle between the vectors Y1 to O3 and Y1 to O2. The third number in line three contains the angle between the vectors Y1 to O3 and Y1 to O1. Thus bond lengths correspond to the first number in each line and bond angles start from the second number. The numbers after the site name and after the ":" character corresponds to the site equivalent position as found in the *.SG space group files found in the SG directory

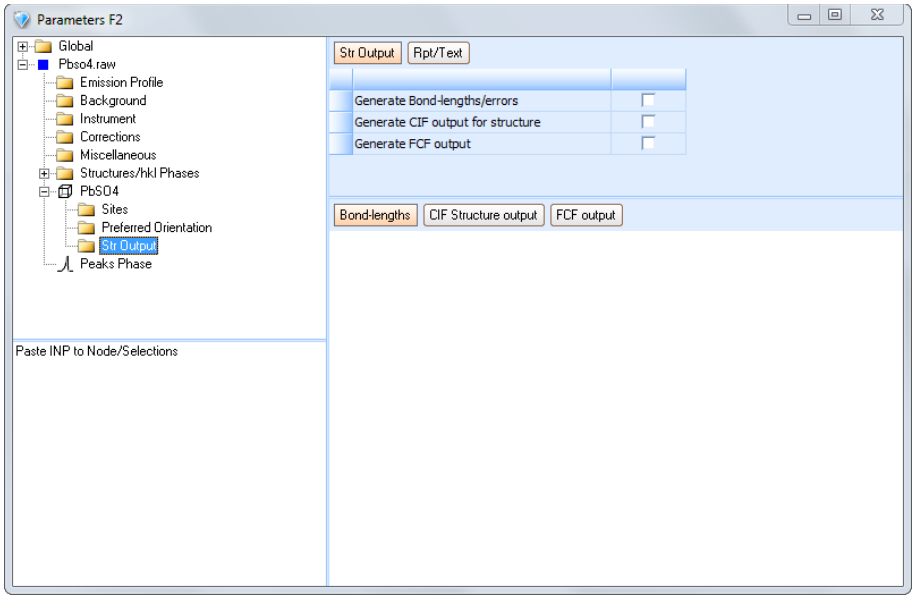


Fig. 4.21: *Str Output* item with its associated data grid.

The shortcut menu of the *Str Output* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

4.2.2.9 Structures / hkl Phases

Allows viewing and manipulating of refinement parameters and options common to all structures and hkl phases (Fig. 4.22).

The data grid associated to the *Structures / hkl Phases* item comprises the following pages: The *Scale* page provides an overview about all loaded structures and hkl phases, which can be included or excluded from the refinement using the "Use" checkbox in front of the *Phase Name* field (see Fig. 4.22). Refineable microstructure parameters can be accessed in the *Cry Size L*, *Cry Size G*, *Strain L*, and *Strain G* pages (for information about the microstructure parameters refer to the Technical Reference manual).

3rd level items are:

- **Internal Standard Method**
  See below
- **External Standard Method**
  See below
- **SG Lattice Parameters**
  Displays lattice parameters for all phases included in the refinement
- **Brindley Correction (Spherical Particles)**
  Corrects microabsorption effects for spherical particles (Brindley, 1945)
- **Display**
  Allows customization of display properties including colors, data point size and line width for each range

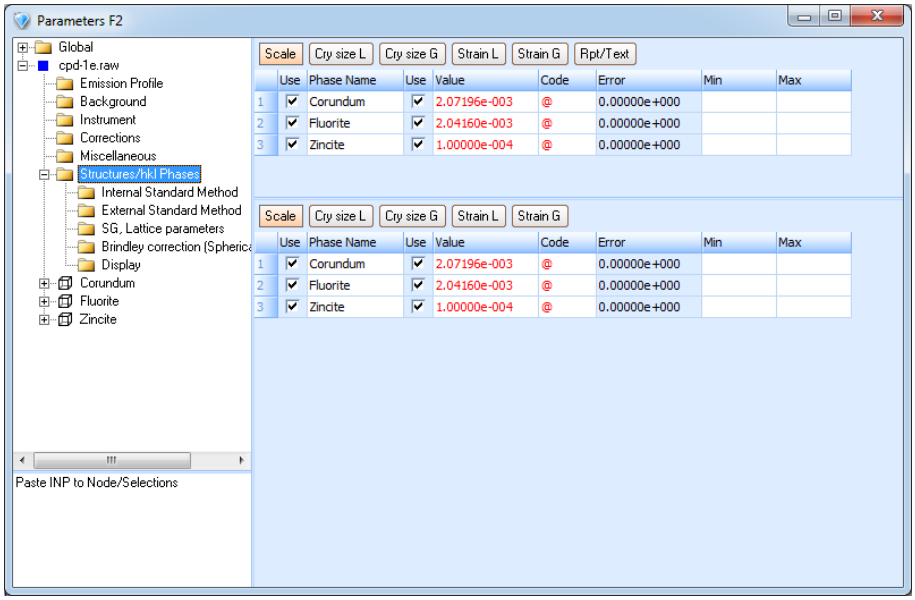


Fig. 4.22: *Structures / hkl Phases* item with its associated datagrid.

The shortcut menu of the *Structures / hkl Phases* item offers the following option:

- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The *Internal Standard Method* item data grid comprises the following pages: *Values*, *Elemental Composition*, and *Degree of crystallinity*.

The Values page

The *Values* page displays quantitative phase amounts for all phases included in the refinement, see Fig. 4.23.

By adding a known weight of an internal standard to the sample (spiking), the amount of the amorphous phase may be measured directly. Furthermore absolute weight fractions will be obtained for all phases.

A single phase can be declared as a spike phase by checking the "Use" checkbox and providing its weighed phase amount (*Wt% of Spike*). Absolute weight percents for all crystalline phases and the amorphous compound are then calculated for both the spiked and the original sample (*Wt% in Spiked sample* and *Wt% in Original sample*, respectively).

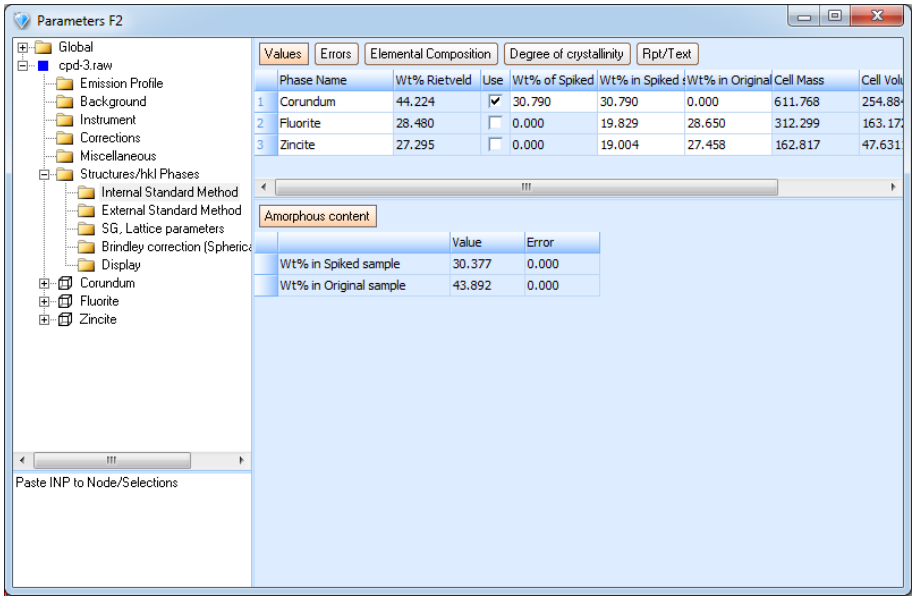


Fig. 4.23: *Quantitative* item showing the *Values* page with its associated datagrid.

Mass absorption coefficients are always calculated and shown for all phases and allow identification of potential microabsorption problems.

The Elemental Composition page

Reports the total elemental composition over all str phases (Fig. 4.24).

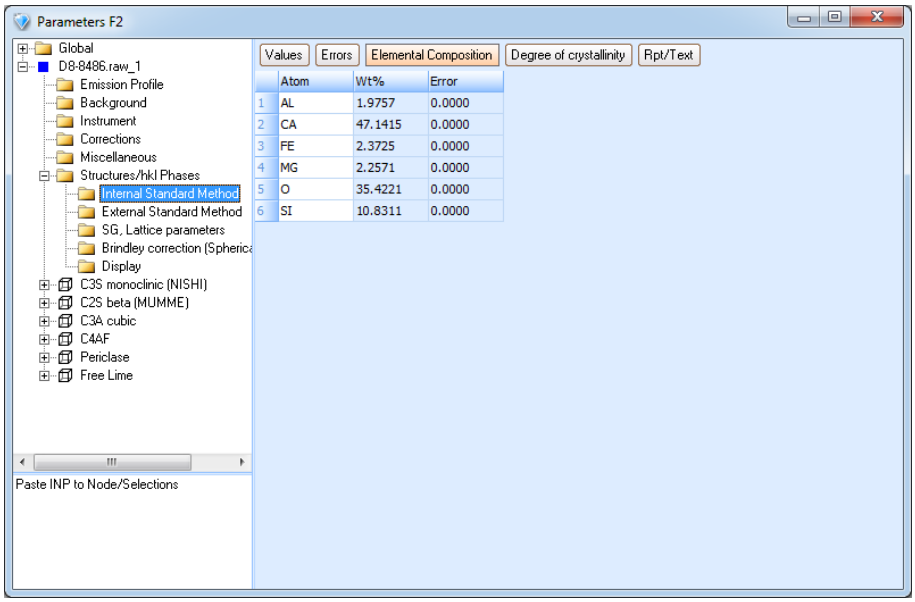


Fig. 4.24: *Elemental Composition* page.

The Degree of crystallinity page

Allows the calculation of the degree of crystallinity of a sample on request, see Fig. 4.25. Note, that degree of crystallinity calculations can be performed using *Peak Phases*, *hkl Phases* and *Structures* in any combination; the number of which used for modeling both crystalline and amorphous contributions is not limited.

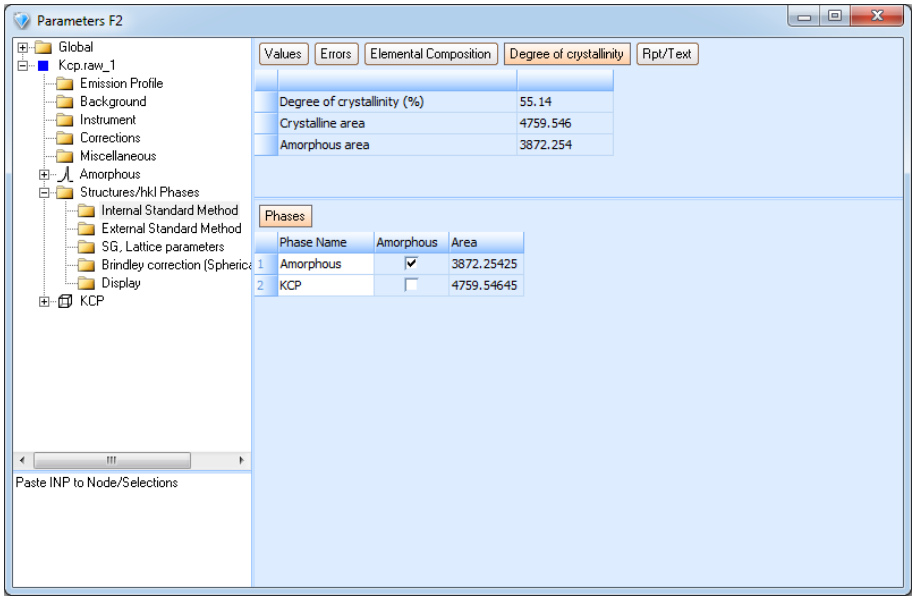
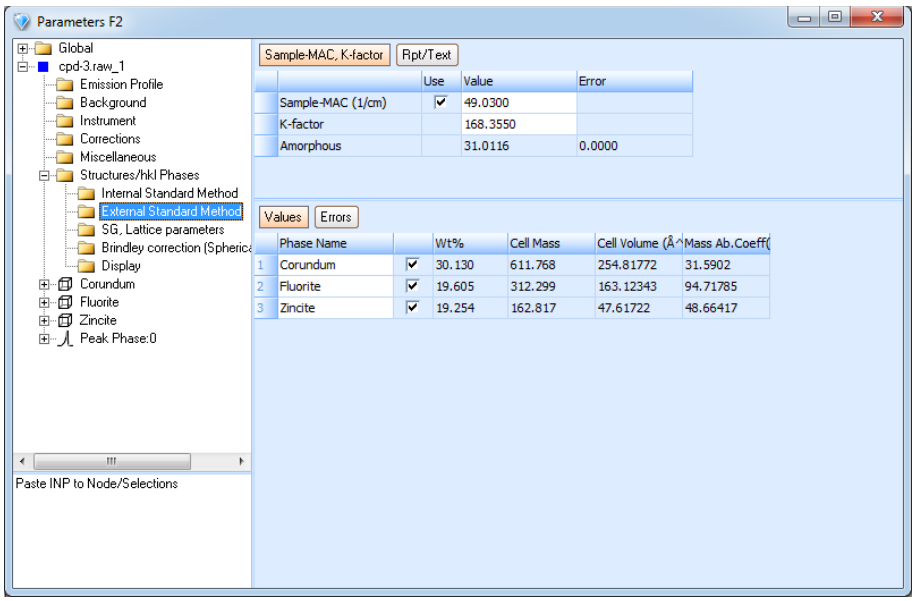


Fig. 4.25: *Quantitative* item showing the *Degree of crystallinity* page with its associated datagrid.

The *External Standard Method* item data grid allows application of the External Standard Method for quantitative phases analysis (O'Connor and Raven, 1988).

Similar to the Internal Standard Method it puts the determined crystalline components on an absolute scale and derives the amorphous content by difference. Required information comprises a normalization constant K and the mass absorption coefficient (*Sample-MAC*) of the entire sample. For details refer to the Technical Reference manual.



4.2.3 Indexing

Indexing provides access to all LSI-Index relevant parameters and options in GUI Mode. The *Indexing* range data grid (Fig. 4.26) comprises the following pages: *Indexing*, *Data* and *Solutions*.

The *Indexing* page offers the following parameters and options:

Parameter name:	Remarks:
Wavelength [Å]	Wavelength used for indexing
Zero error	Includes a zero error calculation
Max zero error in 2Th	Excludes solutions with zero errors larger than the given value
Max 2Th error for UNI	Lines with a 2θ error larger than the given value will be considered as unindexed (UNI)
Max ratio Nc/No	Determines the maximum ratio of the number of calculated to observed lines
Max number solutions	Determines the maximum number of solutions kept
Try space groups	Defines space group(s) to be used for indexing
Set x0 from 2Th	Defines x0 in the reciprocal lattice equation, see the index_x0 keyword in the Technical Reference manual.
Bravais lattices to include	Defines Bravais lattices to be used for indexing

Note that indexing can be performed on both selected space groups and Bravais lattices simultaneously.

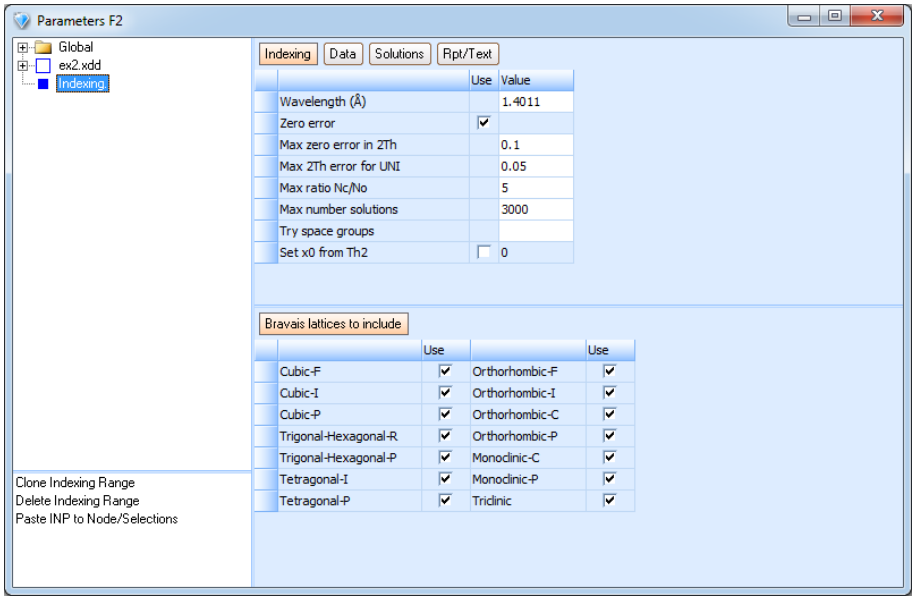
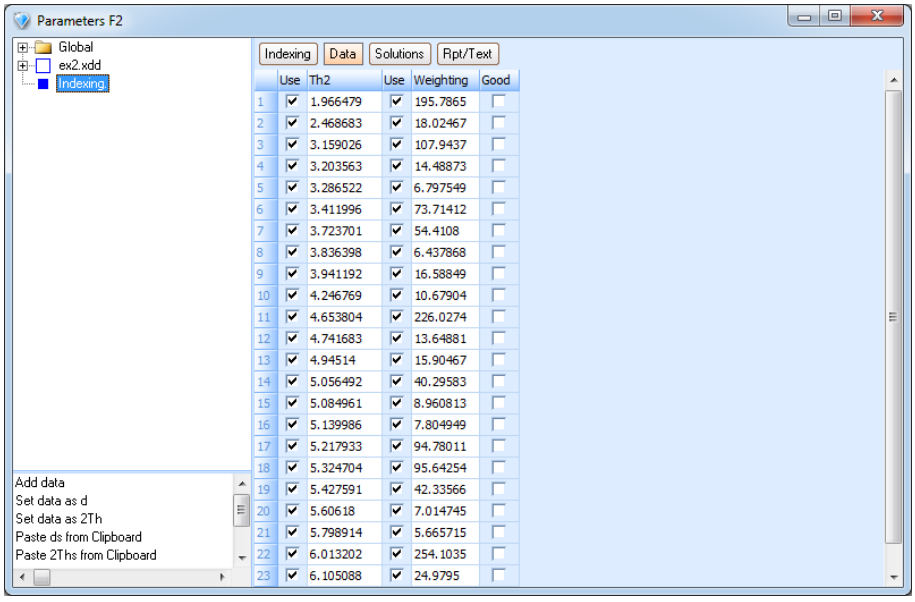


Fig. 4.26: *Indexing* range with its associated data grid showing the *Indexing* page.

Shortcut menu options of the Indexing page are:

- **Clone Indexing Range**
  Clones the selected indexing range
- **Delete Indexing Range**
  Deletes the selected indexing range
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The *Data* page contains the d or 2θ values to be used for indexing (Fig. 4.27). The "Use" parameter allows including or excluding individual lines from calculations. Optionally each line can be weighted, and typically observed line intensities serve as weights. The "Good" parameter, which can be used only once, indicates that the corresponding d-spacing is not an impurity line. A single use of "Good" on a high d-spacing decreases the number of possible solutions and hence speeds up the indexing process.



	Use	Th2	Use	Weighting	Good
1	☑	1.966479	☑	195.7865	☐
2	☑	2.468683	☑	18.02467	☐
3	☑	3.159026	☑	107.9437	☐
4	☑	3.203563	☑	14.48873	☐
5	☑	3.286522	☑	6.797549	☐
6	☑	3.411996	☑	73.71412	☐
7	☑	3.723701	☑	54.4108	☐
8	☑	3.836398	☑	6.437868	☐
9	☑	3.941192	☑	16.58849	☐
10	☑	4.246769	☑	10.67904	☐
11	☑	4.653804	☑	226.0274	☐
12	☑	4.741683	☑	13.64881	☐
13	☑	4.94514	☑	15.90467	☐
14	☑	5.056492	☑	40.29583	☐
15	☑	5.084961	☑	8.960813	☐
16	☑	5.139986	☑	7.804949	☐
17	☑	5.217933	☑	94.78011	☐
18	☑	5.324704	☑	95.64254	☐
19	☑	5.427591	☑	42.33566	☐
20	☑	5.60618	☑	7.014745	☐
21	☑	5.798914	☑	5.665715	☐
22	☑	6.013202	☑	254.1035	☐
23	☑	6.105088	☑	24.9795	☐

Fig. 4.27: *Indexing* range with its associated data grid showing the *Data* page.

Shortcut menu options of the *Data* page are:

- **Add data**
  Adds a new line at the bottom of the list
- **Set data as d**
  Defines given peak positions as d-values
- **Set data as 2Th**
  Defines given peak positions as 2θ-values
- **Paste ds from Clipboard**
  Pastes a list of peak positions from the clipboard and defines them as d-values
- **Paste 2Ths from Clipboard**
  Pastes a list of peak positions from the clipboard and defines them as 2θ-values
- **Paste d/Is from Clipboard**
  Pastes a list of peak positions vs. intensities from the clipboard. Defines peak positions as 2θ-values and intensities as weights.
- **Paste 2Th/Is from Clipboard**
  Pastes a list of peak positions vs. intensities from the clipboard. Defines peak positions as 2θ-values and intensities as weights.
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

The *Solutions* page contains the results of the current indexing run. For each solution the following information is listed: space group proposal (Sg), status (Sts), number of unindexed lines (UNI), volume (Vol), goodness-of-fit (Gof), zero point error (Zero) and lattice parameters. Furthermore a goodness-of-fit versus volume plot as well as detailed information about the selected solution are available (Fig. 4.28 and Fig. 4.29).

The possible values and meanings of status (Sts) are :

- Status 1: Weighting applied as defined in Coelho (2003)
- Status 2: Zero error attempt applied
- Status 3: Zero error attempt successful and impurity lines removal attempt successful
- Status 4: Impurity lines removal attempt successful

In the *Scan Window*, for the selected solution a stick pattern is overlaid with the observed line positions (dashed red lines indicate theoretical peak positions not matched with an observed line, dashed blue lines indicate unindexed observed peaks). The associated powder pattern can be overlaid as well (Fig. 4.30).

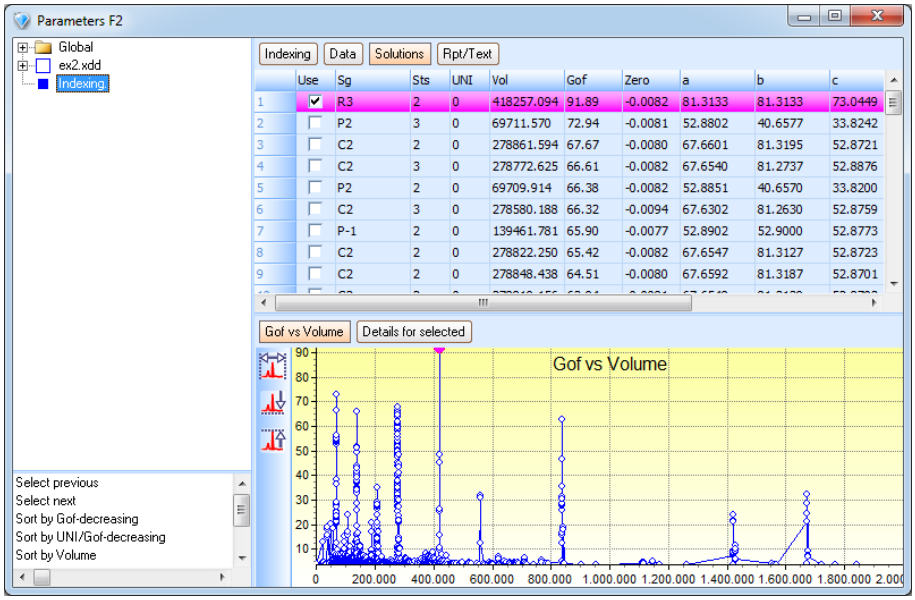


Fig. 4.28: *Indexing* range with its associated data grid showing the *Solutions* page and the goodness-of-fit (Gof) versus volume plot.

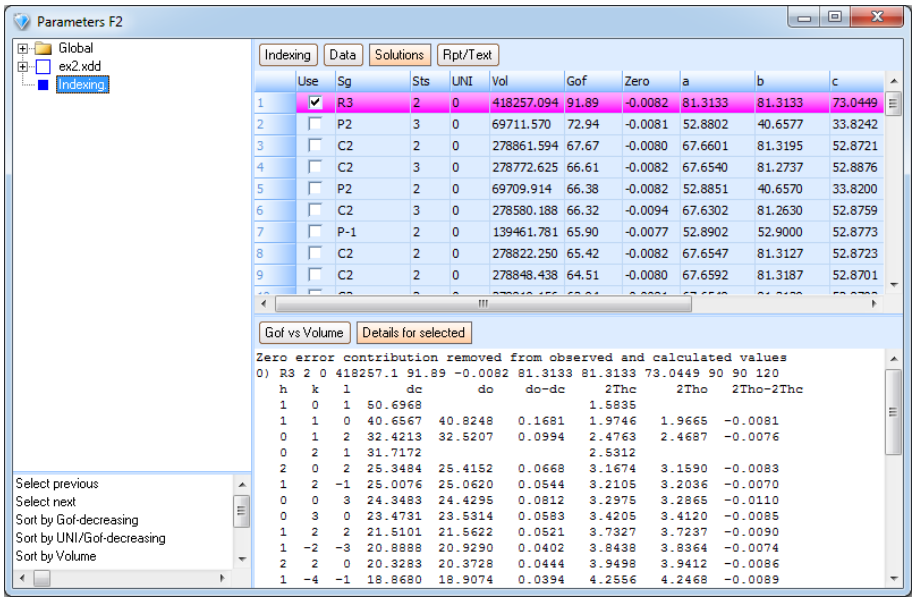


Fig. 4.29: *Indexing* range with its associated data grid showing the *Solutions* page and detailed results for the selected solution.

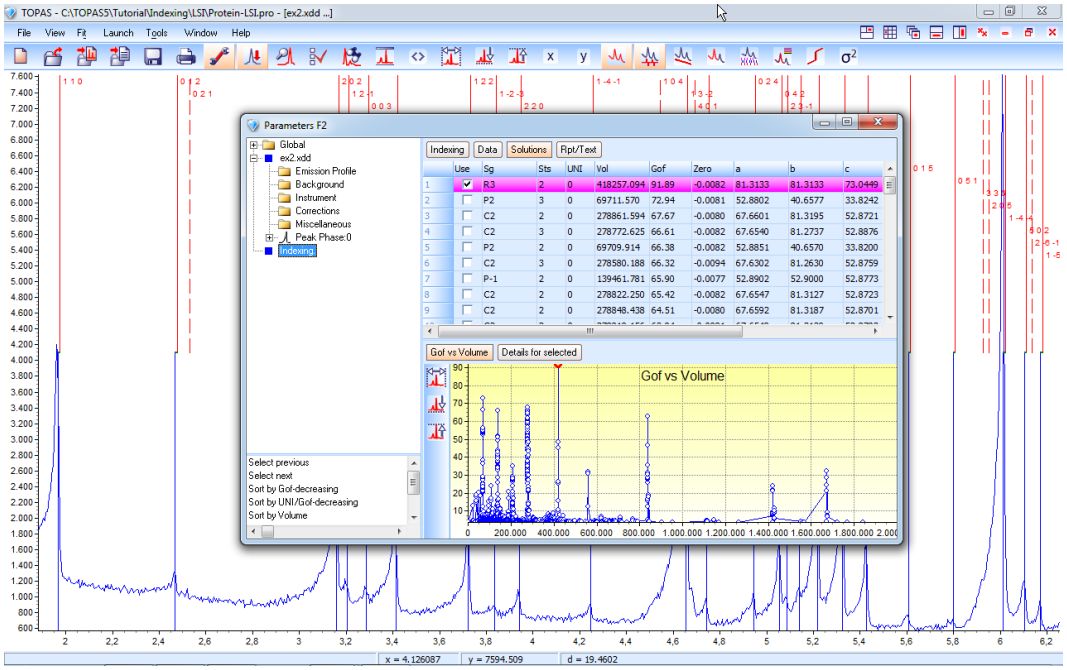


Fig. 4.30: *Scan Window* showing a stick pattern for the selected solution overlaid with the observed line positions and the associated powder pattern.

Shortcut menu options of the Results page are:

- **Select previous**
  Selects the previous solution
- **Select next**
  Selects the next solution
- **Sort by Gof-decreasing**
  Sorts the indexing results by decreasing goodness-of-fit
- **Sort by UNI/Gof-decreasing**
  Sorts the indexing results by decreasing number of unindexed lines / goodness-of-fit
- **Sort by Volume**
  Sorts the indexing results by volume
- **Sort by selected columns**
  Sorts by the selected columns
- **Save to NDX file**
  Saves the indexing results into an NDX file
- **Paste INP to Node/Selections**
  Supports pasting of information in INP format from the clipboard

Multiple Pawley / Le Bail refinements

hkl Phase items provide all parameters required for whole powder pattern decomposition including both the Pawley and the Le Bail method. The dependent *Indexing Details* item allows fully automatic Pawley or Le Bail refinement of all selected solutions of an indexing run (Fig. 4.31).

The *Indexing Details* page is similar to the *Results* page of the *Indexing* range and contains the same information; additionally R_{WP} is obtained after Pawley or Le Bail refinements. The "Ref" checkbox is used to flag solutions to be refined, and is cleared automatically after refinement; repeated refinements are possible.

For multiple Pawley / Le Bail refinements it is not necessary to provide any space group and lattice parameter information in the *Phase Details* page of the *hkl Phase* item; these fields are automatically completed. For each selected solution R_{WP}, the refined zero point error as well as refined lattice parameters are provided after termination of refinement.

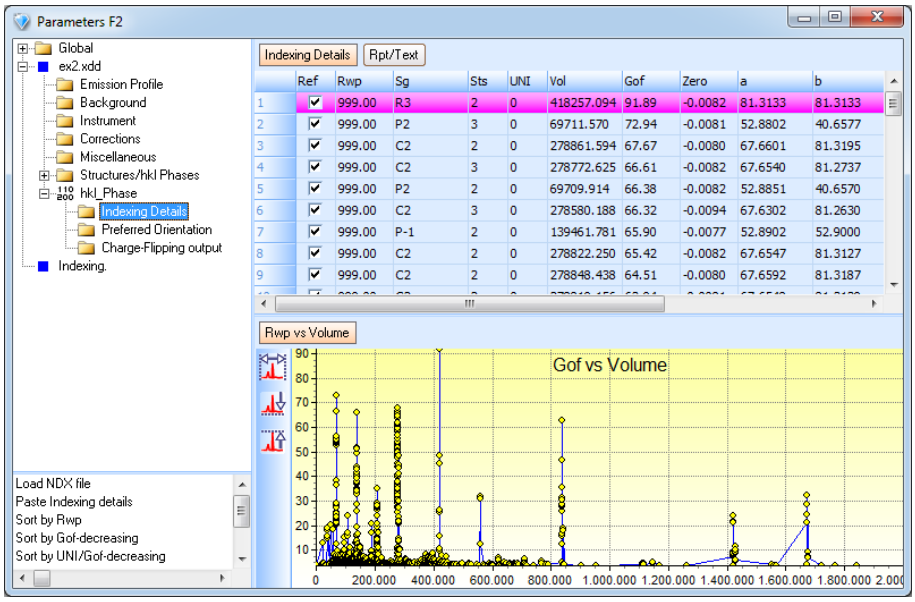


Fig. 4.31: *Indexing Details* page showing a multiple Pawley refinement of selected indexing solutions. The solution highlighted by a blue bar is currently being refined on. For solutions not refined yet R_{WP} is displayed as 999.

Shortcut menu options of the *Indexing Details* page are:

- **Load NDX file**
  Loads previously saved indexing results
- **Paste Indexing details**
  Pastes indexing details from clipboard
- **Sort by Rwp**
  Sorts the indexing results by R_{WP}
- **Sort by Gof-decreasing**
  Sorts the indexing results by decreasing goodness-of-fit

- **Sort by UNI/Gof-decreasing**
  Sorts the indexing results by decreasing number of unindexed lines / goodness-of-fit
- **Sort by Volume**
  Sorts the indexing results by volume

4.2.4 Charge Flipping

The *Charge Flipping* range data grid comprises the following pages: *Charge Flipping*, *Atoms in Unitcell* and *Pick/Omit Atoms* (Fig. 4.32). The respective parameters and options are discussed in the Technical Reference and Tutorial manuals.

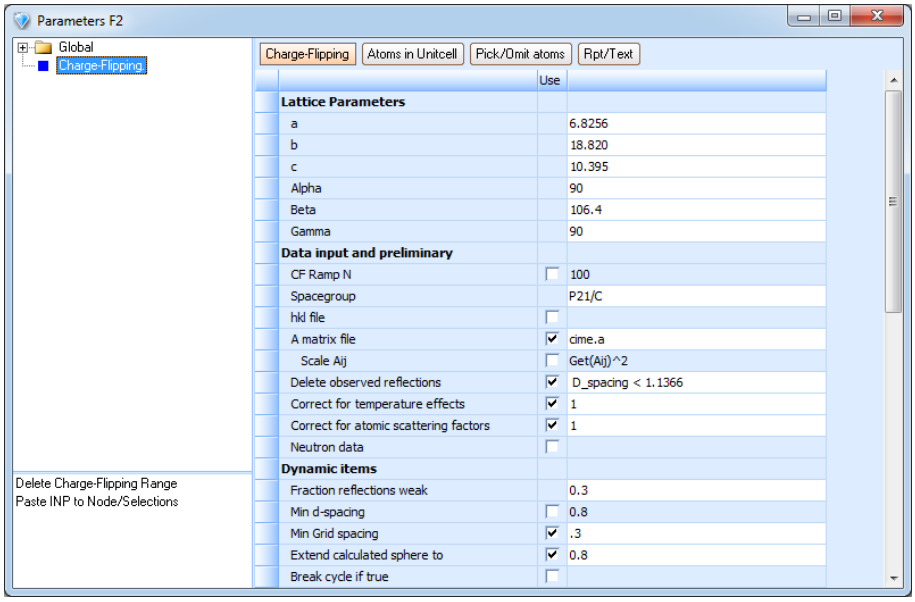


Fig. 4.32: *Charge Flipping* range data grid.

4.3 Printing and reporting

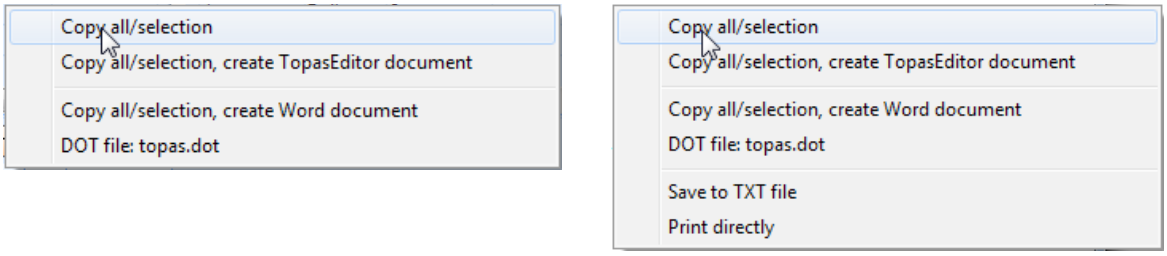
Any data grid information can be copied to the clipboard or transferred to the TopasEditor or Microsoft Word (if available) using the shortcut menu of the data grid shown in Fig. 4.33a. Available options are:

- **Copy all/selection**
  Copies the full page or the selection to the clipboard
- **Copy all/selection, create TopasEditor document**
  Launches the TopasEditor and transfers the full page or the selection into a new document in RTF format
- **Copy all/selection, create Word document**
  Launches Microsoft Word (if available) and transfers the full page or the selection into a new document in DOC format based on the currently defined document template (default is TOPAS.DOT)
- **DOT file: topas.dot**
  Allows to define a document template file for Microsoft Word (if available). This feature allows for the creation of customized reports with, for example, user-defined headers, footers, page numbering and more.

Note: Topas.dot does not contain macros, however to use DOT files containing macros the security level of Microsoft Word for opening files must be set to either "low" (not recommended) or "medium"; if set to "high" any macros will be automatically disabled. For more information please refer to the Microsoft Word user's manual.

Additionally, any contents of text fields including the *Text* and *Report Format* page as well as the *Bondlengths*, *CIF Str Output*, and *FCF Str Output* pages can be saved as plain ASCII files or printed on the default printer (Fig. 4.33b):

- **Save to TXT file**
  Saves the full text field or the selection to a plain ASCII file
- **Print directly**
  Sends the full text field or the selection to the default printer



a) b)

Fig. 4.33: Shortcut menu of the data grid showing available options for (a) grid pages and (b) text fields.

5 THE STRUCTURE VIEWER

The Structure Viewer allows to view crystal structures (Fig. 5.1) and 3D electron densities including atom picking (Fig. 5.2). As a refinement continues the structure viewer is updated providing animated refinement in 3D.

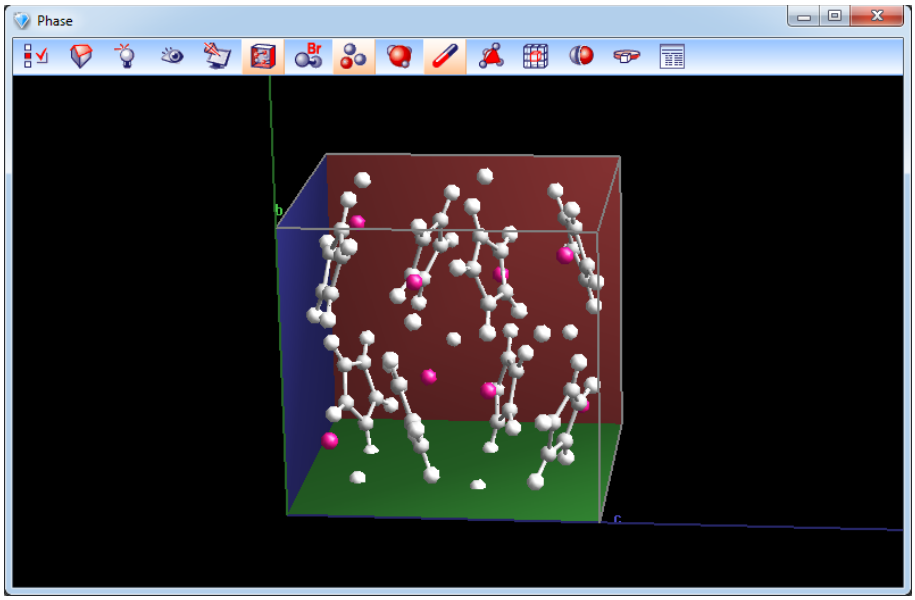


Fig. 5.1: *Structure Viewer* window with crystal structure (Rietveld refinement).

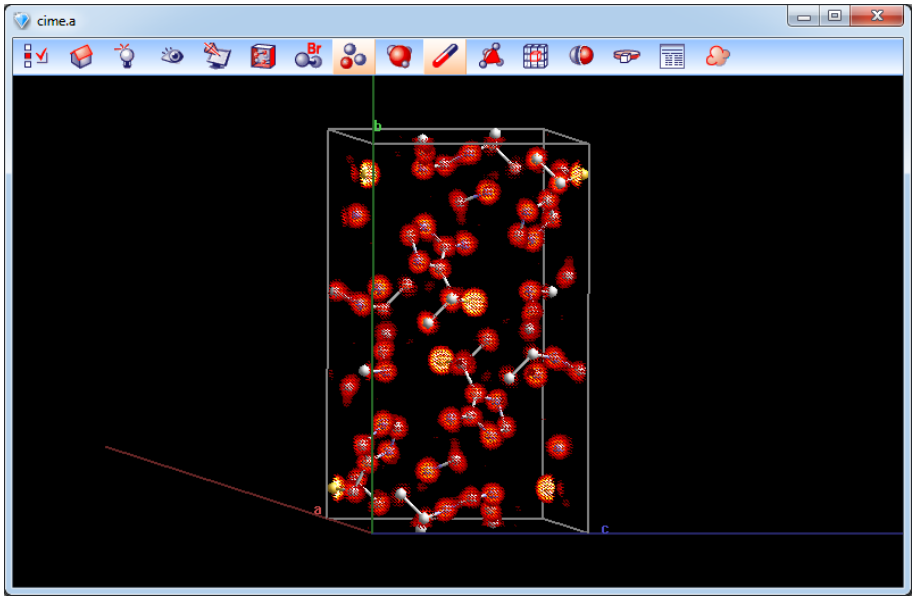


Fig. 5.2: *Structure Viewer* window displaying a 3D electron density map, obtained from Charge Flipping, including automatic atom picking results.

In GUI Mode the *Structure Viewer* can be opened using the shortcut menu item *View/Hide Structure* of the *Structure* item, see section 4.2.2.8. In Launch Mode the structure dependent keyword *view_structure* can be used, see the Technical Reference manual.

Structures / 3D electron densities can be freely rotated by pressing the LMB and dragging the mouse. Zooming is performed by pressing the RMB and dragging the mouse.

To rotate a structure / 3D electron density around an axis perpendicular to the computer screen, press both the Shift key and the LMB and the drag the mouse. The sensitivity of rotation decreases as the mouse position moves away from the center of the window and allows precise positioning of objects.

The *Structure Viewer* comprises the following icons and actions:

Icon:	Result:
	Switches between *Structure Viewer* and *Rigid Body Editor* windows
	Displays in perspective or orthogonal view
	Displays the lighting options
	Displays viewing options incl. rotation and translation dialogs
	Wiggles the view about an axis perpendicular to the computer screen
	Displays / hides the unit cell walls
	Displays / hides atom names
	Displays / hides balls
	Displays atoms in their expected size
	Displays / hides sticks connecting atoms
	Displays / hides polygons
	Display multiple unit cells
	Clip atoms appearing outside of the unit cell
	Display clipping planes and clipping dialog
	*Temporary Output* window. Displays selected atoms in z-matrix format
	*Cloud Options* dialog. Displays viewing options for the 3D display of electron densities and offers atom picking capabilities with or without symmetry consideration

Clicking the RMB anywhere in the *Structure Viewer* will open a shortcut menu offering access to the following features:

- **Set rotation position at geometric center**
- **Set rotation position at atom under mouse**
- **Set rotation position at eye**
- **Keep rotation position at center of view**
- **Show connecting atoms for selection**
- **Hide selection**
- **New polygon connecting selected atoms**
- **Show connecting atoms**
- **Hide connecting atoms**
- **Isolate atom / polyhedra under mouse**

6 THE RIGID BODY EDITOR

The *Rigid Body Editor* (Fig. 6.1) provides for creation, viewing and editing of rigid bodies and structures. It can load and save files in INP format (*.INP, *.STR, *.RGD), furthermore cloud files (*.CLD) can be loaded; for details refer to the Technical Reference manual.

The top *Browser* area can be displayed / hidden using the "Load/Hide" menu item, the *Preview* area offers a preview for the currently selected file (read only).

One or more files can be loaded into the *Main View* area (LMB double-click on file name). The associated rigid body / structure definition is displayed in the *Editor* area at the left of the *Main View* area. The "Loaded" page in the "Options" box provides an overview about all loaded files; selected items can be unloaded using the "Del" key.

The *Editor* allows creation of new / modification of loaded rigid bodies or structures. The *Main View* is updated on using the "Update" menu item or by pressing Alt+F1. New rigid bodies or modifications can be saved using the "Save" and "Save As" options.

The "First Guess Z-matrix" menu item can be used to convert a rigid body definition given in Cartesian or fractional coordinates into a more useful internal coordinate description; a Z-matrix representation of this description is provided in a temporary output window.

The "Add torsion angle" menu item allows to introduce a refineable torsion angle along two selected atoms.

The *Main View* offers most of the features and options available in the *Structure Viewer*, see section 5.

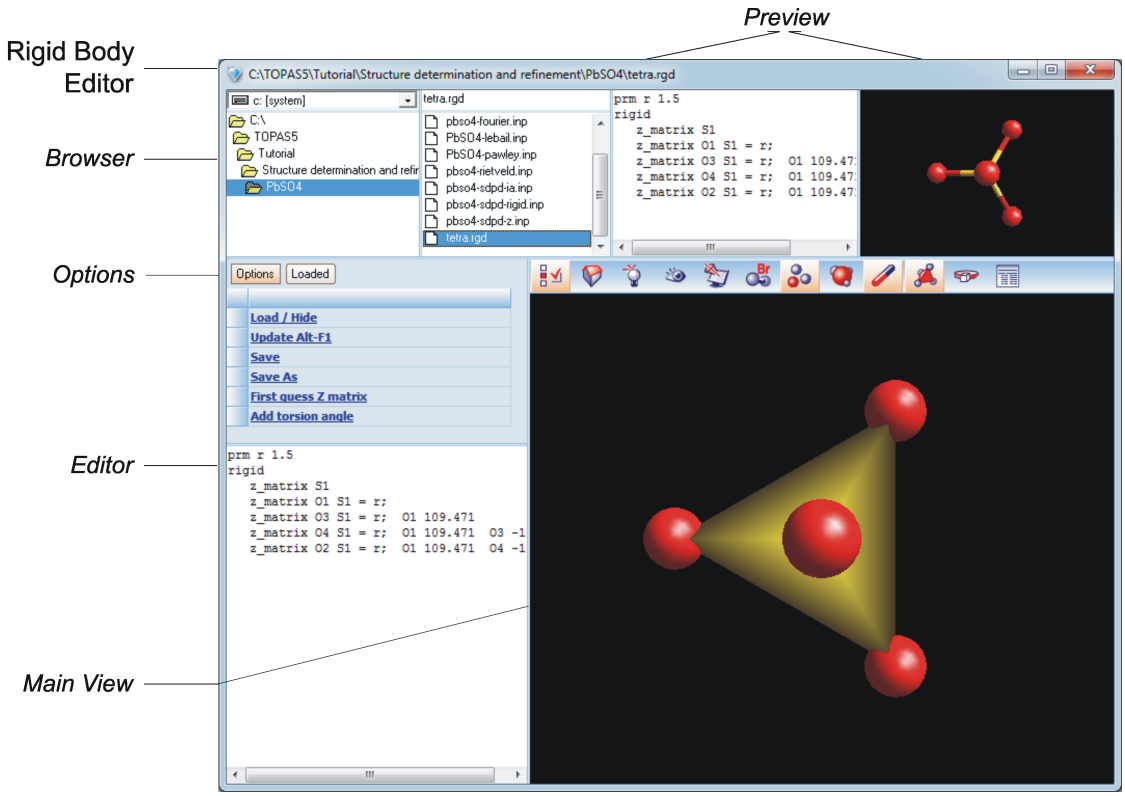


Fig. 6.1: *Rigid Body Editor*.

7 OPERATION IN GUI AND LAUNCH MODE

The GUI Mode is active by default. Launch Mode becomes active if an input file (*.INP) is set by the menu command *Launch - Set INP File*.

Input files contain all information for controlling TOPAS in Launch Mode and can be modified by a text editor (for details please refer to the Technical Reference manual). Notepad is set as the default editor, user specified can be made permanently active by selecting *Launch - Editor*. Editing can be performed by selecting *Launch - Edit INP File*. In Launch mode refinement results are always written to an output file (*.OUT) which has the same format as the INP file. The output file can be inspected by selecting *Launch - Edit OUT File*.

As in Launch Mode the refinement will be completely controlled by the input file, the following windows in the Working Area will have no effect:

- *Peak Search Dialog* (section 3.3.4)
- *Peak Details Dialog* (section 3.3.5)
- *Parameters Window* (section 4)

Scans loaded from Launch mode are placed into the *Parameters Window* for graphical display purposes. Editing of these entries has no bearing on the fitting process which is controlled entirely from the INP file.

Note: It is possible to work in GUI and Launch Mode at the same time. Once an input file is set the *Launch* button in the *Fit Window* allows for the switching between GUI and Launch Mode.

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