HighScore Plus Quick Start Guide

- Introduction
- Getting Acquainted
- Opening a Diffractogram in HighScore Plus
- HighScore: Phase Identification
 - Background determination
 - Search Peaks
 - Strip K-2 Signal (OPTIONAL)
 - Peak Search and Match
- HighScore: Rietveld Analysis: Quantitative Phase Analysis
 - Starting Quantitative Phase Analysis
 - Setting up your desktop layout
 - Start Analysis
 - Display the Phase Amounts
 - Export and Save a File
 - Archive Versions

Introduction

This guide is an introduction to the HighScore Plus software used to solve crystalline phase identification.

HighScore for scientists is available on a virtual computer onboard in the Core Lab.



To access the Remote desktop click on the Windows tab and type 'remote desktop', this will bring you to a window to enter an IP address (*Figure 1*). Click **Connect**.

Only one person can use the remote desktop at one time. The remote desktop can be accessed from any PC onboard (Windows system only).

IP address: 165.91.150.141

Username: daq

Password: dag

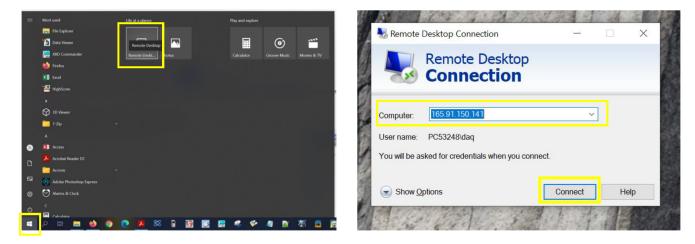
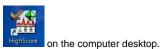


Figure 1. Connection to the Remote Desktop to use HighScore plus software

To disconnect from the virtual computer, move the mouse up screen to reveal the disconnecting options.

Getting Acquainted



To open the software double click the HighScore Plus icon

You can customize the main screen for your own requirements; the most common desktop used is **Phase-ID** for phase identification (see **HighScore: Phase Identification** below). The **Phase-ID** desktop can be selected on the bottom right toolbar in the dropdown menu (*Figure 2*, arrow A) or via Select **Vi ew > Desktop > Desktop Name > Phase-ID** in the dropdown menu (*Figure 2*, arrow B).

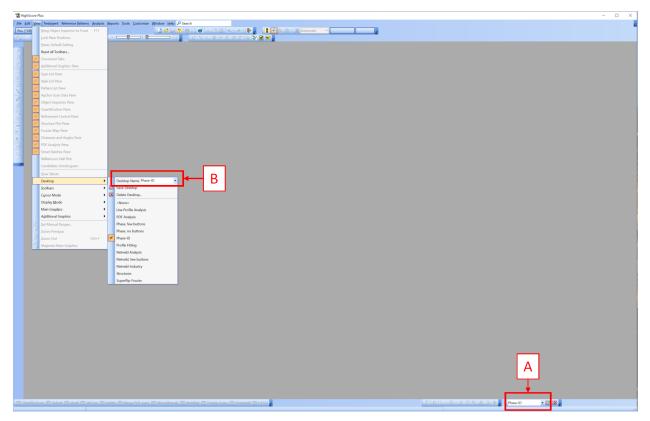


Figure 2. Setting the main screen to Phase-ID desktop

For the **Phase-ID** Desktop display, there are three main windows (*Figure 3*):

- The Main Graphics pane shows the scan and/or scans in the Analyze View tab (circled in green).
- The Additional Graphics pane displays the zoom overview.
 The Patterns and List pane is the third panel on the right of the screen. It is used to examine the Peak List, Scan List, Quantification Graphs, and the Anchor Scan Data tabs.



Opening a Diffractogram in HighScore Plus

To open a diffractogram for evaluation, click File>Open (Figure 4).

The HighScore Plus													
Eile	Edit	t <u>V</u> iew Treat <u>m</u> ent Reference <u>P</u> atterns <u>A</u> nalysis <u>B</u> eports <u>T</u> ools <u>C</u> ustomize <u>W</u> indow <u>H</u> elp P Search											
I.		New Ctrl+N 🕨 🖸 🐔 🖄 🖆 📾 🐟 - 🖈 - 🕪 💂 🚺 🛣 🔊 - 💥 Au											
)	<u>Ωpen</u> Ctrl+O <u>Λ Λ Δ / Ϩ / Ε Λ / ξ / Ξ 및 </u>											
	22	Open all XRDML from Ctrl+M											
	<u>*/</u>	Insert Ctrl+I											
		Import from Clipboard Table											
		Close Ctrl+W											
		Save Document Ctrl+S											
	ч.	Save As											

Figure 4. Open Scan

Select the file you want to open. HighScore Plus software can import files produced by Bruker D4 XRD or Panalytical AERIS XRD in the X-ray lab. The valid file formats include .raw, .uxd (Bruker) and .xrdml (Aeris) files.

- If you do not see the file you are looking for: in the field **Files type**, select **All files (.)**. This will bring up XRD files produced by the Bruker D4 or Panalytical AERIS.
- You can select multiple files; each one will open in its own window.

Click Open. The scan is now open and ready for evaluation (see HighScore: Phase Identification).

HighScore: Phase Identification

Diffraction pattern treatment is used for phase and crystallographic analyses. The two most important treatments are background determination and peak search. A proper background determination is very important for phase analysis.

There are several steps used when you want to determine the identity of the unknown phases in the diffraction pattern:

- 1. Background determination
- 2. Peak search
- 3. OPTIONAL Strip K-2 Signal
- 4. Peak Search and Match

Background determination

Background fitting is often easier if the y-axis is set to 'Square Root Y-axis' (*Figure 5*). To determine the background, select **Treatment** > **Determine Background** (*Figure 6*). The background is automatically determined (fluo green line on the diffractogram in *Figure 6*). The **Determine Background** window will show on the screen (*Figure 6*).

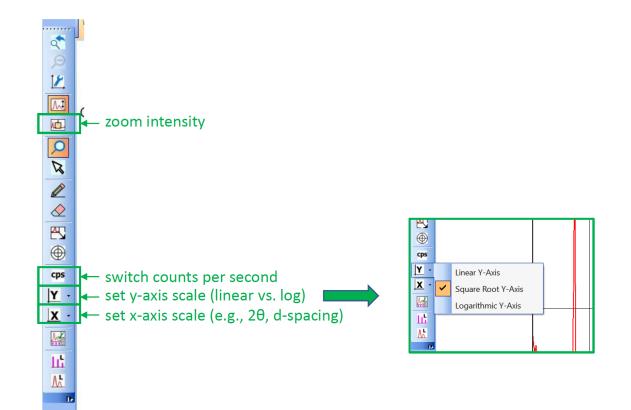


Figure 5. Useful display options

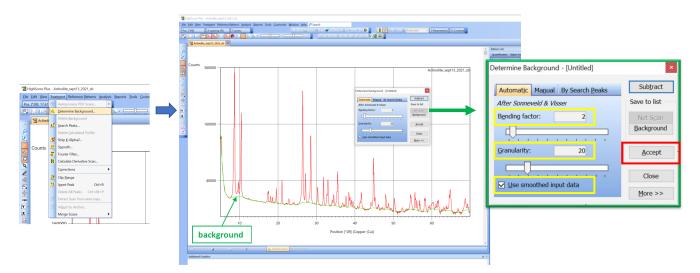


Figure 6. Determining Background

Automatic background fitting is most often used. Adjust parameters until the green background line is a good fit to the data, without overfitting or underfitting the data. You will want to choose your **bending factor** and a **granularity**.

You can change the **Bending factor** by moving the slider on the Determine Background window. This field adjusts the nonlinearity and curvature of the background. Typical values are between 0 and 4 (normally a small number 1-2 fits well).

You can change the **Granularity** by moving the slider. This field changes the number of intervals used for background determination. Typical values range between 10 and 30. The default value of '20' fits most ordinary scans.

Tick the box 'Use smoothed input data' to avoid oversampling.

When the background is fit, click **Accept** to accept the background (*Figure 6*). The **Determine Background** window is closed and the accepted background is displayed as a dark green line on the **Main Graphics** pane (see *Figure 7*).

Search Peaks

Select **Treatment** > **Search Peaks.** The **Search Peaks** window opens (*Figure 7*). Adjust the peak search parameters if needed (Figure 7). The default settings are a good starting point. The **significance** is a calculation of the probability that a possible peak is not noise-induced. A large **minimum significance** above 2 or more is useful for noisy data. Normally you should not have to adjust the other values. Once you are happy with the parameters, click **Search Peaks** (*Figure 7*).

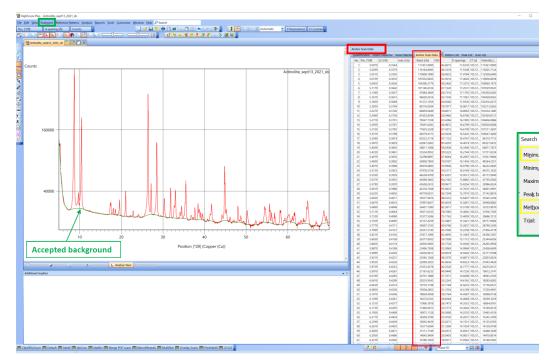


Figure 7. Search Peaks

Detected peaks are displayed above the **Main Graphics** pane by orange lines (green box in *Figure 8*). A calculated pattern based on the peak search is shown in pink over the experimental data shown in red.

Click on (Set Display of Peaks') to toggle the display of peaks.

- K-1 peaks are indicated by solid lines
- K-2 peaks are indicated by dotted lines
- Peaks that are not explained by a reference pattern have a little 'V' mark.

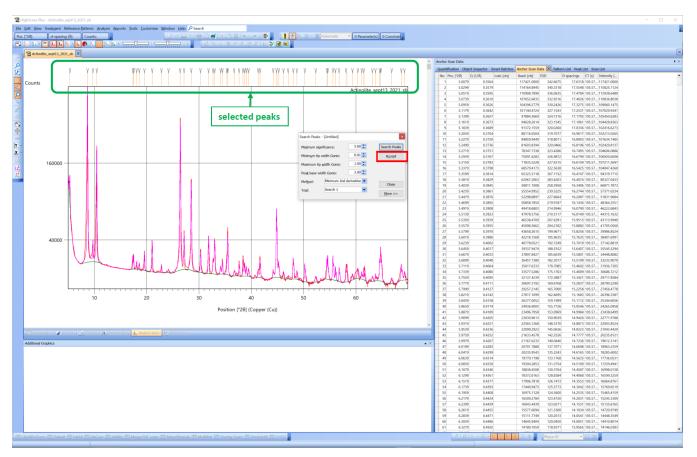


Figure 8. Selected peaks and accept peaks.

When you are happy with the results, click Accept button (Figure 8).

In the **'Patterns and List'** pane, click on **'Peak List'** tab to show the numerical details on every detected peaks (*Figure 9*). In the **Peak List**, you can right click on a row to '**Add Peak'** or '**Delete Peak'** or '**Remove Selected Peak Features from Scan'** (*Figure 9*). Peaks derived from the K-2 wavelength are indicated by a different (gray) background color (*Figure 9*). Deleting certain peaks will help the software to focus on specific peaks for mineral searching. This can be helpful if you have a multiphase (multi mineral) bulk sample.

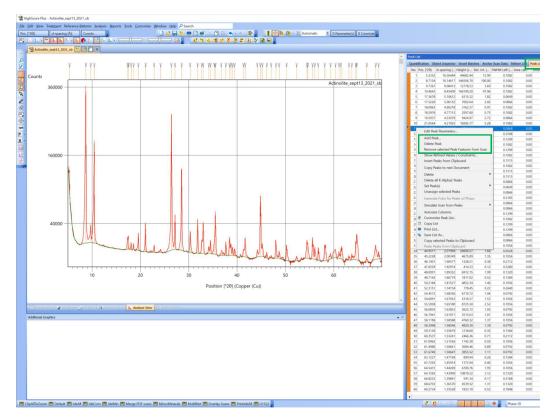


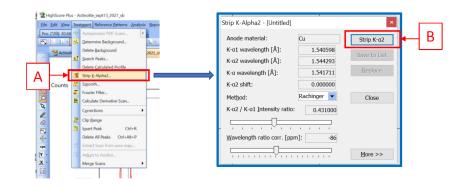
Figure 9. Peak List displaying the numerical details of the detected peaks from the Peak Search

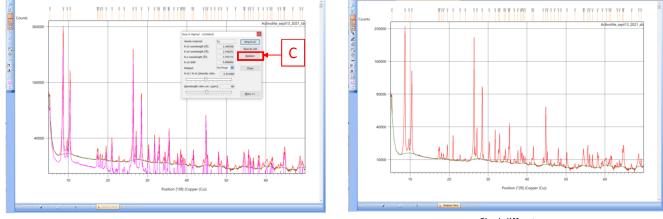
Strip K-2 Signal (OPTIONAL)

This step is **OPTIONAL** and not necessary for phase analysis or other analyses you can do in HighScore Plus. K-2 radiation can be computationally stripped from the data because the relationship between K-1 and K-2 radiation is very well known.

Primarily you remove the K- peaks from the diffraction pattern to clean up the data or make it easier to evaluate good and/or poor matches. To remove the K-2 signal form your data:

Select Treatment>Strip K-Alpha2 (Figure 10, arrow A). Click the Strip K-2 button (arrow B), then click the Replace button (arrow C).





Removing the K-α2 signal

Final diffractogram

Figure 10. Stripping the K-2 signal

Peak Search and Match

Once you have determined the background and searched for peaks the next step is to match the peaks.

Select Analysis >Search & Match > Execute Search & Match (Figure 11).

The Search & Match window appears with a default parameters set (*Figure 11*). For initial searching the 'Default' parameters are fine to start with. You can adjust the parameters as necessary.

- Data Source: Best results when you use Peak & Profile Data
 - Peak Data is the peak list produced from your peak search.
 - Profile Data is all observed intensity above the background model produced when you fit the background
- Scoring Scheme: Set this to Multi Phase. Only use single phase if you want to force program to use a single phase to match all the observed data.
- Auto Residue: Make sure you select this. When you accept a candidate as a good match all of the remaining candidates are rescored based on how well they fit the unmatched features.
- Match Intensity: if this is off, the quality of the match is based only on agreement of peak positions; if this is on, the score reflects the quality of the intensity match as well.
- Demote unmatched strong: if on, if a candidate has one strong (>50%) peak missing in the observed data then the candidate is discarded—no matter how well the rest of the peaks match.
- Allow pattern shift: if on, each reference pattern is shifted for an optimal fit with the data. The maximum allowed pattern shift is ± 4 x FWHM.

Click Search (Figure 11, red box).

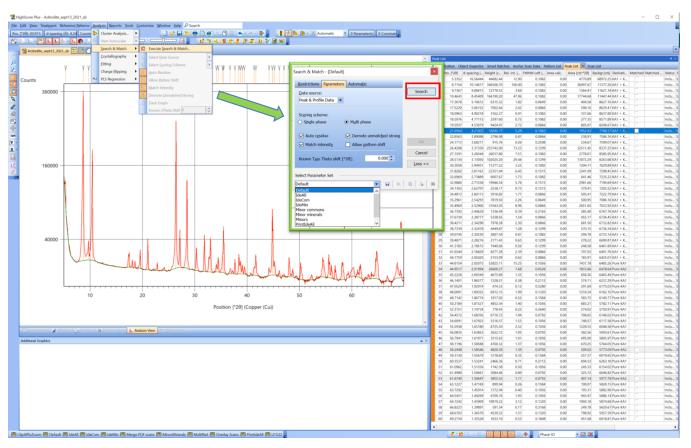


Figure 11. Peaks Search and Match

List of the possible matches is displayed on the lower portion of the **Pattern List** pane (*Figure 12*). Click **OK** to accept (*Figure 12*, red box). The **Candidates** list shows entries ordered by high to low score based on how well they match the experimental data (*Figure 12*).

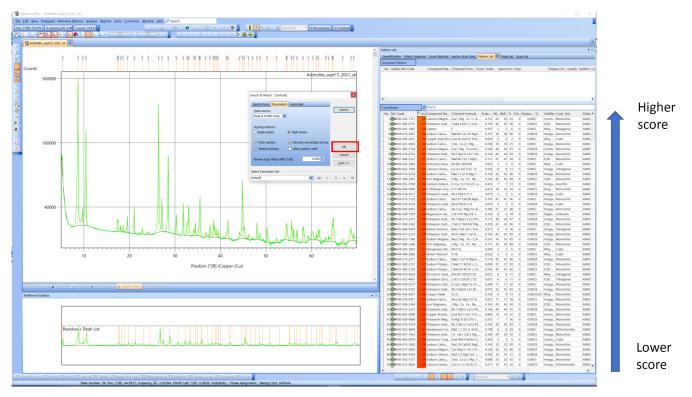


Figure 12. Candidates List ordered by strongest and weakest matches.

The next step is to manually accept candidates that have a high score and that match the peaks and features of the measurement.

For that, select and drag the matching candidate patterns from the **Candidates** list to the **Accepted Ref. Patterns** list in the upper half of the **Pattern List** tab to accept it (*Figure 13*). When you select matching patterns, it is highlighted in gray and lines are displayed in the **Main Graphics** and **Additional Graphics** panes (*Figure 13*). Peaks will lose the "V" mark above the line. These peaks have been explained by the reference pattern. Peaks with a "V" have not been explained and/or matched (*Figure 13*).

There are several views in the Additional Graphics pane to support the visual comparison between reference pattern sticks and the measurement. For instance:

- Select View > Additional Graphics > Compare Mode or
- Select View > Display Mode > Show Calculated Profile

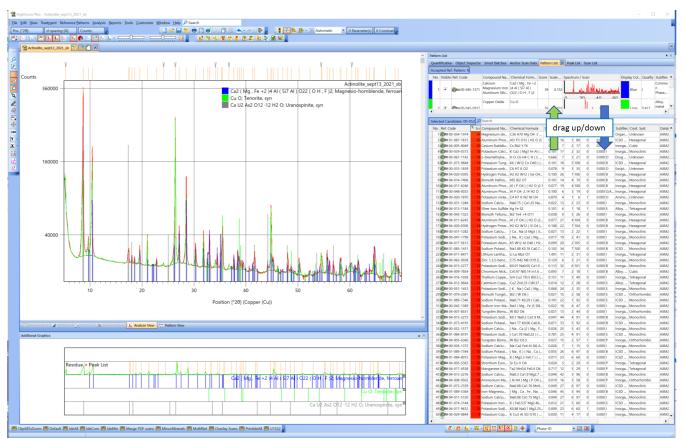


Figure 13. Move accepted peaks from the Candidates sub-panel the Accepted Ref. Patterns sub-panel

To check if the accepted patterns are indeed minerals and comply with the sample description, right-click a specific reference pattern in the Accepted Ref. Patterns list. Select Show Pattern to view the subfile information of that pattern (Figure 14).

							X		
Pattern List		_							
	Quantification Object Inspector Smart Batches Anchor Scan Data Pattern List X Peak List Scan List								
_	Accepted Ref. Pattern: 0							Reference Pattern: 00-045-1	13/1
Enclose a construction of the second	No. Visible Ref. Code Compound Na Chemical Form Score Scale Spectrum / Scan Display Col Quality Subfiles *								
NO. VISIDIE REF. Code	Calcium Ca2 (Mg , Fe +2	e scale s	spectrum / sc	an	Usp	nay Col Quality	Subtries -	Name and formula	a
1 🗹 🛱 ato 00-04	IS-13 / Execute DDM Semi-QPA Method			diller.		81	n		
• Man co-or	Analyze Pattern Lines	0.152		IIII AL AL	A REAL	DILLE I	Phase,	Reference code:	00-045-1371
	Show Pattern		0 20	40	60		ICSD		
2 🔽 🛱 🕅 01.05		0.541		Maler	اشان.		Pattern, *	Mineral name:	Magnesio-hornblende, ferroan
4	Retrieve Pattern by						•	Compound name:	
Selected Candidate: 00-	Paste Patterns from Dataset						_		Calcium Magnesium Iron Aluminum Silicate Hydroxide
No. Ref. Code	copy to			n Dimbra D			Dutyl 4	PDF index name:	Calcium Magnesium Iron Aluminum Silicate Hydroxide
No. Ref. Code 1 #300 00-054-1974	Remove Pattern	le ML		IL Displac Q		The second	Datat AXM2		
2 4730 04-011-6246		1.077 19		0 0.000 8		. Hexagonal	a kinz	Empirical formula:	Al ₂ Ca ₂ H ₂ Mg ₄ O ₂₄ Si ₇
3 3 00 04-020-0305		1.099 26		0 0.000 8		. Hexagonal	AXM2	Chemical formula:	Ca2(Mg,Fe+2)4Al(Si7Al)O22(OH,F)2
4 200 01-087-1615		077 16		0 0.000 B		Hexagonal	AXM2	Mineral classification	: Amphibole (Family), 1M-Ca (Subgroup)
5 00-033-1839		.077 9	2 35	0 0.000 O		Unknown	AXM2		
6 Ctt 001-073-9684		1.101 18	5 108	0 0.000 B	ICSD	Hexagonal	AXM2		
7 00 04-020-0306		.101 22	3 104	0 0.000 B	Inorga	Hexagonal	AXM2	Crystallographic p	
8 00-048-0033		100 6	3 19	0 0.000 O;	A., Inorga	. Hexagonal	AXM2	Crystallographic p	arameters
9 10 00-031-1284		.022 13		0 0.0001		Monoclinic	AXM2		
10 10 04-008-9502		· 0.019 16		0 0.000 P		Orthorhombic	AXM2	Crystal system:	Monoclinic
11 11 04-012-9664		1.014 12		0 0.000 S		Tetragonal	AXM2	Space group:	C2/m
12 12 12 10 04-001-8551 13 12 10 04-011-6245		0.021 13		0 0.000 1		Orthorhombic	AXM2 AXM2	Space group number:	12
14 1200 00-067-1142		203 7		0 0.000 B		. Hexagonal Unknown	AXM2 AXM2		
15 15 00-016-1038		151 11		0 0.0001		. Tetragonal	AXM2	a (Å):	9.8500
16 1500 00-047-1799		017 19		0 0.0001		Monoclinic	AXM2	b (Å):	18.0720
17 17 10 00-042-1323		0.037 9		0 0.000 1		Monoclinic	AXM2	c (Å):	5.3070
18 00-031-1282		021 13		1 0.000 1		Monoclinic	AXM2		
19 19 04-017-5613	Copy selected Patterns to Clipboard	.099 20	2 105	0 0.000 B	Inorga	Hexagonal	AXM2	Alpha (*):	90.0000
20 Ctab 00-065-0938	Pasta Patterns from Cliphourd	1.129 6	3 21	0 0.000 1	Inorga	Monoclinic	AXM2	Beta (*):	104.9500
21 21 00-042-1369		1.021 16		0 0.000 1		. Monoclinic	AXM2	Gamma (*):	90.0000
22 000 01-079-2381		0.021 15		0 0.000 S		Orthorhombic	AXM2		
23 04-017-1607		0.014 19		0 0.000 P		Orthorhombic	AXM2	Volume of cell (10 ⁶ pr	n ³): 912.72
24 24 25 00-026-1372 25 25 00-005-6360		0.026 7	1 10	0 0.000 1		Monoclinic	AXM2	Z:	2.00
26 argta 00-052-1659		0.027 15		1 0.000 P 0 0.000 B		Orthorhombic Monoclinic	AXM2 AXM2		
27 27 27 27 27 27 27 27 27 27 27 27 27 2		0.038 13				Orthorhombic	AXM2	C > Swin Ar Conv B	Print Graphics Print All Intensity Scale Angle Scale Wavelength 1.54060
28 28 00-038-0359				0 0.0001		Monoclinic	AXM2	s > _ save As copy +	The organization of the state o
29 gat 04-005-5363		0.024 5		0 0.000 P		Tetragonal	AXM2		
30 01-074-3144	9 Potassium Iron K (Fe0.537 Mg2.46	0.008 21	1 60	0 0.000 S		Monoclinic	AXM2		
31 00 01-089-7343	Sodium Potassi (Na K) (Na Ca) (0.037 20	3 94	0 0.000 I	ICSD	Monoclinic	AXM2		
32 32 00-068-0243				0 0.000 I	Ceram	Anorthic	AXM2		
							11/1 12		

Figure 14. Information on an accepted reference pattern

If you wish to save the complete file (including the candidate list and the accepted reference patterns), click **File > Save Document**. HighScore Plus file format is **.hpf**.

HighScore: Rietveld Analysis: Quantitative Phase Analysis

To complete a quantitative phase analysis you will use the Rietveld refinement in HighScore Plus software.

Starting Quantitative Phase Analysis

Before completing the quantitative phase analysis, make sure you have completed all of the phase identification steps (see **HighScore: Phase Identification** above):

- 1. Determine the background
- 2. Search peaks
- 3. OPTIONAL: Strip the K-2 signal
- 4. Peak Search and Match

Now you are ready to complete quantitative phase analysis using the **Rietveld method**. This method is a full pattern fit method. It is typically used for standardless, quantitative phase analysis.

Setting up your desktop layout

Select the desktop layout from Phase-ID to Rietveld Analysis on the Desktop toolbar (*Figure 15*, arrow A). Or View > Desktop > Desktop Name > Rietve Id Analysis.

In addition to the **Main Graphics** and **Additional Graphics** pane, available on the Phase-ID desktop as well, there are two other panes: **Refinement Control** and **Object Inspector** (*Figure 15*).

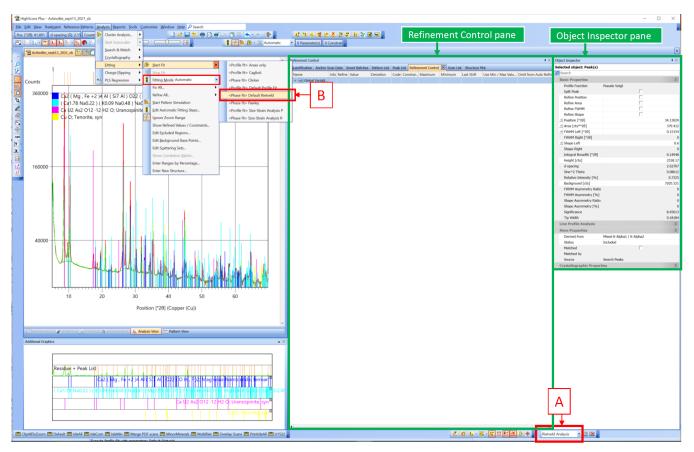


Figure 15. Rietveld Analysis Desktop Layout

Start Analysis

Select Analysis > Fitting. Check that the option Fitting Mode is set to Automatic (*Figure 15*). Correct if necessary. Select Analysis > Fitting > Start Fit> and then select <Phase fit> Default Rietveld to start the refinement with the default Rietveld parameters set (*Figure 15*, arrow B). Wait until it is finished, this may take a few minutes.

The peaks and the calculated profile are in the **Main Graphics** pane. In the 'Refinement Control' tab in the **Refinement Control** pane on the main display, double-click the **Global Variables** (*Figure 16*, arrow A) to open the 'Object Inspector' pane (*Figure 16*). Select **Global Settings>Agreement Indices** to view the values of the **Goodness of Fit** and **Weighted R profile** (*Figure 16*, arrow B).

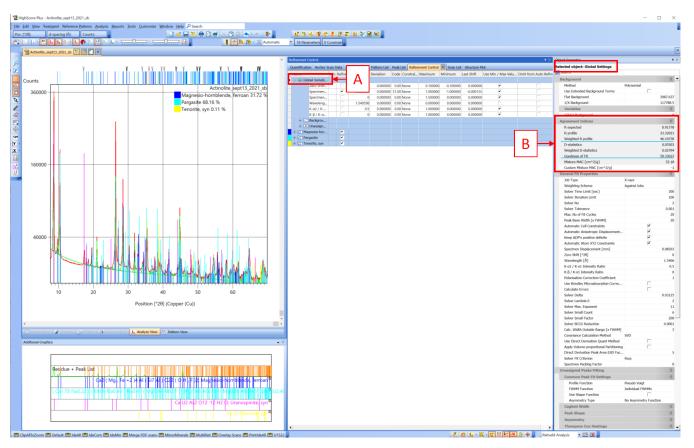


Figure 16. Refinement Control pane displaying the **Refinement Control, Global Variables**. The **Object Inspector** can be used to look at the fit properties and agreement indices.

Display the Phase Amounts

The phase amounts will automatically be displayed together with the phase legend in the **Main Graphics** pane once the Rietveld analysis is completed (*Fig ure 17A*). Alternatively, you can open the 'Quantification' tab in the **Refinement Control** pane and view a pie chart of the quantification (*Figure 17B*).

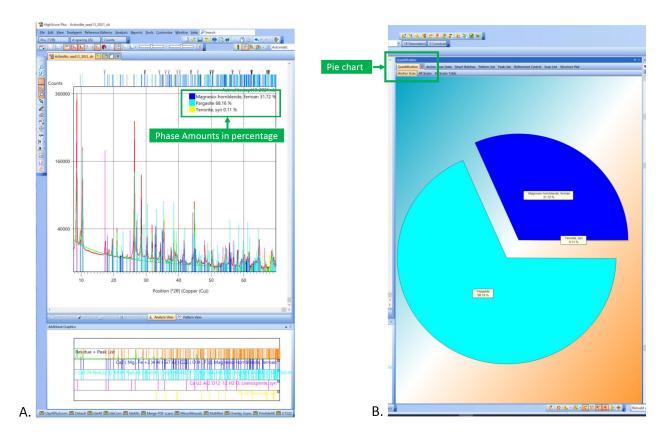


Figure 17. A. Phase amounts as a percentage are displayed on the main graphics window. B. Pie chart of the phase amounts for the quantification of the phases

Export and Save a File

To export a file or save the file as a working file, click **File>Save As.** Save the document as a **.hpf** file (this is the HighScore working file format).

Archive Versions

HighScore Software QSG (Original Version 378) - 290220