HighScore Plus Quick Start Guide

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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: daq

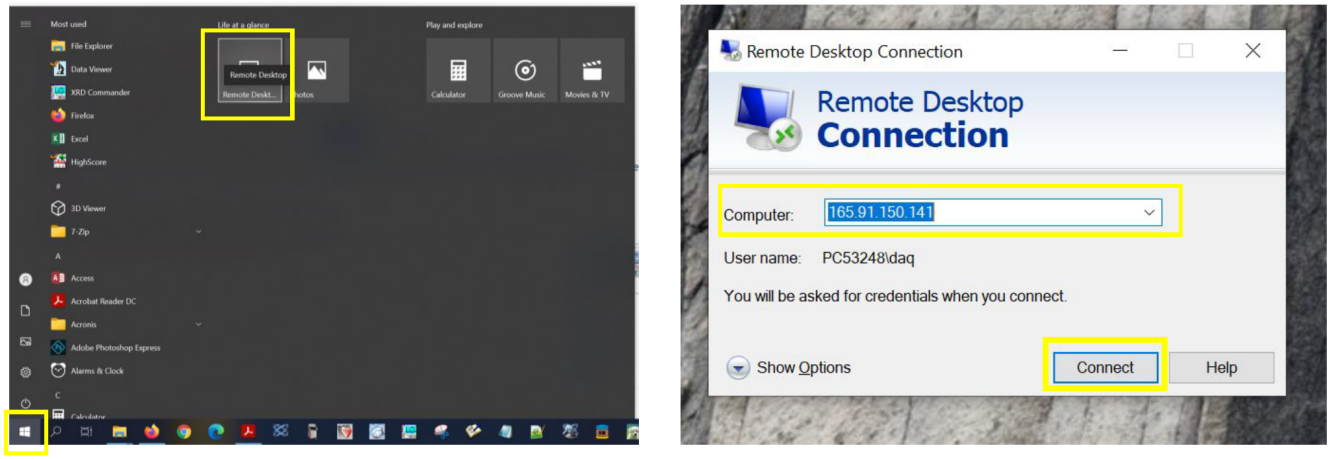

Figure 1. Connection to the Remote Desktop to use HighScore plus software

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 

on the computer desktop.

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 **View > Desktop > Desktop Name > Phase-ID** in the dropdown menu (*Figure 2*, arrow B).

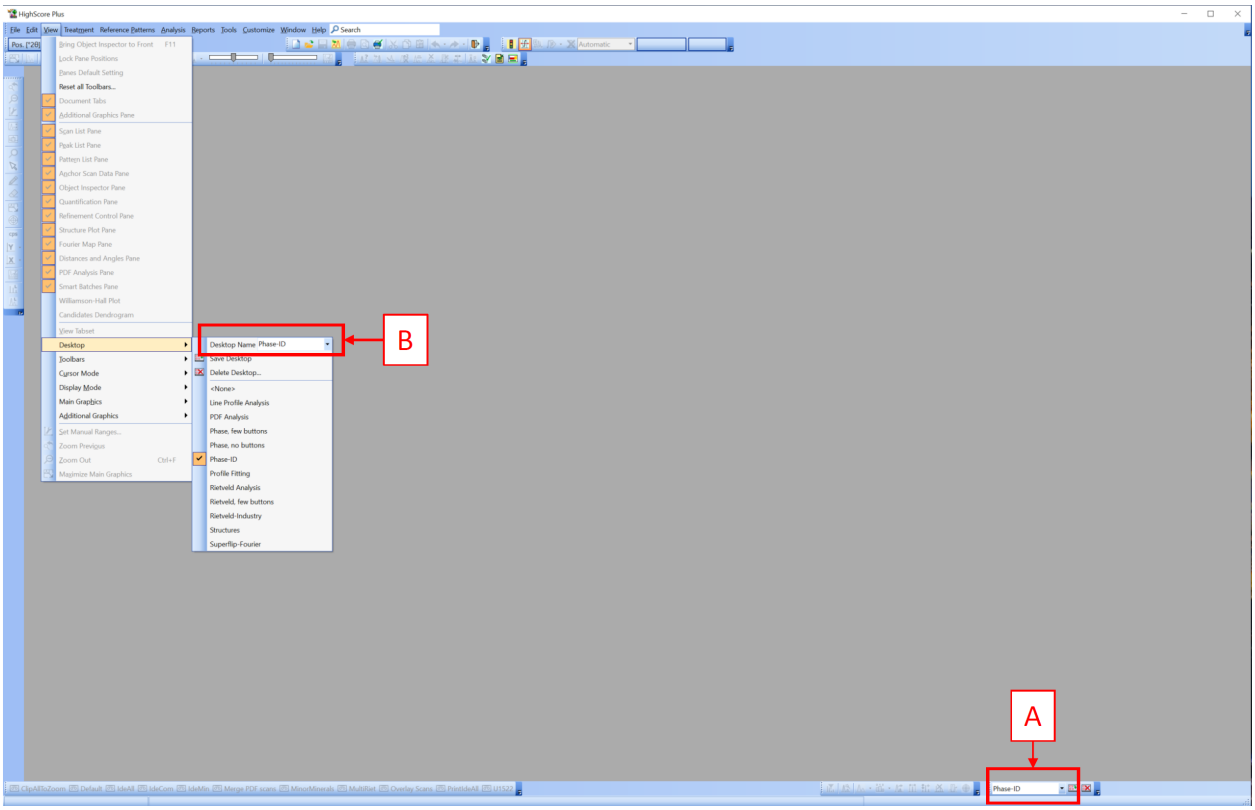

Figure 2. Setting the main screen to Phase-ID desktop

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.

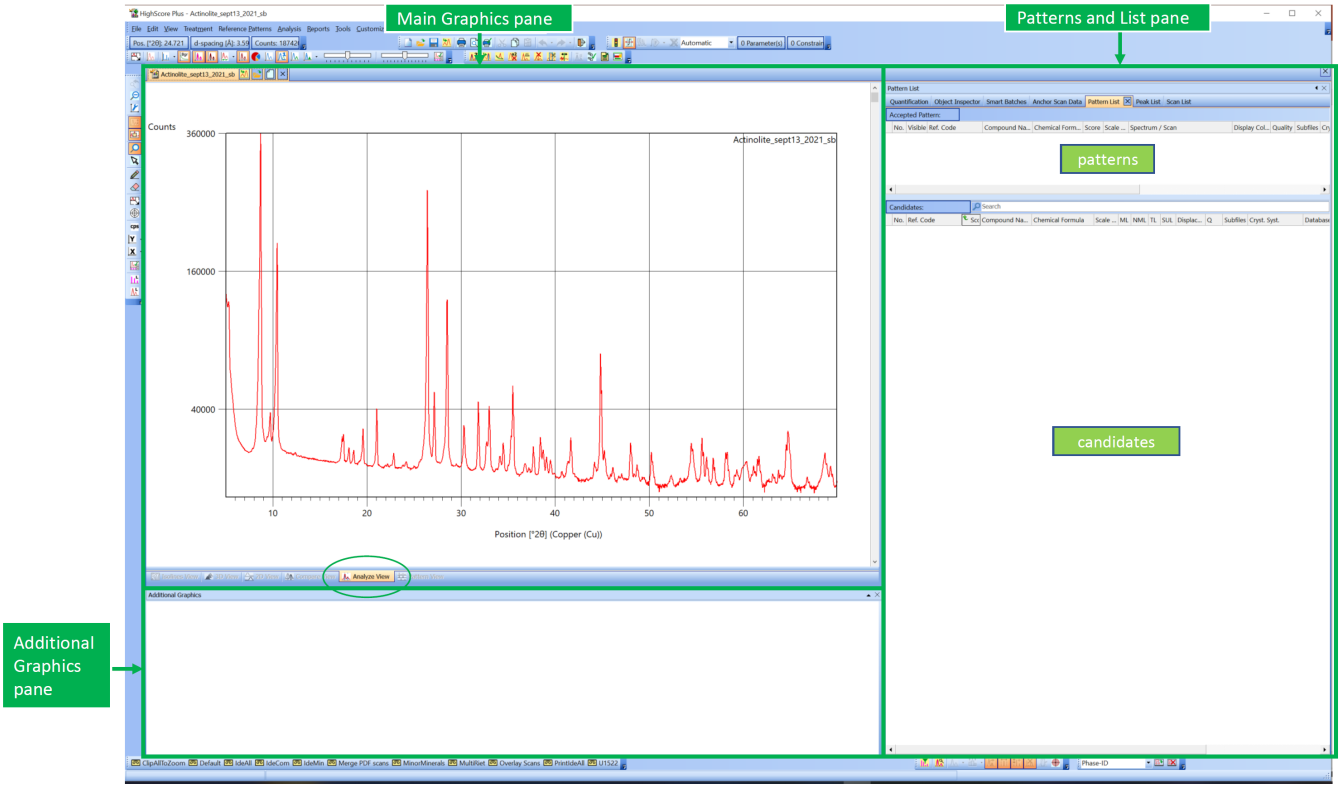

Figure 3. Basic Layout of the "Phase-ID" desktop

Opening a Diffractogram in HighScore Plus

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

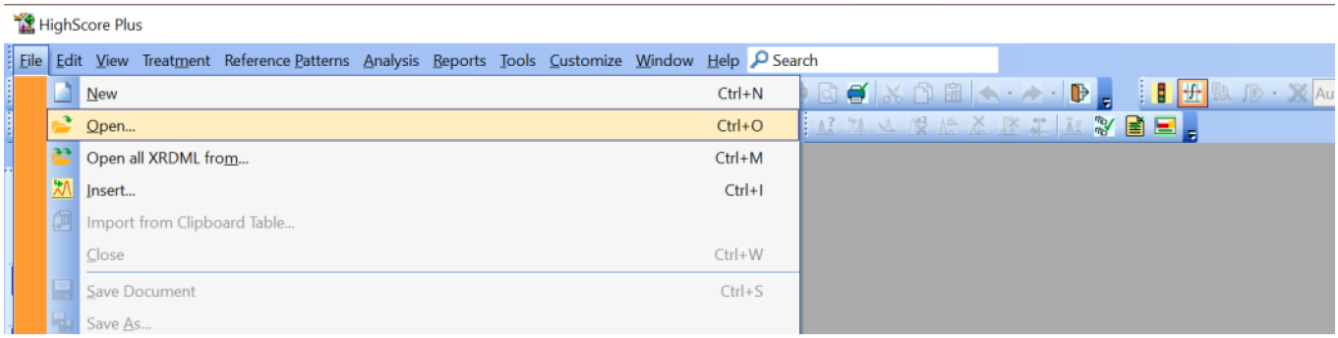

Figure 4. Open Scan

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*).

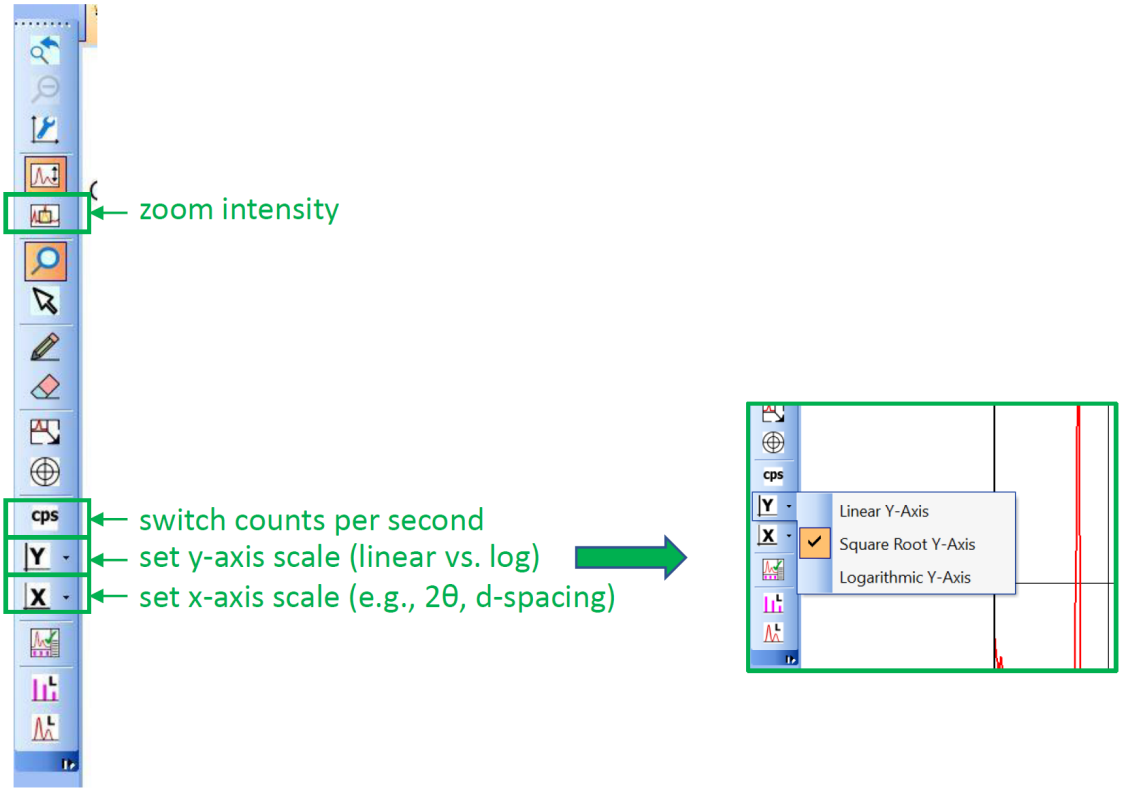

Figure 5. Useful display options

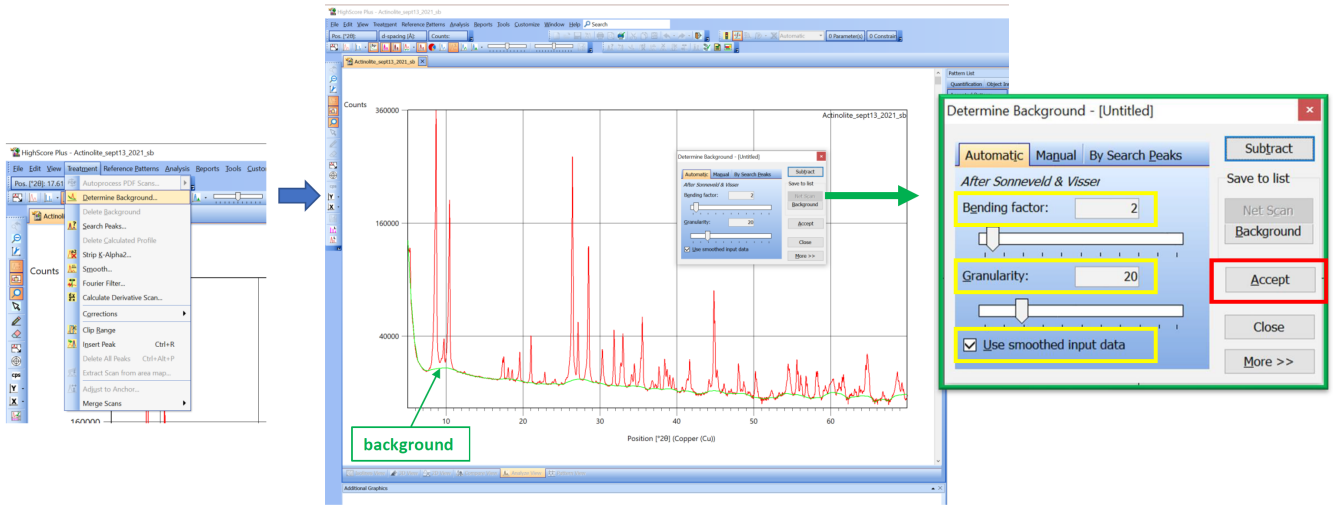

Figure 6. Determining Background

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*).

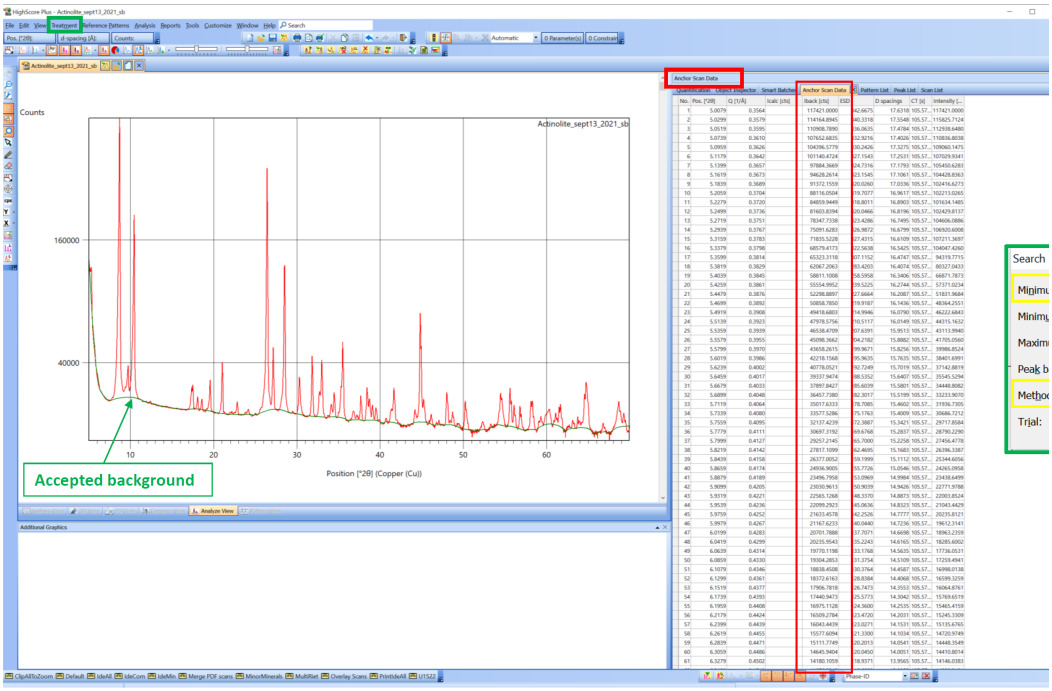

Figure 7. Search Peaks

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.

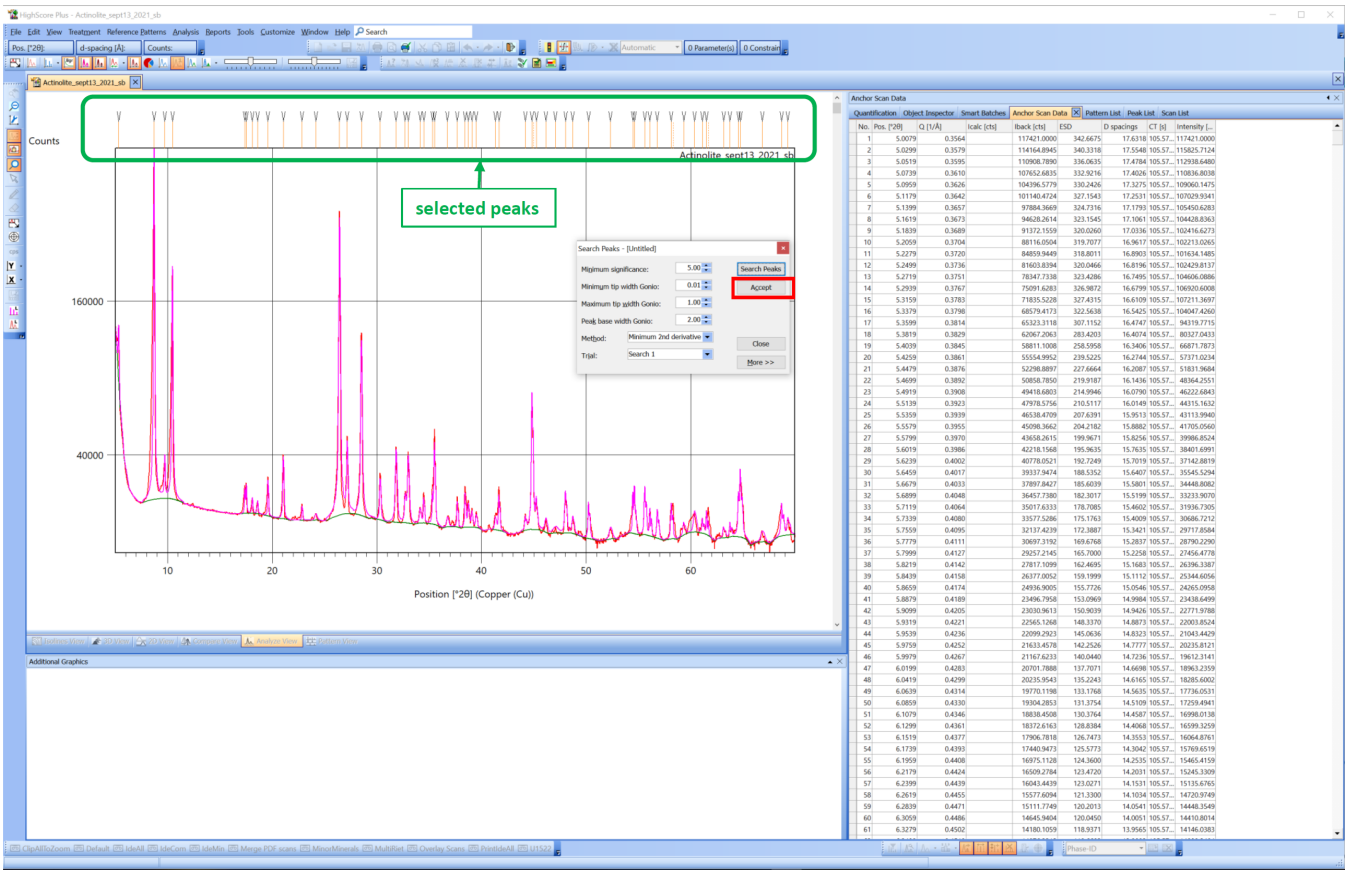

Figure 8. Selected peaks and accept peaks.

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.

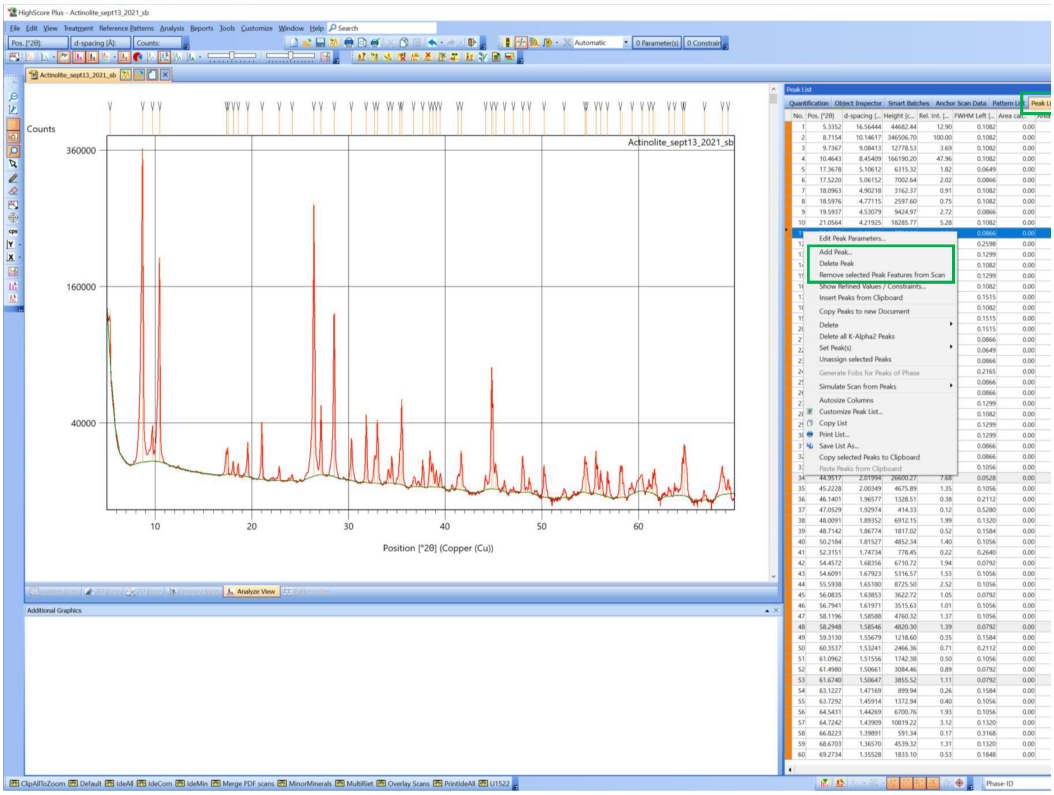

Figure 9. Peak List displaying the numerical details of the detected peaks from the Peak Search

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).

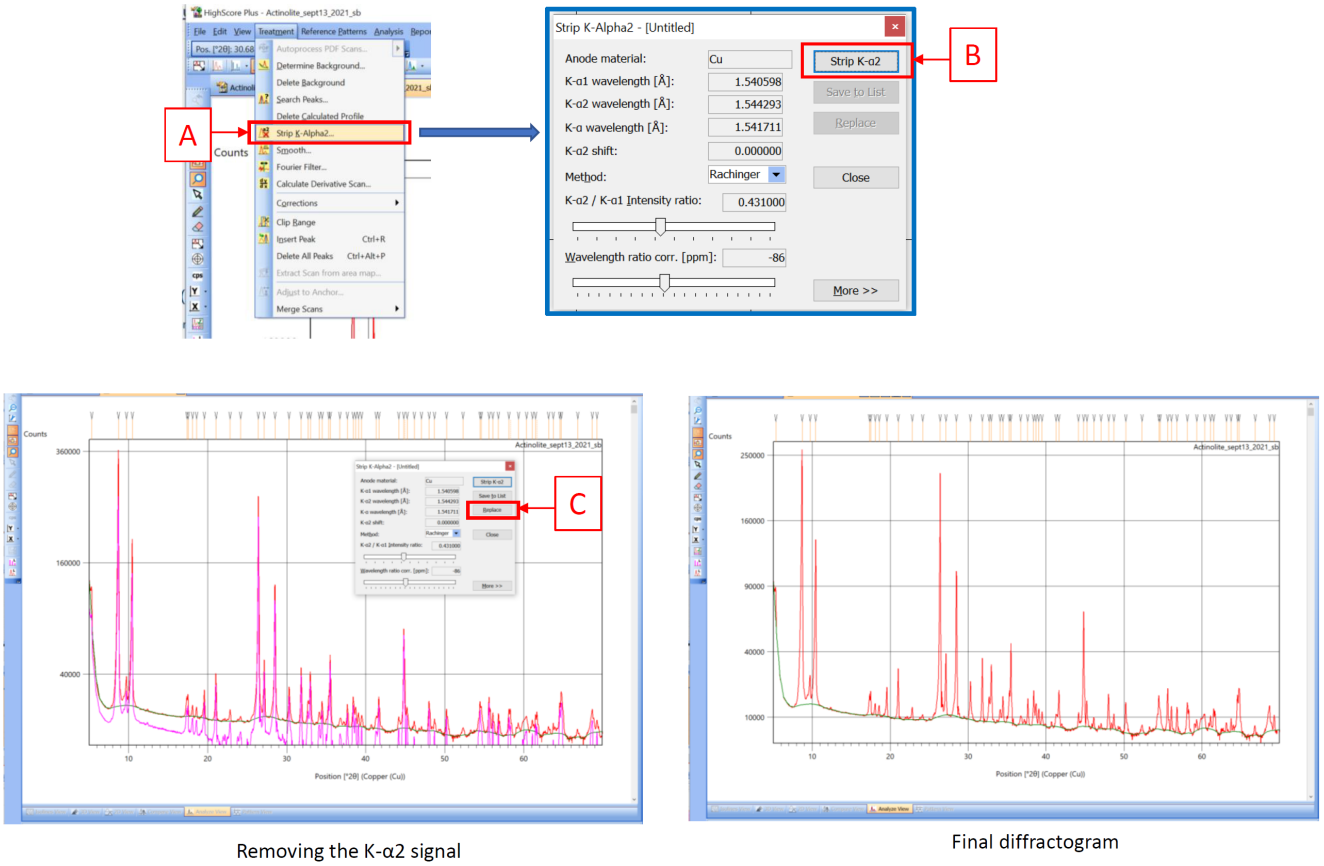

Figure 10. Stripping the K-2 signal

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).

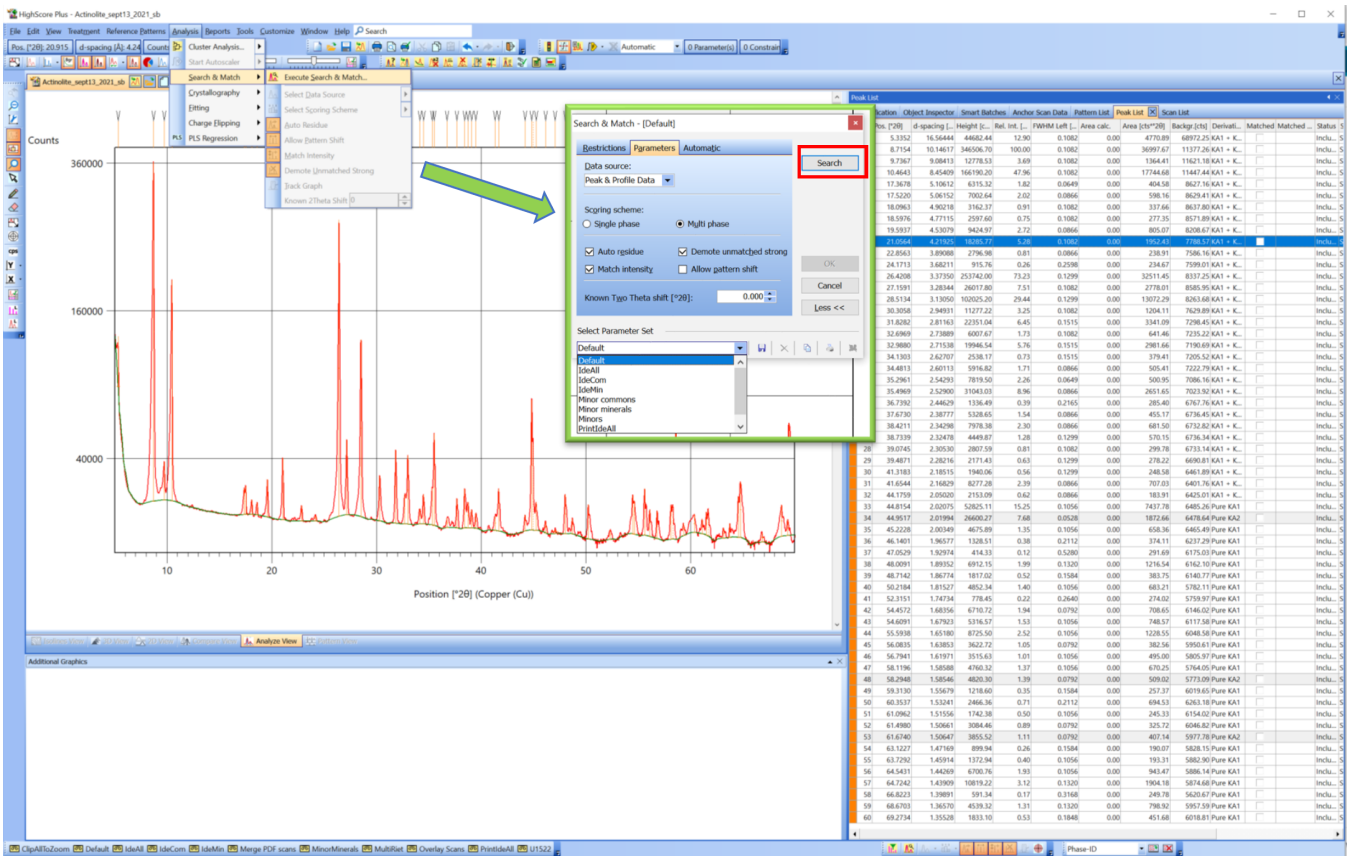

Figure 11. Peaks Search and Match

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*).

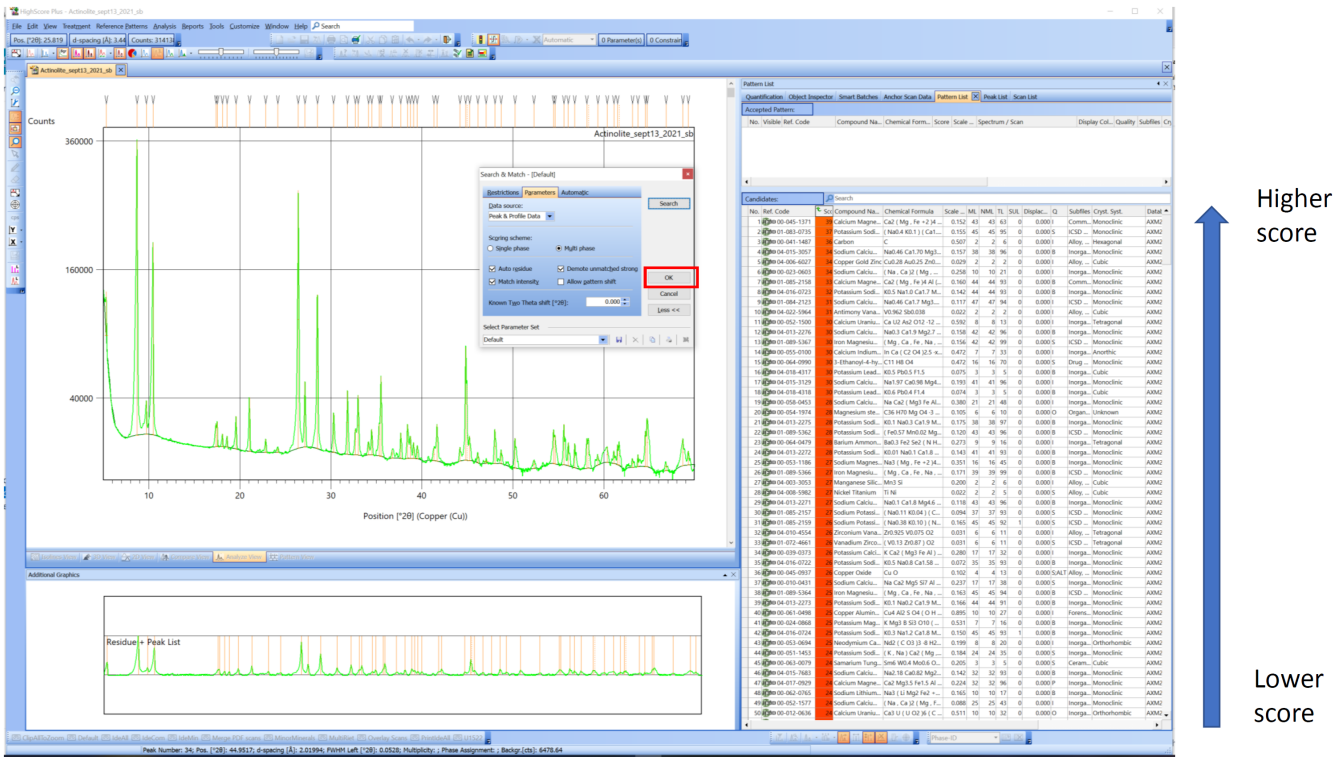

Figure 12. Candidates List ordered by strongest and weakest matches.

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**

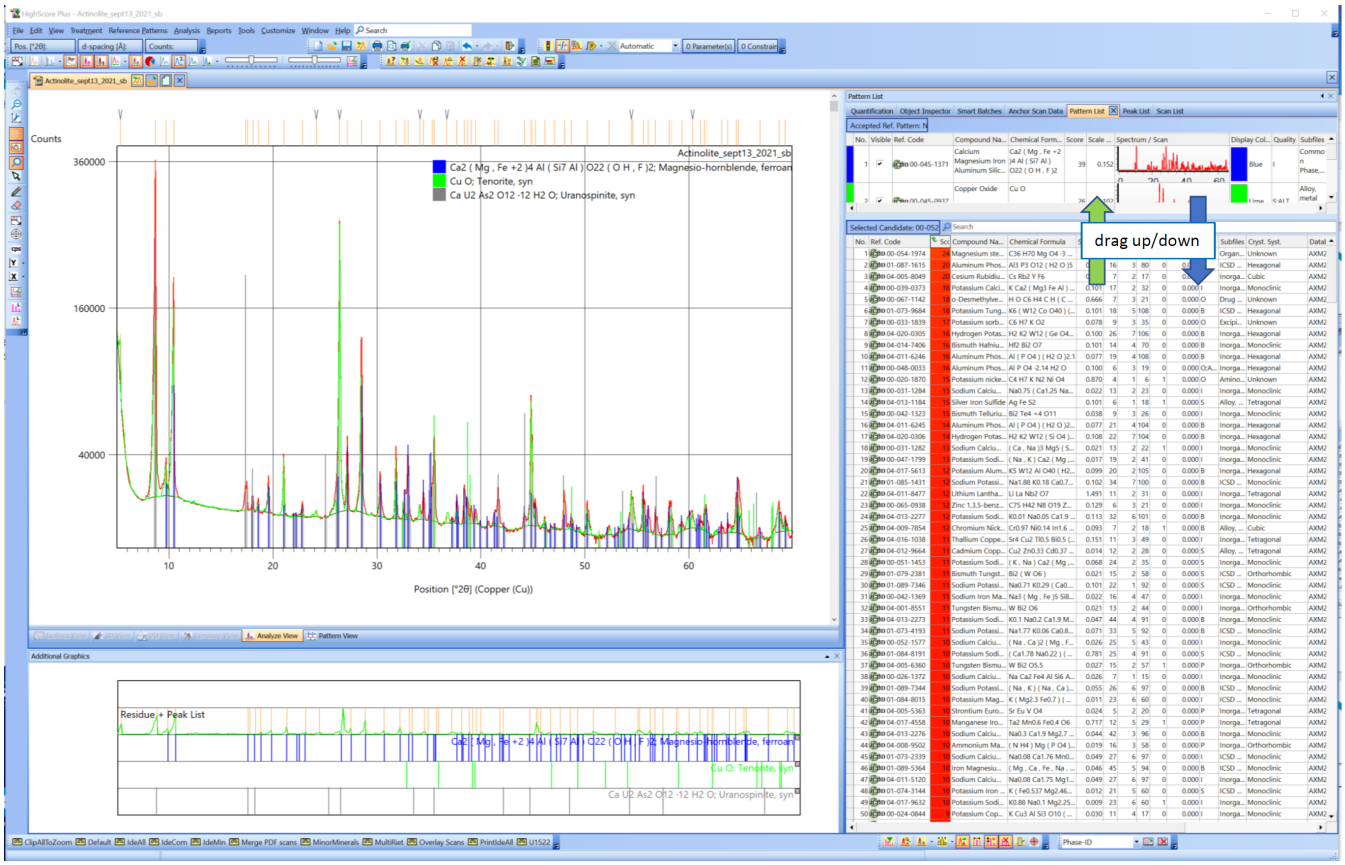

*Figure 13. Move accepted peaks from the **Candidates** sub-panel the **Accepted Ref. Patterns** sub-panel*

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).

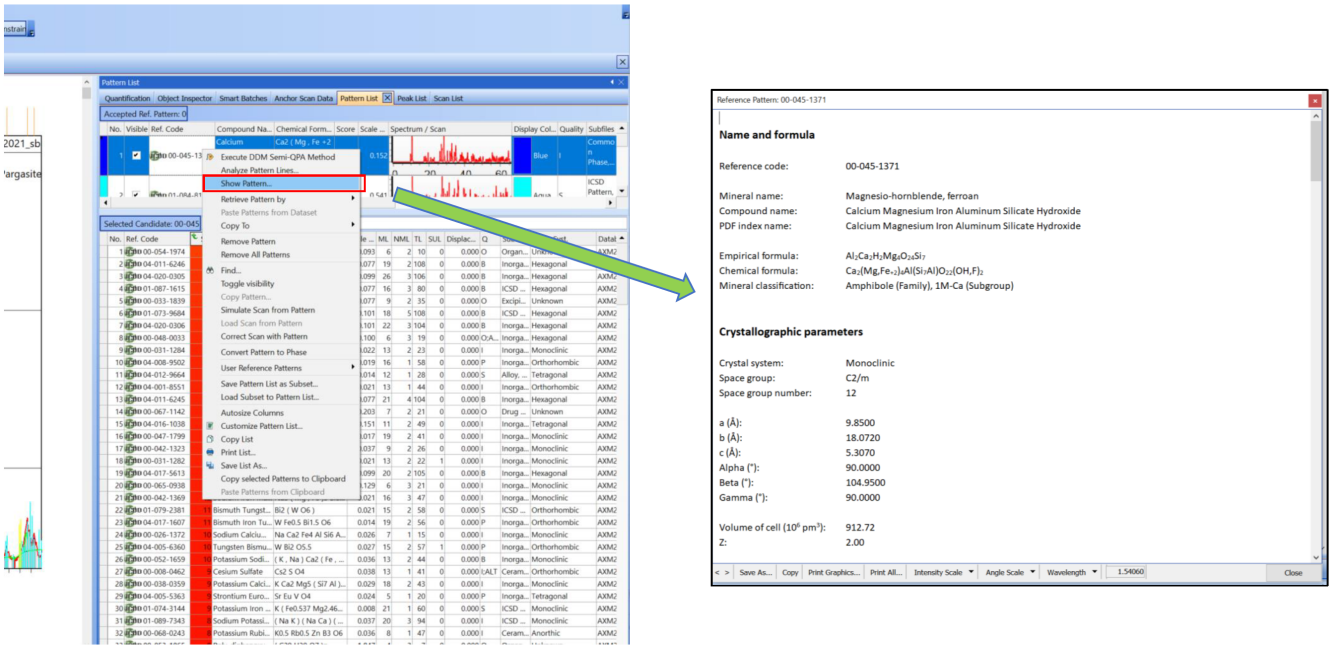

Figure 14. Information on an accepted reference pattern

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 > Rietveld 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*).

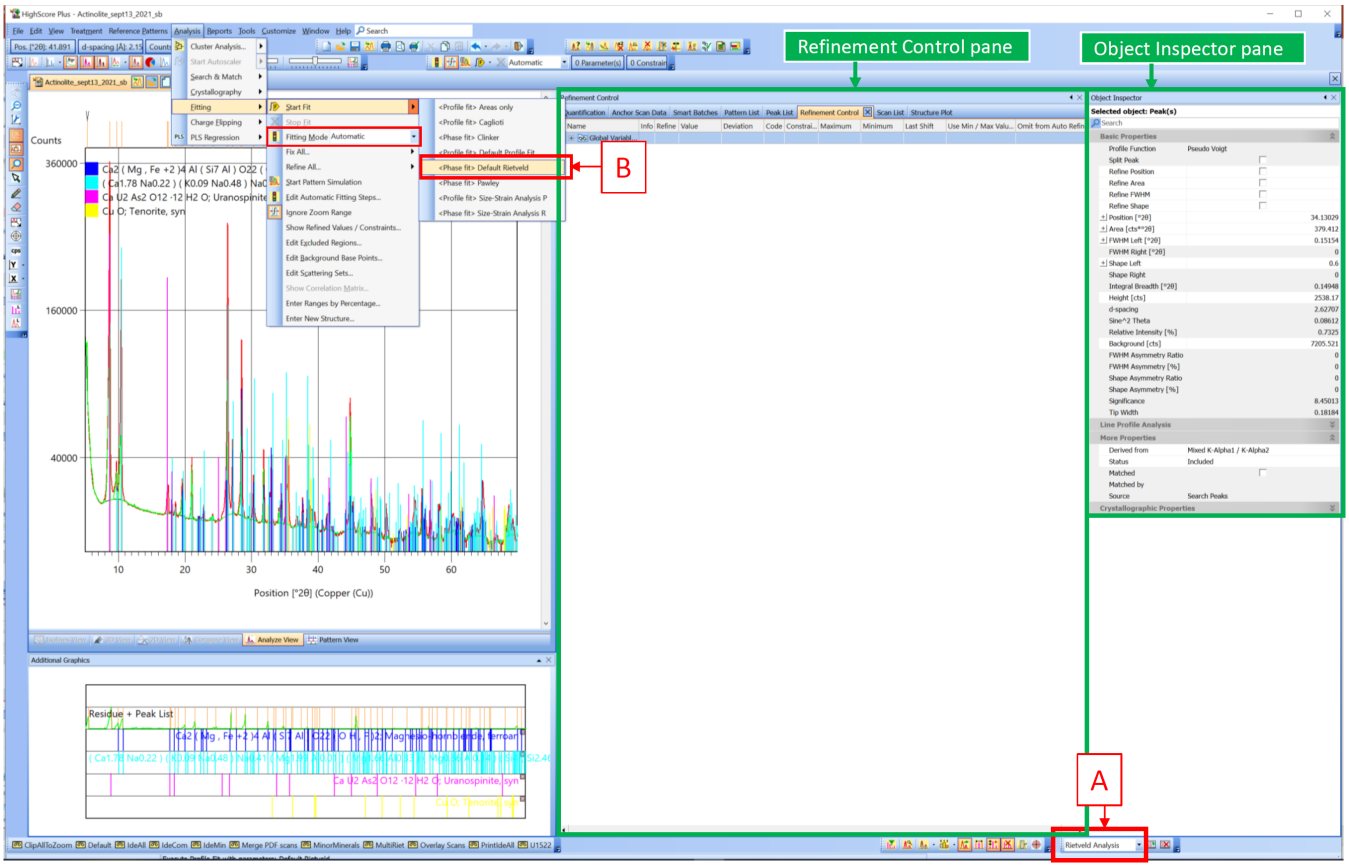

Figure 15. Rietveld Analysis Desktop Layout

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).

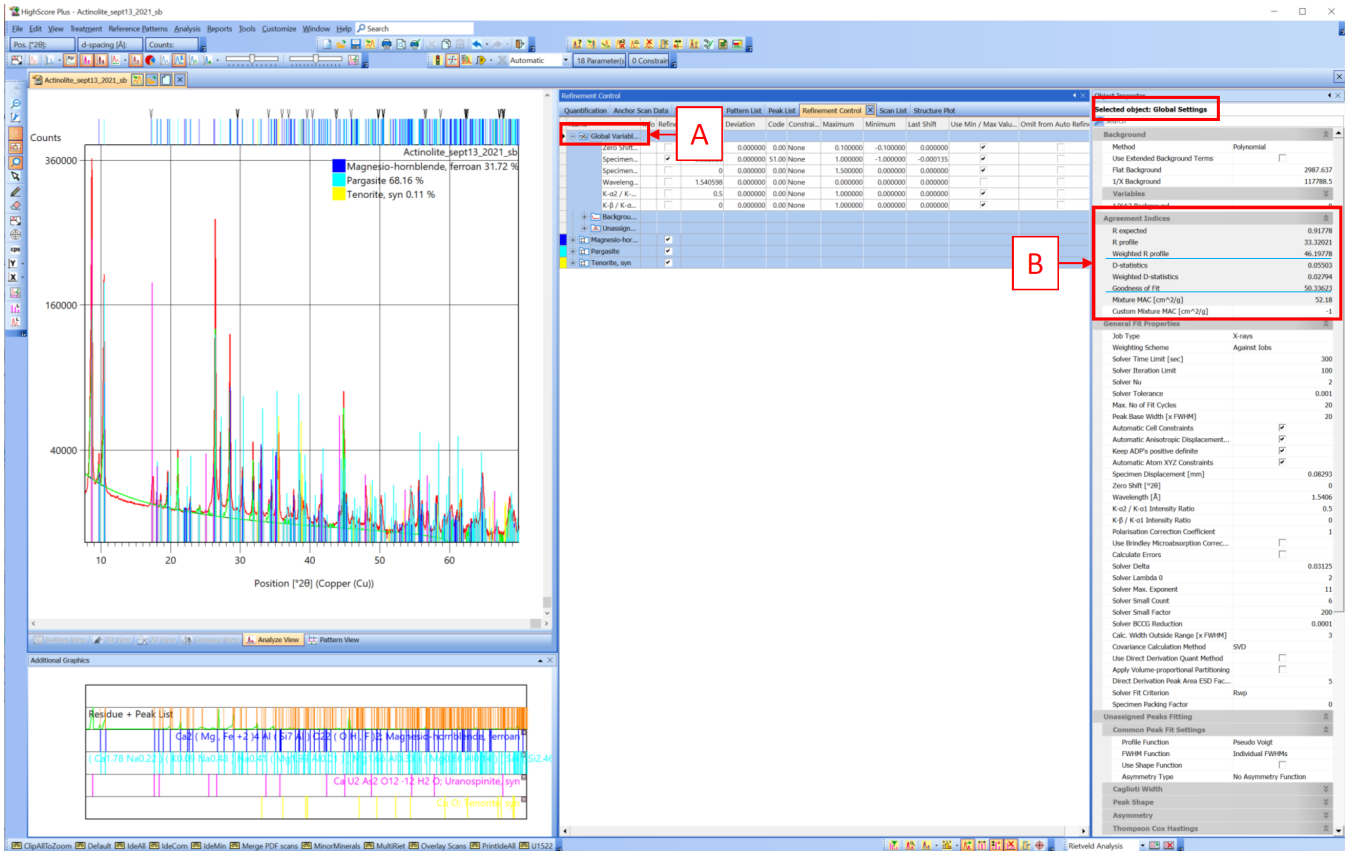

*Figure 16. Refinement Control pane displaying the **Refinement Control, Global Variables**. The **Object Inspector** can be used to look at the fit properties and agreement indices.*

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 (*Figure 17A*). Alternatively, you can open the 'Quantification' tab in the **Refinement Control** pane and view a pie chart of the quantification (*Figure 17B*).

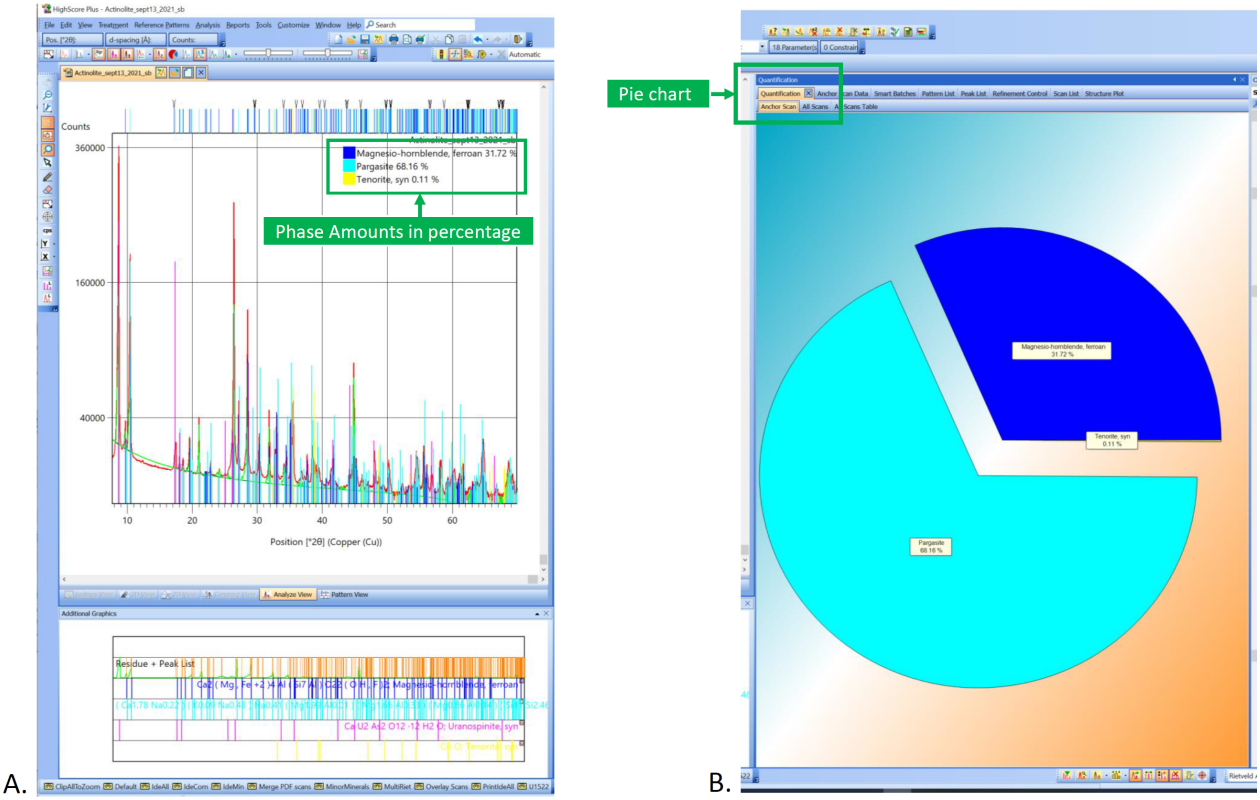

Figure 17. A. Phase amounts as a percentage are displayed on the main graphics window. B. Pie chart of the phase amounts for the quantification of the phases

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