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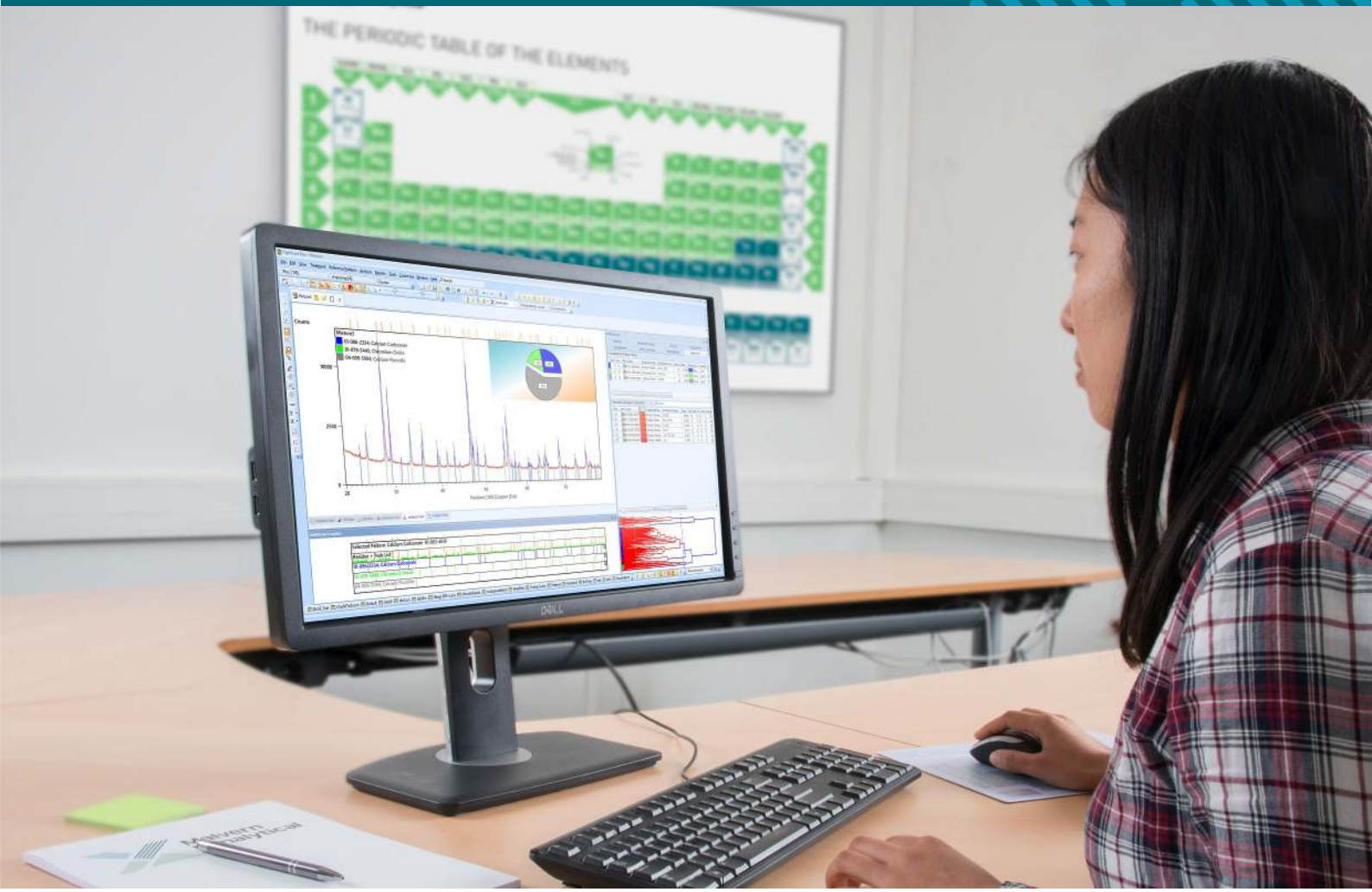
HIGHSCORE AND HIGHSCORE PLUS
QUICK START GUIDE



HIGHSCORE AND HIGHSCORE PLUS
QUICK START GUIDE

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This is the original publication of Edition 3 of this document, to be used with the HighScore and HighScore Plus version 4.9 or higher.

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CHAPTER 1 INTRODUCTION

1.1 Introduction

This Quick Start Guide helps you to get started with HighScore and HighScore Plus quickly.

The examples in this Quick Start Guide show you how to use the software to do simple tasks.

You will use the measurement "Mixture3.xrdml" for many examples in the Quick Start Guide. The sample used for this measurement is an artificial mixture of 3 minerals. The PANalytical Example Database, supplied with HighScore and HighScore Plus, is applicable to this measurement.

For all the tasks in the examples, there can be other possible procedures to complete them, but then you must experiment with HighScore or HighScore Plus and learn the other possibilities yourself.

There can be differences between the example screens in this Quick Start Guide and what you see on your screen. If that occurs, follow the instructions on your screen.

1.2 HighScore Plus

The software packages HighScore and HighScore Plus are almost the same, only that HighScore includes more functionality, such as Rietveld, Pawley, LeBail refinements and crystallographic analysis.

CHAPTER 2 GET STARTED

2.1 Introduction

In this chapter, you will do these tasks:

- Start the software.
- Set the desktop to default settings.
- Customize the desktop layout.
- Select PANalytical Example Database, which is supplied with the software.

2.2 Start the software

To start HighScore, use one of these procedures:

- On the desktop, double-click the icon  .
- Click the Windows Start button and then on the list of apps or programs, go to **PANalytical HighScore > HighScore**.

To start HighScore Plus, use one of these procedures:

- On the desktop, double-click the icon  .
- Click the Windows Start button and then on the list of apps or programs, go to **PANalytical HighScore Plus > HighScore Plus**.

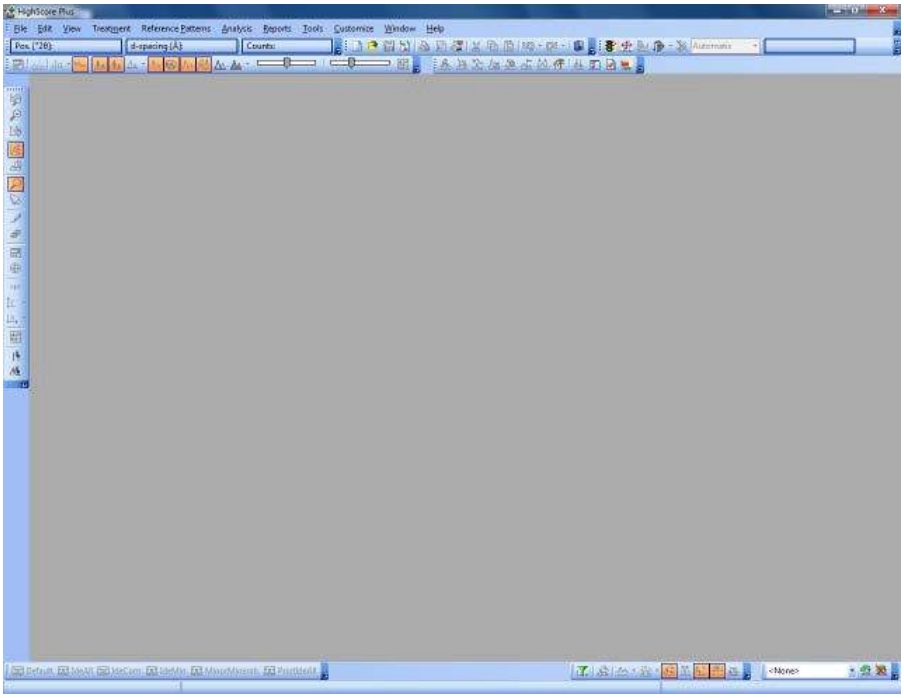


Figure 2.1 *Initial window of HighScore Plus*

2.3 Set the desktop to default settings

Before you start with the examples in this Quick Start Guide, we recommend that you set the desktop back to its default settings.

NOTE: After you completed the examples, you can change the settings to your preferences.

1. On the menu bar, go to **View > Toolbars > Customize** to open the **Customize** window.
2. Go to the **Options** tab.
3. Clear the **Show full menus after a short delay** check box.
4. Clear the **Menus show recently used commands first** check box.

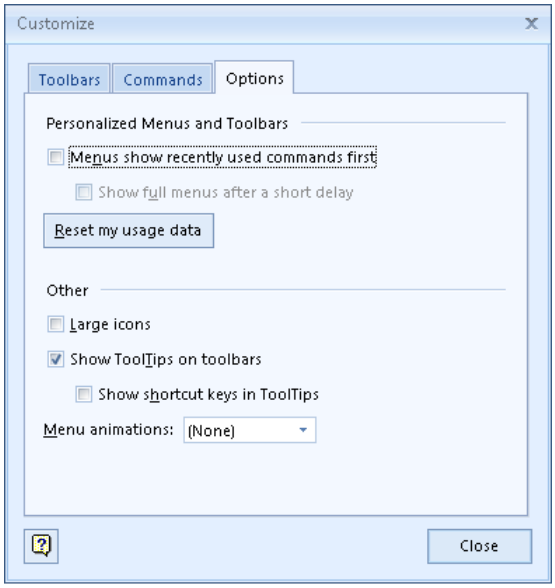


Figure 2.2 Customize window

5. Click **Close**.
6. On the menu bar, go to **Customize > Program Settings** to open the **Program Settings** window.
7. Click **Reset All to Default**.

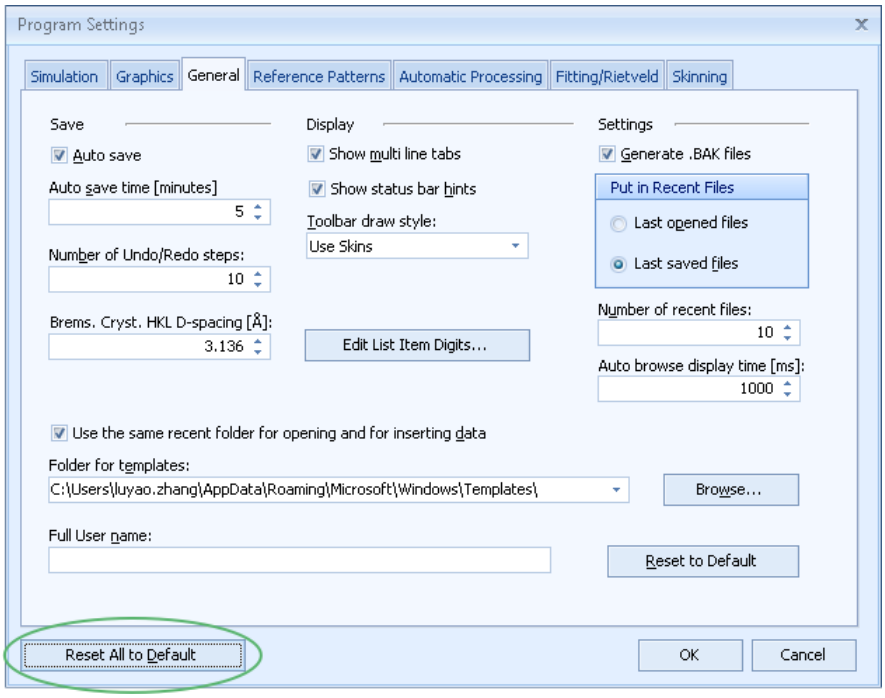


Figure 2.3 Reset All to Default

8. When the **Confirm** windows opens, click **Yes**.

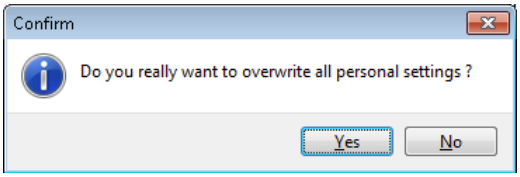


Figure 2.4 Confirm to overwrite all personal settings

9. In the **Program Settings** window, click **OK**.
10. On the menu bar, go to **Customize > Defaults** to open the **Default** window.
11. Click **Restore Factory Defaults**.

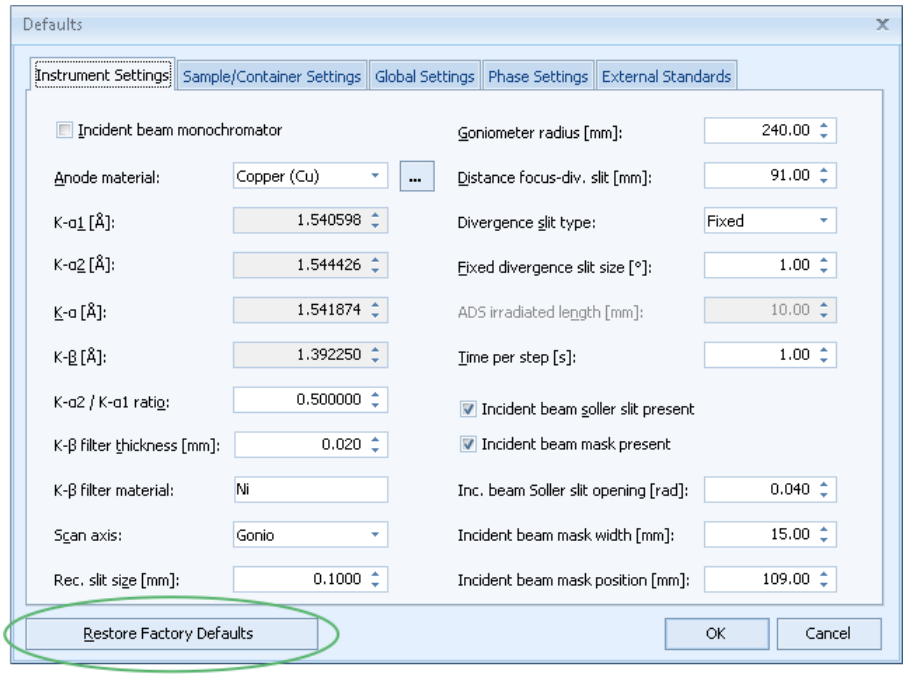


Figure 2.5 Restore Factory Defaults

12. Click **OK**.
13. On the menu bar, go to **File > New** to open a new empty document.
14. On the menu bar, go to **View > Reset all Toolbars**.
15. When the **Confirm** windows opens, click **Yes**.

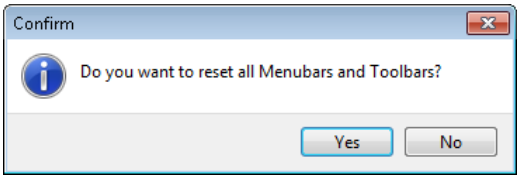


Figure 2.6 Confirm to reset all menu bars and tool bars

16. On the menu bar, go to **View > Panes Default Setting**.
17. On the menu bar, go to **View** and make sure that **Lock Pane Positions** is selected.

2.4 Customize the desktop layout

1. To show a toolbar or a pane on the desktop, on the menu bar, go to **View > Toolbars** and select the pane.
2. To hide a toolbar or a pane on the desktop, on the menu bar, go to **View > Toolbars** and clear the selection of the pane.
3. To adjust the relative size of a pane, drag the horizontal and vertical splitter bars of the pane.
4. To save changes to the desktop layout, do as follows:
   a. On the menu bar, go to **View > Desktop**.
   b. If you save the desktop layout under the current name, click **Save Desktop**.
   c. If you save the desktop layout under a new name, enter the name in the **Desktop Name** field and click **Save Desktop**.

5. To put different panes between 2 monitors, do as follows:
   a. On the menu bar go to **View**.
   b. Make sure that **Lock Pane Positions** is not selected.
   c. Put panes between 2 monitors.
   d. Save it as a desktop layout.
6. To set the desktop layout to a pre-programmed desktop layout, for example **Phase-ID**, do as follows:
   - On the **Desktop** toolbar, select **Phase-ID** in the **Desktop Name** field.

Figure 2.7 Select the desktop layout

   - Alternatively, on the menu bar, go to **View > Desktop** and select **Phase-ID**.

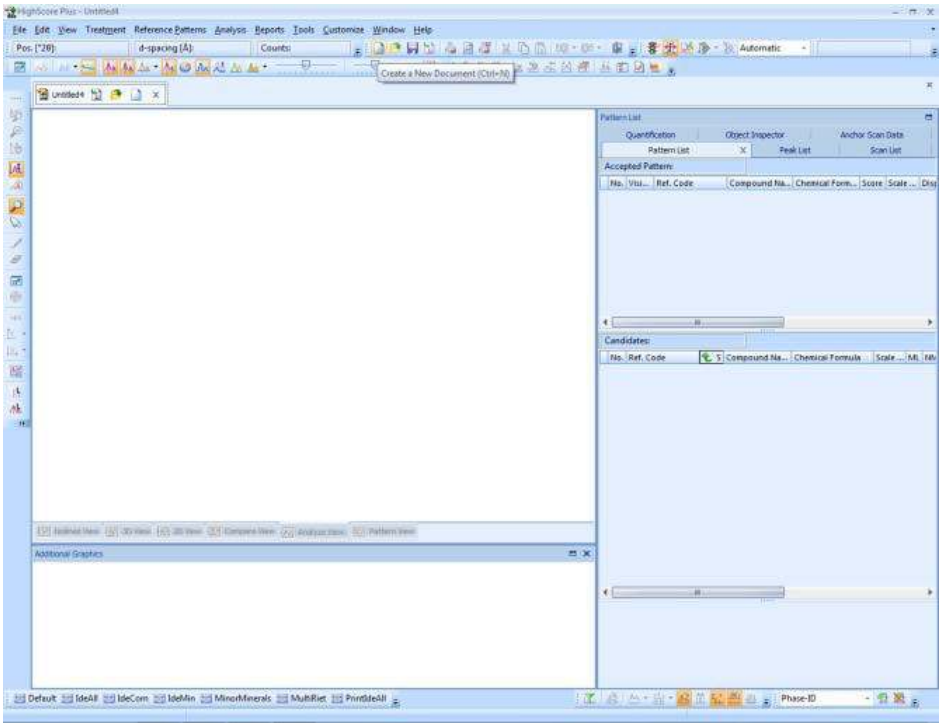


Figure 2.8 Desktop layout "Phase-ID"

You can switch among different desktop layouts and examine the differences in the desktop layouts.

NOTE: You can also set the desktop layout to **<None>** to use no pre-programmed desktop layout. Then the software will save your settings automatically when you close the last document.

For the examples in this chapter, we recommend that you set the desktop layout to **Phase-ID**, because a desktop layout with the **Main Graphics** pane at this size is the most usual layout to start with.

2.5 Get PANalytical Example Database prepared

A very small database PANalytical Example Database is supplied with the software. You will use it for the examples in the Quick Start Guide.

NOTE: Do not use PANalytical Example Database to do phase analysis on unknown samples.

Do not use PANalytical Example Database to do a test of the functionality or examine the phase identification capabilities of the software. Use a large reference database with at least 100,000 patterns instead, for example an ICDD product or the free COD database.

Do not add your own user reference patterns to PANalytical Example Database. Refer to the HighScore Help file for the information about how to make a new, empty reference database.

To get PANalytical Example Database prepared for patterns retrieval, do as follows:

1. On the menu bar, go to **Customize > Manage Databases** to open the **Manage Databases** window.
2. Examine if **PANalytical Example Database** is in the list.

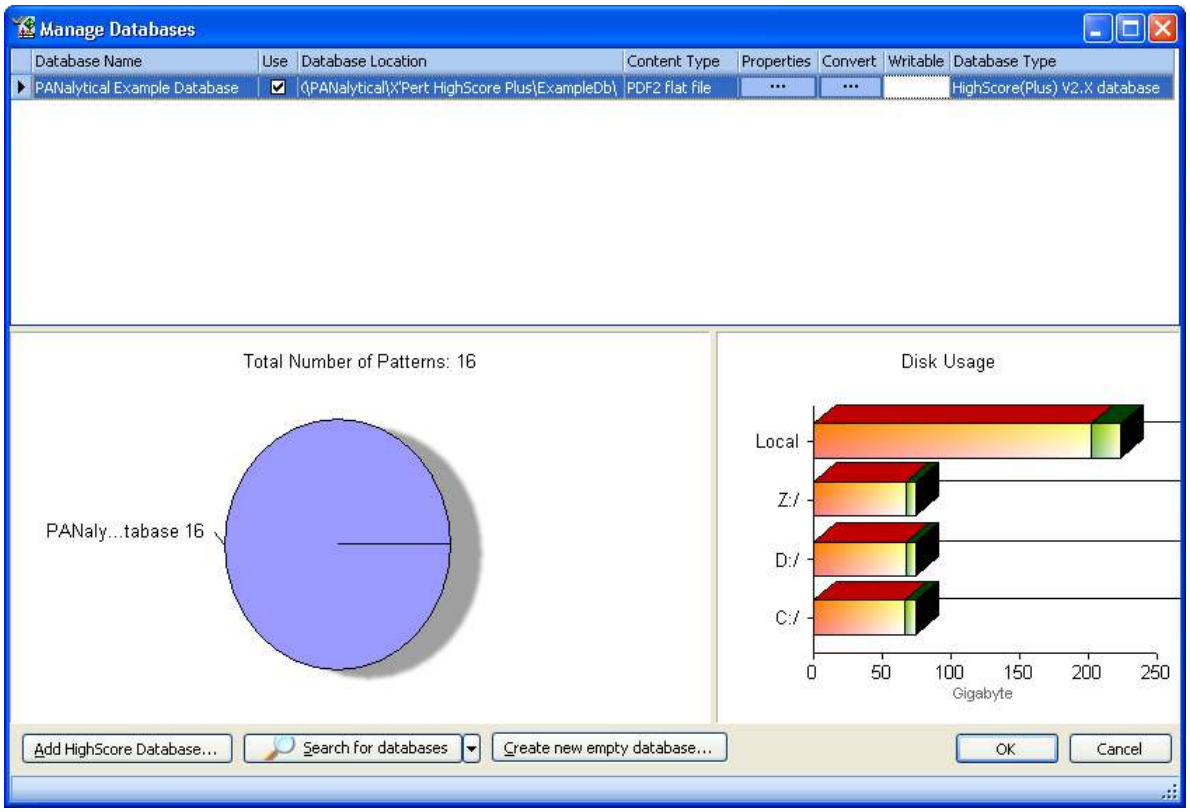


Figure 2.9 PANalytical Examine Database in the list

3. If **PANalytical Example Database** is not in the list, add it to the list:
   a. Right-click in the **Manage Databases** window.
   b. From the pop-up menu, select **Add HighScore Database**. The **Open** window opens.
   c. Open the folder **C:\Documents and Settings\user.name\My Documents\PANalytical \HighScore Plus\ExampleDb**.
   d. Select the file "Codes.pdb".

e. Click **Open**. **PANalytical Manage Database** is added to the list.

4. In the list, make sure that the **Use** check box is selected for **PANalytical Example Database**.
5. In the list, click **Properties** for **PANalytical Example Database**. If the properties are shown, the database can be used.

   NOTE: It is not necessary to convert PANalytical Example Database before you use it.

   In the **Manage Database** window, the pie chart shows the total number of patterns in the database.

6. Click × or go to **File > Close** on the menu bar to close the document.

CHAPTER 3 LOAD AND SHOW DATA

3.1 Introduction

The most frequent tasks in HighScore and HighScore Plus are to load and show data. In this chapter, you will do some simple examples.

There are more than 1 possible procedures to show data. Different views can also be used together.

3.2 Load a scan

1. If the software is not started, start it. Refer to Section 2.2.
2. On the menu bar, go to **File > Open**. The Open **window** opens.
3. Open the folder **C:\Documents and Settings\user.name\My Documents\PANalytical \X'Pert HighScore Plus\Tutorial**
4. In the **Open** window, select **All files (*.*)** from the drop-down list to show all files in this folder.

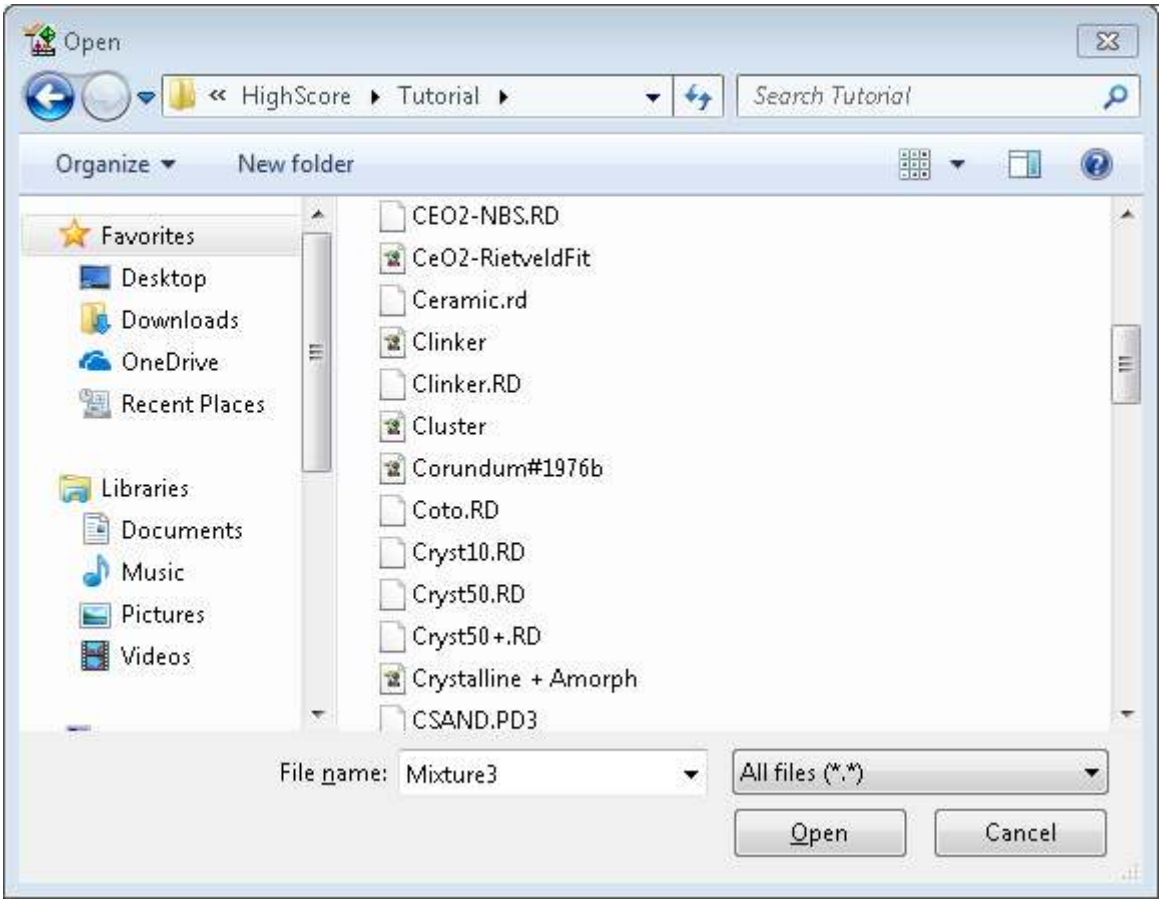


Figure 3.1 Tutorial folder with all files shown

5. Select the file "Mixture3.xrdml".
6. Click **Open** to open the file.

A copy of the selected file is loaded into a new document. Because this is the first scan of the document, it automatically becomes the anchor scan.

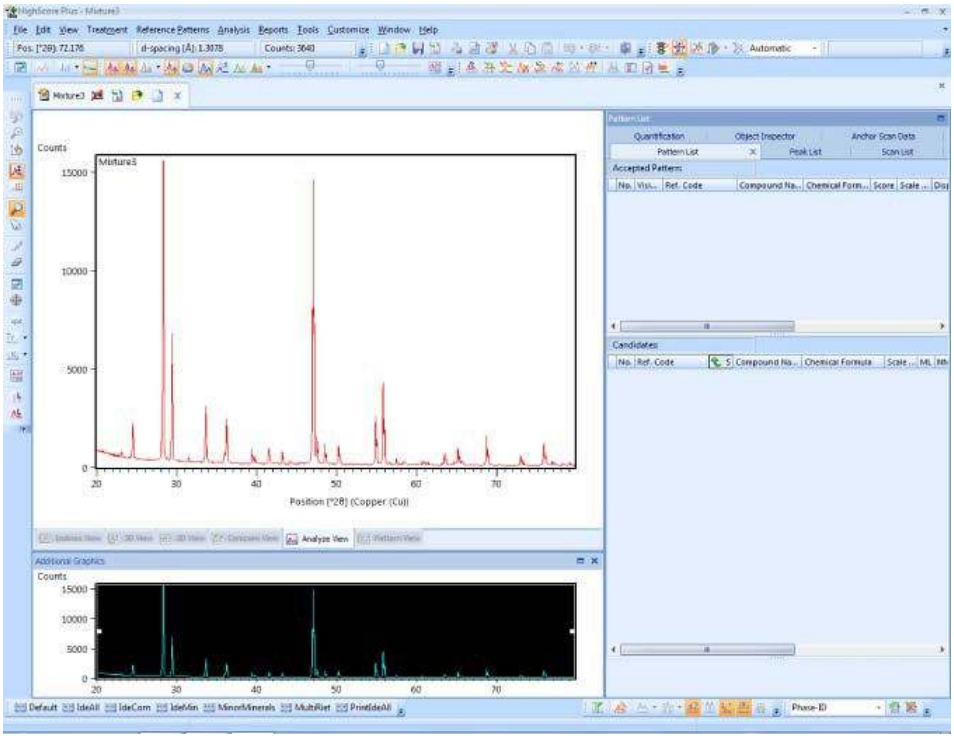


Figure 3.2 Mixture3.xrdml

3.3 Show a scan

3.3.1 About display panes

There are different panes in the software. Take the desktop **Phase-ID** as an example.

At the left side of the window, the **Main Graphics** pane shows the full anchor scan in **Analyze View**.

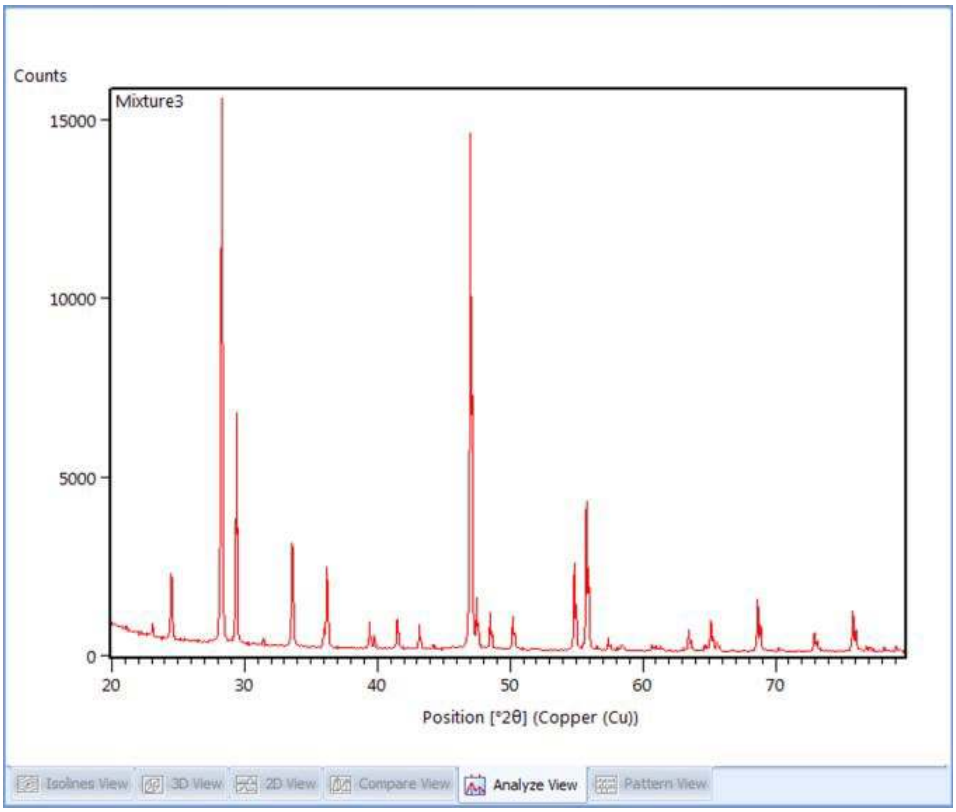


Figure 3.3 Main Graphics pane in Analyze view

Below the **Main Graphics** pane is the **Additional Graphics** pane. It shows where the zooming is done on the scan in the **Main Graphics** pane. If there is no zooming, it shows the full range of the scan in opposite colors.

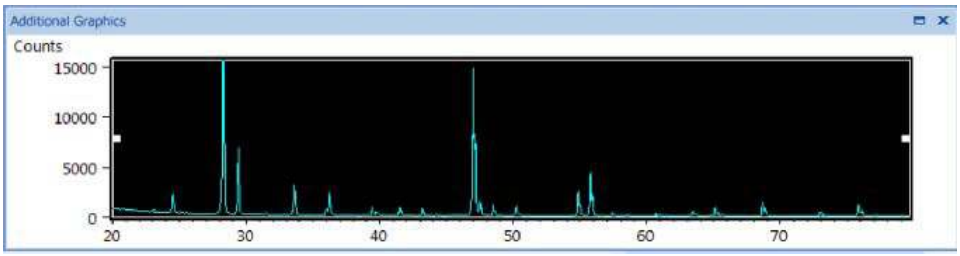


Figure 3.4 Full range in opposite colors

On the right side of the **Main Graphics** pane and **Additional Graphics** pane, there are more panes, for example the **Peak List** pane, the **Refinement Control** pane and the **Scan List** pane. The first time the software is used, the **Pattern List** pane is shown on top. Then the pane that is used the latest is shown on top.

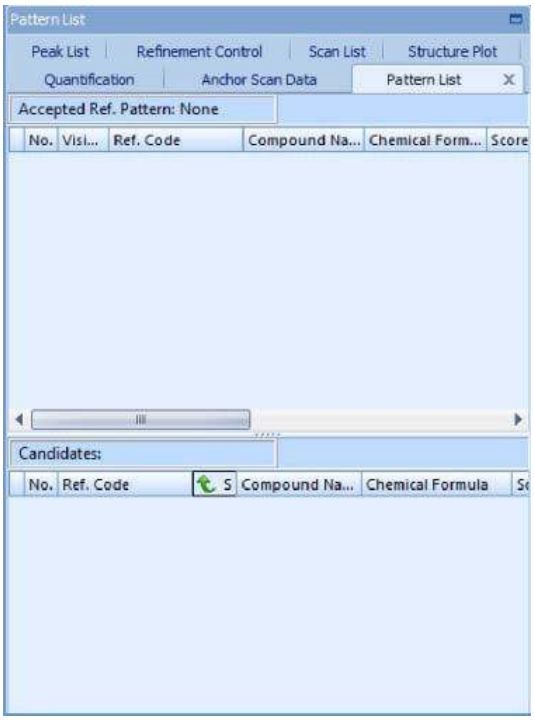


Figure 3.5 Pattern List pane on top of other panes

3.3.2 Use display panes

1. In the **Main Graphics** pane, drag a square on a peak to zoom in. Try to show only part of a peak and examine the changes of the graph in the **Additional Graphics** pane at the same time.
2. Double-click in the **Main Graphics** pane to zoom out.
3. Zoom in along the y-axis:

   a. On the **Tool Palette**, click  to switch on the **Zoom Intensity** function.

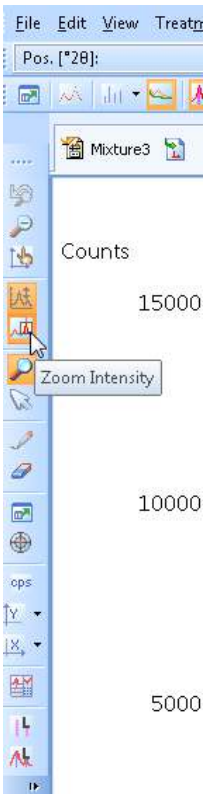


Figure 3.6 Zoom Intensity button

b. In the **Main Graphics** pane, drag a square along the y-axis. The graph is zoomed in along y-axis. Try to show only part of the peak and examine the changes of the graph in the **Additional Graphics** pane at the same time.

4. Set the scale of the y-axis:

a. On the **Tool Palette**, click the small arrow next to .

b. Select one of these scales:

- **Linear Y-Axis**.
- **Square Root Y-Axis**
- **Logarithmic Y-Axis**

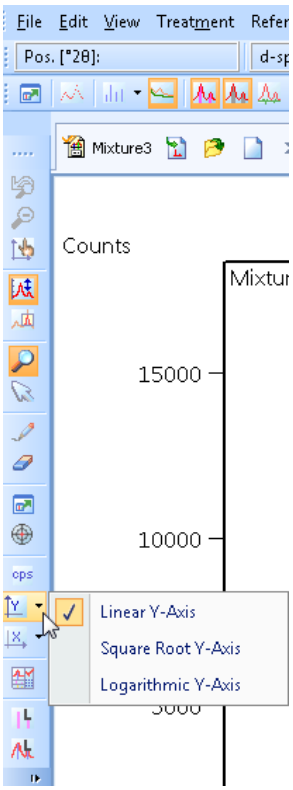


Figure 3.7 Set y-axis scale

3.4 Retrieve a reference pattern

1. On the menu bar, go to **Reference Patterns > Retrieve Pattern by > Restrictions**. The **Restrictions** window opens.
2. Go to the **Strings** tab.
3. In the **Mineral Name** field, enter "calcite".
4. Click **Load**.

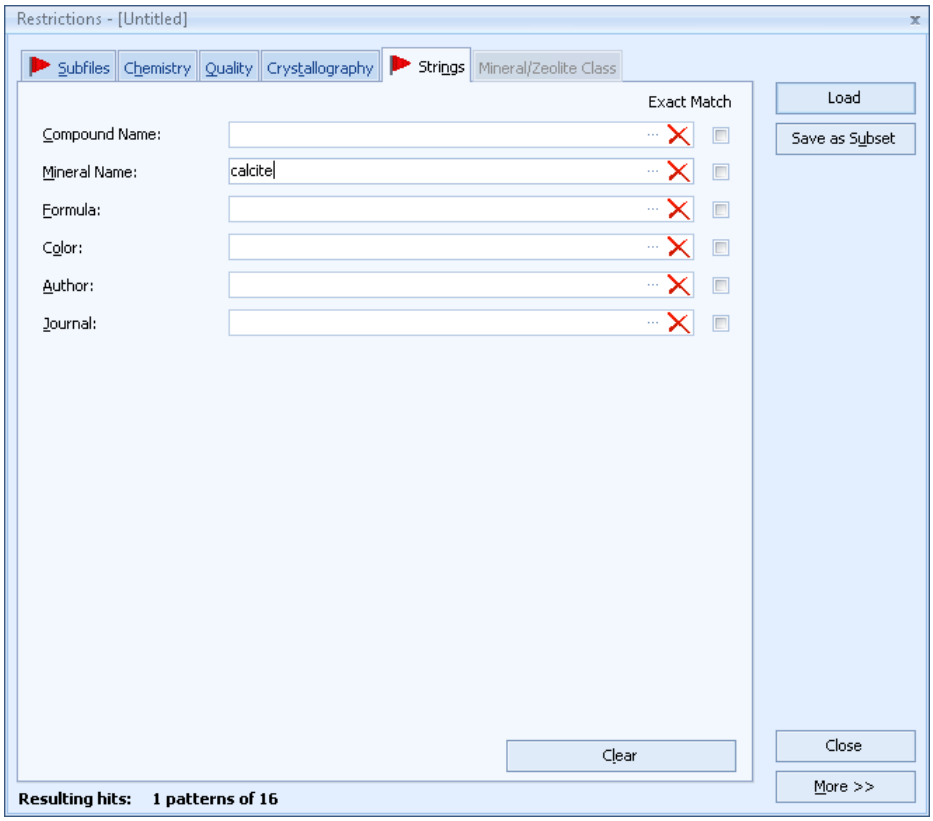


Figure 3.8 Retrieve a reference pattern by restrictions

5. Click **Close** to close the **Restrictions** window.
6. Examine these changes in the display panes:
   - The **Main Graphics** pane shows the reference pattern lines in **Analyze View**.
   - The **Additional Graphics** pane shows the reference pattern lines in most views.
   - The **Pattern List** pane shows a summary of the retrieved reference pattern.

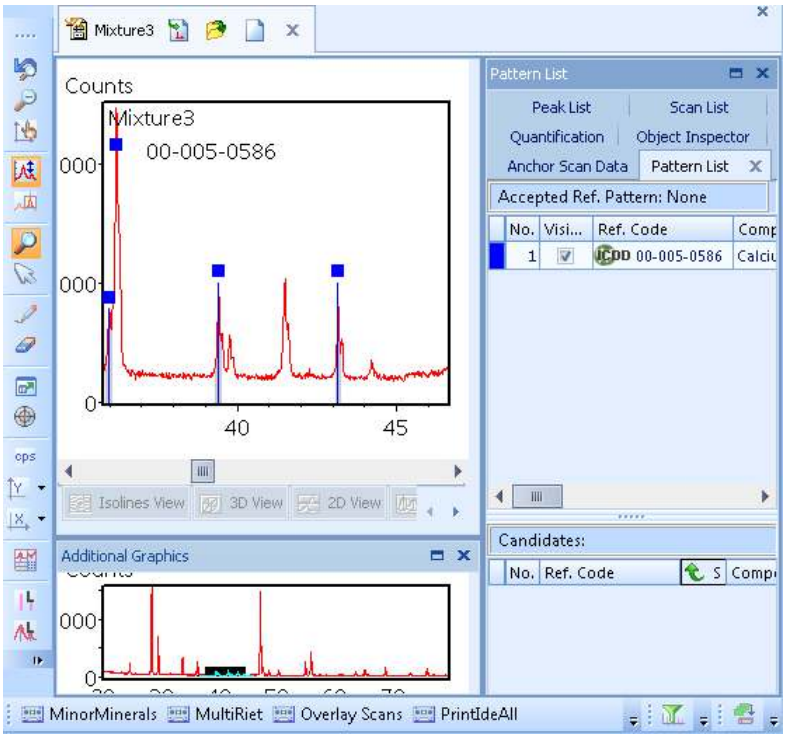


Figure 3.9 Reference pattern shown in display panes

7. In the **Main Graphics** pane, go to the **Pattern View** tab. The reference pattern lines are shown without the scan data. No peaks are shown in **Peak List**, because the **Peak List** pane is empty.

Figure 3.10 Reference pattern in Pattern View

3.5 Show a reference pattern

1. Go to the **Pattern List** pane.
2. Show the details of the reference pattern:
   - Right-click in the **Pattern List** pane and select **Show Pattern** from the pop-up menu.

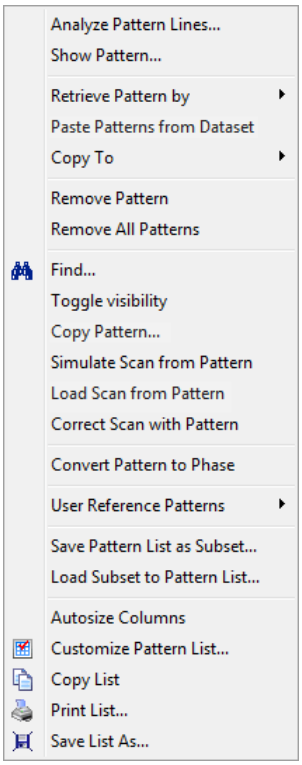


Figure 3.11 *Accepted reference patterns pop-up menu*

- Alternatively, in the **Pattern List** pane, double-click in the **Accepted Ref. Pattern** grid. The **Reference Pattern** window opens and shows the details of reference pattern.

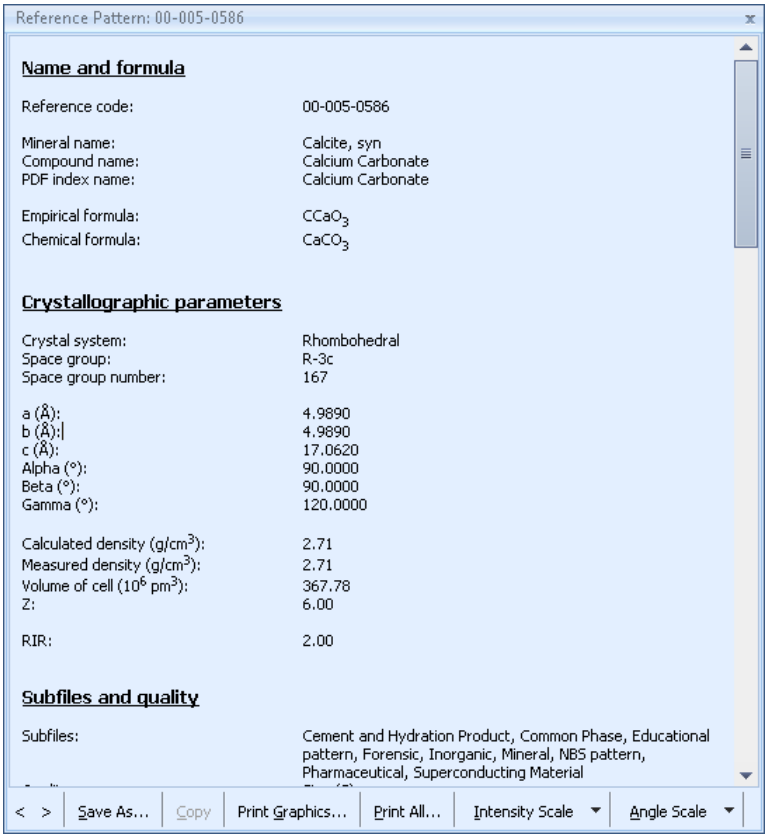


Figure 3.12 *Reference pattern details*

3. Close the **Reference Pattern** window.
4. Open the **Analyze View** again:
   - In the **Main Graphics** pane, go to the **Analyze View** tab.
   - Alternatively, on the menu bar, go to **View > Main Graphics > Analyze View**.
5. On the **Display Mode** toolbar, click  to switch on the **Show Reference Patterns** function. Then you can adjust the display of reference pattern lines in the **Main Graphics** pane.

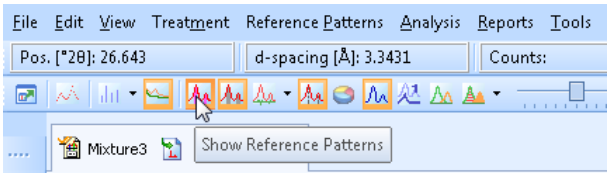


Figure 3.13 Show Reference Patterns button

NOTE: The high intensity areas of the scan marked in gray are those 'features' of the scan explained by the loaded reference pattern. The first 20 reference patterns are automatically matched and scored against the anchor pattern.

6. Save the document:
   a. On the menu bar, go to **File > Save Document**.
   b. Use these settings:

File name	Mixture3
Save as type	HighScore Plus (*.HPF)

   c. Click **Save**.
7. Click × or go to **File > Close** on the menu bar to close the document.

CHAPTER 4 USE PATTERN TREATMENTS

4.1 Introduction

Pattern treatment is used to prepare data for phase analysis and crystallographic analysis and sometimes for a structure refinement. With pattern treatment, you can make changes to the data and get additional derived data.

The 2 most important pattern treatments are to find background and peaks.

For phase analysis with the measured net profile data, it is very important to find background correctly. If you include peak data in the search-match-identification process or if you do profile fitting or indexing, peak search is necessary.

4.2 Find background

1. If the software is not started, start it. Refer to Section 2.2.
2. Open the document "Mixture3.xrdml". Refer to Section 3.2.

   NOTE: Be careful that you do not open the document "Mixture3.hpf".

3. On the menu bar, go to **Treatment > Determine Background** to find the background. The **Determine Background** window opens. The background is immediately found and shows as a bright green line in the **Main Graphics** pane.

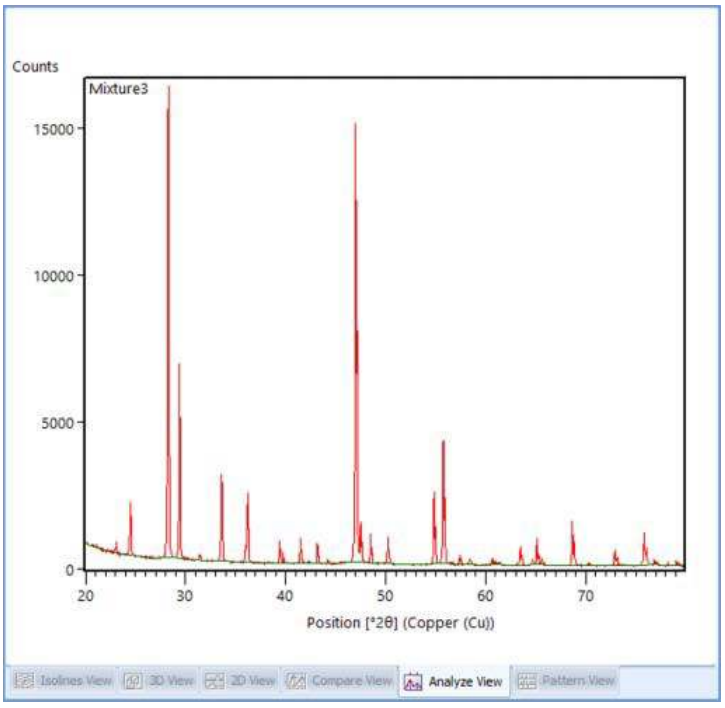


Figure 4.1 Anchor scan with the background

NOTE: Usually the software starts to process data only after you click a button, but after you go to **Treatment > Determine Background**, the software finds the background immediately.

4. Read the title bar of the **Determine Background** window. The title bar shows the parameter set that you use.

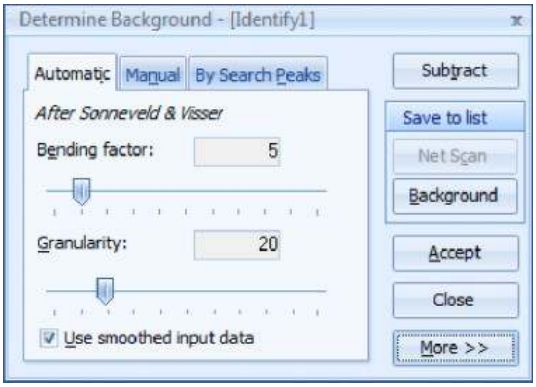


Figure 4.2 Use the Identify1 parameter set

5. Make sure that you use the **Identify1** parameter set.
6. If not, change the parameter set to **Identify1**:
   a. Click **More** to expand the window.
   b. In the **Select Parameter Set** field, select **Identify1** from the drop-down list.
7. Change the bending factor and examine how the background changes:
   a. On the **Automatic** tab, write down the value in the **Bending factor** field. You will set **Bending factor** back to this value later.
   b. Move the slider below the **Bending factor** field to change its value and examine how the background changes in the **Main Graphics** pane at the same time.
   c. If necessary, zoom in to examine small changes.
8. Change the granularity and examine how the background changes:
   a. On the **Automatic** tab, write down the value in the **Granularity** field. You will set **Bending factor** back to this value later.

      NOTE: This parameter changes the number of intervals which are used to find the background. The default value "20" can be used for most scans.

   b. Move the slider below the **Granularity** field to change its value and examine how the background changes in the **Main Graphics** pane at the same time.
   c. If necessary, zoom in to examine small changes.
9. Read the title bar of the **Determine Background** window again. At this time, the title is changed to **Determine Background - [Untitled]**. This shows the parameter set is changed and not saved under a specific name.
10. Set the **Bending factor** and **Granularity** fields back to the initial values that you wrote down in steps 7 and 8.
11. Click **Accept** to accept the background. The accepted background shows in the **Main Graphics** pane.

    NOTE: The color of the background is set on the **Graphic Colors** tab in the **Document Settings** window. Right-click in the **Main Graphics** pane and select **Document Settings** from the pop-up menu to open the **Document Settings** window.

12. Examine the anchor scan data:
    a. Go to the **Anchor Scan Data** pane.

b. Examine the data in the **Iback (cts)** column. This is the background data that you just made.

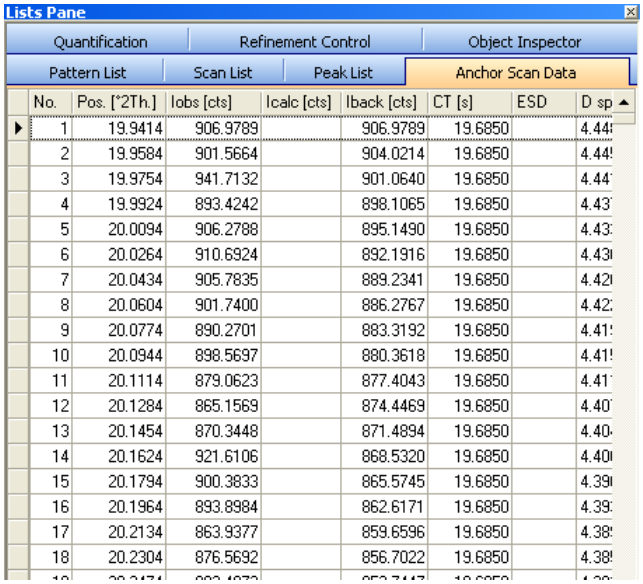


Figure 4.3 Anchor Scan Data tab

4.3 Search peaks

1. On the menu bar, go to **Treatment > Search Peaks** to open the **Search Peaks** window.
2. Read the title bar of the **Search Peaks** window. The title bar shows the parameter set that you use.

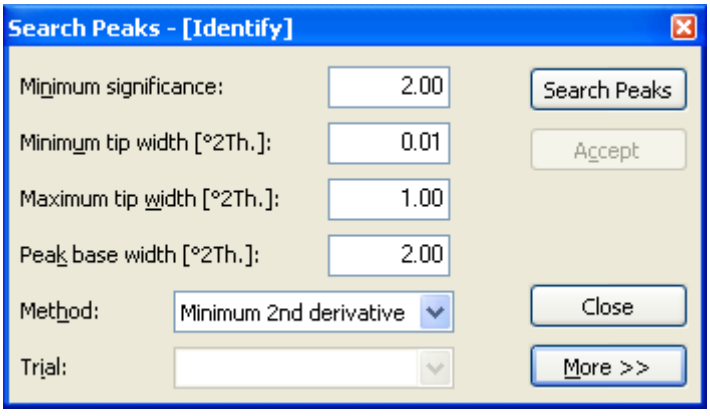


Figure 4.4 Use the Identify parameter set

3. Make sure that you use the **Identify** parameter set.
4. If not, change the parameter set to **Identify**:
   a. Click **More** to expand the window.
   b. In the **Select Parameter Set** field, select **Identify** from the drop-down list.
5. Click **Search Peaks**. The peaks are found and are shown in the **Main Graphics** pane:

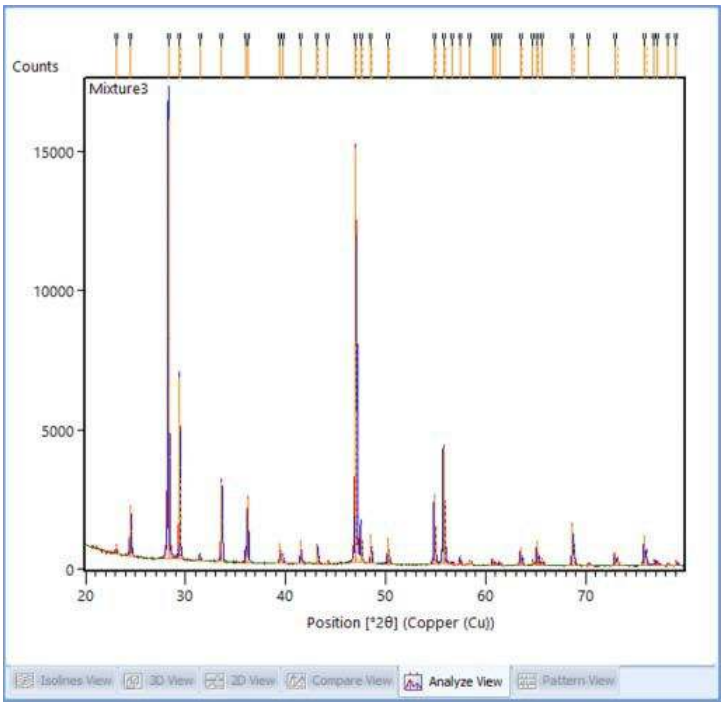


Figure 4.5 Anchor scan with peaks and background data

- Solid lines: $K\alpha_1$ and $K\alpha_{mixed}$ peaks.
- Dashed lines: $K\alpha_2$ peaks.
- **V** marks above: peaks that are not explained by a reference pattern
- Blue line: the calculated profile

  NOTE: The default color of the calculated profile is blue. The color can be changed on the **Graphic Colors** tab in the **Document Settings** window. Right-click in the Main Graphics pane and select **Document Settings** from the pop-up menu to open the **Document Settings** window.

  This is only a preview of the result. If you close the window, no peaks are written into the diffraction document. You can do a check of the quality of the peak search by the peak lines and the profile before you accept any result. You can zoom in for more details.

6. In the **Search Peaks** window, click **Accept** to add the peaks that are found to the document.
7. Change the display of peaks:
   - On the **Display Mode** toolbar, click the small arrow next to  to select the display of peaks.

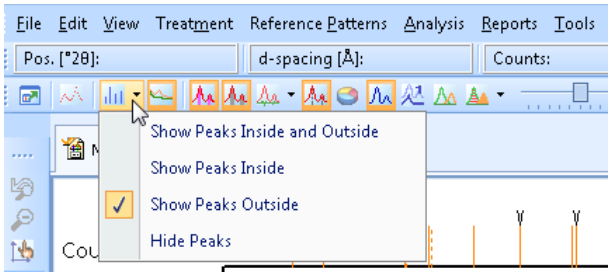
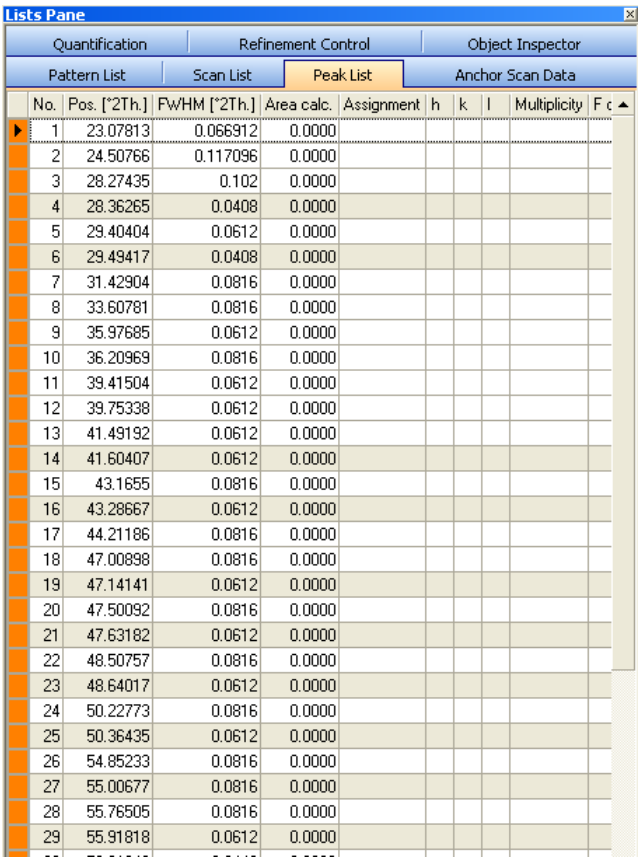


Figure 4.6 Display of peaks

- Alternatively, click  to change the display of peaks. Each time you click, the display of peaks is changed.

8. Go to the **Peak List** pane to examine the details of each peak. Peaks derived from the $K\alpha_2$ wavelength are shown with gray background.



No.	Pos. [°2Th.]	FWHM [°2Th.]	Area calc.	Assignment	h	k	l	Multiplicity	F c
1	23.07813	0.066912	0.0000						
2	24.50766	0.117096	0.0000						
3	28.27435	0.102	0.0000						
4	28.36265	0.0408	0.0000						
5	29.40404	0.0612	0.0000						
6	29.49417	0.0408	0.0000						
7	31.42904	0.0816	0.0000						
8	33.60781	0.0816	0.0000						
9	35.97685	0.0612	0.0000						
10	36.20969	0.0816	0.0000						
11	39.41504	0.0612	0.0000						
12	39.75338	0.0612	0.0000						
13	41.49192	0.0612	0.0000						
14	41.60407	0.0612	0.0000						
15	43.1655	0.0816	0.0000						
16	43.28667	0.0612	0.0000						
17	44.21186	0.0816	0.0000						
18	47.00898	0.0816	0.0000						
19	47.14141	0.0612	0.0000						
20	47.50092	0.0816	0.0000						
21	47.63182	0.0612	0.0000						
22	48.50757	0.0816	0.0000						
23	48.64017	0.0612	0.0000						
24	50.22773	0.0816	0.0000						
25	50.36435	0.0612	0.0000						
26	54.85233	0.0816	0.0000						
27	55.00677	0.0816	0.0000						
28	55.76505	0.0816	0.0000						
29	55.91818	0.0612	0.0000						

Figure 4.7 Peak List pane

9. Save the document:
   a. On the menu bar, go to **File > Save As**.
   b. Use these settings:

File name	Mixture3
Save as type	HighScore Plus (*.HPF)

   c. Click **Save**.
   d. If the **Confirm Save As** window opens, click **Yes**.

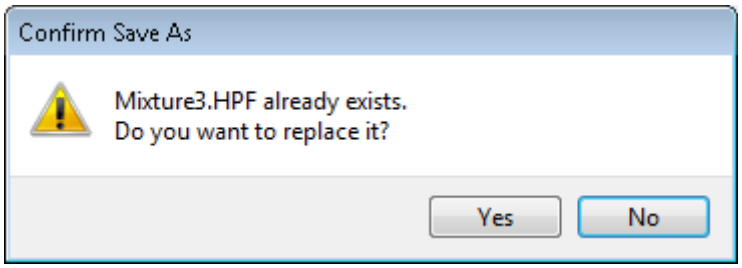


Figure 4.8 Confirm Save As window

The document is saved with the background data and the peak data.

CHAPTER 5 DO AN AUTOMATIC PROFILE FITTING

5.1 Introduction

In this chapter, you will fit a profile with an automatic fit parameter set. This is called non-phase peak fitting or single peaks profile fitting.

To do a non-phase peak fitting is to put a calculated profile around each peak and make each peak follow the original measured scan data as close as possible.This mathematical description of the profile shows peak parameters much better than a simple peak search. It is used for peak deconvolution, unit cell refinements, line profile analysis or structure solution.

For phase identification, it is usually not necessary to do a non-phase peak fitting.

5.2 Prepare for an automatic profile fitting

1. If the software is not started, start it. Refer to Section 2.2.
2. On the menu bar, go to **File > Open** to open the document "Mixture3.hpf" that you saved. Refer to Section 4.3. "Mixture3.hpf" contains background data, peak data and profile data, but the profile data is calculated from the peak data and not fitted and not refined.
3. In the **Additional Graphics** pane, show the differences between measured scan data and profile data:
   a. Right-click in the **Additional Graphics** pane.
   b. From the pop-up menu, go to **Show Graphics**.
   c. Select **Difference Plot**.
4. In the **Main Graphics** pane, show the calculated sum profile in **Analyze View**:
   - On the **Display Mode** toolbar, click  to switch on the **Show Calculated Profile in Analyze View** function.

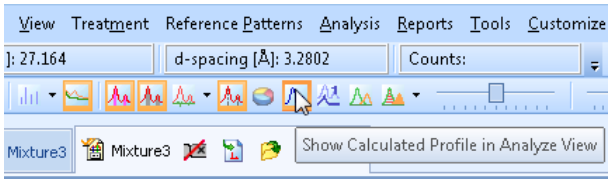


Figure 5.1 Show Calculated Profile in Analyze View button

   - Alternatively, on the menu bar, go to **View > Display Mode** and select **Show Calculated Profile**.

     The the calculated sum profile shows in the **Analyze View** in the **Main Graphics** pane.
5. On the **Desktop** toolbar, set the desktop layout to **Profile Fitting**.

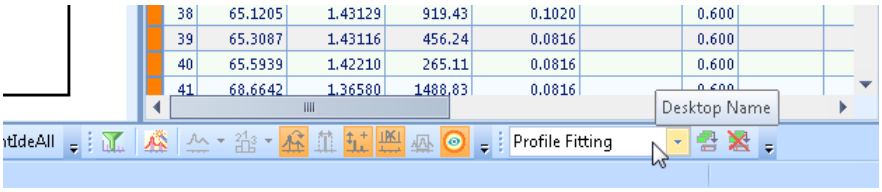


Figure 5.2 Select the desktop layout

6. Make sure that the background is used and not changed during profile fitting:
   a. Go to **Refinement Control** pane.
   b. Select **Background** to open the **Object Inspector** pane.
   c. In the row of **Method**, click the arrow at the end to open the drop-down list.
   d. Select **Use available background**.

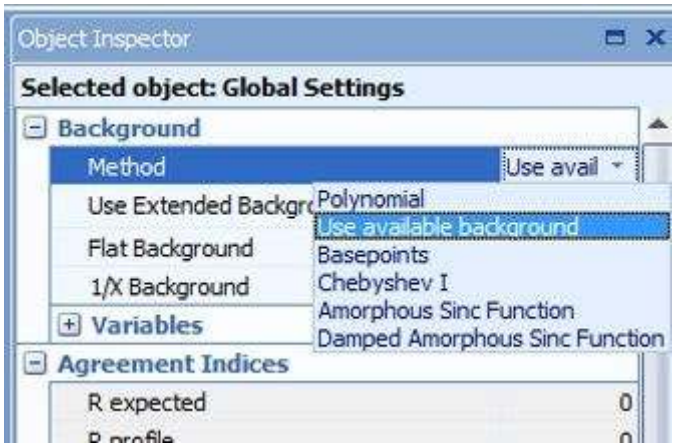


Figure 5.3 Use available background

   e. Press **Enter** to save the change.
7. Set the fitting mode to **Automatic**:
   - On the **Fitting** toolbar, select **Automatic**.

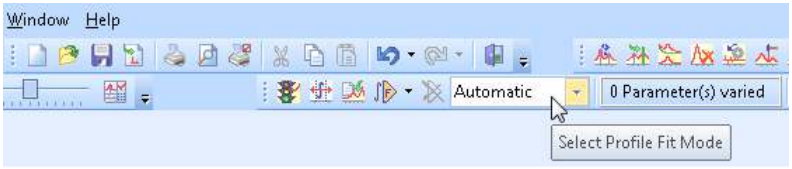


Figure 5.4 Select the fitting mode

   - Alternatively, on the menu bar, go to **Analysis > Fitting** and set **Fitting Mode** to **Automatic**.

5.3 Start an automatic profile fitting

1. Make sure that you prepared for the automatic profile fitting correctly. Refer to Section 5.2.
2. Make sure that the profile fitting will be done to the full range of the scan:
   - Fully zoom out the scan in the **Main Graphics** pane. Profile fitting is only done to the part of the range that is shown.

- Alternatively, on the **Fitting** toolbar, click to switch on the **Ignore actual Zoom Range for fitting** function, or go to **Analysis > Fitting** on the menu bar and select **Ignore Zoom Range**.

Figure 5.5 Ignore actual Zoom Range for fitting button

3. Start an automatic profile fitting:
   - On the **Fitting** toolbar, click and select **<Profile fit> Default Profile Fit**.

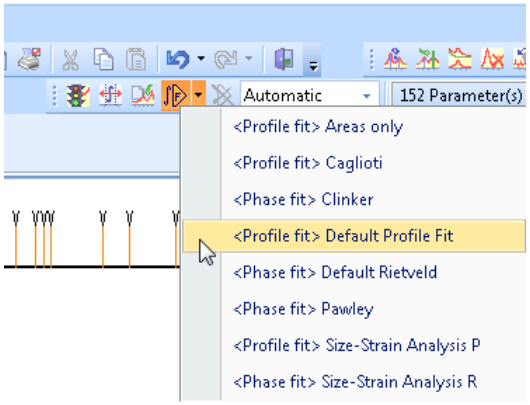


Figure 5.6 Select <Profit fit> Default Profile Fit

   - Alternatively, on the menu bar, go to **Analysis > Fitting > Start Fit** and select **<Profile fit> Default Profile Fit**.

     The profile fitting starts. The **R-Value** window opens.

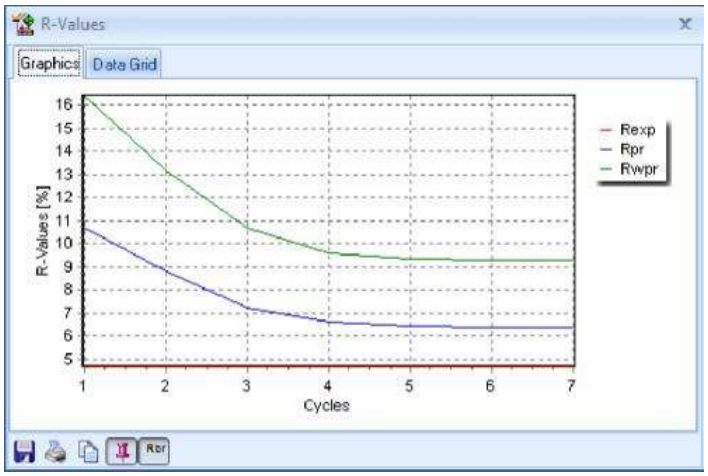


Figure 5.7 R-Value window

4. Examine how the overall quality of the fitting changes during the fitting process.

   After the profile fitting is completed, the profile is fitted to the peaks. The background is used and not changed. The background is high and cuts into the peak feet. This is good for phase identification, but is not optimal for profile fitting.

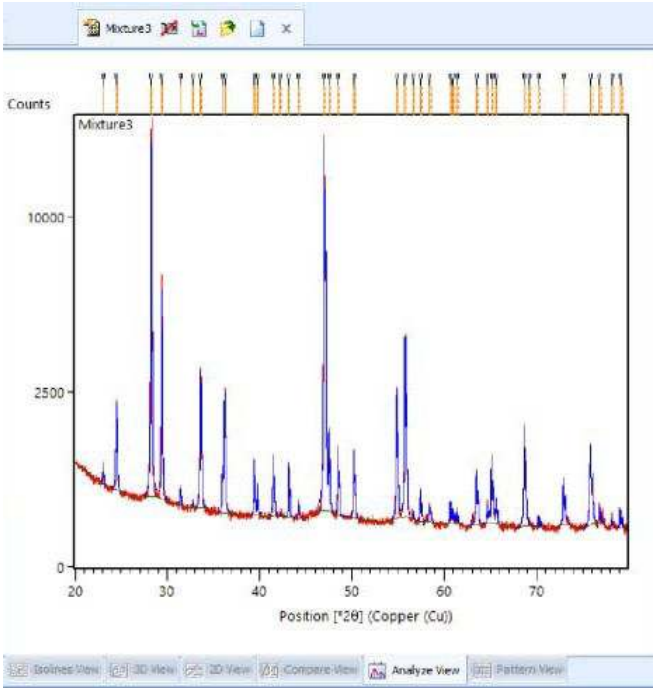


Figure 5.8 Anchor scan and profile after the first profile fitting

5. Examine the agreement indices:
   a. Go to the **Refinement Control** pane.
   b. Double-click **Global Variables** to open the **Object Inspector** pane.
   c. Open **Agreement Indices**.
   d. Examine the R-values and the value of **Goodness of Fit**.

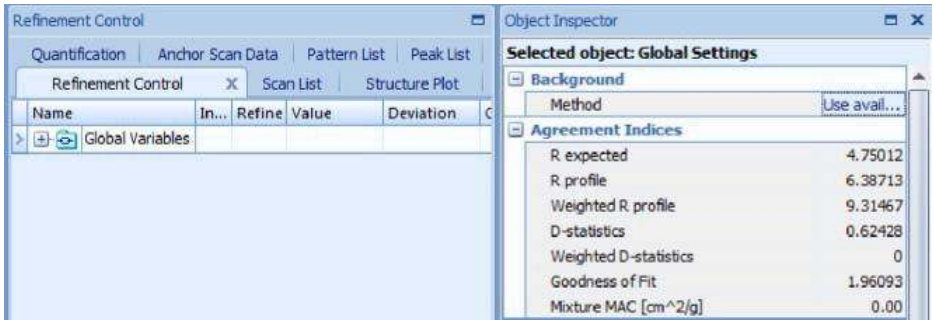


Figure 5.9 Agreement Indices after the first profile fitting

6. Change the settings which are used to find the background:
   a. On the menu bar, go to **Treatment > Determine Background**.
   b. In the **Determine Background** window, change these settings:

Bending factor	0
Granularity	30
Use smoothed input data	Not selected

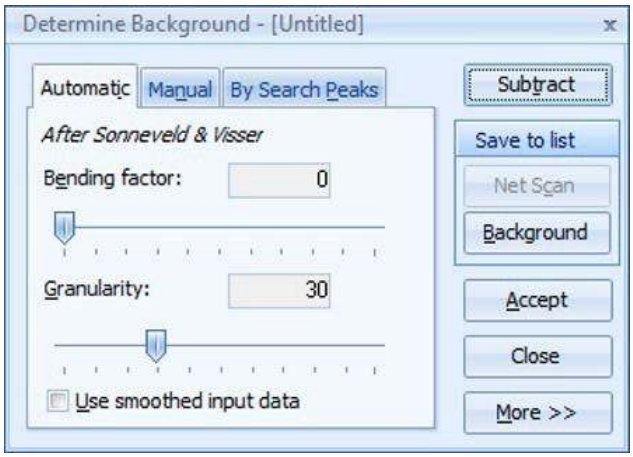


Figure 5.10 New settings

c. Click **Accept** to save the new settings.

7. Set the asymmetry type for peak fitting:
   a. Go to the **Refinement Control** pane.
   b. Double-click **Global Variables** to open the **Object Inspector** pane.
   c. Open **Unassigned Peaks Fitting**.
   d. Open **Common Peak Fit Settings**.
   e. In the row of **Asymmetry Type** , click the arrow at the end to open the drop-down list.
   f. Select **Split Width**. This shows the small asymmetry of the peaks at low angles better.

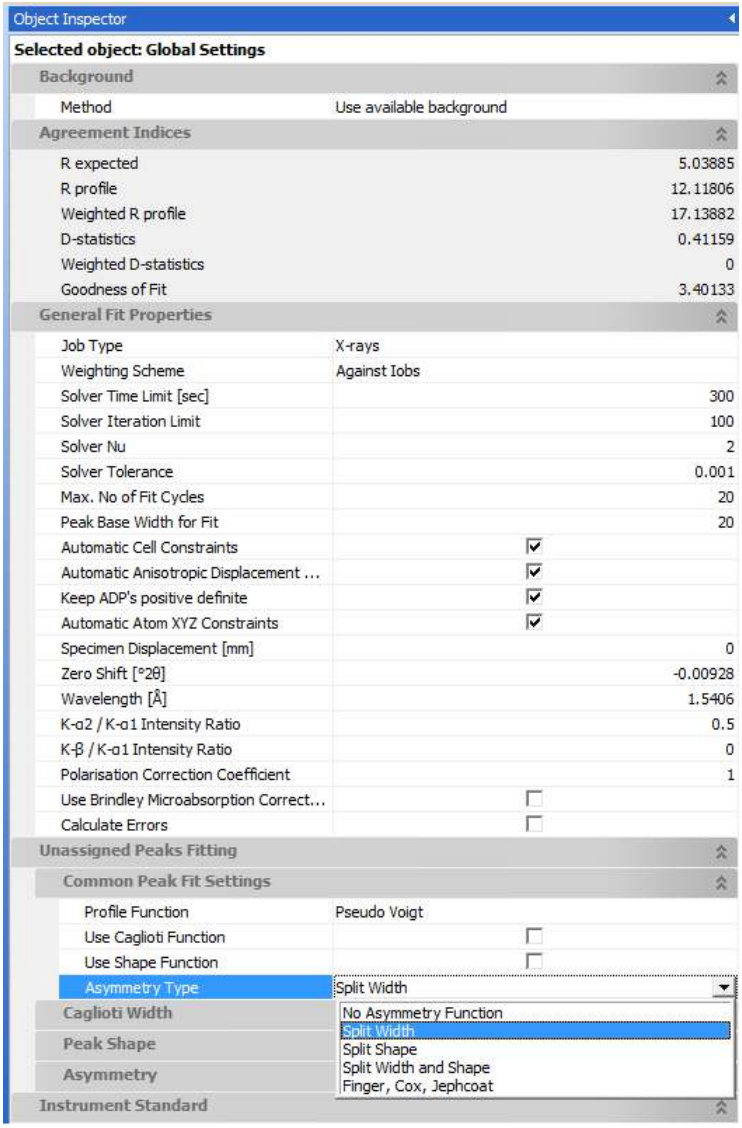


Figure 5.11 Select Spit Width

g. Press **Enter** to save the change.

8. Do step 3 to start an automatic profile fitting again with **<Profile fit> Default Profile Fit**. The profile fitting becomes better this time.

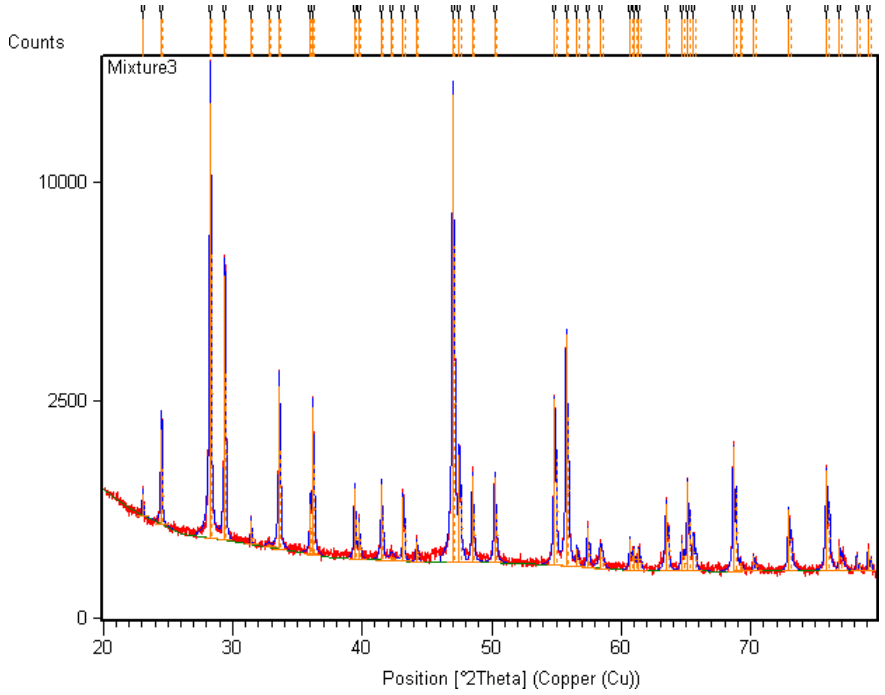


Figure 5.12 Anchor scan and profile with better fitting

9. Examine the agreement indices:
   a. Go to the **Refinement Control** pane.
   b. Double-click **Global Variables** to open the **Object Inspector** pane.
   c. Open **Agreement Indices**.
   d. Examine the values of **Weighted R profile** and **Goodness of Fit**:
      - The value of **Weighted R profile** must be approximately 6.80.
      - The value of **Goodness of Fit** must be approximately 1.44.

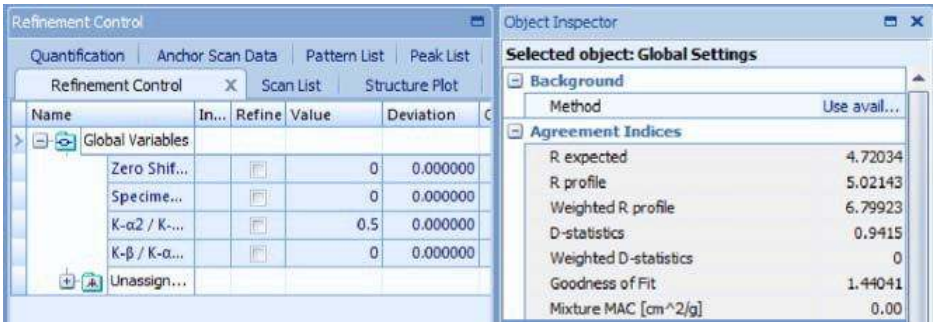


Figure 5.13 Agreement Indices with better fitting

10. Click × or go to **File > Close** on the menu bar to close the document. Do not save the changes.

CHAPTER 6 DO SEARCH - MATCH - IDENTIFY

6.1 Introduction

In this chapter, you will search and match possible candidates and then manually identify the phases of the example document. You will use PANalytical Example Database, which is supplied with the software. This gives you a good example of what to do in real work.

NOTE: Do not use PANalytical Example Database to do a test of the functionality or examine the phase identification capabilities of the software. Use a large reference database with at least 100,000 patterns instead, for example an ICDD product or the free COD database.

6.2 Search and match

1. If the software is not started, start it. Refer to Section 2.2.
2. On the menu bar, go to **File > Open** to open the document "Mixture3.hpf" that you saved. Refer to Section 4.3. "Mixture3.hpf" contains background data, peak data and profile data, but the profile data is calculated from the peak data and not fitted and not refined.
3. On the **Desktop** toolbar, set the desktop to **Phase-ID**.

Figure 6.1 Set the desktop layout

4. On the menu bar, go to **Analysis > Search & Match > Execute Search & Match**. The **Search & Match** window opens.
5. Read the title bar of the **Search & Match** window. The title bar shows the parameter set that you use.
6. Make sure that you use the **Default** parameter set.

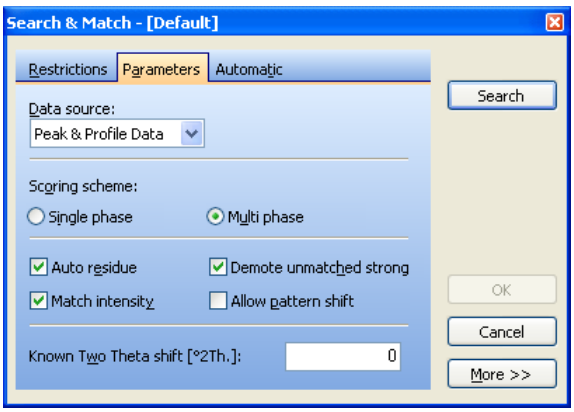
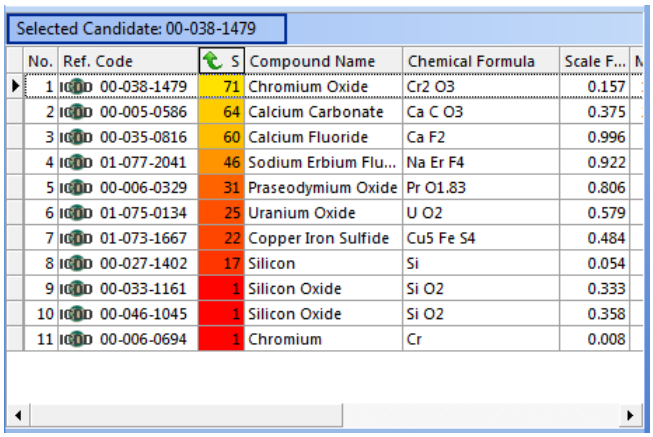


Figure 6.2 Use the Default parameter set

7. If not, change the parameter set to **Default**:
   a. Click **More** to expand the window.
   b. In the **Select Parameter Set** field, select **Default** from the drop-down list.
8. In the **Search & Match** window, click **Search**. The scan shows in the **Additional Graphics** pane in the **Compare Mode** view.
9. Go to the **Pattern list** pane. The **Candidates** list in the lower part of the pane gives a preview of the candidates.



No.	Ref. Code	S	Compound Name	Chemical Formula	Scale F...
1	00-038-1479	71	Chromium Oxide	Cr2 O3	0.157
2	00-005-0586	64	Calcium Carbonate	Ca C O3	0.375
3	00-035-0816	60	Calcium Fluoride	Ca F2	0.996
4	01-077-2041	46	Sodium Erbium Flu...	Na Er F4	0.922
5	00-006-0329	31	Praseodymium Oxide	Pr O1.83	0.806
6	01-075-0134	25	Uranium Oxide	U O2	0.579
7	01-073-1667	22	Copper Iron Sulfide	Cu5 Fe S4	0.484
8	00-027-1402	17	Silicon	Si	0.054
9	00-033-1161	1	Silicon Oxide	Si O2	0.333
10	00-046-1045	1	Silicon Oxide	Si O2	0.358
11	00-006-0694	1	Chromium	Cr	0.008

Figure 6.3 Candidates list

10. If necessary, click the column header **Score** to change the sequence of the candidates by score.
11. In the **Search & Match** window, click **OK** to accept the result.

6.3 Identify

In this section, you will manually accept candidates that have high scores and that match the peaks and features of the measurement. Some views in the **Additional Graphics** pane support a visual comparison of reference pattern lines and the measurement.

1. Set the **Additional Graphics** pane to the **Compare Mode** view:
   - On the menu bar, go to **View > Additional Graphics** and select **Compare Mode**.
   - Alternatively, right-click in the **Additional Graphics** pane, from the pop-up menu, go to **Show Graphics** and select **Compare Mode**.
2. Set the display of peaks to **Show Peaks Outside**:
   - On the **Display Mode** toolbar, click the small arrow next to  and select **Show Peaks Outside**.

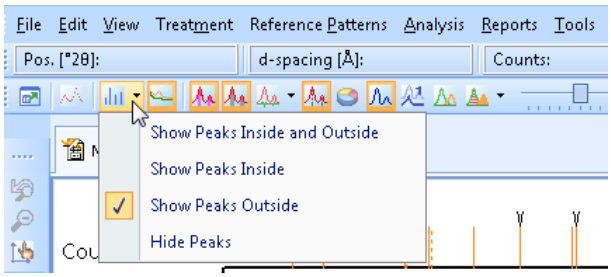


Figure 6.4 Select Show Peaks Outside

- Alternatively, on the menu bar, go to **View > Display Mode > Peaks in Main Graphics** and select **Show Peaks Outside**.

3. Hide the calculated profile to show the measured scan in a better view:
   - Go to to **View > Display Mode** and make sure that **Show Calculated Profile** is not selected.
   - Alternatively, make sure that on the **Display Mode** toolbar is not selected.

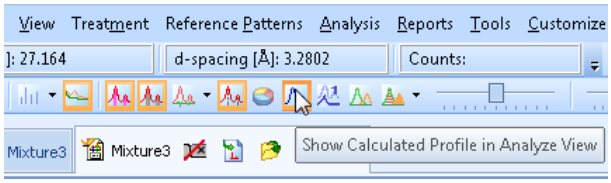


Figure 6.5 Show Calculated Profile in Analyze View button

4. Go to the **Pattern List** pane.
5. Drag the first candidate pattern "00-038-1479, Chromium Oxide" from the **Candidates** list to the **Accepted Ref. Patterns** list to accept it. The **Accepted Ref. Patterns** list is above the **Candidates** list. When you select this pattern, it is highlighted and its lines are shown for comparison in the **Additional Graphics** pane.
6. Examine these changes in the display panes:
   - Some peaks in the **Main Graphics** pane lost the **V** mark. These peaks are explained by the accepted reference pattern. Peaks marked with **V** marks are still not explained.
   - High intensity areas in the **Main Graphics** pane are shown in gray. These scan features are explained by the accepted reference pattern.

     NOTE: If no scan features have gray marks, click in the **Display Mode** toolbar to switch on the **Show Explained Features** function.

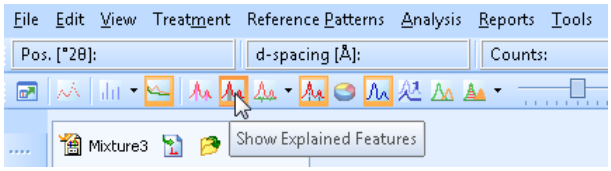


Figure 6.6 Show Explained Features button

   - In the **Candidates** list, the candidate "00-005-0586, Calcium Carbonate" moves to the top. The score of this pattern has a small difference than before.

     NOTE: If necessary, go to **Edit > Undo** and **Edit > Redo** to make these changes occur again to examine them.

7. Drag the top 2 patterns from the **Candidates** list one by one to the **Accepted Ref. Patterns** list to accept them. When you accept the third reference pattern, examine the large changes of the scores of the remaining candidates at the same time.
8. Examine if the accepted patterns are minerals and its subfile information:
   - In the **Accepted Ref. Patterns** list, examine the information in the **Subfiles** column.

     NOTE: When the information is not fully shown, you can hover the cursor over the cell to see the full information.

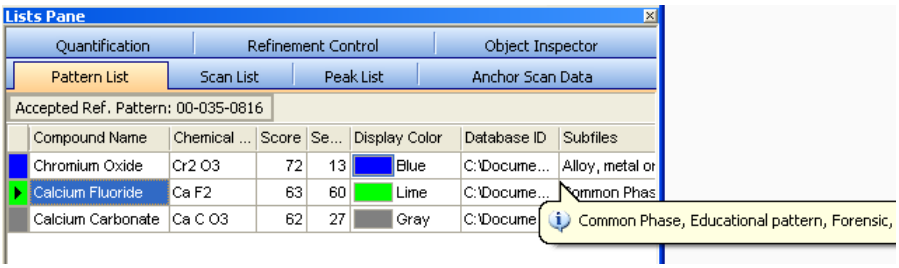


Figure 6.7 Complete information of the cell

- Alternatively, right-click a reference pattern in the **Accepted Ref. Patterns** list and from the pop-up window, select **Show Pattern** to show the subfile information.

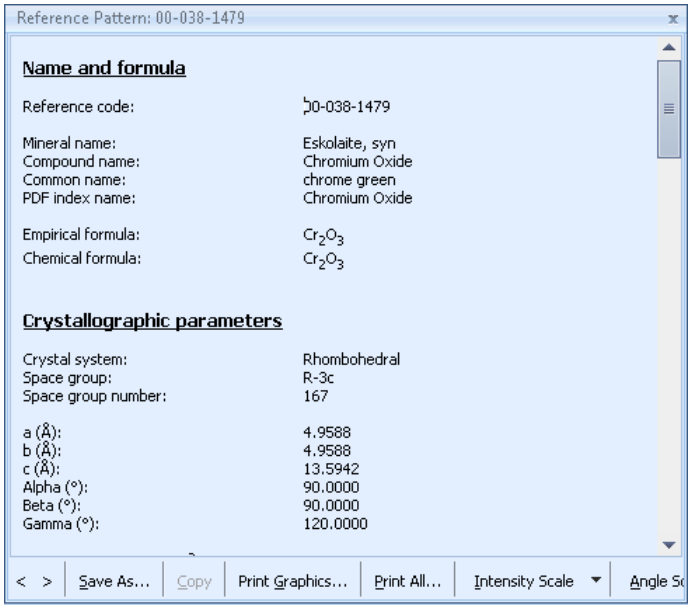


Figure 6.8 Reference Pattern window

9. In the **Display Mode** toolbar, click  to switch on the **Show Reference Patterns** function. The reference pattern lines show in the **Main Graphics** pane.

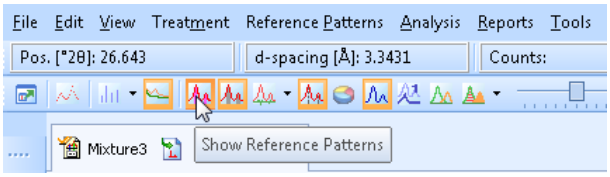


Figure 6.9 Show Reference Patterns button

For this time, these peaks or features cannot be explained:

- A small peak around 42 °2theta is not explained, but this can be a real peak or just some noise.
- A second unexplained peak around 79.3 °2theta is a $K\alpha_2$ peak, which is incorrectly assigned as a $K\alpha_1$ peak. You can go to **Tools > Spectral Lines** to do a check of it.

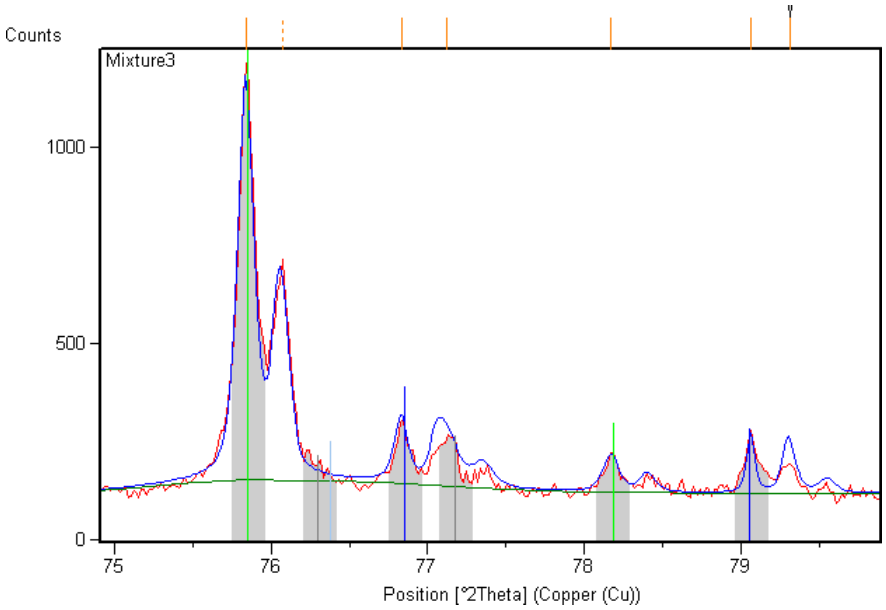


Figure 6.10 Unexplained peaks or features

10. Save the complete document:
    a. Go to **File > Save As**.
    b. Use these settings:

File name	Mixture3
Save as type	HighScore Plus (*.HPF)

    c. Click **Save**.
    d. If the **Confirm Save As** window opens, click **Yes**.

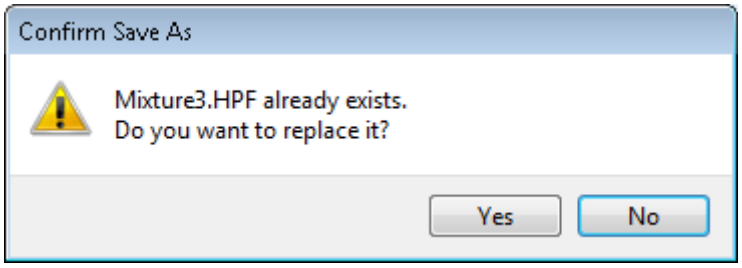


Figure 6.11 Confirm Save As window

The document is saved with background data, peak data, reference patterns and a candidate list.

CHAPTER 7 CHANGE SCORES

7.1 Introduction

In this chapter, you will change the scores of the search and match results in the **Candidates** list.

The scores, shown in the **Candidates** list and the **Accepted Ref. Patterns** list, are related to the parameter sets used for the search and match procedures. These scores can also be changed at any time after the search and match. In real work, if you change scores, other candidates can move to the top of the **Candidates** list and this can help you find more phases.

7.2 Change scores

1. If the software is not started, start it. Refer to Section 2.2.
2. On the menu bar, go to **File > Open** to open the document "Mixture3.hpf" that you saved. Refer to Section 6.3. "Mixture3.hpf" contains background data, peak data, reference patterns and a candidate list.
3. Drag the reference pattern "00-035-0816, Calcium Fluoride" from the **Accepted Ref. Patterns** list to the **Candidates** list.
4. Make sure that the **Pattern** toolbar is shown:
   a. Go to **View > Toolbars**.
   b. Select **Pattern Toolbar**.

Figure 7.1 Pattern toolbar

5. Click to change the data source and examine the changes of the score of the Fluorite reference pattern at the same time.

Figure 7.2 Click Data Source button

NOTE: The look of the **Select Data Source** button is related to the selected data source:

- : **Profile**
- : **Peak List**
- : **Both**

6. Click to include or exclude the quality of matching relative intensity in the score and examine the changes of the scores at the same time.

   NOTE: Steps 5 and 6 are frequently used to change the scores of a **Candidates** list.

7. Click to change the scoring scheme and examine the changes of the scores at the same time.

Figure 7.3 Click Scoring Scheme button

NOTE: The look of the **Select Scoring Scheme** button is related to the selected scoring scheme:

: **Single Phase**

: **Multi Phase**

8. Click to switch on and off the pattern shift and examine the changes of the scores and scale factors at the same time.

   NOTE: Steps 7 and 8 are almost never used to change the scores. Usually, the scoring scheme is set to **Multi Phase** and the pattern shift is switched off.

9. Click or go to **File > Close** on the menu bar to close the document. Do not save the changes.

CHAPTER 8 USE A USER BATCH

8.1 Introduction

All actions that you did about pattern treatments and search, match and identification can be done automatically with pre-programmed pattern restrictions. The default user batches in the software have pre-programmed pattern restrictions. With user batches, you can do a full analysis with just a click of a button.

Your knowledge about the sample is the most powerful tool that you have for phase identification. On the **Restrictions** tab of the **Search & Match** window, you can use some reference patterns for 1 or more conditions which will be used for search and match.

In this example, you will not use any restrictions, but search fully through the small PANalytical Example Database.

8.2 Use a user batch

1. If the software is not started, start it. Refer to Section 2.2.
2. On the menu bar, go to **File > Open** to open the document "Mixture3.xrdml". Refer to Section 3.2.

   NOTE: Be careful that you do not open the document "Mixture3.hpf".
3. Start the pre-programmed batch "IdeALL":
   - On the **Batches** toolbar, click **IdeAll**.

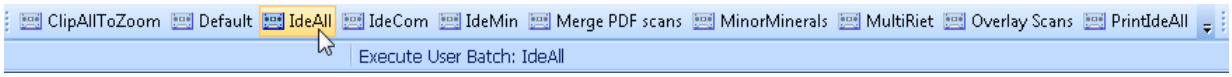
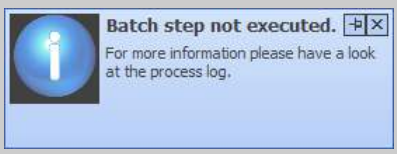


Figure 8.1 User batch IdeAll

   - Alternatively, on the menu bar, go to **Tools** and select **IdeAll**.

Table 8.1 What IdeALL does

Sequence	Step
1	Find the background.
2	Search peaks.
3	Convert divergence slit from automatic to fixed divergence slit intensities. **NOTE:** This step is not used in this example, because the measurement "Mixture3.xrdml" was done with fixed divergence slit.  *Figure 8.2 A batch step is not used*
4	Use the full PANalytical Example Database without restrictions for search and match.
5	Automatically identify candidates that have high scores.

4. After the operation of the user batch is completed, examine the peaks or features that cannot be explained in the **Main Graphics** pane.
5. Examine if the accepted patterns are minerals and its subfile information:
   - Go to the **Pattern List** pane and in the **Accepted Ref. Patterns** list, examine the information in the **Subfiles** column.

     NOTE: When the information is not fully shown, you can hover the cursor over the cell to see the full information.
   - Alternatively, right-click a reference pattern in the **Accepted Ref. Patterns** list and from the pop-up window, select **Show Pattern** to show the subfile information.
6. Examine the details of the batch:
   a. Go to **File > Properties** to open the **Properties of Mixture 3** window.
   b. Go to the **Process Log** tab.

      Process log shows that a batch step is not used.

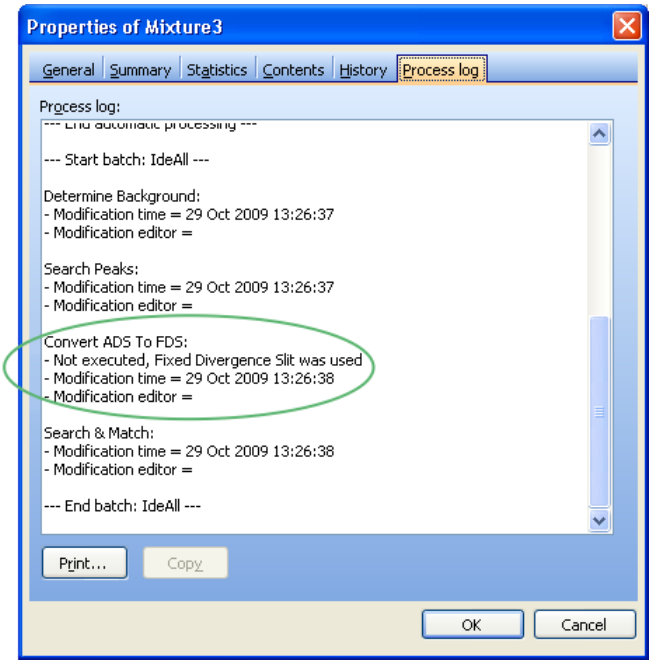


Figure 8.3 This batch step is not used

7. Save the document:
   a. On the menu bar, go to **File > Save Document**.
   b. Use these settings:

File name	MixtureBatch
Save as type	HighScore Plus (*.HPF)

   c. Click **Save**.

      The document is saved with the identified phases.

CHAPTER 9 PHASE IDENTIFICATION STRATEGY AND TROUBLESHOOTING

9.1 Introduction

There is no recipe for phase identification. However, this phase identification strategy can give you some simplified and schematic steps on how to identify an unknown sample in the software.

NOTE: In the identification examples, at the start you alway search the full reference database with no restrictions. This is done to show the capabilities of HighScore in the 'worst scenario', but it is not necessarily the best approach to all phase identification problems.

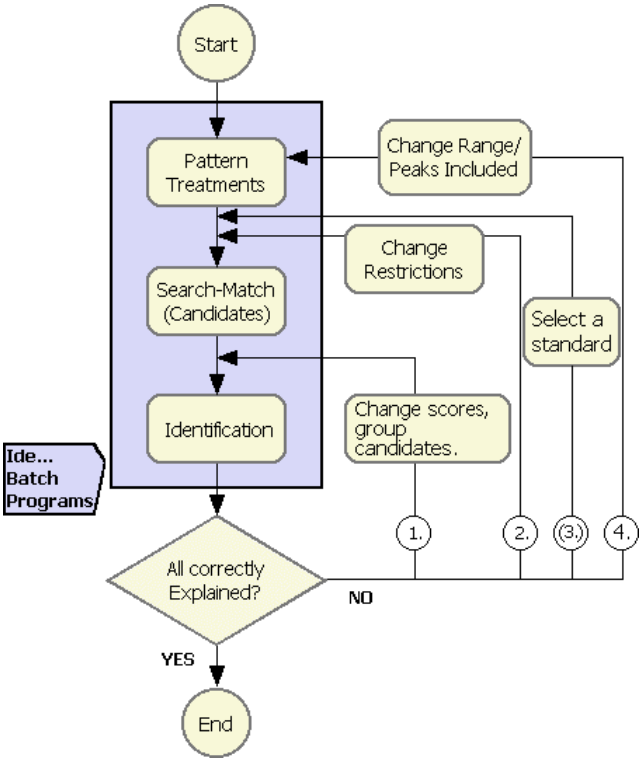


Figure 9.1 Phase identification strategy

Table 9.1 Description of the strategy steps

Step	Description
Start	• Load a measurement.
Pattern Treatments	• Find the background. This helps before you search peaks and it is very important when you use profile data for identification. • Search peaks with a high significance. Do not try to use very small peaks. Also use profile data for input. • Convert intensities to fixed slit intensities when an automatic theta-compensating divergence slit was used. This step is optional, but it makes measured intensities match the reference data better. • Strip $K\alpha_2$. This step is optional. When you do not use the **Strip K Alpha2** function( ), this step is done by the search and match algorithm. The process is done in the background and not shown to you. Most users find it is better not to see the process because then they can see the original measured data in the graphics.
Search and Match	• Start search and match to get a new **Candidates** list.
Identification	• Use score and scale factor values to identify and accept candidates. • Do a check of the graphics. • Examine the lists for more textual information.
Ide... Batch Programs	• You can use the user batch programs with names that start with "Ide" to do the steps above automatically.
Change Range/ Peaks Included	• Examine the unexplained peaks or features with the **Track Graphics Range** function ( ) or exclude the explained peaks and start search and match again to get a new **Candidates** list.
Change Restrictions	• Change restrictions. It is possible that the restrictions that you used were too tight and excluded some phases in the sample, or they were too wide and included many isotypical patterns and made the **Candidates** list full.
Change scores, group candidates	• Change scoring parameters to put other patterns to the top of the **Candidates** list, or put the candidates together to make similar patterns into a group. The idea is to identify as many phases as possible from the **Candidates** list before you start search and match again.
Select standard	• Select 1 identified pattern with simple, fixed chemistry as an internal standard.
All Correctly Explained?	• Do a check of all the patterns that were identified automatically. An automatic identification is not always correct.
End	• Save your result as a diffraction document in HDF format.

It is always good to include all information about the sample that you have. Use the subfiles made by ICDD or give special restrictions for the samples and analytical problems that occur to you.

9.2 Pattern treatment sequence

9.2.1 Find background

You must find the background before you search peaks. When there is no background data, peak search automatically makes its own background data.

When profile data is used for identification, it is very important to find the background correctly. If you are not sure, usually for phase identification, it is better to have the background too high than too low.

9.2.2 Search peaks with a high significance

Do not try to find all small peaks. When you select profile data for phase identification, all intensity above the background is used as input, which also includes very small peaks that are difficult to find.

9.2.3 Convert intensity

Convert intensity to fixed slit intensity when an automatic theta-compensating divergence slit was used. This is optional. The converted intensity will match the reference data better, which gives better scores when the **Match Intensity** function ( ) is used.

9.2.4 Strip $K\alpha_2$

If you do not use the **Strip K Alpha2** function ( ) to strip $K\alpha_2$, $K\alpha_2$ stripping is done by the search and match algorithm. The process is done in the background and not shown to you. Most users find it is better not to see the process because then they can see the original measured data in the graphics.

9.3 Identify

Some tools are available to get information about the match of a candidate or accepted pattern:

- For information about the explained scan regions, click **Show Explained Feature** button to switch on the **Show Explained Feature** feature.
- For information about matched peak, do a check of the **V** marks in the **Main Graphics** pane and the **Matched** column in the **Peak List** pane.

  NOTE: If the **Matched** column is not shown in the **Peak List** pane, you can right-click in the **Peak List** pane, select **Customize Peak List** from the pop-up menu to open the **Customization** window, and then double-click **Matched** to add it as a column in the pane.
- For information about matched peaks by reference codes, do a check of the **Matched by** column in the **Peak List** pane.
- For information about the matched reference pattern lines, right-click in the **Pattern List** pane and select **Analyze Pattern Lines** from the pop-up menu.
- For information about the number of matched lines, do a check of the the **Matched Lines** column in the **Accepted Ref. Patterns** list in the **Pattern List** pane.

When you cannot identify all phases from the **Candidates** list of the initial search and match, do as follows:

1. Change the scores to find more candidates from the **Candidates** list.
   - Change the data source with the **Change Data Source** button , , or on the Pattern toolbar.
   - Match the parameter with the **Match Intensity** button on the **Pattern** toolbar.
   - When you look for minor phases, do not select the **Demote Unmatched Strong** button on the **Pattern** toolbar.

- If you do not have strong solid-solution effects or an incorrect sample height, do not select the **Allow Pattern Shift** button on the **Pattern** toolbar. For the usual small pattern shifts, this parameter is usually more applicable to patterns that do not fit very well to the measurement.
- If necessary, change the scoring scheme with the **Select Scoring Scheme** button and do not select the **Auto Residue** button on the **Pattern** toolbar.

2. Start a search and match again with new, different restrictions or different parameters. It is possible that the restrictions that you used were too tight and excluded some phases in the sample, or they were too wide and included many isotypical patterns and made the **Candidates** list full.
3. If necessary, do step 1 again with the new **Candidates** list. This can solve many identification problems.
4. Optionally, when you identified 1 or several phases , but cannot identify more components in a mixture, this can be caused by a sample height or an alignment problem. If this occurs, it helps to select an identified pattern with a well defined, stable chemistry and use it as an internal standard:
   a. Right-click in the **Accepted Ref. Pattern** list.
   b. From the pop up menu, select **Correct Scan with Pattern**. The measurement with peaks and background is then shifted to fit optimally to the standard selected pattern.

   NOTE: Be careful when you do this step. When you select a pattern with variable chemistry (= solid solutions possible) as internal standard, it can have a bad effect on the correct identification of more phases.

   Make sure that the **Allow Pattern Shift** button is not selected on the **Pattern** toolbar when you select an internal standard.
5. If you still cannot identify all phases from the **Candidates** list, do as follows:
   a. Switch on the **Track Graphics Range** function with on the **Pattern** toolbar.
   b. Zoom in on the low-angle region which contains peaks and features that are not explained and examine the **Candidates** list again. For this time, only the peaks and features in the shown, zoomed-in region are used as input.
   c. If there are no new matches found in the top of the **Candidates** list, start a new search and match only with the zoomed-in range as input.
   d. Alternatively, exclude all matched peaks and do a search and match again with only the remaining peak data as input.
   e. If necessary, do steps 1-3 again with the new **Candidates** list.

9.4 Troubleshooting

If you still cannot identify all phases from the **Candidates** list or get very bad result, you can do troubleshooting:

1. Examine the background. A background level that was found too low has an effect on each reference pattern which match your measurement.
2. Examine the document wavelength, which is usually derived from the anchor scan. If the document wavelength is not in the anchor data, it is supplied by the default instrument settings and cannot match the actual scan data.

3. Examine which databases are used for search and match. Make sure there is a reference pattern in these databases applicable to your measurement.

   NOTE: All patterns in the pattern list are treated as 'known' when you start search and match. They are kept in the pattern list and the first 20 are scored. When the **Auto Residue** function () is used, these first 20 patterns also have an effect on the scores of all candidates.

CHAPTER 10 SEARCH AND REFINE A UNIT CELL (HIGHSCORE PLUS)

10.1 Introduction

In this chapter, you will do these tasks:

a. Load a measurement, set the correct wavelength, and convert the document format from RFL to RD, a format in which the data about the used wavelength can be saved.
b. Search diffraction peaks. The first 8 peaks (reflections) are used to start a unit cell search, which is also called indexing, with the TREOR and DICVOL indexing routines.
c. Refine a good cell candidate with all diffraction peaks as input.
d. Save the results.

10.2 load a measurement

1. If HighScore Plus is not started, start it. Refer to Section 2.2.
2. On the **Desktop** toolbar, set the desktop layout to **Structures**.

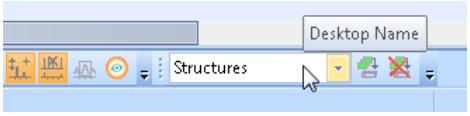


Figure 10.1 Set desktop layout

3. On the menu bar, go to **File > Open** to open the document "TaSSE.rfl" in this folder **C:\Users \user.name\Documents\PANalytical\X'Pert HighScore Plus\Tutorial**.
4. Set the the wavelength for the data set to Copper $K\alpha_1$ only:
   a. Go to the **Scan List** pane.
   b. Double-click in the list to open the **Object Inspector** pane.
   c. Open **Instrument Settings**.
   d. Select **Incident Beam Monochromator**.

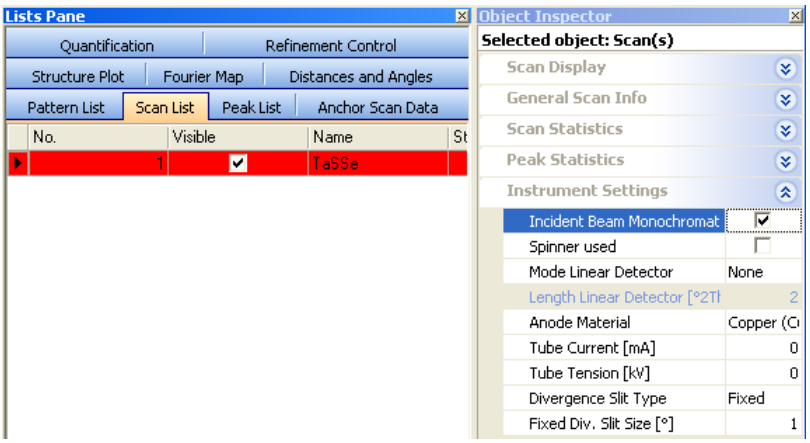


Figure 10.2 Select Incident Beam Monochromator

5. Go to **File > Save As** to save the document as a PHILIPS binary scan in the RD format. The wavelength with the scan data is saved in this format.

By this time, the important instrument information is corrected and the scan is converted from RFL file format to RD file format.

10.3 Search peaks

1. On the menu bar, go to **Treatment > Search Peaks**.
2. Click **More** to expand the window.
3. In the **Select Parameter Set** field, select **Default** from the drop-down list. The title bar of the **Search Peaks** window shows the name of the parameter set that is used.

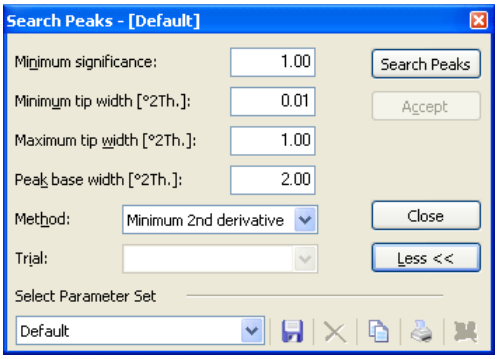


Figure 10.3 Use the Default parameter set

4. In the **Search Peaks** window, click **Search Peaks** to search peaks.
5. Click **Accept** to accept the results. The **Main Graphics** pane shows the peaks and a theoretical profile.
6. Zoom in around 60 to 65 °2theta. You can see that a peak is incorrectly found at about 61 °2theta.

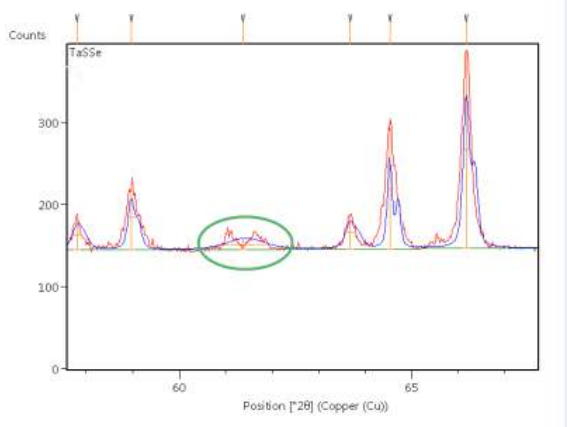


Figure 10.4 Peak incorrectly found

7. Hover the cursor over the peak to see detailed information.
8. On the menu bar, go to **View** and select **Peak List Pane**.
9. Find the only peak that is not correctly at its place: No. 17. This peak is selected when you hover the cursor over it.

10. Delete the peak:
    - Press **Delete**.
    - Alternatively, in the **Peak List** pane, right-click in the row of the peak and from the pop-up menu, select **Delete Peak**.

10.4 Search and refine a unit cell

1. On the menu bar, go to **Analysis > Crystallography > Search Unit Cell**. The **Search Unit Cell** window opens.
2. Go to **General**.
3. Set **Indexing Method** to **Treor**.

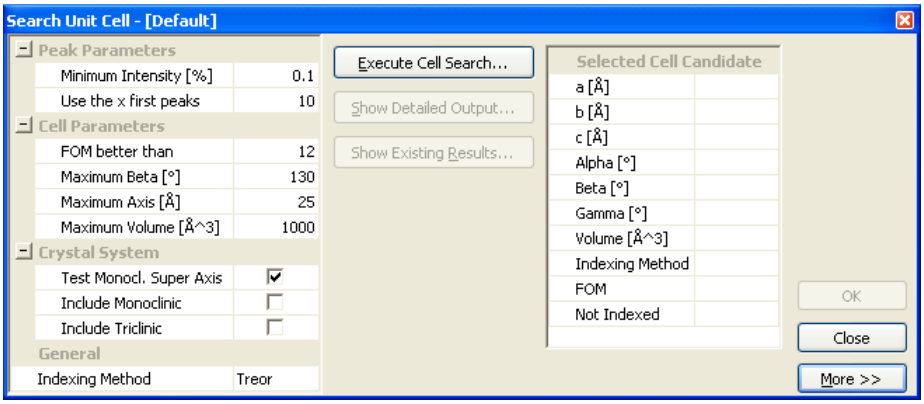


Figure 10.5 Set Indexing Method

4. Click **Execute Cell Search**. The **Cell Candidates** window opens. It shows some cell candidates.
5. Click **Cancel** to close the **Cell Candidates** window.
6. In the **Search Unit Cell** window, set **Indexing Method** to **Dicvol**.
7. Click **Execute Cell Search**. The **Cell Candidates** window opens. All cell candidates are almost the same. Therefore, there is a very possible unit cell.

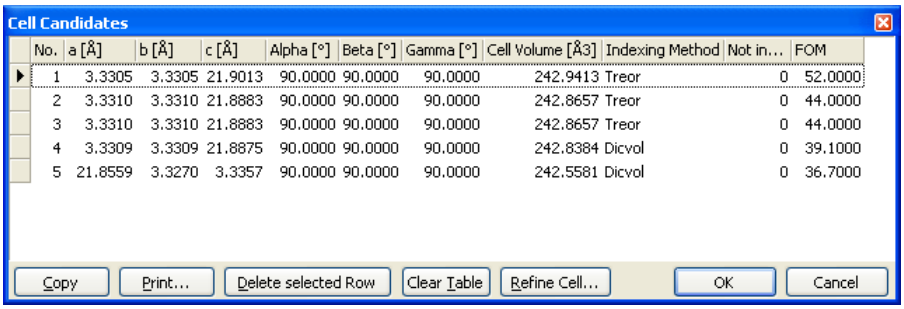


Figure 10.6 Cell Candidates window

8. Select one of the cell candidates.
9. Click **Refine Cell**. The **Refine Unit Cell** window opens.

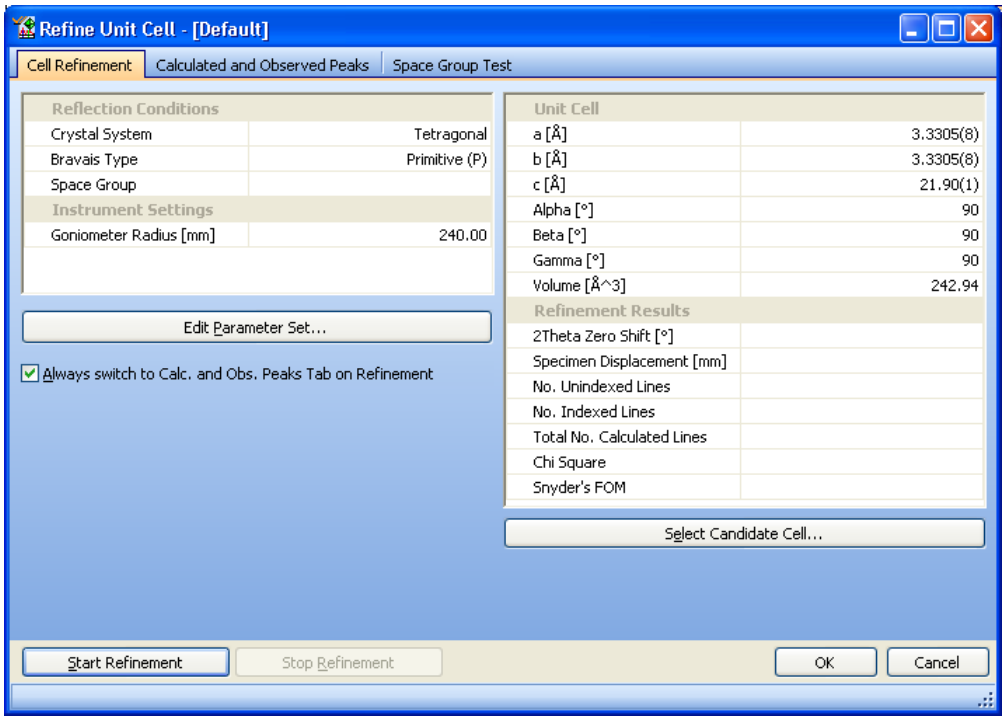


Figure 10.7 Refine Unit Cell window

10. Read the title bar of the **Refine Unit Cell** window. The title bar shows the parameter set that you use.
11. Make sure that you use the **Default** parameter set.
12. Click **Start Refinement**.
13. Go to the **Calculated and Observed Peaks** tab. It shows the calculated and indexed peaks and their deviation from the theoretical values.

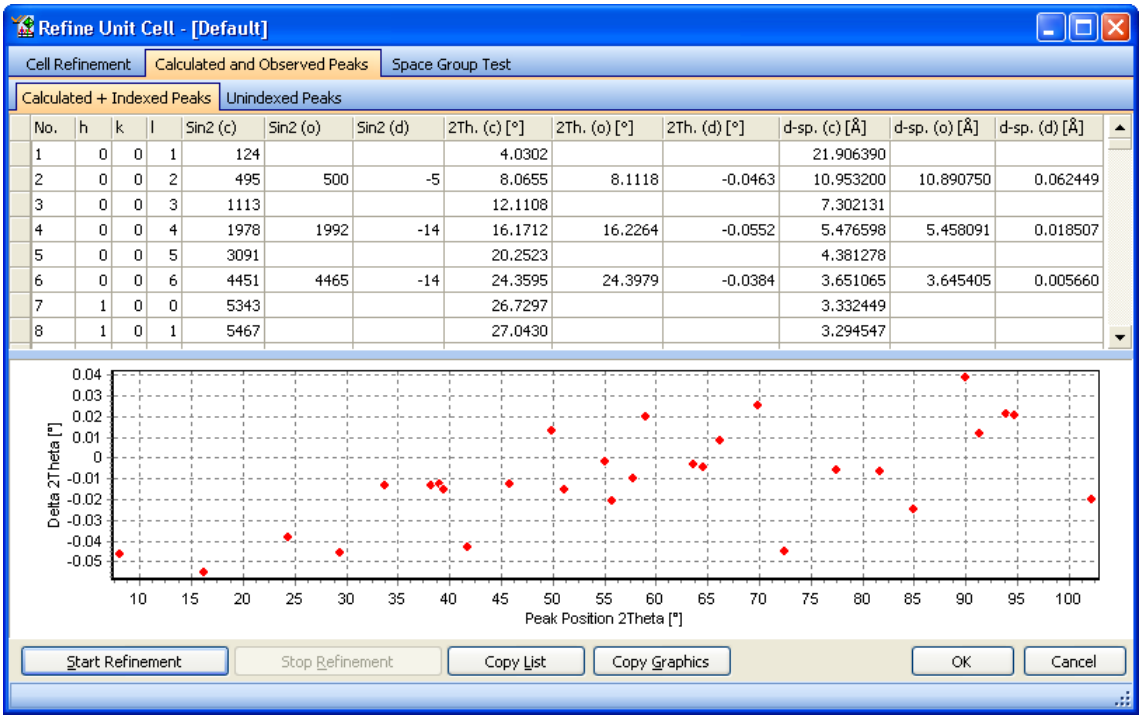


Figure 10.8 Cell refine results

14. Go to the **Unindexed Peaks** tab. It must be empty for this time.
15. Click **OK** to accept the results and close the window.
16. Examine the refined unit cell parameters:
    a. On the menu bar, go to **View** and select **Refinement Control Pane**.
    b. Open the **Search Unit Cell Result 1** phase.
    c. Open **Unit Cell**. The refined unit cell parameters is shown.
17. Examine the derived data:
    a. Double-click **Search Unit Cell Result** to go to the **Object Inspector** pane.
    b. Examine the derived data, for example the cell volume and the estimated cell volume error. It also shows the error of the unit cell axis or angle selected in the refinement control.
18. When a phase color is accidentally red, change the display color:
    a. Double-click the phase to open the **Object Inspector** pane.
    b. Open **Phase Display**.
    c. In the row of **Display Color**, click the arrow at the end to open the drop-down list.
    d. Select a different color.

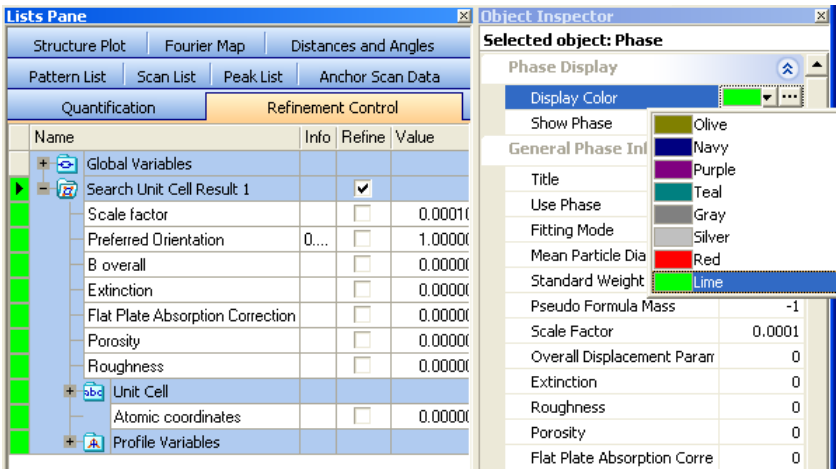


Figure 10.9 Display color

    e. In the **Main Graphics** pane, examine the peaks. All peaks are assigned to the phase and therefore have the phase color. By this time, there are no non-phase peaks in this example.

10.5 Save results

1. Go to **File > Save Document**.
2. Use these settings:

File name	TaSSe
Save as type	HighScore Plus (*.HPF)

3. Click **Save**.
4. If the **Confirm Save As** window opens, click **Yes**.

   The document is saved with scan data, background data peaks, phase data, list of cell candidates and history of the analysis steps.

CHAPTER 11 DO AN AUTOMATIC RIETVELD REFINEMENT (HIGHSCORE PLUS)

11.1 Introduction

This chapter shows you the Rietveld refinement of an artificial mixture of 2 minerals.

You will do an example for quantitative phase analysis. The example has pre-defined refinement steps. An automatic fitting parameter set for phase fits is used for this Rietveld refinement.

11.2 Load data

1. If HighScore Plus is not started, start it. Refer to Section 2.2.
2. Go to **File > Open** to open the document "25-75.rd" in this folder **C:\Users\user.name \Documents\PANalytical\X'Pert HighScore Plus\Tutorial**.
3. Go to **File > Insert** to insert "Example.cry" in this folder **C:\Users\user.name\Documents \PANalytical\X'Pert HighScore Plus\Structures**. The **CIR / CRYSTIN Import Structures** opens.

   NOTE: The "Structures" folder contains some crystal structures supplied with HighScore Plus. "Example.cry" contains the missing crystal structure data.

4. Select **Fluorite** and **Corundum**.

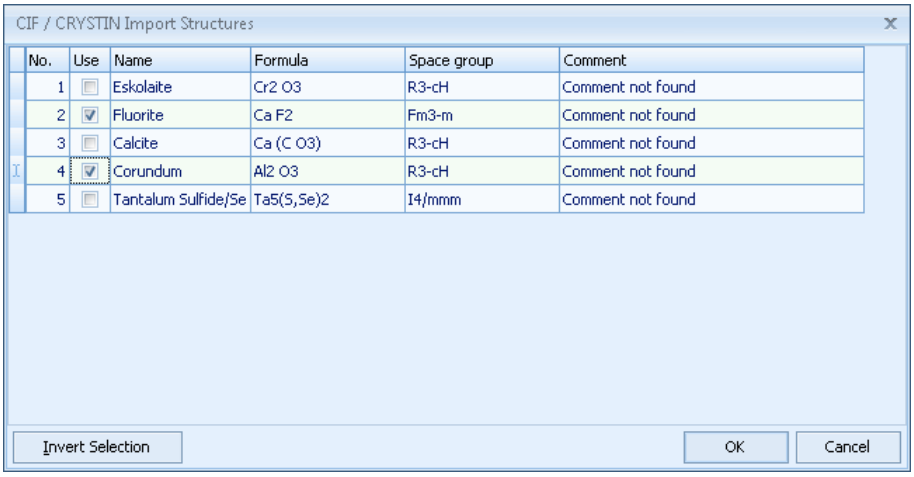


Figure 11.1 Fluorite and Corundum selected

5. Click **OK**. Fluorite and Corundum are inserted into the diffraction document.

11.3 Do an automatic refinement

1. On the **Desktop** toolbar, set the desktop layout to **Rietveld Analysis**.

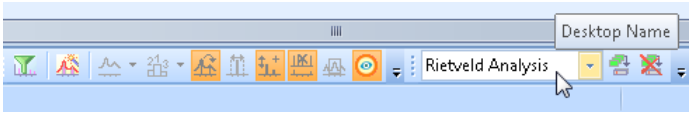


Figure 11.2 Set the desktop layout

2. On the menu bar, go to **Analysis > Fitting**.
3. Set **Fitting Mode** to **Automatic**.
4. On the menu bar, go to **Analysis > Fitting > Start Fit** and select **<Phase fit> Default Rietveld** to start the refinement.

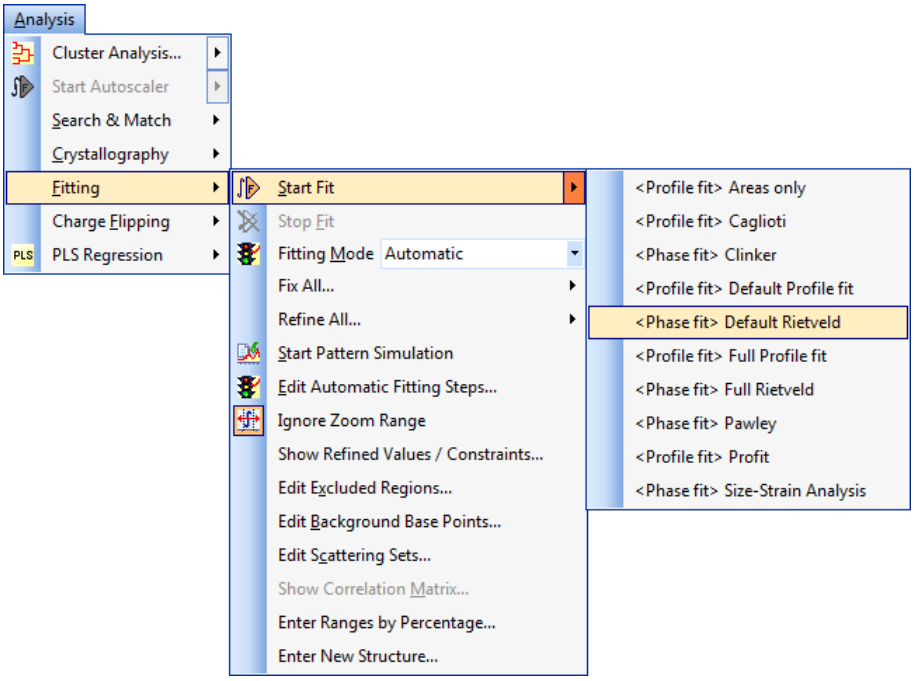


Figure 11.3 <Phase fit> Default Rietveld selected

The **R-Values** window opens.

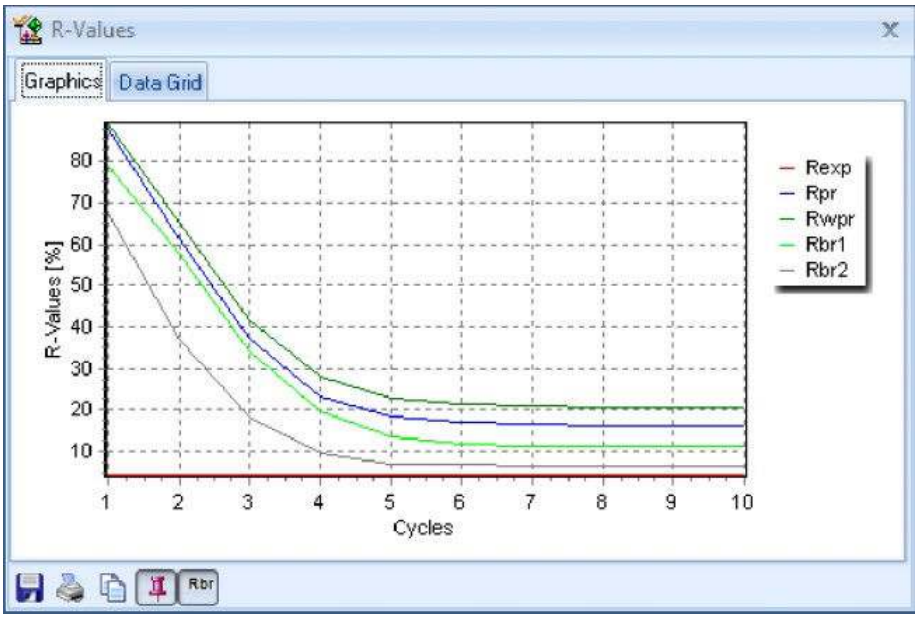


Figure 11.4 Rietveld refinement

After the automatic refinement is completed, the peaks and the calculated profile show in the **Main Graphics** pane.

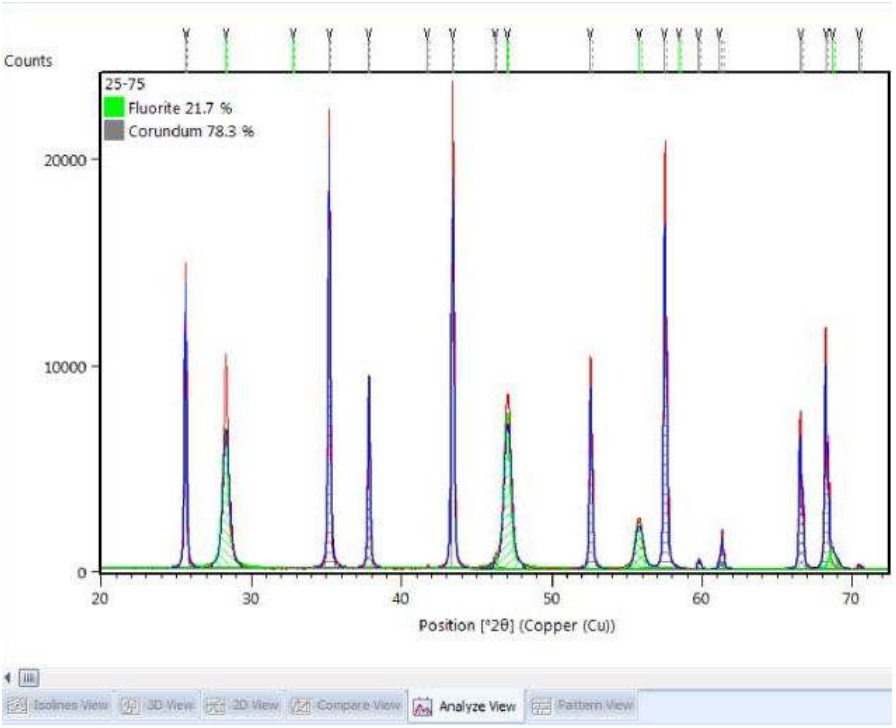


Figure 11.5 Peaks and the calculated profile after the refinement

5. Set the **Additional Graphics** pane to show the difference plot:
   a. Right click in **Additional Graphics** pane.
   b. From the pop-up menu, select **Show Graphics > Difference Plot**.
6. Hide the dynamic difference scale to show the small differences better:
   a. Right-click in the **Additional Graphics** pane.

b. On the pop-up menu, make sure that **Dynamic Difference Scale** is not selected.

7. In the **Main Graphics** pane, zoom in and examine different parts of the profile and the difference plot.
8. Examine the agreement indices:
   a. Go to the **Refinement Control** pane.
   b. Double-click **Global Variables** to open the **Object Inspector** pane.
   c. Open **Agreement Indices**.the values of **Goodness of Fit** and **Weighted R profile**.

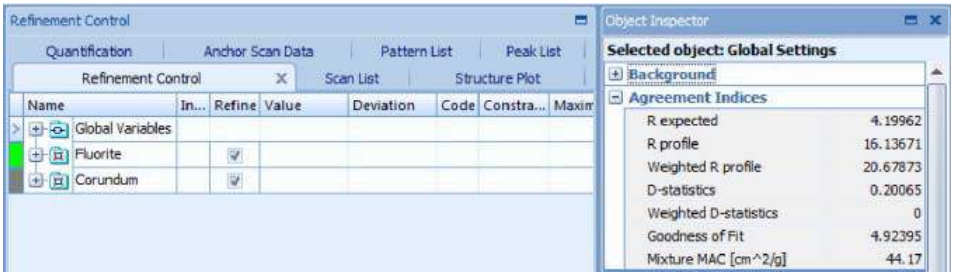


Figure 11.6 Agreement Indices

9. Examine the phase amounts:
   - Go to **Quantification Pane**. The pie chart shows that the result is 78.3 % Corundum and 21.7 % Fluorite, which is close to the correct weight percentages, 75 % and 25 %.

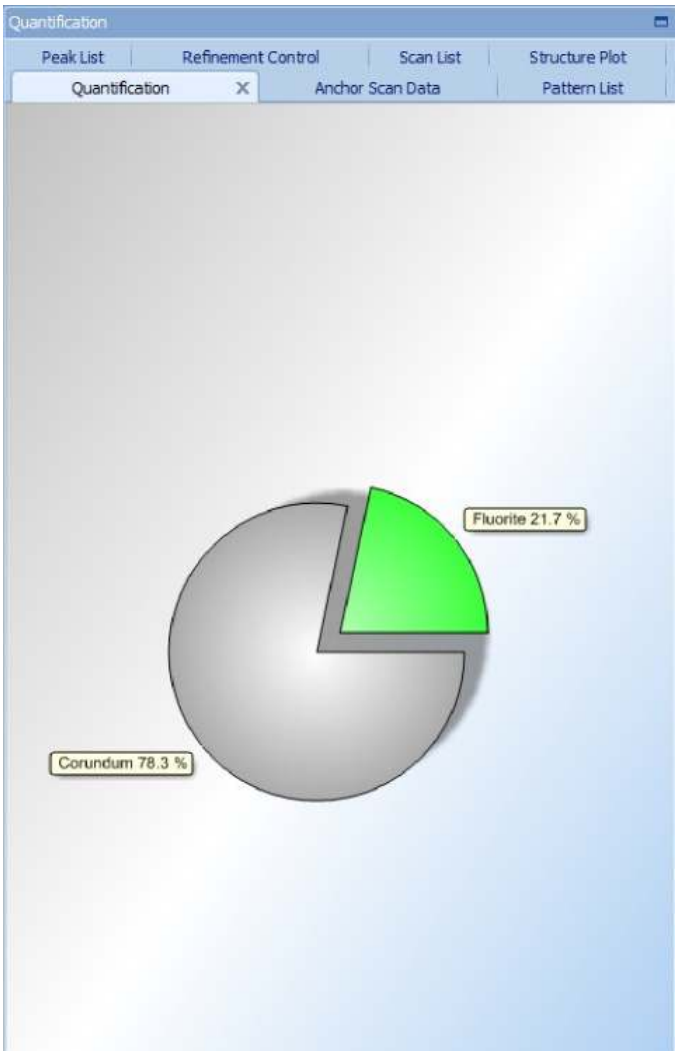


Figure 11.7 Quantification pane

- Alternatively, go to the **Main Graphics** pane. The phase amounts are usually shown with the phase legend.

11.4 Do a better refinement

1. On the menu bar, go to **Analysis > Fitting**.
2. Set **Fitting Mode** to **Manual**. In this mode, you can switch on and off the items that can be refined and automatic fitting parameters are no longer used.
3. Go to the **Refinement Control** pane.
4. Right-click to open the pop-up menu.
5. Go to **Refine All**.
6. Select **V's**.

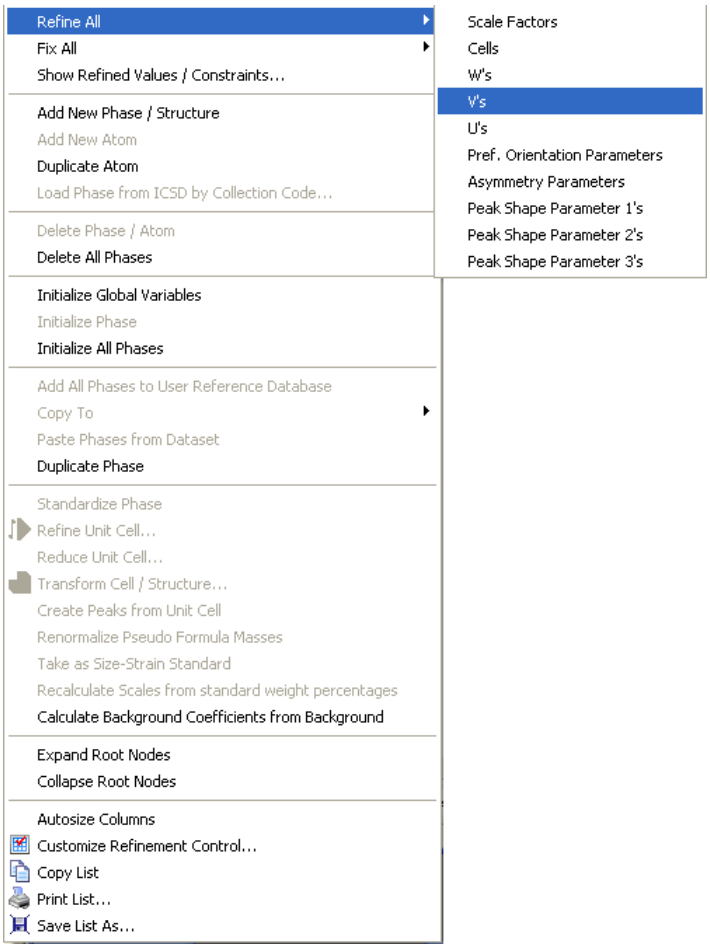


Figure 11.8 V's selected

7. Right-click to open the pop-up menu again.
8. Go to **Refine All**.
9. Select **U's**.
10. Start the refinement again:
    - Go to **Analysis > Fitting >Start Fit**.
    - Alternatively, on the **Fitting** toolbar, click .

Figure 11.9 Execute Fitting button

11. Right-click in the **Refinement Control** pane to open the pop-up menu.
12. Go to **Refine All**.
13. Select **Peak Shape Parameter 1's**.
14. Do step 10 to start the refinement again.
15. Change the FWHM to 30:
    a. Go to the **Refinement Control** pane.

b. Double-click **Global Variables** to open the **Object Inspector** pane.
c. Open **General Fit Properties**.
d. Change **Peak Base Width for Fit** from "20" to "30".

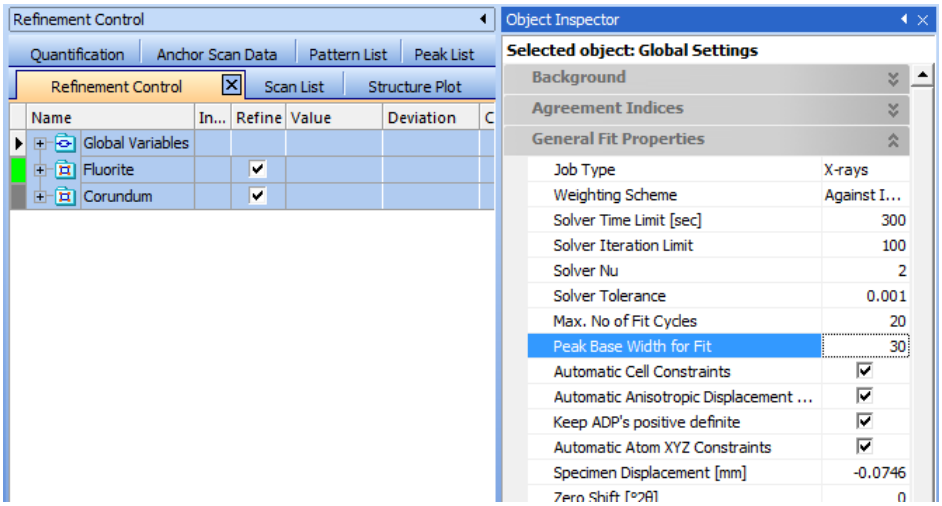


Figure 11.10 Change Peak Base Width for Fit

e. Press **Enter** to save the change.

16. Do step 10 to start the refinement again.
17. Examine the agreement indices:
    a. Go to the **Refinement Control** pane.
    b. Double-click **Global Variables** to open the **Object Inspector** pane.
    c. Open **Agreement Indices**.

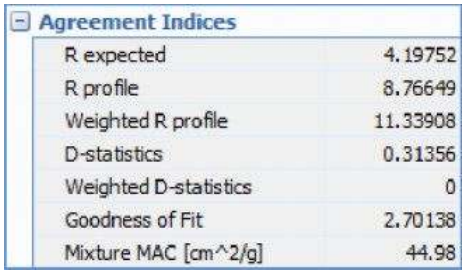


Figure 11.11 Agreement Indices

    d. Examine the values of **Goodness of Fit** and **Weighted R profile**:
        - The value of **Goodness of Fit** is 2.70.
        - The value of **Weighted R profile** is < 11.34.

          You have a better calculated profile with smaller differences to the measurement.

18. Go to **Quantification Pane** to examine the phase amounts. The phase amounts have small changes: 23.1 % for Fluorite and 76.9 % for Corundum, and they are closer to the given values. The phase quantification is within 2 % of the true values, which is a good result for a phase analysis without standard.

    NOTE: A quantitative phase analysis is not always correct with a good fit and low R-values.