


2.1 INTRODUCTION

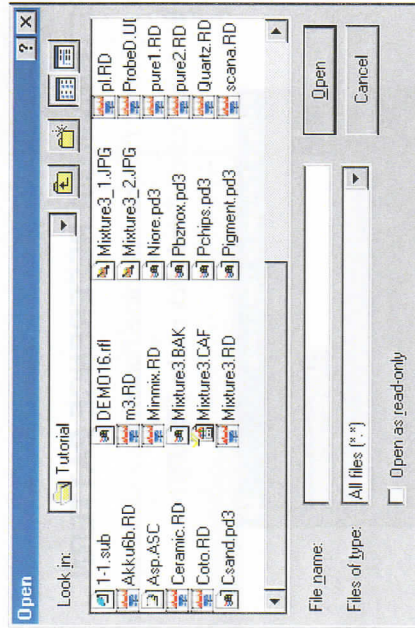
Loading and then displaying data is the most common task when you are using graphical software. There are many ways to display data and the different “Views” allow even more combinations. We will perform some simple examples in this chapter.

2.2 LOADING A SCAN

- If X'Pert HighScore is not already running, double-click on the  icon to start the program.
- Select *File - Open...*

A standard file selection dialog is shown.

- Select Files of type: (*.*) to display all the files in the Tutorial folder. The Tutorial folder can be found in: //.../HighScore.



- Select “Mixture3.RD” and click the Open button to open this file.




A copy of the selected file is loaded into a new X'Pert HighScore document. Since this is the first scan of the document it automatically becomes the anchor scan.

2.3 DISPLAYING THE SCAN

The main graphics pane shows the complete anchor scan in analyze view.

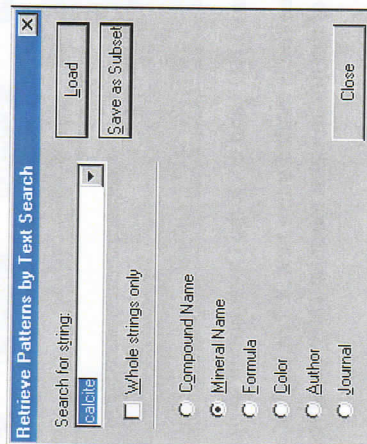
The additional graphics pane displays the zoom overview. (Since you have not zoomed in yet the complete scan range is shown in reverse colors.)

If it is the first time that this program is used, the lists pane has the "Pattern list" on top, if it has been used before, the last used tab will be on top.

- Zoom in by dragging in the main graphics pane. Zoom out by double-clicking in the main graphics pane. Do this a few times to see how it works.
- Use the Zoom Intensity On/Off () button on the Tool Palette button bar to switch zooming in Y-axis direction on or off. Now try zooming in and out with this switched on and off to see the various effects.
- You change the Y-axis scale by toggling with the Set Y-axes Scale () button on the Tool Palette button bar. To switch directly to a "Linear Y-Axis", "Square Root Y-Axis" or "Logarithmic Y-Axis" scale use the small  button next to it.

2.4 RETRIEVING A REFERENCE PATTERN

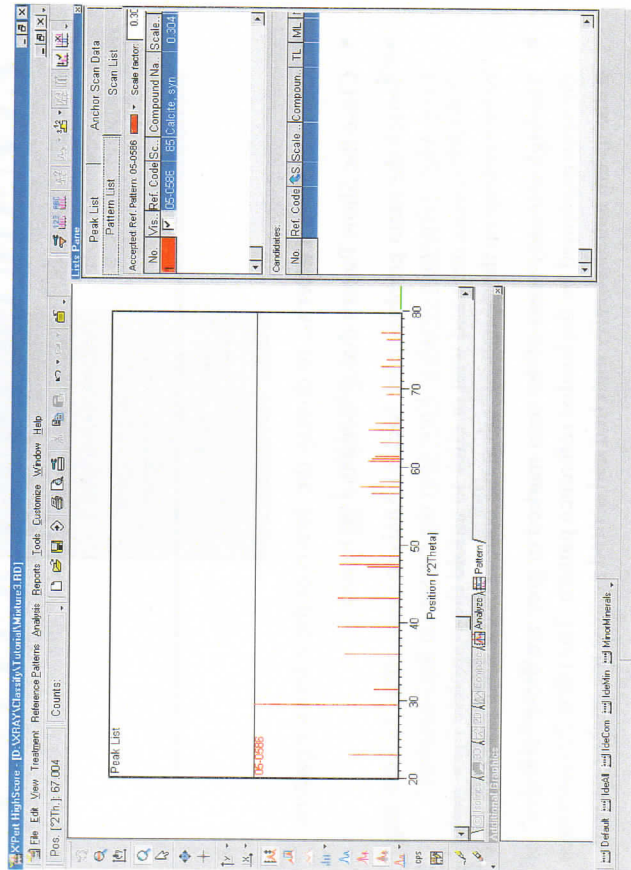
- Select *Reference Patterns - Retrieve Pattern by >* - *Text Search...*
- Enter the string "calcite" and click on the **Load** button.



- Close the dialog with the **Close** button.

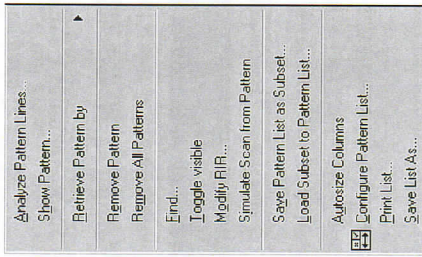
Several changes took place in your diffraction document:




1. The main graphics pane shows the sticks of the reference pattern in pattern view.
 2. The additional graphics pane is empty.
 3. The Pattern List displays a summary of the reference pattern.
- If it is not already on top, click on the Pattern List tab to bring it to the top.



2.5 DISPLAYING A REFERENCE PATTERN

- Right-click in the list of accepted reference patterns to open the pop-up menu.



- Click on **Show Pattern...** to display the details of the reference pattern.
- Close the Show Pattern dialog using the  button.
- Switch the main graphics pane back to Analyze view, either by selecting *View-Main Graphics-Analyze View* (put the cursor on the “Expand” () menu item and wait or click to unfold the complete *View* menu), or by clicking on the “Analyze” tab at the bottom of the main graphics pane.
- The high intensity areas of the scan marked in grey 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.
- Save your work by selecting *File-Save Document* and then accepting the proposed name: “Mixture3.CAF and press .
- Close your document.

CHAPTER 3

PATTERN TREATMENTS

Contents


	Page
3.1 Introduction	3 - 3
3.2 Background Determination	3 - 3
3.3 Peak Search	3 - 6

3.1 INTRODUCTION

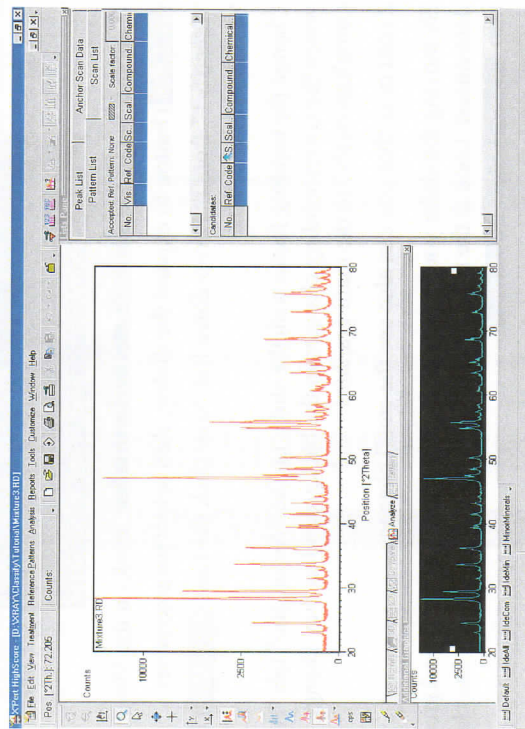
Pattern treatment allows you to modify existing data and generate additional derived data. Pattern treatment is also used to prepare the data for phase analysis.

The two most important treatments are background determination and peak search. A proper background determination is very important for phase analysis using the measured profile; peak search is required if you want to include peak data in the search-match-identification process.

3.2 BACKGROUND DETERMINATION

- If X'Pert HighScore is not already running, double-click on the  icon to start the program.
 - Load the file "Mixture3.RD" again. Do not load the document "Mixture3.CAF".
- Select *File - Open...*

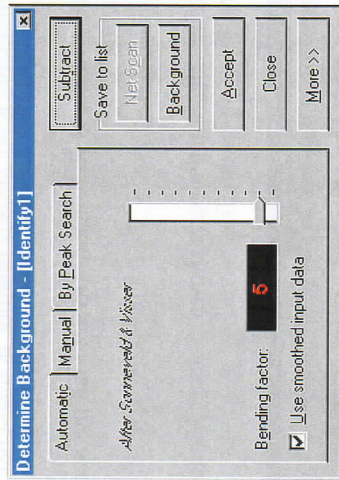
A standard file selection dialog is shown. Select "Mixture3.RD" and click the **Open** button to open this file.



A copy of the selected file is loaded into a new X'Pert HighScore document. Since this is the first scan of the document it automatically becomes the anchor scan.

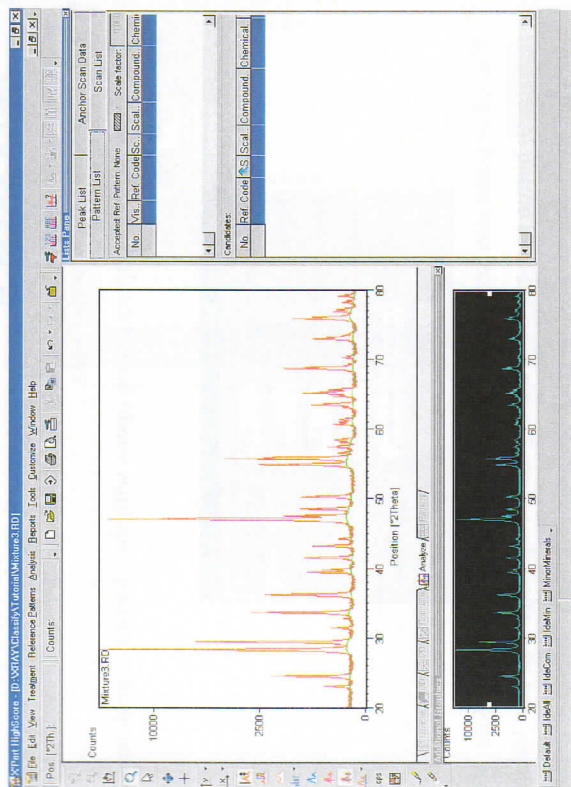
- Select *Treatment - Determine Background...*
- A pop-up dialog opens and the background is automatically determined. The background is shown as a green line in the main graphics. Look at the title bar of the dialog to confirm you are using the "Identify1" parameter set.

Note: This is an exception. Usually you have to invoke a function by clicking on a button in the pop-up dialog.



If another parameter set is shown in the title bar, click on the More (More >>) button to expand the dialog, then select parameter set "Identify1" from the drop-down list. Now click the Less (Less <<) button to collapse the dialog.

- Change the bending factor with the slider on the "Automatic" tab. You can watch the corresponding changes in the background as they change (simultaneously) in the main graphics.
- Zoom in so that you can also see the small changes.
- By moving the slider you have actually changed the way the background is determined. Look at the title bar of the pop-up dialog again. It has changed into Determine Background - [Untitled].
- Press the **Accept** button to accept the background. The pop-up dialog is closed and the accepted background is shown in its color in the graphics.

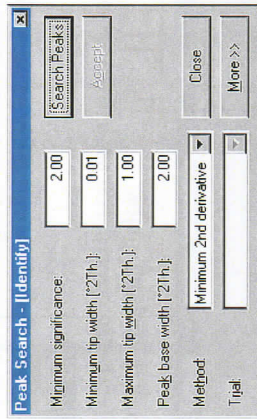


- Click on the "Anchor Scan Data" tab in the Lists Pane. The "Iback" column shows the numerical background data that you just determined.

No.	Pos. [2Th]	Intens. (cts)	Scale (cts)	Iback (cts)
1	20.0200	52	53	53
2	20.0500	49	53	53
3	20.0600	47	53	53
4	20.0800	71	53	53
5	20.1000	53	53	53
6	20.1200	50	53	53
7	20.1400	48	53	53
8	20.1600	52	53	53
9	20.1800	50	53	53
10	20.2000	44	53	53
11	20.2200	52	53	53
12	20.2400	58	53	53
13	20.2600	53	53	53
14	20.2800	46	53	53
15	20.3000	61	53	53
16	20.3200	56	53	53
17	20.3400	53	53	53
18	20.3600	49	53	53
19	20.3800	55	53	53
20	20.4000	46	53	53
21	20.4200	50	53	53
22	20.4400	52	53	53
23	20.4600	56	53	53
24	20.4800	49	53	53
25	20.5000	59	53	53
26	20.5200	56	53	53
27	20.5400	45	53	53
28	20.5600	45	53	53
29	20.5800	41	53	53

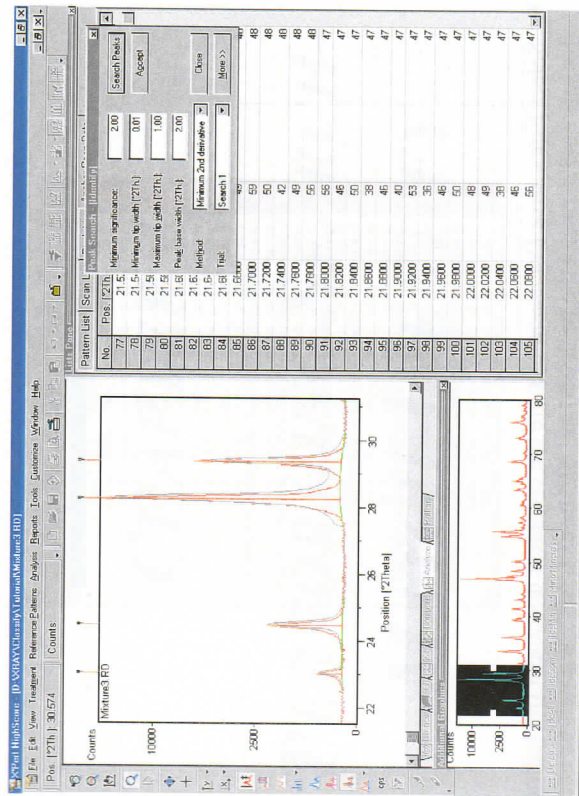
3.3 PEAK SEARCH

- Select *Treatment - Search Peaks...*
The “Peak Search” pop-up dialog opens with the “Identify” peak search parameter set.



If another parameter set is shown in the title bar, click on the More (More >>) button to expand the dialog, then select parameter set “Identify” from the drop-down list. Now click the Less (Less <<<) button to collapse the dialog.

- Press the **Search Peaks** button. Detected peaks are shown inside and above the main graphics by solid ($K\alpha_1$ and $K\alpha_{\text{mixed}}$ peaks) or dashed ($K\alpha_2$ peaks) red lines. If you have peaks displayed at the top of the main graphics, peaks not explained by a reference pattern have a little “v” mark. A calculated profile in preview color is also shown.



Note:

- This is only a preview of the result. If you close the dialog now, no peaks are written into the diffraction document. You can check the quality of the peak search by the peak sticks and the profile before accepting any result. Zoom in for more details.
- Press the **Accept** button to store the detected peaks permanently and to close the pop-up dialog.
- Toggle the display of peaks with the “Set Display of Peaks” button on the Tool Palette button bar. You can also directly switch to one of the four possibilities with the small button next to it.
- The “Peak List” tab is automatically opened in the Lists Pane. It shows numerical details on every detected peak. Peaks assigned to the $K\alpha_2$ wavelength are indicated by a grey background color in the peak list.

No.	Peak (hkl)	Height (cps)	FWHM (°)	Separation (°)	Peak Int.	FWHM (°)	Tip Width (°)	Matched by
1	24.4510	1694.05	0.1161	3.53472	12.03	0.1000	0.1000	
2	29.2570	15739.20	0.1375	3.15985	100.00	0.1400	0.1400	
3	31.3575	5242.71	0.0984	3.04044	30.95	0.1000	0.1000	
4	31.3575	207.05	0.0984	2.84684	1.32	0.1200	0.1200	
5	32.7572	49.37	0.1181	2.73399	0.31	0.1000	0.1000	
6	33.8520	2624.81	0.0984	2.66869	17.95	0.1000	0.1000	
7	35.9516	495.06	0.0720	2.49599	3.15	0.0000	0.0000	
8	35.9516	2241.12	0.0757	2.46334	14.24	0.0000	0.0000	
9	37.3513	107.19	0.0757	2.36765	4.46	0.0000	0.0000	
10	39.7529	587.50	0.0720	2.29855	2.33	0.0000	0.0000	
11	41.4519	793.76	0.0660	2.17811	5.04	0.0000	0.0000	
12	41.6590	401.03	0.0720	2.17529	2.55	0.0000	0.0000	
13	43.1398	567.92	0.0650	2.05629	3.61	0.0000	0.0000	
14	43.2669	279.05	0.0720	2.05461	1.77	0.0000	0.0000	
15	44.1832	110.25	0.0650	2.04832	0.70	0.0000	0.0000	
16	46.9731	1462.73	0.0650	1.93260	92.91	0.0000	0.0000	
17	47.1170	7511.00	0.0480	1.93205	47.73	0.0000	0.0000	
18	47.4744	1493.97	0.0720	1.93959	9.27	0.0000	0.0000	
19	47.6071	701.53	0.0720	1.93300	4.46	0.0000	0.0000	
20	48.9519	107.19	0.0720	1.81891	5.79	0.0000	0.0000	
21	51.1989	992.67	0.1320	1.81892	5.67	0.1000	0.1000	
22	51.3424	427.96	0.0720	1.81595	2.72	0.0000	0.0000	
23	54.8242	2088.83	0.0650	1.67215	15.81	0.0000	0.0000	
24	54.9519	1220.05	0.0720	1.67287	7.75	0.0000	0.0000	
25	55.7357	4350.11	0.0650	1.64793	27.64	0.0000	0.0000	
26	55.8890	2236.95	0.0720	1.64770	14.21	0.0000	0.0000	
27	55.5451	110.43	0.0720	1.62625	0.70	0.0000	0.0000	
28	57.3022	245.06	0.0650	1.50450	1.59	0.0000	0.0000	

- Select *File - Save Document* to store the complete document (including background data and the peak list) as "Mixture3.CAF". We will be using this document in the search-match procedure in Chapter 4.

CHAPTER 4

PERFORMING SEARCH-MATCH-IDENTIFY

Contents


4.1	Introduction	4 - 3
4.2	Search-Match	4 - 3
4.3	Identification	4 - 5

4.1 INTRODUCTION

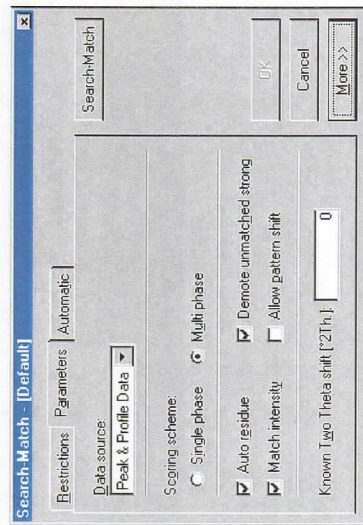
We are now going to search-match possible candidates and then manually identify the phases of the example file. Since we will be using the small reference database "ExampleDb" delivered with X'Pert HighScore this is not a real life situation, but it gives you a good example of what to do.

Note: This example database is not suited in any way to demonstrate the phase identification capabilities of X'Pert HighScore. You need an ICDD licensed PDF2 database to compare this software with other packages on the market.

4.2 SEARCH-MATCH

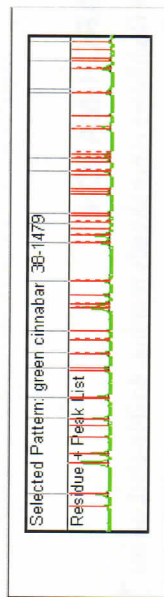
- If X'Pert HighScore is not already running, double-click on the  icon to start the program.
- Select *File - Open...*
- A standard file selection dialog is shown. Select "Mixture3.CAF" and click the **Open** button to open this document which contains the peak and background data which was assembled in the pattern treatments example.
- Select *Analysis - Search-Match...*

The "Search-Match" dialog pops-up with a parameter set.



- Make sure that the program has the correct parameters for the search-match procedure you are going to perform (they are called "Default"): Press the button and then select Default from the drop-down (▾) list. The parameter set name is shown in the dialog title bar.
- Press the button.

After a while a preview of the "Candidates List" is shown on the lower half of the Pattern List tab. Simultaneously the additional graphics is changed to the All-in-One plot.



- Press the **OK** button to accept the result.

The candidates list shows eight entries ordered from high to low scores.

Candidates:										
No.	Ref. Code	Scale	Compound	Chemical	TL	ML	NML	SUL	Displac.	
1	38-1479	72	0.160 green cinnabar	Cr2 O3	16	15	15	0	0.000	
2	06-0586	67	0.302 Calcite, sy	Ca C O3	24	23	23	0	0.000	
3	35-0816	54	0.950 Fluorite, sy	Ca F2	8	8	8	0	0.000	
4	77-2041	53	1.058 Sodium Er	Na Er F4	8	8	8	0	0.000	
5	06-0329	39	0.700 Praseodym	Pr O1.83	8	8	8	0	0.000	
6	75-0134	37	0.859 Uranium O	U O2	8	8	8	0	0.000	
7	73-1667	37	0.707 Bornite	Cu5 Fe S4	8	8	8	0	0.000	
8	06-0694	2	0.022 Chromium, Cr		2	1	1	1	0.000	

4.3 IDENTIFICATION



The next step is to manually accept candidates which have a high score and match the peaks/features of the measurement. Several "Views" in the additional graphics pane support the visual comparison between reference pattern sticks and measurement.

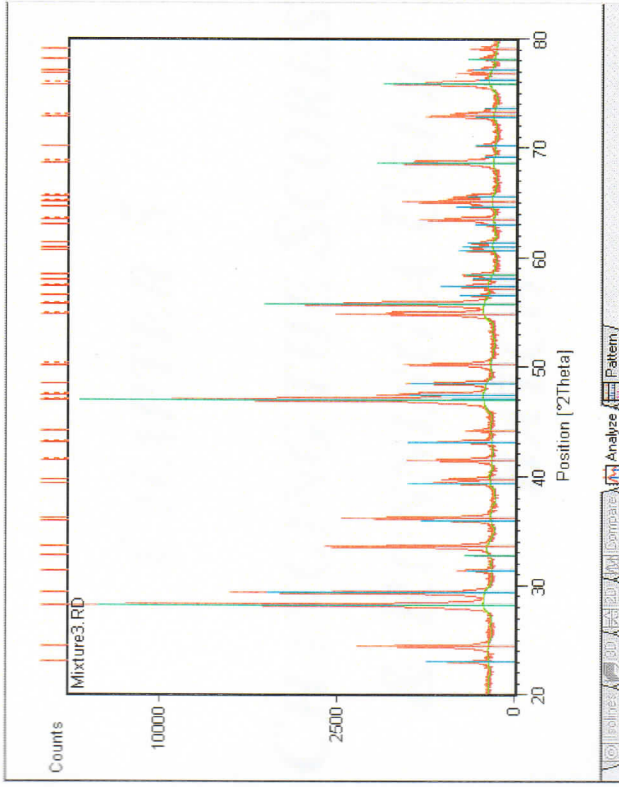
- Right-click in the additional graphics pane and select *Show Graphics - Compare Mode*.



- Toggle to only display peaks outside the main graphics (see: Chapter 3: "Pattern Treatments").
- Drag the first candidate pattern (38-1479, green cinnabar) from the candidates list to the pattern list tab to accept it, this pattern is highlighted. Its lines are shown (for comparison) in the additional graphics pane.

Candidates:										
No.	Ref. Code	Scale	Compound	Chemical	TL	ML	NML	SUL	Displac.	
1	05-0585	68	0.302 Calcite, sy	Ca C O3	24	23	22	0	0.000	
2	35-0816	56	0.950 Fluorite, sy	Ca F2	8	8	7	0	0.000	
3	77-2041	56	1.058 Sodium Er	Na Er F4	8	8	7	0	0.000	
4	06-0329	40	0.704 Praseodym	Pr O1.83	8	8	7	0	0.000	
5	75-0134	39	0.862 Uranium O	U O2	8	8	7	0	0.000	
6	73-1667	38	0.709 Bornite	Cu5 Fe S4	8	8	7	0	0.000	
7	06-0694	2	0.022 Chromium, Cr		2	1	1	1	0.000	

- Observe the following changes:
 1. Some peaks above the main graphics have lost the "V" mark. These peaks are explained by the accepted reference pattern. The peaks still marked with a "V" are not yet explained.
 2. High intensity areas in the main graphics are indicated in grey. These scan features are explained by the accepted reference pattern. If no scan features are marked in grey, press the "Show Explained Features On/Off" () button on the Tool Palette button bar.
 3. The candidates list now has candidate 05-0586, calcite on top. The score of this pattern has slightly changed with respect to the previous situation.
- Accept the next two patterns (one after the other) from the top of the candidates list. Observe the dramatically changing scores of the remaining candidates, while you accept the third reference pattern.
- Check to see if the accepted phases are indeed minerals and comply with the sample description given in the Introduction. Use the "Show Pattern..." function and look for the subfile information of all accepted reference patterns.
- Look for any unexplained peaks/features in the main graphics. Use the  button on the Tool Palette button bar to display the reference pattern lines in the main graphics.






- Select *File - Save Document* to store the complete document (now including the candidates list and accepted reference patterns) as "Mixture3.CAF".

CHAPTER 5

CHANGING THE SCORES & RUNNING A USER BATCH

Contents



	Page
5.1 Introduction	5 - 3
5.2 Changing the Scores	5 - 3
5.3 Running a User Batch	5 - 4

-  Data source = Profile
-  Data source = Peak List
-  Data source = Both (Default)

- Click the “Match Intensity” () button to include/exclude the quality of the intensity match in the score and watch the changing scores.

These two buttons are often used to change the scores of a candidates list.

- Click the “Select Scoring Scheme” button and switch between single/multiple phase scores.

-  Multi Phase scoring (Default)
-  Single Phase scoring

- Click the “Allow Pattern Shift” () button and watch the scores and scale factors changing (default = Allow Pattern Shift Off).

These two buttons are not often used to influence the scores.

5.3 RUNNING A USER BATCH

All of the actions that you performed in Chapter 3: “Pattern Treatments” and Chapter 4: “Performing Search-Match-Identify” can be done automatically in a pre-programmed way. User batches allow to perform a complete analysis by just pushing a button. Default user batches using pre-programmed restrictions are installed with the software.

Using all your knowledge about the sample is the most powerful tool for phase identification you have. Restrictions only allow certain reference patterns fulfilling one or more conditions to be used for search-match.

In this simple example a restriction to the “Minerals” subfile prevents the software from identifying an isotype of fluorite in place of the true mineral.

5.3.1 Running User Batches

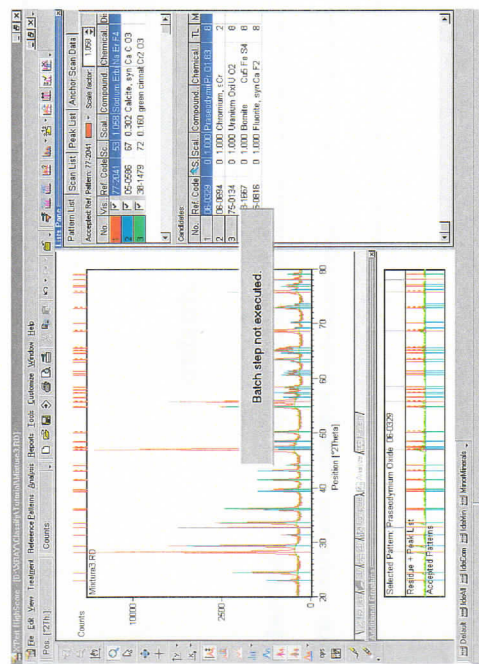
- Select *File - Open...*
- A standard file selection dialog is shown. Select “Mixture3.RD” and click the **Open** button to open this document.

Note: Do not open the Mixture3.CAF file this time.

- Select *Tools - IdeAll*

The pre-programmed batch program “IdeAll” is executed. It performs the following steps:

1. Background Determination.
2. Peak Search.
3. Divergence slit conversion from automatic to fixed divergence slit intensities.
4. Search-Match on the complete reference database (=no restrictions).
5. Automatic Identification of high-scoring candidates.



Step 3 was not executed because the measurement “Mixture3.RD” was done using a fixed divergence slit.

- Look for any unexplained peaks/features in the main graphics.
- Double-click on the first accepted pattern, "77-2041, Sodium Erbium Fluoride" to display its complete information. Check to see whether or not it belongs to the minerals subfile, then close the "reference pattern display".
- Select *Edit - Undo Batch IdeAll* to discard all results of the user batch.
- Select *Tools - IdeMin*
The pre-programmed batch "IdeMin" is executed. It performs exactly the same tasks as batch "IdeAll", but restricts possible candidates to reference patterns of the minerals subfile.
- Look at the list of remaining candidates: it is much shorter now, because it contains only minerals.
- Select *File - Save Document* to save the document including the correctly identified phases, give it the name "MixtureBatch.CAF".

CHAPTER 6

PHASE IDENTIFICATION

STRATEGY

	Page
6.1 Introduction	6 - 3
6.2 Pattern treatment sequence	6 - 5
6.3 Identification	6 - 6

6.1 INTRODUCTION

There is no general recipe for phase identification. Figure 6-1 shows the major steps in X'Pert HighScore (simplified and schematic) to identify an unknown sample. More information about Pattern Treatment and Identification is given in sections 6.2 and 6.3.

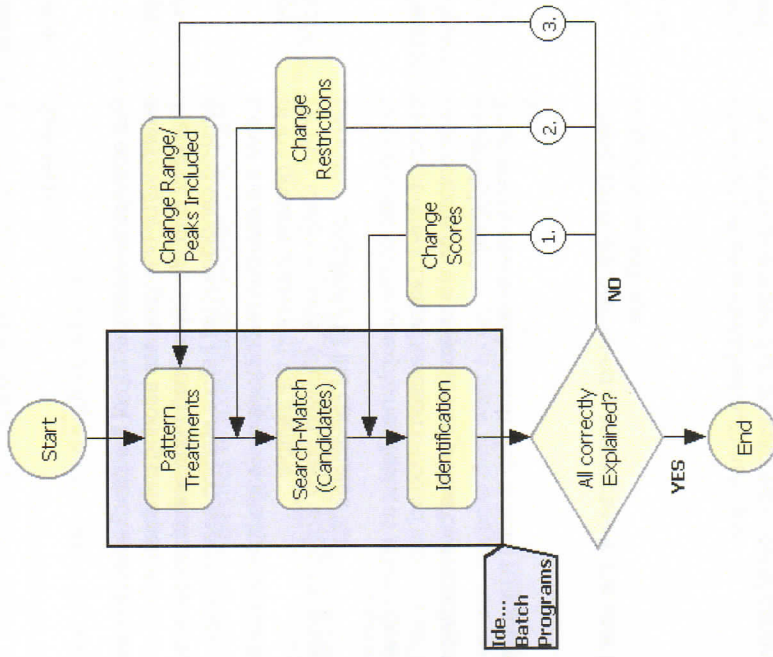


Figure 6-1 : Phase Identification Strategy Diagram

Each of the steps shown in Figure 6-1 are described in slightly more detail here:

Step/Action	Explanation / Description
Start	Load the measurement.
Pattern Treatments	See section 6.2.
Search-Match (Candidates)	Start search-match to build a new candidates list.
Identification	See section 6.3.
1. Change Scores	Put other patterns to the top of the candidates list (and to your attention) by changing the scoring parameters. The idea is to identify as many phases as possible from the existing candidates list before starting search-match again.
2. Change Restrictions	Either the previous restrictions were too tight, excluding some of the phases present in the sample. Or they were too wide, allowing many isotypical phases to pass and completely fill the candidates list.
3. Change Range/Peaks Included	Concentrate on the unexplained features and/or peaks by using the Track Graph function. Concentrate on the unexplained peaks by excluding already explained peaks. Then start a new search-match step to build a new candidates list.
Ide... Batch Programs	These steps are performed automatically in the user batch programs named "Ide...."
All correctly Explained?	Check all patterns identified automatically. There is no guarantee that an automatic identification is correct.
End	Save your result as a diffraction document in *.CAF format.

It is always good to include all information about the sample you have. Use the subfiles defined by ICDD or build dedicated restrictions tailored to the samples and analytical problems you encounter.

6.2 PATTERN TREATMENT SEQUENCE

The sequence that you should use to treat the pattern is as follows:

1. **Determine** the background.
This should be done before the peak search. If no background data is present, peak search automatically generates its own background data.
A proper background determination is crucial when profile data is used for identification.
2. **Search Peaks** with a high significance.
Do not try to locate all the little peaks. When you also select profile data for phase identification, all intensity above the background is taken into account, including very little peaks difficult to locate.
3. **Convert** intensities to fixed slit intensities if an automatic (θ -compensating) divergence slit was used.
This is an optional step, however, after the conversion measured intensities will match the reference data better.
4. **Strip $K\alpha_2$** .
This step can be performed in the background (unseen) if required. Many people prefer the $K\alpha_2$ stripping to be performed in the background (unseen) because they want to see the original measured data in the graphics.

6.3 IDENTIFICATION


There are several tools available to control the "match" of a candidate/accepted phase, they are:



- ◆ Explained scan regions (graphically)
- ◆ Matched peaks (graphically, textually)
- ◆ Matched peaks by reference codes (textually)
- ◆ Reference pattern lines matched (textually)
- ◆ Number of matched lines, number of new matched lines (textually)

If you cannot identify all phases from the candidates list of the initial search-match, proceed as follows:


1. Change the Scores to find more candidates from the existing candidates list.

Change the Data Source ( ) first, then the Match Intensity parameter (). If you are looking for minor phases also switch off the Demote Unmatched Strong parameter ().

Do not use the Allow Pattern Shift () parameter unless you are dealing with strong solid-solution effects or with an incorrect sample height. In case of the usual, small pattern shifts this parameter often favors patterns that do not fit the measurement very well.

You can also experiment with the Scoring Scheme () and the Auto-Residue () parameter.

2. The next step is to repeat search-match with new, different restrictions (and different parameters). It is possible that the correct phase was excluded by the restrictions used in the initial search-match being **too tight**, or **too wide**, allowing many isotypical phases to pass and completely fill the candidates list, leaving no space for other candidates. If necessary, repeat step 1. with the new candidates list. Many identification problems can be solved this way.

3. If you still have no success, you should first switch on the Track Graph function (). Now zoom in on the low-angle region containing unexplained peaks and/or features and check the candidates list. Now only the peaks/features in the visible (= zoomed) region are taken into

account.

If there are no new matches found on top of the candidates list, you could also start a new search-match using only the zoomed range as input.

An alternative is to exclude all of the already matched peaks and to search-match again using the remaining peak data as input.

If necessary, repeat steps 1. and 2. with the new candidates list.

6.4 TROUBLESHOOTING

You have no success or very strange results?

First look at the background. A background level that was determined too low results in every reference pattern matching your measurement.

Check the document wavelength, which is usually derived from the anchor scan, or if missing in the anchor data, is supplied by the instrument defaults.

Check, which database is used for search-match. Are you sure there is a reference pattern fitting to your measurement in the database?

Tip:

All patterns present in the pattern list are treated as knowns when you start search-match (again). This means, they are kept in the pattern list and the first 20 of them are scored. If auto-residue is set to on, these first 20 patterns influence the scores of the other candidates.