

PROCEDURE TO IDENTIFY THE MINERALS IN A SPECIMEN

Computer-automated search-match of the PDF database

Overview

It is assumed that the user has scanned a powdered specimen following the appropriate procedure and has acquired powder diffraction data.

The search-match routine used to identify minerals in a specimen must be preceded by a peak search of the diffraction data. Peak search parameters specify how peaks are differentiated from background and noise. Peaks found during the peak search are compared to powder diffraction data for minerals and other crystalline substances in a large database (powder diffraction files, PDF). Search-match parameters specify how the database is searched, what constitutes a match, and how the matches are scored.

1. From **X'Pert Organiser**, launch **X'Pert Graphics & Identify** software.

Click on the **X'Pert Graphics & Identify** icon in the tool bar or select **X'Pert Graphics & Identify** from the **Modules** menu.

2. Create a new graph from a measured scan.

Select **File / New Graph...** .

If necessary, click **Filter...** to browse/select Users and Projects, etc.

Select the data for the specimen you wish to identify; double click the data name or select the data name and click **OK**.

A graph showing the scan should appear in a new window.

Alternatively, you can create a new graph from a scan file (PC-APD pattern) or open an existing graph.

3. Set peak search parameters.

Select **Edit / Peak Search Parameters...** , if you wish to change or customize a peak search parameter set.

*This is probably not necessary; default fault peak search parameters for **measured intensities** are probably appropriate. In which case, you can skip to step 4.*

Some of the peak search parameters are described below.

Define peak position using the **Minimum of 2nd derivative** for best results in the typical situation of overlapping peaks.

Use 0.00 and 1.00 for the **Minimum** and **Maximum peak tip widths** for most scans. If particularly broad peaks are of interest, increase the maximum peak tip width.

Peak base width is used for the calculation of background. Generally, this parameter should be twice the maximum peak tip width.

Typical values for **Minimum significance** are between 0.6 and 0.75. The lower minimum significance (0.6) ensures that virtually all peaks are found at the risk of including some "peaks" caused by noise. Some peaks may be missed if a higher minimum significance is used, but peaks that are found will be more reliable.

Make adjustments, as necessary, to the peak search parameters in the **Edit Peak Search Parameters** window. Click **Save As...** if you wish to save (and use) your peak search parameters.

In the **Save Peak Search Parameter Set** window, enter a name and a description, and click **OK**.

Click **OK** (or **Cancel**) in the **Edit Peak Search Parameters** window.

4. Execute the peak search.

Select **Analyse / Peak Search** .

In the resulting window (**Select Peak Search Parameter Set**), select the peak search parameter set you wish to use; double click the set name or select the set name and click **OK**. Unless you have created a custom parameter set, this will probably be the default set (**As Measured Intensities**).

Peaks are identified; they are indicated on the graph and are listed in a table (**Edit Peak List** window).

Select **Window / Tile Horizontally** to view both the graph and the table of peaks at the same time.

Assuming that these peaks are acceptable, click **OK** in the **Edit Peak List** window.

Note that you are able to edit these peaks (delete or insert peaks, or change values in the table). However, this is probably not necessary.

5. Set restrictions for the search of **powder diffraction files (PDF)**.

Select **Edit / New Restrictions ...** (bottom of **Edit** menu).

The resulting window (**Edit Restrictions**) has five tab selections, Subfiles, Elements, Chemical Groups, Reference Patterns, and Comments.

You probably wish to narrow the search to minerals only; select the **Subfiles** tab and select (check box) the **Minerals** subfile.

You may also narrow the search based on chemical elements (**Elements** tab) or chemical groups (**Chemical Groups** tab) that you assume (or suspect) to be present or absent.

Click **Help** for explanation of these restrictions options.

Save and name the new restrictions set; click **Save As...** in the **Edit Restrictions** window.

In the **Save Restrictions Set** window, enter a restrictions set name and a description of the set, and click **OK** .

Click **OK** (or **Cancel**) in the **Edit Restrictions** window.

6. Set search-match parameters.

Select **Edit / Search-Match Parameters...** , if you wish to change or customize a search-match parameter set.

*This is probably not necessary; the **MinSearch** parameter set, which is available to all users, is probably appropriate. In which case, you can skip to step 7. The **MinSearch** parameter set restricts the PDF database to minerals only (6,000+ reference patterns in identdb/set50) and has good general-purpose values for search-match parameters.*

The resulting window (**Edit Search-Match Parameters**) has five tab selections, Parameters, Databases, Restrictions, Sort, and Comment.

Search-match parameters (**Parameters** tab) are described below.

Intensity threshold is the relative intensity, below which peaks in the peak list are disregarded during the search of the database. Values between 2% and 5% are normally appropriate.

Confidence threshold is the minimum confidence required before a reference pattern is accepted as a potential match. Values between 10% and 40% are normally appropriate.

Number of strong lines is the number of strongest lines used from each reference pattern during the search.

Length of score list is the maximum number of potential matches that will be found during the search. The search will cease when this number of potential matches are found. In general, use approximately 5 times the number of peaks in the peak list (result of step 4), but not less than 100; set the **length of score list** to about 150 for a list of 30 peaks, or 250 for a list of 50 peaks.

Minimum, Maximum, and Delta displacements are used by the software to iteratively correct the peak list for sample displacement and search the database. Negative 50, 50, and 100 microns are realistic displacements.

Penalty for missing lines can be specified to help compensate for preferred orientation. If a strong preferred orientation is expected, a relatively low penalty for missing lines should be used. The default value of 0.45 works well for most analyses.

Make adjustments, as necessary, to the search-match parameters.

Select the **Databases** tab and select (check box) the **identdb/set50** database.

Select the **Restrictions** tab and select (radio button) the restrictions set you wish to use.

Select the **Sort** tab and select (radio button) **score*relative score** and **descending** order.

Click **Save As...** if you wish to save (and use) your search-match parameters.

In the **Save Search-Match Parameter Set** window, enter a name and a description, and click **OK**.

Click **OK** (or **Cancel**) in the **Edit Search-Match Parameters** window.

7. Execute the search-match routine and study the results.

Select **Analyse / Search-Match** .

In the resulting window (name), select the search-match parameter set you wish to use; double click the set name or select the set name and click **OK**.

*Try the **MinSearch** parameter set for analyses of minerals.*

After the search-match routine is finished, a list of scored potential matches (the match score list) is shown in a new window (**Identity**).

Select **Window / Tile Horizontally** to view both the graph and the match score list at the same time.

As per step 6 (or the **MinSearch** parameter set), the match score list shows potential matches from the PDF database in descending order based on the **score*relative score** . This is generally the best type of scoring to use.

In the **Identity** window, check the box for **Correct position of plotted peaks for specimen displacement** . This will usually improve the alignment of peaks in the measured diffraction pattern and reference patterns.

Select a high scoring entry in the match score list; click on the name of the substance (mineral). Note that the reference pattern for the selected substance is displayed on the graph with black lines. If the reference pattern provides a good match to the measured diffraction pattern, accept it; click the check box in front of the entry (**A** column).

Reference patterns for accepted substances are shown on the graph in different colors.

Go down the list; continue the process of selecting an entry, comparing its reference pattern (shown in black) to that the measured diffraction pattern, and either accepting or not accepting it, until all peaks in the measured diffraction pattern are accounted for.