

PROCEDURE TO SCAN A SPECIMEN

Overview

It is assumed that the user has started or turned up the X-ray generator and has mounted a powdered specimen in the specimen chamber following the appropriate procedures.

As described here, the program-controlled scan is preceded by a quick manual scan. The manual scan is used to determine appropriate measurement conditions, which vary from sample to sample. Ultimately, this strategy saves time and ensures good, dependable data.

1. Launch the Philips **X'Pert Organiser** software.

Double click the Philips **X'Pert Organiser** icon on the computer's desktop or launch the software from the Start menu; select **Start / Programs / Philips X'Pert Software / X'Pert Organiser** .

2. At the introductory screen, sign in with your **User name** and **Password** .

See J. Mies for a user name and password.

3. You may wish to set up a new project.

"Users" and "Projects" constitute a hierarchical database, in which measurements, scan programs, search parameters, etc. are stored.

Select **Users & Projects / Edit Projects...**

In the resulting window (**Edit Projects**), click **New** .

In the **New Project** window, type in a project name. If you wish, also type in a description and a comment. Click **OK**.

If you are done editing projects, click **OK** in the **Edit Projects** window.

4. Select a project to work under.

Select **Users & Projects / Select Project...** .

In the **Select Current Project** window, click on the name of the project you wish to work under and click **OK** (or double click the project name). (*This is likely to be the new project you created in step 3.*)

5. From **X'Pert Organiser**, launch **X'Pert Data Collector** software.

Click on the **Data Collector icon** in the tool bar or select **X'Pert Data Collector** from the **Modules** menu.

6. Go On-line, i.e. establish communication with the XRD.

Select (click on) the **Control!** menu. The **Control!** "menu" behaves more like a "button"; it is not a menu of choices. Instead, you are presented with a succession of windows.

In the 1st window (**Go On-line**), select **Standard Configuration** and click **OK** .

In the 2nd window (**X'Pert Data Collector**), you'll be alerted as to what slits are recorded to have been installed. Click **OK** .

In the 3rd window (**Control [Standard]**), you'll be given the opportunity to ...

- a. Change the goniometer position. This is generally not necessary.
- b. Change the tension and current. Change these to **40 kV** and **40 mA** .

Click on **Change kV-mA** . Type in **40 kV** for tension and **40 mA** for current and click **OK**. Confirm these settings and click **OK** in the next alert (!) window.

- c. Change the incident and diffracted beam optics

If the slits you have installed are different than what was reported in the 2nd window, make the changes here. Otherwise, leave these settings as they are.

If you close the **Control [Standard]** window, be careful not to go Off-line.

7. Perform a quick (10 to 15 minute) manual scan to determine appropriate measurement conditions for the detailed (programmed) scan.

Back at the computer (X'Pert Data Collector software), select **Measure / Manual Scan** .

Set up the **Scan Mode (Prepare Manual Scan** window) as follows:

Scan axis:	Gonio	Step size:	0.040°
Scan mode:	Continuous	Time per step:	0.40 s
Range:	80°		

You must click in another field, Step size for example, to see the calculated values described below.

Scan speed is calculated, 0.100 °/s in this case.

Total time is calculated, 13 min. 20 sec. in this case.

Set up the **Position** as follows:

2 Theta: **45°** *This will be the center position of your scan (5° to 85°).*

Unit cell: **None**

Before you click **Start** , open the shutter (or confirm that the shutter is open).

An open shutter is indicated by the SHUTTER OPEN light.

Shutters are not controlled by computer software. Instead, they are controlled by buttons at the upper left and lower right corners the PW 1830 (X-ray generator) control panel. Simultaneously press the **Shutters button** (upper left corner) and the **Open button** for Shutter 4 (lower right corner).

Click **Start** to begin the manual scan.

The goniometer should move rapidly to the starting angle of the scan (5°). Then, it should move slowly from 5° to 85°, as it performs the scan. You should also see a graph appear in a new window on the computer display as data are collected.

Realize how the 5°-to-85° scan has been specified. The starting angle of the scan is the 2-theta position (45°) minus half of the range ($80^\circ/2 = 40^\circ$); the ending angle of the scan is the 2-theta position plus half of the range. Thus, the scan will run from 5° to 85°, with a step size of 0.040° and a time per step of 0.40 seconds. This is appropriate for most minerals. For clays, you may wish to start the scan at 2° or 3°.

When the manual scan is completed, you may wish to close the shutter, particularly if you anticipate it being some time before the detailed (programmed) scan is executed. To close the shutter, press the close button for shutter 4.

9. Study the result of your manual scan to determine appropriate scan range, counting time, and step size.

Change axes

Click the **Axes...** button. In the **Change Axes** window change units to **Counts/s** and click **Close** .

Scan range

Note the range of 2θ over which reflections (peaks) occur. It is only necessary to scan this range.

For the purpose of an example, lets assume that reflections only occur between 10° and 50° .

Zoom in and acquire statistics on a tall, low-angle peak

Position the pointer on the graph just above and to the left of a tall low-angle peak. Press the left mouse button and drag the mouse over the area of the graph to be zoomed (enlarged), such that it includes the tall low-angle peak.

*If you make a mistake here, press the right mouse button and select **Fullsize** from the resulting menu.*

Click the right mouse button and select **Peak mode** from the resulting menu. **Net height** (count rate), **FWHM** (Full Width Half Maximum) and other statistics for the tallest peak in the zoomed area are reported.

Counting time

You should collect 1,000 to 10,000 counts on this tall peak. Divide 1,000 counts by the count rate to determine the minimal counting time. For improved statistics, use longer counting times; divide up to 10,000 counts by the count rate.

For example, if the count rate (Net height) is 180.96 counts/s, divide 1,000 counts by 181 counts/s. This yields 5.5 seconds. Six seconds at 181 counts/s will provide nearly 1100 counts; whereas 7 seconds will provide 1270 counts. Thus, 6 or 7 seconds would probably work well for this example. Note that these are rather long counting times and that larger slits may improve the situation (provide higher count rates); counting times of a second or less are more typical.

Step size

Precise definition of the peak requires at least 8 to 10 steps (evenly spaced points of measurement) across its FWHM. Divide the FWHM by 10 to determine an appropriate step size. Smaller steps contribute to long scan times. Twenty steps or more are excessive.

For example, if the FWHM is 0.2082° , divide 0.2082° by 10. This yields a step size of $0.02^\circ/\text{step}$.

Record the measurement conditions (scan range, counting time, and step size), as determined above.

You can now close all windows related to the manual scan, but do not go off-line.

10. Create a scan program.

Select **File / New Program...** You are presented with a succession of windows.

In the 1st window (**New Program**), select **Absolute scan** and click **OK** .

In the 2nd window (**Prepare Absolute Scan [Program 2]**), make the following settings:

Configuration: **Standard**

Scan Axis: **Gonio**

Scan mode: **Continuous**

Start angle: ... as per results of manual scan. *For example, 10°*

End angle: ... as per results of manual scan. *For example, 50°*

Step size: ... as per results of manual scan. *For example, 0.020°*

Time per step: ... as per results of manual scan. *For example, 7 s*

Scan speed and Total time are calculated. *However, you must click in another field to see these calculated values.*

In the case of the example, the total time is 3 hours and 42 minutes!

Without closing this 2nd window (**Prepare Absolute Scan [Program 2]**), select **File / Save As...** .

In the save dialog, give the program a meaningful name and click **OK** .

In the case of the example, a name like 10,50,0.020,7s might work well.

Close the **Prepare Absolute Scan [Program 2]** window.

11. Open the shutter (shutter 4), or confirm that it is open. (See step 7.)
12. Execute the scan program.

Select Measure / Program... .

In the 1st window (**Open Program**), select a scan program and click **OK** .

This is likely to be the new program you created in step 10.

In the 2nd window (**Start**), enter a Data set name and click **OK** .

If you wish to export this data, use a DOS-compliant name (5 characters, etc.).

Confirm that the shutter is open and click **OK** in the next alert (!) window.

The detailed scan should now be underway.

The diffraction data is added to the database, i.e. written to disk, automatically; it need not be saved.

When the detailed scan is completed, you may wish to close the shutter, particularly if you have no immediate need for it to be open.

When the detailed scan is completed, you can exit **X'Pert Data Collector** software or minimize the window.