

PANalytical X-Ray Diffractometer with PW3830 Generator

Standard Operating Procedure

Revised on 02/15/2007

Booking of XRD time

1. Please book the XRD schedule first come/first serve in advance on the sheets kept out side of XRD Lab. Do not book more then two slots (2hrs) at a stretch for the convenience of others. If you need longer time, Please see **Prof Kowach** (Room Number 1116, Ext *5247 and *6039)(*Write your name, Lab name, room number and telephone number*)
2. If your scan requires > 6h, please run overnight
3. Stick to time of the schedule and please call next user after you are finished
4. Fill up the log book (Kowach group Only) kept near the XRD machine

Information to be filled in log book

1. Date and Time,
2. Program
3. Name of user
4. Sample name and file name
5. Angle range
6. Duration of run
7. Operation power
8. Off time if you are the last user.

TO TURN ON XRD:

1. Check the status of water chiller in the middle of the room. The cooling water switch should be located in the middle between XRD and XRF.
2. Turn the key switch clockwise to the on position in the front panel of X-ray machine.

3. Now the X-ray yellow light will illuminate on the front panel and the X-ray warning lamp.
4. If X-ray does not turn on, one of the fault lamps will be lit.
5. The starting voltage is 20 kV and starting current is 10 mA.
6. Using the + button, slowly increase the voltage to 40 kV. Wait for at least a minute before going to the next step. Green okay light will be on when the power stabilizes.
7. Then, increase the current to 20 mA. Again, green okay light will be on when the power stabilizes. (*presently it is allowed to increase to 30 mA*)
8. Wait for half an hour to warm up the machine completely.

INSERTING THE SAMPLE:

1. Loosen the set-screw on the sample chamber cover. Then, gently pull the cover to open up the sample chamber
2. Gently push down the wire clip inside the chamber by a finger or tweezers, then insert your sample. Your sample should be facing up. Make sure the sample is well centered.
3. Slip the cover back on and make sure it fits properly. You should hear a click. Tighten the set-screw back. If the cover is not closed properly, the interlock system will not let you open the shutter for X-ray to come into the sample stage.
4. Push “shutter open enable” button and at the same time push “4 open” button. The shutter lamp will light on when the shutter is on.

X-RAY SCAN PROGRAM:

1. Double click “Philips X’pert Data Collector”.
2. Type user name as “User-1” (case-sensitive) and password as galaxy.
3. Select CONTROL! from the menu. And select “CONFIGURATION1” and click ok.

4. Messages regarding power generator, divergence slit, and etc will pop up. Check if there's any type E message (E = error). If everything message is type A and click ok.
5. Then, configuration window will pop up. Do not change any options except the voltage and current of power generator.
6. To create a new scan program, go to FILE menu and select NEW PROGRAM.
To open an existing program, go to FILE menu and select OPEN PROGRAM.
7. For the new program, choose "absolute scan" and click ok. Type in X-ray scan values for your program.
8. Close the window by clicking x in the upper right corner. Pop-up window will ask you if you want to save the program. Click yes.
9. Name your program name and make it available to all users or to User-1 only.
10. Now go to MEASURE menu and select PROGRAM. Open your program and name the filename of the X-ray scan.
11. When you click okay, pop-up window will ask you if the shutter is open. Make sure the shutter is open and click okay. Now the goniometer should move to the designated position and the scan data will be displayed in the window.

DATA FILE EXPORT:

1. Bring a 3.5 floppy disk. Insert it into A drive.
2. After X-ray scan is completed, open “X’pert Graphics + Identify” program. Sign in as “User-1” (case-sensitive) with the password of “galaxy”
3. Select FILE menu and choose open NEW GRAPH.
4. Select your sample filename of the X-ray scan.
5. Now you will see the graph of your X-ray diffraction scan.
6. To save as a text file, go to FILE menu and choose PRINT.
7. Click the box for “textural report” and scroll right to find a down arrow. Select “XY table” from the drop down menu.
8. Click “OK” and choose text file.
9. Save the file with your sample name as a text file into A: drive.
10. Don’t forget to take your floppy diskette with you.

DATA FILE EXPORT IN OTHER FORMATS

1. Click on “Philips X-pert organizer” to open the program
2. Login as “User-1” Use Password “galaxy”
3. Click on **database** on the upper menubar to see down scroll options
4. See options across the “**Export**” options
5. Click on “**scan**”
6. Select the desired file (Use either scroll down or filter options)

The file will appear in box as

Scan name	Scan type	Folde r	File name	Samp Id	RD	SD	OD	OSD	UDF	DAT	SCN	Dnn	Xnn
		****			√				√	√			

**** Select destination folder by scroll, “locate the floppy drive as **a:**”

√ select the formats to be exported

Once the file exported the table will disappear. Then you can close or go to another file to export in similar manner.

TO TURN OFF:

1. Bring down the current slowly back to 10 mA. Then, bring down the voltage slowly back to 20 kV.
2. Turn off the X-ray generator by toggling key switch to OFF. Do not push power off button.
3. Turn off the cooling water to the XRD in order to prevent any water condensation on the X-ray tube.

Use of XRD software available with XRD unit

XRD unit (PANalytical) has two important additional analysis software along with the data collection software, which are commercial and powerful for analysis of your XRD patterns.

1. The Graphic software “X’pert Graphics + Identify” is used to plot the XRD pattern and data export as two column XY format.
2. This software can be used to analyze the XRD pattern for the phase(s) present in a pattern and to find peaks accurately.

3. To analyze the XRD pattern open pattern as new graph and then find peak from the analysis heading.
4. Click the search match from the same heading.
5. Analyze as precise first and look for your compound or close to your XRD pattern by looking peak position of the observed and reported data.
6. If it does not give suitable results relax by choosing other options

1. The Graphic software “X’pert Organizer” is used to export data to different formatted data suitable for many other softwares. The data can be exported by clicking
Export
Scan

Select the file to be exported and destination where to be exported and the kind of file exported.