Powder X-ray Diffractometer
Instruction Manual

Philips XRG

University of Nevada, Reno

SEM-200

Adapted from Indiana State XRG Operating Manual
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Principles of X-ray crystallography

- A collimated beam of monochromatic x-ray photons, from a copper source, is directed at a finely ground powder or flat sample placed upon a glass slide. The sample diffracts the radiation at characteristic angles.

- The intensity of the diffraction as well as the angle between the incident and diffracted beam (2Θ) can be used to identify the minerals within or characteristics of the measured sample.

Safety Precautions

1. ALL OPERATORS MUST BE TRAINED THROUGH BOTH EH&S AND THROUGH BENJAMIN KING!!

2. All operators must have X-ray safety training through EH&S

3. The Philips XRG-3100 X-Ray Diffractometer utilizes radiation that is readily absorbed by soft tissue. Severe damage to skin and/or longer term cancer risks is a possibility due to prolonged exposure or cumulative exposure over time.

4. The XRG-3100 system was designed many years ago and thus presents a higher risk source of radiation exposure than the double radiation shielding used in modern instrumentation. The radiation danger of this instrument will be frequently monitored by the University, however, any questions or concerns should be immediately directed to Dr. Benjamin King.
Sample Preparation

Glass slides of the appropriate length for the Philips XRG are available through Lakeside Microscope Accessories (Stock # 452 size: 27 mm x 46 mm x 1.16-1.27 mm thick)

Your sample should be placed roughly $\frac{1}{2}$-$\frac{3}{4}$" from the end of the slide in order to allow room to be place in the goniometer's sample holder.

For proper irradiation of the sample it should be ~1”x1” as shown above.
Operating the X-ray generator and Ganiometer

Preliminary Requirements

1. Both water coolers should be turned on before starting the instrument.

2. Observe the setting of the Goniometer’s $2\theta$ angle indicator to ensure that it reads 2.00-50° (degrees) setting outside this range can damage the instrument.

   a) Operating Less than 2° will increase the possibility of a direct x-ray beam impinging onto the detector – this may well damage the scintillator and/or photomultiplier detector.

Observe that the $2\theta$ angle displayed is 40.00 degrees.
Turning on the X-RAY Generator:

1. Be sure that the KV is set to 25 (minimum setting) and the MA to 10 (starting).

2. Turn on the Power key switch (clockwise) on the left side of the generator control panel and notice that the red X-RAYS OFF light comes ON.

3. Be sure that the Current Control Switch (at the right end of the panel) is set to OPERATE, look for red LED (not to Minimum MA) and use the DISPLAY button switch to set the Panel Meter display to KV, again look for the red LED.

4. Be sure that the sample compartment cover is on, the tension screw is turned CW to lock it in place, and that the mechanical beam shutter mechanism has enabled the beam shutter.
5. Push the X-RAYS ON button (the second switch to the right of the key switch) and hold until the KV panel meter reading stabilizes at 25 KV.

6. Bring up the kV (high voltage) SLOWLY (5 kV steps) to **40 kV** maximum (for a copper, (CU), anode x-ray tube) by pushing the vertical stepping toothed dial down and waiting for the Panel Meter to stabilize after each increment before proceeding.

7. Toggle the DISPLAY switch to indicate mA (Milliamperes) and use the mA stepping switch to SLOWLY increase the current in one milliampere steps – toggle both digit switches at once in going from 19 to 20 mA. Do not exceed 20 mA for a Cu x-ray tube.
a. Using less than 20 MA may prolong the life of the x-ray tube, but will reduce the intensity of the X-Ray beam and diffraction peak heights.

8. Push the Shutter Open button on the right side of the Generator Panel.

9. Observe that the orange X-Ray beam light at the X-Ray source comes ON and know that an X-Ray beam is now passing into the sample compartment.
Running Samples:

1. Close the Beam Shutter – using the white pushbutton on the right side of the generator panel. Notice that the orange beam shutter light is OFF and know that the X-Ray beam is OFF.

![Beam Shutter Image]

2. Open the Sample Compartment cover, release the tension screw and then while gently pressing the lever as shown above pull the cover toward you. (This mechanical shutter lockout is a secondary mechanism to ensure that the x-ray beam is blocked from entering the sample compartment.

![Sample Compartment Cover Image]

3. Remove any sample holder in the chamber by pushing down on the spring clip with a spatula or similar tool and remove the sample holder. With a damp tissue wipe out any residual sample powder from the chamber.

![Sample Holder Removal Image]

4. Insert a new sample/holder using the reverse procedure above – being sure that the sample is centered on the holder pedestal. Release the spring clip carefully and replace the chamber cover and lock it in place with modest CW tension on the locking screw.

![New Sample Holder Insertion Image]
**Setting Up Datascan Software**

1. Open the MDI Datascan 4 program on the CPU desktop

2. Using the controller window, drive the goniometer to your starting 2 theta value between 2-50 deg.

3. From the “scan” drop down menu, there are two main options
   
   a. *Quick Scan*- this is for a single run that has a specific set of parameters. When opened one simply enters the parameters and starts the run.

   b. *Routine Scan*- this is used to run a series of scans or a scan that will be used multiple times. When open you must double click the sample name to enter the data and instrument parameters

**Shutting Down the XRG System:**

1. Close the Shutter and Remove the Sample – replace the sample compartment cover.

2. Reduce the HV current in one mA steps to 10 mA (starting setting).

3. Toggle the digital display selector to read KV and bring down the voltage in one KV steps to 25 kV (the minimum setting).

4. Push the X-RAYS OFF button.

5. Turn off the Generator with the Key Switch.

6. Fill out the System Log Book noting the hours used, date, operator and enter any malfunctions noted in the Maintenance Notes section, etc.

7. Turn off the Water Coolers