

XRD Quick Start Guide

The following instructions are provided as a Quick Start Guide for powering up, running measurements, and shutting down the X-ray diffractometer (XRD) system.



Warning: X-ray diffractometers produce potentially hazardous radiation. Only users who have completed the *Radiation Safety* section of the DMSE Safety Training, and who have received training by qualified department staff or faculty are permitted to use this equipment. Contact the DMSE for training information.

Notice: You must have an assigned dosimeter ring badge to operate the XRD System. If you do not have one, you will not be provided with user access. Please follow the directions below to order a dosimeter ring badge for this system.

1. Order your dosimeter ring badge by calling **Landauer** at **1-800-323-8830** and tell them that you need to add a dosimeter ring badge to the Johns Hopkins' account.
2. Tell them our account information: **Account #: 2627, Series: A24** (Hufnagel).
3. Give them **your name, date of birth, and social security number**.

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Getting Started

Powering up the XRD system and loading a specimen

1. Sign in to the logbook.
2. Retrieve your dosimeter ring badge and place it on the index finger of your dominate hand.
3. The system should be in *Idle Mode* or *Standby Mode*.
 - a. *Idle Mode* includes
 - i. Water chiller is on (lights on chiller unit are on),
 - ii. The “X-ray On” light on top of the XRD is illuminated,
 - iii. Values for $^{\circ}\theta$ and $^{\circ}2\theta$ should appear on the control panel,
 - iv. High Tension on the Control Panel reads values of 40 kV and 20 mA.
 - b. *Standby Mode* includes
 - i. Water chiller is off,
 - ii. System is powered down – all lights off,
 - iii. The Control Panel has Standby illuminated.

Notice: If the XRD is not in either the *Idle* or *Standby* mode, please contact the DMSE Laboratory Coordinator; DO NOT turn on or use the instrument in this circumstance.

4. If the system is in *Standby Mode*, follow the steps below to power up the system; otherwise, continue to Step 5.
 - a. Turn on the water chiller.
 - b. Ensure that the doors to the XRD system are securely closed.
 - c. Press the Power button on the Control Panel and wait for the system to stop initializing – this will take approximately 1 minute. Afterwards the Control Panel will read values of 30 kV and 10 mA.
5. Turn on the hutch light using the switch on the Control Panel.
6. Slide open the instrument doors and load the specimen into the mounting clip and positioning it against the semicircular sample stage.

Notice: For accurate data, it is very important that the top surface of your specimen be exactly flush with the bottom surface of the sample stage.

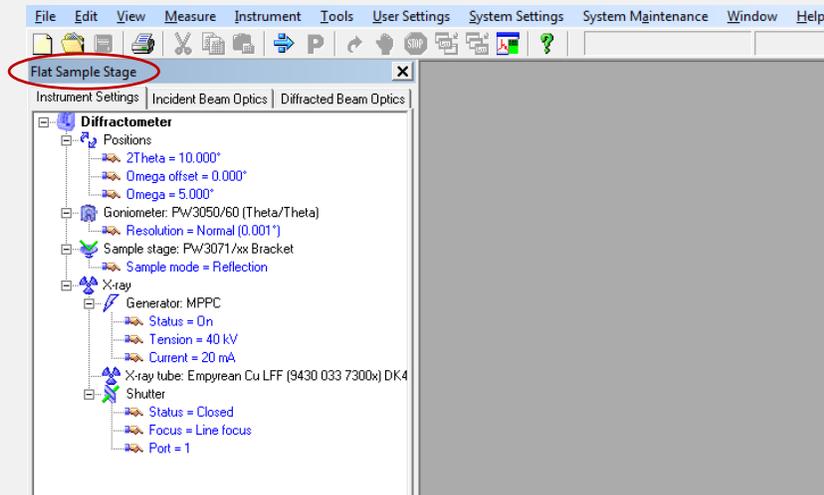
7. Close the doors to the hutch and turn off the light.

Connecting the software to the XRD system

1. Log in to the instrument computer using your JHED ID.
2. Launch the *Data Collector* program and log in.

3. Connect to the diffractometer by selecting *Instrument* from the top menu bar and choosing *Connect (Instrument → Connect)*.
4. Choose the *Flat Sample Stage* configuration when prompted – it's the only option – and confirm the instrument status by clicking OK when the system prompts you with the assumed instrument conditions in the software.

Notice: A control window should appear on the left side of the computer screen that is titled *Flat Sample Stage*.



Running a Scan

Setting scan parameters

1. Open the scan program by selecting *File* from the top menu bar and choosing *Open Program*.
2. Double-click on *1 - XRD Default Scan Parameters* in the list of available programs; this will open a new window with a list of scan properties.
3. Adjust the parameters for Start Angle, End Angle, Step Size, and Time per Step to reflect desired measurement parameters for your sample. Standard default inputs are provided below.
 - a. Start Angle: 5°
 - b. End Angle: 100°
 - c. Step Size: 0.02°
 - d. Time per Step: 10 seconds
4. Choose *File* from the top menu bar and *Save* the program.

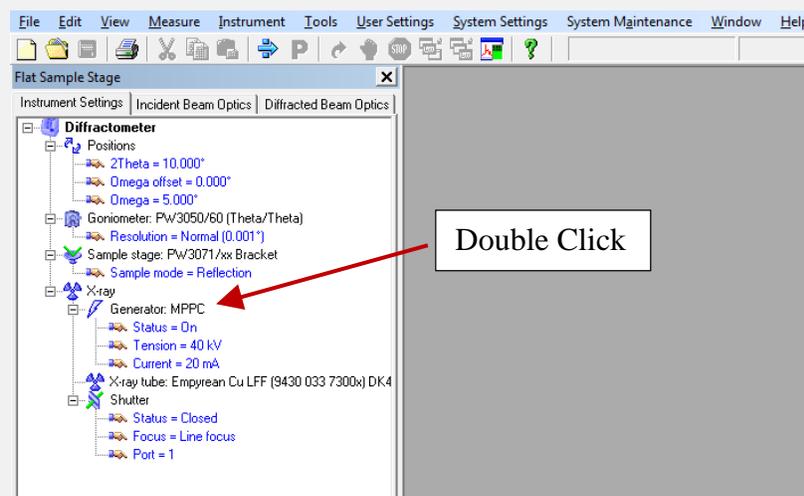
Notice: If any of the settings have been changed by clicking the *Settings...* icon, please save the program using the *Save As* selection and save it under a different file name. Normal use should not require a change to the settings.

5. Close the window by selecting the red “X” box in the top right of the window.

Initiating the scan

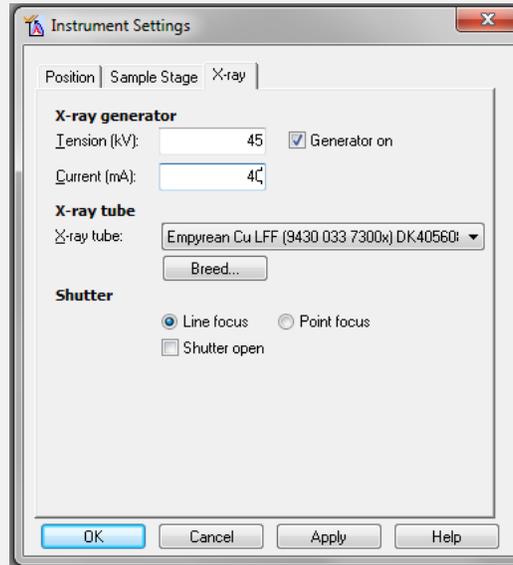
1. Double click on the *Generator* heading in the *Flat Sample Stage* window to open the instrument settings.

Notice: The *Generator* heading is indicated by the red arrow in the image below.



2. Set the Tension to 45 kV and the Current to 40 mA by entering the values in the appropriate boxes and clicking OK; after pressing the OK button, the window will disappear and the control panel on the XRD system will reflect the changes in the voltage and current display settings.

Notice: No other setting should be changed in the *Instrument Setting* window.



3. Choose *Measure* from the top menu bar and select *Program (Measure → Program)* and double click on the program *1 - XRD Default Scan Parameters* in the pop-up window.
4. Enter the file name and check to ensure the proper folder is selected for storing the data.
5. Start the scan by selecting OK. The system will automatically open the shutter, run the scan and close the shutter.
6. When the scan is complete, close the scan window. The data are saved automatically.

Notice: If you have other scans or specimens to run, you may leave the x-rays at this power while you run new scans or load new samples to run - the shutter on the X-ray tube automatically closes after a scan is complete and you will be able to safely open the doors when the interlocks are not engaged. Otherwise, if all scans are complete, place the system in stand-by using the instructions in the next section.

Standby and shut down procedures

There are two options for shutting down the XRD system. Standby Option #1 is used when the system will be used again in the next 24 hours, Standby Option #2 is used when the system will be not be used for over 24 hours.

Notice: Use shut down procedures for Standby Option #1 when using the XRD system Sunday morning through Friday afternoon.

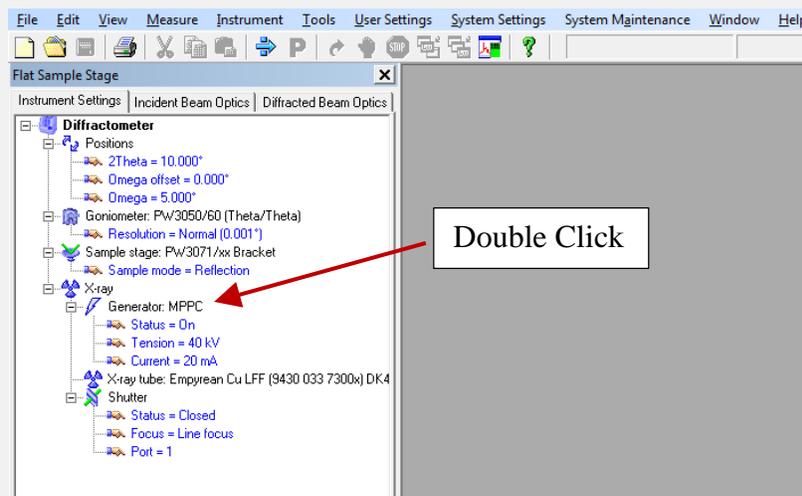
Use shut down procedures for Standby Option #2 when using the XRD system after 3 pm on Friday and on Saturday.

Standby Option #1

Use this procedure when using the XRD system Sunday morning through Friday afternoon.

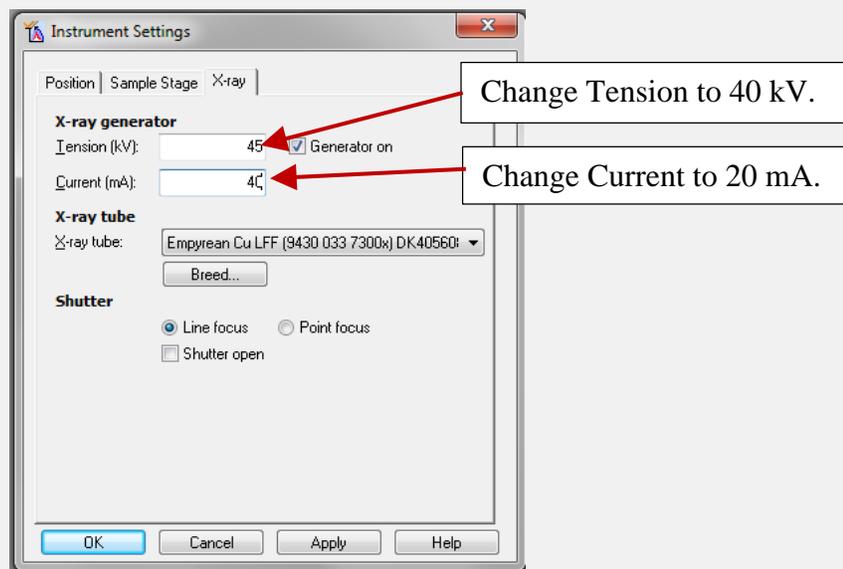
1. Double click on the *Generator* heading in the *Flat Sample Stage* window to open the instrument settings.

Notice: The *Generator* heading is indicated by the red arrow in the image below.



2. Set the Tension to 40 kV and the Current to 20 mA by entering the values in the appropriate boxes and clicking OK; after pressing the OK button, the window will disappear and the control panel on the XRD system will reflect the changes in the voltage and current display settings.

Notice: Change the Tension to 40 kV and the Current to 20 mA; no other setting should be changed in the *Instrument Setting* window.



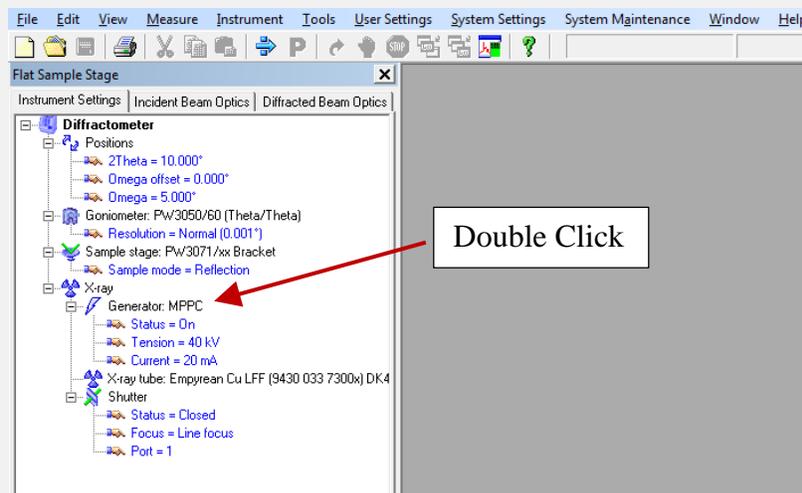
3. Disconnect to the diffractometer by selecting *Instrument* from the top menu bar and choosing *Disconnect (Instrument → Disconnect)*.
4. Close the Data Collector program and log off of the computer.

Standby Option #2

Use this procedure when using the XRD system after 3 pm on Friday and on Saturday.

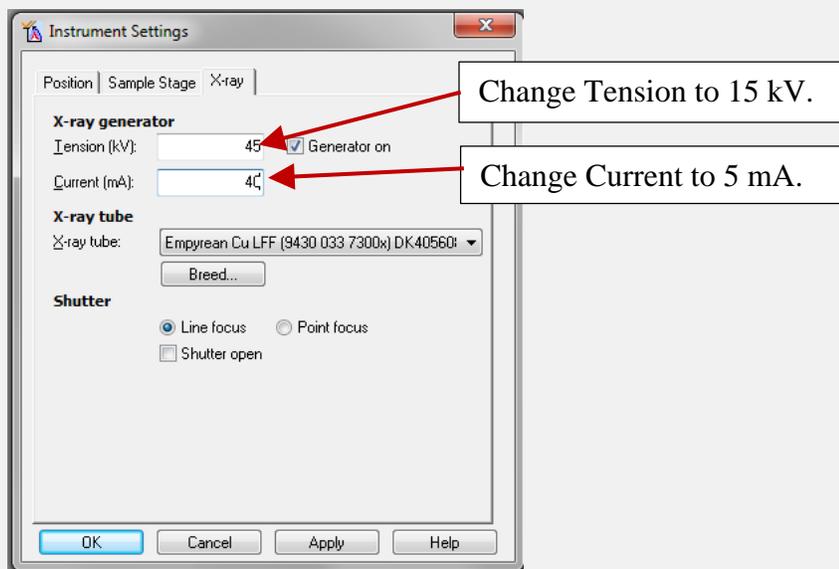
1. Double click on the *Generator* heading in the *Flat Sample Stage* window to open the instrument settings.

Notice: The *Generator* heading is indicated by the red arrow in the image below.



2. Set the Tension to 15 kV and the Current to 5 mA by entering the values in the appropriate boxes and clicking OK; after pressing the OK button, the window will disappear and the control panel on the XRD system will reflect the changes in the voltage and current display settings.

Notice: Change the Tension to 15 kV and the Current to 5 mA; no other setting should be changed in the *Instrument Setting* window.



3. Disconnect to the diffractometer by selecting *Instrument* from the top menu bar and choosing *Disconnect (Instrument → Disconnect)*.
4. Close the Data Collector program and log off of the computer.
5. Wait 1 minute.
6. Press the *Standby* button on the *XRD Control Panel*.
7. Turn off the water chiller using the rocker power switch on the front of the chiller unit.