

Standard Operating Procedure: Rigaku Miniflex 600 X-Ray Diffractometer

(You MUST be trained by an authorized BSCMC trainer to use this instrument)



600 W Generator with copper target

+2° to +145° 2Θ scan range

NaI scintillation detector with graphite monochromator

D/teX Ultra 1D Si detector – ultra fast scans

Six sample automatic sample changer (ASC)

Zero background and airtight sample holders

Hazards: This unit produces a high intensity X-ray beam. Take all precautions to avoid exposure to the x-rays.

Warning: Beryllium! Do not touch the front window of either the x-ray tube or the detector as they contain Beryllium. Fumes of the dust from Beryllium and its compounds can be hazardous if inhaled!

Emergencies

Power failure

The diffractometer will shut down safely. When power is restored, it is necessary for the BSCMC staff to restart the instrument manually.

Cooling Water Suspension

The Miniflex 600 uses chilled-water for cooling the X-ray tube. The Huskris water chiller is installed on the right side of the instrument. In case of abnormal water flow or failure of the chiller instrument, the X-ray tube will shut down automatically and an error message will be displayed on the computer.

Emergency Stop

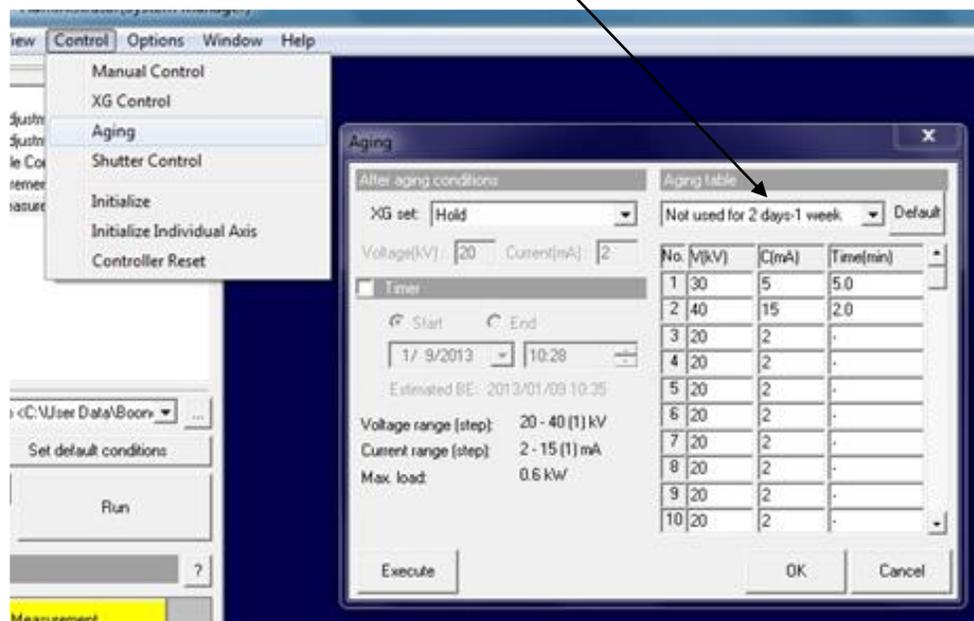
In case of fire or severe flood or water leak, shut off power using the RED colored Emergency OFF button located on the front side of the instrument. Do NOT remove the key! Avoid this method of shutdown unless absolutely necessary.



A. Start-up Procedure

1. Switch ON Huskris water chiller
2. Switch ON the computer
3. Press the “Power ON switch” on the front side of the instrument to turn on the power of the instrument
4. The Door Lock Button will blink. Press the Door Lock Button. The door is locked now.
5. Launch the “**Miniflex Guidance**” software. The initialization of goniometer, detector and any other attachments will start automatically.
6. Confirm that the “OPERATE lamp” on the front side of the instrument is ON in yellow, which indicates the normal status of the instrument.
7. Starting up the X-ray generator: Click on **Control Menu -> Aging**

Select the appropriate aging condition and execute



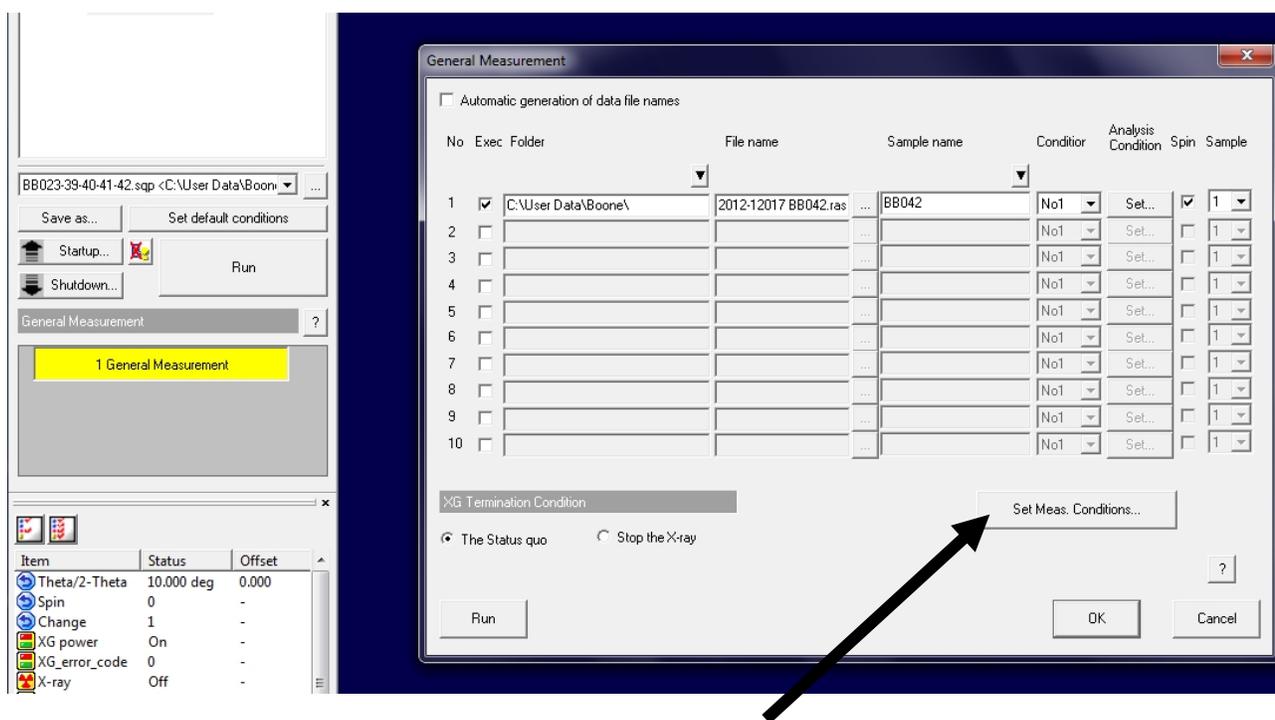
The normal operating condition is **40kV & 15mA**.

B. Sample Loading and Measurement

D/teX high speed detector along with the standard plate type sample holder is the default instrument configuration. If you are in need of the scintillation detector + monochromator (or) any other type of sample holder (six-sample automatic sample changer (ASC), air sensitive and zero background), contact the BSCMC staff.

1. Load the sample filled plate type holder on to the instrument. **Note that the sample holder also rotates with respect to the detector and hence, make sure to secure everything in place.**
2. Close and lock the door.
3. Select **General Measurement**

In this window, specify the names of the folder and file where you want to save measurement data and the sample location on the ASC.



The number in the condition box corresponds to the number of measurement condition set to be used. Measurement conditions can be entered by clicking on the **Set Measurement Condition** button.

In the Set Measurement condition dialogue box, enter your measurement conditions under appropriate measurement condition number tab.

Measurement condition

Slit Condition Background Condition

Variable slit system Variable+Fixed slit system Background Measurements

Optical devices

Soller(inc.) IHS DS

5.0 deg 10.0 mm 1.250 deg

SS Soller(rec.) RS Monochromatization

8.0 mm 5.0 deg 13.0 mm(Qper) kb filter (x1)

Measurement condition Edit the measurement conditions File Create

Preset Condition... Copy... Reset Individual configuration files

No1 No2 No3 No4 No5 No6 No7 No8 No9 No10

ScanAxis: Theta/2-Theta Mode: Continuous

Exec	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Comment	Voltage (kV)	Current (mA)	Background Conditions
<input checked="" type="checkbox"/>	10.000	70.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...
<input type="checkbox"/>	3.0000	120.000	0.02	10.0		40	15	Set...

Calculated scan duration : 00:06:49

OK Cancel

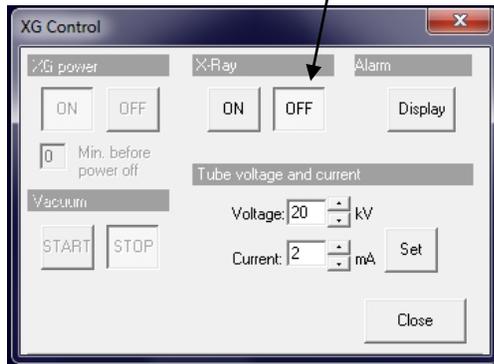
Save and close the conditions / general measurements dialogue.

Click **Run** to execute your scan.

The data will be saved automatically to the specified location at the end of scan.

C. End of Session

1. Remove your sample(s) and clean the area
2. Click **Control – XG Control** and then click the **OFF** button. When the x-ray generation stops, the yellow X-ray warning lamp on the top of the instrument will turn off.



3. End Miniflex Guidance
4. Wait for at least **15 or more minutes** and then press the Power OFF switch.
5. Turn OFF the Haskris water chiller
6. Update the iLab online calendar and you must enter the **number of scans** performed by using the “Add additional service charge” section on your iLab booking window.