Standard Operating Procedure

Rigaku SmartLab XRD

I. Powder X-ray Diffraction (PXRD)

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room A119C
810 West Campus Drive
West Haven, CT 06516

Version 1.6, June 2025

> **FOLLOW** the SOP strictly to keep the instrument in good condition. **No** explorations allowed on software unless permitted by lab manager

> **NEVER** use your own USB drive on the XPS computer. Data can be either uploaded to Yale Box, or copied to the Jump Drive provided by the Core.

> **NEVER** surf the web on the XPS computer to minimize the risk of the computer being hacked

> Users should **acknowledge** MCC in their publications. Please check the following link for details:
http://ywcmatsci.yale.edu/publications

> The core reserves the right to use the data for core promotion

Table of Contents

Rigaku SmartLab XRD Standard Operating Procedure

1 Introduction

a) Instrument features:

> Full automated alignment under computer control
> A high-efficiency 2D detector (Hypix 3000) with high-count rate
> Cross Beam Optics (CBO) permits easy switching between focusing (BB) and parallel beam (PB) geometries without reconfiguration
> In-plane diffraction arm for in-plane measurements without reconfiguration
> High temperature (~1500 C) measurements in air, vacuum and helium
> Air-Tight Sample Container for contamination free measurement

2) Location

Materials Characterization Core
Room A119C
810 West Campus Drive
West Haven, CT 06516

b) Primary Staff Contact

Dr. Min Li
Tel: 203-737-8270
Email: min.li@yale.edu
Office: ESC II, Room E119D

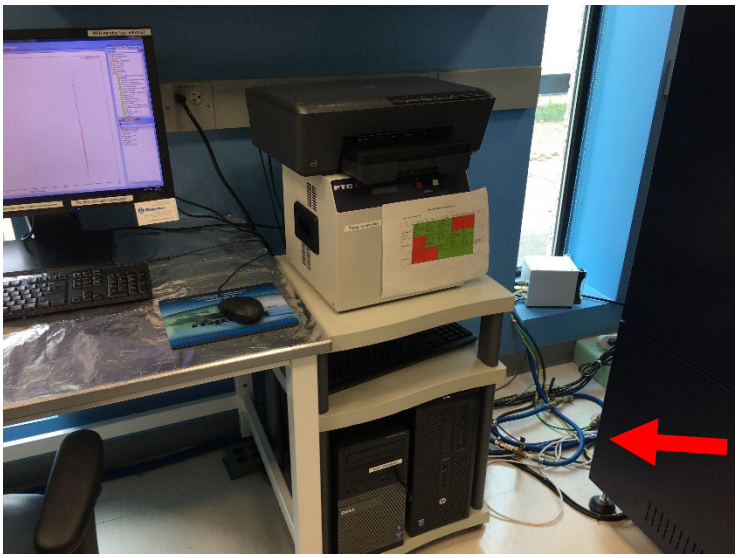
The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please **contact** the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. **No** explorations on program allowed unless approved by core manager.

2 System Status Check

1) Please check to make sure the instrument sliding door is closed and there is no beeping sound once getting into the XRD room.
2) If there is a beeping sound from the instrument and meanwhile the OPERATE light is flashing as show below. Hit the white OFF button  on the front panel above and contact manager immediately.

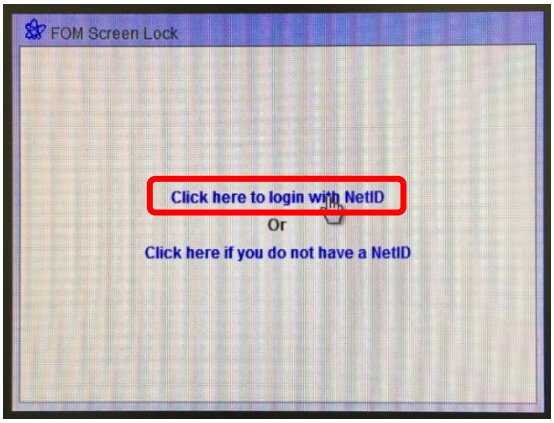
3) Meanwhile, please check the floor at the back corner of the machine and make sure no water leaking on the floor. If no water leaking and the manager can't be reached, before leaving, put a machine down note on the machine sliding door.



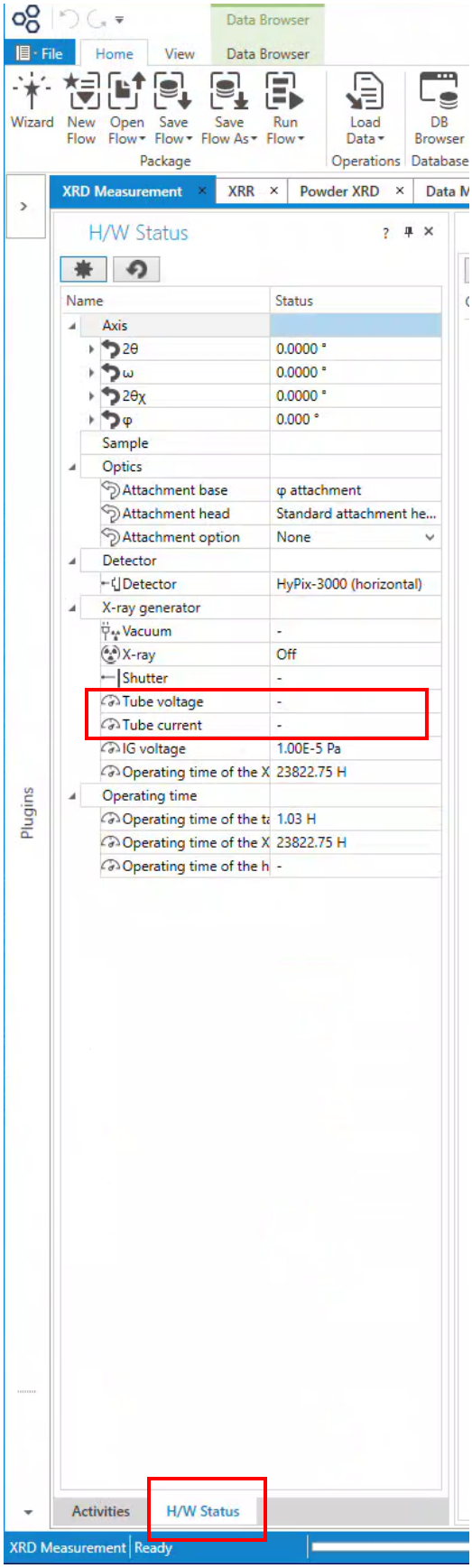
4) If there is water leaking, it must be taken care before leaving. Please go to the service corridor **A117** outside the Core in the next door and shut down the **XRD chiller** at the end of the corridor. The key is hang on the shelf right across the manager's office.
5) Come back to the XRD room and clean the water leak with paper tower.
6) Leave a machine down note on the machine sliding door.

3 XRD Computer Login

1) Sign in on the logbook.
2) Login FOM system: click on **Click here to login with NetID** to unlock the screen lock.



3) Check system status:
   a) Open the **SmartLab Studio II** software if it was closed (login: **administrator**, password: **rigaku**).
   b) Check the **H/W Status** tab below on the bottom left corner of the software window as shown below:
      > No number display on **Tube voltage** and **Tube current** if the X-ray is at shutdown status.


Data Browser
File
Home
View
Data Browser
Wizard
New Flow
Open Flow
Save Flow
Save Flow As
Run Flow
Package
Load Data
Operations
DB Browser
Database
XRD Measurement
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Detector
Detector
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X-ray generator
Vacuum
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X-ray
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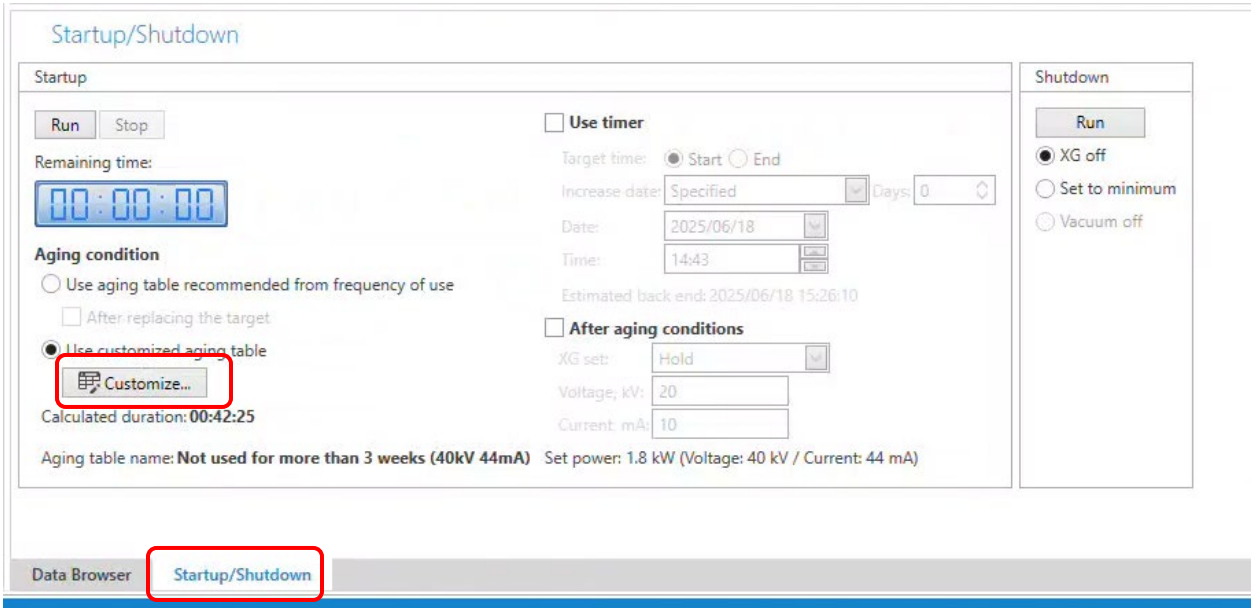
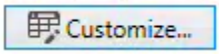
Plugins
Activities
H/W Status
XRD Measurement
Ready

c) If the **Tube voltage** and **Tube current** read **40 kV** and **44 mA**, skip **Step d)** and **e)** below.

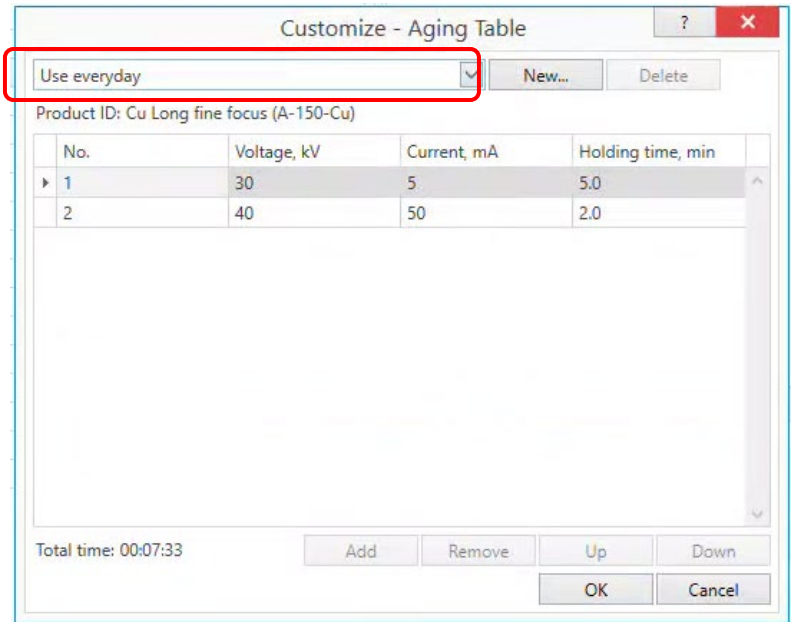
d) If the system is at shutdown status, click the 

button on the top menu to open **Startup/Shutdown** tab window below:

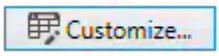


e) Click the 

button in the **Startup/Shutdown** window above to open the **Customize – Aging Table below**:



> Check the logbook record, if the machine was used within 24 hours choose **Use everyday** and click **OK** button to go back to the Startup/Shutdown tab window and click **Run** button to warm up the X-ray tube. It takes **15 minutes** for the system to reach the operation power of **40 kV** and **44 mA**.

> If the machine was NOT used for more than 24 hours, click the 

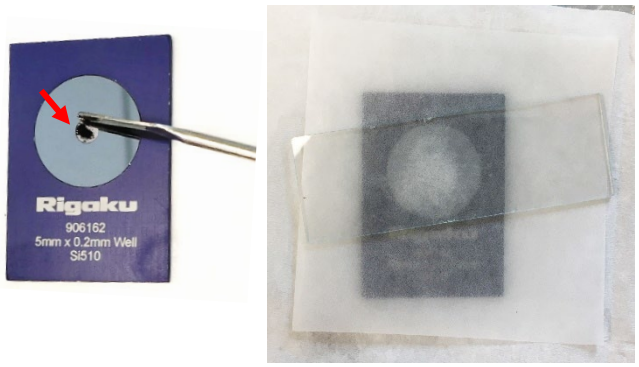
button in the **Startup/Shutdown** window to open the **Customize – Aging Table below** above. Choose **Not used for 2days-1week** and click **OK** button to go back to the **Startup/Shutdown** window and click **Run**. It takes 30 minutes for the system to reach the operation power of **40 kV** and **44 mA**.

4 Sample Preparation

1) **Wear gloves** and clean the sample holder with Kimwipe and provided **isopropanol solvent**.
2) Powder samples should be milled to below **5 µm** to minimize measurement deviations due to particle size effects.
3) Two types of powder sample holders, the **zero-background** holder and **glass** holder are provided as shown below. An amorphous hump signal could appear in the spectra on glass holder, which can be avoided if use the **zero-background** holder.

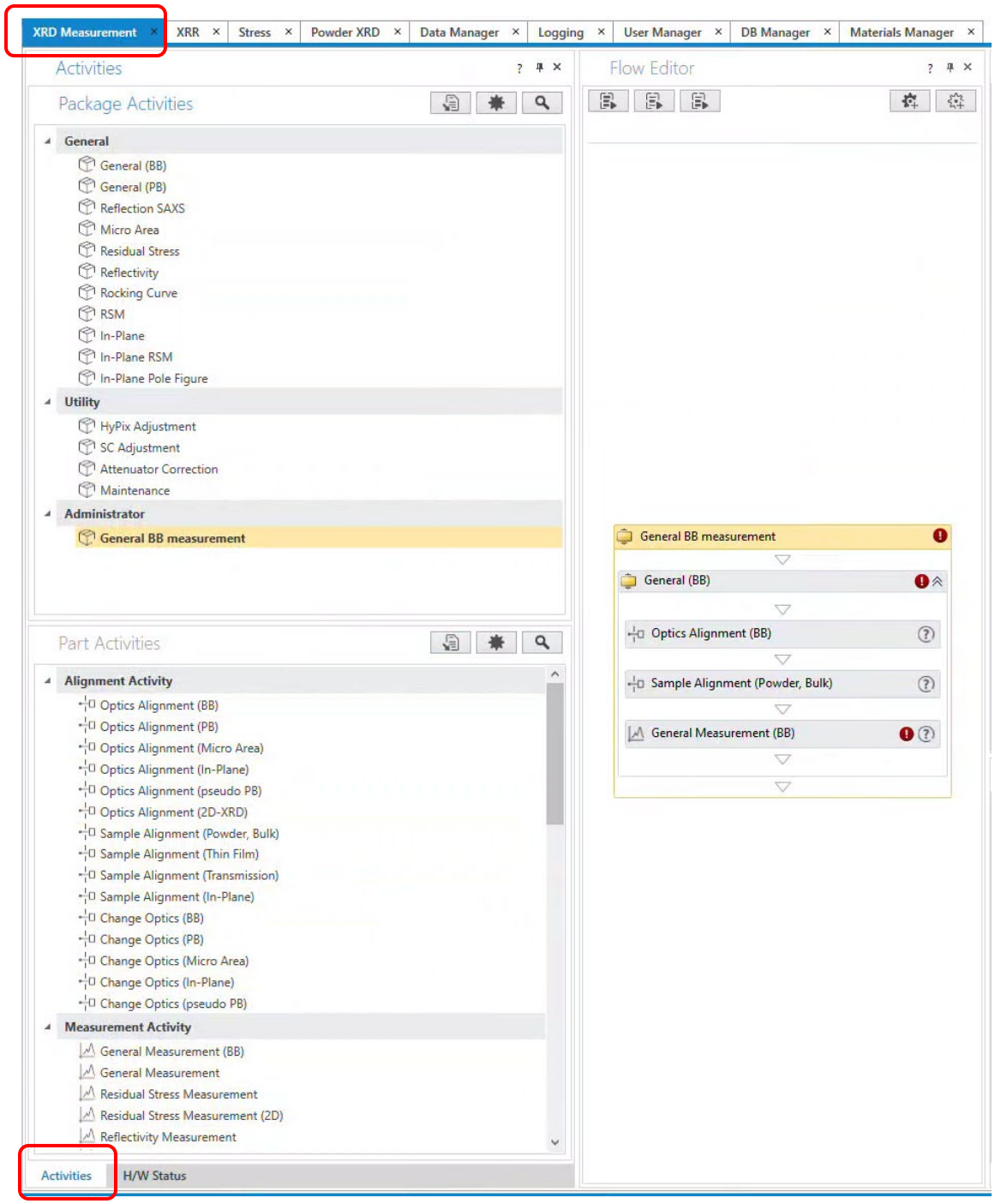
Zero-background | Glass holder

4) Put powder samples at the center of the circle on the zero-background holder or square on the glass holder as shown below; cover the powder with a **weighing paper** and press and rotate sample with a glass slide to flat the sample surface.



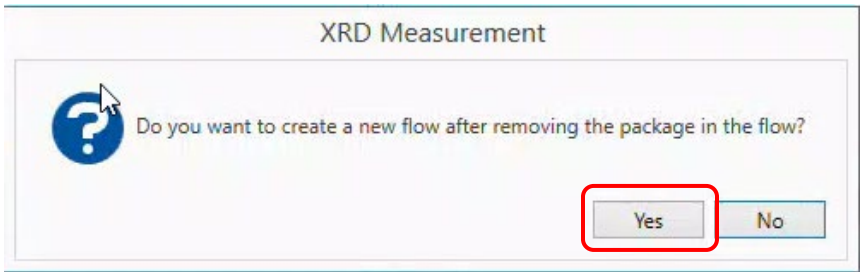
5 User flow handling (create, save and reload)

1) Click **XRD Measurement** tab on the top left corner of the window below the menu bar to open the measurement window, and click the **Activities** tab on the bottom left corner of the window to open the window below:

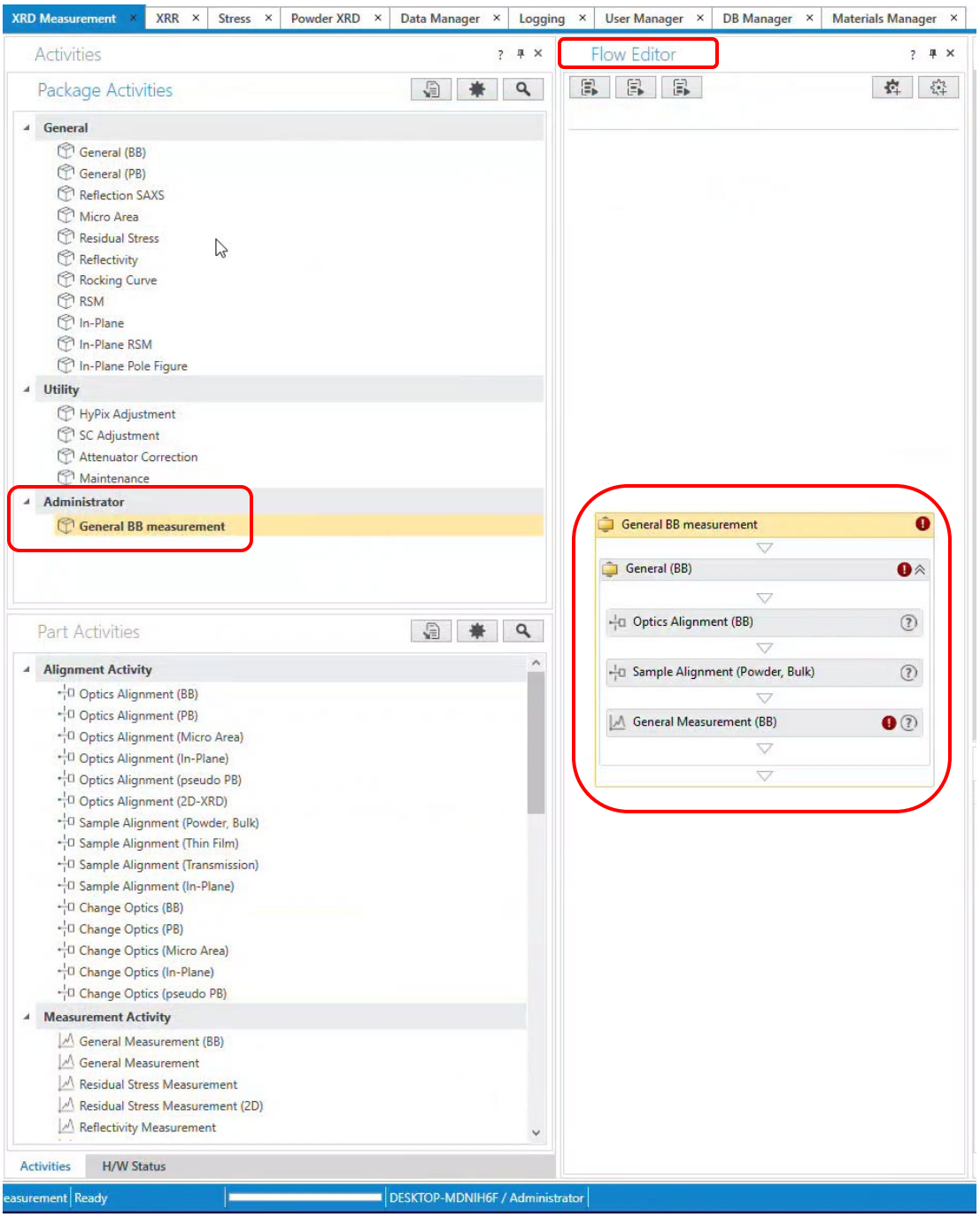


2) To create a new flow, click 

button on the top menu bar and click OK on the popup window below:



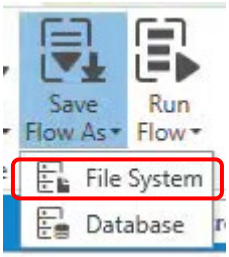
3) Double click the **General BB measurement** in the **Administrator** region as highlighted below to open the flow in the **Flow Editor** window on the right side.



4) To save modified/customized flow, click and highlight the flow under **Administrator** and click 

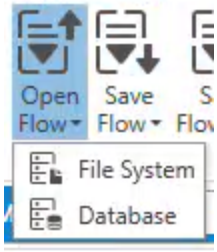
on the top menu bar. Select **File System** to save the flow in .rmrf format into

the specified user folder. The **Database** option should be used to save a flow for a user lab.



5) To reload a flow from **File System** or **Database**, click 

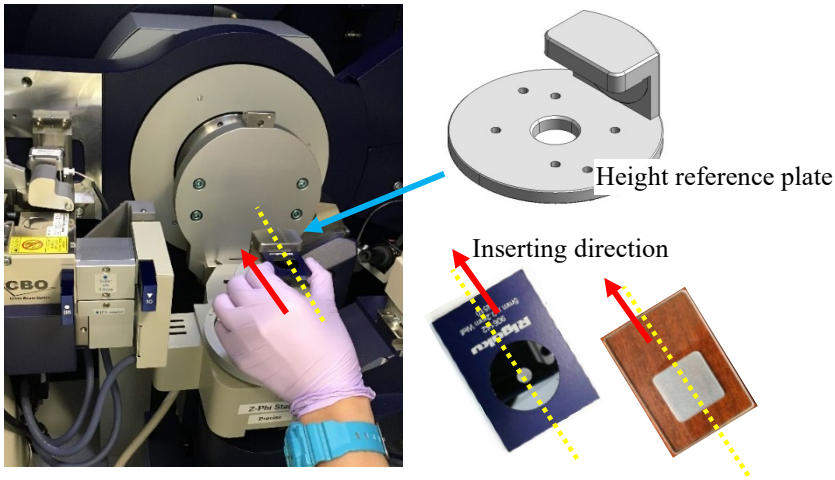
to clear the **Flow Editor** space. Click **Open Flow** button on the top menu bar, select **File System/Database** to open the flow in the **Open Flow** popup window.



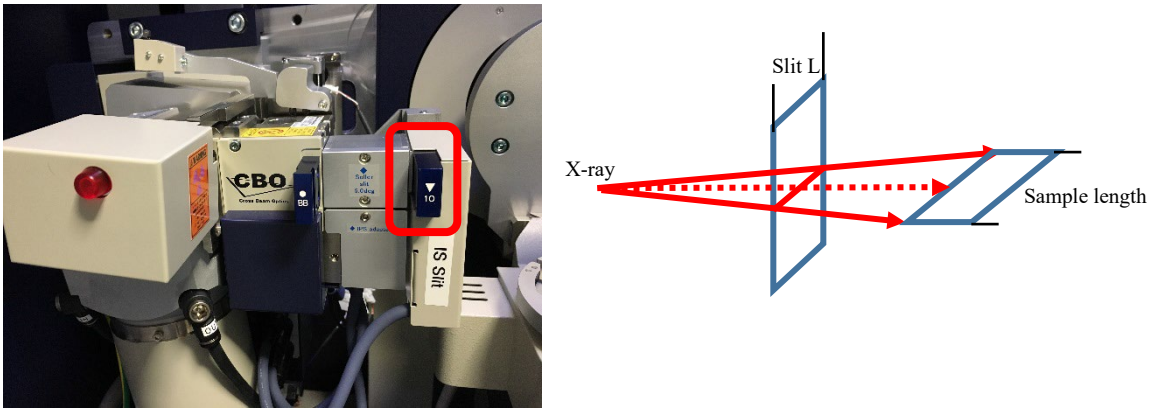
6 Sample Loading

1) Press the **Door Lock** button on the instrument cabinet door, wait till the button **flashes**, and then grab the handle (highlighted below) to slide the door open.

2) Push the glass or zero background holder all the way into the **height reference sample plate** as shown below. Align the holder to the center of the **plate** as highlighted by a dashed yellow line.
Note: If no enough sample to cover the entire holder, try to **extend** the sample area along the **longer** side of holder (dashed yellow line)



4) Insert the right slit size **IS L** (length limiting incident slit) as shown in the left picture below. Choose typical sizes from **2, 5,** or **10 mm**. The x-ray beam size is doubled when reaching sample surface as shown on the illustration below. For instance, if choosing 5 mm slit size, the exposed sample surface area/length will become ~ 10 mm across the X-ray beam.

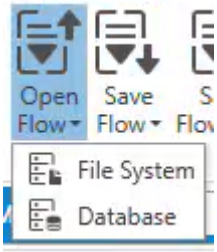


5) Close the cabinet door and press the **Door Lock** button.

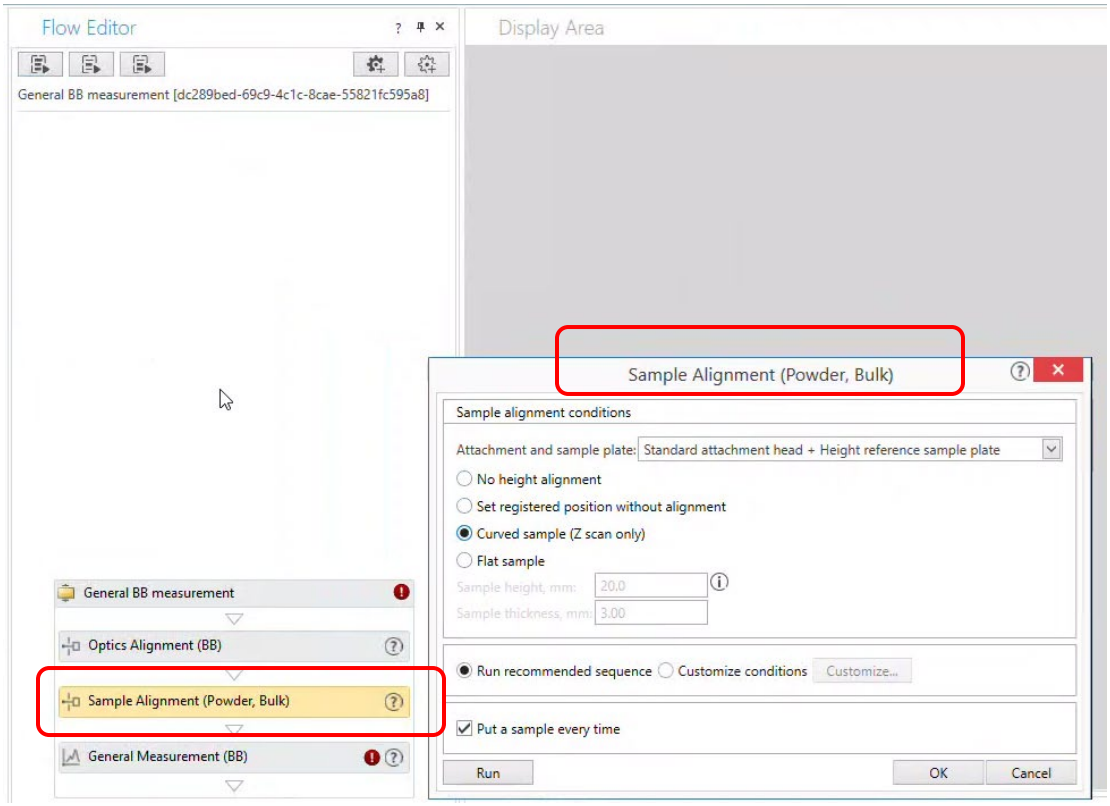
7 Sample Alignment

1) To open/reload a flow from **File System** or **Database**, click 

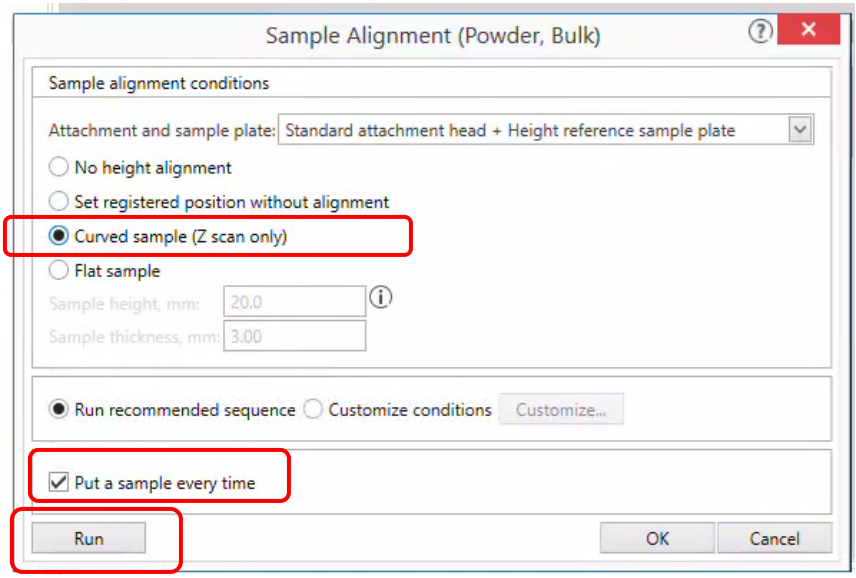
to clear the **Flow Editor** space. Click **Open Flow** button on the top menu bar, select **File System/Database** to open the flow in the **Open Flow** popup window.



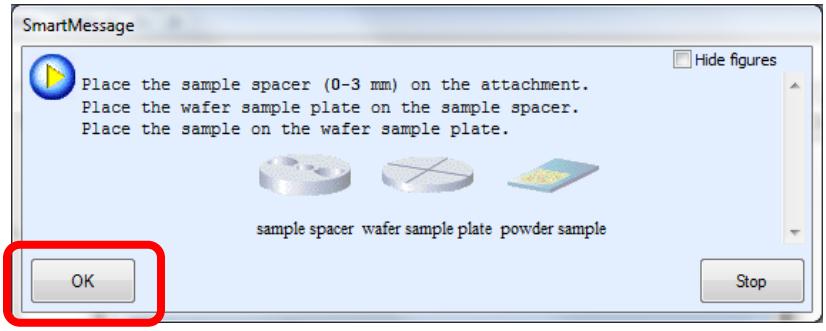
1) Click on **Sample Alignment (Powder, Bulk)** as highlighted below to open the alignment window.



2) In the Sample Alignment (Powder, Bulk) window below:



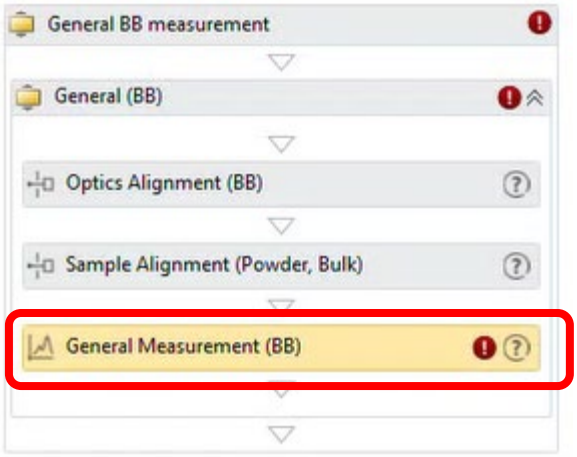
a) Choose **Curved sample (Z scan only)** for powder and bulk samples.
b) Check the box next to **Put a sample every time** click **Run** button.
c) For multiple samples with similar thickness or if powders flush with the holder, **no need** to repeat sample alignment.
d) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment (BB)** window will be back active in ~ 2 minutes after finish.



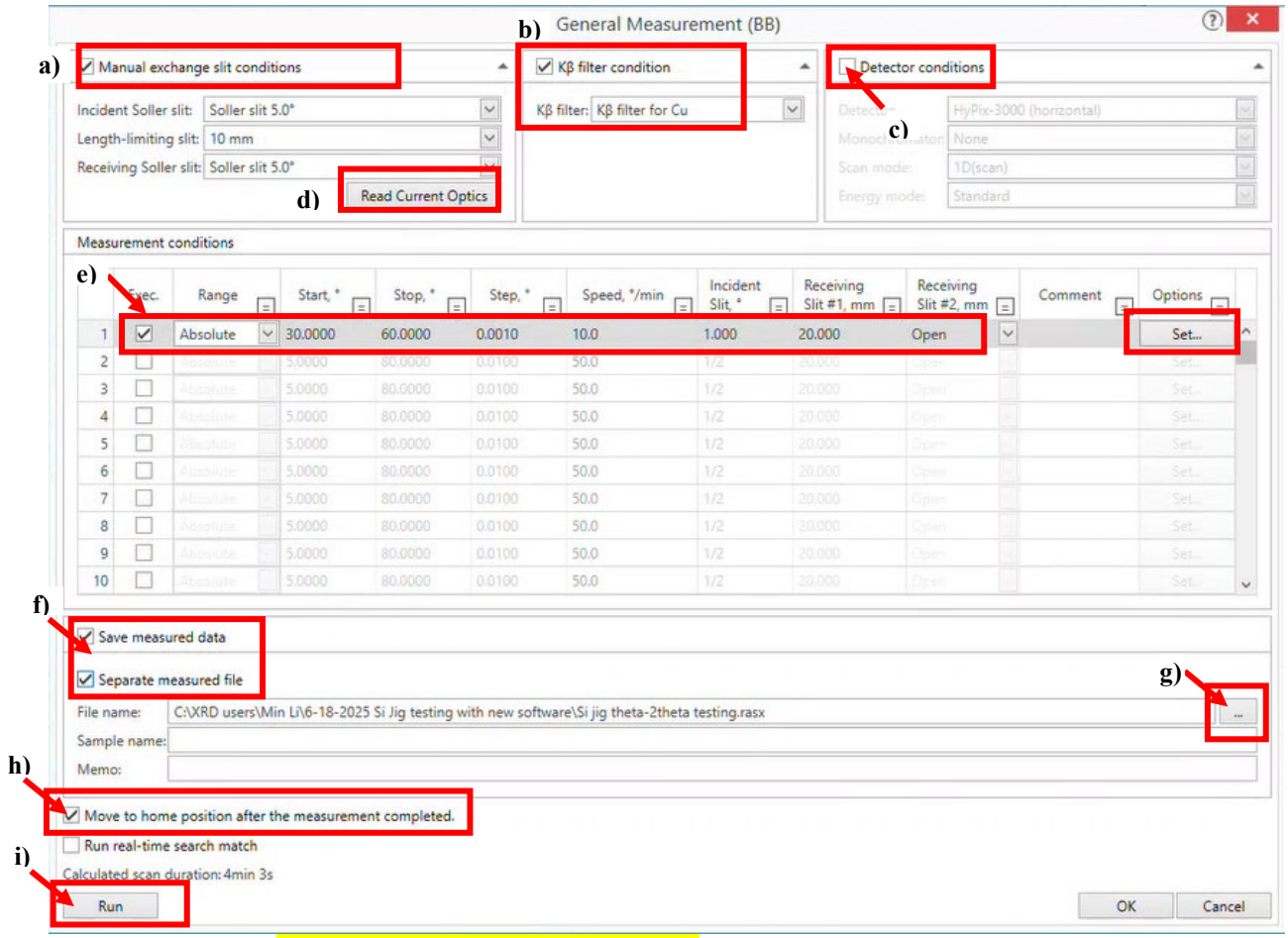
3) Wait till the sample alignment finishes and click **OK** on the **Sample Alignment (BB)** window in **Step 3a)** above to finish.

8 Sample Measurement

1) Click on **General Measurement (BB)** in the window below:

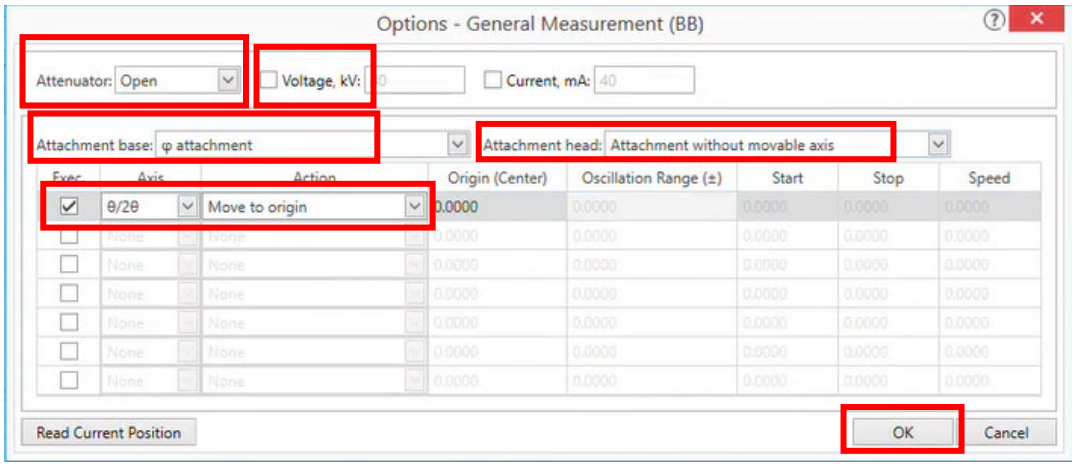


2) Perform following steps on the popup **General Measurement (BB)** window below:



a) Select **Manual exchange slit condition**.
b) Check **Kβ filter condition.**
c) Uncheck **Detector conditions**.
d) Click **Read Current Optics.**
e) Set measurement conditions:
> **Exec:** check small box to activate **Row 1**.

> **Range:** Absolute
> **Start (deg):** 10.0000 or larger**.** Never change to below **5**. The detector will be damaged.
> **Stop (deg):** 90.0000 or below. Typical scan range: 10 – 90 degrees.
> **Step (deg):** 0.0010
> **Speed/min:** 10.0000 or lower**,** choose lower speed to improve signal to noise ratio.
> **Incident Slit:** 1.
> **Receiving Slit#1, mm:** 20.000.
> **Receiving Slit#2, mm:** Open.
> Click **Set…** button to open the **Options-General Measurement (BB)** window below:

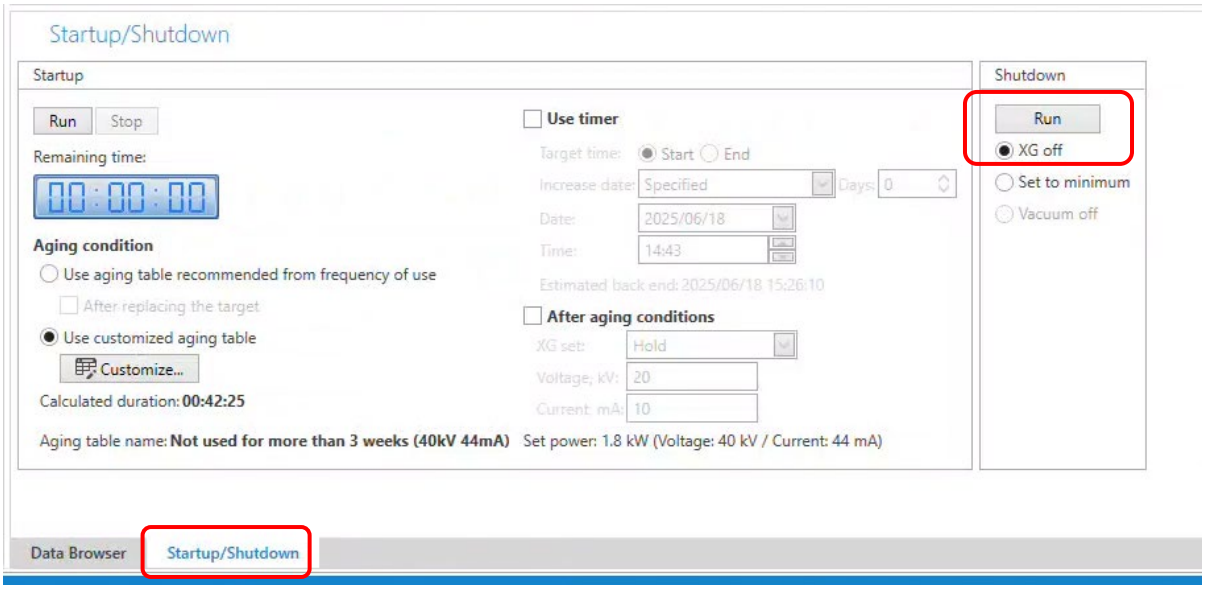


> **Attenuator:** Open**.**
> Uncheck **Voltage.**
> **Attachment base**: φ attachment.
> **Attachment head**: Attachment without movable axis.
> **Check Row 1:** select Θ/2Θ, Move to origin
> **Click OK.**

f) Check **Save measured data** and **Separate measured file** box.
g) Click the box to specify **File name** and folder.
h) Check the box next to **Move to home position after the measurement completed.**
i) Click **Run** button to start measurement.

9 Checklist after Experiment

1) Back up your data to Yale OneDrive cloud drive or using Core USB drive. **Do not** use a personal USB drive.
2) **Remove** sample from the stage. **DO NOT** leave samples inside XRD.
3) Shutdown instrument if there is no reservation within next hours. Make sure **XG off** is checked and click **Run** button in the **Startup/Shutdown** window below:



4) Wait until the **yellow light** on top of the instrument is turned off.

5) **DO NOT minimize** or **close** **SmartLab Studio II** software**.**

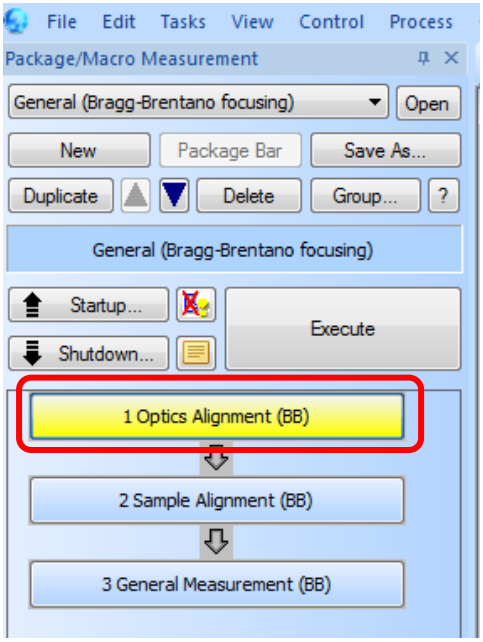
6) Logoff **FOM** program: click the  icon on the taskbar below to activate the FOM program and click **Logoff** button in the FOM window. If any issues occurred during scan, check "**Something wrong**" and type message in the **Comments** space. The message will be sent to the lab manager.

7) **Sign off** on the logbook.
8) **Clean** the bench sample preparation area, the sample holders and glass slides with clean wipes and isopropanol.
9) **Put** the sample holder and other tools back into the tool box.

Appendix: Optics Alignment

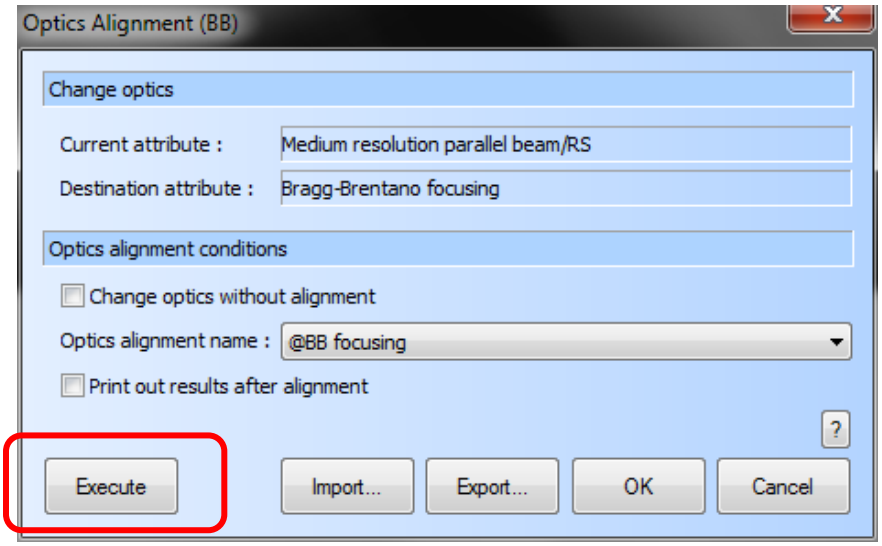
Only required if **PB** (parallel beam) mode was used and needs to be switched back to **BB** mode**.**

1) Insert the **BB** slit into the **CBO** adapter inside the machine
2) In the **SmartLab Guildance** window, click on 1 Optics Alignment (BB) in the **Package/Macro**



Measurement window below:

3) Click **Execute** button on the popup **Optics Alignment (BB)** window below to start optics alignment:



4) Follow the instructions on the popup **SmartMessage** window below to replace required parts and click **OK** to continue. The **Optics Alignment (BB)** window will be active after finish in ~5 mins.

5) Click **OK** on the **Optics Alignment (BB)** window in Step 2) above after finish.
6) Insert the **K-Beta** filter back the receiving slit box #2.
7) Remove the **Center_slit** from the stage.