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 1.1, February 2019

> **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

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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

Zishan Wu, Lab Assistant
zishan.wu@yale.edu
203-824-5563 (cell)
Office: ESC II

Yiren Zhong, Lab Assistant
yiren.zhong@yale.edu
203-710-9820 (cell)

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.

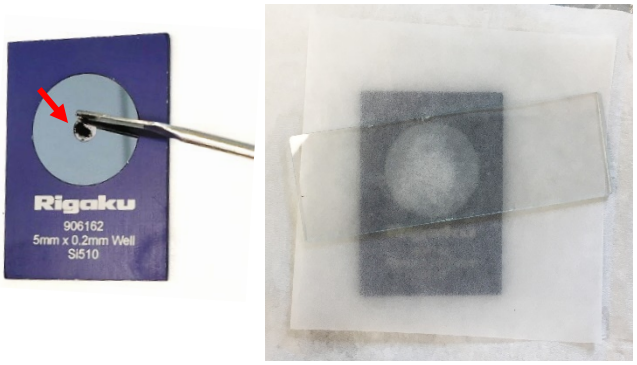
Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. **No** explorations on program allowed unless approved by core manager.

2 Sample Preparation

1) **Wear gloves** and clean the sample holder with provided **IPO**.
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.

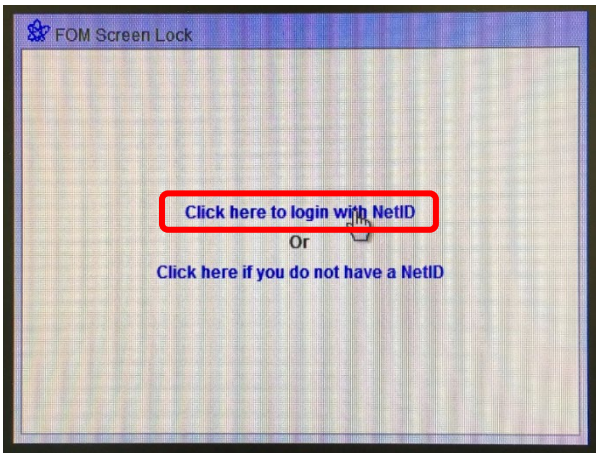
Zero-background | Glass holder

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.

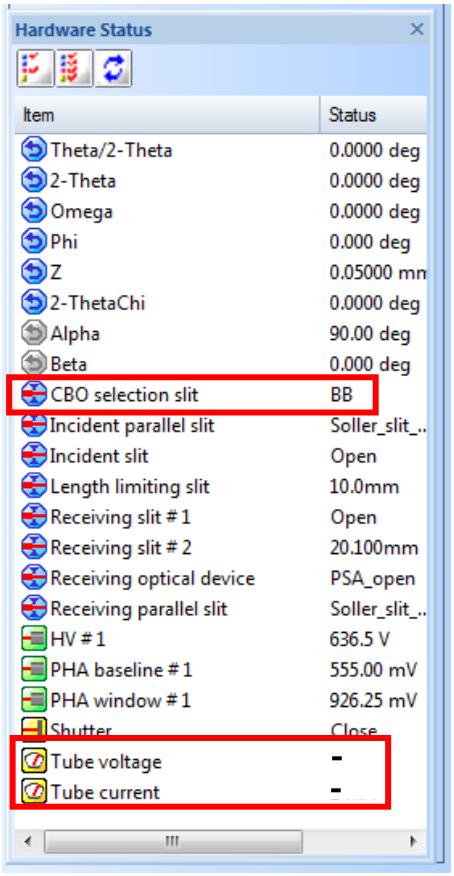


3 XRD Computer Login

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

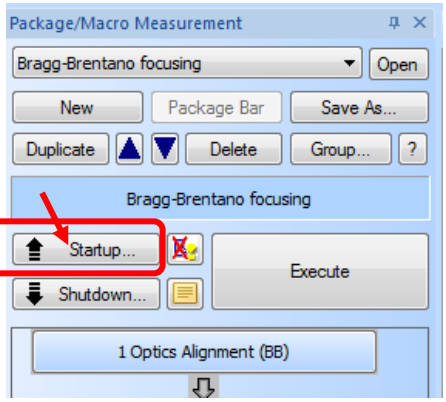


2) Check system status:
   a) Open the **SmartLab Guidance** software if it was closed (login: **administrator**, password: **rigaku**).
   b) Check the **highlighted boxes** on the bottom left of the window as shown below. Make sure that the "**CBO selection slit**" is set at **BB,** Bragg-Brentano focusing mode, and if the X-ray is at shutdown status, no number display for **Tube voltage** and **Tube current**.

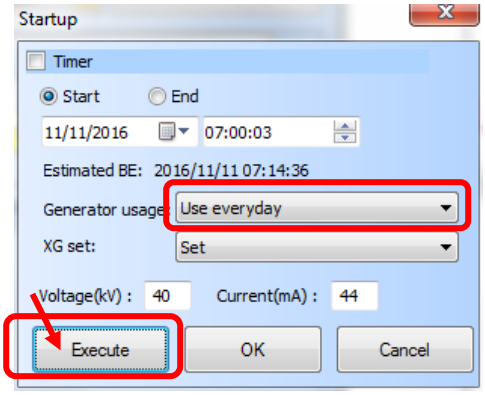


   c) If the **Tube voltage** and **Tube current** read **40 kV** and **44 mA**, skip **Step d)** and **e)** below.
   d) If the system is at shutdown status, click

button in the **Package/Macro Measurement** window below:

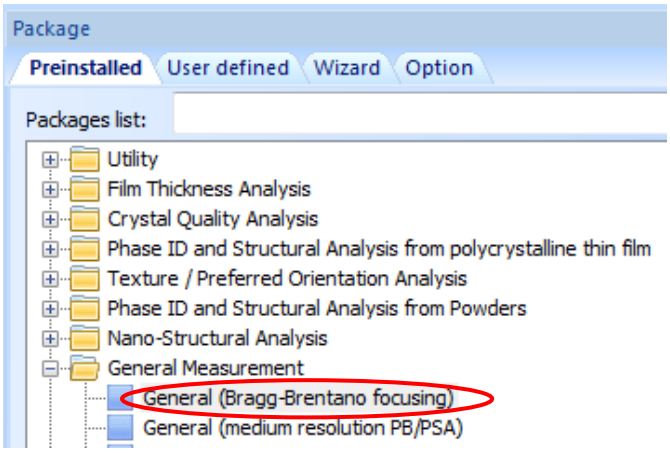


e) In the popup **Startup** window below, choose **Use everyday** if the machine was used within 24 hours and click **Execute** button. It takes 15 minutes for the system to reach the operation power of **40 kV** and **44 mA**.

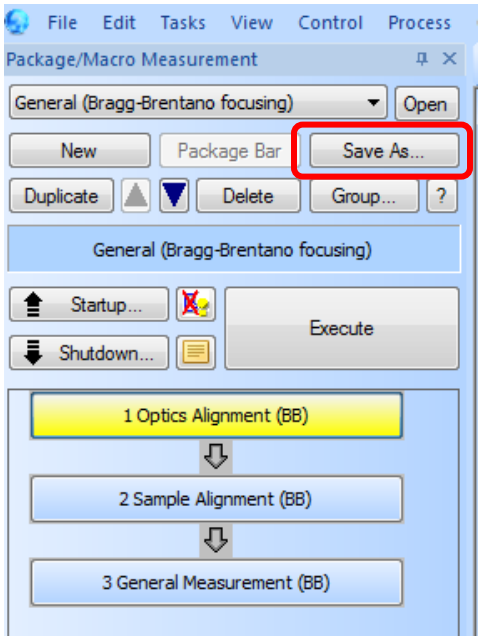


4 User Package Definition

1) On the **Package** window at the top right of software window, find **Preinstalled**> **General Measurement** > **General (Bragg-Brentano Focusing)** package as shown below:



2) Double Click on **General (Bragg-Brentano Focusing)** to open the package in the **Package/Macro Measurement** window below which composes of three parts as shown below:



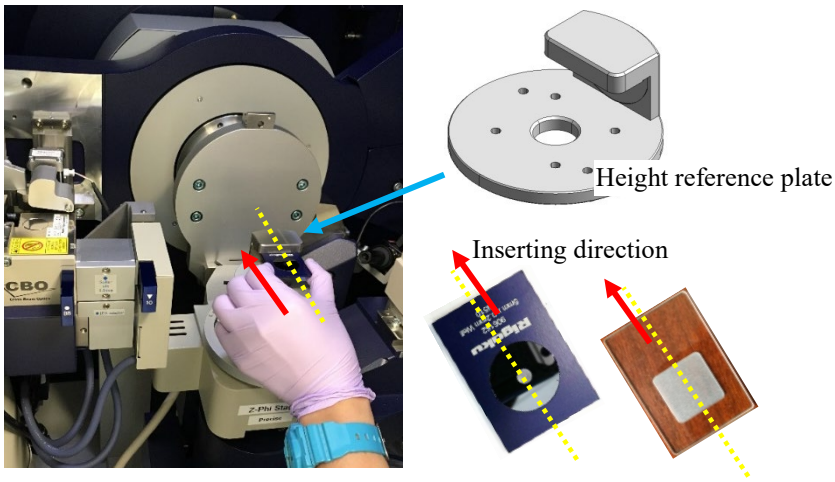
3) Click on **Save As** button on above window to save the package as user defined package which will appear in the **User defined** tab on the top right side of the software window. On the bottom of **User defined** tab click to create a user folder and move saved package file into the folder by clicking .

5 Sample Loading

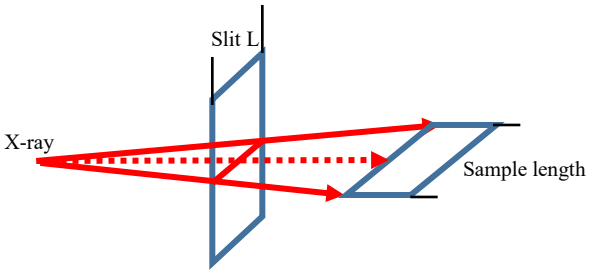
1) Press the **Door Lock** button on the instrument cabinet door, wait till the button **flashes**, and then grab the handle (highlighted below) to slide the door open.

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)



3) 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.

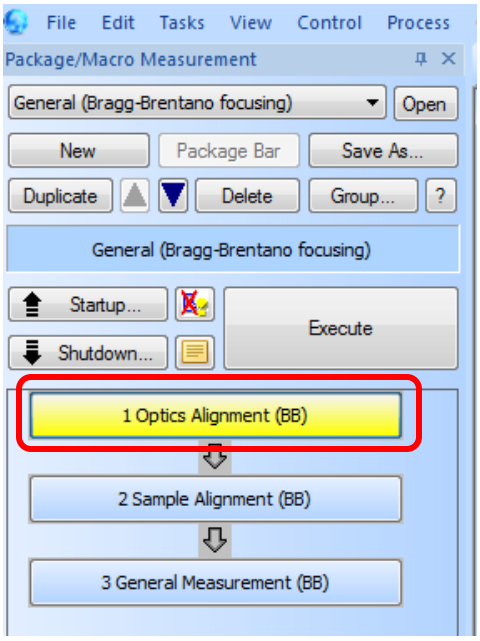


4) Close the cabinet door and hit **Door Lock** button

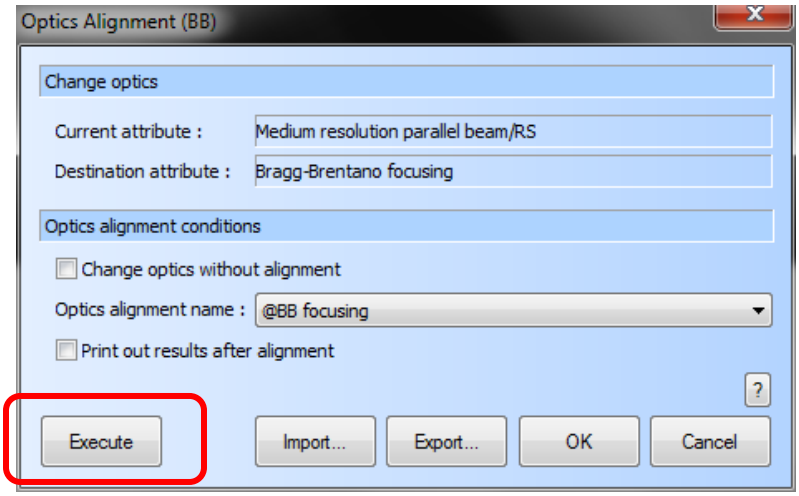
6 Optics Alignment

1) Only required after **parallel beam (PB)** measurement. **Skip** steps below if the **BB** is shown in **Hardware Status** window in **Section 3 > Step 2b).**

2) Insert the **BB** slit into the **CBO** adapter and 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:

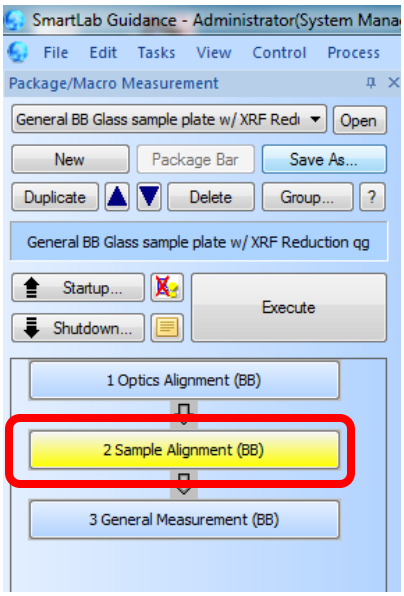


4) Follow the instructions on the popup **SmartMessage** window below to replace required parts and click **OK** to continue. The **Optics Alignment (BB)** window will be active after finish in ~5miniutes.

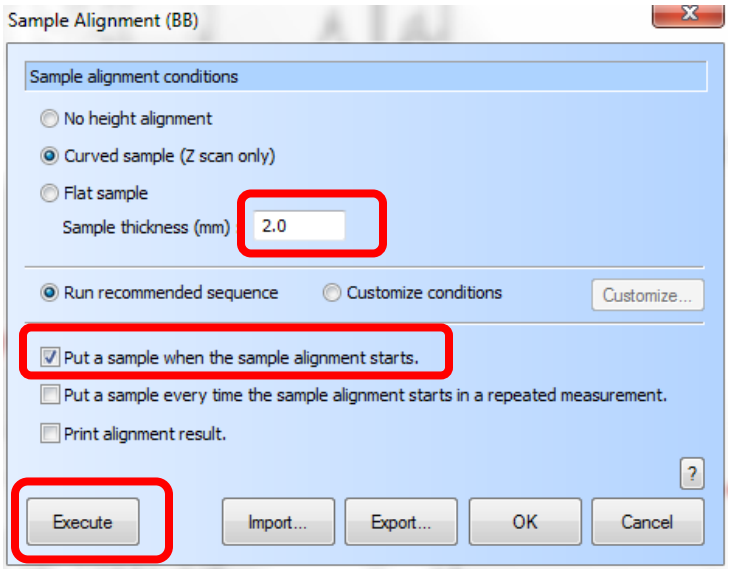
5) Click **OK** on the **Optics Alignment (BB)** windowin Step 2) above after finish.

7 Sample Alignment

1) Open user specific package file in **User defined** tab created in **Section 4 User Package Definition.**
2) Click on 2 Sample Alignment (BB) as highlighted in the **Package/Macro Measurement** window below:



3) In the pop-up **Sample Alignment (BB)** window below:
   a) Choose **Curved sample (Z scan only)** for powder samples.
   b) Input **2.0** as **Sample thickness (mm)** if all sample surfaces are flush with holder surface;

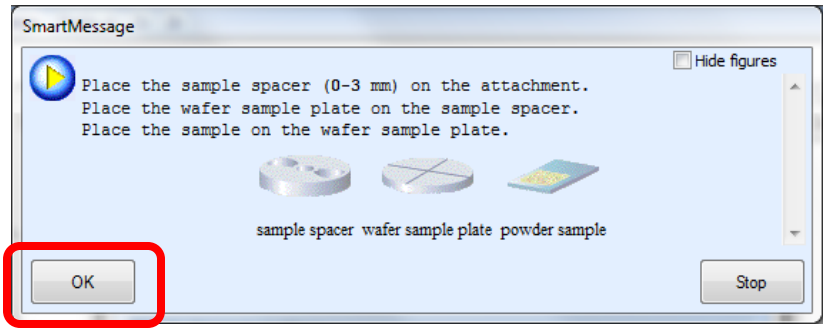


c) Input **2.0 + solid sample height above holder** as **Sample thickness (mm)** if the sample is higher than the holder.

d) For multiple samples with similar thickness or powders flush with the holder, **no need** to repeat sample alignment.

e) Check the box next to **Put a sample when the sample alignment starts** in above **Sample Alignment** window and click **Execute** button

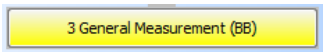
f) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment (BB)** window will be back active in ~ 2 minutes after finish.



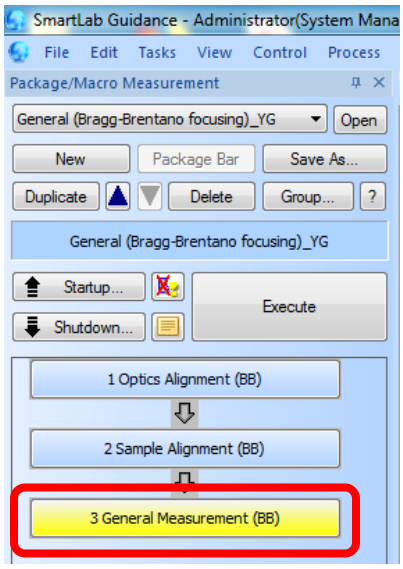
4) Click **OK** on the **Sample Alignment (BB)** window in **Step 3)** above to quit the window.

5) **No need** to repeat sample alignment if the next sample surface is flush with the holder or at the same height as the previous one.

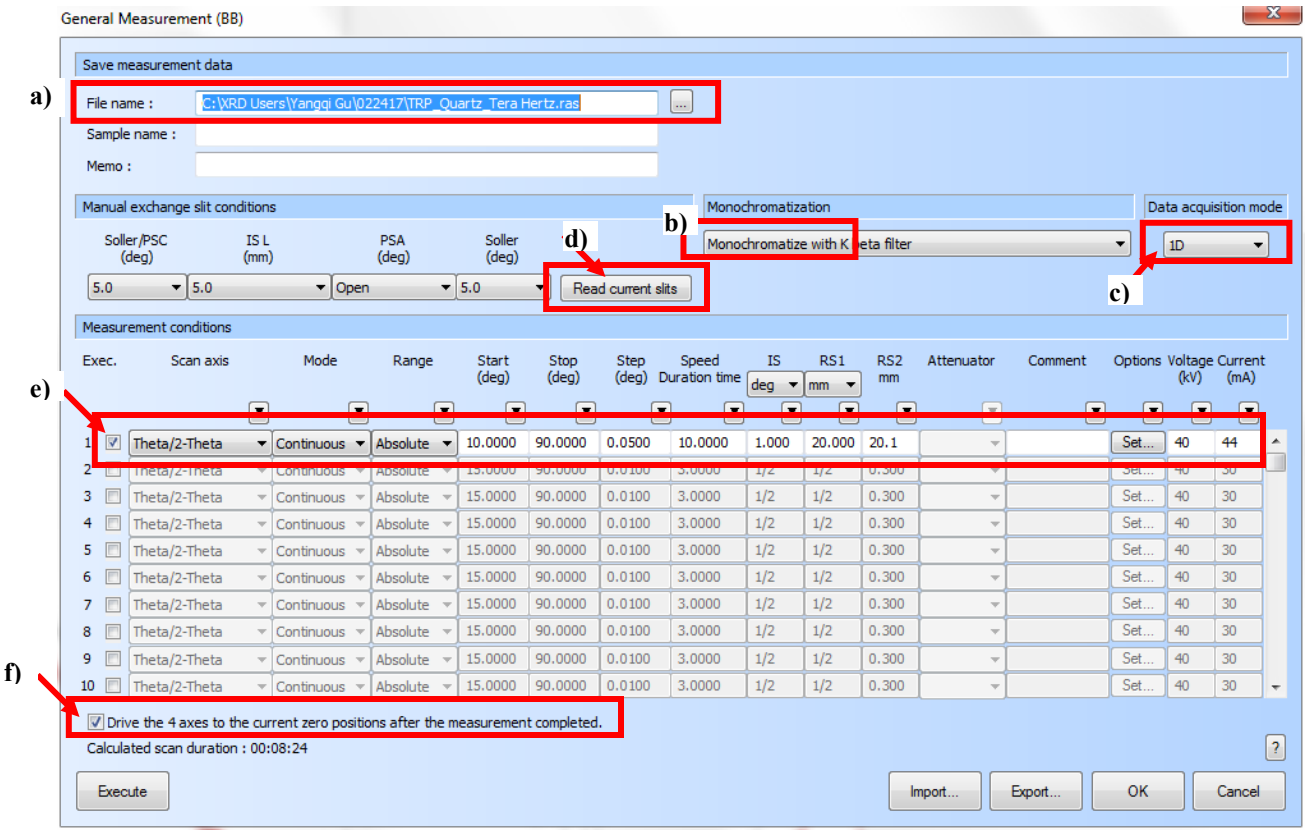
8 Sample Measurement

1) Click on 

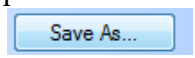
button as highlighted in the **Package/Macro Measurement** window below:

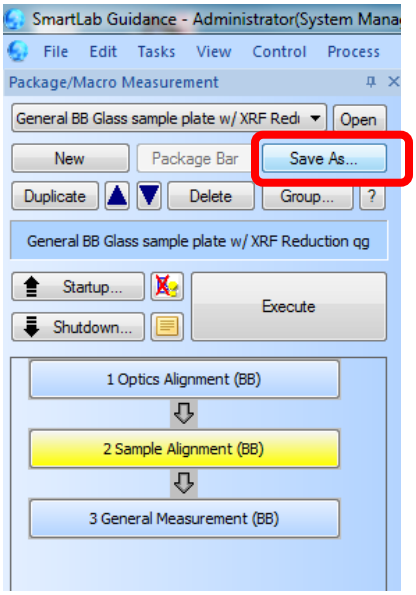


2) Perform following steps on the popup **General Measurement (BB)** window below:
   a) Specify **File name** and folder as highlighted in the window below.
   b) Check **K beta filter method**
   c) Select **1D mode**
   d) Click **Read current slits** button
   e) Set measurement conditions:
      > **Exec:** click/check small box to activate line **1**.
      > **Mode: Continuous**
      > **Range: Absolute**
      > **Start (deg): 10.0000.** Never change to below **5**. The detector will be damaged.
      > **Stop (deg): 90.0000,** the upper limit.
      > **Step (deg): 0.0500**
      > **Speed Duration time (degree/min): 10.0000,** choose lower speed to smooth spectra.
      > **IS mm**: **1,** recommended size.
      > **RS1 mm: 20.000,** recommended size.
      > **RS2 mm: 20.1,** recommended size.
      > Voltage (kV): **40**, maximal voltage. **Never** change to above 40 to damage the X-ray tube.
      > **Current (mA): 44**, maximal current. **Never** change to above 44 to damage the X-ray tube.
   f) Select box near **Drive the 4 axes to the current zero positions after measurement completed**.
   g) Click **Execute** button to start measurement
   h) Follow the instruction on SmartMessage windows if appear and hit **OK**

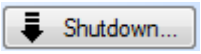


3) XRD spectra will be automatically saved to specified user folder after scan

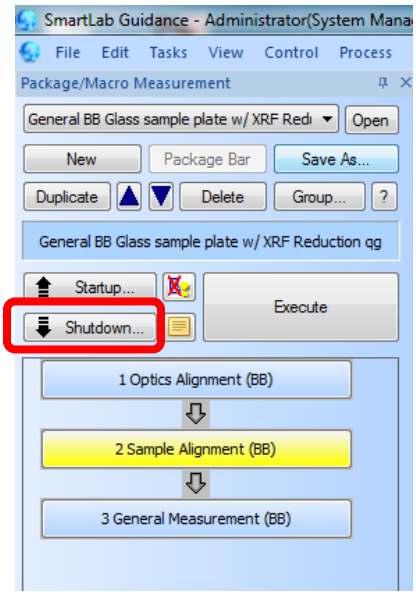
4) Click  button highlighted in the window below to save modified measurement parameters into user defined package.



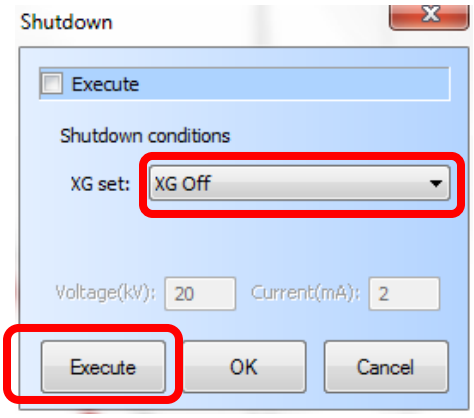
9 Checklist after Experiment

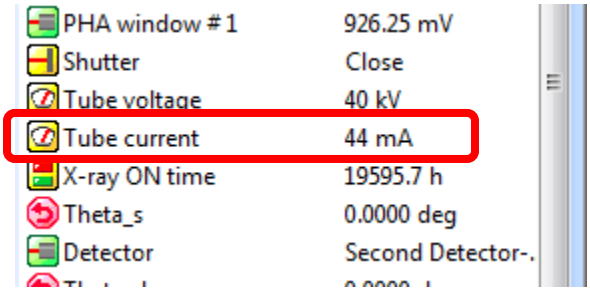
1) Back up your data to Yale Box cloud drive (box.yale.edu) or using Core USB drive. **Do not** use personal USB drive.
2) **Remove** sample from the stage. **Never** leave samples inside XRD.
3) The user who finishes near **6pm** at regular time or **anytime** in off-peak hours should shutdown the instrument by clicking 

in the window below:



4) Make sure **XG Off** is chosen and click **Execute** button in the popup **Shutdown** window below and watch the **Tube current** start to drop from **44 mA** before logoff FOM.





5) **Never minimize** or **close SmartLab Guidance** 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.

7) **Sign off** on the logbook.
8) **Clean** the sample holders and glass slides with clean wipes **IPA**.
9) **Store** the sample holder and other tools back into the tool box.