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 2021

> **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

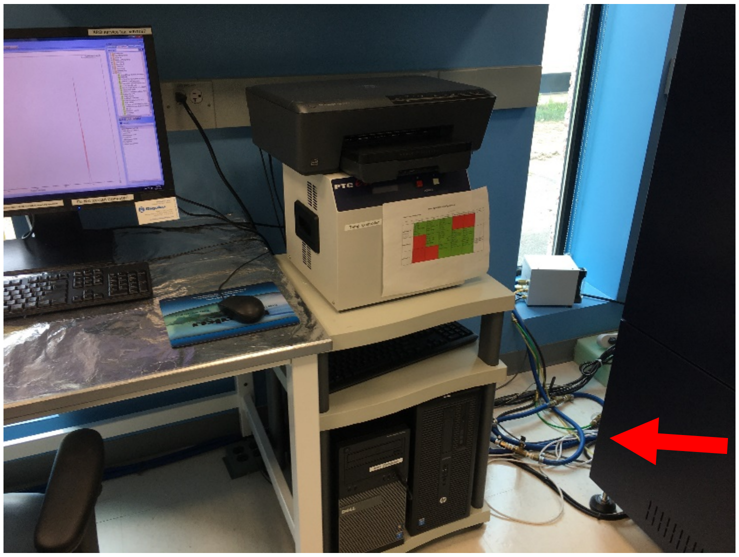
The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please **contact** the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should,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

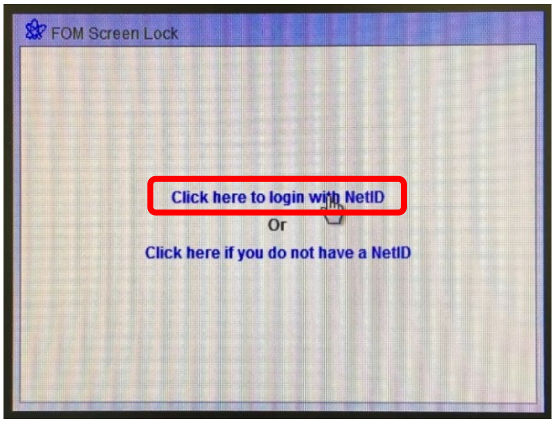
3) Meanwhile, please check the floor at the back corner of the machine and make sure no water leaking on the floor. If no water leaking and the manager can't be reached, before leaving, put a machine down note on the machine sliding door.



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

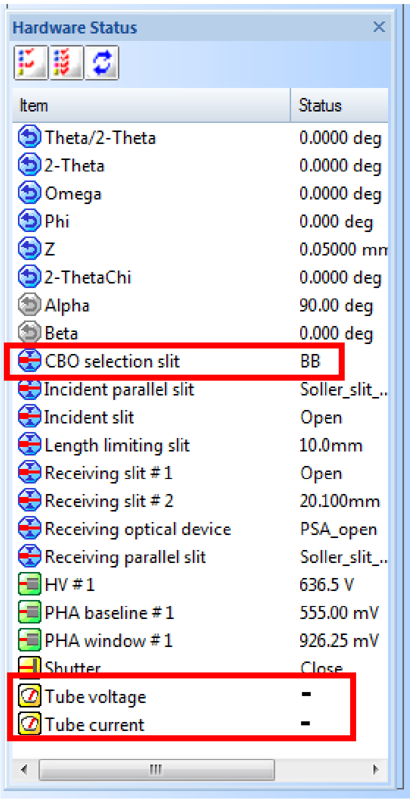
1) Sign in on the logbook.
2) Login FOM system: click on **Click here to login with NetID** to unlock the screen lock.



3) Check system status:
   a) Open the **SmartLab Guildance** software if it was closed (login: **administrator**, password: **rigaku**).
   b) Check the **highlighted areas** on the bottom left of the Hardware Status window as shown below:

      > Make sure that the "**CBO selection slit**" is on **BB (**Bragg-Brentano focusing) mode,

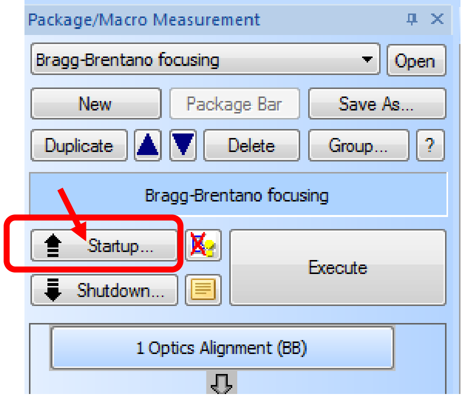
      > No number display on **Tube voltage** and **Tube current** if the X-ray is at shutdown status.



Item	Status
Theta/2-Theta	0.0000 deg
2-Theta	0.0000 deg
Omega	0.0000 deg
Phi	0.000 deg
Z	0.05000 m
2-ThetaChi	0.0000 deg
Alpha	90.00 deg
Beta	0.000 deg
CBO selection slit	BB
Incident parallel slit	Soller_slit_..
Incident slit	Open
Length limiting slit	10.0mm
Receiving slit #1	Open
Receiving slit #2	20.100mm
Receiving optical device	PSA_open
Receiving parallel slit	Soller_slit_..
HV #1	636.5 V
PHA baseline #1	555.00 mV
PHA window #1	926.25 mV
Shutter	Close
Tube voltage	-
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 Startup... button in the **Package/Macro Measurement** window below:

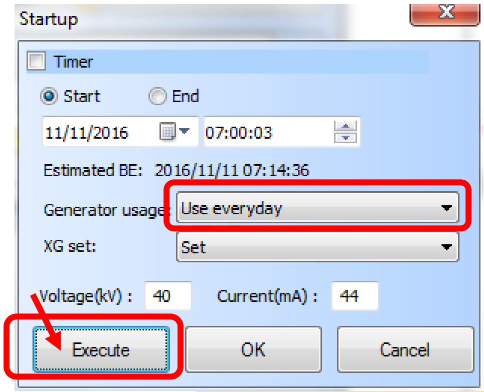


Package/Macro Measurement

Bragg-Brentano focusing

e) In the popup **Startup** window below

> Check the logbook record, if the machine was used within 24 hours choose **Use everyday** and click **Execute** button. It takes **15 minutes** for the system to reach the operation power of **40 kV** and **44 mA**.



Startup

☐ Timer

● Start ○ End

11/11/2016 07:00:03

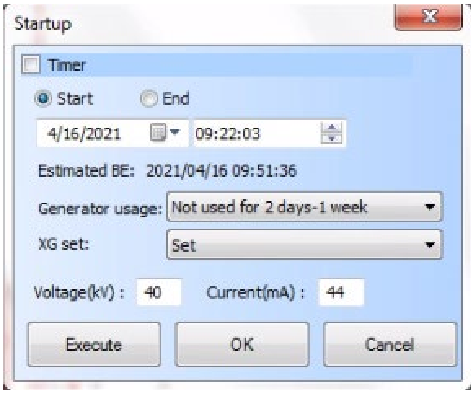
Estimated BE: 2016/11/11 07:14:36

Generator usage: Use everyday

XG set: Set

Voltage(kV) : 40 Current(mA) : 44

> If the machine was NOT used for more than 24 hours choose **Not used for 2days-1week** and click **Execute** button. It takes 30 minutes for the system to reach the operation power of **40 kV** and **44 mA**.



Startup

☐ Timer

● Start ○ End

4/16/2021 09:22:03

Estimated BE: 2021/04/16 09:51:36

Generator usage: Not used for 2 days-1 week

XG set: Set

Voltage(kV) : 40 Current(mA) : 44

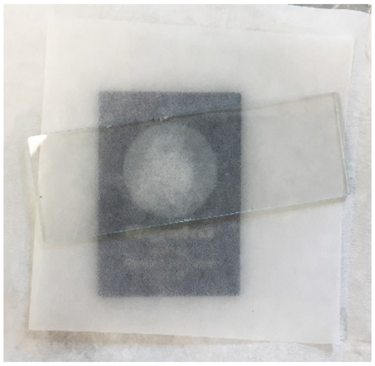
4 Sample Preparation

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

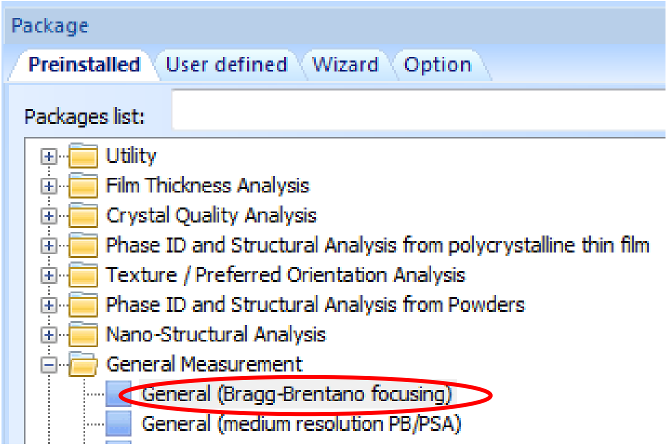
Glass holder

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.

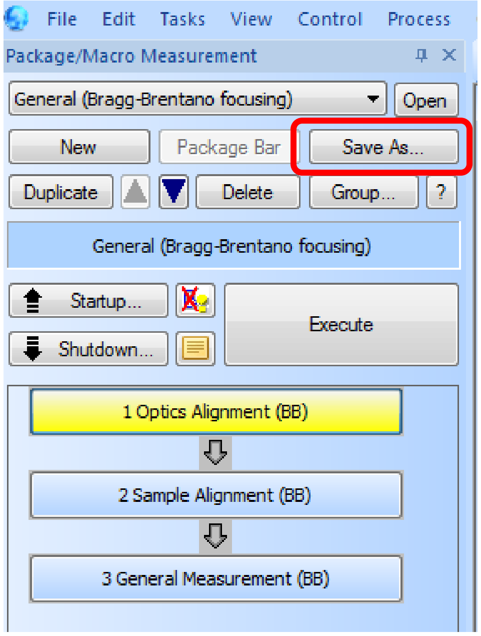


5 User Package Definition for the first time

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:

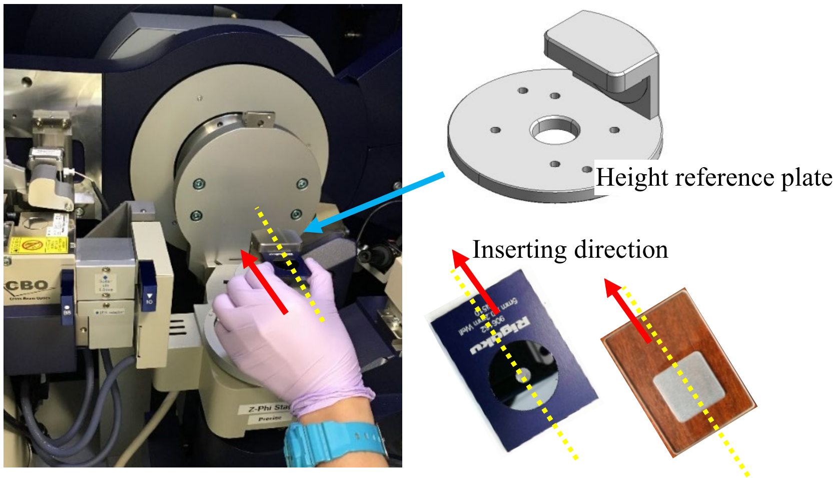


3) Click on **Save As** button on above window to save the package as user defined package which will appear in the **User defined** tab on the top right side of the software window. On the bottom of **User defined** tab click ⊞ to create a user folder and move saved package file into the folder by clicking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

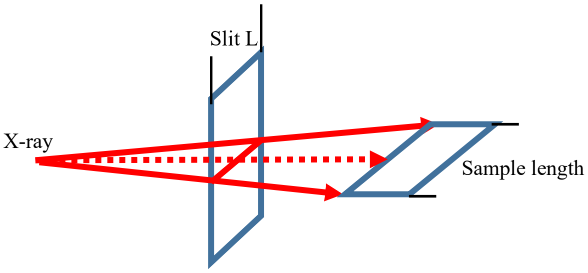
6 Sample Loading

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.

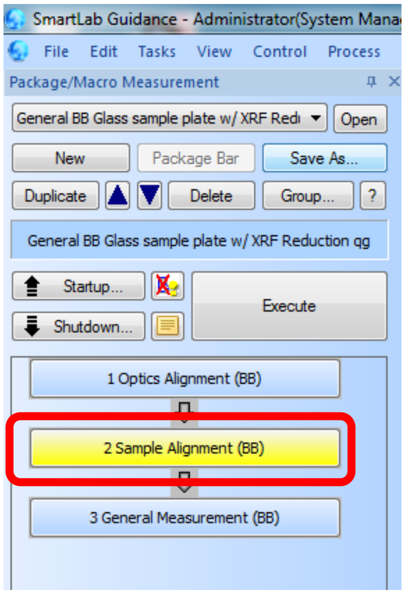


5) Close the cabinet door and press the **Door Lock** button.

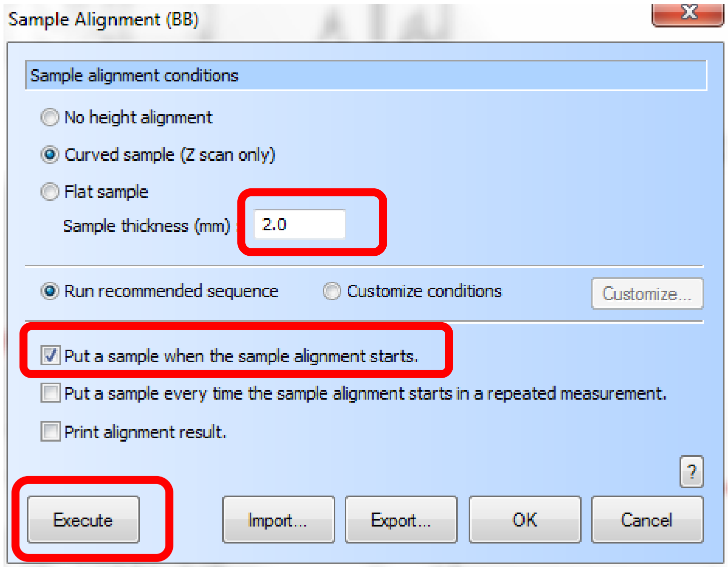
7 Sample Alignment

1) 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.



Sample Alignment (BB)

Sample alignment conditions

○ No height alignment
● Curved sample (Z scan only)
○ Flat sample
Sample thickness (mm) 2.0

● Run recommended sequence ○ Customize conditions

☑ Put a sample when the sample alignment starts.
☐ Put a sample every time the sample alignment starts in a repeated measurement.
☐ Print alignment result.

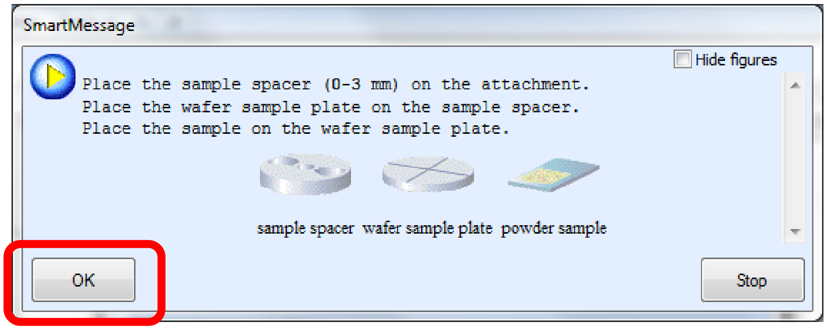
b) For powder samples, input 2.0 as **Sample thickness (mm)** if the sample surface is flush with holder surface;

c) For solid samples, 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 if powders are 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.



SmartMessage

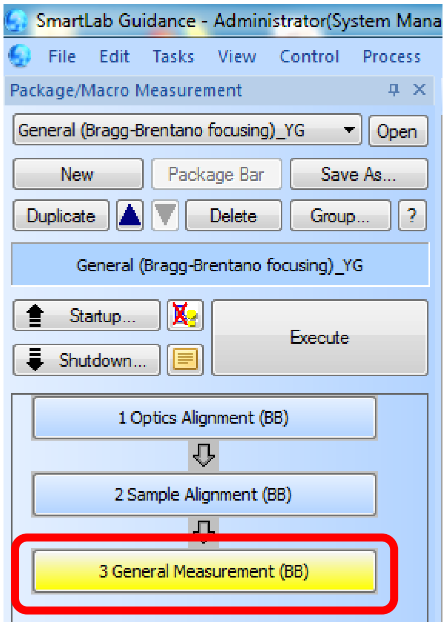
☐ Hide figures

```
Place the sample spacer (0-3 mm) on the attachment.
Place the wafer sample plate on the sample spacer.
Place the sample on the wafer sample plate.
```

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

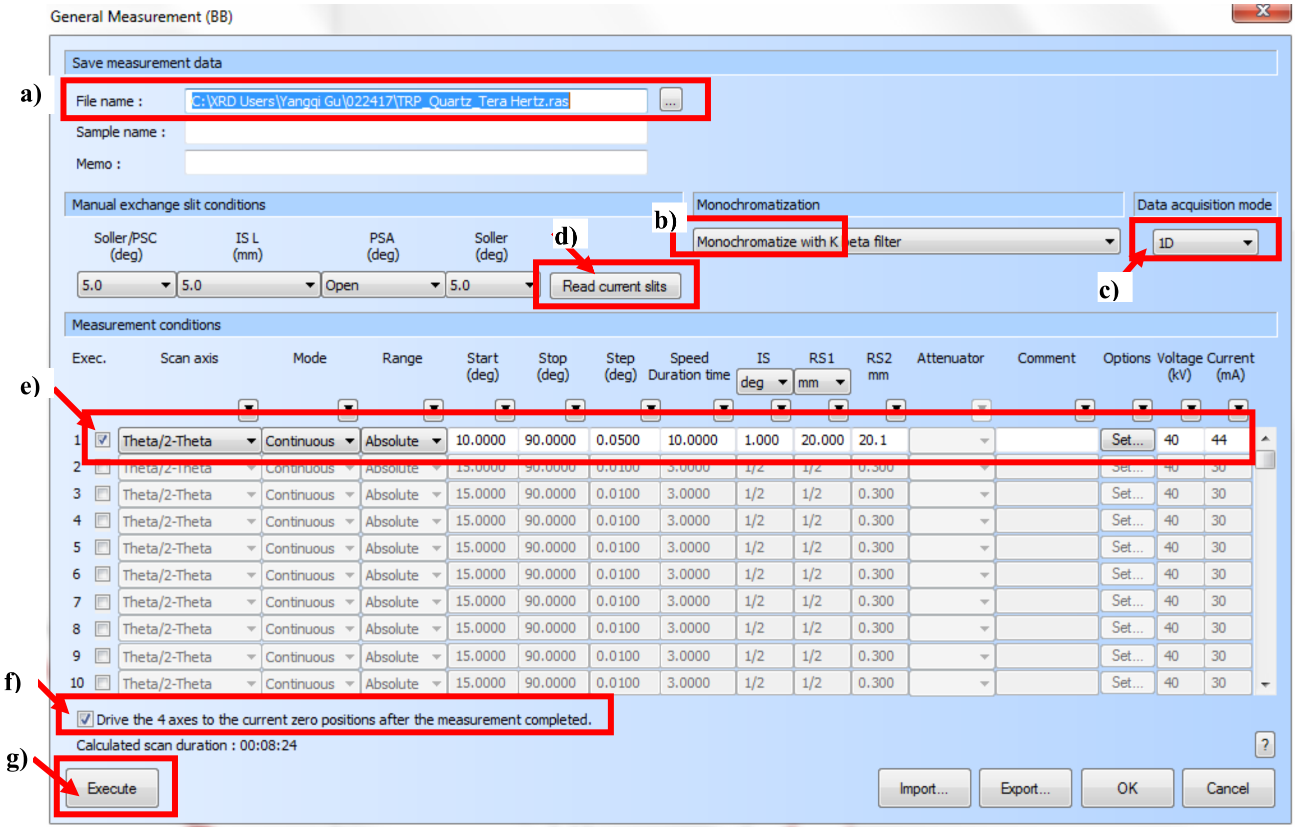
8 Sample Measurement

1) Click on 3 General Measurement (BB) 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**



General Measurement (BB)

Save measurement data

File name : C:\XRD Users\Yangqi Gu\022417\TRP_Quartz_Tera Hertz.ras

Sample name :

Memo :

Manual exchange slit conditions

Soller/PSC (deg)	IS L (mm)	PSA (deg)	Soller (deg)
5.0	5.0	Open	5.0

Monochromatization: Monochromatize with K eta filter

Data acquisition mode: 1D

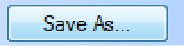
Measurement conditions

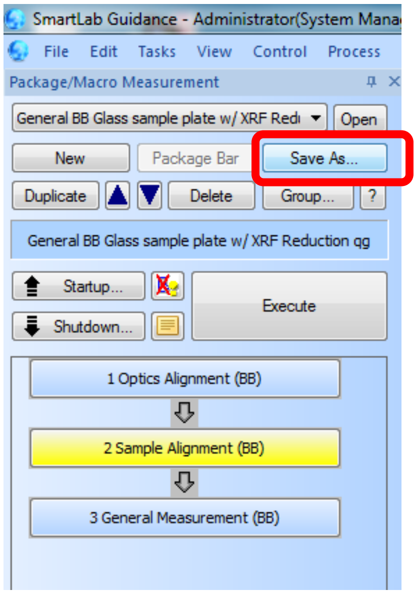
	Exec.	Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed Duration time	IS deg	RS1 mm	RS2 mm	Attenuator	Comment	Options	Voltage (kV)	Current (mA)
1	☑	Theta/2-Theta	Continuous	Absolute	10.0000	90.0000	0.0500	10.0000	1.000	20.000	20.1			Set...	40	44
2	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
3	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
4	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
5	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
6	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
7	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
8	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
9	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30
10	☐	Theta/2-Theta	Continuous	Absolute	15.0000	90.0000	0.0100	3.0000	1/2	1/2	0.300			Set...	40	30

☑ Drive the 4 axes to the current zero positions after the measurement completed.

Calculated scan duration : 00:08:24

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.

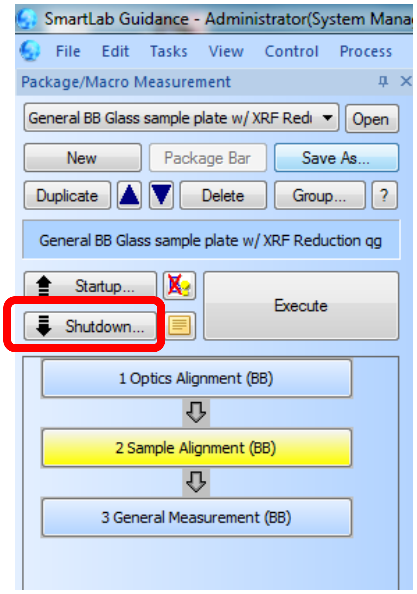


General BB Glass sample plate w/ XRF Redu

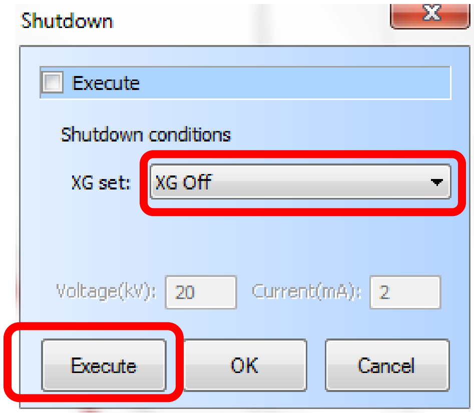
General BB Glass sample plate w/ XRF Reduction qg

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. **DO NOT** leave samples inside XRD.
3) Shutdown instrument if there is no reservation within one hour. Click Shutdown... in the window below:

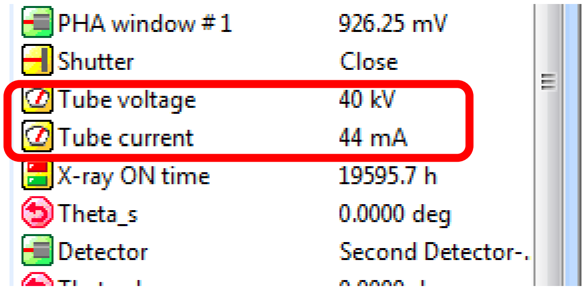


4) Make sure **XG Off** is chosen and click **Execute** button in the popup **Shutdown** window below and watch the **Tube voltage and current** drop down to zero and the **yellow light** above the machine is turned off.



XG set: XG Off

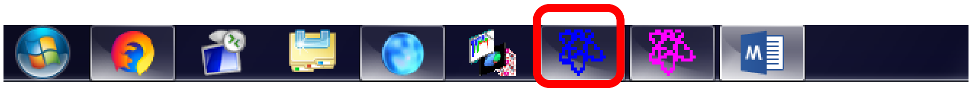
Voltage(kV): 20 Current(mA): 2



PHA window # 1	926.25 mV
Shutter	Close
Tube voltage	40 kV
Tube current	44 mA
X-ray ON time	19595.7 h
Theta_s	0.0000 deg
Detector	Second Detector-.

5) **DO NOT 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. The message will be sent to the lab manager.

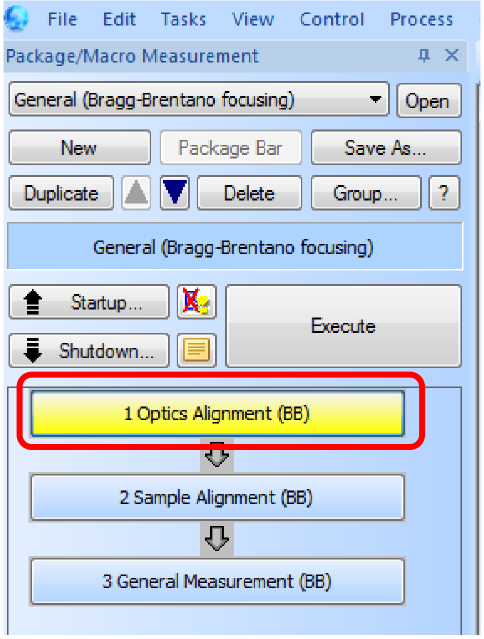


7) **Sign off** on the logbook.
8) **Clean** the bench sample preparation area, the sample holders and glass slides with clean wipes and isopropanol.
9) **Put** the sample holder and other tools back into the tool box.

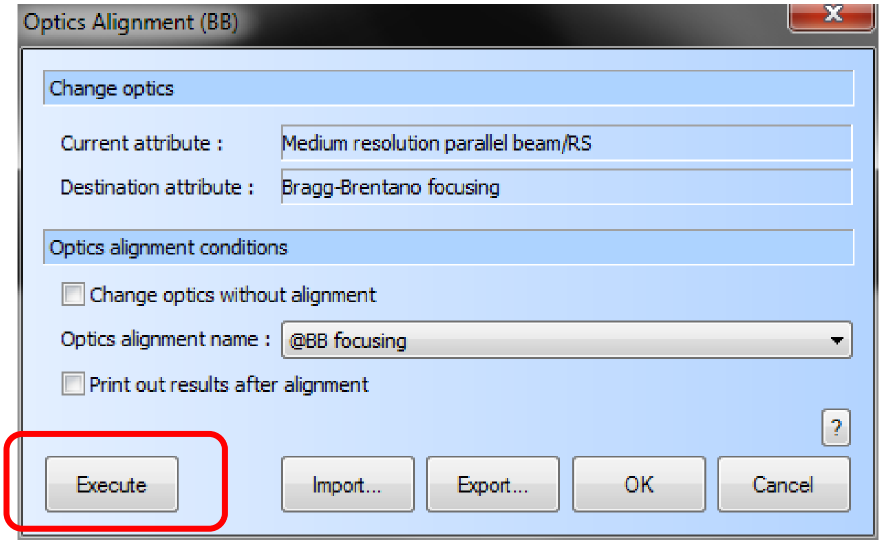
Appendix: Optics Alignment

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:



Current attribute : Medium resolution parallel beam/RS

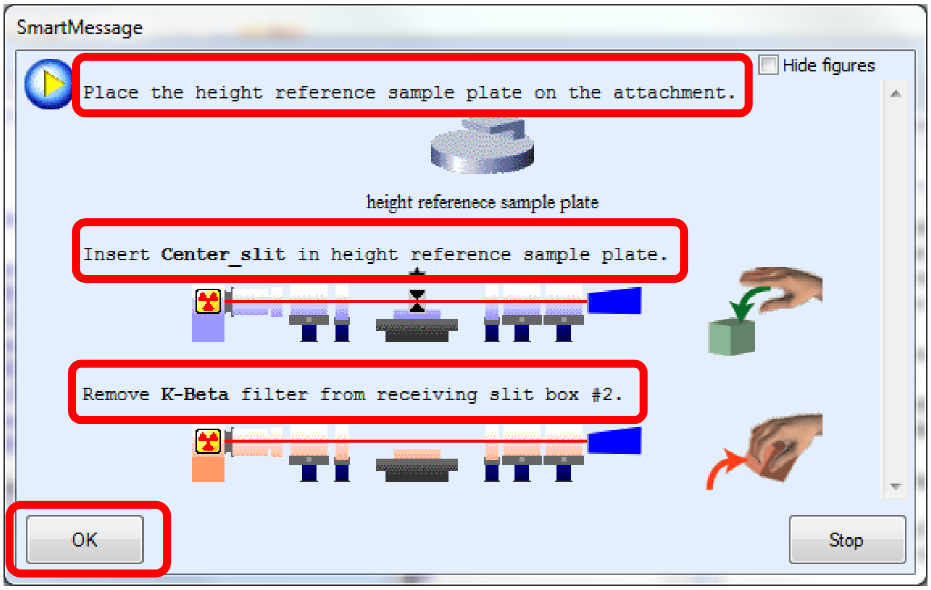
Destination attribute : Bragg-Brentano focusing

☐ Change optics without alignment

Optics alignment name : @BB focusing

☐ Print out results after 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.



SmartMessage

Place the height reference sample plate on the attachment.

Insert **Center_slit** in height reference sample plate.

Remove **K-Beta** filter from receiving slit box #2.

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.