Standard Operating Procedure

Rigaku SmartLab XRD

II. Grazing Incidence XRD (GIXRD)

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

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

Version 1.1, August 2020

> **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

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

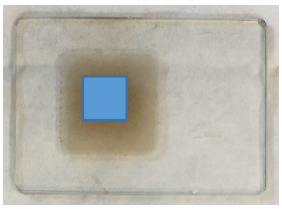
1) Please check to make sure the instrument sliding door is closed and there is no beeping sound once getting into the XRD room.
2) If there is a beeping sound from the instrument and meanwhile the OPERATE light is flashing as show below. Hit the white OFF button  on the front panel above and contact 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 not 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.

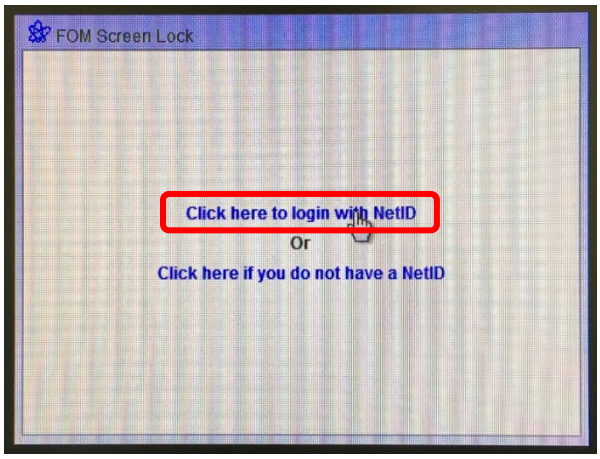
3 Sample Preparation

1) **Wear gloves** and clean the sample holder with provided **IPO**.
2) The film sample can be put on the back of the glass powder sample holder. Make sure the sample is placed at the center of the square as shown below.



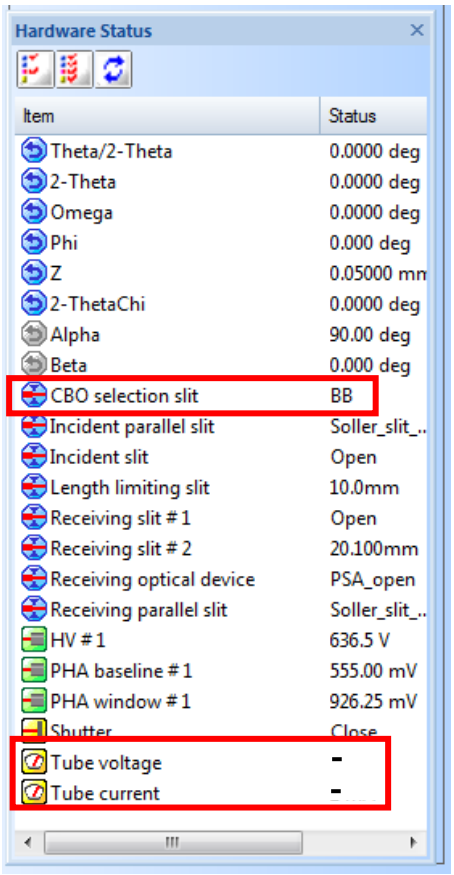
4 XRD Computer Login

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 Guildance** software if it was closed (login: **administrator**, password: **rigaku**).

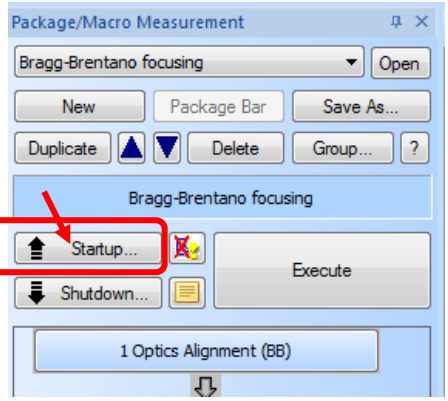
b) Check the **highlighted boxes** on the bottom left of the window as shown below. Make sure that the "**CBO selection slit**" is set at **BB,** Bragg-Brentano focusing mode, and if the X-ray is at shutdown status, no number display for **Tube voltage** and **Tube current**.



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 

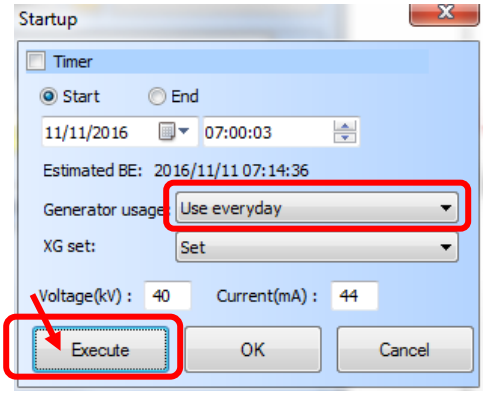
button in the **Package/Macro Measurement** window below:



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

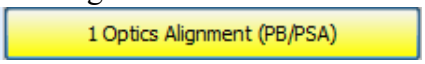
> If the machine was used within one day, choose **Use everyday** and click **Execute** button. It takes 15 minutes for the system to reach the operation power of **40 kV** and **44 mA**.

> If the instrument has not been used for more than one days, choose **Not used for 2days-1 week**. It takes 30 minutes to warm up the X-ray tube.

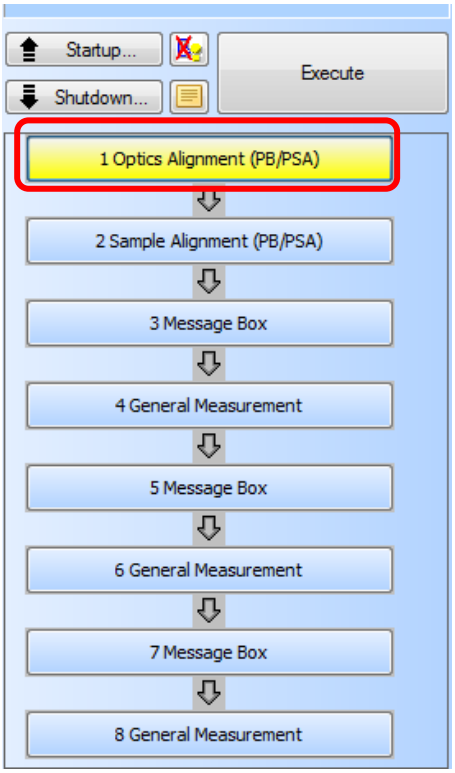


5 Optics Alignment

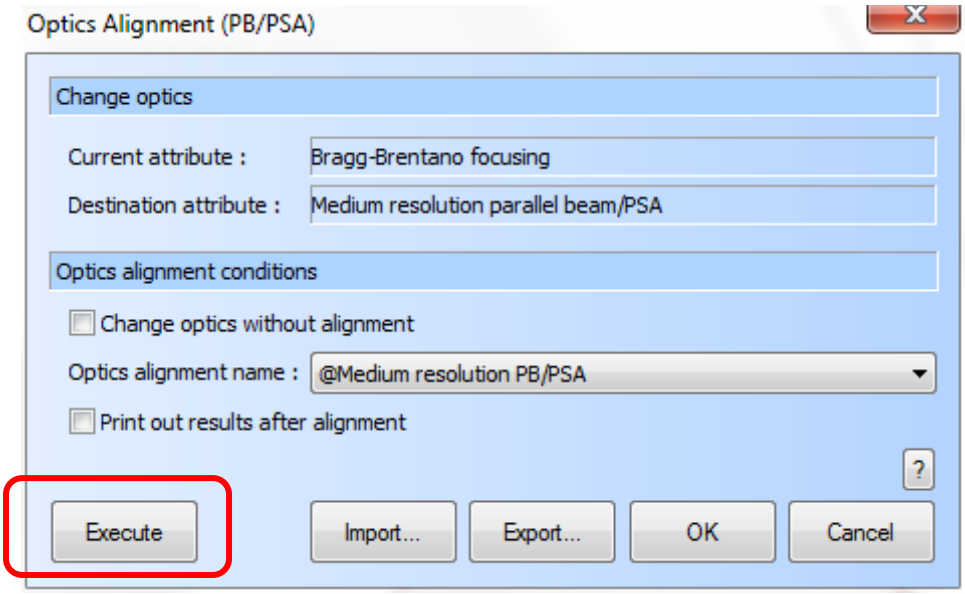
1) Open the GIXRD package file in User defined tab on the right side of the software window.

2) Insert the **PB** slit into the **CBO** adapter and click on 

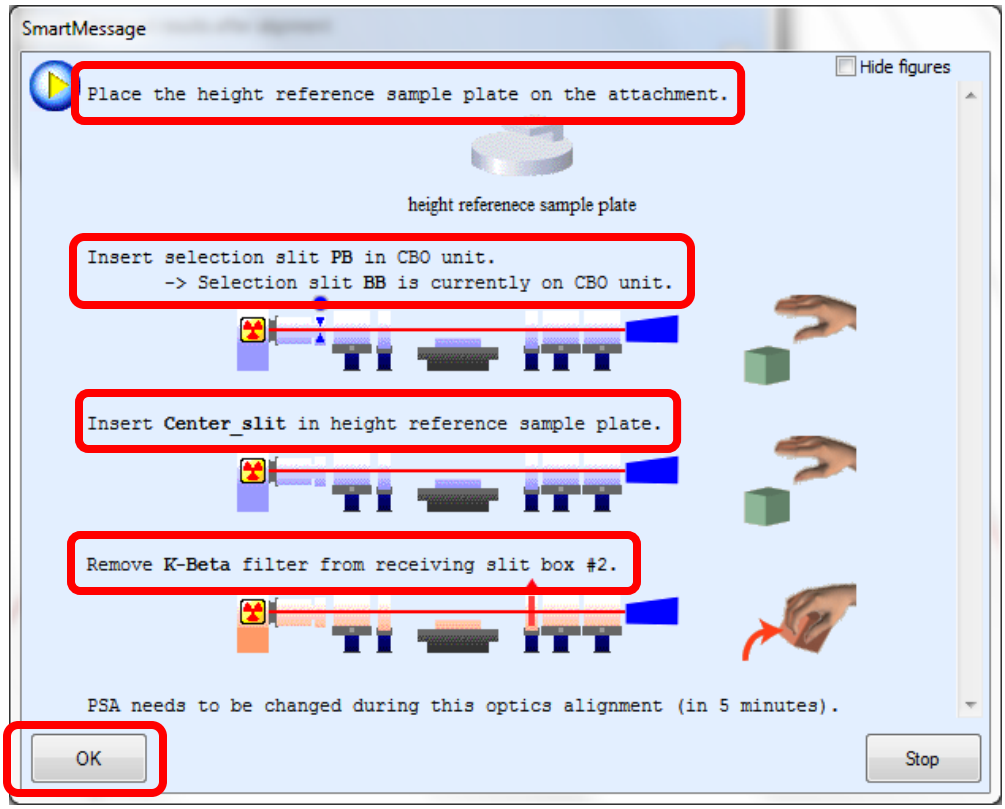
in the **Package/Macro Measurement** window below:

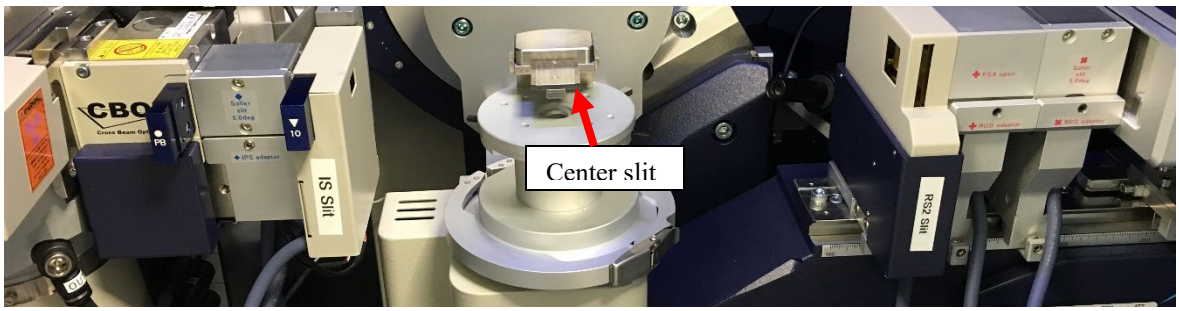


3) Click **Execute** button on the popup **Optics Alignment (PB/PSA)** window below to start optics alignment:

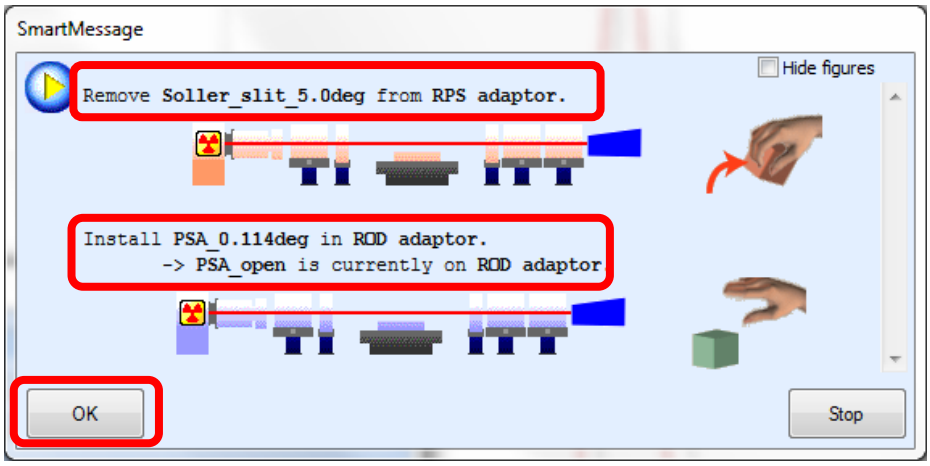


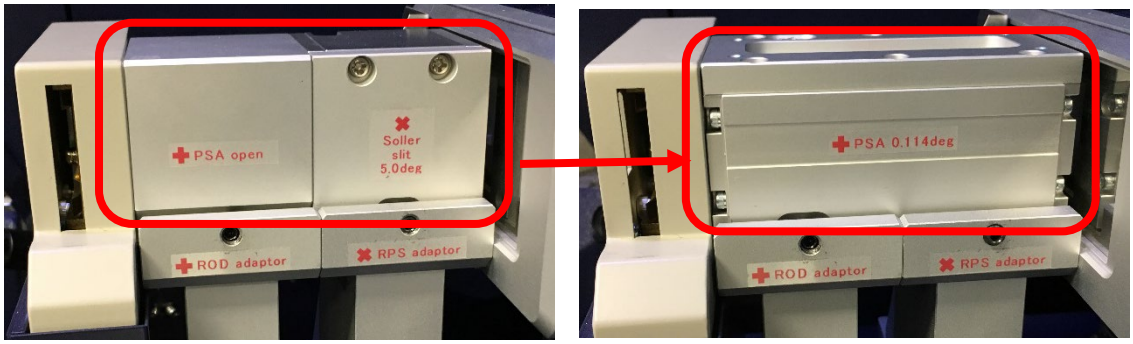
4) Follow the instructions on the popup **SmartMessage** window below to replace required parts and click **OK** to continue. The **Optics Alignment (PB/PSA)** window will be active after finish in ~**10 minutes**.
Note: Keep an eye on alignment process, if the calibration curves become stuck for more than 5 minutes, please contact manager. It could indicate the software communication issue.





5) If the following window appears, follow the instruction on the window to proceed and click **OK** after finish. **Make sure** the units are tightened. **Remember** also remove the PSA open in order to place PSA_0.114 unit.

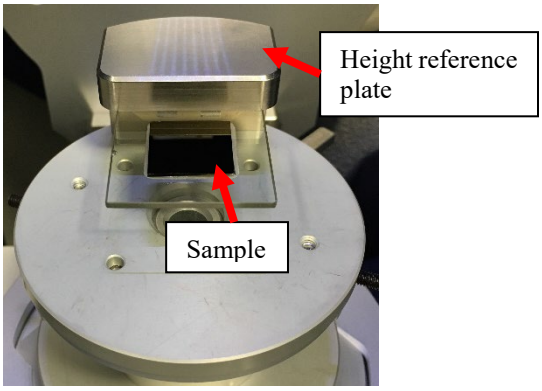


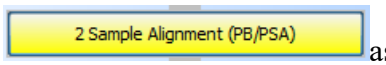


6) Click **OK** on the **Optics Alignment (PB/PSA)** window in **Step 2)** above after finish.

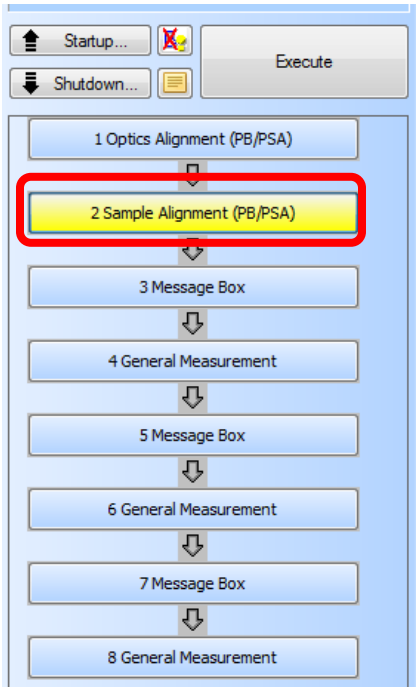
6 Sample Alignment

1) Remove the Center slit from the **height reference sample plate.**
2) Flip the glass powder holder over and push the holder all the way into the **height reference sample plate** and place the sample on top of the square as shown below.

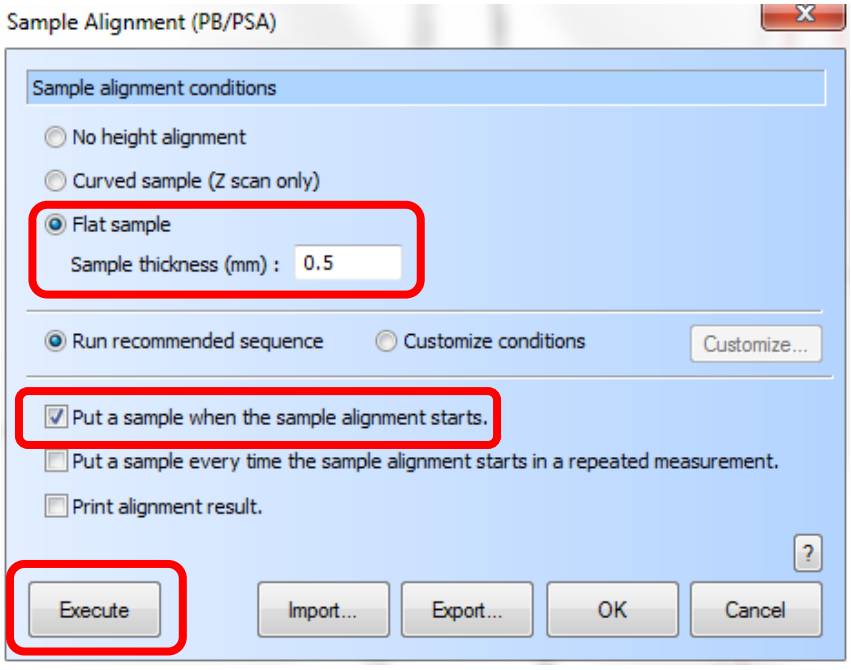


3) Click on 

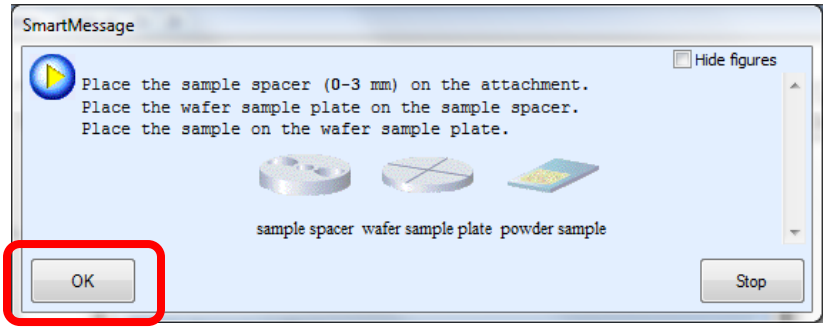
as highlighted in the **Package/Macro Measurement** window below. For samples with similar heights, only the first sample alignment is required.



4) In the pop-up **Sample Alignment (BB)** window below, choose **Flat sample** and Input the **Sample thickness (mm)** above the glass holder; choose **Put a sample when the sample alignment starts** and click **Execute** button.



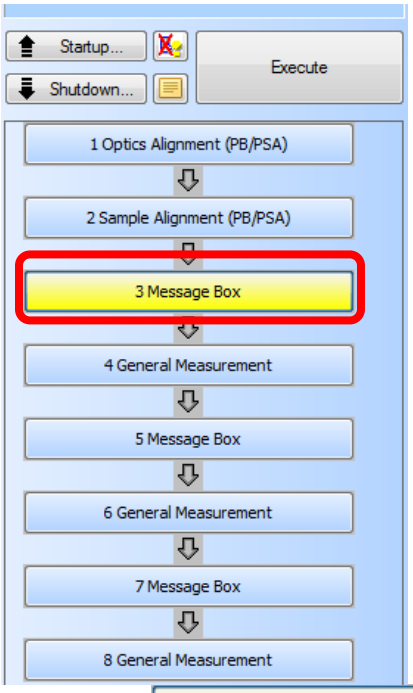
5) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment (PB)** window will be back active in ~ 2 minutes after finish.

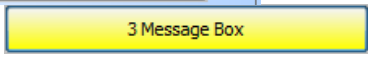


6) Click **OK** on the **Sample Alignment (PB/PSA)** window in **Step 4)** above to quit the window.

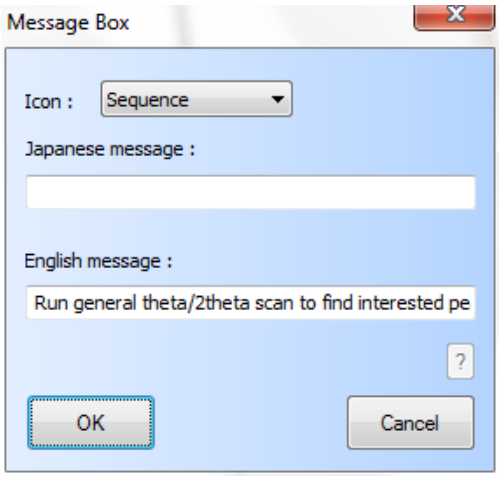
7 Sample Measurement

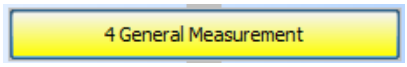
1) **Skip Steps 3-5** inside the window below if the interested film peak position is already known from previous measurements. Otherwise proceed from the steps below.

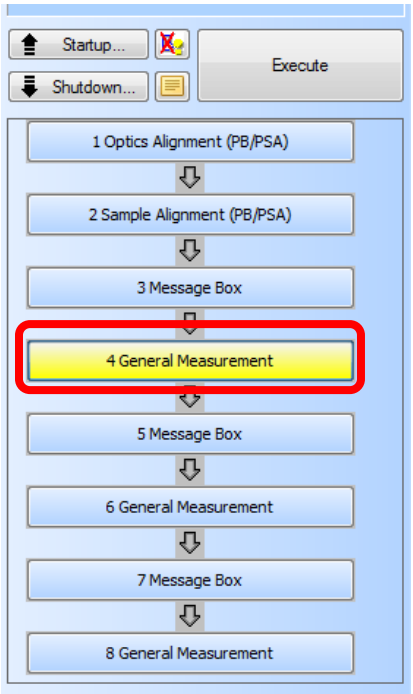


2) Click on 

button as highlighted in the **Package/Macro Measurement** window below. Read the message and click OK to close.



3) Click on  button as highlighted in the **Package/Macro Measurement** window below. This measurement is to have a full range scan of the thin film to find peak of interest.

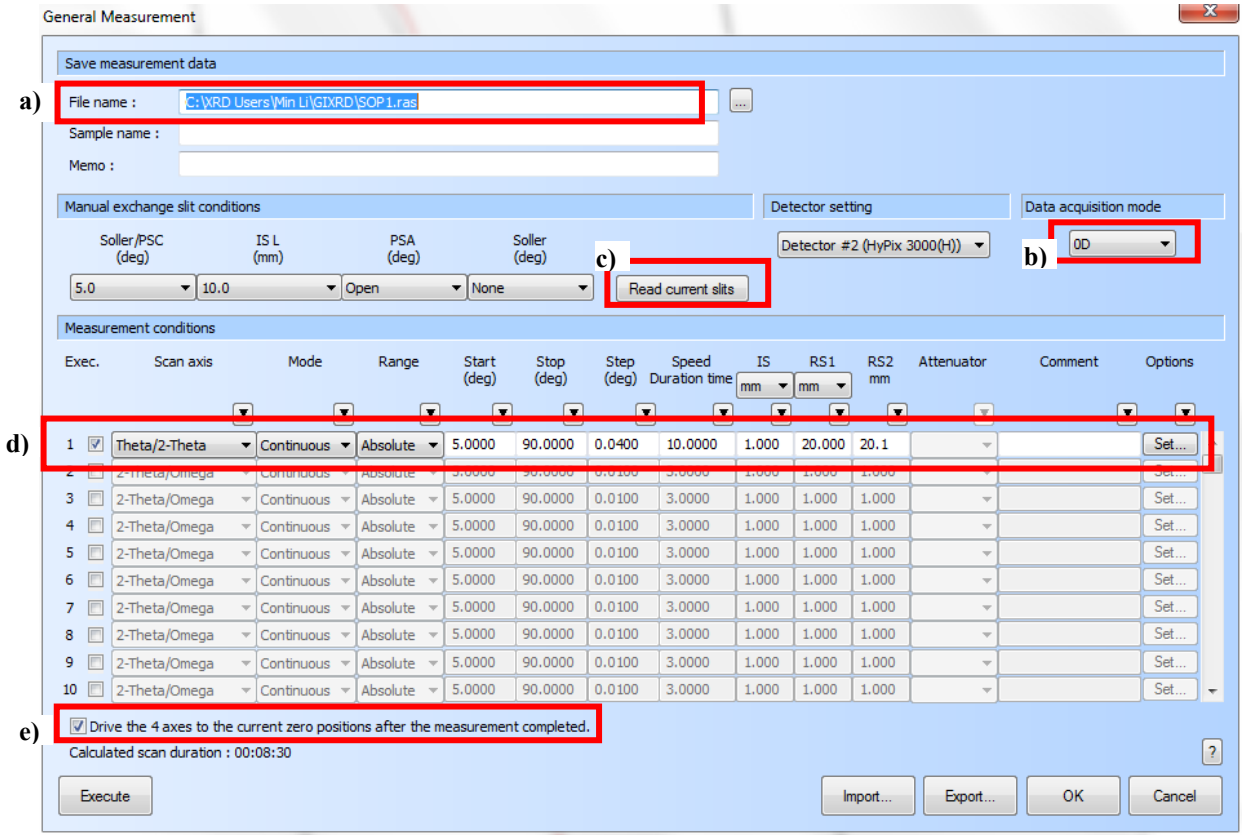


4) Perform following steps on the popup **General Measurement** window below:
   a) Specify **File name** and folder as highlighted in the window below.
   b) Check **K beta filter method**
   c) Select **1D mode**
   d) Click **Read current slits** button
   e) Set measurement conditions:
      > **Exec:** click/check small box to activate line **1**.

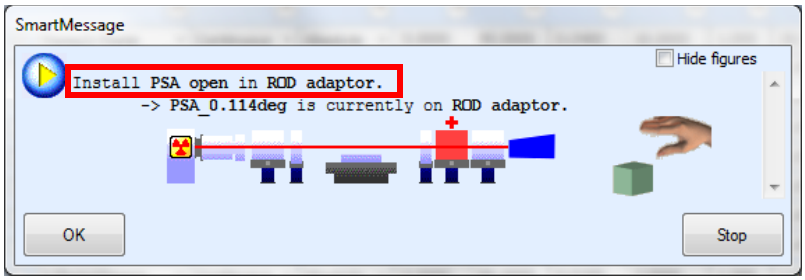
> **Mode: Continuous**
> **Range: Absolute**
> **Start (deg): 10.0000.** Never change to below **5**. The detector will be damaged.
> **Stop (deg): 90.0000,** the upper limit.
> **Step (deg): 0.0500**
> **Speed Duration time (degree/min): 10.0000,** choose lower speed to smooth spectra.
> **IS mm**: **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.

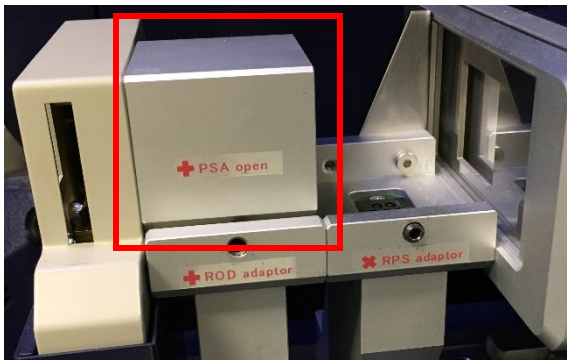
f) Select box near **Drive the 4 axes to the current zero positions after measurement completed**.

g) Click **Execute** button to start measurement

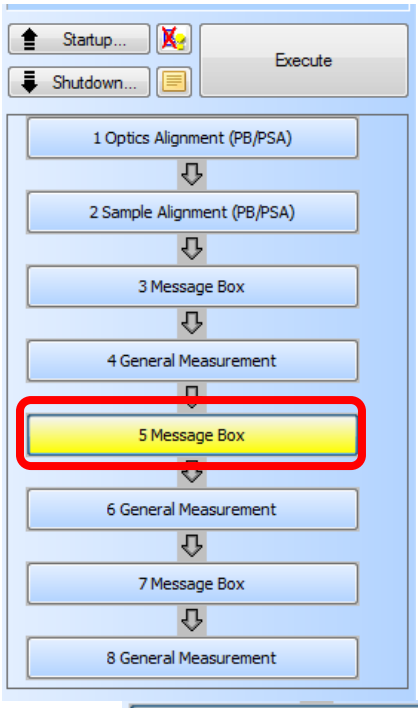


5) Follow the instruction on SmartMessage window below to replace the **PSA_0.114deg** unit with **PSA_open** and click **OK**:



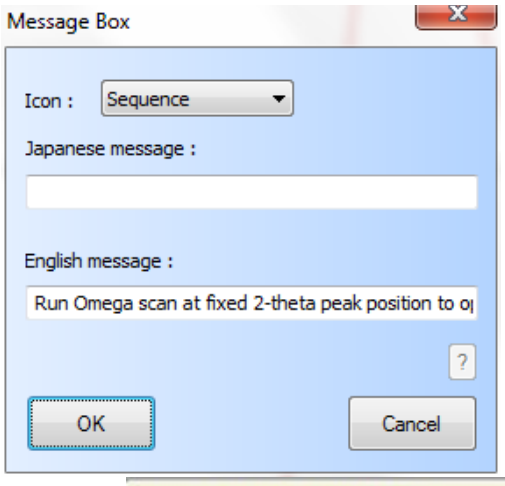


6) Click **OK** button on **General Measurement** window and popup **File Save** window and find the peak position of the interest in saved spectrum.
7) Skip the **steps 5-7** inside the window below if the X-ray grazing angle is known from previous measurements on similar samples. Otherwise proceed with steps below.

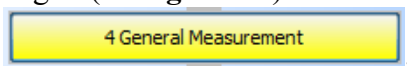


8) Click on 

button above and read the message in the Message Box below and click **OK** to close.



9) Click the 

in the window below decide the X-ray grazing angle (**Omega** scan) with the detector fixed at the 2-theta peak position found in 

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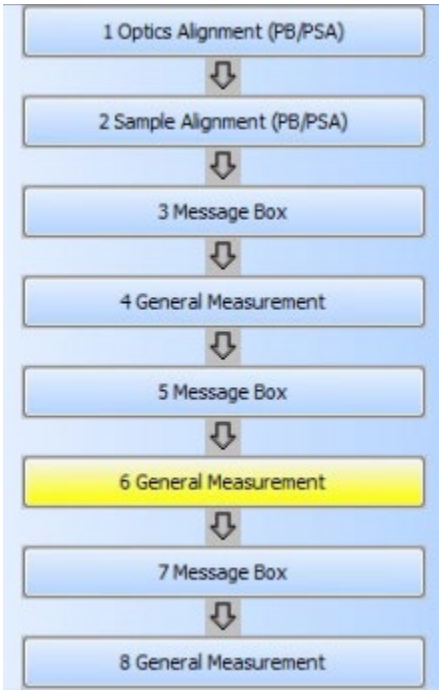
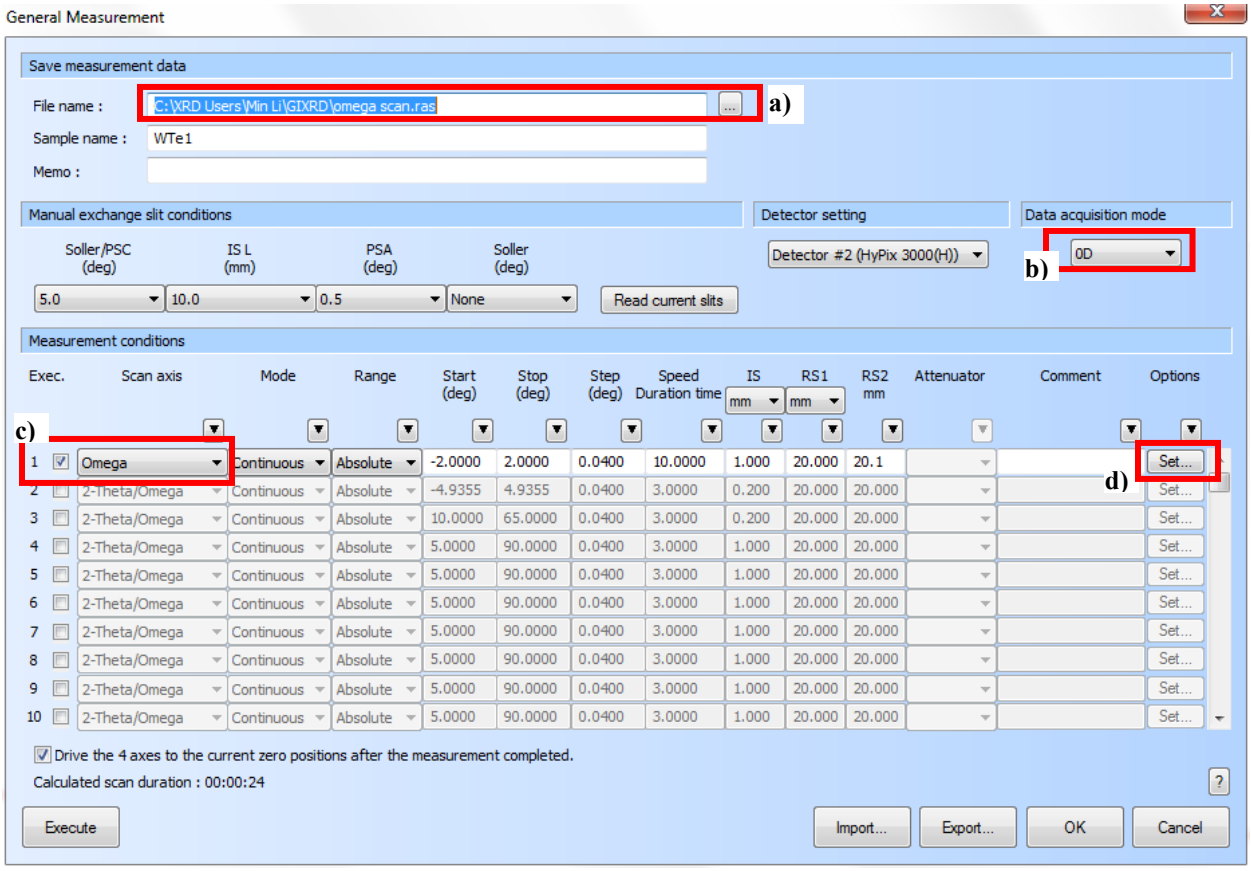
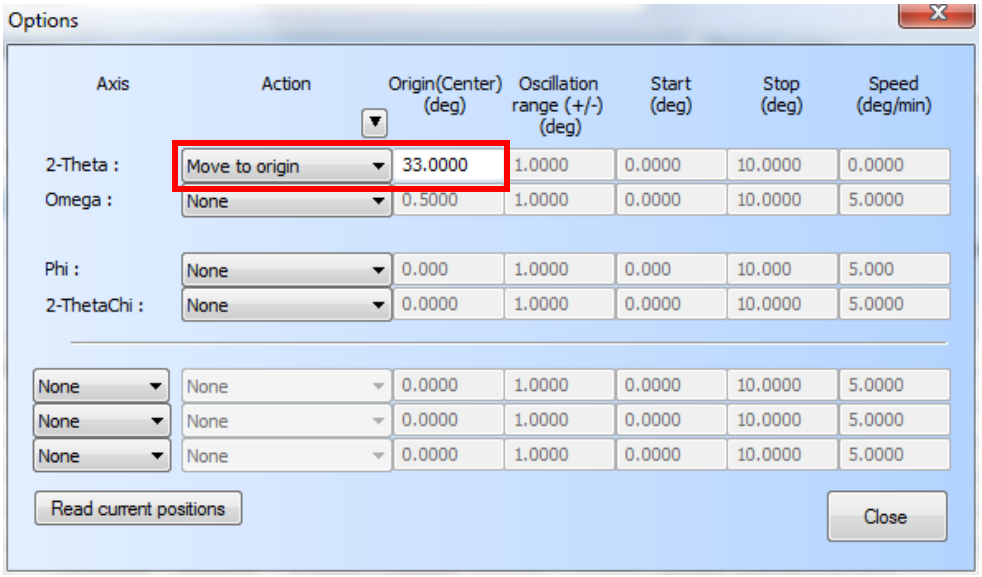


10) Perform highlighted steps on the popup **General Measurement** window below:
    a) Specify file name and folder,
    b) Make sure 0D is selected in Data acquisition mode,

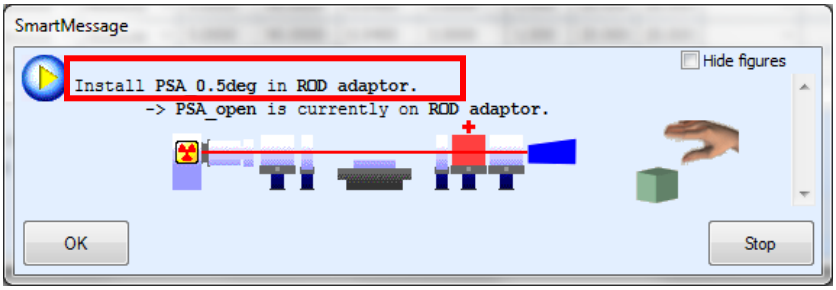
c) Select Omega in Scan axis.

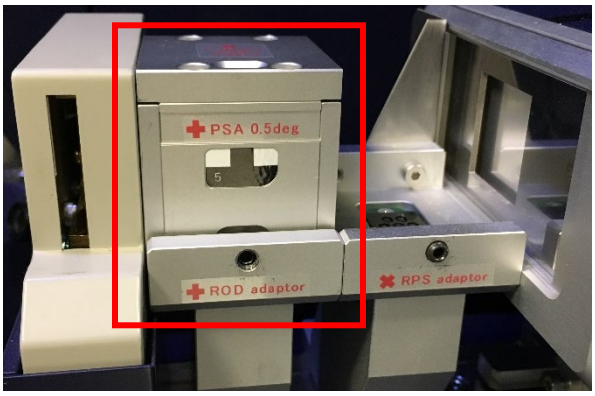


11) Click **Set** button to open the Options window below. In the highlighted area, select **Move to origin** and input 2-Theta angle, the peak of interest found in **Step 5)** and click **Close** button.

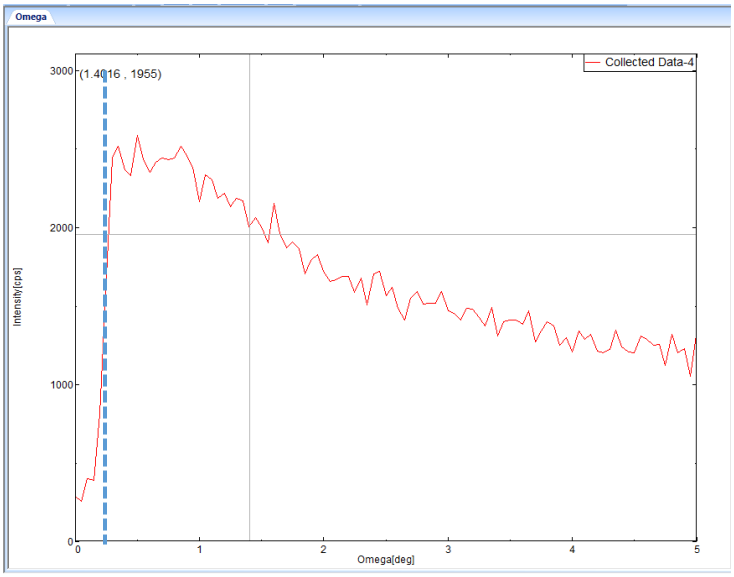


12) Go back to **General Measurement** window in **Step 11)** above and click **Execute** button.
13) Follow instruction on SmartMessage window below to install the **PSA_0.5deg** unit and click **OK** button.



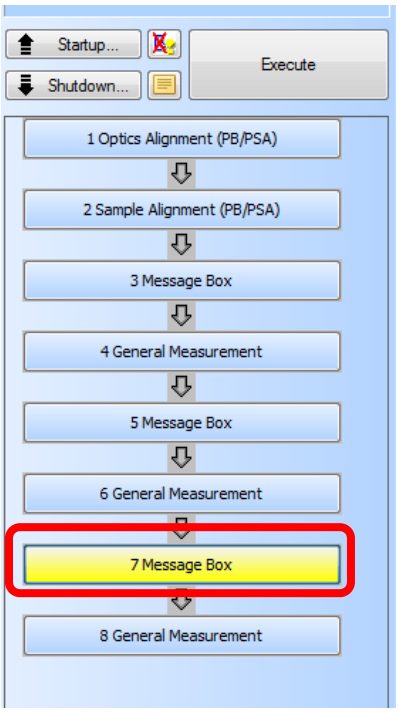


14) Choose the Omega angle close to the plateau of the Omega scan curve as shown below. Typically the angle at or slightly below dashed line is good to have enough signal intensity.

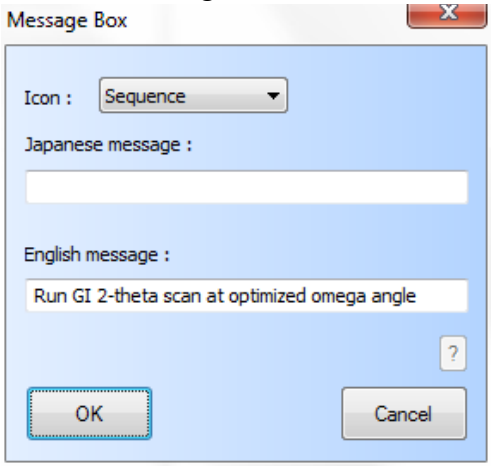


15) Click on 

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

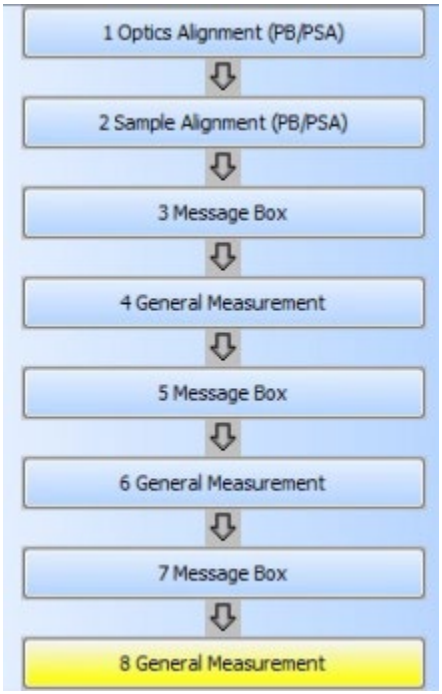


16) Read the message and click **OK** to close the window below.

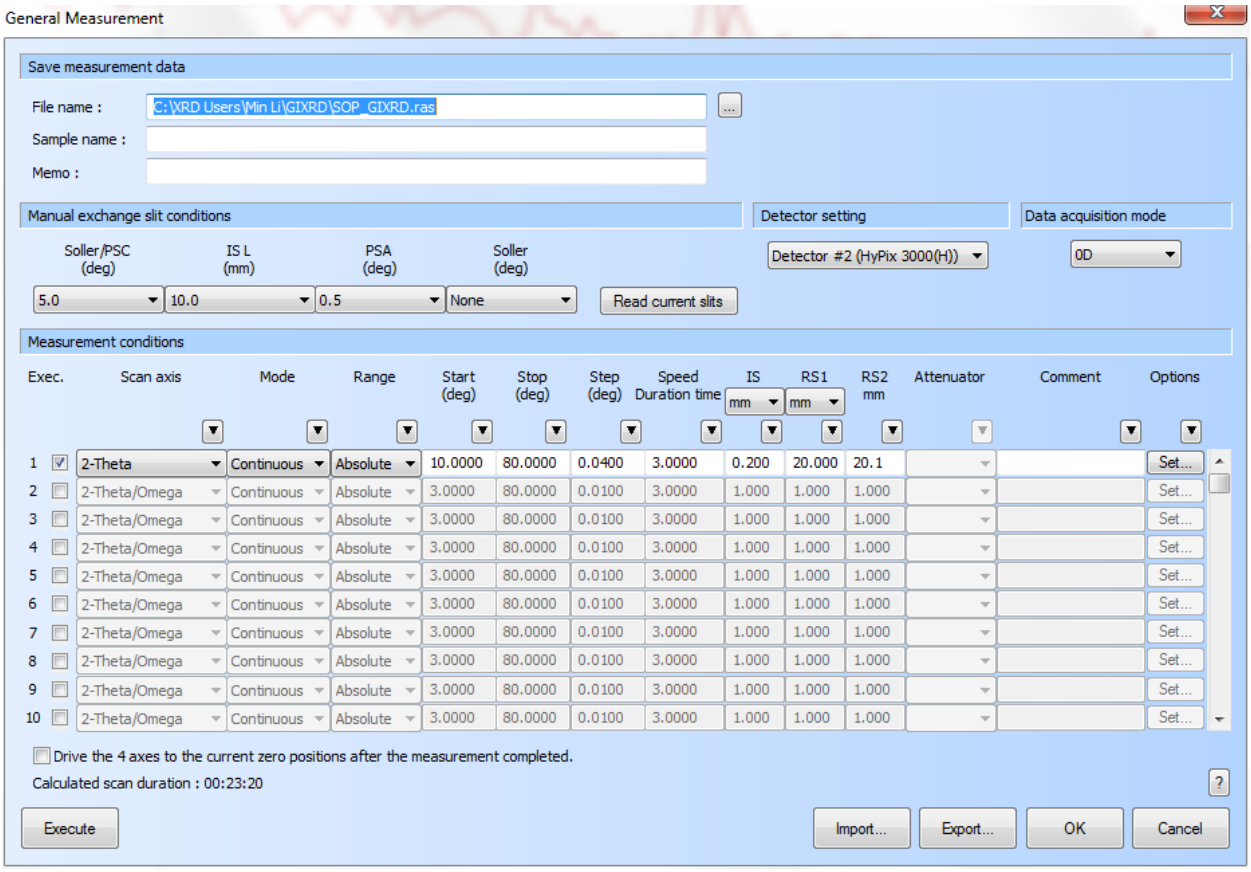


17) Click 

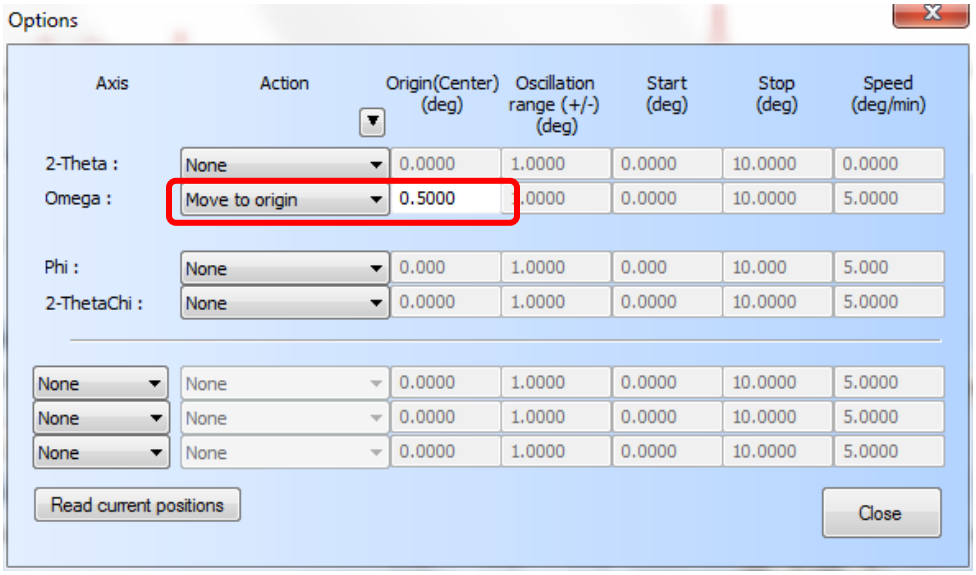
in the window below to perform the final grazing incidence scan.



18) Perform highlighted steps on the popup **General Measurement** window below:
   a) Specify file name and folder,
   b) Make sure 0D is selected in Data acquisition mode,



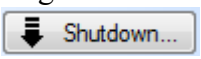
19) Click **Set** button to open the Options window below. In the highlighted area, select **Move to origin** and optimized Omega angle found in 6 General Measurement and click **Close** button.



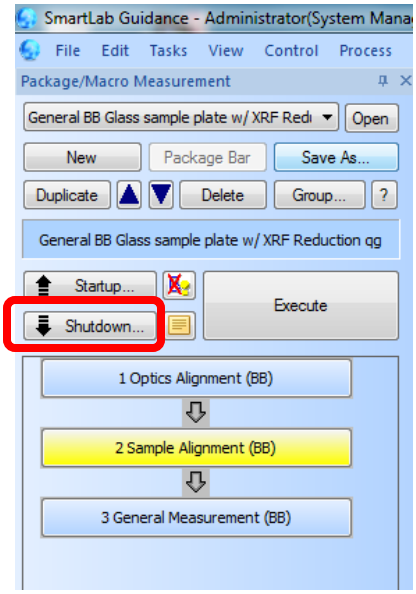
20) Go back to **General Measurement** window in **Step 18)** above and click **Execute** button.

8 Checklist after Experiment

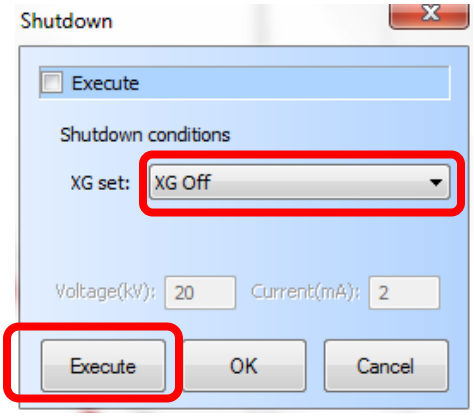
1) Back up your data to Yale Box cloud drive (box.yale.edu) or using Core USB drive. **Do not** use personal USB drive.
2) **Remove** sample from the stage. **Never** leave samples inside XRD.
3) Open a BB scan package file in your folder and **perform BB optical alignment**. Please check the powder XRD SOP for further instructions.
4) Put back the **K-Beta filter** after finish.
5) Remove the **center slit** from the stage.,

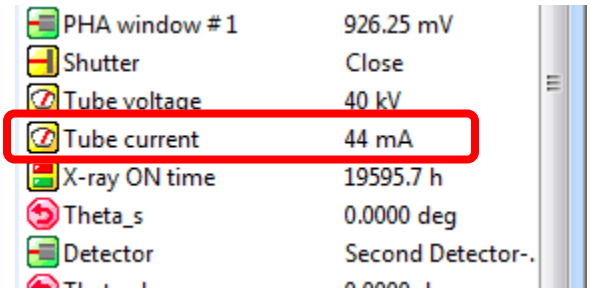
6) The user who finishes near **6pm** at regular time or **anytime** in off-peak hours should shutdown the instrument by clicking 

in the window below:



7) 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 logging off FOM.





8) **Never minimize** or **close SmartLab Guidance** software**.**

9) 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.

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