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

- 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.

AR FUM Scree	HI LUCK
	Click here to login with NetID
	Or 💆
	Click here if you do not have a NetID

- 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 **Startup...** 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.

Startup	
Timer	
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	
Execute OK Cancel	

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 **1** Optics Alignment (PB/PSA) in the **Package/Macro Measurement** window below:

Startup Shutdown Execute	
1 Optics Alignment (PB/PSA)	
4	
2 Sample Alignment (PB/PSA)	
<u>. </u>	
3 Message Box	
4 General Measurement	
₽	
5 Message Box	
₽	
6 General Measurement	
₽	
7 Message Box	
8 General Measurement	

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

Optics Alignment (PB/PS/	A)							
Change optics								
Current attribute :	Bragg-Brentano focusing							
Destination attribute :	Medium resolution parallel beam/PSA							
Optics alignment condition	ns							
Change optics without	Change optics without alignment							
Optics alignment name :	@Medium resolution PB/PSA 🗸							
Print out results after the second	er alignment							
	2							
Execute	Import Export OK Cancel							

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 2 Sample Alignment (PB/PSA) as highlighted in the Package/Macro Measurement

window below. For samples with similar heights, only the first sample alignment is required.

Startup Ke Execute
1 Optics Alignment (PB/PSA)
2 Sample Alignment (PB/PSA)
3 Message Box
<u>. </u>
4 General Measurement
<u></u>
5 Message Box
6 General Measurement
7 Message Box
8 General Measurement

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.

Sample Alignment (PB/PSA)
Sample alignment conditions
🔿 No height alignment
Curved sample (Z scan only)
Flat sample
Sample thickness (mm): 0.5
Run recommended sequence Customize conditions Customize
✓ Put a sample when the sample alignment starts.
Put a sample every time the sample alignment starts in a repeated measurement.
Print alignment result.
?
Execute Import Export OK Cancel

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.

Startup	Execute
1 Optics A	lignment (PB/PSA)
	Ŷ
2 Sample A	Alignment (PB/PSA)
	0
3 M	lessage Box
	\checkmark
4 Gener	al Measurement
	₽
5 M	lessage Box
	Û
6 Gener	al Measurement
	₽.
7 M	lessage Box
	Ω
8 Gener	al Measurement
Clister	3 Message Box

2) Click on <u>3Message Box</u> button as highlighted in the **Package/Macro Measurement** window below. Read the message and click OK to close.

Message Box	×
Icon : Sequence Japanese message :	•
English message : Run general theta/2theta	scan to find interested pe
ОК	? Cancel

3) Click on

4 General Measurement

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.

Startup 🔀 Execute
1 Optics Alignment (PB/PSA)
$\overline{\Omega}$
2 Sample Alignment (PB/PSA)
3 Message Box
V
5 Message Box
6 General Measurement
7 Message Box
8 General Measurement

- 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.
- <mark>> Step (deg): 0.0500</mark>
- > 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

-	Save r	measuremen	it data															
ſ	File n	ame :	C:\XRD L	Jser	rs\Min Li\GIXF	RD\	SOP1.ras											
1	Samp	le name :																
	Memo	:																
Г	Manua	al exchange :	slit conditi	ons									De	tector sett	ina		Data acquisition n	node
1		Celles /DCC					DCA			Caller								
		(deg)		(1	mm)		(deg)			(deg)	c) —		P	etector #2	2 (HyPix)	3000(H)) 🔻	b)	•
	5.0		▼ 10.0		-	0	pen	_	▼ None	-	Rea	d current slits						
	Money	rement cons	ditions			_												
	-	rement conc	uluons				_			-								
	Exec.	Scar	n axis		Mode		Range		Start (deg)	Stop (deg)	Step (deg) [Speed Duration time	IS	RS1	RS2 mm	Attenuator	Comment	Option
			ſ	•	ſ	-	ſ	Ŧ										
	1 🔽	Theta/2-T	hota	Ţ	Continuous	Ţ	Absolute	•	5.0000	00.0000		10.0000	1 000	20.000	20.1			Set
			neta		Contandudus					90.0000	0.0400	10.0000	1.000	20.000	20.1	· · · ·		
	2	z-meta/o	mega		continuous	Ξ	ADSOIUTE	Ţ	5.0000	90.0000	0.0400	3.0000	1.000	1,000	1.000			
	3	2-meta/0 2-Theta/0	mega)mega		Continuous Continuous	÷	Absolute Absolute	÷	5.0000	90.0000	0.0400	3.0000	1.000	1.000	1.000			Set
	2 🖸 3 🗖 4 🗖	2-meta/0 2-Theta/0 2-Theta/0	mega Omega Omega	-	Continuous Continuous Continuous	•	Absolute Absolute Absolute	· ·	5.0000 5.0000 5.0000	90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000	1.000 1.000 1.000	1.000 1.000 1.000	1.000 1.000			Set Set
	2 E 3 E 4 E	2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O	mega Omega Omega Omega		Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute	• • •	5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000			Set Set Set
	2 E 3 E 4 E 5 E 6 E	2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O	imega Dimega Dimega Dimega Dimega		Continuous Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute Absolute	• • •	5.0000 5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000			Set Set Set Set Set
	2 2 3 2 4 2 5 2 6 2 7 2	2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O	Imega Imega Imega Imega Imega Imega		Continuous Continuous Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute Absolute	* * * * *	5.0000 5.0000 5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000			Set Set Set Set Set Set Set
	2 0 3 0 4 0 5 0 6 0 7 0 8 0	2-meta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0	omega Omega Omega Omega Omega Omega		Continuous Continuous Continuous Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute Absolute Absolute	* * * *	5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000			Set Set Set Set Set Set Set Set Set
	2 3 2 4 2 3 4 2 5 2 5 2 5 2 5 5 5	2-meta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0	omega Omega Omega Omega Omega Omega Omega		Continuous Continuous Continuous Continuous Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute Absolute Absolute Absolute	* * * * *	5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000			Set
	2 1 3 1 4 1 5 1 6 1 7 1 8 1 9 1 10 1	2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O 2-Theta/O	inega Dimega Dimega Dimega Dimega Dimega Dimega		Continuous Continuous Continuous Continuous Continuous Continuous Continuous Continuous Continuous		Absolute Absolute Absolute Absolute Absolute Absolute Absolute Absolute	* * * * * * * *	5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000	90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100	10.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000			Set Set
	2 0 3 0 4 0 5 0 6 0 7 0 8 0 9 0 10 0	2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0 2-Theta/0	Imega Imega Imega Imega Imega Imega Imega Imega Imega Imega		Continuous Continuous Continuous Continuous Continuous Continuous Continuous Continuous Continuous ent zero posi		Absolute Absolute Absolute Absolute Absolute Absolute Absolute Absolute ns after th		3.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 5.0000 eesuremen	90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000 90.0000	0.0400 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100 0.0100	3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000 3.0000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000	1.000 1.000 1.000 1.000 1.000 1.000 1.000 1.000			Set Set

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.

Startup Ke Execute Shutdown	
1 Optics Alignment (PB/PSA) 2 Sample Alignment (PB/PSA)	
3 Message Box	
4 General Measurement	
5 Message Box	
7 Message Box	
8 General Measurement	
8) Click on 5 Message Box	button above and read the message in the Message

Box below and click **OK** to close.

Message Box
Icon : Sequence Japanese message :
English message : Run Omega scan at fixed 2-theta peak position to or
? OK Cancel

9) Click the ^{6 General Measurement} in the window below decide the X-ray grazing angle (**Omega** scan) with the detector fixed at the 2-theta peak position found in

4 General Measurement								
1 Optics Alignment (PB/PSA)								
₽ ₽								
2 Sample Alignment (PB/PSA)								
Ŷ								
3 Message Box								
Ŷ								
4 General Measurement								
Ŷ								
5 Message Box								
Ŷ								
6 General Measurement								
Ŷ								
7 Message Box								
Ŷ								
8 General Measurement								

- 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.

General N	leasurement												×
Save m	easurement data												
-1				_					•				
File na	me:	ers (Min LI (GIXRD	vomega scan.r	85				. a	,				
Sample	ename: WTe1												
Memo	:												
Manual	exchange slit condition	IS						Det	ector set	ting		Data acquisition m	ode
S	oller/PSC	ISL	PSA		Soller			6	atactor #		2000/41)		
	(deg)	(mm)	(deg)		(deg)				elector #	2 (Hyrix .	JUUU((1)) +	b)	
5.0	▼ 10.0	▼ [0	.5	▼ None	•	Rea	id current slits						
Measur	ement conditions												
Ever	Scan avie	Mode	Pange	Start	Stop	Sten	Speed	15	DS1	P \$2	Attenuator	Comment	Ontions
EXCC.	Scarraxis	Houe	Runge	(deg)	(deg)	(deg) I	Duration time	mm 🔻	mm 👻	mm	Attendator	connent	options
c)	T					▼		•					
1 🗸	Omega 🗸	Continuous 🔻	Absolute 🔻	-2.0000	2.0000	0.0400	10.0000	1.000	20.000	20.1			Set
2	2-Theta/Omega 🔹	Continuous 👻	Absolute 👻	-4.9355	4.9355	0.0400	3.0000	0.200	20.000	20.000	-	d)	Set
3 🔳	2-Theta/Omega 🔹	Continuous 👻	Absolute 🔻	10.0000	65.0000	0.0400	3.0000	0.200	20.000	20.000			Set
4 🔳	2-Theta/Omega 🛛 👻	Continuous 👻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
5 📃	2-Theta/Omega 👻	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
6 📃	2-Theta/Omega 🔹	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
7 🔳	2-Theta/Omega 🔹	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
8 🔳	2-Theta/Omega 🔹	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
9 📃	2-Theta/Omega 🔻	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
10	2-Theta/Omega 🔹	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set 👻
V Driv	/e the 4 axes to the cu	rrent zero positio	ons after the m	easuremen	t completed								_
Calcula	ited scan duration : 00	:00:24											?
Exec	ute								In	nport	Export	ок	Cancel
2.00													

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.

Avis	Action		Origin(Center)	Oscillation	Start	Stop	Speed
0,00	Action	▼	(deg)	range (+/-) (deg)	(deg)	(deg)	(deg/min)
2-Theta :	Move to origin	-	33.0000	1.0000	0.0000	10.0000	0.0000
Omega :	None	•	0.5000	1.0000	0.0000	10.0000	5.0000
Phi :	None	-	0.000	1.0000	0.000	10.000	5.000
2-ThetaChi :	None	-	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	-	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
Read current po	ositions					[Close

- 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	7 Message	Box	button as	highlighted	in the	Package/Macro
Measuren	nent window belo	w.		88		
Startup	Execute					
1 Optic	s Alignment (PB/PSA)					
	<u>₽</u>					
2 Samp	le Alignment (PB/PSA)					
	<u></u>					
	3 Message Box					
4.60						
466						
	5 Message Box					
						
6 Ge	neral Measurement					
	7 Message Box					
	⊘					
8 Ge	neral Measurement					
16) Read the r	nessage and click	OK to close	the window	v below.		
Message Box		x				
Icon : Sec	juence 🔻					

	Icon : Sequence	
	Japanese message :	
	English message :	
	Run GI 2-theta scan at optimized omega angle	
	?	
	OK Cancel	
17)	Click 8 General Measurement	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,

ieneral Measurement															
Save m	easurement data														
File na	me : C:\XRD	User	rs\Min Li\GIXRD	SOP_GIXR	D.r	as			(
Sample	name :														
Memo	:														
Manual	exchange slit condit	ions								De	etector sett	ina		Data acquisition m	ode
S	oller/PSC (deg)	I (1	IS L mm)	PSA (deg)			Soller (deg)			(Detector #	2 (HyPix 3	3000(H)) 🔻	OD	•
5.0	▼ 10.0		▼ [0	.5		▼ None	•	Rea	ad current slits						
Measur	ement conditions														
Exec.	Scan axis		Mode	Range		Start (deg)	Stop (deg)	Step (deg)	Speed Duration time	IS mm	RS1	RS2 mm	Attenuator	Comment	Options
	(T) (T										
1 🔽	2-Theta	•	Continuous 🔻	Absolute	•	10.0000	80.0000	0.0400	3.0000	0.200	20.000	20.1			Set 🔺
2 🔳	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
3 🔳	2-Theta/Omega	-	Continuous 💌	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000	-		Set
4 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
5 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
6 📃	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
7 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000	-		Set
8 📃	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000	-		Set
9 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
10 📃	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set 👻
Drive the 4 axes to the current zero positions after the measurement completed. Calculated scan duration : 00:23:20															
Exec	ute										In	nport	Export	ок	Cancel

GIXRD

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.		

Axis	Action		Origin(Center) (deg)	Oscillation range (+/-) (deg)	Start (deg)	Stop (deg)	Speed (deg/min)
2-Theta :	None	-	0.0000	1.0000	0.0000	10.0000	0.0000
Omega :	Move to origin	•	0.5000	.0000	0.0000	10.0000	5.0000
Phi :	None	•	0.000	1.0000	0.000	10.000	5.000
2-ThetaChi :	None	•	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔹	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
Read current or	sitions					ſ	~

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 Shutdown... 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.

Shutdown		
Execute		
Shutdown conditions	🗐 PHA window # 1	926.25 mV
XG set: XG Off 🔹	🕂 Shutter	Close
	Tube voltage	40 kV
	🕜 Tube current	44 mA
Voltage(kV): 20 Current(mA): 2	🔚 X-ray ON time	19595.7 h
	😏 Theta_s	0.0000 deg
Execute OK Cancel	Detector	Second Detector
	AT1 . 1	0.0000 1

- 8) Never minimize or close SmartLab Guidance software.
- 9) Logoff FOM program: click the ¹/₂ 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.