User guide for RIGAKU powder diffractometer

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• X-ray set

1. Open "XG Control" window and click "**SET kV, mA**" to set X-ray tube at ___**kV and ___mA** for the operation (standby setting is 20kV, 2mA).

💰 JXG			
File Edit Instrument Tools Tasks Window Help			
XG Control			
XG Control XG State			
XG TYPE	XG S	TATUS	
SealedTube (Mo, LongFine) kV : min = 20, max = 54, step = 2 mA : min = 2, max = 54, step = 2	NOT READY READY	X-RAY ON ALA	RM
kW : max = 2.70	1,		
POWER X-RAYS	X-RAY POWER	FUNC	TION
ON ON	50 50 mA 2.	50 kW Abort X	GTask
ON ON OFF	50 kv 50 mA SET kV,mA	Reset	Narms
Alarms	Alarm Code	Shutters	
[CW] Cooling Water [PF] Pow	er Failure No.1 7	SHUTTER A CLOSED	
🥚 [HV] High Voltage 🛛 😑 [LV] Low	Voltage No.2 ?		
😑 [TC] Tube Current 🛛 😑 [FC] Filar	nent Current No.3 ?	SHOTTER B CLOSED	
[OL] Overload	Current No.4 ?		
	No.5 ?		
E Messages			_ <u>_</u>
kV changed to 50, mA changed to 50 (2.50	kW)		
kV changed to 50, mA changed to 30 (1.50	kw)		
kV changed to 40, mA changed to 20 (0.80	kW)		
ky changed to 30, mA changed to 10 (0.30	R.00) 533		
kV changed to 24, mA changed to 2 (0.04 k)	a)		
kV changed to 28, mA changed to 2 (0.06 k	(a)		-
			R

2. Wait until the X-ray tube ramp up to 50kV, 50mA on the Rigaku x-ray diffractometer.



• Sample set on the machine

- 1. Open the **RINT RAPID** control software, and **Goniometer control** (Manual>Goniometer control). Select **Phi** position as **Init.**
- 2. Set your sample on the holder. Open the door (press the "door" button before opening machine).
- 3. Mount goniostad on the manual XYZ stage.

• Centering

- 1. Rotate Phi to 0°.
- Select Omega window, toggle move and enter a value of ____°. Center and focus the area for x-ray measurement on the cross section using X, Y and Z screws. Rotate Omega to ____°and adjust the area of interest using only Z screw.
- 3. Rotate Phi to 360° to check your sample is centered properly.

• Measurement

1. Open the RINT RAPID control software.

Quit Device check San	No shutter CCD camera XG control Display	Stop
Project Data folder : C:(raxwish)ima Available disk: 38.09GB	ge\Group_name	
Device status	Standard Measurement Image	Log
Omega-axis : 0.000	- File/Sample	
Phi-axis : 360.000	File name	
	The number of IcaAlsite	Target Mo
IP position : Pood	_ Sample I	X-ray:Measureme 50 🛨 KV 50 🛨 mA
n position read	Operator name User name	after Meas. 📀 hold C mini. C off
Eraser lamp : Off	Memo IRINT RAPID	Exposure time
Shutter : Close	Resolution 100x100 Micrometer	Collimator
IP read conditions		
Read line : 0	2Teata-I Auto Conversion	
Read range : Wide	C Execute Conversion (Don't Execute Conversion	Version Conditions
- 	Goniometer	0 mm
1 1/61 31/6 . 4700/2300	Attachment stage . [Manual-X1-stage]	
Image file list	Sample holder : [Reflection(small)]	Change
	Omega-axis : Oscillation , Position (deg); 0 <-> 40 ,	Speed (deg/sec); 1 Mode
	Phi-axis : Oscillation , Position (deg); -180 <-> 0 , S	Speed (deg/sec); 3
	Drive	test
	Measure/E	Execute
Process(PID):: Gonio server: 3/	18 XG server: 548 Video: 0 Xa control: 0 Display: 0	9/12/2013 10:19

- 2. Change the folder and file name. Go to **Project>data folde**r and choose your folder. Then give the new file name, sample and operator name.
- 3. Set exposure time (10-15 min), stage, sample holder conditions and axis motions. See Table 1.



Table1. Appropriate stage, sample holder, axis motions and speed for different samples.

Samples	Attachment stage	Sample holder	Axis motions		Speed	Range
			Omega	Fixed		
				Oscillation	1 degree/min	0 <omega<40< td=""></omega<40<>
on glass fibers	Manual-XY-stage	Reflection (small)		Fixed		
of tubes			Phi	Oscillation	3 degree/min	-180 <phi<0< td=""></phi<0<>
			Spin	3 degree/min	-180 <phi<0< td=""></phi<0<>	
on solid surfaces Ma		Reflection (small)	Omega	Fixed	1 degree/min	35 <omega<60< td=""></omega<60<>
				Oscillation		
	id Manual-XY-stage Reflection (sma		Phi	Fixed		
				Oscillation	3 degree/min	-180 <phi<0< td=""></phi<0<>
				Spin	3 degree/min	-180 <phi<0< td=""></phi<0<>

• Run test measurement

Click the "Drive test" button to see there is no collision.

• Press the measure/Execute button

Now your raw data (X-ray image file) is saved under c:\raxwish\image\Group_name\your_name*.img on RIGAKU instrument computer.

- 2DP Pattern Integration (software to integrate the X-ray pattern)
 - 1. **Load**>double click the file



2. Convert to profile. Select the region you want to analyze.



3. Click Run.

Convert Image to Profile				
Load			xxv_RTF10411 #1	_
Correct Background				
T.	· · · · · · · · · · · · · · · · · · ·			
Convert to Profile				
T.	I I			
Smooth				
	20 -			
Cabined Devicement				
Subtract Background	-			
V				
Strip Ka2				
4	18-			
Find Peaks	<u></u>			
	i i i i i i i i i i i i i i i i i i i			
	8)			
	2 10-			
	Iter			
d				
	14-			
2 302 40 818				
11.120 21.187				
Differences				
-38.515				
-10.066				
	have been been as a fight of the second s			
	There is a second s	10		
			2-Theta (deg)	
	CG 2D Image Set	2D Image Set	L 1D Profile Set	Inser
	Ge 1 Goe_Cr	BG_Goe_Cr	1DConv_BG_Goe_Cr	Remo
	2 Goe_Cr2	BG_Goe_Cr2	1DConv_BG_Goe_Cr2	5.00
	Goe_Al	BG Goe 2wk	1DConv_BG_Goe_2wk	Jave
		ou_uou_erm	incom_nd_dot_time	Bena

Now your integrated data (RINT ASCII file) is saved under **z:\Group_name\your_name*.asc** on RIGAKU instrument computer.

• Data analysis

For the data analysis using JADE program, the data you saved on z:\ drive is also shared under c:\Document and Settings\All Users\Documents\data\Group_name\your_name\ on the JADE computer.

• Finishing session

- 1. Set the X-ray standby mode: If no one is booked after your measurement is done right away, turn down the X-ray to the standby setting at ____kV and ___mA. (Keep the X-ray ON.)
- 2. Turn off the light for specimen illumination and the monitor.
- 3. Record your activities (data, your name, PI's name, the number of your sample patterns) on the log book.