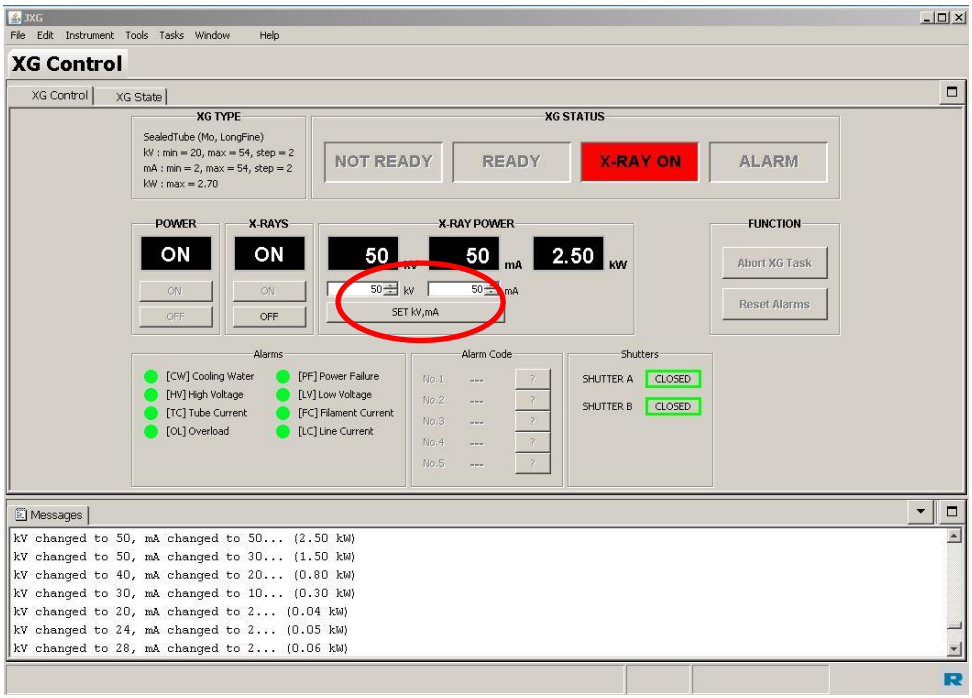
User guide for RIGAKU powder diffractometer

September 2013 by Dr. Rie Fredrickson

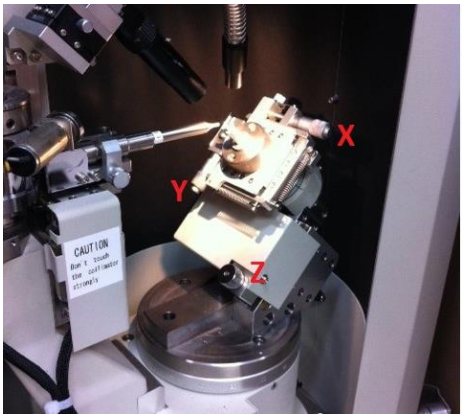
- **X-ray set**
  1. Open "XG Control" window and click "**SET kV, mA**" to set X-ray tube at **__kV and __mA** for the operation (standby setting is 20kV, 2mA).



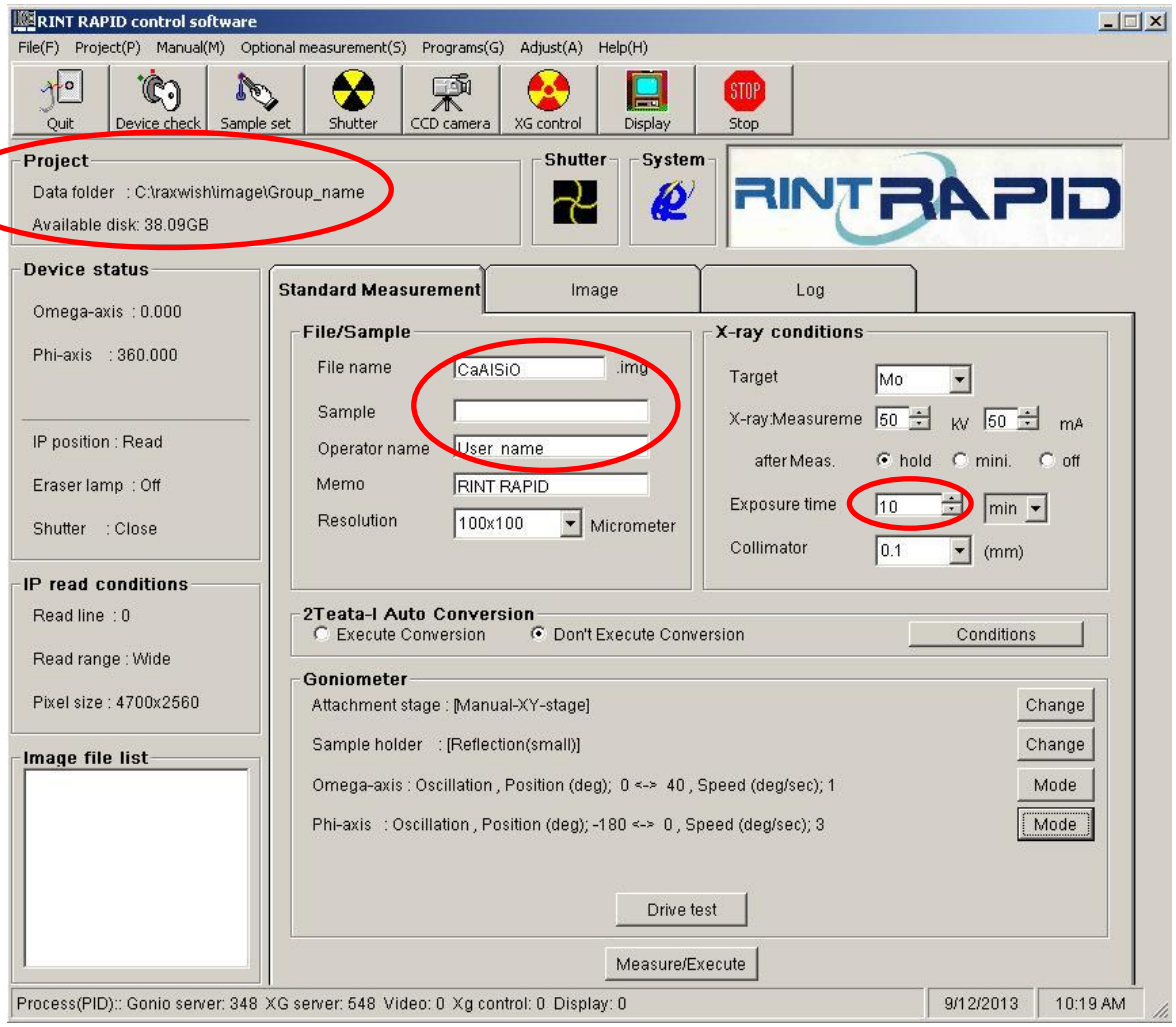
  2. Wait until the X-ray tube ramp up to 50kV, 50mA on the Rigaku x-ray diffractometer.

- **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°.
  2. 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.



  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
on glass fibers or tubes	Manual-XY-stage	Reflection (small)	Omega	Fixed		
				Oscillation	1 degree/min	0<omega<40
			Phi	Fixed		
				Oscillation	3 degree/min	-180<phi<0
				Spin	3 degree/min	-180<phi<0
on solid surfaces	Manual-XY-stage	Reflection (small)	Omega	Fixed	1 degree/min	35<omega<60
				Oscillation		
			Phi	Fixed		
				Oscillation	3 degree/min	-180<phi<0
				Spin	3 degree/min	-180<phi<0

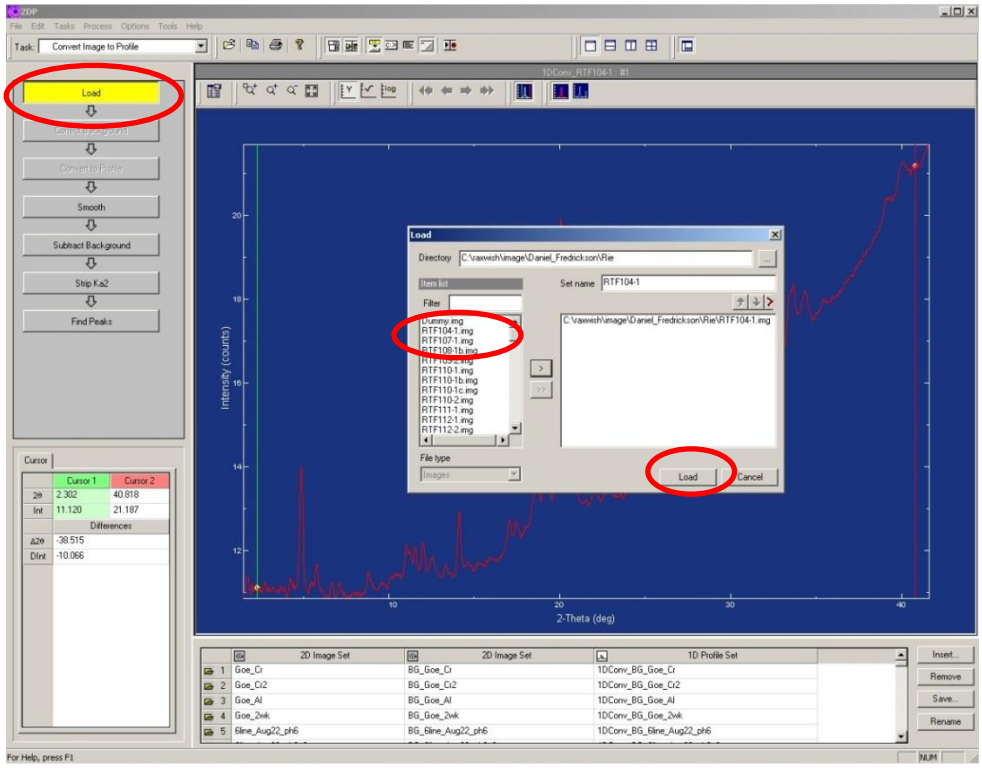
- **Run test measurement**

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

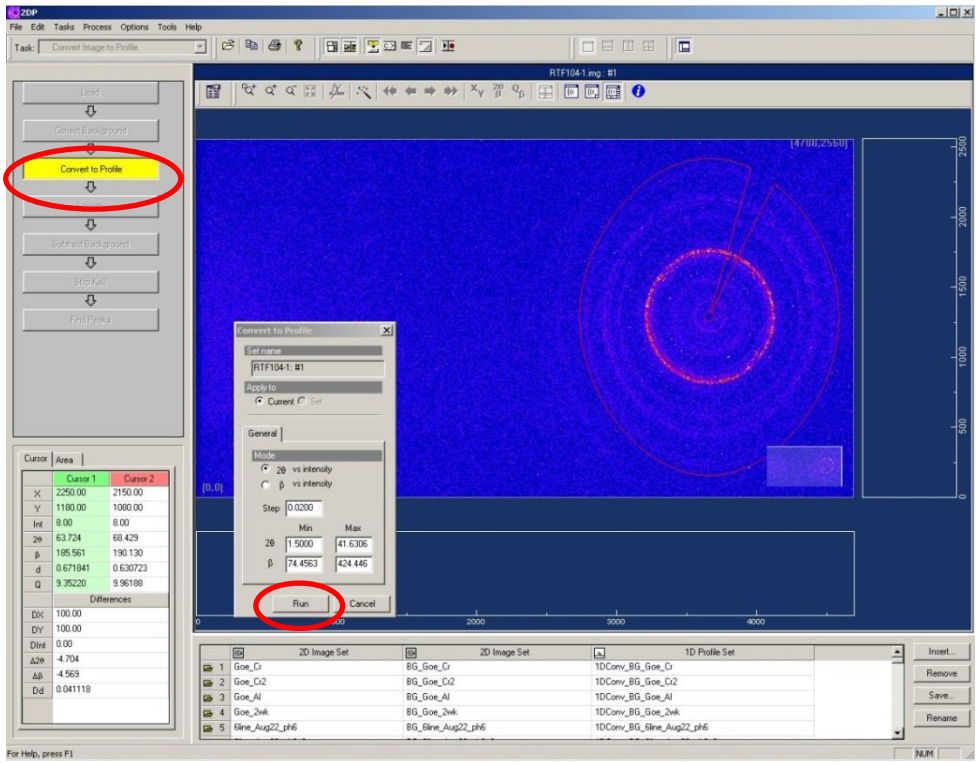
- **Press the measure/Execute button**

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

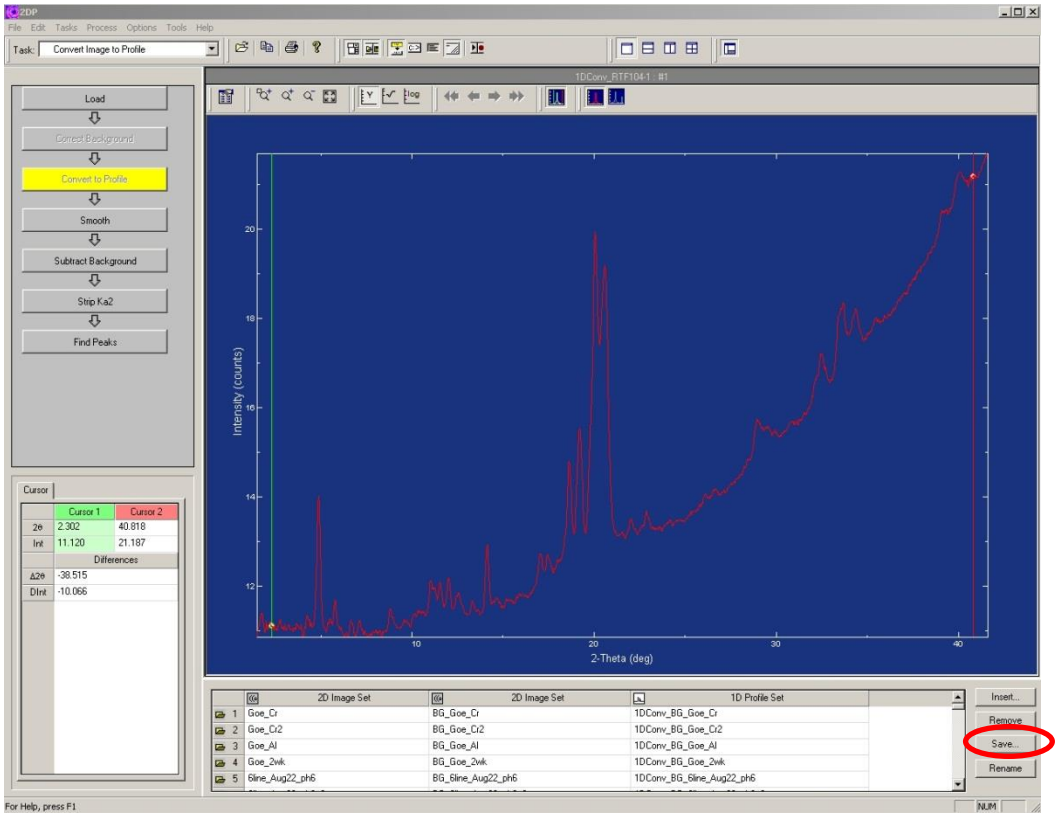
- **2DP Pattern Integration** (software to integrate the X-ray pattern)
  1. **Load**>double click the file



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



3. Click **Run**.



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.