

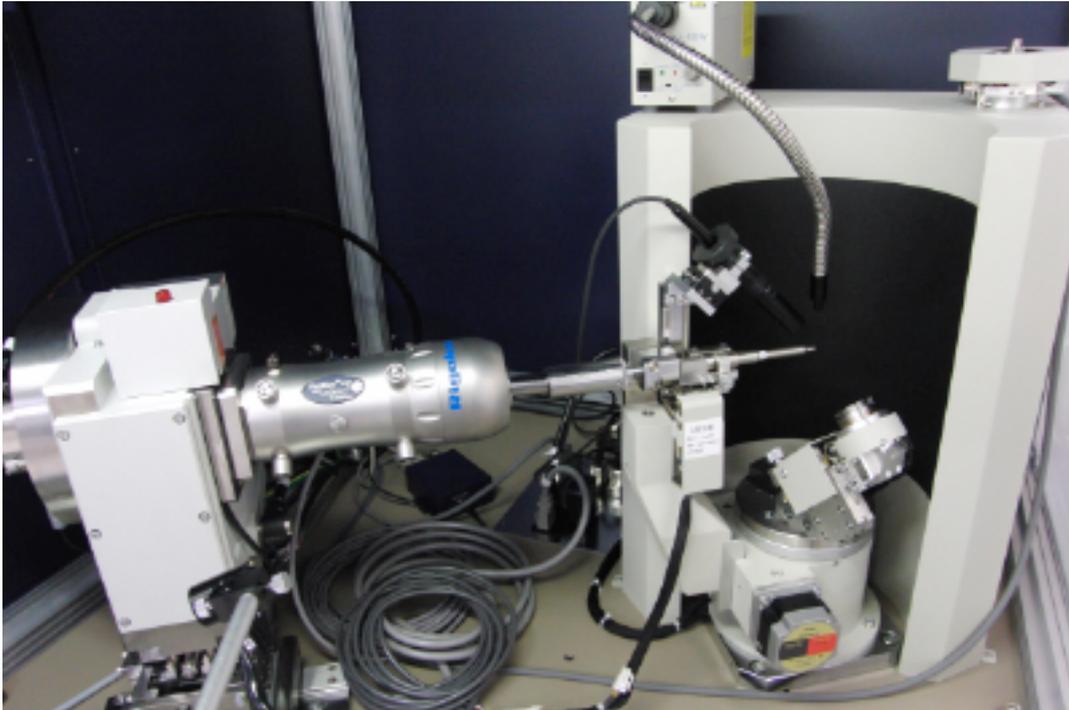
RINT RAPID II Quick Manual

This is quick manual for RINT RAPID II. For details, refer to original manual and references. Note that CMS DW focusing mirror was new, so it is not mentioned in most of the manuals. If you have questions, and for target change, alignment etc, please ask Dr. Kanzaki.

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RINT RAPID II CMF-DW

Summary of RINT RAPID II CMF-DW (Rigaku Co.)

Cu/Mo rotating anode targets (1.2 kW)

Focusing mirror (VariMax DW)

Goniometer (ω, ϕ axes rotation)

Collimator: 10, 30, 50, 100, 300 and 800 μm (beam itself is smaller than last two)

Curved imaging plate detector (Camera length: 127.30 mm)

Warning:

1. Please cleanly use the instruments, the tables, and the room. Don't use sand paper in this room. Current situation is worst ever. If this continues, I have to force severe restrictions for all users.

2. Please wear film badge before use.

3. Please handle zoom lens of CCD camera gently.

(In followings, “” indicates switches etc on the main unit, and [] shows objects (button etc) in PC software.)

Operations:

Startup

Normally following five steps are not necessary. If power is not coming, check these steps.

1. At the back side of RINT RAPID II main unit, turn the circuit breaker to ON position.
2. Push “Controller SW” in “Power ON/OFF” of the front panel of the main unit.
3. Turn breaker at back side of X-ray generator (XG for short) unit to ON position.
4. Open front door of XG unit, and push “ON” button in “MAIN POWER”.
5. Turn breaker switch in front panel of cooling water unit (behind PC) ON. LED display will show READY and turns to current temperature reading.

Normally start from here

6. Current target should be Cu, but make sure by checking display on “target selection box” in the shield door of RINT RAPID II. If the target is not for your need, please ask Kanzaki for change.
7. Flow helium gas: Open a valve of He gas cylinder behind SmartLab. Set flow to 25 mL/min using a valve of a flow meter. At the He gas controller board on the XG unit, gas will stop when valve is at “Direct” position. Currently “He-path” side is disabled. If no gas flow, check the valve in front of the flow meter.
8. Start PC (if not started yet). Double click [RAPID_XRD] icon on desktop. It will ask whether last condition to be used. Normally answer yes (type “y”).
9. Data folder for your data will be displayed. You can not make new folder at this point. So make folder before this and select your data folder. Or just select default one. You can change later.

10. Main measurement window will open. In order to switch to English menus, hold down Alt-key, and push P, L and E keys.
11. If vacuum is not ON yet, push “START” button in “VACUUM” on the front panel of XG. Current pressure can be monitored from “STATE” display. Push “MODE” switch several times, to see IG (ion gauge). Wait until IG value becomes lower than 200 mV (0.4 mPa).
12. When “READY” lamp of “X-RAYS” turns on, and $IG < 200$ mV, then you can start X-ray generator. Push “ON” switch of “X-RAYS”, cooling water will start to flow. The voltage and current will become 20 kV and 10 mA automatically. If IG exceeds 200 mV during this process, it will be halted. Wait a while, and push “ON” switch of “X-RAYS” again.
13. Slowly increase voltage up to 40 kV by pushing voltage up key. Then slowly increase current to 30 mA for Cu target (for Mo, 50 kV and 24 mA). If current is unstable, decrease current, and wait until it stabilizes. Also check IG. Stabilization of focused beam position will take 1.5 H. This means that measured intensity will be low, if you measured just after start-up.

Sample exchange

14. In order to open shield doors, push “DOOR” button in “Safety Release”. Beeping will start, and “DOOR” button blinks. Then you can open the doors. If you skip this procedure, X-ray will be automatically turned off.
15. If light is necessary, turn fluorescent tube light switch inside of the diffractometer cabinet ON
16. When large samples or special sample holder are placed on the goniometer, then it will interfere with collimator during initialization of axes. Therefore, remove these before the initialization. Remove sample and sample holder, except “standard manual XY stage” which is default one.
17. When removing holder, it might be necessary to rotate axes. To rotate axes, select [Goniometer control] from [Manual] menu. [Gonio control] window will be displayed. Select axis tab to move, select [Move], and input angle, then press [OK] button, or use jog buttons. Please watch carefully when you are rotating any axes.
18. Click [Device check] button on the main window. Initialization will start.
19. After initialization, put sample stage. For this, rotate $\omega=45^\circ$, $\phi=0$ by [Gonio control] window. Fix the stage by two hex bolts.
20. Check [Attachment stage] in Goniometer area of the main window. If it is not correct, change it by clicking [Change] button. Do same for [Sample holder]. Soft limit for rotation is defined by here, so selecting wrong sample holder might cause collision with the collimator when rotated, or it will not rotate to the angle you specified. The angle limit is displayed in [Gonio control] window. Some collimator is longer than others, i.e., 300 μm one, rotation to lower ω angle needs close attention.
21. Put your sample on one of adequate sample holders, and put the sample holder on the manual XY stage. Move ω to 90° to observe sample by CCD camera.
22. Click [CCD camera] button. RAXVIDEO window will appear. Turn on fiber light source, and illuminate the sample. Pan the CCD camera, and by using XY stage and sample Z axis, move sample to cross of the display, and focus to the sample. Zoom the CCD camera, and move and

focus again. Rotate ϕ and watch. If the cross does not match with center of rotation, alignment is necessary (ask Kanzaki for alignment) or read alignment procedure in last part of this manual. Scale of this CCD image is 20 μm for single division (at max. magnification).

23. You can record the position of the sample you took the diffraction by taking picture. To do so, selecting menu in CCD camera window.
24. For reflection diffraction measurement, move ω to lower angle (30 to 20°) with closely watching. At lower ω angle, sample holder and collimator will interfere. If sample position moves away in the CCD image, move the sample using sample Z axis. If focus is out after ω rotation, focus adjustment of the CCD camera is necessary. Please ask Kanzaki for adjustment.
25. For transmission mode, set beam stopper on the collimator. For reflection measurement, the beam stopper is normally not necessary. But if you see direct beam image (central spot) in taken IP image, use the beam stopper.
26. After sample set, turn off fiber light source, close all shield doors. Beeping will stop.

Measurement

27. In the main window, set file name, memo etc. **Set ω to current value** (if you forget this, ω changes when the measurement started).
28. If you want to rotate axis during the measurement for better Debye-Scherrer rings, set condition. For reflection measurement, ω should be fixed as present value. ϕ could be rotated if no interference with the collimator. To check rotation, click [Test move]. If test is OK, click [STOP] button.
29. Confirm measurement conditions, and click [Measure/Execute] button. IP initialization, exposure, IP reading will be conducted automatically. It takes about exposure time plus additional few minutes. Current state can be checked at [Current status] in the main window.
30. After measurement finished, 2D image will be displayed in [Image tab] of the main window. For image processing, separate software is provided. Double click [2DP] icon on the desktop. Essentially you just need to click buttons in the flow bar in 2DP. Once the data is converted to 1D profile, you can use PDXL software for processing (for identification etc.). Now, you can also use QualX2 software for identification. You can install this software to your PC. A manual for QualX2 is available.
31. For emergency situation, press “EMO” emergency stop switch. In order to restore, rotate this button clockwise. Once you confirm safety, start from step 1 of this manual. Also restart [RAPID_XRD] software.
32. If the room is hot, decrease set temperature of air conditioner.
33. If the system does not run, or diffraction intensities are very weak, or other troubles, please contact to Kanzaki.

Stop

34. Record IG and filament current (FC) values to the log notebook. Filament current is displayed in inside of the front door.
35. Make sure shutter is closed (red lamp is OFF), and “OPEN” lamp of “SHUTTER2” is out.

36. First slowly reduce current to 10 mA, then slowly reduce voltage to 20 kV.
37. Push “OFF” in “X-RAYS” in the operation panel. The voltage and current become zero.
38. Wait few minutes before push “OFF” in “POWER”. Cooling water flow will stop automatically.
39. Stop PC if necessary.
40. Close valve of the He gas cylinder. If not much gas left, order new gas cylinder.
41. Keep vacuum ON more than 1H after X-ray stopped.
42. Stop vacuum. Push “STOP” button in “VACUUM” in the operation panel. If someone use this diffractometer soon (within a day), then keep vacuum ON.
43. Record your name, date, time used, IG, FC values, current He gas pressure, water temperature etc clearly in the log notebook. **These are necessary for a report to MEXT annually how much this instrument is used, and may help to replace it in near future. Those who belongs to other university, please write your institute name too (as I often have difficulty to identify).**
44. Don't forget to recover your sample and an USB memory.

Memo

1. “SHUTTER1” in the operation panel of XG is not used for current setting.
2. “He” gas is to protect focusing mirrors from oxidation and also to reduce absorption of X-ray by air. Latter is effective for Cu target.
3. After power shutdown, the instrument will beep. This is due to target switching unit, and beep can be stopped by turning ON vacuum pump.
4. For reflection measurement, in order to obtain lower angle data, you need to use smaller sample holder, and make ω angle as small possible (but not interfere with the collimator). Note that irradiated area will be elongated for this direction.
5. If XG can not be controlled from PC, check “SHT MODE 2” switch behind front panel (open front panel) is turned to “REMOTE”.
6. If abnormal temperature error occurred in water cooler unit, turn breaker off once.
7. If water level of the water cooler unit is lower than lowest level indicated (backside of the unit), add water or call Kanzaki.
8. Since beam is focused down to about 150 μm by mirrors, use of collimators of 300 and 800 μm does not result beam size of their nominal size.
9. If trouble persists, shutdown all units, and then turn on again. If problem can not be fixed, please contact to Kanzaki.

Check for current beam position (added 2017/01/03)

1. Beam position may displace from the center with time (after earthquake etc). In this part, I will describe how to check it by the users. Do “Cross-hair adjustment” first. Put it on sample
2. The sample holder with fluorescence disk is stored in the desiccator. Fix it on the manual XY stage. Focus on top of the fluorescence disk using the camera at max. magnification. Then, turn off sample fiber light and fluorescence tube. Close all shield windows

3. Open the front panel, and turn “SHT MODE 2” switch to “XG” side. Then, push OPEN button of SHUTTER2 in the display panel.
4. Watch the camera image, and you may see X-ray beam image (very faint). Note that the beam image is elongated in vertical direction (since the fluorescence disk is inclined). If the center of beam is shifted more than 20 μm , please report to Kanzaki.
5. Once finished, push CLOSE button of SHUTTER2, and then turn the switch of SHT MODE 2 to “REMOTE” side.

The jig (vertical pinhole jig with 30 μm hole on top) to be used for following adjustments is missing. If you took it, please return it. If you can not do the adjustment by yourself, please ask Kanzaki for the adjustment.

Cross-hair adjustment (quite often this adjustment is necessary)

1. When you are observing the sample using the CCD camera at maximum magnification, rotate ϕ axis. If the point of the sample placed under cross-hair walks off from the cross-hair during ϕ rotation, cross-hair adjustment is necessary.
2. If current collimator is 300 μm , then exchange to other (since 300 μm one is longer than others, and it interferes with the alignment jig).
3. Place vertical pinhole jig (with 30 μm hole on top), which is stored in a dry box, on the manual XY stage.
4. Move ω axis to 90°. Observe the top surface of jig by the CCD camera, and move the hole under cross-hair at maximum magnification. Focus at top of the hole.
5. Rotate ϕ axis (e.g., 0 to 180° or back). During the ϕ axis rotation, find the point which is not moving (i.e., the center of ϕ axis rotation).
6. Move the cross-hair to the center of rotation by choosing [Move Cross (Mouse)] from [Cross] menu in the CCD capture window, and then click mouse at the center of rotation. The cross-hair moves to the clicked point. If the point of rotation is outside of the view area, please contact Kanzaki.
7. Move the center of the hole under the cross-hair by XY stage. Move ω to 30°. If the hole moves a bit, set it back to under the cross-hair using sample Z-axis. Then cross point of ω axis and ϕ axis is defined by the position of the cross-hair and the position of focus. If the hole defocus and move during ω rotation, focus adjustment of CCD camera is necessary, and see below or please contact Kanzaki.

Focus adjustment of CCD camera

1. This adjustment is necessary when defocusing happens when you rotate ω axis. For this adjustment, moving stage at the base of the CCD camera unit is adjusted.
2. Firstly, adjust cross-hair position (see previous part). ω axis should be 90°.

3. Place vertical pinhole jig (with 30 micron hole on top), stored in dry box, on the manual XY stage. Observe hole at maximum magnification. Move center of the hole to the cross-hair position. Focus to top of the hole.
4. Loose lock screw of focus adjustment for the CCD camera.
5. Rotate ω axis to 30° . The hole will move and defocus during the rotation. Using focus adjustment of camera, move back a bit.
6. Rotate back to 90° . Using sample Z-axis, focus it, and move the hole to the cross-hair by XY stage. ¥
7. Repeat 5 and 6, until the hole does not move, and focus is OK.
8. Tighten the lock screw.