

Manual for SmartLab powder diffractometer

Quick manual for “Quick Theta/2-Theta Measurement Package”. For details or sophisticated measurements, see the original instruction manuals or References. Or try help menu of SmartLab “Guidance” software. In these manuals, new incident monochrometer is not described. For information of this monochrometer, please ask Kanzaki.

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Features of SmartLab diffractometer (Rigaku Co.)

Cu tube (40kV, 30mA)

Ge 111 incident monochrometer ($K\alpha_1$)

θ - θ type goniometer (no rotation for sample)

para-focus (Bragg-Brentano) (default) or parallel beam geometry are available

1D detector (D/teX) for rapid data collection (scintillation detector is also available)

Please clean the instrument, the tables, and the room when you use. Current condition is worst ever. If this continues, I have to force severe restrictions for all users.

Warning: Put on film badge before use!

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

Procedure

Startup

Normally following three steps are not necessary. If power is not coming, try these steps.

1. At the back side of the machine, turn the circuit breaker to ON position.
2. Turn key switch (“HV Enable”) to horizontal position.
3. Push “I (POWER)” switch in the front panel, next to the key switch. “OPERARE” lamp will lit in red few seconds, and then turn to green. Sometimes the “Door Lock” button will flash and beep. In this case, push the “Door Lock” button once.

Normally start from here

4. Make sure “OPERATE” lamp (lower right of the main unit) is green.
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.
6. Start PC (if not started yet). For login, just press Enter key.
7. Double click [SmartLab Guidance] icon on the desktop, or launch it from [Start] menu.
8. Login as an user ‘eng’ for English menus to be displayed (no password so far). If you login as Administrator, Japanese menus will be displayed.
9. Initialization of the diffractometer will be executed. It will take few minutes. Wait until [Starting...] window goes away.
10. Press [Startup] button on the Package Measurements window (flow bar), to start aging. If there is no such window, select [Package Measurement] from Tasks menu. [Package Measurement] flow bar will appear.
11. The [Startup] window will pop up. Depending on how long passed after last use (check the log notebook), select appropriate one from [Generator usage] popup list. Set [XG set] as ‘Hold’. Click [Execute] button. The aging starts immediately. Cooling water unit will start to flow water.

For default, final set voltage and current will be 40 kV and 30 mA, respectively. The time necessary for aging will be displayed in [Hardware Control] window. Wait until the aging finish. Meantime, prepare sample. If last use was few months ago, the aging will take few hours. **Don't skip this aging!**

12. Lamp at the top of the main unit will be lit when X-ray generator is ON. When shutter is open, red lamp near the tube will be lit.

Sample exchange

13. Press [Door Lock] button on the left door. It starts to beep. "Close door" message will appear on the PC display, and disallow any operation on the PC.
14. Slide open the doors, and replace/put sample holder in sample stage.
15. Close door.
16. Press "Door lock" again. Beep will stop. The message window will disappear.

Measurement

17. From Package Measurements flow bar, select "Quick Theta/2-Theta Scan (Bragg-Brentano) D/teX (if the item is folded under [Phase ID and Structural Analysis from Powders], unfold it). The procedure list (called 'part' in Rigaku's manual) of the package will appear. Do not RUN or select part No. 1-3. Click [4. Quick Theta/2-Theta Measurement [BB]] part.
18. [Quick Theta/2-Theta Measurement [BB]] window will appear. Put file name of this measurement, and memo etc (unfortunately when you want to change file location, file dialog box is displayed in Japanese). If you are fine for [Run recommended sequence], check this radio button, and select [Sample] list, check radio buttons for step and speed. Go to step 20.
19. If you want to customize the conditions or to know what is recommended conditions, check [Customized Conditions] radio button, and click [Customize...] button. Here you can set slits and scan conditions. For recommended conditions, click [Set recommended values] button. Default value of incident slit (IS) is $2/3^\circ$ (corresponding 1° for standard diffractometer). A relation of IS and irradiated width at the sample is shown in the diagram (last page). For quantitative analysis, irradiated sample width should be smaller than the sample width at lowest 2theta angle you measure. 'IS L' defines irradiated length, and 10 mm is for standard sample holder (if you change this, you will be asked to replace this slit). RS1 is 4° and PSA is 'No_unit'. Standard Soller slits we use are 5° for both. Set RS2 to 13 mm, corresponding length of D/teX detector. Then set 'Measurement conditions'. This quite depends on your sample. In most case, 5 to 90° with step size of 0.02° is fine, but for sophisticated works, you should change these parameters. Depending on S/N ratio, change scanning speed. Attenuator should be 'Open'. If rotational stage is mounted, you can select phi rotation speed (the unit displayed here seems incorrect, it should be read as rpm instead).
20. Once all conditions are set, then click [Execute] button. Message window showing "Prepare for change ..." will pop up. For present quick measurement, you do not need to exchange anything.

So click [OK]. The measurement will start. **Please note that if you don't click OK button, the measurement will not start.**

21. The diffraction pattern will be displayed. By left mouse button click, you can expand the pattern etc. When you select slow scanning speed, it takes a while to start scanning (may take more than few minutes, because of nature of 1D detector. During the measurement, [Hardware Control] will be displayed. You can stop the measurement by clicking [Abort] button.
22. For analysis of your obtained data, use PDXL software, installed on the PC. You can run the program during the measurement for already acquired data. Same software is also available in the PC for micro-focus X-ray diffractometer.
23. 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 SmartLab guidance software.
24. If the room is hot or water temperature is high, decrease set temperature of air conditioner.

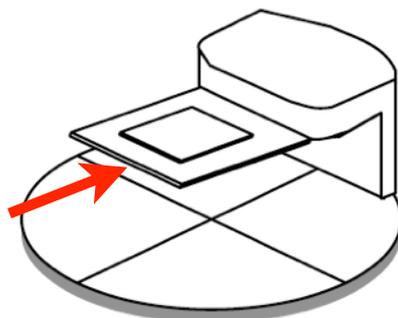
Shutdown

25. Press [Shutdown] button on the Task flow bar, to start shutdown procedure.
26. Select [XG Off] from list in [Shutdown] window, and press [Execute] button. Tube current and voltage will become zero. After few minutes later, the cooling water will stop. Meantime, take out sample.
27. **If the cooling water stopped, then you can quit SmartLab guidance software.** If necessary, copy data to your USB memory.
28. Shutdown PC if necessary.
29. (Not necessary) Turn off breaker switch of the cooling water unit.
30. Write log notebook (Name, date, how long used etc). For outside users, please write your institute name too. **We have to report how much and who used this instrument to MEXT annually, and it may help to replace it in near future.**
31. Don't forget to recover your sample and an USB memory.

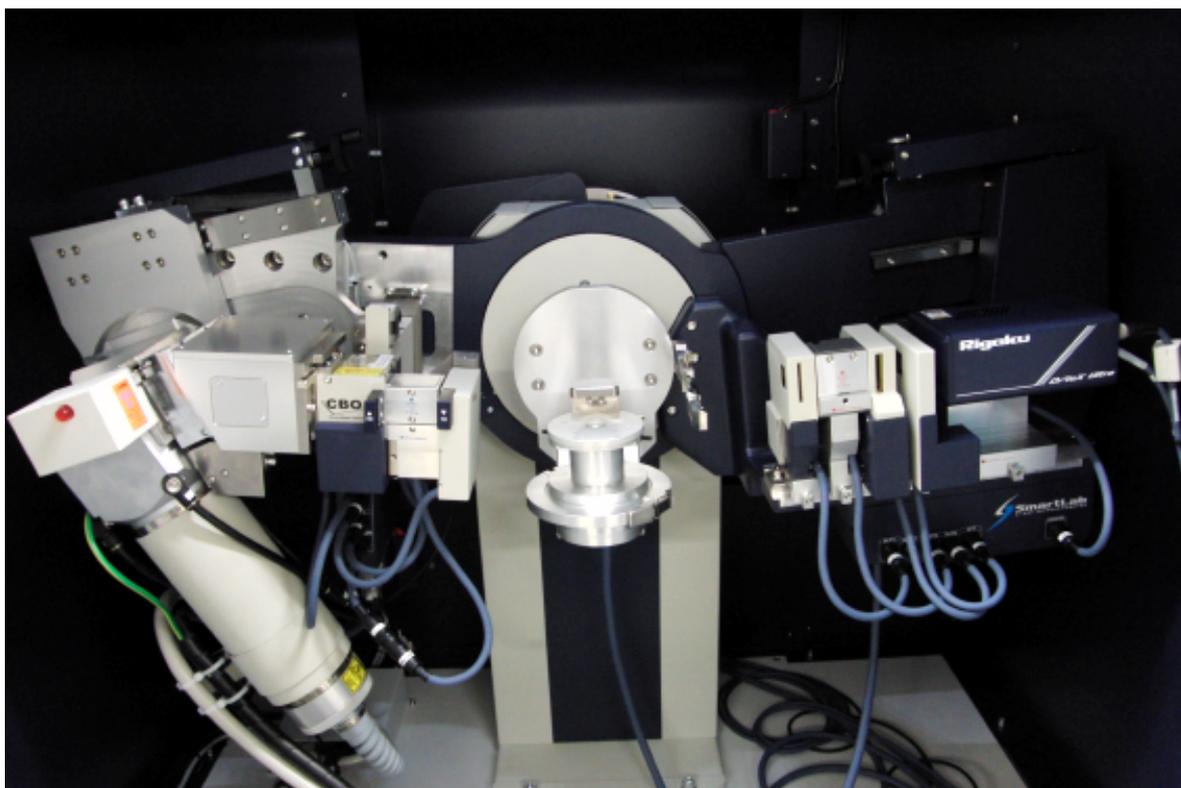
Notes

- 1) If hardware error reported during startup, turn off SmartLab's main power, and start from very initial part of this manual. If the error message still exists, please report to Kanzaki.
- 2) Do not apply $K\beta$ filter. $K\beta$ is already removed by incident monochrometer.
- 3) Because D/teX should be used with opened receiving slit, background at lower angle will be higher than ordinary diffractometers.
- 4) If your sample contains iron or other heavier elements which emit fluorescent X-ray, background of the pattern becomes high. One way to reduce fluorescent X-ray is to use Low Fluorescence mode of D/teX detector. From [Utility], select [D/teX Energy Resolution Adjustment]. Then select [XRF reduction]. You will be asked to insert standard Si sample. Please remember to return to [Standard] mode for other users.

5) Rotational stage is also available. Ask Kanzaki for instruction. You should use another sample holder designed for the rotational stage (the one shown below can not be used). You can use Rigaku's standard glass sample holder, but Si and quartz plates can not be used.



Standard sample stage. Insert glass sample holder in upper part of the stage.



SmartLab inside the cover. Left side is incident optics, central part is sample stage, right side is detector side optics. The box between the tube and CBO unit is Ge 111 monochromator. A box at right with Rigaku logo on the surface is D/teX Ultra detector.

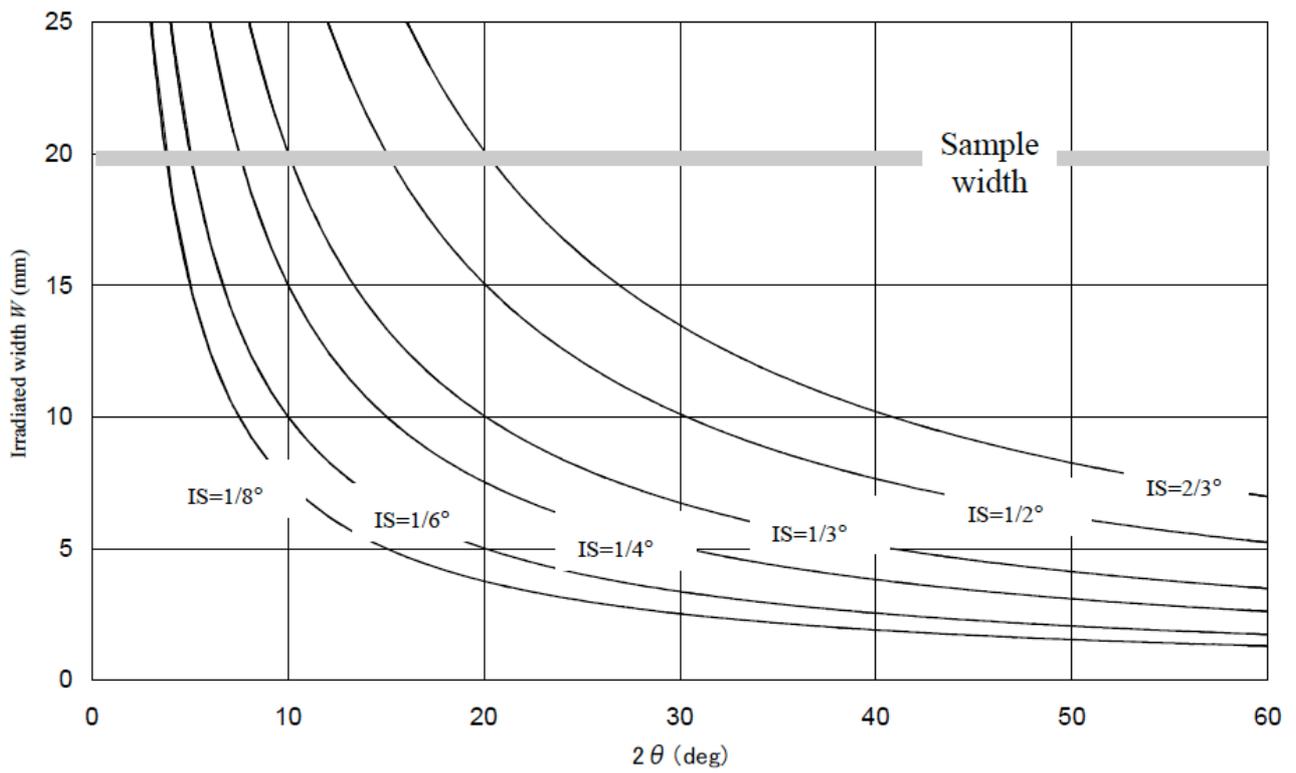


Fig. 4.2.6 Irradiated width of incident x-ray beam in para-focusing optics for goniometer radius of 300 mm

Make sure your beam width is always smaller than that of your sample using above diagram.