

Rigaku Ultima IV Powder Diffractometer Operation Summary

11/29/2012 A.E.

Location: 1709 Gilman Hall

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Safety

All users of Rigaku Ultima IV must complete the EH&S “X-ray Safety Fundamentals” course available on-line at www.ehs.iastate.edu. The valid “X-ray Safety Fundamentals” Certificate is mandatory to enter 1709 Gilman Hall X-ray lab.

This class demonstrates the correct procedures for the authorization and use of X-ray producing devices. Topics include X-ray production and hazards, hazard control measures, and regulatory requirements. Certification is awarded upon successful completion of the quiz at the end of the course. You must recertify every year.

All diffractometer enclosures in CIF X-Ray Laboratory are checked by EH&S annually and certified for data collection operations. All systems are also equipped with multiple interlock switches blocking the X-ray shutter to avoid any possible operator exposure.

Training Requirements

Before operating diffractometer, you **MUST** be properly trained and authorized by CIF Supervisor of the X-ray lab!

This training manual includes information about the diffractometer and step-by-step instructions on sample preparation and data collection.

All referenced documents, including the detailed “Instruction Manual” and the “User’s Manual” are available in 1709 Gilman Hall.

I. Starting Up

1. Log into computer.

2. Double click icon XG Operation  to open XG Operation RINT 2200 window.



3. Increase tube voltage and tube current by 10 then hit the “set” button. Continue increasing the voltage and tube current in this manner until a voltage of **40 kV** and a current of **44 mA** is reached. (*note: the first setting should be 30 kV and 14 mA to avoid having to increase the amperage by 2 mA at the end*)
4. Close the XG Operation window by selecting **File → Quit**.

II. Preparing your sample

1. Grind sample to a fine powder in 1710 Gilman
2. Place sample in the ground glass depression in the sample holder.
3. Flatten sample using a glass slide

NOTE!!! This is a very sensitive instrument and sample preparation makes a big difference in the quality of your observed diffraction pattern. A good sample is smooth and well packed; if there are ridges the pattern quality degrades considerably. Small sample sizes can be accommodated by only filling portion of the holder, ask and I'll show you how.

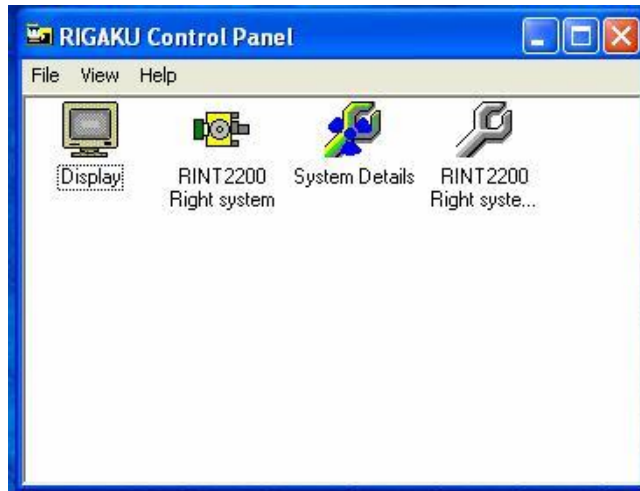
Inside the Cabinet

4. Press door lock button and insert sample into instrument. [3.1.2;14] The sample is inserted by sliding the holder into the clip assembly at the center of the goniometer.

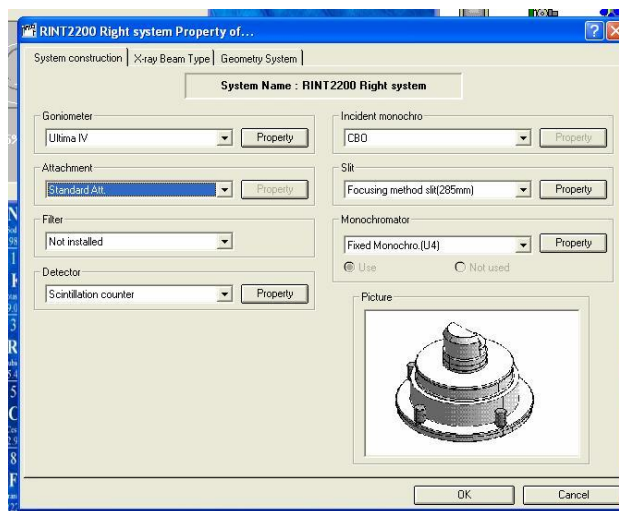
1. For powder samples, the following items must be in place:
 - a. Cross-Beam Optic slit, "BB" for standard powder measurements
 - b. Divergent Height Limiting (DHL) slit
 - c. Monochromator should use "Bent" mirror setting
5. Close cabinet doors and press door lock button

III. Running your scan

1. Open the Rigaku Control Panel by double-clicking on the icon

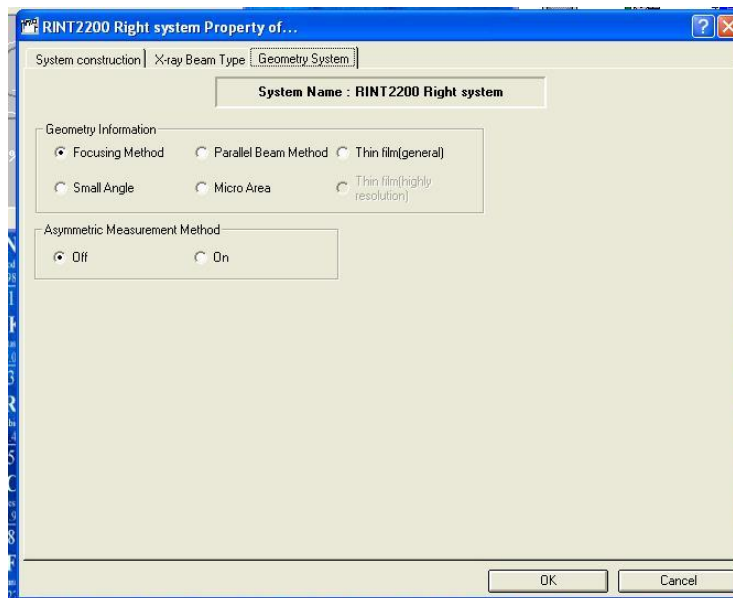




2. Start RINT 2200 by double clicking the icon in the Control Panel window.

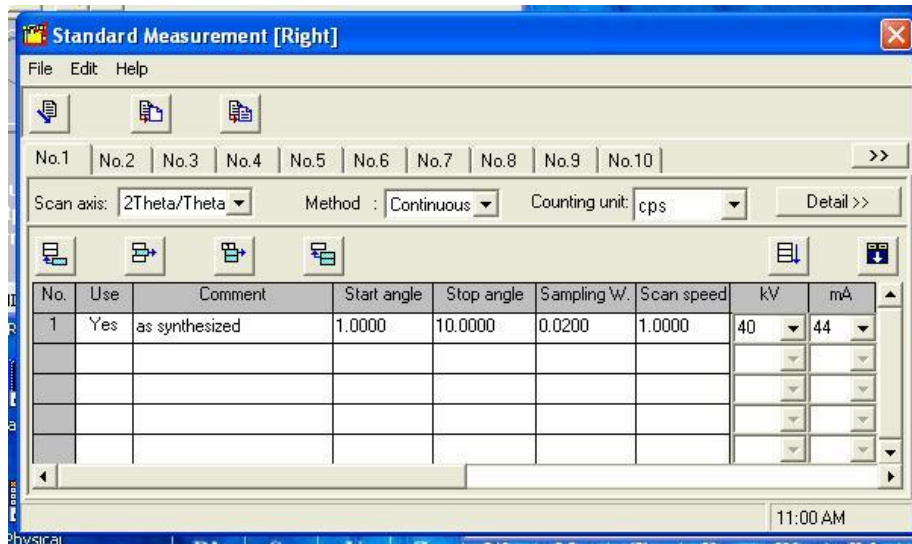


3. Fill in the following fields accordingly:
 - a. Goniometer – Ultima IV
 - b. Attachment – standard
 - c. Filter – not installed
 - d. Detector – Scintillation counter
 - e. Incident Monochromator - CBO
 - f. Slit – Focusing method (285mm)
 - g. Monochromator – fixed monochromator (U4)

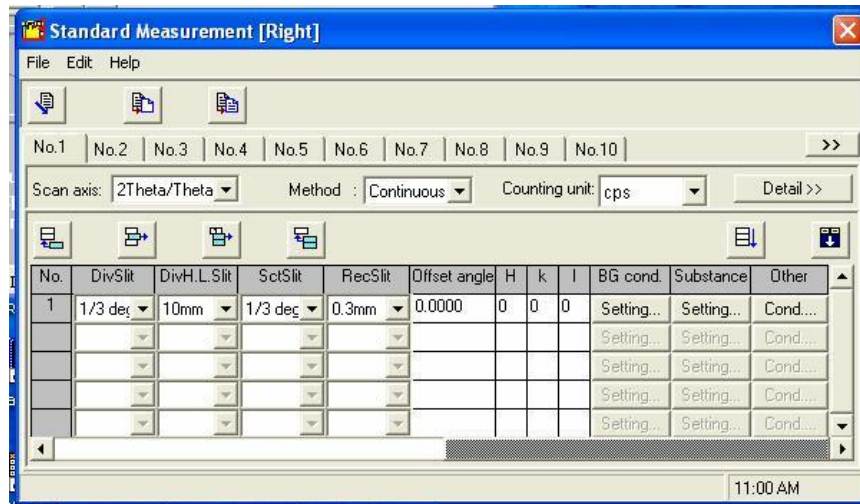
4. Select the “Geometry System” tab and ensure the following fields are checked:
 - a. Geometry Information – Focusing Method
 - b. Assymmetric Measurement Method - Off



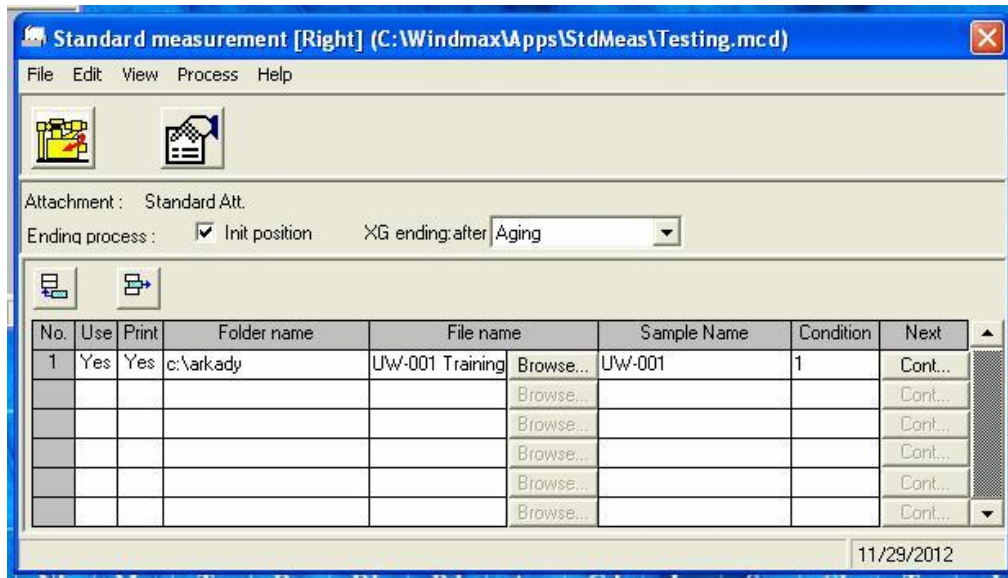
5. Click the “OK” button and select “yes” when asked if you would like to save the configuration.
6. **See last page for standard measurement conditions.** Open the “Standard Measurements” program by double-clicking the desktop icon  .
7. Click the “Measurement Condition” icon  . This opens the Standard Measurement conditions window.



8. Click on the first field “use” and select yes. Press tab to cycle to the next field and proceed to enter the relevant data into the fields. For a complete explanation of all fields please see the manual!
9. Click on the horizontal scroll bar to access the next window.




10. Enter the relevant data into the fields.
11. Ensure that 2theta/theta is selected. Close the Standard Measurement Conditions window, no saving is necessary.




12. Edit the “Folder Name” and “File Name” to set up the file for outputs. After that Check the box “Init position” to have the Goniometer arms return to their standard position ($10^\circ 2\theta$)

13. The dialog box following “XG ending: after” should contain the settings:

- a. “Present Condition” – If and only if you are running another scan or you are sure someone is using the instrument after you.
- b. “Aging” – if and only if someone is going to be using the instrument sometime later the same day, but not within 30 minutes of your last scan.

14. Before clicking the “Execute measurement” box , please ensure that the sample is in place and the slit marked “BB” is in the CBO module and the “DHL” slit is in place and matches the settings you entered into the “Measurement condition” window.

15. Click “Execute measurement” . If you have changed the value of the DHL slit, the program will ask you to insert “X mm DHL”. This should already be done, click “Ok” when ready. The scan should begin.

16. When your scan is complete press the “door lock” button, open the doors and remove your sample. Close the doors and press the “door lock” button to silence the alarm.

- a. **Clean your sample holder and ANY mess you may have left behind**

IV. Processing Your Diffraction Pattern

1. Start the MDI Jade 9 program by double-clicking the desktop icon.
2. Click the “Close” button in the “Job Selection” window.
3. Click “File” → “Pattern”. Select your folder and ensure the program is looking for “RINT – 2000 Binary Pattern Files (*.raw)”
4. Your file is saved as “xxxx.raw” where “xxxx” is the file name entered in the Standard Measurement main window.

5. Once your file is detected it can also be opened from the upper left file window.
Processing may now begin
Note!!! Diffraction patterns may also be processed using the software described in manual # ME13435A05 Part 4. Basic Data Processing.
6. If you wish to make a copy of your file that can be read in standard spreadsheet software you can use the “Binary – ASCII Conversion” utility.
7. Click the “Input File Name...” button and select the directory which contains your files and then the file you wish to convert. You should now see your file name in the dialog box.
8. Click the “Output File Name...” button and select the directory and input the name of the converted file. This information should also now appear in the dialog box.
9. Under the “Conversion Conditions” heading the following commands should be used for files that will be processed using Microsoft Excel:
 - a. Field Separator – TAB (**<TAB>**)
 - b. Divided data – Vertical line output
 - c. Separator – Not use
10. You can then also select the additional data you would like exported with your diffraction pattern.
11. Alternatively if you would like to export a processed pattern from MDI Jade 9 select “File” → “Save in XML” and select the directory and file name to which you would like to send your processed, spread-sheet readable pattern.
12. When you are finished with data processing “log off” of the computer, do not “Switch User”.

NOTE!!! Using the Switch User option will cause the diffractometer to try to be controlled by multiple incidences of the same software, this will cause errors.

V. Important Information for Happy Data Collection

1. Do not scan below 0.5° for a standard powder measurement.
2. Use the DHL 5 mm slit for all scans starting below 1° .
3. **Read the manuals**, relevant sections are reproduced for this SOP; however the reproductions do not provide data on all aspects of the software. **Read the manuals.**
4. For training to acquire data from thin films or SAX please contact ellern@iastate.edu

Appendix I. Standard Measurement Conditions

Scans from $1 - 10^\circ$ 2theta (MSN)

DHL of 10mm, slit sizes $1/3$, 10 mm , $1/3$, 0.3 mm
scan speed of $1 - 2^\circ/\text{min}$

Scans from $0.75 - 10^\circ$ 2theta (LP-MSN)

DHL of 5mm, slit sizes $1/3$, 5 mm , $1/3$, 0.3 mm

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