Rigaku XRD-System Instruction Manual v4/19/03

The Krishnan Group/Wilcox 132, University of Washington

Contents:

Seattle 3/3/03 Peter Blomqvist

General information

The XRD-system in Wilcox 132 is a Rigaku rotating anode high brilliance (RU-200BH) X-ray diffractometer (Serial# L17171). It generates high intensity X-rays with a wavelength of 1.54 A (Cu-anode) or 0.71 A (Mo-anode). The maximum power is 12 kW (60 kV and 200 mA). ***At all times safety is paramount in using this instrument.***

The XRD-system was installed in Wilcox Hall 132 on February 25-26th , 2003. The instrument is owned by the University of Washington, Seattle and installed in the laboratory of Professor Kannan M. Krishnan (Office: 323 Roberts Hall, Tel.: 206-543-2814; Fax: 206-543-3100; Email: kannanmk@u.washington.edu) for use by members of his research group (http://depts.washington.edu/kkgroup/). Other qualified users may have access to the instrument provided they are approved by Prof. Krishnan. This requires appropriate safety training and a demonstrated ability to operate the instrument independently. Training and certification for use will be administered by members of the Prof. Krishnan's research group. For all such Users, a standard rate of $80/hr will be charged for the use of the instrument and their advisors/supervisors bear responsibility for any damage caused by them to the instrument. The use of the XRD system is restricted to normal working hours (M-F, 9:00-5:00). Weekend use is possible with advance permission and the guaranteed presence of someone else in the laboratory, along with the user, at all times.

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Safety

The XRD-system is a very expensive and advanced scientific instrument and it generates X-rays with high intensity. Careless use can result in personal injury and damage to the instrument. The user is therefore expected to follow the safety rules and guidelines in this manual.

If you have a general or a specific question regarding the operation of the XRD-system do not hesitate to contact any of the contact persons.

The XRD-system has been radiation tested by the office of Environmental Health and Safety (EH&S) at the University of Washington. *No radiation was detected outside or inside the radiation enclosure with X-ray source on and shutter open (50 kV, 200 mA)!* The radiation tests were performed on February 27th 2003 by Ms. Molly McGee, Health Physicist, Radiation Safety Environmental and Health, University of Washington. If further details about these radiation tests are required she may be contacted at Hall Health Center, Box 354400, Seattle, WA 98195-4400
Tel.: 206-685-5311 Fax: 206-543-3351
Email: mkmcgee@u.washington.edu Web: www.ehs.washington.edu

The following access rules must be followed at all times

- Only certified Users are allowed to operate the XRD-system independently.
- Other persons requiring some simple measurements must be accompanied by an authorized User at all times.
- Only authorized Users are allowed to deactivate the radiation protection safety system.
- Only an authorized User is allowed to change any hardware or software settings.
- The procedures to turn the X-ray source on and turn off (outlined below) must be followed.

- Users are not authorized to carry out any major alignments. This is the exclusive privilege of the SuperUsers. Whilst carrying out alignments, ring badges MUST be worn.
- Any major modifications, repairs or maintenance of the instrumentation will be scheduled by the SuperUsers in consultation with Prof. Krishnan.

The XRD-system has a built-in safety system

- The main power to the XRD-system is shut off if the metal safety panels surrounding the power source are removed. This is to protect the user from electric shock.
- The XRD-system will not operate without sufficient water cooling. The anode (target), tube tower (housing) and the turbo pump (TMP) need to be cooled during operation. A red warning light (CW) on the front panel will come on if the water flow is too low, if the system is on it will automatically be turned off.
- The X-rays can not be turned on if the anode is not rotating. The X-rays will automatically be turned off if the anode for some reason stops rotating to prevent it from overheating.
- The radiation enclosure and the sample chamber are to protect the user from exposure to X-ray radiation. If the X-rays are on, *indicated by the red warning light inside the radiation enclosure and a red warning light on the front panel*, and the radiation enclosure is opened the X-ray source will automatically be turned off, no matter if the shutter is open or closed. In order to open the shutter the lid must be on the sample chamber. There is a red warning light on the tube tower when the shutter is open
- **Important:** the radiation protection safety system can be deactivated by turning the fail-safe (FS) key. *Only authorized persons are allowed to do that!* When the safety system is deactivated a red flashing light on the front panel will come on and you will hear a beeping sound.

How to turn on the X-rays

- Make sure that the XRD-system has enough cooling water. If the water flow is too low the red warning light (CW) on the front panel will come on. The flow rates should be above the black markings on the flow meters on the water chiller. The temperature of the cooling water should be roughly 70 degrees Fahrenheit.
- **Check for water leaks. Pay particular attention to the areas above and around the anode. The X-ray source should NOT be turned on if there is a water leak!**
- Make sure that the vacuum pumps are on. Both the green RUN light on the TMP DRIVE UNIT and the green OPERATE light on the VACUUM CONTROLLER unit should be on.
- Check the pressure inside the tube tower. The X-rays can only be turned on if the pressure inside the tube tower is 10^{-4} Pa (two green lights on the VACUUM CONTROLLER unit) or less (three green lights). *If there are only two green lights do NOT continue!*
- Make sure that the lid on top of the sample chamber is on.
- Make sure that the radiation enclosure is closed.
- Turn on the anode rotation by pressing the T-REV button on the XG CONTROLLER.
- If everything is ok the orange READY light should come on.
- Turn the TUBE VOLTAGE and the TUBE CURRENT knobs to 20 kV and 10 mA, respectively.
- Turn on the X-rays by pressing the ON button on the XG CONTROLLER. The red warning lights inside the radiation enclosure and on the front panel should come on.
- Wait 5 minutes for the voltage and current to stabilize. Use a voltmeter while you are waiting to check the pressure inside the tube tower, the voltmeter should read roughly 50 mV*.

- ***First*** increase the ***tube voltage*** in steps of 10 kV to 50 kV, wait 10 seconds between each step. Monitor the pressure inside the tube tower as you increase the voltage.
- ***Then*** increase the ***tube current*** step in steps of 10 mA to desired value, wait 10 seconds between each step. Higher current gives higher intensity. Maximum tube current is 200 mA. Recommended tube current is 100-200 mA. Monitor the pressure inside the tube tower as you increase the current. *If the voltmeter reads more than 200 mV do NOT continue!*
- **Important:** If you open the radiation enclosure while the X-rays are on (shutter can be either open or closed) the safety system will automatically turn off the X-ray source. *This may damage the X-ray source*. To access the goniometer you must turn the FS (fail-safe) RELEASE key. When the safety system is deactivated a red flashing light on the front panel will come on and you will hear a beeping sound. The red light and the sound will disappear when the radiation enclosure is closed again.
- You are now ready to load your sample and start your measurement.

*The actual value depends on the X-ray source: whether it has been aged or not and the condition of the vacuum seal.

How to turn off the X-rays

- ***First*** decrease the ***tube current*** in steps of 10 mA to 10 mA, wait 10 seconds between each step.
- ***Then*** decrease the ***tube voltage*** in steps of 10 kV to 20 kV, wait 10 seconds between each step.
- Wait 10 minutes for the anode and cathode to cool down.
- Turn off the X-rays by pressing the red OFF button. The red warning lights inside the radiation enclosure and on the front panel should go off.
- Remove sample and do not forget to fill out the logbook.
- Remove the fail-safe release key and make sure that everything is in order.

General guidelines on how to use the XRD-system

X-ray source:

The cathode (filament) has a lifetime of about 2000 hours at 50 kV and 200 mA. Reducing the tube current to 100 mA increases the lifetime by 50%. The lifetime of the cathode is also dependent on the number of times the X-ray source is turned on and off. Turning the XRD-system on and off stresses the filament as well as other components. It is therefore strongly recommended to keep the XRD-system on as much as possible and only reduce the power to 20 kV and 10 mA (stand-by settings) when it is not used. Other components that are likely to fail and need to be replaced are the two cooling water seals (about 1000 hours) inside the rotating anode. *See the manual for the lifetime of the different components.*

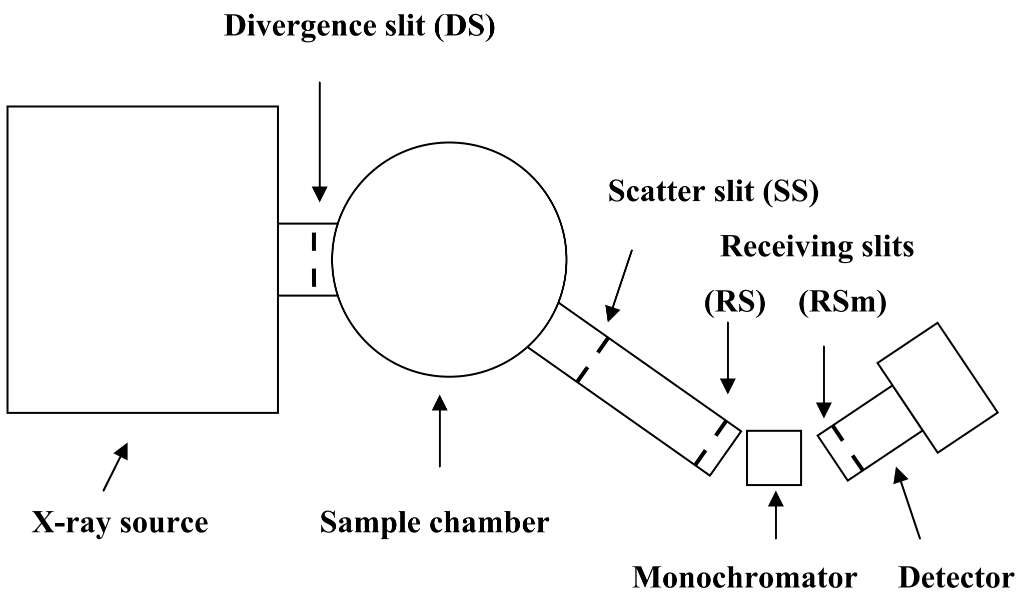
Goniometer:

The goniometer has four different slit positions: the divergence slit (DS) position, the scatter slit (SS), the receiving slit (RS) and the receiving slit at the monochromator (RSm). Each slit is marked with its size in degrees (º) or millimeters (mm). The divergence and the scatter slit are marked with DS and SS, respectively, whereas the receiving slit and the receiving slit at the monochromator are not marked. The slits should be placed in the goniometer with the edge pointing away from you. The scattering geometry used in this X-ray diffractometer is a so called focusing geometry which means that the widths of the divergence and scatter slit should be equal, i.e. if you use a 1/2° DS then you should use a 1/2° SS and so forth. The same general rule applies to the receiving slit and the receiving slit at the monochromator (see figure on next page).

"What slit size should I use?"

- Wide slits give higher intensity but lower resolution.
- Narrow slits give higher resolution but lower intensity.
- Use narrow slits if your sample is very small.

- The irradiated area is proportional to 1/sinθ. For a sample area of width 20 mm the following guidelines apply: the 1/2˚ DS can be used down to a 2θ angle of 10 degrees, the 1˚ DS to 20 degrees and the 2˚ DS to 30 degrees.
- Try different slits and see what work best for your sample.



The sample height is very important when a focusing geometry is used. The correct sample height is given by the vertical flat surface inside the sample chamber.

- If the sample is mounted too low the diffraction peaks will shift towards lower 2θ angles.
- If the sample is mounted too high the diffraction peaks will shift towards higher 2θ angles.

Software:

DataScan v3.2 from MDI is used to control the goniometer. The following different scans are available:

- Coupled 2θ/θ -scan => Powder samples and thin films
- Uncoupled 2θ/θ -scan => Thin films

- θ-scan (rocking curve) => Thin films (and powder samples)
- 2θ-scan (detector scan) => Thin films (and powder samples)

"So what type of scan should I use?"

It depends on what kind of sample (powder sample, thin film, polycrystalline, single-crystalline, textured etc.) you have and what kind of information you are interested in. Contact Peter Blomqvist if you are not sure of which type of scan to use.

There are two different scan modes: continuous and step scan.

- In a <u>continuous</u> scan data is collected continuously as the angular position is increased. The scan speed (degrees/min) and the step size (in degrees) can not be chosen independently, the step size should be of the order of the scan speed /250. Furthermore, the step size should be a multiple of the minimum step size (0.002 degrees), i.e. 0.002, 0.004, 0.01, 0.2 ……. *In a continuous scan the intensity will be given in counts per second.*
- In a <u>step</u> scan the goniometer moves to an angular position and stays there while data is collected. The step size should also in this case be chosen as a multiple of the minimum step size. *In a step scan the intensity will be given in counts.*

The continuous scan is faster while the step scan is more accurate.

File name convention:

For each scan you need to enter a file ID (name of the measured data file) and a sample ID. What you write as sample ID is up to you but the file ID name must consist of eight characters: the two first should be your initials, the next three characters should be the sample name/identification number and the last three should be the scan type/number, see example below.

DD003HA5

It is now easy to see that Donald Duck is the user, the sample is number 003 and this is high-angle (HA) number 5.

Remember:

- Smaller step size gives better angular resolution.
- Longer time per step (dwell time) gives better statistics (less noisy data).
- Measuring time = time per step * (start angle – end angle) / step size
- Counts per second = counts/time per step.
- The scan speed and step size that one should use depend on sample size and the crystalline quality of the sample. Generally one can say that a small sample of poor crystalline quality takes longer time to measure than a large sample of good crystalline quality.

A good idea:

If you have a sample that you have not measured on before it is strongly recommended to first make a quick scan with large step size (0.1 or 0.2 degrees) and a short time per step (0.5-2 seconds per step) over a wide angular range to see where the peaks are and how intense they are. Then you can adjust the slits if it is necessary and make more precise measurements. It will help you to get good data and save you a lot of time.

Comparing data from different samples:

- Use the same angular range (x-axis).
- The intensity (y-axis) should be in counts per second, not counts.
- Use the same type of intensity scale (y-axis), i.e. linear or logarithmic*.
- Remember that the measured intensity (in counts per second) depends on sample size, beam intensity (tube current) and the slits used.

*The DataScan program uses a linear intensity scale.