STANDARD OPERATING PROCEDURES

for the

Rigaku RU300 Rotating Anode X-Ray Generator
185mm Radius Diffractometer

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These instructions describe the proper operation of the Rigaku RU300 Rotating Anode X-ray generator and the MDI DataScan data collection software. The diffractometer attached to the generator is a Rigaku D/Max-b 185mm radius goniometer with diffracted-beam monochromator and scintillation point detector.

If there is any problem with the generator or diffractometer, you are required to report it to Josh Guske immediately (13-3090, x3-0180, jtguske@mit.edu). If the problem poses a safety hazard, turn off the generator (even if a scan is in progress) and post a note on the enclosure (so that no one else tries to use the instrument). Remember, you are not just responsible for your own safety; you are also responsible for the safety of those around you.

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Operating Instructions: Abridged Checklist for Experienced Users

More detailed instructions start on page 5; if you are unsure about the proper way to do something, consult those instructions or ask SEF staff (Josh Guske) for help.

1. Engage Coral to start Logging Time

2. Assess the Instrument Status and Safety
   a) Is the Generator on?
   b) Is the Shutter open?
   c) If the generator is OFF, go to step 3.
   d) If the generator is ON and the shutter is closed, go to step 2.

3. If the generator is ON, then turn it OFF
   a) Check that no scan is running and that the shutter is closed.
   b) Slowly turn down the current (mA)
   c) Slowly turn down the voltage (kV)
   d) Press the red OFF button on the generator control panel.

4. Set up the Diffractometer
   a) Check the slits (DS, SS, RS, and RSM)
   b) Insert the sample and place the sample chamber cap above the sample holder.
   c) Close the Enclosure Doors

5. Turn On the X-Ray Generator
   a) Press the white T-REV button.
      • The amber READY light on the generator control panel should light up brightly
      • If the READY light does not light up, or lights up only dimly, then check:
        o that all enclosure doors are properly closed.
        o that the voltage and current dials are turned fully counterclockwise to their minimum positions.
      • If correcting these does not cause the amber ready light to light up, contact Josh immediately.
   b) Press the white ON button.
   c) If the CW light in the Alarm section illuminates, then:
      • Press the RESET button
        • Start over with step a
      • If any other alarm light is lit, do not attempt to operate the instrument- contact Josh
   d) Turn up the generator power.
      a. Use only the coarse adjust dials for kV and mA.
      b. First turn up the kV slowly to 50 kV, waiting for the gauge to settle between each detent.
      c. Then turn up the mA slowly to 200 mA, pausing between each detent (i.e. click).

REMEMBER:
- The shutter has a pair of “Shutter Open” Warning Lights.
- Never open the doors while the Generator is ON.
5. Using the Data Collection Software
   a) Start MDI DataScan4
   b) Open Control > Rigaku D/Max-b to initialize the diffractometer
      • In the Goniom tab, set the “2Theta” and “Theta” offsets and click the Datum button.
      • In the PHA tab, set the “Base Line”, “Window”, and “Detector HV” values and then click all 3 Set buttons.
   c) Set up the Scan Parameters in Scan > Routine Scan
   d) Start the Scan

   You should not loiter in the XRD lab while your data is being collected.
   However, you must be present when your run finishes so that you can clean-up your sample and turn off the instrument.

6. Turn OFF the Generator when you are Done Collecting Data
   a) You must turn off the generator when you are done collecting data.
   b) First, slowly turn the current all the way down to 10 mA, pausing between each detent
   c) Then, slowly turn the voltage all the way down to 20 kV, pausing between each detent
   d) Press the generator OFF button
   e) Remove your sample from the diffractometer
   f) Close the enclosure doors.

7. Disengage Coral to Stop Logging Time
Operating Instructions: Thorough & Illustrated

I. ENGAGE the Rigaku_Cr_Source in CORAL

II. PRELIMINARY CHECKS

1) Determine whether the X-ray generator is ON or OFF
   a) This is indicated by the red warning light hanging above the tube tower and the red warning light on the generator control panel (labeled X-RAY).

2) If the X-rays are NOT ON, proceed to Section II “Setting up the Diffractometer”

3) If the X-rays are ON
   a) Check if the shutter is open. This is indicated by:
      • The red warning lights on the side of the tube tower
      • The red warning lights on the shutter control panel.
      • Only Shutter 1 is used on this instrument
      • The X-ray safety shutters can open only when the X-ray generator is on.
   b) If the shutter is open, check DataScan (pg 14) to see if a run is in progress.
      • If a run is in progress, wait until it finishes or manually stop the run.
      The computer will close the shutter.
      • If no run is in progress and the shutter is open, do not attempt to operate the instrument- the shutter might be stuck. Turn the generator off and notify Josh immediately.
   c) If the shutter is NOT open, then turn off the generator
      i) First, slowly turn down the current (mA)
         • Turn the coarse adjust dial one ‘click’, pause, then turn it again.
         • Turn the coarse adjust knob all the way counterclockwise to 10 mA
      ii) Then, slowly turn down the voltage (kV)
         • Turn the coarse adjust dial one ‘click’, pause until the needle stops moving, then turn it again.
         • Turn the coarse adjust knob all the way counterclockwise to 20 kV
      iii) Press the red OFF button on the generator control panel.

PLEASE NOTE:
1. You cannot open the enclosure doors while the generator is on. Opening the doors will automatically cause the generator to shut off.
2. Never operate the shutters from the switches on the Generator Control Panel; the computer will automatically open and close the shutter when collecting data.
3. The shutter and the X-ray generator each has two warning lights indicating open/on. For a pair of warning lights, if you ever observe that one warning light is on and the other warning light is off, turn off the generator (even if a run is in progress) and contact Josh immediately.
II. SETTING UP THE DIFFRACTOMETER

1) Check the slits
   a) Use Appendix A (pg 16-18) to help you decide which slits to use.
   b) The divergence slit (labeled DS) is usually ½ or 1 deg.
      i) The DS is covered by an anti-scatter cap that you must remove.
      (1) Be sure to replace the anti-scatter cap after checking/changing the DS.
      ii) The flat side of the DS faces the X-ray tube when inserted.
   c) The scatter slit (labeled SS) is usually twice the size as the DS (1 or 2 deg).
      i) The flat side of the SS faces away from the sample when inserted.
   d) The receiving slit (labeled RS) is usually 0.3mm.
      i) The flat side of the RS faces towards the sample when inserted.
   e) The monochromator receiving slit (labeled RSM) is usually 0.8mm.
      i) Use a smaller RSM to remove more K-beta intensity (eg single crystal substrate)
      ii) The flat side of the RSM faces the detector when inserted.

2) Insert the sample.
   a) Position the sample holder so that the sample is in the top half and facing you.
   b) After inserting the sample, check that it is snug up against the alignment post.
      i) Make sure that both springs are behind the sample holder.
      ii) If the sample holder is loose, the measured angles may not be correct.
   c) Place the sample chamber cap on the scatter shield above the sample.
      i) Make sure that the microswitch is depressed by the flat tab on the cover.
      ii) The enclosure doors will not register as closed if the sample chamber cap is not in place.

3) Close the enclosure doors.
   a) Line up the white metal plates on the doors with the red dots on the enclosure frame.
   b) Make sure that the right side door is slid all of the way to the right- it sometimes sticks on the enclosure frame.
III. TURNING ON THE X-RAY GENERATOR

1) **Press the white T-REV button.**
   a) The belt drive should begin spinning the anode.
   b) The amber READY light on the generator control panel should light up brightly.
   i) If the READY light does not illuminate, see the troubleshooting section below.

2) **Press the white ON button.**
   a) Wait for the voltage meter to settle at 20kV and the current meter to settle at 10mA.
   b) If the CW alarm light turns on, then:
      i) Press the RESET button
      ii) Press the white ON button again
   c) Then proceed to the next page (pg 9) for instructions on turning up the power

**TROUBLESHOOTING**

When you push the T-Rev button, if the READY light does not light up, or lights up only dimly, then:

- Check that all enclosure doors are properly closed.
  - Check that the voltage and current dials are turned fully counterclockwise to their minimum positions.
    - The Voltage and Current each has 2 dials: a coarse adjust dial and a fine adjust dial. Both must be turned fully counterclockwise to their minimum positions.
  - Check if any ALARM lights on the Generator Control Panel are lit.
    - If the CW alarm light is lit, then press the RESET button.
    - If any other alarm light is lit, do not try to operate the instrument. Press the OFF button and contact Josh immediately.
  - Check the status of the vacuum. The three green lights on the vacuum controller, labeled 10\(^3\), 10\(^4\), and 10\(^5\) should be lit.
    - If only one or two lights are on, do not turn on the generator. Report this to Josh immediately.
  - If none these steps cause the amber READY light to light up, then there may be a serious problem. Contact Josh for help.
3) **Turn up the generator power.**
   a) First slowly turn up the kV to **50 kV**.
      i) Turn the coarse adjust dial one ‘click’ at a time, waiting for the meter to settle at the new kV before turning the dial to the next detent (i.e. click).
         1) Use only the coarse adjust dial.
   b) Then slowly turn up the mA to **200 mA**. Pause between each detent (i.e. click).
      1) Use only the coarse adjust dial
         2) As you approach 200 mA, then needle will not track accurately—it will read slightly below the set current. You should look at the scale on the mA knob to make sure that you do not turn it above 200 mA.
         3) If you do turn the current above 200 mA, the generator will shut off and the **OL** light in the Alarm Indicators will light up (see figure on bottom of previous page). If this happens, then:
            a) Press the RESET button below the Alarm Indicators
            b) Turn the kV and mA knobs all the way counterclockwise to their lowest settings.
            c) Wait 1 minute.
            d) Turn the generator on again, proceeding from step 1 on page 8.

**PLEASE NOTE:**
- Do not turn the voltage and current up to quickly—this will damage the filament.
- If any of the alarm indicator lights other than CW or OL on the Generator Control Panel illuminate, accompanied by the generator shutting off, contact Josh immediately. Do not continue to attempt to operate the instrument.
V. USING THE DATA COLLECTION SOFTWARE

Data from the Rigaku diffractometer is collected with MDI DataScan v4. This program follows most of the conventions for Windows-based programs. Conventions used in this guide:

- Menu items are printed in *italics*, for example: *File, Edit*, etc.
- Nested menus and selections within menus are indicated by >, for example *File>* *Open*.
- Window titles (shown in the blue title bar of each window) are written in **bold italics**.
- Names of tabs are **underlined**.
- Icons or Buttons on a dialogue box that you should click are shown in **bold**, for example: **OK, Apply**.
- All fields are shown in **bold** between “quotation marks”.

See Appendix B (pg 19-22) for more thorough information on scan parameters, writing data collection programs in DataScan, and data collection strategies.

1) **Start MDI DataScan** if it is not already running.

2) **Initialize the Diffractometer**

   *Before data can be collected, the goniometer must be initialized and the detector turned on.*
   - This only needs to be done once per session.
   - The datum offsets and HV/PHA values are posted on the monitor of the data collection computer. **DO NOT USE THE VALUES LISTED IN THIS SOP—USE THE VALUES POSTED ON THE MONITOR!!**

   a) Open the menu *Control>* *Rigaku D/Max-b*
      i) The *Rigaku D/Max-b* control window will open.

   b) **Initialize the Goniometer**
      i) Go to the *Goniom* tab.
      ii) Make sure that the offset values for “2Theta” and “Theta” correspond to those posted on the computer monitor.
      iii) Click the **Datum** button.

   iv) A dialogue window will ask you if you want to “Datum the goniometer?”
   v) Click **OK**

   vi) It may take a minute or two for the Datum to complete. You will know it is done by the message in the lower left portion of the screen, which will read “Datum completed.”
c) Turn on the detector
   i) Go to the PHA tab in the Rigaku D/Max-b control window.
   ii) Make sure that the values for “Base Line”, “Window”, and “Detector HV” correspond to those posted on the monitor of the data collection computer.
   iii) Click SET for all three PHA properties: Base Line, Window, and Detector HV.

d) Close the Rigaku D/Max-b control window after you have initialized the diffractometer.

3) Set Up the Scan Parameters
Part A of these instructions (pg 11-12) tells you how to create a new data collection program by creating a single new Scan Job in the Task List.
Part B of these instructions (pg 13) gives you instructions for collecting data from additional samples by editing a previous Task List.
Appendix B (pg 19-22) provides additional information about saving, editing, and using the Task List and Scan Job, as well as information about scan parameters and data collection strategies.

A) Creating a New Data Collection Program
   a) Go to the menu Scan> Routine Scan ...
      i) This opens the Setup Scans window that holds the Task List.
      ii) Never use the Quick Scan option; it doesn’t work well for our instrument.

   b) If the Task List in the Setup Scans window already contains a Scan Job, delete it.
      i) If you want to use the existing Scan Job (for example, if running a second sample) go to part B (pg 13) for instructions.

   c) Set the field “Output Dir:” to the folder where you want your data to be saved.
      i) Click Browse to change the folder where your data will be saved.

   d) Set the field “Append data filename with” to None

   e) Click on the New Scan Job button.
      i) This will open the New Scan Job window, where you can edit scan items.
f) Edit the **New Scan Job** window
   i) Set the filename in the “Filename” field
   ii) You can provide a sample description in the “Scan ID” field.
   iii) Click on the New Range button.
       (1) The **New Scan Range** window will open.

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f) Define the Scan Parameters in the **New Scan Range** window.

   i) **Start**: The minimum start angle is $5^\circ$ 2theta
   ii) **End**: The maximum end angle is $130^\circ$ 2theta
   iii) **Step**: the recommended step size is $0.02^\circ$
       (1) The step size can be 0.01 to 0.05deg
       (2) You MUST make sure that the scan range is evenly divisible by the step size:
           (a) for example, if using a step size of 0.03, then a scan range from 20 to 40 degrees 2theta (20 deg scan range) would produce an error, while a scan range from 20 to 50 degrees 2theta (30 deg scan range) would be ok.
   iv) **Mode**: Continuous is recommended unless you want a scan rate less than 0.2 deg/min
   v) **Scan Speed** is defined differently for continuous and step scan modes
      (1) For continuous mode, a **Rate** between 0.2 to 3 deg/min is recommended
      (2) For step scan mode, a **Dwell** time between 5 and 10 sec is recommended
   vi) **Axis**: should be **Both axis**
   vii) **Psi**: should be 0.0, unless the sample includes a single crystal substrate
       (1) **Psi** of 1.0 is recommended if the sample includes a single crystal substrate.
   viii) **Appendix B** defines the various parameters and offers suggestions for data collection.
   ix) Click **OK** in **New Scan Range** window and click **OK** in the **New Scan Job** window, so that you return to the **Setup Scans** window.

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h) **Start the Scan**
   i) Click on the Start button (in the bottom of the **Setup Scans** window) to begin data collection.
   ii) Click **OK** on the resulting dialogue
   iii) The shutter will automatically open and the instrument will begin to collect data
   iv) You do not need to remain in the X-ray lab while your data are being collected, but you do need to return to the X-ray lab punctually when your measurement is finished so that you can turn off the instrument and clean up your sample.
B) Collecting Data from Another Sample by Editing a Previous Data Collection Program

After you have collected data from your first sample, you might want to use similar scan conditions to collect data from another sample. To do this:

1) Follow the procedure in Section II “Preliminary Checks” (pg 6) to change your sample.
2) Once your sample is loaded and the enclosure is properly closed, go to the MDI DataScan4 program.
   a) The data from your previous run will be shown on the screen. The data were automatically saved, assuming that you used a routine scan, so there is nothing further for you to do.
   b) You do not need to initialize the diffractometer again.
3) Go to Scan > Routine Scan …
   a) This will open the Setup Scans window that contains the Task List.
4) The Task List in the Setup Scans window will show your previous Scan Job.
5) Many times all you want to do is change the filename for the new sample and then repeat an identical measurement keeping the same comments, scan parameters, etc. To do this:
   a) Change the Filename in the Task List.
   b) Click the Start button.
6) If you want to change more details in the Scan Job, such as the comment and scan parameters, then:
   a) Click on the Scan Job Property icon or double-click on the Scan Job in the Task List.
   b) This will open the New Scan Job window, where you can edit scan items.
   c) In the New Scan Job window, you can edit the “Filename” and “Scan ID”.
      i) If this is all that you want to change, then click OK to return to the Setup Scans window.
   d) To edit scan parameters, click on the Show Item Property icon or double-click on the line in the scan job list.
   e) The New Scan Range window will open. You can edit the scan parameters as before (start, end, step size, scan rate, etc). Then click OK.
   f) When done editing the Scan Job, click OK to return to the Setup Scans window.
   g) When done editing the Task List, click Start to begin the measurement.
4) **During the Scan**
   a) MDI Datascan4 will display the data in real time as it is collected.
   b) The **Scan Progress Bar** (with the text “Scan in progress ...”) will show the %completion of the current scan.
      i) By floating the mouse pointer over this display, a yellow text box will appear showing the time left before the scan is finished.
   c) You can stop a scan in progress:
      i) Click on the **Stop** icon
      ii) Click **OK** in the resulting dialogue
      iii) The partially completed scan is saved automatically
   d) The bottom bar of the display window shows information such as the current detector angle, the filename of the data being collected, the scan parameters of the current scan
   e) You can zoom in the realtime display window by left-click and dragging.

5) **If the MDI DataScan4 Software Stops Communicating with the Rigaku Diffractometer**
   a) If the data collection seems to hang or you get error messages regarding control of the diffractometer, use the following instructions to correct any problem.
      i) Exit MDI DataScan4.
      ii) Reset the Rigaku controller electronics
         1) There is a green cabinet behind the computer desk.
         2) Open the door to the green cabinet.
         3) Find the RESET switch in the NIM-BIN on the right-hand side of the cabinet.
         4) Toggle the Reset switch
      iii) It might be a good idea to restart the computer, but this is not always necessary.
      iv) Start MDI DataScan4 again.
         1) Reinitialize the goniometer and detector (pg 10)
V. WHEN YOUR RUN IS FINISHED
While you do not need to remain in the XRD lab while your data are being collected, you must be present when your run finishes so that you can turn off the diffractometer and retrieve your sample.

When you are done collecting data, turn off the generator. Do not leave the generator on for the next user, unless that user is present in the lab and has their sample ready.

1) **Turn off the X-ray generator**
   a) First, slowly turn down the current (mA)
      i) Turn the coarse adjust dial on ‘click’, pause, then turn it again.
      ii) Turn the coarse adjust knob all the way counterclockwise, to 10 mA
   b) Then, slowly turn down the voltage (kV)
      i) Turn the coarse adjust dial on ‘click’, pause until the needle stops moving, then turn it again.
      ii) Turn the coarse adjust knob all the way counterclockwise to 20 kV
   c) Press the red OFF button on the generator control panel.

2) **Remove your sample from the diffractometer**

3) **Close the enclosure doors.**

4) **Disengage Coral** to stop logging your instrument use time.

Please note:
- Reserve instrument time using Coral
  o You may reserve 30 hours of prime-time (9am to 5pm) use in a 2 week period.
  o You may reserve time up to 14 days in advance

- Repeated failure to show up for reserved instrument time may result in a penalty, at the sole discretion of CMSE administration
  o If you need to cancel a reservation, do so at least 12 hours before the start of your time
- If a person is 15 minutes late for their reserved time slot, they forfeit that reservation
- Repeated failure to log your instrument use may result in a penalty, at the sole discretion of CMSE administration
- Any sample left in the lab will be disposed of and the sample holder confiscated.
APPENDIX A: Selecting Slits for Your Experiment

The Rigaku diffractometer uses a line focus X-ray source - at its origin, the X-ray beam is a line 0.5mm wide and 10mm high. From the origin the X-ray beam diverges. Soller slits are used to limit the divergence in the axial direction; divergence and receiving slits are used to limit the divergence within the diffraction plane. The Soller slits are fixed at a 5° aperture; consequently, the X-ray beam irradiates a 12mm high area on the sample. The divergence, anti-scatter, and receiving slits can be changed to optimize the trade-offs between resolution, intensity, and background noise.

The most common configuration for the Rigaku diffractometer is a 1° divergence slit, a 1° anti-scatter slit, a 0.3mm receiving slit, and a 0.8mm monochromator receiving slit. The ½° divergence and scatter slits are also commonly used.

THE DIVERGENCE SLIT (DS)

The divergence slit (DS) limits the divergence of the incident X-ray beam within the diffraction plane. The DS affects intensity and resolution - a larger DS will yield more intensity at the cost of lower resolution and more background noise in the diffraction pattern. The length of the irradiated area of the sample is determined by the incident angle (omega) and the DS aperture.

Figure 1 and Table I show how the irradiated length varies with DS aperture and the diffraction angle 2theta; for these calculations, it is assumed that the incident angle (omega) is equal to ½ · 2theta.

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<th>1/6° DS</th>
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<th>2° DS</th>
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<td>740</td>
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If your primary concern is maximizing the diffraction peak intensity in your measurement, then you should choose the largest divergence slit that avoids beam spill-off. Beam spill-off (sometimes called “falling off”) refers to a condition where the X-ray beam is larger than the sample and therefore irradiates other stuff such as the sample holder. This may produce diffraction peaks from the sample holder and will increase the background intensity and noise. If the beam becomes larger than even the sample holder, the background intensity and noise will increase more and artifact peaks (aka ghost
peaks) may be produced at low angles 2theta. Beam spill-off will also violate the constant volume assumption, compromising the accuracy of quantitative analysis. Using a smaller DS can be advantageous because it improves the linearity of the background at low angles 2theta, improves the signal-to-noise ratio, and improves the peak shape and resolution. You might particularly want to consider using a smaller DS when determining %crystallinity in polymers, when working with samples that have a rough surface, or when working with small samples.

Choices for the DS are: 1/6, ½, 1, and 2°. The ½ and 1° slits are the most commonly used. The 1° DS is often used when the important data is at angles larger than 20° 2theta; and the ½° DS is often used when there are important data below 20° 2theta or when doing % crystallinity calculations for polymers.

THE SCATTER SLIT (SS)
The scatter slit (SS) is used to reduce background noise from air scatter and other sources. Judicious use of the SS can improve the signal-to-noise ratio by as much as a factor of ten. The SS should be large enough that it does not impinge on the diffracted X-ray beam, but only on largely diverging and randomly scattered X-rays.

Air scatter is dominant at low angles of 2theta and is the cause of typical exponential rise in the background at low angles 2theta.

In general, the SS should be the twice as large as the DS. When collecting data below 20° 2theta, you might choose to make the SS the same size as the DS in order to reduce the background. The SS should never be smaller than the DS.

THE RECEIVING SLIT (RS)
When the X-ray beam irradiates the sample, each diffracting crystallite functions as a point source producing a divergent Debye ring. The receiving slit (RS) defines what portion of the Debye ring passes through to the detector as a signal. Using a smaller RS can produce greater peak resolution at the expense of overall intensity. Contrariwise, a larger RS will offer better intensity in the diffraction pattern at the expense of peak resolution.

Choices for the RS are 0.15, 0.3, and 0.6 mm. The Rigaku diffractometer is optimized for the 0.3mm RS. You may use a larger or smaller RS, but you will give up more than you gain-- a 0.15 RS will gain 15% better resolution but you will lose 21% intensity. The 0.6mm RS will gain intensity but the peak widths will be much larger.

Peak resolution is a function of the width of the diffracted peak, usually defined as the full-width at half-maximum intensity (FWHM). Better peak resolution (smaller FWHM) will allow peaks that are close together to be individually resolved. Improved resolution (smaller RS) is important for resolving patterns with many overlapping peaks and for providing accurate peak positions for indexing and cell refinement. Greater intensity (larger RS) is important when looking for trace impurities, thin films, and analyzing nanoparticles less than 40nm large. The larger RS may be particularly advantageous when analyzing small nanoparticles (<40nm), since the diffraction peaks are broadened by the crystallite size and therefore the loss of resolution will not be significant. Conversely, when analyzing large nanoparticles (>70nm) the resolution is extremely important and so a smaller RS would be very advantageous.
Table II compares the trade-off between peak width and intensity for common combinations of slits using the (220) peak of Si.

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THE MONOCHROMATOR RECEIVING SLIT (RSM)

The RSM works in conjunction with the monochromator to remove spectral contamination (unwanted wavelengths of radiation) from the X-ray signal, such as K-beta, Tungsten L-alpha, and X-rays fluoresced from the sample. The monochromator alone removes most (~70%) of the spectral contamination, but the addition of the RSM can improve the performance removing up to 99% of the spectral contamination, as illustrated in Figure 2.

Most data on the Rigaku is collected with a 0.8mm. A smaller RSM is necessary when strong diffraction peaks produce noticeable K-beta and W lines in the diffraction data, or when the sample fluoresces under the incident radiation producing an unacceptably high background. In such cases, the 0.6 or 0.45mm RSM slits can be used. The smaller the RSM, the more spectral contamination is removed-- but diffraction peak intensity is also lost. The 0.6mm slit is sufficient to remove most fluoresced X-rays. The 0.45mm RSM is required when collecting data from films on single crystal substrates or when doing very slow scans (several hours long). The 0.8mm RSM slit produces a more linear background without sacrificing very much intensity; it is almost always preferable to use the 0.8mm RSM rather than no RSM.

![Figure 2](image-url).

Figure 2. Comparison of the (111) peak of Si collected with (red) and without (black) the 0.45 RSM slit.
Appendix B. Typical Scan Parameters and Data Collection Strategies

The typical scan uses the goniometer in theta/2theta reflectivity mode, where the detector rotates at a rate of 2theta/minute and the sample rotates at a rate of theta/minute. Consequently, the incident angle (theta) is always ½ of the angle between the incident beam and detector (2theta). The theta/2theta mode of data collection ensures the optimal focusing condition.

The most common data collection scheme is a continuous mode scan, both axis, from 20 to 70 deg 2theta with a scan speed of 0.5 to 3 deg/min and a step of 0.02deg. The start and end angles should be changed as appropriate for your sample: consult the PDF cards to anticipate if there are low or high angle peaks that should be observed to best analyze your sample.

Scan Job Parameters, set in the New Scan Range window, are:

Start and End: determines the start and stop angles for the scan range. For phase ID, a scan from 20 to 70° 2theta is often adequate. The PDF database should be consulted to choose the best scan range for the possible phases in a sample. For example, if a potential phase in your sample has low angle peaks (below 20° 2theta) then that range should be included in the scan--because few inorganic phases have low angle peaks, analysis of the low angle data can be less ambiguous. Likewise, if your sample has only 2 or 3 peaks below 70° 2theta (common for many metals) then you should scan to a higher angle--it is very hard to conclusively determine that a phase is present from only 2 diffraction peaks.

The scan range limits on the Rigaku are from 3° to 140° 2theta.

Mode: can be either continuous or step scan.

- In continuous mode, the detector moves at a constant rate. The counts observed over a specified range (the step size) are added together to define a datum point. For example, the data might record 874 counts/sec at 10.02 deg 2theta; this actually indicates that 874 counts/sec were observed over the range of 10.01 to 10.03 (assuming a 0.02° step size ).
- In step scan mode, the detector is moved to a specified position and then sits stationary counting photons for the specified time (the dwell time). The distance between each detector position is specified by the step size.

The difference between the two modes is very subtle and only rarely affects the quality of the data. One rule of thumb is to use continuous mode for faster scans and use step mode for longer scans. If the dwell time would be less than 5 sec/step in step mode, then consider using continuous mode instead; if the scan speed would be slower than 0.2 deg/sec in continuous mode, then consider using step mode instead. This guideline considers the stress on the motor during your data collection.

Additional Detail

In continuous mode, the position is slightly less precise because intensity is integrated over an interval; for example, if the counts at 10.01 °2theta were 10 cps, and the counts at 10.03 °2theta were 1000 cps, then those data might be recorded as 500 cps at 10.02 °2theta. However, all photons at all detector positions are recorded, possibly producing more precise peak intensities. In step mode, the detector might miss the point of highest intensity: if the detector collected 500 cps at 10.02 °2theta and 300 cps at 10.04 °2theta, it might not accurately represent that the peak maximum was at 10.03 °2theta with 1000 cps. It might be considered, then, that continuous mode produces more precise intensities and also greater intensity, since no position is skipped; and step mode produces more
precise positions. However, because most diffraction peaks are relatively wide (>0.2 deg 2theta) and the detector does not truly observe just a single infinitely small point, but rather a small range, 99% of the time you will not be able to tell the difference between data collected in continuous mode or step mode.

**Step:** The step size as designated in 0.002 °/step increments. Typical values are 0.01 to 0.04 °/step.
- **In continuous mode,** the step size determines the sampling interval over which intensity is added together to define a datum point. A larger sampling interval results in more photon counts over a longer range being added together for each datum point, and therefore may produce greater intensity in the pattern. However, a larger sampling interval will produce less sharply defined transitions from background to peak intensity and lower spatial resolution between close or overlapping diffraction peaks. **The sampling interval in continuous mode therefore produces a trade-off between intensity and resolution**
- **In step mode,** the sampling interval determines the distance between successive detector positions at which intensity is measured. A larger step size reduces the total scan time, but also produces lower spatial resolution. **The sampling interval in step mode therefore produces a trade-off between resolution and scan time.**

**Rate (d/m) or Dwell(s):** when the mode is continuous, this parameter is scan rate; when the mode is step, this parameter is dwell time per step.
- **Rate (d/m):** the rate, in degrees/minute, at which the detector moves to collect the data. The total scan time is merely the scan range/scan speed. Most data can be reasonably collected with a scan speed of 0.5 to 5 d/m. Some samples, such as strongly crystalline samples or single crystals, can be scanned at faster rates; while organic materials or poorly crystalline sample may require slower rates.
- **Dwell (s):** the time, in seconds, that the detector spends at each data point counting intensity. The total scan time is determined by the (scan range/sampling interval) * count time. Standard settings are 0.5 sec/step to 10 sec/step.

**Axis:** Can be Both, Detector, Theta, or Rocking psi-tilt.
- **Both Axis** moves both the sample angle (theta) and the detector angle (2theta), collecting data in the optimal para-focusing condition. This option should be used for most scans. Also known as a theta:2theta scan.
- **2theta** axis rotates only the detector; the sample position is fixed. This is most often used for Glancing Incidence Angle X-Ray Diffraction (GIXD). Without a thin film attachment, the 2theta scan will tend to give broad and misshapen peaks.
- **Theta** rotates only the sample; the detector is fixed.
- **Rocking psi-tilt** rotates the sample and the detector at a constant rate (unlike a scan of both axis, where the sample rotates at a rate equal to ½ the rate at which the detector rotates).

**Psi (or Theta, Detector, or 2Theta):** This field varies depending on which scan axis has been chosen.
- **Psi:** Appears when the scan axis is Both. Psi can be used to define an offset between theta and 2theta that is preserved during the entire scan. **If you are collecting data from a film on a single crystal substrate, then Psi should be set to 1 or 2°. Without a psi offset, omega is equal to ½·2theta. With a psi offset, omega is equal to ½·2theta + psi. A psi offset of 1 or 2° is sufficient to eliminate 90%+ of the intensity from a single crystal substrate.**
- **Theta**: Appears when the scan axis is **Detector**. This defines the fixed incident angle omega in a scan where only the detector is moving.
- **Detector**: Appears when the scan axis is **Theta**. This defines the fixed detector angle 2theta in a scan where only the sample moves.
- **2Theta**: Appears when the scan axis is Rocking psi-tilt. This fixes the angle between the detector and the sample. Then, as the sample rotates the detector moves at the same rate, keeping a constant angle 2theta between them.

**Additional Information about the Task List in MDI DataScan4**

DataScan includes two components that define how data will be collected.

- **Scan Job**: specifies parameters, such as scan range, speed, etc., for collecting the XRD data.
  - The Scan Job is show in the New Scan Jobs window that opens when you click on the create or edit scan job button in the Setup Scans window.
  - A single scan job may include several different ranges, which is useful if you want to collect several short scan segments rather than one long scan. All data collected with a single scan job is saved under a single filename.
  - A typical Scan Job for the Rigaku will contain just a single item.

- **Task List**: a list of scan jobs that will be collected in order.
  - The Task List is shown in the Setup Scans window that opens when you select Scans>Routine Scan in the DataScan menu.
  - This is most useful if the diffractometer has a sample changer, which ours doesn’t.
  - Useful on the Rigaku if you want to collect multiple scans from the same sample, or multiple short scan segments, and save each one with a different filename.
  - A typical task list for the Rigaku will contain a single line for a single scan job.

The Task List shown above has three scan jobs. Only scan jobs with a check mark next to them will be run-- in this case, the first and third will be executed in the order in which they appear in the Task List. Each separate scan job in the Task List will be saved in a separate file. Clicking on the New Scan Job button will allow you to add another scan job to the Task List. You can change the Filename for each Scan Job in the Task List, but anything else must be changed by double-clicking on a Scan Job or clicking on the Scan Job Property button to open the New Scan Jobs window.
The Scan Job shown above will collect data from a single range as defined by the single item in the list. Clicking on the **New Item** button will allow you to add another scan range to the list. This additional item would be run after the first; both measurements would be saved in the same file. Clicking on the **Show Item Property** button or double-clicking on an item in the list will allow you to edit the scan parameters.

**You can save a Task List for repeated use.**
While you can create a new data collection program every time you collect data, you can also save a task list as a *.jrx file and load it every time you want to collect data using those scan parameters.

Create a Task List with the scan jobs and scan parameters that you expect to commonly use. To save the Task List, click on the **Save Task** button. This will save the Task List as a *.jrx file.

To load a saved task list (a *.jrx file), click on the **Load Task** icon. The Task List was saved with a specific **Filename**—be sure to change the filename and **Scan ID** before running the measurement.

**Some Data Collection Strategies**

- **When collecting data from a thin film on a single crystal substrate,**
  - the signal from a substrate may dominate the pattern and obscure the signal from the coating.
  - The single crystal substrate may produce many spurious diffraction peaks, such as multiple K-beta and W L-alpha lines and even forbidden reflections.
  - The diffraction peak from the single crystal substrate may also be intense enough to damage the detector!

To avoid the single crystal peak, in the **New Scan Range** window you can set “psi” to a non-zero value, usually 1 or 2° (assuming that your scan axis is set to “Both Axis”). The sample angle will then be offset relative to the detector, so that the single crystal does not diffract. You may observe a small amount of diffraction intensity from the substrate, but it will be reduced by 90% or more.
  - You can use a rocking curve scan to determine how much of a psi offset is required to avoid the single crystal peak. The psi offset should be as small as possible, to avoid distorting the peak shapes. then you can include an angular offset in your data collection to correct for this.
  - The single crystal peak from a substrate will only appear for precisely defined angles between the incident beam and detector; for example, the diffraction peak from the planes parallel to
the substrate surface will only diffract when theta is exactly ½ 2theta (the normal parafo-
ocusing condition in a theta/2theta scan).

- The success of this technique assumes that your coating is a randomly oriented polycrystalline coating. If not, e.g. if the coating is epitaxial, then this offset might cause the peaks from the coating to also not diffract.

- **If you have a sample with only a few peaks or very specific ranges that you are interested in observing**, then you do not have to scan the entire pattern; you can collect data just from the ranges that you are interested in. To do this, you can either list multiple scan ranges in your scan job, or you can list multiple scan jobs in your task list. If you define several scan ranges in a single scan job, all of those data will be saved in a single file. If you define several scan jobs in your task list, then each scan range can have a different filename. This approach will allow you to reduce the total scan time by focusing just on the areas of interest. *In this approach, you make many assumptions about your sample. Be careful that you are not ignoring useful data.*

- **For unit cell or Rietveld refinement**, particularly of high symmetry structures, high angle data are often invaluable. High angle data can greatly increase accurate refinement despite sample displacement errors. You should collect data to as high an angle that still produces distinct diffraction peaks.

**Recommended References for Powder Diffraction:**

- For more references, including links to free online databases, tutorials, and software, go to [http://prism.mit.edu/xray/resources.htm](http://prism.mit.edu/xray/resources.htm)