The following write-up with photographs will show the alignment procedure to be done on Rigaku Ultima III Powder X-Ray Diffractometer (ru-pxrd) once in couple of months. Thanks are due to David Carnevale for help in demonstrating the procedure.

**Note:** This write-up is a beta version and some refinement is needed on some steps. Steps done at the PC (computer) will be shown in Times New Roman font; Sections done at the instrument r-pxrd will be shown in Georgia font.

Rigaku Ultima III Powder X-Ray Diffractometer (shown on the right hand panel) with Mercury CCD and Scintillation Detector is located in CSL 1011 in FSU’s Chemical Sciences Laboratory.

The generator has a Copper fixed anode tube and is cooled by Haskris chiller (originally a refrigerated version; now water-to-water chiller). The Mercury CCD is cooled by Julabo MD F25 chiller.

Two computers control data collection and processing: 1) Control PC on the right and 2) Frame Grabber PC on the left. However, there is only ONE monitor and key board. So you access each computer using a KVM switch.

In the keyboard press, “Scroll Lock” key twice quickly to switch between the two PCs. Almost all of the time the monitor outputs and keyboard inputs are from and to Control PC.

Log in to control PC. It is likely that it is already running.
In the ‘Standard Measurement Window’, select
- File > Save
- Confirm
This allows the Standard Measurement values to be remembered when we restart the computer.

Close RaxVideo window

Left Click on RGKExpand on the left side of System Tray, then select
- Stop AreaMax
This closes the AreaMax window (better way to close it)

Close Standard Measurement Window
Close Manual Measurement Window

Left Click on RGKExpand on the left side of System Tray, then select
  • Exit

In the Ultima III Generator’s Left Top Panel, under the SAFETY RELEASE panel, press the ‘DOOR’ button. While the DOOR’ button is blinking, open the sliding door all the way to left and right.

Inside the x-ray enclosure, locate the DALSA power supply (usually located on the right hand side and sits on the base plate) for the CCD detector. Power it down by switching it OFF (the power switch is located in the front of the unit, however, only the unit’s back is visible) by locating the switch on the far right hand side.

Now, in the computer using the single keyboard press, “Scroll Lock” key twice quickly to switch to Frame Grabber PC.
  • Log in using username and password
  • Wait for the computer to display a DOS window
  • Using the Start Button, shutdown the computer
Now, in the Ultima III Generator’s Left Top Panel,
- Under X-RAYS
  o Press the Grey OFF button, then
- Under POWER
  o Press Red OFF button

Then, in the Ultima III Generator’s Left Bottom Panel, open the door and locate the SERVO AMP UNIT,
- Turn it OFF by pressing the black power switch to off position.

Swap Equipment Now

With Ultima III generator door open, locate the CN ATT2 cable (this is plugged underneath the stage) and unplug it
<table>
<thead>
<tr>
<th>Step</th>
<th>Image</th>
</tr>
</thead>
<tbody>
<tr>
<td>Attach the CN ATT2 cable to the ATTACHMENT ADAPTER sitting on the base plate</td>
<td><img src="image1.jpg" alt="Attachment Adapter" /></td>
</tr>
<tr>
<td>Locate the plastic lid for the CCD and cover the CCD with it</td>
<td><img src="image2.jpg" alt="Plastic Lid" /></td>
</tr>
<tr>
<td>Remove the micro-area stage (used for powder samples)</td>
<td><img src="image3.jpg" alt="Hex Screws" /></td>
</tr>
<tr>
<td>• Use the small hex wrench and undo the two screws until they are flush (on-top) with surface</td>
<td><img src="image3.jpg" alt="Hex Screws" /></td>
</tr>
<tr>
<td>• Gently pull the stage toward the user</td>
<td><img src="image3.jpg" alt="Hex Screws" /></td>
</tr>
<tr>
<td>• Sit it down on the base plate of the instrument</td>
<td><img src="image4.jpg" alt="Stage sitting on base plate" /></td>
</tr>
<tr>
<td>Process</td>
<td>Description</td>
</tr>
<tr>
<td>---------</td>
<td>-------------</td>
</tr>
</tbody>
</table>
| Remove the CCD Camera (with lid on it) | • Hold the CCD in one hand  
• Note down the value on the back side (~105mm)  
• Use larger hex wrench and undo all the six(?) screws flush to the top  
• Slowly wiggle the CCD and remove it  
• Gently place it on the base plate |
| Using larger hex wrench undo all the larger screws flush to the top and remove the black bracket that was holding the CCD and remove it completely (with the screws) and leave it on top of the CCD inside the enclosure. |
| Now, remove the two black brackets from the cabinet above the Control PC (they are marked #1 and #2) | • Put the #1 bracket close to the sample  
• Put the #2 bracket away from the sample  
• Use the hex wrench and secure them to the detector-arm of the instrument (the same place where the long black bracket was secured) |
| Now, we to put the Filter Holder (Part #53D-0466) in front and Scintillation Counter (Part #57 38E401) and at the back with very little gap between them. |
First, put the Filter Holder (FH) in front
- Lightly secure it in the detector-arm with 3 small hex screws. DO NOT fully tighten it
- Look underneath the FH and locate a “Red Line”
- Match this “Red Line” with the “Red Line” underneath the detector-arm
- Now tighten the Filter Holder fully

<table>
<thead>
<tr>
<th>Filter Holder in position now</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.jpg" alt="Filter Holder" /></td>
</tr>
<tr>
<td><img src="image2.jpg" alt="Filter Holder" /></td>
</tr>
<tr>
<td><img src="image3.jpg" alt="Filter Holder" /></td>
</tr>
</tbody>
</table>

Now, put the Scintillation Counter behind the Filter Holder with very little space between them
- Secure it with 2 hex screws

<table>
<thead>
<tr>
<th>Now we have both Filter Holder and Scintillation counter in place</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image4.jpg" alt="Filter Holder" /> <img src="image5.jpg" alt="Scintillation Counter" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Plug in the connector for Scintillation counter</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image6.jpg" alt="Scintillation Counter Plug" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Plug in the RJ1 underneath the Filter Holder</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image7.jpg" alt="RJ1 Plug" /></td>
</tr>
</tbody>
</table>
Now, put the new stage Standard Sample Holder (stored inside the cabinet above the PC)
- Push the stage into the slot where the Micro-Area stage was installed
- The correct position of stage is guaranteed if the three holes in the stage are aligned with three holes on the instrument back panel
- Put the thumb screws and gently tighten
- Insert an adapter for bubble level

<table>
<thead>
<tr>
<th>Adapter for the level and bubble level</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image of adapter and bubble level" /></td>
</tr>
</tbody>
</table>

With the adapter and bubble level in place now gently rotate the stage until the bubble is level both horizontally and vertically. Then hand tighten the thumb screws. The standard sample holder is now set.

<table>
<thead>
<tr>
<th><img src="image2.png" alt="Image of stage with bubble level" /></th>
</tr>
</thead>
</table>

Now open the Right Hand Side Bottom panel on the generator
- Change the SHT MODE switch from CCD to SD (flip it down)

<table>
<thead>
<tr>
<th><img src="image3.png" alt="Image of generator panel" /></th>
</tr>
</thead>
</table>
| Now open the Left Hand Side Bottom panel on the generator  
| ---  
| • Press the third switch from bottom on the left-most PC card once  

| In CBO mirror box remove the H2 slit and put H10 slit. The label should be facing the sample.  
| ---  
| In the back filter holder  
| • Open the box  
| • Put Al foil in the front  
| • (0.3) slit in the back  
| • Ni foil at the back  
| • All labels facing the sample  
| • Close the box  

| Restart in this sequence  
| ---  
| 1. SERVO AMP UNIT on  
| 2. GREEN button on in POWER Panel  
| 3. GREY button on X-RAY panel  
| 4. Confirm Shutter 2 is in EXT mode  

| Turn on the Control PC  
| ---  
| Log in  
| • Administrator  
| • Cmt-521!  
| • RINT2200 Window opens  
| • Right Window opens  
| • Then open the Rigaku Control Panel folder  

|
Now click on the RINT2200 Right System (but not the RINT2200 Right System Add/Delete button). This should open a new dialog box

The transition between earlier and next step needs some clarification

System Details Property Window opens. Change the XG Server Mode from “Standard (Shutter Relay)” to “Standard”

Now click on the System Details button in the Rigaku Control Panel

This will open up the window shown below with the title “RINT 2200 Right System Property of …”
In System Construction

<table>
<thead>
<tr>
<th>Change From</th>
<th>Change To</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goniometer: Ultima 3 theta-theta</td>
<td>Goniometer: Ultima 3 theta-theta</td>
</tr>
<tr>
<td>goniometer+Mercury CCD</td>
<td>goniometer+Mercury CCD</td>
</tr>
<tr>
<td>Attachment:</td>
<td>Attachment: Standard Sample Holder</td>
</tr>
<tr>
<td>Filter:</td>
<td>Filter: K-beta filter</td>
</tr>
<tr>
<td>Detector:</td>
<td>Detector: Scintillation Counter</td>
</tr>
<tr>
<td>Temp Controller: Not installed</td>
<td>Temp Controller: Not installed</td>
</tr>
<tr>
<td>Slit:</td>
<td>Slit: Parallel</td>
</tr>
</tbody>
</table>

In Geometry System
Select: Micro area

Now open the Auto-align (Right system).htm link

- It will open a window and will load lots of stuff
- In the window, select the following
  - 2-Theta Alignment
  - Theta Alignment
  - Profile Measurement

Now click the button “Execute” in the .htm file (center area) and follow the directions

- Confirm everything
• Initializing …… (takes sometime)
• 20 kv and 2mA will go to 40 kv and 40 mA
• Tube will move

MORE TO COME … … [Incomplete procedure!!]