X-Ray Generator Standard Operating Procedure (SOP)

All users of x-ray facility are required to take the Radiation Safety Course offered by FSU Radiation Safety Office. The users should also be trained by the X-Ray Facility Director prior to using the instrument.
RSO (644-8801 or 644-8802) | www.safety.fsu.edu/rad.html
XRF Director (850-644-6448) | tsomasundaram@fsu.edu | www.sb.fsu.edu/soma

The following is the Standard Operating Procedure (SOP) for starting-up and shutting-down Rigaku RU-H3R x-ray generator located in Kasha Laboratory Building 410A (KLB410A; Facility Layout). This write-up is a quick start-up procedure (for someone already familiar with the system). For a complete procedure, please read the X-Ray Generator Manual kept at KLB410A.

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Throughout this write-up the SOP is indicated by numbered steps. Simple trouble-shooting steps are indicated by an alphabet preceded by the special symbol “@” and linked to a Trouble Shooting Guide at the end of the write-up. It is essential that all first-time users get properly trained by the X-Ray Facility Director Dr. Thayumanasamy Somasundaram [aka “Soma”] before actually using the instrument. He can be contacted by phone at 850-644-6448 or by e-mail tsomasundaram@fsu.edu. Soma’s office is KLB414.

1. Wear radiation safety badges (Body and/or Finger badges as appropriate). If you need one, please go to the Radiation Safety Office’s Personal Dosimetry Application page at:
2. Locate the chilled-water circulator’s grey circuit-breaker box. They are labeled P5 and P6 (see Figure 1a) and are located on the North wall of KLB413. Select either Pump5 or Pump6 and turn the 3-way (circular black) switch from ‘OFF’ to ‘HAND’ position. This turns the power to the chilled-water circulators located on the West wall (See Figure 1b).

![Figure 1a. Chilled-water circulator circuit-breakers.](image)
![Figure 1b. Chilled-water circulators (West wall).](image)

3. Usually the valve in the domestic chilled-water inlet is turned to partly ‘OPEN’ position, if not please do so. The building chilled-water pipes can be identified by the shiny silver-like insulation wrapped around them (Figure 2a). When the handle is to parallel to the pipe, water flows fully and when the handle is turned perpendicular to the pipe the flow stops completely. @A.

![Figure 2a. Domestic chilled-water pipe and partly open valve.](image)
![Figure 2b. Haskris water chiller with Pump selection and ON switch high-lighted.](image)

4. Press the ‘ON’ switch in Haskris water chiller. Our Haskris chiller is a water-to-water type and will take several minutes of external chilled-water flow to produce the desired internal temperature. Ensure that the ‘PUMP SELECTION SWITCH’ is at the center [@ BOTH] and ‘TANK WATER LEVEL INDICATOR’ lamp is ‘ON’ (See Figure 2b). @B.

5. In the front panel of the Haskris water chiller, you will notice two dials and three flow meters. The left-hand side (LHS) dial and flow meter give information about the pressure, temperature and flow rate of the internal chilled-water to the target. The right-hand side (RHS) dial and flow meters give the same information for the turbo molecular pump and tube housing.
6. At the start of the Haskris operation, temperature reading on the both dials will be close to the ambient (~70°F). The pressure gauges should be reading ~38 psi (LHS) and ~28 psi (RHS). The flow rate should be ~3.5 GPM (LHS) and ~20 GPH & ~1.6 GPM (RHS) and close to the blue lines scored on top of the flow-meters.

7. After several minutes of Haskris chiller operation, the temperature dials should read 48-52°F, indicating that the internal water supply has reached equilibrium. @C.

8. Write in the logbook your name, the date, time, hour-meter reading of the generator and a general description of your experiment (e.g., screening, data collection, etc.)

9. Flip the handle in the x-ray power circuit breaker box on the wall to ‘ON’ position (grey box with a red-handle, Figure 3). This will supply power to the x-ray generator and vacuum system.

![Figure 3. X-Ray Power Circuit breaker.](image)

![Figure 4a. Primary and Reserve Helium cylinders.](image)

10. Ensure that enough helium gas is available either on the Primary or the Reserve (labeled as P and R) gas cylinders strapped to the wall (Figure 4a). High-pressure gauge at the cylinder should read between 500-2500 psi and low-pressure gauge should read 10-15 psi (See Figure 4b). @D.

![Figure 4b. Helium cylinder valve and flow gauges.](image)

11. One white tubing each from Primary and Reserve cylinders is connected to the back side of Bio-Switch (Figure 5b), an automated gas cylinder switching device (See Figure 5a).
12. One braided hose is connected from Helium cylinder Switcher to the Flow Control Box (Figure 6) on each of the generators (via a hidden Y connector on the ceiling). **Ensure** that the flow selection knob on the Flow Control Box is set to ‘SLOW PURGE’ mode, the flow indicator is at 55 and helium is flowing to Osmic mirror system (Figure 7). **NOTE:** Failure to flow helium will irreversibly damage the mirrors and is extremely costly to fix, so ensure the flow. @E.

13. Walk to the back of the generator to ensure the following (default) conditions are met:
   - **(Left Hand Side):** In Vacuum control panel, yellow POWER lamp is lit.
   - **(LHS):** In TMP Drive Unit, both POWER and READY orange lamps are lit (Figure 8a).
   - **(LHS):** In the bottom panel big LINE orange lamp is lit and switch marked ELB1 is up and on ‘ON’ position (Figure 8b).
   - **(RHS):** All four lamps marked Temp, Flow (Tube), HP, and Flow (Target) is lit green. Figure 8c shows when conditions are met. @F.

14. After about 20 minutes of chiller operation, all conditions seen in Step 6 should remain stable.
15. **Press** the ‘START’ button in the Front ‘VACUUM’ Control Panel (Figure 9). This should execute an automated sequence of starting the rotary pump, the turbo molecular pump, and the ion gauge, indicated by the activation of respective green lamps. This sequence will take approximately 10 minutes to complete indicated by the lit yellow ‘OPERATE’ light. 

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16. **Press** the ‘ON’ switch in the ‘X-RAY’ panel (Figure 10). Two LED will light up below ‘TUBE VOLTAGE’ and ‘TUBE CURRENT’ each indicating a value of zero (Figure 11).

17. **Confirm** that the amber light at VACUUM panel is activated indicating it is ready to ‘OPERATE’ At this point, check to see the digital multi meter (located at far left in the front panel) reads a value of 0.200V or less (Figure 12).

18. Wait until the digital multi meter’s reading to fall below 0.150V. Then **press** the White ‘ON’ button located just below TARGET (Figure 10). The amber light just below READY at the X-RAY panel is activated. 

19. The target will start rotating. Simultaneously a green light in the Lamp Post (Figure 13) will also be activated.
20. **Wait** until the digital multi meter reading falls below 0.120V, preferably below 0.100V. Then **press** the White ‘ON’ button below X-RAY. Now the red light below X-RAY will be activated and the red light in the Lamp Post will also be activated.

21. The values shown in the LED’s will slowly increase and reach a value of 20 in TUBE VOLTAGE and 10 in TUBE CURRENT.

22. Simultaneously the needle in the Filament Current Monitor will go from 0A to approximately 0.75A (Figure 14). 

23. Allow 5 minutes warm-up at 20 kV and 10 mA. Slowly increase the TUBE VOLTAGE and the TUBE CURRENT according the following sequence (Make certain that you **DO NOT** exceed the maximum power of the generator for a particular filament). 

24. **Increase** the voltage by six (6) kV; Wait for a minute.

25. **Increase** the current by ten (10) mA; Wait for a minute.

26. **Repeat** Steps 23-24, until desired or the maximum allowable power is reached (Figure 15).

27. The maximum working power of the generator for a particular filament is a fixed value and **should never be crossed**. For example, for a 0.3 x 3.0 mm² filament, that value is 5.4 kW (e.g., 40 kV and 125 mA) and for 0.2 x 2.0 mm² filament, it is 2.8 kW. The applied load (power) for any filament can be calculated by simply multiplying the TUBE VOLTAGE and TUBE CURRENT.
28. **Enter** the voltage, tube current, filament-current, hour meter reading, time of the day, vacuum, filament used and other pertinent details in the logbook. @K.

29. To carry out an experiment an x-ray port and a particular shutter need to be opened.

30. Only the Right Hand Side port of the x-ray generator is coupled to an Osmic Confocal Mirror system and the mirror system in turn is coupled either to an R-Axis IP or a marCCD 165 detector.

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31. **Important Note for R-AXIS IP detector:** The opening and closing of the x-ray generator shutter for the R-AXIS IP is controlled solely by data collection computer whisper.sb.fsu.edu and therefore the shutter switch on the x-ray generator should to left at “EXT” position (Figure 16a).

32. **Important Note for marCCD detector:** The opening and closing of the x-ray generator shutter for marCCD is controlled manually and therefore the shutter switch on the x-ray generator should be left at “OPEN” position (Figure 16b). The computer spruce.sb.fsu.edu controls another shutter down-stream of the x-ray shutter and the user need not worry about being exposed to x-rays while mounting their samples.

33. If there is a concern regarding the proper use of x-ray shutters, please remember that keeping the shutters at “CLOSE” position is the best procedure while loading and unloading your samples. However, remember to switch it back either to “EXT.” (R-Axis IV ++ IP) or “OPEN” (marCCD) before you start your data collection.

34. The amber light at the Lamp Post will indicate which of the two x-ray ports is open.

35. Carry out your experiment.

36. **IN AN EMERGENCY:** If anytime during the experiment for any emergency reason the user needs to shut down the generator, please push the Red Round Button labeled “EMERGENCY” (Figure 17) located between the X-RAY and VACUUM Panels. This should completely shut-down the generator.

37. **IN AN TOTAL EMERGENCY:** If for any reason the user is unable to reach the generator but needs to shut down the system due to any emergency, please push the Red Circular Button labeled “EMERGENCY STOP” (Figure 18) located near the entrance to the Facility. Power to the X-Ray Facility will be completely cut-off.

38. **NOTE:** It is a good practice to locate these two EMERGENCY buttons, as soon as you start using the Facility. Soma will show the location during the initial training.

39. Complete the experiment.

40. Reduce the TUBE VOLTAGE and the TUBE CURRENT in steps. They can be brought down rapidly unlike the power up procedure.
41. Enter the final value of hour meter, time of the day and other details in the log book.
42. After reaching 20 kV and 10 mA, press the X-RAY ‘OFF’ red button located in X-RAY panel (Figure 10).
43. Press the red STOP button in the VACUUM panel (Figure 9).
44. Turn the knob (counter clock-wise rotation) in Helium Control Box to ‘Stop’ position (Figure 6).
45. Flip the red handle to ‘OFF’ position in the circuit breaker at the wall (Figure 3).
46. Wait for 15 minutes for the generator and the target to cool down.
47. Turn the black circular knob from ‘HAND’ to ‘OFF’ position on the circuit-breaker for water-chiller (Figure 1a).

48. Switch OFF the Haskris water chiller (Figure 2b).
49. Return the Radiation Finger/Body badge back to the storage box at the Facility.
50. Report any problems or concerns to Thayumanasamy Somasundaram.
## Trouble Shooting Guide

<table>
<thead>
<tr>
<th>Link</th>
<th>Symptom</th>
<th>Remedy</th>
</tr>
</thead>
<tbody>
<tr>
<td>@A</td>
<td>If the valves are not fully opened, temperature of the internal cooling water will rise leading to complete shut-down of the generator and x-ray output.</td>
<td>Open all the valves fully. If the generator shuts-down contact Soma for further assistance.</td>
</tr>
<tr>
<td>@B</td>
<td>If pump selection switch is not in the middle position, or the water level is below, the Haskris chiller will not run properly leading to generator shut-down and x-ray output.</td>
<td>Keep the Pump selection switch in the middle position. Add distilled water if needed.</td>
</tr>
<tr>
<td>@C</td>
<td>If the temperature indicator in Haskris chiller does not drop below 55°F, it means either that the building chilled water valves are not open properly or there is a problem with the chilled water.</td>
<td>Open the valves fully or contact Soma for further assistance.</td>
</tr>
<tr>
<td>@D</td>
<td>If helium level falls below 15 psi either in Primary or the Reserve cylinder, the Bio-Switch will automatically switch to the cylinder with more helium accompanied by an audible alarm.</td>
<td>Flip the silver switch toward the Primary or Reserve (whichever red light is ‘ON’) to silence the alarm.</td>
</tr>
<tr>
<td>@E</td>
<td>Flow rate can be adjusted by rotating the knob just below the Slow Purge Flow Gauge. Failure to flow Helium will damage the mirrors, and VERY EXPENSIVE to replace. So always check this!!!</td>
<td>Helium flow can be ascertained by watching the bubbler on MarCCD generator.</td>
</tr>
<tr>
<td>@F</td>
<td>If any of the Flow Control Lamps are off that indicates poor flow or obstructed tubes.</td>
<td>Contact Soma for further assistance.</td>
</tr>
<tr>
<td>@G</td>
<td>If vacuum ‘ALARM’ light comes ‘ON’. Press the ‘RESET’ button and try the vacuum sequence again.</td>
<td>If the problem persists contact Soma for further assistance.</td>
</tr>
<tr>
<td>@H</td>
<td>If the amber ‘READY light on X-RAY panel does not light up it could be due to: 1) the 12V/110mA Ready light on the Lamp Post has burnt out, 2) the OL-387 lamp for the X-RAY has burnt out, 3) both the lamps have burnt out.</td>
<td>Contact Soma for further assistance.</td>
</tr>
<tr>
<td>@I</td>
<td>If the Filament Current needle does not stay around 0.75A but fluctuates between 0.0 and 0.5A rapidly it means that the x-ray filament has burnt out. Experiment cannot proceed further.</td>
<td>Contact Soma for filament replacement and reschedule the data collection.</td>
</tr>
<tr>
<td>@J</td>
<td>Maximum allowable values for 0.3 x 3.00 mm² filament and a Copper anode (default) are: Tube Voltage: 40-44 kV Tube Current: 90-100 mA</td>
<td>Stay with the lower range to extend the filament life and prevent premature stoppage of your experiment.</td>
</tr>
<tr>
<td>@K</td>
<td>Entering the values in the log book will very beneficial in the event that the user wants to repeat the same but forgotten condition.</td>
<td></td>
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</tbody>
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