

SAFETY FEATURES, LAB-SPECIFIC STANDARD OPERATING PROCEDURES, AND USER RULES FOR THE CMRR RIGAKU D/MAX-IIB X-RAY DIFFRACTOMETER

I. EQUIPMENT LOCATION AND CONFIGURATION

The Rigaku Geigerflex model D/Max-IIB x-ray diffractometer system consists of a 2 kW x-ray generator (s/n LD2724N), a left-side wide angle goniometer (s/n H11892), a goniometer/detector control console (s/n L10971), and a computer with MDI DataScan software for remote operation. The diffractometer system is currently located in Lab 1B of the Center for Magnetic Recording Research. The goniometer and x-ray tube are located on top of the x-ray generator cabinet and are surrounded by a protective radiation enclosure. A 1.8 kW Co sealed tube source (K_{α} energy = 6.93 keV) is currently in use, but sealed tubes containing other target materials producing x-rays with different K_{α} energies (e.g., Cu, Cr, Fe, W etc.) can be accommodated. The x-ray tube is mounted inside a horizontal tube shield having two electronically controlled shutters which provide access to the incident x-ray beam. Shutter #1 is located on the left side of the tube shield and controls the incident beam directed towards the wide angle goniometer. Shutter #2 is located on the right side of the tube shield and is currently disconnected; it cannot be opened as long as it remains disconnected. Shutter #1 can be activated either manually or remotely, depending on the position of the SHUTTER #1 switch mounted on the front control panel of the x-ray generator. Manual control of the SHUTTER #1 switch is achieved by selecting either the OPEN or CLOSE position; the shutter will respond accordingly irrespective of any external control signals. When the SHUTTER #1 switch is placed in the EXT position, the shutter responds only to external control signals from the computer. Tube potential and current can be independently selected, up to 60 kV and 50 mA respectively, via controls on the generator front panel. In order to protect the tube from power levels that exceed the tube rating, power to the x-ray tube is automatically cut off when the product of potential and current exceeds the value selected on the generator front panel LOAD control (currently set to 1.8 kW for the Co sealed tube). Tube power is also interrupted when cooling water pressure and flow rate are not within specified limits, or whenever tube current and potential are not being properly regulated; power shutdowns caused by these events are indicated by LED lamps in the ALARM section of the generator front panel.

When operated in accordance with the lab-specific standard operating procedures and user rules described in this document, the x-ray diffraction instrument is considered an "enclosed beam system", meaning all possible primary and diffracted x-ray paths are completely enclosed so that no part of the human body can be exposed during normal operation.

I. SAFETY HAZARDS

The Rigaku x-ray diffraction instrument produces x-rays when the tube is energized. Warning signs indicating this hazard are attached to the front panel of the x-ray generator, the x-ray tube housing, the X-RAY ON lamp inside the radiation enclosure, and on the door of the laboratory where the instrument is located.

High voltages are present inside the fused electrical cut-off box mounted on the wall behind the instrument, inside the x-ray tube tower, and in the circuits and wiring located behind the doors of the x-ray generator cabinet underneath the front control panel. A warning sign indicating that a high voltage hazard is present is attached to the x-ray tube high voltage cable. Normal operation does not require operators to handle any components inside areas where high voltage hazards are present. Authorized operators will not be exposed to high voltages when the equipment is operated in accordance with the standard operating procedures and user rules outlined in this document.

III. SAFETY FEATURES AND DEVICES

X-ray On. Two red warning lamps indicate when the tube is energized, whether or not the shutters are open and regardless of power level applied to the tube. One lamp is inside the radiation enclosure mounted on a post attached to the ceiling above the x-ray tube, and the other is located on the generator front panel control section labeled X-RAY. The x-ray tube cannot be energized if either lamp is inoperative or removed.

Shutter Open. Each x-ray shutter has two red lamps which indicate when the shutter is open. One lamp is mounted on the shutter itself and located above the shutter opening, and the other is located on the generator front panel control section labeled SHUTTER directly above the switch controlling the selected shutter. As with the x-ray warning lamps, the shutter associated with the two shutter lamps cannot be activated if either lamp is inoperative or removed. In addition to being interlocked to these warning lamps, the shutter will not open if the specimen chamber cover is not in place or is improperly positioned. If open, the shutter will immediately close whenever the cover is removed. A mechanical interlock activates a secondary outer shutter door whenever the entrance slit housing is removed from the shutter assembly. This secondary shutter door will block the incident beam if the entrance slit housing is pulled out of the shutter assembly with the x-ray tube energized and the inner shutter door open.

Radiation Enclosure Interlocks. The top and rear panels of the radiation enclosure are steel. The front and side panels are made of transparent plastic impregnated with heavy metals to absorb any radiation which may have escaped from the incident and diffracted beam tunnels, the specimen chamber radiation shield, the slit housings, and the monochromator assembly. Lead plates are mounted to internal movable plastic panels on the left and right sides of the enclosure. These panels act as incident beam stops and are positioned to attenuate the direct beam if all fail-safe features have somehow been defeated and shutters are inadvertently opened without the required specimen radiation shields in place. The external movable front, side, and rear panels are interlocked to the shutter and power controls so that all shutters will close and tube power will be turned off immediately if the panels are opened while the tube is energized. The interlock can be defeated via use of a key switch on the generator front panel to activate the FAIL-SAFE RELEASE mechanism. Both a flashing red alarm light above the key switch and an audible alarm will turn on to indicate when the interlock mechanism has been defeated; both alarms will remain on until the radiation enclosure is returned to "fail-safe" mode by properly closing all panels. The FAIL-SAFE RELEASE key is stored in a designated location remote from the x-ray generator to prevent unauthorized individuals from opening the radiation enclosure while the x-ray tube is energized.

Standard Operating Procedures and User Rules. All authorized operators must agree to operate the x-ray diffractometer system in accordance with the lab-specific operating procedures and rules contained in this document. These procedures and rules are therefore considered an integral part of the x-ray diffractometer safety features. A reference copy of this document is available in the lab where the x-ray diffractometer is located.

IV. AUTHORIZED OPERATORS

Only authorized individuals may operate the Rigaku x-ray diffraction instrument, either manually or via computer control. Only the Principal Investigator responsible for the equipment and the lab radiation safety coordinator assigned by the Principal Investigator can authorize individuals to operate the x-ray diffraction system. A current list of authorized operators is posted on the side of the generator cabinet facing the computer.

Before being authorized to use the diffractometer, individuals must have successfully completed all of the necessary radiation safety courses and periodic refresher courses required by USCD Environmental Health & Safety, and received personal instruction on the safe operation of the x-ray diffraction instrument from either the Principal Investigator responsible for the equipment or the lab radiation safety coordinator assigned by the Principal Investigator.

All authorized users must agree to use the instrument in accordance with the lab-specific standard operating procedures and user rules outlined in this document, and all authorized users assume responsibility for any events resulting from their failure to comply with these procedures and rules.

V. TESTING OF SAFETY DEVICES

All x-ray diffractometer safety devices must be checked for proper operation at least every 30 days, and whenever a safety device malfunction is suspected. It is the responsibility of each operator to verify that safety devices have been checked within the past 30 days and, if not, to proceed with the following test procedure. The name of the operator conducting the test and the results of all safety device checks must be noted on the Safety Device Inspection Report attached to the x-ray generator cabinet. The instrument must not be used if any of the five safety device tests listed below fails.

Authorized operators must wear both finger and body dosimetry badges at all times while performing the safety device inspection. Finger badges must be worn on the hand that is closest to the location of the incident x-ray beam. The active area of the finger badge should be oriented so that it faces the location of the x-ray beam. Body badges should be worn at chest level.

Before performing the tests listed below, authorized operators must complete the following steps:

- i) Put on finger and body dosimetry badges.
- ii) Move both SHUTTER #1 and SHUTTER #2 switches to their CLOSE positions to prevent remote operation of the shutters.
- iii) Open the cooling water supply valves (return side first, supply side second).

- iv) Verify that the beam tunnels, the specimen chamber radiation shield, and the covers of the specimen chamber and slit housing are in place and properly positioned.
- v) Verify that both the tube potential (kV) and tube current (mA) selector knobs are positioned at their minimum settings (both knobs fully counterclockwise).
- vi) Turn on the x-ray generator by depressing the POWER ON switch on the front panel, **but do not apply power to the x-ray tube until instructed to do so in TEST 4.**
- vii) Ensure that all radiation enclosure panels are properly closed, that the XRAY READY light is at full brightness level, and that no LED's are illuminated in the generator front panel ALARM section. (If an ALARM LED is illuminated, check water flow first, and then try resetting the system by depressing the RESET switch. If any ALARM LED cannot be turned off by these means, the x-ray generator will not operate and the tests cannot be performed. In this case, turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; note the problem in the diffractometer logbook along with your name and notify the Principal Investigator and/or lab radiation safety coordinator).
- viii) Record your name, the date, and the x-ray generator clock hours ("x-ray on" hours) in the x-ray diffractometer logbook.

NOTE: The following safety device tests must be performed in the sequence listed below. Tests 4 and 5 must never be performed if any of the preceding tests fail.

TEST 1: Radiation Enclosure Interlocks

Open any one of the exterior plastic radiation enclosure panels. The XRAY READY light should turn off. Closing all panels should cause the READY light to illuminate again. Repeat this process for all seven of the exterior plastic panels.

If any panel fails this test, **do not proceed to Test 2; do not use the x-ray diffractometer;** turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; write "FAIL" in the box for Test 1 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; notify the lab safety coordinator and/or Principal Investigator.

If all checks of the radiation enclosure interlocks completed successfully, write "PASS" in the box for Test 1 on the Safety Device Inspection Report, and continue with Test 2.

TEST 2: Fail-Safe Release Mechanism

Check to make sure the XRAY READY light is illuminated. Get the FAIL-SAFE RELEASE key from its designated storage location and insert it into the switch on the generator front panel. Momentarily turn the key 90° clockwise. The audible alarm and the flashing red light above the key switch should now be activated. Open one or more of the seven exterior plastic radiation enclosure panels. The audible alarm, flashing red light above the key switch, and the XRAY READY light should remain on. Next, close all

exterior panels. Both the audible alarm and the flashing red light should turn off; the XRAY READY light should remain illuminated.

If any part of this test fails, ***do not proceed to Test 3; do not use the x-ray diffractometer;*** turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 2 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; notify the lab safety coordinator and/or Principal Investigator.

If all checks of the fail-safe release mechanism completed successfully, write "PASS" in the box for Test 2 on the Safety Device Inspection Report, and continue with Test 3.

TEST 3: X-RAY ON Lamp Interlocks

Ensure that the XRAY READY light is illuminated. Unscrew the red plastic cover from the X-RAY ON indicator on the generator front panel and remove the lamp (don't lose the cover or the lamp!). With all radiation enclosure panels properly closed, the READY light should turn off when the lamp is removed. With the lamp still removed, depress the X-RAY ON switch. Power should *not* be applied to the tube, as indicated by zero readings on both the tube potential and tube current front panel meters. If power is applied to the tube, immediately shut off power to the generator by depressing the red POWER OFF switch and proceed to the next paragraph. If the tube did not energize, replace the lamp and tighten the red plastic cover. The READY light should turn on again if all radiation enclosure panels are properly closed. With the READY light illuminated, momentarily turn the FAIL-SAFE RELEASE key 90° clockwise. The audible alarm and the flashing red light above the key switch should now be activated and the READY light should remain on. Open one or more of the seven exterior plastic radiation enclosure panels. Unscrew the red plastic cover from the X-RAY ON indicator mounted on the post above the x-ray tube and remove the lamp. Verify that the READY light has turned off. Return all panels to their proper closed positions (both the audible alarm and the flashing red light should turn off; this is important because improperly positioned panels also cause the XRAY ON lamp to turn off), and verify that the READY light *remains off*. Depress the X-RAY ON switch. Power should *not* be applied to the tube, as indicated by zero readings on both the tube potential and tube current front panel meters. If power is applied to the tube, immediately shut off power to the generator by depressing the red POWER OFF switch and proceed to the next paragraph. If the tube did not energize, open one or more of the radiation enclosure panels, replace the lamp and tighten the red plastic cover onto the post. Return all radiation panels to their proper closed positions. The READY light should now turn on again.

If any part of this test fails, ***do not proceed to Test 4; do not use the x-ray diffractometer;*** turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 3 on the Safety Device Inspection Report; attach a note to the generator front panel indicating

that the instrument has failed a Safety Device Check and is not safe for use; notify the lab safety coordinator and/or Principal Investigator.

If all checks of the X-RAY ON lamp interlocks completed successfully, write "PASS" in the box for Test 3 on the Safety Device Inspection Report, cycle power to the x-ray generator by first depressing the red POWER OFF switch and then the POWER ON switch, but **do not apply power to the x-ray tube**. Continue with Test 4.

TEST 4: Shutter Interlocks

Before conducting this test, verify the following:

- a. The x-ray tube is *not* energized (both X-RAY ON lamps are off)
- b. The entrance slit housing cover is in place and correctly positioned.
- c. The specimen chamber radiation shield cover is in place and correctly positioned.
- d. All radiation shield panels are properly closed (XRAY READY lamp is illuminated).

Shutter Operation with No Power Applied to the X-Ray Tube. After checking the above items, move the SHUTTER #1 switch to the OPEN position. The shutter should *not* open. Return the SHUTTER #1 switch to the CLOSE position. Repeat this check with the SHUTTER #2 switch. **SHUTTER #2 is electrically disconnected and should never open.**

If either shutter opens when the tube is not energized, **do not proceed with other checks in this test; do not use the x-ray diffractometer**; turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; notify the lab safety coordinator and/or Principal Investigator.

If both shutters did not open, continue with the checks described in the next paragraph.

Shutter Operation with the X-Ray Tube Energized (Part 1). Verify that both SHUTTER #1 and SHUTTER #2 switches are in their CLOSE positions, and that the XRAY READY lamp is illuminated. Unscrew the red plastic cover above the SHUTTER #1 switch and remove the lamp for this shutter. Apply power to the x-ray tube by depressing the X-RAY ON switch and verify that both X-RAY ON lamps are illuminated; the XRAY READY light should turn off. Allow the tube potential and current to stabilize for about 15 seconds. **Do not increase the tube potential and current above their minimum settings.** Move the SHUTTER #1 switch to the OPEN position. The shutter should open and immediately close. The sealed lamp on the left shutter assembly mounted on the x-ray tube enclosure should *not* be illuminated. Move the SHUTTER #1 switch to the CLOSE position and replace the SHUTTER #1 lamp. Again move the SHUTTER #1 switch to the OPEN position. The shutter should now open and both the SHUTTER #1 lamp and the sealed lamp on the left shutter assembly should be illuminated. Move the SHUTTER #1 switch to the CLOSE position. Both Shutter #1 lamps should turn off. Now move the SHUTTER #2 switch to the OPEN position. The

right shutter should *not* open even with both lamps in place. **If SHUTTER #2 opens, immediately depress the red POWER OFF switch to turn off power to the entire x-ray generator. SHUTTER #2 is electrically disconnected and should never open.** Move the SHUTTER #2 switch to the CLOSE position.

If either shutter did not pass these checks, ***do not proceed with the remaining checks in this test; do not use the x-ray diffractometer;*** turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator.

If both shutters passed these checks, continue with the checks described in the next paragraph.

Shutter Operation with the X-Ray Tube Energized (Part 2). Prepare either the TA Associates or the Ludlum radiation survey meter (Geiger-Mueller counter) as follows:

- a. Inspect the UCSD Radiation Safety Office calibration label on the selected survey meter body to verify that the meter calibration has not expired.
- b. Remove the protective cover from the face of the survey meter counter chamber if one is installed.
- c. Rotate the left knob on the TA Associates survey meter to the x1 position; on the Ludlum meter, turn the knob to the x0.1 position.
- d. Turn the volume control of the TA Associates meter clockwise to the MAX position; on the Ludlum meter flip the AUD switch to ON.
- e. Check the batteries of the TA Associates meter by depressing the red BATTERY CHECK button below the volume control knob and verifying that the meter needle moves to the BAT position; on the Ludlum meter, rotate the knob to the BAT and verify that the meter needle moves to the BAT TEST (return knob to the x0.1 position after checking the batteries in the Ludlum meter).

After performing these steps, measure the background radiation level and record its value in the comment section on the Safety Device Inspection Report (the background reading should be ~50 counts/minute if the meter calibration is current and good batteries are installed).

Verify that both x-ray tube shutters are closed via visual inspection of all shutter lamps (all shutter lamps, both on the front panel and on the two shutters attached to the tube shield, should be off). After verifying both shutters are closed, activate the FAIL-SAFE RELEASE mechanism by momentarily turning the key 90° clockwise. Open any one or more of the exterior plastic radiation enclosure panels and use the radiation survey meter to verify that radiation levels around the specimen chamber remain at background levels. If the survey meter indicates radiation levels are not above background levels, remove the specimen chamber cover, and again check the radiation levels around the specimen chamber with the radiation survey meter. **(If the radiation survey meter indicates the presence of radiation levels above background with the sample chamber cover either in place or removed, immediately depress the red POWER OFF switch to turn off power to the entire x-ray generator; shut off water flow to the x-ray generator;**

remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator. **Do not use the x-ray diffractometer**). If the radiation survey meter indicates radiation levels are not above background, continue with the test, close all radiation shield panels and verify that both the audible alarm and the flashing red light above the fail-safe key switch have turned off. Move the SHUTTER #1 switch to the OPEN position. The shutter should not open. Both the SHUTTER #1 lamp and the sealed lamp mounted on the left shutter assembly should remain off. Move the SHUTTER #1 switch to the CLOSE position.

If any of the above checks failed, **Do not use the x-ray diffractometer**; turn off power to the x-ray tube by depressing the XRAY OFF switch; turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator.

If all checks of the shutter interlock completed successfully, **verify that both x-ray tube shutters are closed** via visual inspection of all shutter lamps; all shutter lamps, both on the front panel and on the two shutters attached to the tube shield, should be off. After verifying both shutters are closed, activate the FAIL-SAFE RELEASE mechanism by momentarily turning the key 90° clockwise. Open any one or more of the exterior plastic radiation enclosure panels and use the radiation survey meter to verify that radiation levels around the specimen chamber remain at background levels. (If the radiation survey meter indicates the presence of radiation levels above background, immediately depress the red POWER OFF switch to turn off power to the entire x-ray generator; shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator. **Do not use the x-ray diffractometer**). If the radiation survey meter indicates radiation levels are not above background replace the specimen chamber cover. Close all radiation shield panels and verify that both the audible alarm and the flashing red light above the FAIL-SAFE RELEASE key switch have turned off. Do not turn off power to the x-ray tube. Write "PASS" in the box for Test 4 on the Safety Device Inspection Report, and continue to Test 5.

TEST 5: Radiation Enclosure Leak Check

Make sure that that one of the survey meters is on and ready for use, the specimen chamber cover is in place, both the flashing red light and audible FAIL-SAFE RELEASE alarms are off, and the x-ray tube is operating at minimum applied potential and current as outlined in the last paragraph of Test 4, above. If these conditions have been met, alternately increase the tube potential and current to their maximum allowable settings (50 kV and 32.5 mA for the Co tube). Move the SHUTTER #1 switch to the OPEN position and verify that the shutter is now open by visual inspection of both SHUTTER #1 warning lamps. Position the survey meter's detector surface parallel to and about 1/2" from the radiation shield exterior surface and observe the meter readings as the detector is slowly moved over all exterior surfaces accessible to personnel.

A failure of the radiation shield is indicated by any steady reading three times or greater than the background radiation level. If such readings are observed, close the shutter by moving the SHUTTER #1 switch to the CLOSE position. *Before* searching for the source of the elevated reading, alternately reduce the tube potential and current to their minimum settings, turn off power to the x-ray tube by depressing the XRAY OFF switch, and record the anomalous high reading in the comment section on the Safety Device Inspection Report. With tube power off, visually inspect the beam tunnels, specimen chamber shield, and exterior panels to identify any misaligned or improperly positioned components. Correct any obvious problems, but *do not* modify any shield component or defeat any safety device without written approval of the Principal Investigator or lab radiation safety coordinator.

After correcting any misaligned panel or shield component, close all enclosure panels (READY light should turn on), verify that both the flashing red light and the audible FAIL-SAFE RELEASE alarms are off, depress the X-RAY ON switch (allow the tube potential and current to stabilize for about 15 seconds), alternately increase the tube potential and current to their maximum allowable settings, and repeat the radiation survey.

If the corrections have failed to reduce the elevated readings to background levels, ***do not use the x-ray diffractometer***; close the shutter by moving the SHUTTER #1 switch to the CLOSE position; alternately reduce the tube potential and current to their minimum settings; turn off power to the x-ray tube by depressing the XRAY OFF switch; turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 5 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that instrument has failed a Safety Device Check and is not safe for use; turn off the radiation survey meter; notify the lab safety coordinator and/or Principal Investigator.

If no leaks were detected during the radiation shield survey, write "PASS" in the box for Test 5 on the Safety Device Inspection Report and record the measured exterior radiation level in the comment section. Turn off the radiation survey meter if it is no longer needed. The x-ray diffractometer can now be used for measurements in accordance with the standard operating procedures outlined in the Section VI, below.

If the diffractometer will be used immediately after the safety device inspection, it is not necessary to turn off the x-ray tube. In this case, turn on power to the goniometer/detector control console next to the computer (do not change the settings of any other switch on this console), launch the MDI DataScan program and allow the goniometer to finish its DATUM process, and proceed to Part B of Section VI, below.

If the diffractometer will not be used after completing the safety device inspection, alternately reduce the tube potential and current to their minimum settings and turn off power to the x-ray tube by depressing the XRAY OFF switch; in the logbook record the generator clock reading ("x-ray off" hours) as well as the maximum tube potential and current levels used during this test; turn off power by depressing the red POWER OFF switch and shut off water flow to the x-ray generator; turn off the radiation survey meter; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage location.

VI. STANDARD OPERATING PROCEDURES

The standard operating procedures listed in this section are subdivided into three parts. Part A lists the steps to be taken when beginning with a powered off x-ray diffraction system, Part B lists the procedure for changing specimens and/or goniometer slits with the x-ray tube already energized, and Part C provides the procedure for a normal shutdown of the x-ray diffraction system after concluding measurements.

None of these operating procedures authorize users to operate the x-ray diffraction system as an "open beam system". Authorized operators are therefore not permitted to perform any repairs, adjustments, or alignments of the x-ray diffraction system with the x-ray tube energized and the shutters open while the FAIL-SAFE RELEASE mechanism is activated. Such actions require prior review and written permission of the Principal Investigator responsible for the instrument, or the lab radiation safety coordinator assigned by the Principal Investigator.

All authorized operators must wear both finger and body dosimetry badges while operating the diffractometer whenever the x-ray tube is energized. Finger badges must be worn on the hand that is closest to the location of the incident x-ray beam. The active area of the finger badge should be oriented so that it faces the location of the x-ray beam. Body badges should be worn at chest level.

A. Procedure for Powering Up the X-ray Diffractometer System:

1. Put on finger and body dosimetry badges.
2. Check the Safety Device Inspection Report and verify that an inspection has been performed within the last 30 days.
 - a. If an inspection is not required, continue with step 3 of Part A of this section (this procedure).
 - b. If a safety device inspection has not been conducted during the preceding 30 days, perform the inspection as instructed in Section IV, above. After all safety devices have passed the required tests, and only if the x-ray tube is still energized following these tests, go to step 1 of Part B of this section, below, to insert a specimen and to insert/change goniometer slits.

3. Check the positions of both SHUTTER #1 and SHUTTER #2 switches and move both to their CLOSE positions, if necessary, to prevent accidental external control of the shutters via computer commands.
4. Check both the tube potential (kV) and tube current (mA) selector knobs to verify that they are positioned at their minimum settings (both knobs fully counterclockwise).
5. Open the cooling water supply valves (return side first, supply side second)
6. Apply power to the x-ray generator by depressing the POWER ON switch. **Do not apply power to the x-ray tube at this time** (it is unnecessary for the tube to be energized when first inserting a specimen or selecting goniometer slits).
7. With all radiation enclosure panels properly closed, check that the READY light is at full brightness level and that no ALARM LEDs are illuminated. If an ALARM LED is illuminated, check the water flow first, and then try resetting the system by depressing the RESET switch. If any ALARM LED cannot be turned off by these means, the x-ray generator will not operate. In this case, turn off power to the generator by depressing the red POWER OFF switch, close the cooling water valves (supply side first, return side second), record the problem in the diffractometer logbook, and notify the Principal Investigator and/or lab radiation safety coordinator.
8. Turn on power to the goniometer/detector control console next to the computer (do not change the settings of any other switch on this console).
9. At the computer, launch the MDI DataScan program and allow the goniometer to finish its DATUM process.
10. Open one or more of the exterior plastic radiation enclosure panels, insert the specimen in the specimen chamber, and insert/change goniometer slits as desired.
11. Verify that the covers of both the specimen chamber and the entrance slit housing are in place and properly positioned, and then close all exterior plastic radiation enclosure panels. If all panels are properly closed, the READY light should be at full brightness.
12. Record your name, the date, and the x-ray generator clock reading (“x-ray on” hours) in the x-ray diffractometer logbook.
13. Apply power to the tube by depressing the XRAY ON switch, and allow both tube potential and current to stabilize for about 15 seconds.
14. Increase the x-ray tube power to the desired level by alternately increasing the tube potential and tube current in one-step increments. Do not exceed 50 kV potential and 32.5 mA current settings for the Co tube.
15. Move the SHUTTER #1 switch to the EXT position to permit shutter control via computer commands, and use the MDI DataScan program to perform the desired experiment.
16. To change specimens or insert/change goniometer slits with the x-ray tube energized, go to step 1 of Part B of this section, below.
17. To turn off the x-ray diffractometer and remove samples, go to step 1 of Part C, of this section, below.

B. Procedure for Changing Specimens and Goniometer Slits when the X-Ray Tube is Energized:

1. You should be wearing both finger and body dosimetry badges. If this is not the case, put them on now.
2. Move the SHUTTER #1 switch to the CLOSE position to prevent accidental external control of the shutters via computer commands.
3. Prepare one of the radiation survey meters as described on page 7 (*Test 4: Shutter Interlocks; Shutter Operation with the X-Ray Tube Energized (Part 2)*).
4. ***Verify that both x-ray tube shutters are closed*** via visual inspection of all shutter lamps (all shutter lamps, both on the front panel and on the two shutters attached to the tube shield, should be off).
5. After verifying both shutters are closed, remove the FAIL-SAFE RELEASE key from its designated storage location, insert it in the key switch, and activate the FAIL-SAFE RELEASE mechanism by momentarily turning the key 90° clockwise.
6. Open any one or more of the exterior plastic radiation enclosure panels. Before removing the sample chamber cover, survey the surroundings of the sample chamber with the radiation survey meter to ensure that radiation levels are not above background level. If radiation levels are not above background, remove the sample chamber cover and again survey the area with the meter. ***(If the radiation survey meter indicates the presence of radiation levels above background with the sample chamber cover either in place or removed, immediately depress the red POWER OFF switch to turn off power to the entire x-ray generator; shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write 'FAIL' in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator. Do not use the X-ray diffractometer until the reason for the excessive radiation level has been determined and corrected by the Principal Investigator or the lab radiation coordinator designated by the Principal Investigator).*** If the meter indicates radiation levels are not above background level, change samples and insert/change goniometer slits as desired.
7. Verify that the covers of both the specimen chamber and the entrance slit housing are in place and properly positioned, and then close all exterior plastic radiation enclosure panels. With all panels properly closed, both the flashing red FAIL-SAFE RELEASE alarm light and the audible alarm will turn off.
8. Remove the FAIL-SAFE RELEASE key from the key switch and place it in its designated storage location.
9. If necessary, adjust the tube potential and tube current to the desired levels.
10. Move the SHUTTER #1 switch to the EXT position to permit shutter control via computer commands, and use the MDI DataScan program to perform the desired experiment.

11. To turn off the x-ray diffractometer and remove samples, go to step 1 of Part C of this section, below.

C. Procedure for Normal Shutdown of the X-Ray Diffractometer System:

1. If you are not already wearing your finger and body dosimetry badges, you have failed an intelligence test. Put them on now or you will no longer continue to be an authorized user.
2. Decrease the x-ray tube power by alternately reducing the tube potential and tube current in one-step increments until both have reached their minimum settings (both control knobs fully counterclockwise).
3. Depress the XRAY OFF switch, and verify that the tube is not energized by inspecting both XRAY ON lamps. Allow the cooling water to flow until step 7, below.
4. In the diffractometer logbook record the maximum tube potential and current levels used, and record the x-ray generator clock reading ("x-ray off" hours) next to the "x-ray on" entry previously recorded at step 12 of Part A of this section, above, or step viii of Section V, Testing of Safety Devices.
5. Open the exterior plastic radiation enclosure panels, and remove the specimen.
6. Replace the specimen chamber cover in its correct position, close all radiation shield panels, and turn off power to the generator by depressing the red POWER OFF switch.
7. Close both cooling water valves (supply side first, return side second).
8. Exit the MDI DataScan program.
9. Turn off power to the goniometer/detector control console next to the computer.

VII. EMERGENCY PROCEDURES

A. Emergency Shutdown in Case of Abnormal Operation and It Is Safe to Approach the Instrument:

1. Depress RED switch on the front panel (POWER OFF switch).
2. Turn off main power at the electrical cutoff on the wall behind the instrument.
3. Close both cooling water valves (supply side first, return side second).
4. Discontinue further use of the instrument.
5. Notify Principal Investigator and/or designated lab radiation safety coordinator.

B. Emergency Shutdown In Case of Abnormal Operation and It IS NOT Safe to Approach the Instrument (e.g., standing water surrounding instrument):

1. Shut off electrical power to the instrument at breaker panel 1L10A in the hallway outside of the laboratory (across from Room 105) using breaker switches 34 and 36.
2. Notify Environmental Health and Safety. If life-threatening, call (858) 534-4357 (4-HELP).
3. Only if it is now safe to approach the instrument, close both cooling water valves (supply side first, return side second).
4. Discontinue further use of the instrument.
5. Notify Principal Investigator and/or designated lab radiation safety coordinator.

C. In Case of Suspected, Accidental, or Abnormal Exposure to Radiation

1. Depress RED switch on the front panel (POWER OFF switch).
2. Notify Radiation Safety. If life-threatening, call (858) 534-4357 (4-HELP).
3. Turn off main power at the electrical cutoff on the wall behind the instrument.
4. Close both cooling water valves (supply side first, return side second).
5. Discontinue further use of the instrument.

VIII. LAB-SPECIFIC OPERATING RULES

All persons authorized to operate the Rigaku x-ray diffraction instrument described above must have successfully completed all of the necessary radiation safety courses and periodic refresher courses required by USCD Environmental Health & Safety.

All persons authorized to operate the Rigaku x-ray diffraction instrument described above must have received personal instruction on the safe operation of the x-ray diffractometer from either the Principal Investigator responsible for the equipment, or the lab radiation safety coordinator assigned by the Principal Investigator.

All authorized operators must wear finger and body radiation dosimeter badges whenever the x-ray tube is energized. Finger badges must be worn on the hand that is closest to the location of the incident x-ray beam. The active area of the finger badge should be oriented so that it faces the location of the x-ray beam. Body badges should be worn at chest level.

All authorized operators must use the x-ray diffractometer logbook, signing their name, and entering the date of use, the maximum tube potential and current used, the generator clock readings (x-ray "on" and "off" hours) to indicate the time the x-ray tube was energized, and any operating problems that were experienced.

All authorized operators are required to verify that all safety devices have been inspected and have passed all required tests within the 30 day period preceding their use of the x-ray diffractometer. Authorized operators must perform the required tests whenever it is necessary to maintain this inspection frequency, and whenever a safety device malfunction is suspected. Results of all safety checks must be noted on the Safety Device Inspection Report attached to the x-ray generator cabinet. All operators must discontinue use of the x-ray diffractometer if any safety device malfunctions or has failed during a regularly scheduled test, and must notify all other users in event of any safety device failure.

All authorized operators must use the x-ray diffractometer in a manner consistent with the "enclosed beam system" designation. This means that all authorized operators must verify via visual inspection of all shutter lamps that both x-ray tube shutters are closed *before* defeating the radiation enclosure safety interlock via the FAIL-SAFE RELEASE mechanism and *before* opening the radiation enclosure while the tube is energized. Only then may the FAIL-SAFE RELEASE mechanism be used for changing specimens and inserting/changing goniometer slits with the x-ray tube energized. **NO EXPERIMENTS OR ADJUSTMENTS WITH AN "OPEN-BEAM SYSTEM" ARE PERMITTED AT ANY TIME WITHOUT PRIOR REVIEW AND WRITTEN APPROVAL FROM THE PRINCIPAL INVESTIGATOR OR THE LAB RADIATION SAFETY COORDINATOR.**

All authorized operators must never leave the x-ray diffraction unit unattended when the radiation enclosure safety interlock is defeated and the tube is energized.

All authorized operators must place the FAIL-SAFE RELEASE key in its designated storage location after changing samples or goniometer slits, and before leaving the x-ray diffractometer unattended.

Modifications to any of the x-ray diffraction safety devices, via means other than proper use of the FAIL-SAFE RELEASE mechanism, are prohibited.

IX. OPERATOR AGREEMENT

By signing below, I certify the following:

I have read, understood, and agree to operate the x-ray diffractometer in accordance with the lab-specific operating procedures and rules described in this document,

I accept responsibility for any events resulting from my failure to comply with these procedures and rules, and

I have received personal instruction on the safe operation of the x-ray diffraction instrument from (check one) the _____ Principal Investigator responsible for equipment, or _____ lab radiation safety coordinator.

Authorized Operator: _____
(Print name) (Signature) (Date)

Lab Radiation Safety Coordinator: _____
(Print name) (Signature) (Date)

Principal Investigator: _____
(Print name) (Signature) (Date)

REFERENCE ONLY OCT 17 2011

ALL XRD USERS

Effective immediately, UCSD Radiation Safety requires all operators to use a radiation survey meter to ensure that radiation surrounding the sample chamber is not above background levels when changing samples with the XRD tube energized. This change has been incorporated into the new updated Lab Specific Operating Procedures now attached to the XRD generator cabinet (see procedures for Shutter Interlock Test 4 beginning on page 6, and the procedure for changing specimens and slits when the XRD tube is energized on page 12). The new procedure states:

Before removing the sample chamber cover, survey the surroundings of the sample chamber with the radiation survey meter to ensure that radiation levels are not above background level. If radiation levels are not above background, remove the sample chamber cover and again survey the area with the meter. *(If the radiation survey meter indicates the presence of radiation levels above background with the sample chamber cover either in place or removed, immediately depress the red POWER OFF switch to turn off power to the entire x-ray generator; shut off water flow to the x-ray generator; remove the FAIL-SAFE RELEASE key from the switch and return it to its designated storage area; write "FAIL" in the box for Test 4 on the Safety Device Inspection Report; attach a note to the generator front panel indicating that the instrument has failed a Safety Device Check and is not safe for use; record the x-ray generator clock reading ("x-ray off" hours) in the x-ray diffractometer logbook; notify the lab safety coordinator and/or Principal Investigator. **Do not use the X-ray diffractometer until the reason for the excessive radiation level has been determined and corrected by the Principal Investigator or the lab radiation coordinator designated by the Principal Investigator.*** If the meter indicates radiation levels are not above background level, change samples and insert/change goniometer slits as desired.

By continuing to use the XRD unit, you are agreeing to abide by this new requirement.