



RIGAKU MINIFLEX II

Standard Operating Procedure (SOP)

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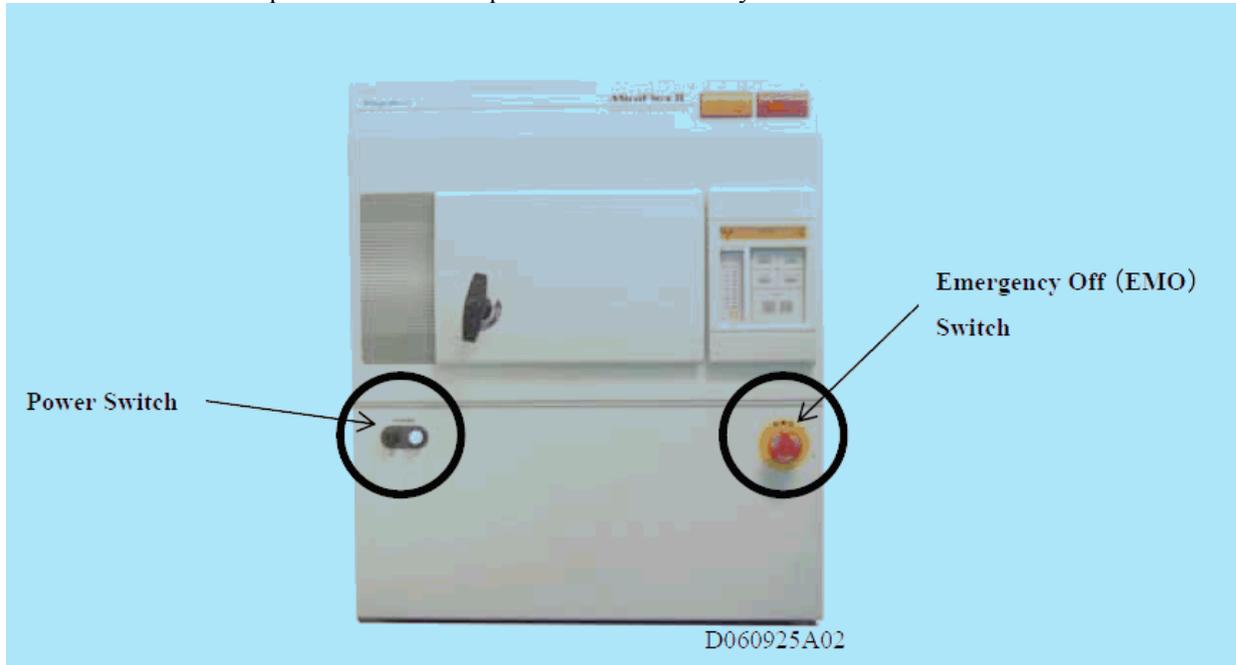
Student Health Services
Oregon state University
Corvallis, OR
97331-5801
Concern: 737-WELL
Schedule App: 737-9355
Medical Advice: 737-2724
IN CASE OF EMERGENCY DIAL 911

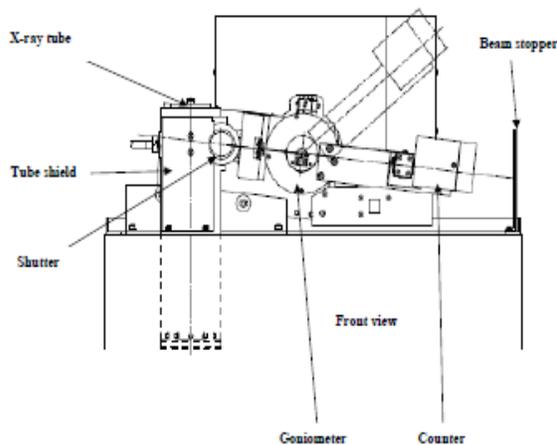
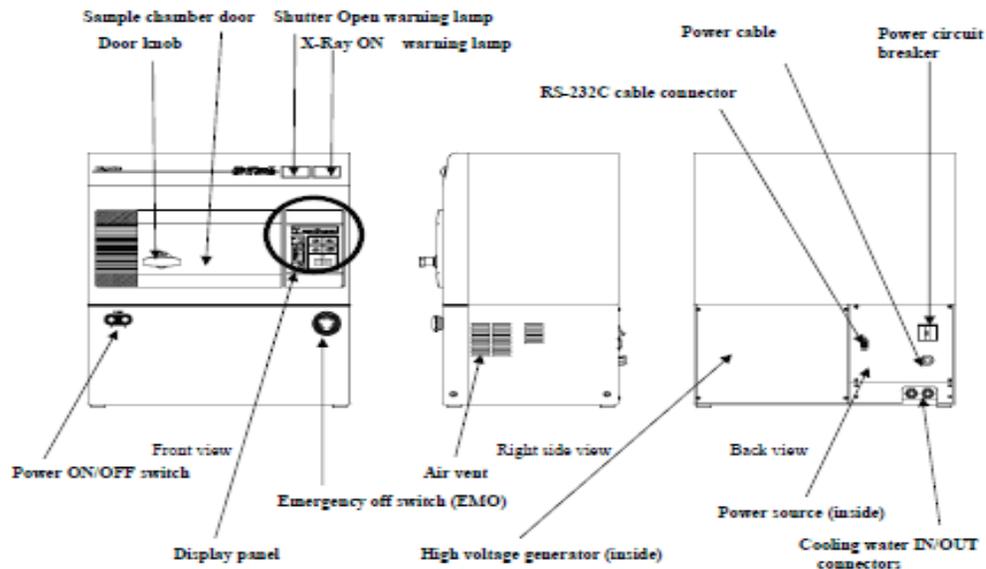
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INTRODUCTION

The Rigaku Miniflex II can be used for a variety of applications from diffraction pattern comparison of polycrystalline materials such as powder samples and metal plates to qualitative and quantitative analyses and quality management of raw materials and products. This Miniflex system also offers use of a real-time angle correction system, a single sample holder, an automated six sample changer (used in standard operation), as well as a monochromator addition that captures X-rays other than Cu K α for use in analysis.

The main components and their specifications of this system are listed and illustrated below:





The specifications of the Miniflex II Components:

X-Ray Generator

- (1) Tube output voltage: 30 kV (fixed)
- (2) Tube output current: 15 mA (fixed)
- (3) High-voltage generation method: High-frequency Cockcroft-Walton method
- (4) Stability: Within $\pm 0.05\%$ for both the tube voltage and tube current, with reference to $\pm 10\%$ of input power variation

Tube shield

- (1) Applicable X-ray tube: Toshiba A-20, 41, Phillips PW22XX, etc.
- (2) Focusing-position adjustment mechanism: ± 1 mm, continuously variable through the use of a spring plate (patent pending)
- (3) X-ray shutter: Mechanical rotary shutter interlocked with the radiation-protection cabinet door (patent pending)

(4) $K\beta$ suppression filter: Ni-filter (for the Cu-tube). Note that at present the Miniflex uses a graphite monochromator not the Ni-filter.

Goniometer

- (1) Type: Vertical
- (2) Goniometer radius: 150mm
- (3) Scanning axis: $\theta/2$. interlocked
- (4) 2θ scanning range: $-3^\circ \sim +145^\circ$
- (5) Positioning speed: $500^\circ/\text{min}(2\theta)$
- (6) Scanning speed: $0.01\sim 100^\circ/\text{min}$. (Keyboard input)
- (7) Slit: DS 1.25° (Building in), 0.625° , Height limitation of 10mm
Auxiliary DS Variable slit to keep the radiation width within the "sample width + α " range
SS 1.25° ,fixed (AS:Antiscatter slit)
RS 0.3mm,fixed
Roller slit $\pm 2.5^\circ$ (divergence angle)
- (8) Datum angle: $2\theta = 10^\circ$
- (9) X-ray take-off angle: 6° ,fixed

Detector (SC)

- (1) Scintillator: NaI (T1)
- (2) Window material: Be
- (3) Effective window diameter: $\phi 23$ mm
- (4) Dimension: L 80 mm

Counting control circuit

- (1) $\theta/2$ θ axis minimum step angle: $0.01^\circ/(2\theta)$
- (2) Counter high-voltage power source: 0 to 1000 V
- (3) Wave height discriminator circuit: Fixed pulse height system
- (4) Scaler: 32-bit counter

(5) Indication functions

- a. SCAN: This lamp is on while the goniometer is scanning.
- b. HV: Lights when high voltage is applied to the detector
- c. PHA: Lights when the detector detects and counts X-rays
- d. HVL: When the tube voltage exceeds 31.5 kV, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- e. LVL: The moment the tube voltage falls to 27.4 kV, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- f. HTC: The moment the tube currents exceeds 15.75 mA, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- g. LTC: The moment the tube current falls to 13.7 mA, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- h. HFC: The moment a filament current in excess of approximately 4.3 A is applied to the X-ray tube, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- i. LFC: The moment the filament current of the X-ray tube falls to approximately 3 A, X-rays are cut off, this indicator lights, and the alarm buzzer sounds.
- j. CW: Lights when the cooling-water flow falls below 3.0 l/min. Simultaneously, the alarm buzzer sounds, and X-rays are cut off.

The alarm buzzer does not sound if X-rays are off.

- k. X-RAY: Remains lit when the X-ray tube is generating X-rays
- l. SHUTTER: This lamp lights when the sample chamber door is shut and you turn the door knob clockwise with the X-ray shutter opened.
- m. READY: Specification A (Used for Standard Operating Conditions):
This lamp is on when the lamps “d” to “j” above are off (no alarm), the door is shut, and the shutter is opened.
Specification B: This lamp is on when there is no alarm or other anomaly.

Lamp Indications

- When the X-rays are being generated and the shutter is open:
The PHA lamp stays illuminated. (The shutter is open and strong X-rays are counted.)



- When the door is being unlocked
The PHA lamp is off. (X-rays are not counted because the shutter is closed. Since natural counting is performed, the lamp may sometimes light intermittently.)
* The SHUTTER lamp stays on for safety until the door knob is turned counterclockwise completely to release the locking.



- When the door is completely unlocked (X-rays are turned off and the X-RAY lamp and SHUTTER lamp go off).



Cooling-Water Unit

Flow rate: A flow rate of 3.0 l/min. or less is detected on the water-drainage side.

Hose: $\Phi 9$ I.D. x $\Phi 15$ O.D. x 10 m in length each (with Teflon blade), provided with four hose bands

INFORMATION ON SAFETY

A. Personal Protective Equipment (PPE)

The Miniflex generates strong X-rays that can be harmful to the human body upon exposure. Furthermore, the X-ray tube window is made of metallic beryllium that is harmful upon contact or inhalation. In response to this, the following PPE are mandatory for use of the Miniflex II:

- Radiation Dosimeter Badge and Finger rings (left and right) :
 - These are administered by the OSU Radiation Safety Department upon completion of their Radiation Safety Seminar and the Miniflex II training by a Miniflex II Administrator. Please contact a Miniflex II Administrator for further questions on this.

Recommended PPE to also be used while operating the Miniflex II are:

- Safety Glasses
- Gloves
- Lab Coat

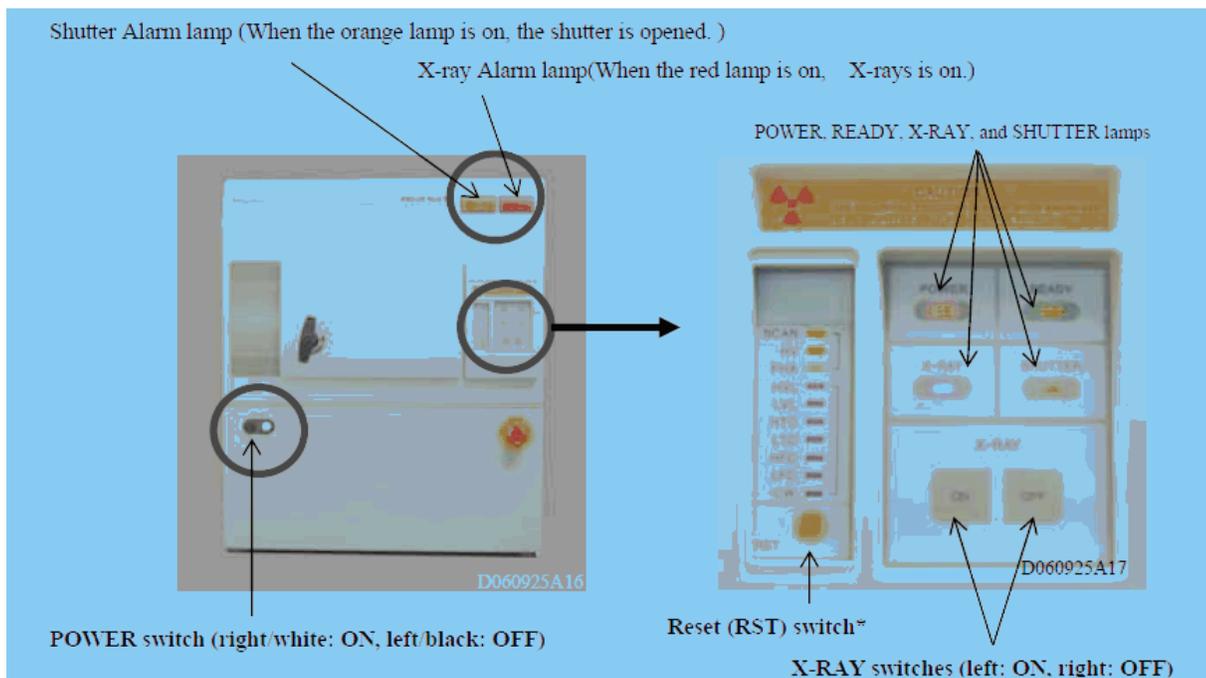
For information on the safety precautions and features relating to the Miniflex II system please refer to *Appendix 1*

PROCEDURE FOR MEASUREMENT

(With Six Sample Changer and Monochromator Option)

A. System Start Up

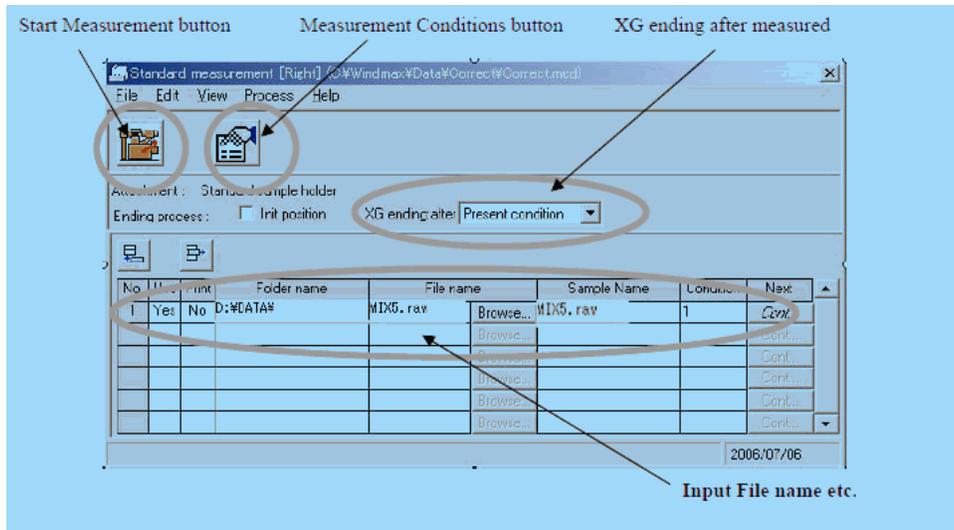
1. Turn on the water pump (Haskris Chiller System).
2. Turn on power to the Miniflex II system using the power button shown in the diagram below.



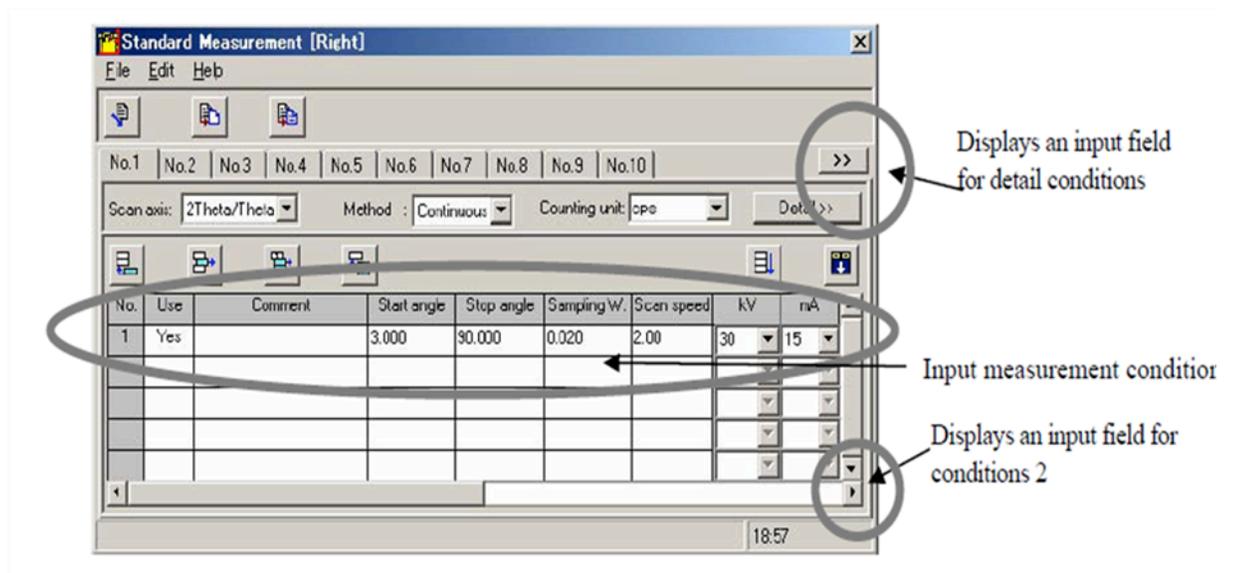
3. Insert sample(s) carefully onto the sample holder's magnetic troughs. Be careful to ensure that no sample spills and note the number of the trough corresponding to each sample.
4. Close the door, and turn the knob clockwise to lock it. With the rotation of the knob, the shutter opens, and the warning light lamp of the shutter on the right of the device will illuminate as shown in the diagram above.
5. Check that the READY lamp on the upper right of display panel is on. When this lamp is on, the system is ready to generate X-rays

B. Measurement

1. Select the Rigaku-Standard Measurement icon on the computer desktop. This will open the Standard Measurement as shown:



2. In this window, input the names of the folder and file where you want to save measurement data and the name of the sample. Within the C:\Windmax\Data file all users will have a folder which they may store their diffraction data. If you do not have a data folder please contact a Miniflex Administrator.
3. Each user is assigned a Measurement Condition number in which they may input different measurement settings to be used. If you do not have a Measurement Condition number, please contact a Miniflex Administrator.
4. The measurement conditions button will lead to the following window in which users can select which settings to use within their particular Measurement Condition number.



5. Selecting “Yes” under the use column identifies the settings to be used for the Measurement Condition.
6. **Within the Standard Measurement window ensure that the sample numbers listed in Sample column (the utmost right column) correspond to the trough number on the sample holder in which your samples lie. This is crucial as it determines the sample location at which x-ray beams will be targeted by the instrument.**
7. When certain all specifications for your measurements are set, click the “Start Measurement” button.
8. You can view your diffraction pattern in real time in the Measurement window that appears on the screen.

C. Shutting Down the Miniflex

1. When Measurement completes, the X-ray lamp will turn off and the PHA light will also turn off.
2. The diffraction data will be available in the folder assigned. The data may be viewable in using software such as JADE available on the PC.
3. Make sure to turn off the “Yes” selection in Use column in the Standard Measurement Window before closing the window.
4. Open the door slowly to retrieve your samples. The Shutter Open lamp will turn off.
5. Remove your samples carefully ensuring no sample spills. Close and lock the door.
6. The Miniflex II power off button (black) can then turned off.
7. ALLOW FOR 15 MINUTES TO PASS BEFORE TURNING OFF THE CHILLER PUMP.

REFERENCES AND FURTHER READING

- Rigaku Miniflex II System Manual (available in Rm. 214, Gibert Hall)
- Rigaku Software Manual (available in Rm. 214, Gilbert Hall)
- Beryllium [MSDS](#)
- Radiation Safety Website (oregonstate.edu/ehs/rso)
- Oregon State University Department of Chemistry-[Safety Web](#)

Appendix 1

To increase safety, the Miniflex II incorporates the following safety features:

- (1) The system has a circuit breaker. The power of the system cannot be turned on if the circuit breaker is not on. If an electric leak or any other abnormal condition arises, the circuit breaker is turned off immediately.
- (2) The system has its major components housed in an enclosure. Removing the enclosure cover turns off the power of the system.
- (3) In order for the system to generate X-rays, the READY lamp on its front panel must be on.
- (4) While the system is generating X-rays, the X-RAY lamp stays on. If this lamp experiences any anomaly, such as line disconnection or short circuit, the generation of X-rays will be stopped.
- (5) While the X-ray shutter is open, the SHUTTER lamp stays on.
- (6) While the system is generating X-rays, the voltage and current of the X-ray tube are 30 kV and 15 mA, respectively.
- (7) If the tube voltage exceeds 31.5 kV, the HVL lamp will light and the generation of X-rays will stop.
- (8) If the tube voltage drops below 27.4 kV, the LVL lamp will light and the generation of X-rays will stop.
- (9) If the tube current exceeds 15.75 mA, the HTC lamp will light and the generation of X-rays will stop.
- (10) If the tube current drops below 13.7 mA, the LTC lamp will light and the generation of X-rays will stop.
- (11) The shutter closes as you turn the door knob counterclockwise to unlock and open the door. When you attempt to open the door while X-rays are being generated, the behavior of the system differs depending on its specification.
Specification A (used in standard operation for this system): The shutter will close, and the generation of X-rays will stop.
Specification B: The shutter will close, but the generation of X-rays will continue.

The above mechanisms help to reduce the risk of exposure to X-rays. In addition to these features, please ensure that before running a measurement to check the following:

- Neither the inside nor the outside of the Miniflex enclosure is wet.
- No abnormal odor is coming from the main unit, power cable, water pump or any other component.
- No alarm in the maintenance display is on.
- No unusual features are in or around the Miniflex.

If one or more of these cannot be confirmed, please seek assistance from a Miniflex Administrator. Furthermore, it is prohibited to do the following:

- Remove warning labels from their original locations. If any come off, please affix them again or seek assistance from the Miniflex Administrator.
- Attempt to generate X-rays or operate the Miniflex in any other manner than that specified in this SOP and in the official Rigaku Miniflex II manual supplied with the instrument.

Additional Precautions of the Miniflex II System.

Please note that the following precautions primarily relate to maintenance operations for the Miniflex II system which should always be performed by a Miniflex Administrator or other well qualified and authorized personnel.

Shutter and Other X-ray protection Mechanism

Direct beam: A strong X-ray beam is emitted from the tube window mainly in the direction 6 degrees below the horizontal. The direct beam is blocked by the beam stopper.

Scattered X-ray: Weak X-rays are emitted in all directions inside the enclosure including the sample chamber. The scattered X-rays are blocked by the interior walls of the enclosure.

When the system is generating X-rays with the shutter closed, the X-rays are emitted inside the tube shield. Because the shutter is closed, however, there is no X-ray radiation inside the enclosure. When the system is generating X-rays with the shutter opened, a direct beam passes through the inside of the enclosure (including the sample). Scattered X-rays are also present. These X-rays are blocked by the beam stopper and enclosure walls and door, thus making the outside of the system enclosure safe.

Do not remove or disassemble the shutter. Do not remove any part from the system. If the equipment is operated with the X-ray shutter, slit box, slit plate, or direct beam stopper of the optical system moved from their normal assembled positions or modified, the X-rays may escape to the outside of the instrument. Do not remove or modify said parts under any circumstances. In particular, removal or disassembly of the shutter could result in exposure to a massive dose of X-rays for the operator and those working in the same room.

Removing the upper part of the cabinet to replace the X-ray tube

Turn off the power circuit breaker before removing or attaching the X-ray tube. When installing or replacing the X-ray tube, remove the three screws on either side of the enclosure and then remove the upper-side cover of the enclosure. Turn off the power circuit breaker before doing this task. To remove the upper-side cover, push it backward a little and then lift it up. Exercise due care not to drop the cover.

Handling of the X-ray tube

Immediately after the disconnection of the power to the X-ray unit, the X-ray tube's bulb is at a high temperature and may cause burns if touched. Before removing the tube, cool it by allowing the water to run for at least 30 minutes.

The X-ray window is made of beryllium (Be). Metallic beryllium or its corroded substances or compounds may be harmful if touched or swallowed. To prevent this risk, observe the following precautions:

- (1) Do not touch the metallic beryllium with any part of the body.
(If you have inadvertently touched it with your bare hand or contacted it with any part of your body, immediately wash it off with soapy water.)
- (2) Prohibitions relating to the part made of metallic beryllium portion
 - Do not get it wet. (If it accidentally gets wet, dry it by absorbing the water with hygroscopic paper.)

- Do not polish, chip, cut, or otherwise damage it.
 - Do not clean it with chemicals, burn it, or reduce it to a powder or vapor.
- (3) Procedure in the event beryllium is accidentally broken
- Do not touch it with your bare hand.
 - Do not inhale its powder.
 - Recover all the fragments, place them in a bag, and have a specialized contractor dispose of them.
 - If you have difficulty finding such a specialized contractor, contact Rigaku:

Rigaku Americas
 Tel: 1-888-362-2324
 Email: support@Rigaku.com

Mechanical parts

Do not touch the gear of the goniometer. Putting a finger between the goniometer gear teeth while the goniometer is scanning may result in the finger getting caught. Also, closing the sample chamber door hurriedly may result in your finger or fingers getting caught in the door. Exercise care to avoid such accidents.

High voltages

Turn off both the power switch of the X-ray generator and the wall power switch before performing any maintenance work. While the equipment is generating X-rays, the X-ray tube is fed with a high voltage through a high-voltage bushing. To prevent accidents caused by electric shock, observe the following safety precautions when cleaning the head of the high-voltage bushing, applying the insulating grease, replacing the X-ray tube, or checking the interior of the X-ray generator.

- (1) Switch off the equipment and turn off the wall power switch to ensure that the power supply is cut off.
- (2) The equipment retains a high-voltage charge in the high-voltage bushing and inside the transformer. Wait for at least 30 minutes after cutting off the power before starting work.
- (3) This task should be carried out by appointed maintenance personnel well versed with this system and having high-level electrical expertise.
- (4) If the head of the high-voltage bushing or other part must be touched directly by hand, start working after discharging the electric charge by grounding the head.

Applying a direct beam to the counter

Do not allow any direct X-ray beam into the counter. If a direct X-ray beam must be applied to the counter, always insert an absorber to attenuate the X-rays. The counter will be damaged severely if it counts direct X-ray beams directly.

Emergency off (EMO) switch

Please push EMO switch (red, at the right of the front side of the device) when you urgently need to stop running the device **in an emergency**. The LED of the display panel will be turned off. The button of the EMO switch will need to be first rotated clockwise when resuming standard operations.

Checking Safety Functions

Note that these safety checks should be performed by a Miniflex Administrator or other well qualified and authorized personnel.

To check the diffracted rays count using the instrument counter (SC) :

- Turn on the power of the main unit. Ensure the HP PC is on.
- Select "Right Measurement" - "Manual Measurement".
- Click the "Initialize" button to perform execution.
- Set Si of the setting jig on the sample holder.
- Close the door of the sample chamber and turn the door knob clockwise to lock the door.
- Move the $2\theta/\theta$ axis to the peak position (28.4°) of Si (1 1 1).
- (Perform execution by specifying control target = $2\theta/\theta$, control = movement, and target position = 28.4° .)
- Press the X-Ray ON button.

To check that the shutter and door mechanisms are operating safely:

- With the instrument on standby, that is no X-rays are being generated but the door is locked, shutter is open and the PHA lamp is on. Turn the door knob counterclockwise bit by bit to release the locking until the PHA lamp goes off. This indicates that the shutter has closed. The lamp may sometimes lights weakly because natural counting is performed.
- Further turn the door knob counterclockwise. The following behavior is observed depending on the specification. Specification A is used under standard operation conditions and in line with this the X-rays are turned off and the X-RAY lamp goes off before the locking of the door is released.
- This observation ensures that the door of the sample chamber can be opened safely when the shutter closes.