

MiniFlex XRD Performance Monitoring and Maintenance

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MiniFlex XRD Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the MiniFlex X-ray diffractometer (XRD). The Rigaku MiniFlex XRD uses an X-ray source, a goniometer, and a detector to generate an X-ray diffraction pattern for the sample being analyzed. The generated pattern is compared to X-ray patterns in the library of the International Center for Diffraction Data (ICDD), the pattern of a reference standard, reference material, or other material of known origin. Definitions and guidelines are outlined IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/subdisciplines: Explosives Chemistry, Paints and Polymers, General Chemistry, and Seized Drugs.

3 EQUIPMENT

- Instrumentation
 - Rigaku MiniFlex (or equivalent)
 - PDXL or SmartLab Studio II Software (or equivalent)
 - ICDD
- Materials
 - Silicon reference specimen (or equivalent reference standard)

4 STANDARDS AND CONTROLS

The silicon reference specimen is used to ensure that the sample holder and X-ray tube are aligned and functioning properly. The standard is used to verify performance and continued integrity of the system. This standard does not expire.

5 PROCEDURE

5.1 Daily Checks

- A. Record the remaining disk space on the hard drive. Verify that the hard disk has at least 100MB of free disk space. Do not use if less than 100 MB remain.
- B. Ensure the silicon reference specimen is in the instrument.
- C. Age the X-ray generator.
- D. Ensure appropriate analysis settings under the 'Instrumental Conditions' section are used and start the measurement.
- E. Using the data analysis software, open the collected silicon standard data file and evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the diffraction pattern and peak locations.
- F. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

6 INSTRUMENTAL CONDITIONS

The X-ray generator parameters, detector, and scan mode are fixed within each instrument and will not be adjusted. Adjustments can only be made to the scan speed, step width, and scan range.

X-ray Generator:	40 kV, 15 mA (MiniFlex 6G)
Detector:	D/teX Ultra
Scan mode:	Continuous
Scan speed:	20 deg/min
Step width:	0.02 deg
Scan range:	5 - 80 deg

7 ACCEPTANCE CRITERIA

Check the location of peaks and peak intensity. Results must meet the following criteria:

- A. The peak height of the 100% peak should be greater than 10,000 cps.
- B. Peaks (2θ , deg), all within 0.15 deg:
 - o 28.42
 - o 47.30
 - o 56.10
 - o 69.17
 - o 76.37

If the values lie outside the specified range, verify the instrument parameters and re-analyze the silicon standard. If the results are still outside the specified range, contact appropriate instrument support personnel.

8 LIMITATIONS

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

9 SAFETY

The Rigaku MiniFlex XRD produces X-rays. The unit is equipped with appropriate shielding and electrical interlocks which prevent operation under conditions which would allow the escape of ionizing radiation. These interlocks should never be overridden. All personnel operating the spectrometer are routinely monitored via personal radiation monitors, administered at the unit level and tracked by the appropriate health and safety personnel.

The X-ray tube window contains beryllium and is extremely delicate. In the event of damage to the window, the beryllium dust created could pose an acute health hazard. If this occurs, seal the chamber and seek assistance from the appropriate health and safety personnel.

10 REVISION HISTORY

Revision	Issued	Changes
02	07/01/2022	Revised to match new format requirements. Removed MiniFlex 600 throughout. Section 2- Removed categories of testing. Section 3- Separated instrumentation from software and added separate silicon reference. Removed silicon powder. Section 5- Simplified to be generic and not software specific. Section 6- Updated the silicon scan speed and scan range. Section 7- Updated the peak list. Section 9- Updated health and safety information.
03	11/15/2022	Section 2- Updated Scope to remove location. No other substantive changes to content.