

## **Performance Monitoring Protocol (QA/QC) for the Rigaku MiniFlex X-Ray Diffractometer (XRD)**

### **1 Scope**

This document addresses the performance monitoring (QA/QC) of the Rigaku MiniFlex X-ray diffractometer (XRD). This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Explosives (chemistry), paint, drug chemistry, and Chemistry Unit general physical and chemical analysis.

### **2 Principle**

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 (ICDD), the pattern of a known standard, of the pattern of other material of known origin.

This performance monitoring protocol is based upon the manufacturer's recommendations. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

### **3 Equipment/Materials/Reagents**

- a. Instrumentation – Rigaku MiniFlex II, MiniFlex 600, with PDXL Software (or equivalent)
- b. International Center for Diffraction Data (ICDD) Library
- c. NIST SRM 640c Silicon Powder (or equivalent traceable standard)

### **4 Standards and Controls**

The silicon standard 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 Sampling or Sample Selection

Not applicable.

## 6 Procedures

The following steps will be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

- a. Record the remaining disk space on the hard drive. Use Windows Explorer program to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.
- b. Place the silicon standard in the instrument.
- c. Open the 'General Measurement' or 'Standard Measurement' window, enter the appropriate information pertaining to the standard, and start the measurement.
- d. Using the PDXL software, open the silicon pattern just created by selecting 'Load' and the recently analyzed silicon standard data file.
- e. In the 'Profile View' tab, unclick the buttons for 'ResidualGraph' (optional) and 'Peakbar 1'. Click 'Create Report' and select "Silicon Standard QA/QC Printout" (or equivalent). Click 'Create Report' and resize the diffractogram so that all information fits on one page. Alternatively, print the diffraction pattern and peak list.
- f. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable print the report.
- g. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact appropriate instrument support personnel.

## 7 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 600) 30 kV, 15 mA (Miniflex II)
Detector:	D/teX Ultra
Scan mode:	Continuous
Scan speed:	40 deg/min

Step width: 0.02 deg  
Scan range: 20 – 140 deg

## 8 Decision Criteria

Check the location of peaks and peak intensity. Results must fall within the following:

- a. The peak height of the 100% peak should be greater than 10,000 cps.
- b. Peaks ( $2\Theta$ ), all +/- 0.15:  
28.42  
47.30  
56.10  
69.17  
76.37  
88.06  
94.97  
106.73  
114.13  
127.57  
136.93

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

## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

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

## 12 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 Health and Safety Group.

The detector window is comprised of 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 Health and Safety Group.

## 13 References

Manufacturer(s)'s Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual*.

*FBI Laboratory Safety Manual*.

<u>Rev. #</u>	<u>Issue Date</u>	<u>History</u>
0	4/25/16	New document, previously existed in the Explosives Unit.
1	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Updated heading in Section 5. Removed unchecking of 'ResidualGraph' in Section 6 e. Added 'appropriate instrument support personnel' to Sections 6 g and 8. Updated 'Instrument Operation and Systems Support' in Section 13 and header.

**Approval**

Redacted - Signatures on File

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