Performance Monitoring Protocol (QA/QC) for the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS). This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The SEM/EDS is utilized primarily to characterize the elemental composition of a material. It is necessary to ensure the instrument is performing optimally because this instrumentation is dependent upon a determination of the energy of detected X-rays.

SEM can be utilized for morphological and metrological determinations; therefore, image quality and measurement accuracy will need to be determined for these examinations. For routine analysis the magnification accuracy is sufficient. When a measurement is required with reportable accuracy, a calibrated measurement standard will be employed. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Scanning Electron Microscope (SEM) – JEOL JSM IT300 (or equivalent)

b. Energy Dispersive Spectrometer (EDS) – Thermo Noran System 7 (or equivalent)

c. Energy Dispersive X-ray processing software – Noran NSS (or equivalent)

d. Manganese (Mn) standard, polished (or equivalent)

e. Carbon planchets (or equivalent)

f. Aluminum pin stub specimen mounts (or equivalent)

g. Liquid Adhesive (Duro-tak or equivalent)

h. Acetone(Reagent grade)

4 Standards and Controls

Prior to each use of the SEM/EDS, a determination of the ability to perform elemental identification is made by confirming system energy calibration using the X-ray lines of the pure element standard manganese (Mn).

5 Sampling

Not applicable.

6 Procedures

6.1 Daily Checks

a. Collect a spectrum from the pure element standard manganese (Mn).

b. Analyze spectra. Observe peak shape, peak width, high to low energy peak ratio, shape of background, peak artifacts, and system peaks.

c. If the peaks observed are shifted from their previously established positions by more than 30 eV, contact the appropriate instrument support personnel.

d. If all requirements are within specification, prepare the documentation as outlined in the “General Instrument Maintenance Protocol.” If any requirements fail, contact the appropriate instrument support personnel.

7 Instrumental Conditions

7.1 Imaging

Values for accelerating (high) voltage, working distance, spot size, beam intensity, stigmation, focus, brightness, and contrast are established at the operator’s discretion based on image quality desired.

7.2 Detector

Detector response: Mn
Beam voltage: 25KV

Working distance and beam intensity/spot size will be set at the operator’s discretion.

8 Decision Criteria

a. Detector response:
In order for the instrument to be considered in good operating condition, the most abundant peak in the manganese spectrum must be at 5.9 eV. The spectrum should exhibit a similar high to low energy peak ratio, Gaussian peak shape, a minimum SNR of 3:1, and the presence of any spectral artifacts. Changes in the low to high peak intensity ratio may indicate accumulation of ice on the crystal face.

b. Energy characterization:
Calibration of the EDS will only be performed if the x-ray lines are shifted from their expected positions in the spectrum of the pure element standard manganese (Mn) by more than 30 eV. This procedure will be performed by properly trained personnel by following the procedure for recalibration in the manufacturer's instrument manuals.

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel shall perform duties involved in the operation, maintenance, or troubleshooting of the SEM and/or EDS.

12 Safety

General precautions common to electron microscope laboratories include: minimization of contact with cryogens, venting of P-10 gas, venting or filtering of roughing pump discharge, and avoidance of direct exposure to beryllium metal. Under normal operator conditions the instrument poses no known hazards.

Use universal precautions when handling potentially biohazardous materials. Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis.

13 References

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

Goldestein, Newbury, Echlin, Joy, Romig, Lyman, Fiori, Lifshin, *Scanning Electron Microscopy and X-ray Microanalysis,* Second Edition, Plenum Press, 1992.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual

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0	10/04/18	New document that specifies instrument protocol for the Huntsville facility.

Redacted - Signatures on File

Approval

Scientific Analysis
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TL Approval

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