

SEM/EDS Performance Monitoring and Maintenance

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SEM/EDS Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Scanning Electron Microscope (SEM)/Energy Dispersive X-ray Spectrometer (EDS). The SEM/EDS is utilized primarily to characterize the elemental composition of a material. Because this instrumentation is dependent upon a determination of the energy of detected X-rays, it is necessary to ensure the instrument is performing optimally for the intended analysis.

SEM can be utilized for morphological and metrological determinations. If measurements will be made that require an estimate of the magnification accuracy, see Section 5.2. These measurements are not traceable and should not be considered reliable, quantitative measurements. 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, General Chemistry, Metallurgy, Paints and Polymers, and Seized Drugs.

3 EQUIPMENT

- Instrumentation
 - JEOL JSM 6510LV SEM, EDAX EDS detector, JEOL software, EDAX software (or equivalent)
 - JEOL JSM 6610 SEM, EDAX EDS detector, JEOL software, EDAX software (or equivalent)
 - JEOL JSM IT300 SEM, Thermo UltraDry EDS detector with Noran System 7 software, JEOL software (or equivalent)
 - TESCAN Vega 4 SEM, EDAX EDS detector, Vega software, EDAX software (or equivalent)
- Materials
 - Manganese (Mn) standard, polished (or equivalent)
 - Geller MRS-3 SEM magnification standard (or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

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

5 PROCEDURE

5.1 Daily Checks

- A. Collect a spectrum from the pure element standard manganese (Mn).
- B. Recall verification spectrum of previous pure element.

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- C. Compare spectra. Observe and compare peak shape, peak width, high to low energy peak ratio, shape of background, peak artifacts, and system peaks.
- D. If the peaks observed are shifted from their previously established positions by more than 30 eV, contact appropriate instrument support personnel.
- E. Prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 Magnification Standard

Magnification standardization will be performed as needed.

- A. Place the Geller MRS-3 SEM magnification standard into SEM chamber and evacuate.
- B. Set specimen tilt to 0 degrees (perpendicular to the electron beam).
- C. Adjust the instrumental conditions to be similar to those used to image the object from which an accurate measurement is required.
- D. Bring the Geller MRS-3 SEM magnification standard into focus using stage Z.
- E. Take one digital image of an appropriate pattern on the Geller standard. Different pattern selections are available depending on the magnification selected. Examples are provided below:

Pattern	Spacing
50X	largest bar plus space = 0.5 mm
100X	largest bar plus space = 0.5 mm
1,000X	middle bar plus space = 50 μ m
10,000X	smallest bar plus space = 2 μ m

- F. Using a ruler, measure the features in the digital images and calculate the actual magnification.
- G. Calculate % magnification error, and apply the correction to the measurement from the structure of interest.
- H. Record the results in the appropriate instrument log.
- I. Once a magnification error has been determined for a specific set of analytical conditions, error correction can be applied to any subsequent measurements obtained under those conditions.

6 INSTRUMENTAL CONDITIONS

6.1 Imaging

Detector type (e.g., secondary or backscatter) and values for accelerating (high) voltage, working distance, spot size, beam intensity, stigmatism, focus, brightness, and contrast are established at the operator's discretion based on image quality desired.

6.2 Magnification

Instrumental conditions will be the same as those required to image a material of interest from which a measurement is required.

6.3 EDS Detector

Detector response:	Mn
Beam voltage:	25 KV
Working distance:	Set at the operator's discretion
Beam intensity/spot size:	Set at the operator's discretion

7 CALCULATIONS

- magnification = (image dimension)/(object dimension)
- % magnification error = [(displayed magnification - measured magnification)/measured magnification] x 100

8 ACCEPTANCE CRITERIA

8.1 Detector Response

Compare the manganese spectrum to a previously collected manganese spectrum. The spectrum should exhibit a similar high-to-low energy peak ratio, Gaussian peak shape, a minimum SNR of 3:1, and the absence of any significant spectral artifacts.

8.2 Energy Characterization

If the measured peak centroid energy is more than 30eV from the theoretical average Mn K α peak energy of 5.895 keV, a detector calibration adjustment will be performed in accordance with the manufacturer's recommendations.

9 LIMITATIONS

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

10 SAFETY

General precautions common to electron microscope laboratories include: 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.

11 REVISION HISTORY

Revision	Issued	Changes
03	10/04/2018	Section 1- Updated scope to include applicable disciplines/categories of testing. Section 3- Added JSM6610. Section 7- Removed specific software titles. Section 7- Added 'appropriate instrument support personnel'. Section 8- Added detector types. Section 12- Added theoretical value. Section 13- Removed outdated cryo hazard. Section 14- Updated 'Instrument Operation and Systems Support'.
04	10/01/2022	Revised to match new format requirements. Section 1- Clarified function and measurement. Section 3- Updated Tescan to Vega 4, added IT300. No other substantive changes to content.