

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.

Quantitative measurements (i.e., elemental concentration, dimensional measurements) and their associated measurement traceability, measurement uncertainty, and validation are outside the scope of this procedure and will be addressed in discipline/subdiscipline-specific technical procedures, as applicable.

Definitions and guidelines are outlined in 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)
 - JEOL JSM IT510 SEM, EDAX EDS detector, JEOL software, EDAX software (or equivalent)
 - TESCAN Vega 4 SEM, EDAX EDS detector, Vega software, EDAX software (or equivalent)
- Materials
 - Manganese (Mn) standard, polished (or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

Prior to use of the EDS, a daily check 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 EDS Daily Check

- A. Collect a spectrum from the Mn pure element standard using a beam voltage of 25 kV.
- B. Compare the Mn spectrum to a previously collected Mn 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 unexplained spectral artifacts.
- C. If the measured peak centroid energy is more than 30eV from the theoretical average Mn K α peak energy of 5.895 keV, contact the Instrument Operation and Systems Support (IOSS) team for assistance.
- D. Prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate IOSS personnel.

5.2 Magnification Standardization

Magnification standardization and the associated quantitative measurements will be addressed in the applicable discipline/subdiscipline-specific technical procedure(s).

6 INSTRUMENTAL CONDITIONS

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.

7 LIMITATIONS

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

8 SAFETY

A dosimeter should be worn when operating the SEM.

9 REVISION HISTORY

Revision	Issued	Changes
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.
05	08/01/2024	Section 1- edited to clarify scope regarding quantitative measurements. Section 3- added JEOL JSM IT510, removed Geller MRS-3 SEM magnification standard. Section 4- edited to clarify 'daily' check vs. 'each use'. Section 5- clarified check is for the EDS and is conducted with 25 kV beam voltage. Moved acceptance criteria into Section 5 for ease-of-use and deleted previous Section 8- Acceptance Criteria. Section 5.1 C.- revised to contact IOSS rather than the 'instrument manufacturer's recommendation'. Section 5.2- removed procedural content and referred to discipline/subdiscipline-specific technical procedures. Pared down Section 6 as relevant content was moved to other sections for ease-of-use; deleted Sections 6.2- Magnification, and 6.3- EDS Detector. Removed previous Section 7- Calculations. Section 8- removed generic safety information and removed reference to beryllium metal; added dosimeter badge info.