TECHNICAL IMPLEMENTING PROCEDURE

TIP-CM-3

Operator Calibration of Scanning Electron Microscopes

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OPERATOR CALIBRATION OF SCANNING ELECTRON MICROSCOPES.

1.0 PURPOSE

This procedure describes requirements necessary for operator calibration of Scanning Electron Microscopes (SEM's) in support of the Yucca Mountain Project (YMP). These requirements are established to assure that SEM calibrations are documented, and that measurements made on SEM images are accurate.

2.0 SCOPE

This procedure applies to operator calibration of scanning electron microscopes for scientific investigations for the Yucca Mountain Project. It applies to measurements made using photographic images obtained using a Scanning Electron Microscope. Measurements are made on photographic positive prints either produced directly or made from negatives produced by the electron microscope. Calibrations are made in accordance with well established practices.

Scanning Electron Microscope Magnification Standards are available through the National Institute for Standards and Technology (formerly the National Bureau of Standards).

3.0 RESPONSIBILITIES AND AUTHORITIES

The Task Leader (TL) whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure. The Task Leader may delegate this responsibility and authority to a Principal Investigator.

The individual calibrating the SEM and making the measurements is responsible for following the requirements of this procedure, documenting calibrations and assuring that the latest revision of this document is followed.

The YMP Quality Assurance Manager (QA Manager) is responsible for monitoring work to assure proper implementation of this procedure and for assuring its continued effectiveness.

4.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with procedure 033-YMP-QP 5.0, "Technical Implementing Procedures." Procedures are issued in accordance with procedure 033-YMP-QP 6.0, "Document Control."

4.1 Selection

The Task Leader, or his designate, will ensure that the SEM's used are of the proper type, design, range, accuracy, and tolerance to accomplish their required function.

4.2 Identification

The SEM's are identified by using unique identification numbers (DOE Nos.). The identification number is recorded on data sheets, laboratory note books, logs, etc., along with the measurements taken to assure traceability to the electron microscope used to make the measurement.

4.3 Calibration

The standards used for calibration of the SEM are traceable to the National Institute for Standards and Technology (formally the National Bureau of Standards). Traceability requires the ability to relate individual measurement results through an unbroken chain back to NIST or other nationally recognized standard. The chain of calibration must be documented and auditable.

4.4 References

The present calibration procedure was derived from the following procedures for establishing magnification calibration for an SEM:

1. David B. Ballard, "A Procedure for Calibrating the Magnification of a Scanning Electron Microscope Using NBS SRM 484", NBSIR 77-1248, U.S. Department of Commerce, National Bureau of Standards, June 1977.

2. J. Paul Calli, "National Bureau Of Standards Certificate, Standard Reference Material 484a, Scanning Electron Microscope Magnification Standard (A Stage Micrometer Scale)", U.S. Department of Commerce, National Bureau of Standards, December 5, 1977.

5.0 PROCEDURE

This procedure includes calibration with an NBS Standard Reference Material (SRM) 484a scanning electron microscope standard (a stage micrometer scale). This SRM is intended for use in calibrating the scanning electon microscope (SEM) magnification scale to an accuracy of 5% or better within the range of 1,000 to 60,000X. Each SRM bears an identification number and has been individually measured.

5.1 Calibration

Place the magnification standard in a specimen holder and insert it into the microscope column. Select an operating voltage consistent with that for which calibration is desired. Bring the electron microscope into operation in accordance with the procedures specified by the manufacturer. Saturate the filament and select the area of the SRM containing the Knoop indentation and gold-filled scribed lines for imaging. Adjust apertures and stigmators for optimum resolution.

Note 1 -- For the Hitachi S-800 field-emission scanning electron microscope, DOE No. 4616427, the operating procedures recommended by the manufacturer are located in the room with the instrument. The document is entitled "Instruction Manual for Model S-800 Field Emission Scanning Electron Microscope, Part No. 580-8600-2, YR-G (FT-LT)" and Chapter 3, pages 3-1 to 3-37, defines the operating procedures.

5.1.1

Select a working distance and set the lens current for focussing at this distance. Using the Z-axis control, move the SRM into focus. Record the microscope identification number, the SRM identification number and the working distance.

5.1.2

Select magnification settings of 1,000X, 10,000X, 20,000X, 50,000X and 60,000X. At each magnification, focus the image (by Z-axis control) and expose the photographic film.

5.1.3

For magnification calibration, use a commercial scale to measure the line spacings on each of the images and determine the image magnifications from these spacings.

$$\text{Magnification} = \frac{\text{Distance measured between image lines on the micrograph}}{\text{Certified distance between same lines}}$$

6.0 HANDLING AND STORAGE

Electron microscopy standards shall be handled and stored so as to minimize their exposure to dusty or corrosive atmospheres and mechanical shock. Normally, isolating the standard inside a container that identifies the contents is sufficient to satisfy those requirements.

7.0 RECORDS

The laboratory note book will identify the calibration status and the calibration procedure (including revisions) used to perform each calibration.

SEM calibrations will be recorded in the laboratory note book in accordance with procedure 033-YMP-QP 3.4, "Scientific Notebooks."

Documentation of NIST traceability of the standards will be maintained in a record file kept in the laboratory, with copies sent to the Local Records Center. SEM's are identified by make, model, serial number, and LLNL property number.