



Designation: E766 – 14 (Reapproved 2019)

# Standard Practice for Calibrating the Magnification of a Scanning Electron Microscope<sup>1</sup>

This standard is issued under the fixed designation E766; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers general procedures necessary for the calibration of magnification of scanning electron microscopes. The relationship between true magnification and indicated magnification is a complicated function of operating conditions.<sup>2</sup> Therefore, this practice must be applied to each set of standard operating conditions to be used.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

**E7 Terminology Relating to Metallography**

**E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications**

**E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods**

**E456 Terminology Relating to Quality and Statistics**

**E691 Practice for Conducting an Interlaboratory Study to**

**Determine the Precision of a Test Method**

2.2 *ISO Standard:*

**ISO Guide 30: 1992 Terms and Definitions Used in Connection with Reference Materials**<sup>4</sup>

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of metallographic terms used in this practice see Terminology **E7**.

3.1.2 The definitions related to statistical analysis of data appearing in Practice **E177**, Terminology **E456**, and Practice **E691** shall be considered as appropriate to the terms used in this practice.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibration*—the set of operations which establish, under specified conditions, the relationship between magnification values indicated by the SEM and the corresponding magnification values determined by examination of a reference material.

3.2.2 *calibration method*—a technical procedure for performing a calibration.

3.2.3 *certified reference material*—reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence (see ISO Guide 30:1992).

3.2.4 *pitch*—the separation of two similar structures, measured as the center to center or edge to edge distance.

3.2.5 *reference material*—a material or substance one or more of whose property values are sufficiently homogeneous, stable, and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (see ISO Guide 30:1992).

3.2.6 *reference standard*—a reference material, generally of the highest metrological quality available, from which measurements are derived.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee **E04** on Metallography and is the direct responsibility of Subcommittee **E04.11** on X-Ray and Electron Metallography.

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<sup>2</sup> See **Annex A1**.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3.2.7 *traceability*—the property of a result of a measurement whereby it can be related to appropriate international/national standards through an unbroken chain of comparisons.

3.2.8 *verification*—confirmation by examination and provision of evidence that specified requirements have been met.

#### 4. Significance and Use

4.1 Proper use of this practice can yield calibrated magnifications with precision of 5 % or better within a magnification range of from 10 to 50 000X.

4.2 The use of calibration specimens traceable to international/national standards, such as NIST-SRM 484, with this practice will yield magnifications accurate to better than 5 % over the calibrated range of operating conditions.

4.3 The accuracy of the calibrated magnifications, or dimensional measurements, will be poorer than the accuracy of the calibration specimen used with this practice.

4.4 For accuracy approaching that of the calibration specimen this practice must be applied with the identical operating conditions (accelerating voltage, working distance and magnification) used to image the specimens of interest.

4.5 It is incumbent upon each facility using this practice to define the standard range of magnification and operating conditions as well as the desired accuracy for which this practice will be applied. The standard operating conditions must include those parameters which the operator can control including: accelerating voltage, working distance, magnification, and imaging mode.

#### 5. Calibration Specimen

5.1 The selection of calibration specimen(s) is dependent on the magnification range and the accuracy required.

5.2 The use of reference standards, reference materials, or certified reference materials traceable to international/national standards (NIST, Gaithersburg, MD; NPL, Teddington, UK; or JNRLM, Tsukuba, Japan) calibration specimens is recommended. However, the use of internal or secondary reference materials validated against reference standards or certified reference materials may be used with this practice.

5.3 Where traceability to international or national standards is not required, internal reference materials, verified as far as technically practicable and economically feasible, are appropriate as calibration specimens and may be used with this practice.

5.4 The most useful calibration specimens should have the following characteristics:

5.4.1 A series of patterns allowing calibration of the full field of view as well as fractional portions of the field of view over the range of standard magnifications. Suitable standards allow for the pattern “pitch” to be measured,

5.4.2 Pitch patterns allowing calibration in both X and Y without having to rotate the sample or the raster,

5.4.3 Made from materials which provide good contrast for the various imaging modes, especially secondary electron and backscatter electron imaging.

5.4.4 Made of or coated with electrically conductive, electron beam stable materials, and

5.4.5 Made of materials which can be cleaned to remove contamination which occurs during normal use.

5.5 Under typical use some contamination of the calibration specimen should be expected. When cleaning becomes necessary always follow the manufacturer’s instructions. Improper handling, especially during cleaning, may invalidate the calibration specimen’s certificate of accuracy or traceability and require re-certification. Care should be taken to prevent the standard from sustaining mechanical damage which may alter the standard’s structure.

5.6 The facility using this practice shall have arrangements for the proper storage, handling, and use of the calibration specimen(s) which should include but is not limited to:

5.6.1 Storage in a desiccating cabinet or vacuum container,

5.6.2 Using finger cots, clean room gloves or tweezers when handling, and

5.6.3 Restricting its use to calibration only, unless it can be shown that the performance of the calibration specimen will be unaffected by such use.

5.7 The facility using this practice shall establish a schedule for verification of the calibration specimen(s), where verification should include but is not limited to:

5.7.1 Visual and microscopical inspection for contamination and deterioration which may affect performance,

5.7.2 Photomicrographic comparison (and documentation) of the present state of the calibration specimen(s) to the original state, and

5.7.3 Validation or re-certification of calibration specimen(s) distance intervals against other reference standards or certified reference materials.

#### 6. Procedure

6.1 Mounting of the calibration specimen.

6.1.1 Visually inspect the calibration specimen surface for contamination and deterioration which may affect performance. Remove any dust or loose debris using extra care not to damage the specimen surface. One safe method is to use clean dry canned air to remove the loose surface debris.

6.1.2 Ensure good electrical contact by following the SEM and calibration specimen manufacturers’ directions for mounting. In some instances the use of a conductive cement may be required.

6.1.3 Mount the calibration specimen rigidly and securely in the SEM specimen stage to minimize any image degradation caused by vibration.

6.2 Evacuate the SEM chamber to the desired or standard working vacuum.

6.3 Turn OFF the tilt correction and scan rotation circuits. These circuits should be calibrated independently.

6.4 Set the specimen tilt to 0° such that the surface of the calibration specimen is perpendicular to the electron beam. A technique for checking specimen surface perpendicularity is to observe the image focus as the specimen is translated twice the

picture width in the X or Y direction. The change of image focus should be minimal at a nominal magnification of 1000X.

6.5 Adjust the accelerating voltage, working distance, and magnification to the desired or standard operating conditions.

6.6 The instrument should be allowed to fully stabilize at the desired operating conditions. The time required will be pre-determined by the facility using this practice.

6.7 Minimize residual magnetic hysteresis effects in the lenses by using the degauss feature, cycling lens circuits ON-OFF-ON two or three times, or follow manufacturers recommendations.

6.8 Adjust the image of the calibration specimen on the viewing display.

6.8.1 Bring the image of the specimen into sharp focus. The sample working distance should be pre-selected to determine magnification accuracy since different working distances may have different magnification errors. The specimen height (Z axis) is then adjusted to attain focus on the viewing display. If the SEM has a digital working distance display, the desired value may be selected by adjusting the objective lens focus.

6.8.2 Mechanically rotate the calibration specimen so the measurement pattern(s) is parallel to the X or Y directions of the image display, or both. Never use the scan rotation circuits to rotate the image since the circuit may introduce distortions or magnification error, or both.

6.8.3 Translate the calibration specimens so the fiducial markings of the measurement pattern(s) span 90 % of the full display of the viewing display using the SEM specimen stage X and Y controls. It is desirable to see both edges of each fiducial marking in order to ascertain the line-center or repeated pitch distance on the calibration specimen

6.9 Digital image calibration:

6.9.1 Follow the manufacturer’s calibration instructions by choosing two points or drawing a line between the centers or edges of the repeating structure on the calibration specimen. Enter the known distance into the SEM imaging software and save a calibration file for each set of conditions as the manufacturer provides. The content of the calibration file is a distance per pixel value that varies with digital resolution as well as microscope conditions.

6.10 Analog Image Display Calibration Method:

6.10.1 Measure with an appropriate ruler and record the pitch distance (D) between two of the fiducial markings (in mm ± 0.5 mm) which are separated by the largest spacing in the field of view. This step must be carried out for both the X and Y directions of the image display.

6.10.1.1 If the fiducial markings are lines the measurement must be made perpendicular to the fiducial lines and from line center to line center or line edge to the corresponding line edge.

6.10.1.2 With some calibration specimens, it may be necessary to rotate the specimen by 90° in order to determine magnification in both the X and Y directions. If this is the case, follow 6.10 – 6.10.2 before rotating the sample. Then follow 6.8.2 and 6.8.3 to re-align the calibration specimen in the new orientation and repeat 6.10 and 6.11.

6.10.2 Calculate the magnification by using 6.12.

6.11 Recording CRT calibration method (for older SEMs).

6.11.1 Photograph the field used in 6.10 with sufficient signal to noise ratio and image contrast to allow for accurate measurements.

6.11.2 Allow sufficient time for the photographic material to stabilize prior to measurement. This will minimize the effects of dimensional changes in the film caused by temperature and humidity.

6.11.3 Measure and record the pitch distance (D) between two of the fiducial markings (in mm ± 0.5 mm) which are separated by the largest spacing in the photomicrograph for the best precision.

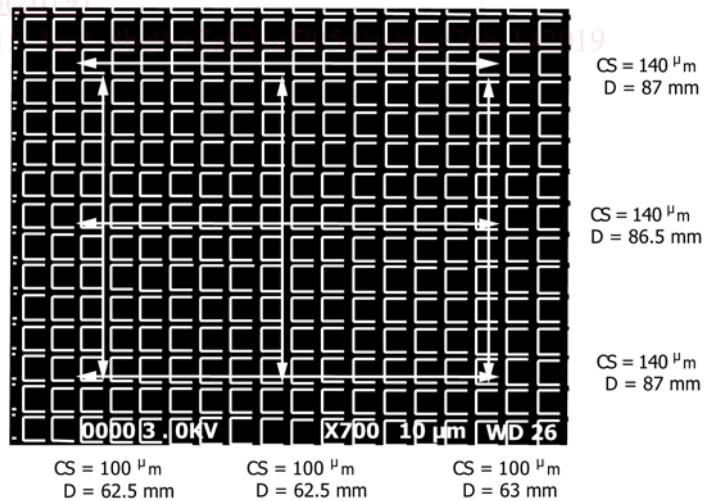
6.11.4 It is recommended that the fiducial markings used for the pitch measurement be at least 10 mm from the photo edges to minimize edge distortion effects.

6.11.5 If the measurement pattern consists of lines which span the length or width of the photomicrograph, then repeat the measurement in 6.11.3 at least three times at locations separated by at least 3 mm so that the average spacing may be determined (see Fig. 1).

6.11.6 Calculate the magnification for each measurement using 6.12. When multiple measurements have been made determine the mean and standard deviation for the set of measurements.

6.12 Calculation of Magnification:

6.12.1 Calculate the true magnification (M) by dividing the measured distance (D), usually in mm, by the accepted, certified, or 'known' spacing (CS), usually in micrometers and then multiplying by the appropriate length units conversion factor (CF). Conversion factors do not have to be used if the same units in the calculation are used. For instance, if the magnified pitch distance is measured in mm, divide that



NOTE 1—A 4 × 5 in. (101.6 × 127 mm) photomicrograph of a reference material, a 10 µm pitch square grid imposed on a silicon wafer, used as a calibration specimen. This calibration specimen is not certified. This micrograph was obtained using 3 kV accelerating voltage, 26 mm working distance, and a magnification setting of 700X. Measurements of D (in mm ± 0.5 mm) in both the horizontal and vertical directions are made approximately 10mm from the edges of the micrograph and in the center. In this example the horizontal magnification is 620X (± 2X) and the vertical magnification is 627X (± 3X).

FIG. 1 Photomicrograph of a Reference Material