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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Calibrating the Magnification of a Scanning Electron Microscope[1]

This standard is issued under the fixed designation E 766; 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 (ϵ) 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.[2] 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 the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 7 Terminology Relating to Metallography[3]

E 29 Standard Practice For Using Significant Digits in Test Date to Determine Conformance with Specifications[4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[4]

E 456 Terminology Relating to Quality and Statistics[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

2.2 *ISO Standard:*

ISO Guide 30: 1992 Terms and Definitions Used in Connection with Reference Materials[5]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of metallographic terms used in this practice see Terminology E 7.

3.1.2 The definitions related to statistical analysis of date appearing in Practice E 77, Terminology E 456, and Practice E 691 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 standard*—a reference material, generally of the highest metrological quality available, from which measurements are derived.

3.2.6 *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).

3.2.7 *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.

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[1] This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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[2] See Annex A1.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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 CRT.

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 CRT. 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 CRT, 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 CRT 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

6.8.4 A ruler of known accuracy should be used for these measurements.

6.9 Viewing CRT "micron" marker calibration method.

NOTE 1—This measurement determines the micron marker accuracy on the CRT for the indicated magnification (which is assumed to be correct), and not the magnification accuracy. Often the viewing CRT is a different size than the record CRT and resultant micrograph. The displayed magnification of the viewing CRT may therefore be incorrect as it was probably intended for the final image.

6.9.1 Measure the length of the "micron" marker (in mm ± 0.5mm) with an appropriate ruler of known accuracy. Record this value (D) and the indicated magnification. Due to the thickness of the CRT face plate be careful that parallax errors in the measurement do not affect the accuracy.

6.9.2 Calculate the true micron marker size by multiplying the indicated magnification by the displayed micron marker length. Calculate the percentage error by diving these two values. If error is more than the allowable tolerance, the micron marker should be adjusted. This may be accomplished by the manufacturer of the SEM or by following the manufacturer's documented procedures.

6.10 *Viewing CRT 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 view CRT.

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.

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*:

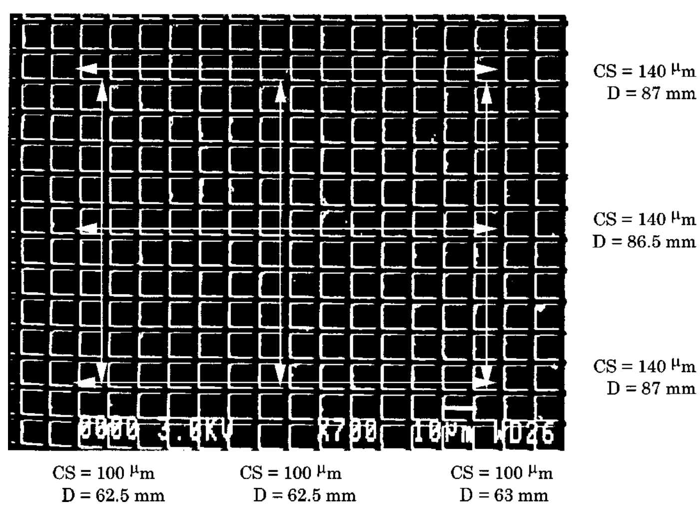
6.12.1 Calculate the true magnification (M) by dividing the measured distance (D), usually in mm, by the accepted, certified, or 'known' spacing (CS), usually in micrometers and then multiplying by the appropriate length units conversion factor (CF). Conversion factors do not have to be used if the same units in the calculation are used. For instance, if the magnified pitch distance is measured in mm, divide that number by the actual distance in mm (that is, 10/0.01 mm = 1000X).

$$M = (D/CS)*CF$$

Units (D/CS)	CF
millimetres/micrometre	1000
millimetres/nanometre	1000000
inches/micrometre	25400
inches/nanometres	25400000

NOTE 2—Practice E 29 provides guidance in the use of significant digits in calculating and reporting results.

6.13 *Digital Recording Method*—It is recommended that the magnification not be recorded with the image since the final image size will not be fixed. The micron marker should be used because it will scale with the final image.



NOTE 1—A 4 × 5 in. (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

7. Report

7.1 The results of each calibration shall be reported accurately, clearly, unambiguously, and objectively. Each report shall include at least the following information:

7.1.1 Calibration report title.

7.1.2 Name and address of the laboratory.

7.1.3 Reference to this standard practice.

7.1.4 Name and unambiguous identification of the instrument being calibrated.

7.1.5 Name or identification of the reference standard(s), or both, certified reference material(s), or reference material(s) used with this practice.

7.1.6 Name of the person conducting the calibration.

7.1.7 The date and time of the calibration

7.1.8 The specific operating conditions - accelerating voltage (kV), working distance (mm), imaging mode, and indicated magnification.

7.1.9 Other operating conditions which may influence the results such as degradation of the reference standard or power fluctuations.

7.1.10 The calibrated magnification in both X and Y.

7.1.11 The relative error of the calibrated magnification (see Section 8) or some other measure of the statistical reliability of the result.

7.2 Tabulating the results in a "conditions" matrix will facilitate the identification of improper instrument settings or instrumental problems. Fig. 2 is an example of such a matrix.

8. Precision and Bias

8.1 The precision and bias of the calibrated magnifications are directly related to the operator's ability to obtain identical operating conditions. Many factors influencing these are given in Annex A1.

8.2 Each facility using this standard practice should determine the precision and bias for the calibrated magnifications.

8.3 A measure of the precision can be easily determined by estimating the maximum error of the measured distance *D*. For example, if *D* was measured to the nearest 1mm then the maximum error on *D* would be delta $= \pm$ 0.5 mm. The precision is estimated by:

$$\text{Precision in } \% = \pm (\text{Delta}/D)*100$$

Fig. 3 illustrates estimated precision for three typical situations.

8.4 The relative error associated with the calibration will be approximately equal to the sum of the relative error of the reference standard and the relative error of *D*.

9. Application of Magnification Accuracy to the Measurement of Sample Detail

9.1 The preceding measurements involve measuring pattern pitch. Pitch is specifically used to avoid errors in locating the measurement starting and stopping points. These errors are related to edge effects from electron beam penetration, shadowing of sample detail, changes in sample geometry and the choice of imaging modes. All these errors offset each other when pitch patterns are utilized.

9.2 Once the magnification accuracy is established measurements with the same accuracy can be made on a sample only if the sample contains a repeating structure. For instance, the measurement of a sphere's diameter will not necessarily have the same accuracy as that given by the accuracy of the magnification. The location of the edges are subject to some uncertainty and will vary depending upon contrast settings and the accelerating voltage.

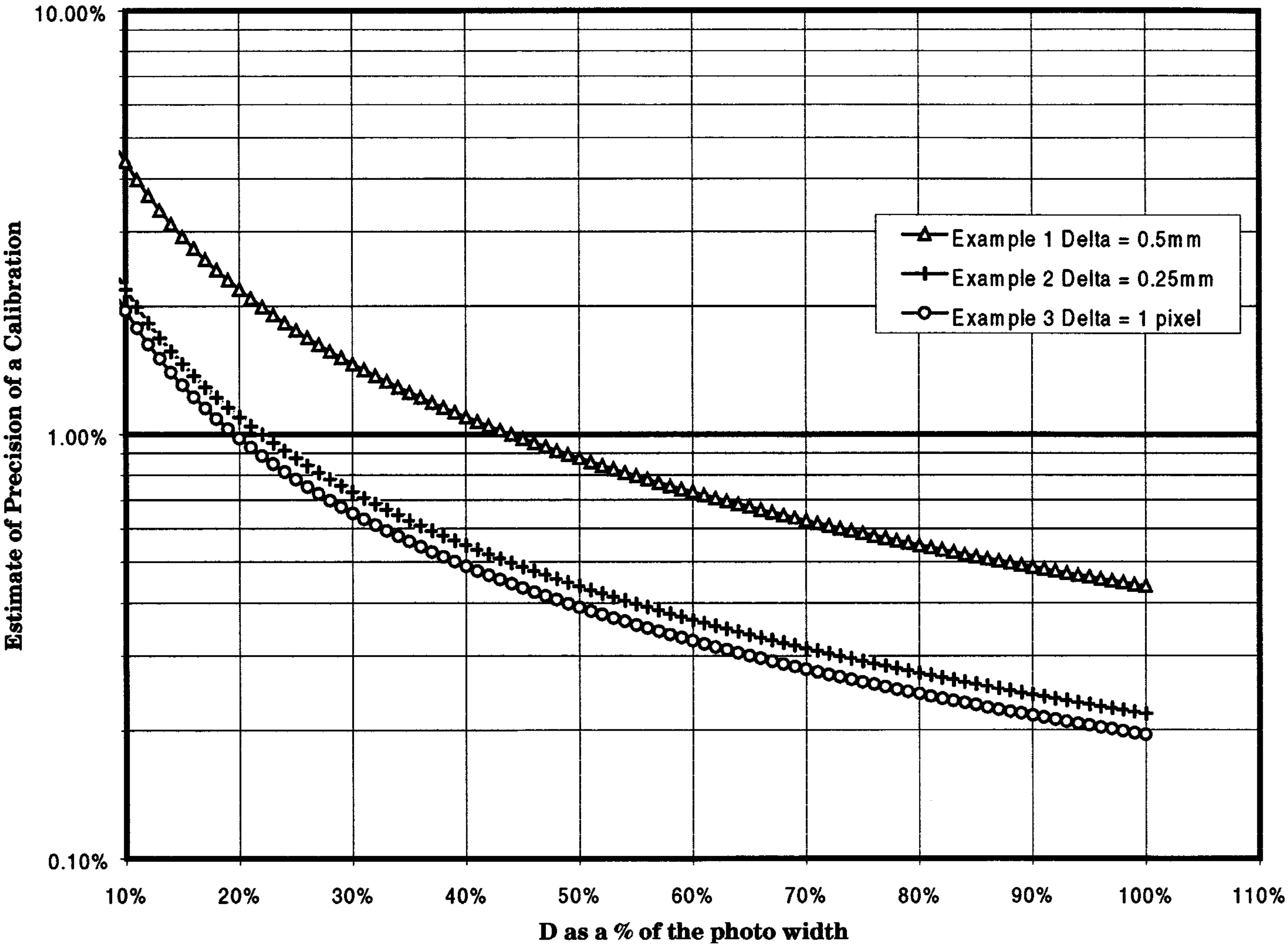
Magnification Calibration Table									
Acc. Voltage		2keV		5 keV		10 keV		20 keV	
Magnification	WD	X	Y	X	Y	X	Y	X	Y
100	10								
	20								
	30								
1,000	10								
	20								
	30								
10,000	10								
	20								
	30								
Instrument:									
Operator:									
Date:									

NOTE 1—Insert the actual magnification measured against the calibration standard.

NOTE 2—The above accelerating voltage, magnifications, and working distances are for example only. These items should be adjusted to represent those settings that are used in actual practice. You may use a different number of settings than in this example.

NOTE 3—The data should be plotted in a control chart to show variability over time.

FIG. 2 Example of a "Conditions" Matrix for Tracking Calibrated Magnifications as Part of a Quality Control Program



Example 1—D measured to the nearest 1mm on a 4 × 5 in. (mm) photomicrograph.
Example 2—D measured to the nearest 0.5 mm on a 4× 5 in. (mm) photomicrograph.
Example 3—D measured to the nearest 1 pixels on a 512 × 512 image.

FIG. 3 Estimate of Precision of a Magnification Calibration Where the Measured Length D is Given as a Percentage of the Photo Width

10. Keywords

10.1 calibration; magnification; pitch; scanning electron microscope; SEM

ANNEX

(Mandatory Information)

A1. PARAMETERS THAT INFLUENCE THE RESULTANT MAGNIFICATION OF AN SEM

A1.1 The parameters listed below may interact with each other, and are considered in order of their location in the instrument from electron source to the recorded photograph.

A1.1.1 Electron gun high-voltage instability or drift can change the energy of the electrons, therefore changing the final focus.

A1.1.2 Different condenser lens strength combinations change the focal point of the final lens.

A1.1.3 Uncorrected final lens astigmatism can give false indication of exact focus.

A1.1.4 Residual magnetic hysteresis, particularly in the final lens, can change the focal conditions.

A1.1.5 Long depth of focus, particularly at low magnification and small beam divergence controlled by lens and aperture selection, can lead to incorrect focus.

A1.1.6 Nonorthogonal deflection (x-y axis) can be produced by the scan coils.

A1.1.7 Scan generator circuits may be nonlinear or change with aging of circuit components or both.

A1.1.8 Zoom control of magnification can be nonlinear.

A1.1.9 Nonlinearity of scan rotation accessory can distort magnification at different degrees of rotation.

A1.1.10 Distortion of the electron beam sweep may occur from extraneous magnetic and electrostatic fields.

A1.1.11 The percent error in magnification may be different for each magnification range. The range is usually determined by a separate resistor chain.

A1.1.12 A tilted sample surface (not perpendicular to the beam axis) will introduce foreshortening and magnification variation.

A1.1.13 The tilt correction applied may not be at 90° (in the plane normal to the electron beam) to the tilt axis of the specimen or of a particular area on the specimen surface.

A1.1.14 Signal processing, particularly differentiation or homomorphic processing, can give a false impression of focus. However, it is permissible to use d-c suppression (sometimes called differential amplification, gamma processing, black level/gain, dark level, or contrast expansion) because its effect on the image is isotropic (that is, nondirectional).

A1.1.15 The objective lens on some instruments may be electrically coupled to the magnification meter; thus focus and magnification are operator dependent.

A1.1.16 For the same apparent magnification, two different combinations of working distance and beam scan raster will produce different linear magnifications.

A1.1.17 Thermal and electronic drift of circuit components related to the above parameters can affect magnification with time in random manner.

A1.1.18 Distortion of faceplate and non-orthogonal beam deflection of recording CRT can produce nonlinear magnification.

A1.1.19 The image magnification on the recording CRT may not be the same as that on the viewing CRT.

A1.1.20 Recording camera lens distortion and change in ratio of the photographic image to CRT image can lead to magnification errors.

A1.1.21 Expansion or contraction of photographic material, photographic enlarging, and control of contrast, can all have a significant effect on the final apparent image magnification.