ASTM INTERNATIONAL

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Standard Test Methods for
Rating and Classifying Inclusions in Steel Using the Scanning Electron Microscope[1]

This standard is issued under the fixed designation E2142; 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 test method covers procedures to obtain particle size distribution, chemical classification, and Test Methods E45 ratings of inclusions in steels using an automated scanning electron microscope (SEM) with X-ray analysis and automatic image analysis capabilities.

1.2 There are three discrete methods described. Method 1 is the SEM analog of Test Method E45, which uses image analysis and light microscopy to produce automated Test Methods E45 ratings. Method 2 produces similar ratings based predominantly on sorting inclusions by chemistry into the traditional classes defined in Test Methods E45. Method 3 is recommended when explicit detail is needed on particular inclusion types, not necessarily defined in Test Methods E45, such as to verify the composition of inclusions in inclusion-engineered steel. Method 3 reports stereological parameters such as volume or number fraction, rather than Test Methods E45 type ratings.

1.3 This test method deals only with the recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability for any grade of steel or other alloy where the method is appropriate.

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

1.5 *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.*

1.6 *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:*[2]
E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E45 Test Methods for Determining the Inclusion Content of Steel
E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope
E768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel
E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
E1508 Guide for Quantitative Analysis by Energy-Dispersive Spectroscopy

2.2 *Adjuncts:*
ANSI/IEEE STD 759 IEEE Standard Test Procedure for Semiconductor X-Ray Energy Spectrometers[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Analysis Rules*—

3.2.1.1 *acquisition analysis rules*—include the criteria to terminate X-ray collection (counts or time, or both), the list of elements to be analyzed, the number of fields or particles to be analyzed, morphologies of particles from which spectra will be collected, etc. (see Appendix X1 for a more complete listing of typical Acquisition Rules).

3.2.1.2 *post-acquisition analysis rules*—define ratios of X-ray intensities or elemental compositions required to identify

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[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., P.O. Box 1331, Piscataway, NJ 08854-1331, http://www.ieee.org.

an inclusion as belonging to a particular chemical classification and, for Methods 1 and 2 herein, define the main inclusion class (A, B, or C) to which each chemical classification belongs.

3.2.2 *chemical classification*—defined compositional categories in which inclusions are placed according to the analysis rules. Categories may be broad (for example, sulfide, aluminate, silicate) or more precise (for example, calcium sulfide, calcium silicate, anorthite, etc.).

3.2.3 *critical aspect ratio*—the aspect ratio of a single inclusion that defines the boundary between "globular" and "elongated".

3.2.4 *discontinuous stringer*—two or more Type C or three or more Type B inclusions aligned in a plane parallel to the hot working axis and offset from the stringer centerline by no more than 15 μm, with a separation of < 40 μm between any two nearest neighbor inclusions.

3.2.5 *stringer*—an individual oxide inclusion that is highly elongated in the deformation direction; or two or more Type C, or three or more Type B, inclusions aligned in a plane parallel to the hot working axis and offset (from the stringer centerline) by no more than 15 μm, with a separation of < 40 μm between any two nearest neighbor inclusions.

4. Summary of Test Method

4.1 A properly prepared as-polished metallographic specimen is mounted in a computer-controlled SEM equipped with image analysis and X-ray analysis subsystems such that inclusions intersecting the plane of polish can be imaged and analyzed.

4.2 During analysis, an inclusion is detected by its gray level in the back-scattered electron signal. Once detected, size parameters of the inclusion are automatically determined and its X-ray spectrum collected. Particle morphology, chemistry, and location are stored in the computer, allowing re-examination of the data or the particle itself. In this manner, a complete or partial size distribution of inclusions by chemical class can be determined.

4.3 There are three methods described (see Fig. 1):

4.3.1 Method 1 is most similar to Test Methods E45 which uses light microscopy, and is intended to be its SEM analog. As such, it uses morphology as the primary basis for sorting particles into classes. As in Test Methods E45, a critical aspect ratio of 2 is defined. Chemistry is used to identify sulfide inclusions and to discriminate among sulfides when more than one type is present, as well as to ensure that exogenous inclusions and surface scratches and debris are not included in the ratings. Inclusions will be classified into four categories, Types A, B, C and D as described in Test Methods E45. Elongated sulfides are Type A. Other inclusions are oxides and are classified as Types B, C or D, depending on their morphology, as follows: discontinuous stringers of three or more inclusions with member particles having low aspect ratio are Type B; discontinuous stringers of two or more inclusions with member particles having high aspect ratio and single elongated oxide inclusions are Type C; remaining isolated inclusions are Type D. The categories will be further subdi-

vided as thin, heavy and oversized, according to their width (tabulated in Table 1). E45-equivalent ratings are determined and recorded, and reported particles are usually limited to those ≥ 2 μm in size.

4.3.2 The inclusion classification strategy in Method 2 is based predominantly on chemistry, but uses morphology when necessary, such as to classify Type D (globular) inclusions and to compute severity ratings for Type B and C inclusions (which require determination of stringer lengths). Method 2 is based on the underlying intention of Test Method E45, namely, that Type A inclusions are deformable sulfides, Type B inclusions are non-deformable oxides (typically alumina), and Type C inclusions are deformable oxides (typically silicates). Each defined chemical class is assigned to one of these categories. Once classifications are made based on chemistry, the globular particles from each classification, or from designated classifications, can be re-classified as Type D inclusions. E45-equivalent ratings are determined and recorded, and reported particles are usually limited to those ≥ 2 μm in size.

4.3.3 Method 3 allows inclusions to be analyzed and reported in a manner individualized to the material and application of interest. It allows complete freedom in formulating chemical classes. Aspect ratio definitions can be chosen appropriately for the application. Termination of the analysis can be based on detecting a certain number of inclusions rather than sample area. Size and morphology distributions of all inclusions by chemical class are determined. Indirect terminology such as "Type A, B, C and D" and "thin" and "heavy" is not used; rather, inclusions are classified directly by chemical class and size range of interest. Particles may be further subdivided by morphology. Method 3, as in Practice E1245, reports basic stereological parameters, such as volume and number fractions of inclusions within each field, as well the maximum Feret's diameter for each inclusion. This method would be used for custom analyses, such to report all non-sulfide particles by thickness, as may be useful in tire cord applications.

5. Significance and Use

5.1 This test method is established to cover automated SEM/EDX-based procedures for:

5.1.1 Rating the inclusion content of steels based on procedures listed in Standards E45 and E1245, with the significant difference that the composition of the individual inclusions, as determined by X-ray analysis, is utilized to sort them into chemical classes.

5.1.2 Determining the number, size and morphological distribution of inclusions in steels sorted by chemical class.

5.2 Methods 1 and 2 of this test method are primarily intended for rating the inclusion content of steels deoxidized with silicon or aluminum, both silicon and aluminum, or vacuum-treated steels without either silicon or aluminum additions. Guidelines are provided to rate inclusions in steel treated with rare earth additions or calcium-bearing compounds (13.4). When such steels are evaluated, the test report should describe the nature of the inclusions rated according to each inclusion category (A, B, C, D).

5.3 Methods 1 and 2 will provide a quantitative rating of the inclusion content in half-severity number increments from 0 to
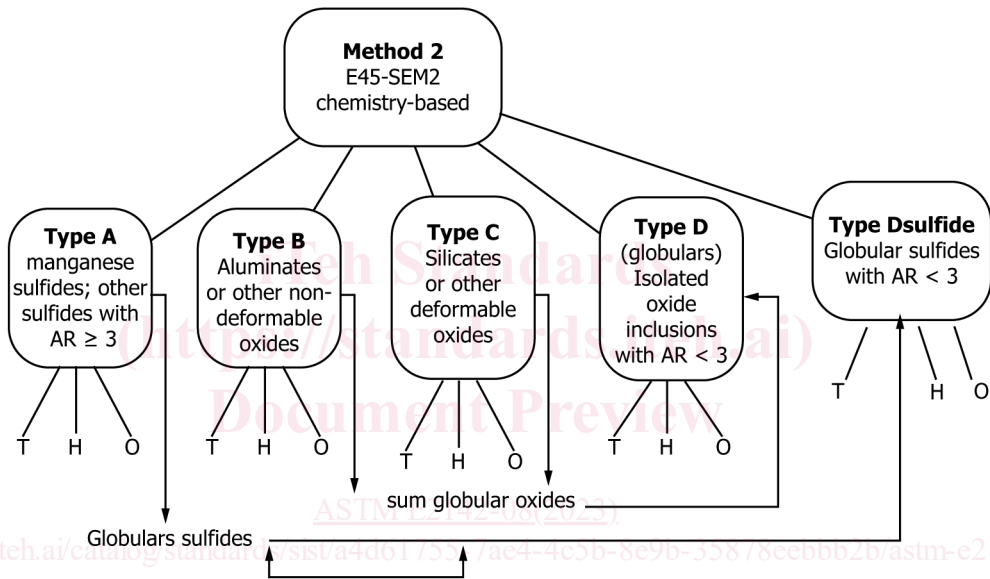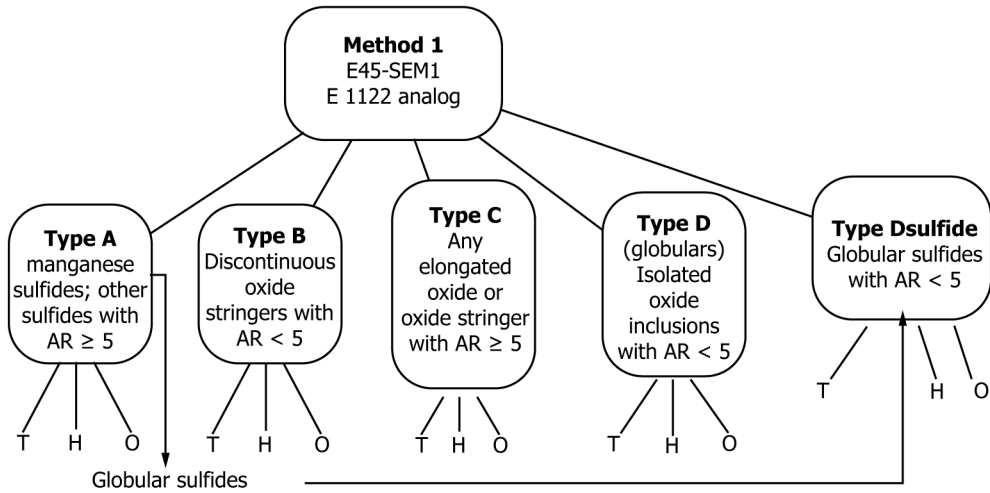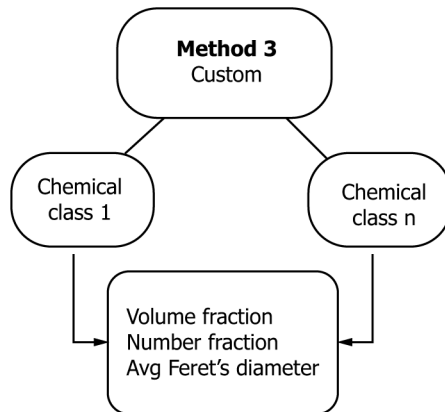
FIG. 1 Illustration of Classification Methods

TABLE 1 Inclusion Width Parameters

Inclusion Type	Thin		Heavy		Oversize
	Minimum Width (μm)	Maximum Width (μm)	Minimum Width (μm)	Maximum Width (μm)	Minimum Width (μm)
A	≥2	4	>4	12	>12
B	≥2	9	>9	15	>15
C	≥2	5	>5	12	>12
D	≥2	8	>8	13	>13

5 for each inclusion type and thickness (Method D of Test Method E45), and in tabulated in Table 2. Test Method E45 ratings by SEM may differ from those determined following E45 because of the use of chemistry in the classifications. In order to differentiate E45 ratings obtained using the SEM from traditional ratings using light microscopy, the ratings obtained using Method 1 or 2 of this Test Method shall be identified as E45-SEM1 and E45-SEM2, respectively.

5.4 Method 3 defines procedures to analyze and report inclusions by arbitrary size distribution and chemical classifications. It may be made applicable to any material by appropriate choice of these classifications.

5.4.1 Method 3 determines and reports basic (as used in Test Method E1245) stereological measurements (for example, volume fraction of sulfides and oxides, the number of sulfides or oxides per square millimeter, and so forth). This test method, however, does not address the measurement of such parameters. E45 ratings are not produced in Method 3 because the inclusion classifications do not follow those defined in Test Method E45.

5.5 The quantitative results are intended to provide a description of the types and amounts of inclusions in a heat of steel. This test method contains no guidelines for such use.

6. Apparatus

6.1 An automated computer-controlled Scanning Electron Microscope equipped with the following accessories:

6.1.1 *Digital Imaging* hardware and software.

6.1.2 *Computer-Controlled Motorized X-Y Stage.* It is conceivable that the method described in this standard may be performed without an automated stage. Performing this method with a manual stage will be tedious, and accuracy will suffer due to difficulty in tracking inclusions across field boundaries.

Therefore, an automated stage, although not essential, is highly recommended and is assumed throughout this document.

6.1.3 *Energy Dispersive X-Ray (EDX) Analyzer.* A "light element detector" (one equipped with a sufficiently thin window to effectively transmit the low energy X rays characteristic of the elements below sodium) is required if oxygen is to be discretely measured, or if analysis of carbides, borides, or nitrides is to be included. The EDX detector must have resolution better than 155 eV as measured in accordance with ANSI/IEEE STD 759.

6.1.4 *Backscattered Electron Detector* and electronics with ability to set one or more thresholds to enable discrimination between inclusions and matrix.

6.1.5 *Control and Image Collection Software* to control the beam and stage and to collect images and spectra according to user-specified parameters.

6.2 *Automated Feature-Analysis Software,* capable of:

6.2.1 Allowing analysis rules to be set such that chemical classifications can be made and features sorted according to chemistry as well as size and morphology.

6.2.2 Distinguishing between elongated and globular particles based on aspect ratio.

6.2.3 Separating the stringered oxides according to the difference in morphology (Type B or C) and measuring the stringer lengths per field of each type.

6.2.4 Connecting stringers which cross field boundaries.

6.2.5 Generating standardized reports.

6.3 *Special Considerations*—The environment housing the equipment must be controlled. Computer equipment, SEMs and EDX systems all require control of temperature and humidity and the air must be relatively dust free.

7. Sampling

7.1 Sampling is done in accordance with the guidelines given in Test Method E45.

8. Test Specimens

8.1 The location and orientation of test specimens shall be as described in Test Method E45. In all cases, the polished surface shall be parallel to the hot-working axis. Studies have demonstrated that inclusion length measurements are significantly affected if the plane of polish is angled more than 6 degrees from the longitudinal hot-working direction. Test specimens should not be cut from areas influenced by shearing which alters the true orientation of the inclusions.

8.2 The surface to be polished must be at least 160 mm^2 (0.25 in^2) in area. It is recommended that a significantly large

TABLE 2 Minimum Values for Inclusion Severity Rating Levels for Measurements in Micrometers (For expression in other units, see Test Methods E45, Table 2)

Test Method E45 Rating Limits (μm at 1× or count)				
Severity	A	B	C	D
0.5	37.0	17.2	17.8	1
1.0	127.0	76.8	75.6	2
1.5	261.0	184.2	176.0	4
2.0	436.1	342.7	320.5	9
2.5	649.0	554.7	510.3	16
3.0	898.0	822.2	746.1	25
3.5	1181.0	1147.0	1029.0	36
4.0	1498.0	1530.0	1359.0	49
4.5	1898.0	1973.0	1737.0	81
5.0	2230.0	2476.0	2163.0	100

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area should be obtained so that the measurement may be made within the defined area away from the edges of the sample.

9. Specimen Preparation

9.1 Metallographic specimen preparation must be carefully controlled to produce acceptable quality surfaces for image analysis. Guidelines and recommendations are given in Method E3 and Standards E45 and E768.

9.2 Polishing must reveal the inclusions without interference from artifacts, foreign matter, or scratches, although the use of chemistry will minimize the errors associated with these features. Polishing must not alter the true appearance of the inclusions by excessive relief, pitting, and pull-out. Use of automatic grinding and polishing devices is recommended.

9.3 Inclusion retention is generally easier to accomplish in specimens that are hardened rather than in annealed condition. If inclusion retention is inadequate in annealed specimens, they should be subjected to a standard heat treatment cycle using a relatively low tempering temperature. After heat treatment, the specimen must be descaled and the longitudinal plane must be reground below any decarburization. This recommendation only applies to heat-treatable steel grades.

9.4 Mounting of specimens is not required if unmounted specimens can be properly polished.

9.5 Polishing practice should follow Practice E768.

10. Calibration and Standardization

10.1 The SEM magnification should be calibrated according to E766. It is important to calibrate the magnification of the SEM to obtain accurate E45 ratings and to ensure that analysis time is minimized. The number of particles of a given size increases strongly as size decreases; if particles below the desired low size limit are included due to magnification error, the number of spectra collected, and therefore the total analysis time, will increase significantly.

10.2 The EDX energy calibration should be done according to section 8.1 of E1508.

10.3 The EDX energy resolution should be checked periodically. The energy resolution, defined as the Full Width at Half Maximum (FWHM) height of the Mn Kα X-ray line, after background has been subtracted, should be measured according to the practice suggested by the manufacturer, provided that it is in accordance with the IEEE methodology.

11. Procedure

11.1 Prepare specimens following the standard protocol set forth in Practice E768. At this time, a small piece of aluminum tape or other reference material may be placed on the edge of the sample. The tape may later be used as a target in order to determine the proper setting of the electron probe current or to check its stability.

11.2 Position the sample in the SEM at a working distance that is suitable for both BSE and EDX.

11.3 Set the beam accelerating voltage appropriately for the elemental range of interest, bearing in mind that excessive voltage will give rise to an (unwanted) increase in matrix contribution to the spectrum. Use of 10–15 kV is typical, although slightly lower or higher voltages may be appropriate depending on the particular application. Use the microscope manufacturer's procedures for saturating the filament, aligning the column and setting other parameters to optimize image quality.

11.4 Calibrate the X-ray analyzer such that the collected spectrum will include all the elements of interest; 0–10 keV is recommended. If there are X rays of interest above 10 keV (such as Pb L lines), use 0–20 keV.

11.5 Set electron probe current by direct measurement using a pico-ammeter and Faraday cup, if the optimum probe current has previously been determined. Alternatively, the current can be set by moving the aluminum tape under the beam and recording X-ray counts. Probe current (or "spot size", which is proportional to probe current) is adjusted until approximately 40 % dead time, if possible, is achieved. The steel matrix itself may be used as the basis of current setting in place of the tape, but this will likely result in the least consistent setting of the described methods.

11.6 Select the BSE imaging mode, which is used because the brightness of a feature in the BSE image is directly related to its average atomic number. The matrix, which consists primarily of iron, will be brighter than some inclusions (for example, MnS) and darker than other inclusions (for example, Pb). Since inclusions are discriminated by the BSE gray level, the threshold(s) must be set appropriately using the procedure recommended by the manufacturer.

11.7 Select and store the region of the sample to be examined following the stage control manufacturer's recommended procedure. The region can be larger than but not smaller than 160 mm^2; if the sample region is larger, then the software shall select a contiguous area of exactly 160 mm^2 wholly contained within the user-selected region to analyze. In Method 3 of this Test Method, analysis can be based on the number of inclusions detected rather than sample area.

11.8 As the beam rasters the selected region, the software recognizes features that fall within the previously defined range of gray-levels. Morphological and chemical parameters are immediately calculated and stored or, alternatively, raw data is stored for off-line processing.

11.8.1 In Test Method E45 inclusions are examined using field areas of 0.50 mm^2 and magnifications of 100×. The inclusions can be examined and discriminated by type using magnifications other than 100× and field areas other than 0.50 mm^2 as long as the severity ratings (see Section 12) are based on the required 0.50 mm^2 field area.

11.9 *Define the Analysis Rules:*

11.9.1 The EDX acquisition should continue until sufficient statistics are accumulated to classify the inclusion. For a discussion on X-ray counting and chemical classification statistics, see Appendix X2 and standard text books.[4] The

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[4] Goldstein, et al, *Scanning Electron Microscopy and X-Ray Microanalysis*, 2nd ed, Plenum Publishing Corporation, New York, NY, 1992 , pp 493-505.

minimum number of counts in a peak necessary for peak identification must be entered.

11.9.2 Define the relevant chemical classes and their analysis rules. In Method 2, for example, at least three chemical classes are defined: sulfides, aluminates, and silicates. Additional classes may be defined, depending on the application. For example, a "calcium silicate" class may be defined and included as Type B, as such inclusions appear similar to and have the same detrimental effects as traditional Type B inclusions. Each chemical class and the main inclusion class to which it is assigned should be reported.

11.9.3 Define the measure of intensity in the X-ray spectrum which must be met in order to identify the particle as belonging to a certain classification. Each class should be defined in terms of one or more of the following: (*1*) peak intensity range, (*2*) peak to background ratio, (*3*) peak intensity ratios, (*4*) elemental percentage as calculated by established methods, or (*5*) other chemical measurement(s) that characterizes a specific type of inclusion. This choice is either narrowed or made by the system or software manufacturer.

11.10 Set the relevant imaging parameters such as the magnification(s) to be used, the minimum and maximum particle sizes to be recorded, and the critical aspect ratio defining an elongated inclusion (see 11.14). Appendix X1 provides a more complete list of analysis rules.

11.10.1 For the selected magnification, digital imaging resolution should be chosen such that there are an adequate number of pixels in each inclusion for the computer program to accurately make measurements. In order to detect a 2 µm particle, the step size of the electron probe, which is in fact the pixel size, must be at most 2 µm. If a 256×256 image is displayed on a 10 cm screen, the field of view is 512 µm wide, and the magnification is 195.3 × (magnification = 10/0.0512). However, to accurately measure the size of a 2 µm particle to within, say, 10 %, a step size of 0.2 µm would be dictated, corresponding to a magnification of 1953× . Depending upon the inclusion analysis software, such pixel size and magnification may be selected automatically, based on the minimum inclusion size of interest input by the user. In the example given, a magnification of 195.3× could be used to search for inclusions; once detected, the magnification is automatically increased to 1953× to measure the inclusion dimensions. The inclusion analysis software must include this or an equivalent analysis strategy to provide the required accuracy.

11.11 Start the analysis, which will run unattended in a completely automated system.

11.12 Ratings similar to E45 ratings will be determined automatically within Methods 1 and 2 of this Test Method. Inclusions will be classified according to type (or chemistry), morphology and thickness. Since ratings using light microscopy may differ from those using the SEM, ratings resulting from application of this Test Method shall be called E45-SEM1, if method 1 is used, and E45-SEM2, if method 2 is used.

11.13 The acquired raw data should be saved, unaltered by the application of any analysis software. The raw data can then be used at a later time for re-classification of the inclusions based on different criteria.

11.14 A critical parameter in the morphological characterization of an inclusion is the Aspect Ratio (AR), at or above which an inclusion is considered elongated. In Test Methods E45, which relies on morphology to distinguish oxide types, a relatively high AR of 2 is used in order to more reliably differentiate silicates, which are generally highly elongated, from aluminates, which are less elongated. In this Test Method, the X-ray spectrum from the inclusion is directly obtained and will serve to differentiate aluminates from silicates, reducing the dependence on morphology. In Method 2, used for "chemistry-based" E45 ratings, and Method 3, used for custom analyses, a critical AR of 2 is suggested for consistency with E45, but other values of AR may be used. Inclusion analysis software must allow the critical AR to be selectable as an Analysis Rule, with default settings as described above.

11.15 In Method 3, the analysis will automatically terminate when a minimum number of inclusions has been classified (for example, 1000) or when a specified area of the sample has been examined (For example, 160 mm²), whichever occurs first. The use of this two-criteria approach to terminate analysis will ensure that steels with low inclusion content will be examined over a representative area, and that a meaningful number of inclusions will be classified in more typical products in a reasonable amount of time.

12. Classification of Inclusions and Calculation of Severities: Method 1: the SEM analog to Test Methods E45, based on Morphology, Chemistry and Thickness

12.1 Method 1 is the SEM analog to the light-microscopy-based Test Method E45, using the same inclusion classifications and morphological definitions. The primary difference in this method is that the spectrum from a feature is used to determine that it is in fact an inclusion, to differentiate sulfides from oxides, and to classify complex or unusual inclusions.

12.2 Method 1 implies the use of the latest edition of Test Method E45 in the SEM with the addition of EDS qualitative chemical compositions.

12.3 The indigenous inclusions in steels deoxidized with rare earth elements or treated with calcium-containing materials are also classified according to morphology and thickness, with the added requirement that appropriate chemical classes be defined, and inclusions within these classes be tabulated in the report.