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

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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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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
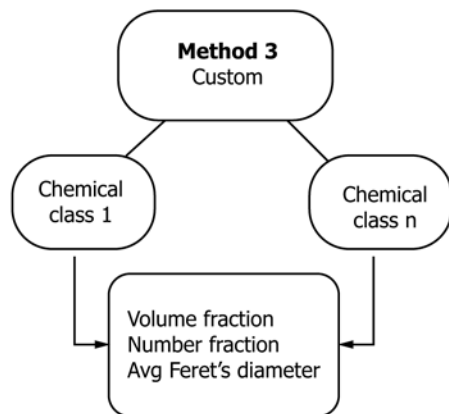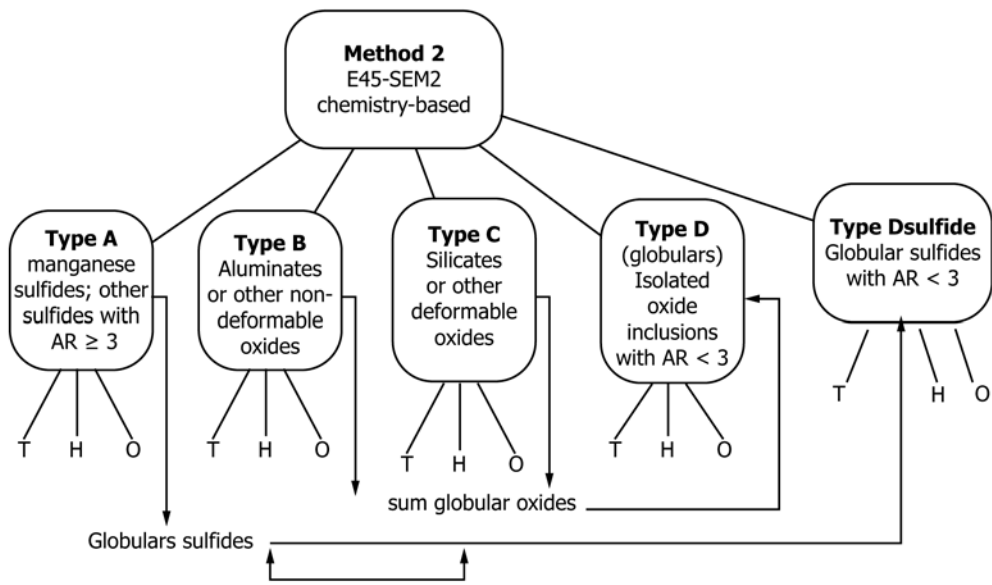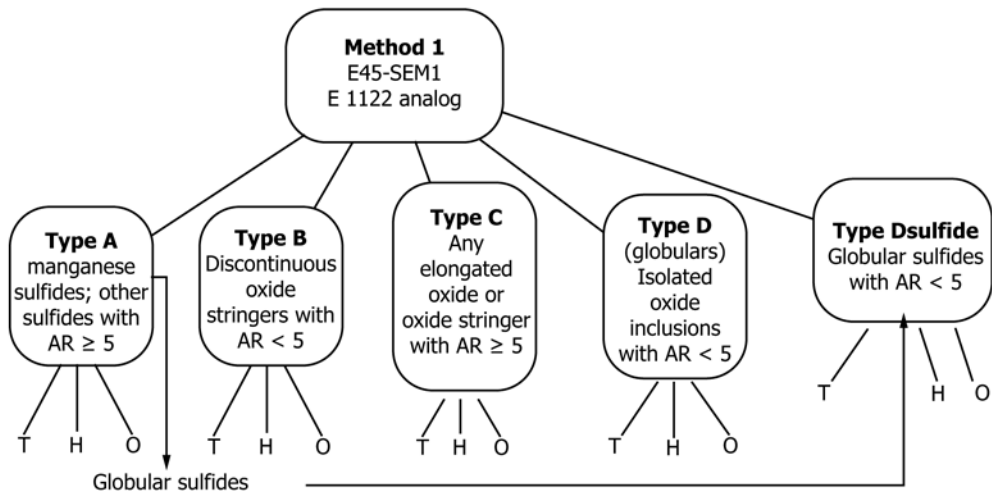
FIG. 1 Illustration of Classification Methods

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

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 mm^2), 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.

13. Classification of Inclusions and Calculation of Severities: Method 2, based on Chemistry, Morphology and Thickness

13.1 The underlying intent of the classification strategy used in Test Method E45 and its usual interpretation is that deformable sulfides, typically manganese sulfides, are Type A, non-deformable oxides, typically alumina, are Type B, and deformable oxides, typically silicates, are Type C. The morphology developed by an inclusion after processing is a result of its deformability. If the deformability of an inclusion type is known, there is no need to rely on its morphology after deformation to classify it. Method 2 of this Test Method, using appropriately defined analysis rules, sorts detected inclusions into chemical classes and then into the appropriate inclusion class, based on known deformability characteristics.

13.1.1 Chemical classifications are defined to cover the inclusion types of interest, and then each chemical class is assigned to one of the main inclusion classes: Type A (deformable sulfides), Type B (non-deformable oxides), and Type C (deformable oxides).

13.1.2 If discriminating sulfide types is unimportant in a given applicatinon, then Type A can be defined more generally as simply "sulfides".

13.2 In some cases, the A, B and C classifications may be sufficient. Typically, however, knowledge of the isolated inclusion content is also of interest. In these applications, a Type D classification is established for globular oxides and, if appropriate, a Type $D_{sulfide}$ classification for globular sulfides. Type D and Type $D_{sulfide}$ classes are populated by removing the globular inclusions from Type B and C (oxide) and Type A (sulfide) classes and restating them as Type D and $D_{sulfide}$, respectively.

13.2.1 It may be appropriate in some applications to retain the globular particles from certain designated chemical classes in the original main inclusion category, rather than restating it as a D-type. For example, if an alumina inclusion is found, but is not present as a stringer, it may still be appropriate to classify it as Type B using this method. Decisions on which classes will be used to form the Type D category should be made according to producer-purchaser agreement.

13.3 Method 2 is used to rate inclusion content according to direct determination of inclusion type using its chemistry, rather than relying on morphologies developed as a result of hot working. In this methodology, the knowledge of chemistry will clearly separate Type B aluminates from Type C silicates.

13.3.1 A critical aspect ratio of 2 is also used to separate deformable sulfides from globular sulfides.

13.3.2 In Test Method E45 as in Method 1 of this Test Method, isolated inclusions with aspect ratios between 3 and 5 will be classified as Type D globulars, whereas in Method 2 they will be more appropriately classified as Type C, if they meet the defined chemical classification criteria.

13.4 The indigenous inclusions in steels deoxidized with rare earth elements or treated with calcium-containing materials are treated similarly. Chemical classifications are defined for the expected inclusion types (such as Calcium Sulfide,

Calcium Silicate, Anorthite, Spessartite, etc.) and assigned to the appropriate main inclusion class.

13.4.1 Calcium sulfide is less deformable than manganese sulfide. Therefore, it is likely that calcium sulfide inclusions will have AR < 2 and will be re-classified as Type $D_{sulfide}$ if such a category is established.

13.4.2 In Method 1 of this Test Method, a brittle non-deformable oxide inclusion, such as calcium silicate, will likely develop the Type B morphology and will be so classified. In Method 2, Type B can be defined to include several chemical classifications, all of which are known to be non-deformable oxides; one such classification could be "calcium silicate". In both methods, calcium silicate will likely be classified as Type B: in Method 1 by virtue of its morphology; and in Method 2, by virtue of its chemistry.

13.5 Type B and Type C inclusions are subdivided into stringers and isolated particles, in order to determine severity ratings. Depending upon the application, globular oxides may be removed from their chemical class and accumulated as Type D (see 13.2), As in Method 1, categories will be further subdivided as thin, heavy and oversized, according to their width (tabulated in Table 1), recorded particles are limited to those ≥ 2 µm in width, and E45-SEM2 ratings are determined.

13.6 Type A inclusions are usually deformable sulfides having AR ≥ 2. and globular sulfides will usually be classified as Type $D_{sulfide}$ (see 13.1.1 and 13.3.1).

13.7 Complex inclusions, such as oxysulfides or duplex inclusions, are also rated according to chemistry and thickness. If the inclusion is predominantly (> 50% by area) sulfide, it is classified as Type A; if it is predominantly a non-deformable oxide such as alumina, it will be classified as Type B; if it is predominantly a deformable silicate, it will be classified as Type C.

13.7.1 Complex inclusions with AR < 2 may be removed from their chemical class and re-classified as Type D, if they are predominantly oxides, or Type D sulfide, if they are predominantly sulfides.

14. Classification of Inclusions: Method 3: Custom

14.1 Method 3 is intended to be used when Methods 1 or 2 are not applicable. During automated SEM/EDX inclusion analysis, parameters such as size, shape, chemistry and inclusion location are stored. Therefore, these parameters may be summarized into any manner that is useful for a given application, process or specification. Examples of its use include the determination of the number and size distribution of inclusions having a specific composition (such as titanium-bearing inclusions in stainless steels); confirming the distribution and composition of inclusions in inclusion-engineered steel; the analysis of inclusions in steel in the as-cast condition; and the sorting of inclusion content in unique ways, such as to classify all non-sulfide particles by thickness, as may be useful in tire cord applications. Also, specific limits or specifications for steel or other alloys may be easily incorporated into this methodology for a given application.

14.2 In Method 3, relevant chemical classifications, size ranges of interest, and morphology classes may be defined for

the specific application of interest. Indirect terminology such as Type A, B, C or D or thin, thick and heavy is not used. Rather chemical classifications are defined and become the inclusion category headings. The size distribution of the inclusions in each category is determined. The minimum size to be included in the analysis is not defined in this document, but rather is determined appropriately for the application. Higher magnifications may be used to determine inclusion populations in smaller size regimes than is able to be done with optical microscopy.

14.3 The stereological parameters of interest are typically volume fraction, which is equal to area fraction and number per unit area. Ratio parameters such as the average inclusion area or mean free path are calculated. See Practice E1245 for definition and further discussion of these parameters.

14.4 There is no rating system suggested for Method 3 in this document, although such rating systems may be devised and implemented for specific intents and with producer/purchaser agreement.

15. Test Report

15.1 Pertinent data regarding the identity of the specimen analyzed should be reported.

15.2 For E45-SEM ratings computed in methods 1 and 2 of this test method, the number of fields of each inclusion type and thickness category are reported for each severity from 0 to 5 in whole or half-severity level increments. For steels with very low inclusion contents, severities may be computed to one-quarter or one-tenth severity level increments. Note that for D-type inclusions, because one inclusion per field is a severity of 0.5, by definition, there can be no D-severity levels between 0 and 0.5. Fig. 2 illustrates a typical report for E45-SEM values as might be produced using Method 1.

15.3 Modifications to the report may be made, based on producer-purchaser agreements. For example, the report may be simplified to include only certain inclusion types, thicknesses or severity values. Other modifications may include the reporting of worst-field severity ratings only or the number of fields at the worst-field severity ratings.

15.3.1 The use of contiguously aligned fields may not produce true worst-field (Method 1 of Test Method E45) ratings. Valid worst-field ratings require more sophisticated data analysis methods that, for example, automatically review the entire data set, searching for the highest severity rating in any 0.5 mm^2 field, regardless of prior field boundaries.

15.3.2 If desired, by producer-purchaser agreement, an index may be calculated to describe the inclusion content.

15.3.3 To produce average results for more than one specimen per lot, the average number of fields for each severity rating, inclusion type and thickness may be calculated as recommended by Test Method E45 (Table 4 on Example of Inclusion Rating (Method D)).

15.3.4 Data for inclusions or stringers that are oversized in either length or width, or both, should be reported separately. Report the width for all inclusions, as well as the length for Types A, B, and C.

15.3.5 Fields with zero severity levels may be further classified, if desired, as either blank (no inclusions of a particular type and width category are present) or non-ratable (inclusions are present but their length is below the 0.5 severity limit or their width is <2 μm).

15.3.6 For complex or duplex inclusions, report the chemical class and describe their nature, for example, "globular calcium aluminates encapsulated with a thin film of calcium-manganese sulfide", or "irregular aluminates partially or fully embedded in manganese sulfide stringers".

15.4 Since elemental composition data will be available for all inclusions, information pertaining to the composition of the inclusions may be reported in Method 1 if desired. For rare earth- or calcium-treated steels, or other steels with non-traditional deoxidation approaches, the chemical composition of the inclusions, in general terms, must be reported with each rating. Methods 2 and 3 will include chemical data, by definition.

15.5 In all Methods, stereological data determined during analysis may be included in the test report as desired. Standardization of such test data is not governed by this method (see Practice E1245).

15.6 Method 3 will provide complete size distribution data by chemical classifications, including stereological data. Although no rating system has been devised for inclusions outside of those defined in E45, such rating systems can be defined by producer-purchaser agreements.

15.6.1 The reported values will include the number and area fraction, the number of particles per unit area, the mean inclusion Feret's diameter or area, and the maximum Feret's diameter for each particle type. The definitions and formulas used to compute the stereological values are defined in Section 12 of Practice E1245.

15.6.2 A report typical of that generated using Method 3 is shown in Fig. 3, along with an illustration of the analysis rules used to define the chemical classes selected.

16. Precision and Bias

16.1 From the chemistry perspective, precision and bias for multiple runs of a given sample are governed to a large extent by the statistics applicable to collecting reliable X-ray spectra (obtaining adequate counting statistics) from individual inclusions and then to sorting inclusions into the proper chemical classes. These topics are more fully developed in Appendix X2; a detailed treatment of the general topic of X-ray counting statistics can be found in standard text books.[4]

17. Keywords

17.1 automatic image analysis; complex inclusions; EDX; globular inclusions; inclusions; inclusion ratings; inclusion stringers; oxides; second-phase particles; SEM; steel; stereology; sulfides; X-Ray microanalysis

Sample report format

Type A
Histogram of field severity ratings.
Warning: Sample area is less than 160 mm² (320 fields)

Area = 148.5 mm² as 297 fields

Class	Width, µm	0.5	1	1.5	2	2.5	3
Thin	2.0–4.0	118	73	57	24	12	2
Heavy	4.0–12.0	10					
Oversize	> 12.0	1					

Type B
Histogram of field severity ratings.
Warning: Sample area is less than 160 mm² (320 fields)

Area = 148.5 mm² as 297 fields

Class	Width, µm	0.5	1	1.5	2	2.5	3
Thin	2.0–9.0	22	34	5			
Heavy	9.0–15.0						
Oversize	> 15.0						

Type C
Histogram of field severity ratings.
Warning: Sample area is less than 160 mm² (320 fields)

Area = 148.5 mm² as 297 fields

Class	Width, µm	0.5	1	1.5	2	2.5	3
Thin	2.0–5.0						
Heavy	5.0–12.0						
Oversize	> 12.0						

Type D
Histogram of field severity ratings.
Warning: Sample area is less than 160 mm² (320 fields)

Area = 148.5 mm² as 297 fields

Class	Max. Dia. µm	0.5	1	1.5	2	2.5	3
Thin	2.0–8.0	31	75	82	4		
Heavy	8.0–13.0	62	12				
Oversize	> 13.0	22	8	1			

FIG. 2 E45-SEM Report - as would be used with Method 1 of this Test Method

Summary of oversized D inclusions

Part no.	Max Feret's diameter µm	x, mm	y, mm
14	17.8	1.442	11.848
128	16.7	3.926	11.680
229	13.8	6.853	11.964
445	22.1	12.246	11.958
677	16.9	17.986	11.990
849	13.8	21.731	11.952
957	15.5	24.110	11.740
1027	21.6	23.691	10.603
1040	16.2	22.713	10.986
1074	15.0	21.340	10.584
1111	17.2	20.103	11.077
1249	26.5	16.326	10.836
1303	15.1	14.454	10.949
1350	14.6	13.677	10.696
1455	14.7	9.974	10.843
1605	16.6	7.631	10.647
1614	13.1	7.819	10.556
1689	16.2	5.993	10.972
1741	16.7	4.750	10.752
1752	14.3	4.815	10.537
1935	18.2	3.817	9.620
2084	14.6	8.064	9.823
2107	15.4	8.491	9.492
2112	18.7	9.257	10.129
2158	21.7	10.334	9.951
2225	13.8	13.094	10.228
2325	17.8	14.832	9.529
2360	13.8	16.108	10.123
2471	14.1	19.145	9.531
2675	13.8	23.408	8.953
2682	19.3	23.786	8.815
2704	13.4	22.609	8.988
2713	13.8	21.787	9.341
2728	18.0	22.013	8.996
2747	26.4	21.929	8.815
2805	17.6	19.192	9.348
2812	20.2	19.126	9.241
2823	20.3	19.303	9.172
2834	15.9	18.906	9.058
2840	22.4	18.100	9.302
2855	15.8	17.940	9.049
2856	14.2	18.204	8.852
2890	13.8	14.486	8.726
2891	15.8	13.899	9.252

FIG. 2 *(continued)*

Nitrides/Sulfides

Project_Number	MAH809566
Sample_Number	123456
Client_Number	#3, A-10
Description	12345
Datafiles	000200_A\12345.*

Magnification	50	400.0
Fileds	38.1	164.9
Area (sq. mm)	120.5	8.1

Classes	#	Num%	Area Frac	Num/sq. cm
$MnS + Cr_2O_3$	168	63.0	4.61E-05	2.06E+03
MnS	84	28.7	3.35E-05	9.40E+02
$TiN + Cr_2O_3$	7	2.6	2.95E-06	8.60E+01
$MnS + Al_2O_3$	5	1.5	2.78E-06	5.00E+01
TiN + MnS	3	1.1	1.74E-06	3.68E+01
TiN	2	0.8	1.11E-06	2.46E+01
MnS + TiN	3	1.1	7.15E-07	3.68E+01
Other	2	0.8	3.09E-07	2.46E+01
$MnS + TiN + Cr_2O_3$	1	0.4	3.09E-07	1.23E+01
Totals	275	100.0	8.96E-05	3.27E+03

Area Dist by Maximum Feret's Diameter (microns)

Classes	Area%	1-2	2-5	5-10	>>>
$MnS + Cr_2O_3$	51.5	30.5	42.5	19.9	7.1
MnS	37.4	11.8	31.7	23.7	32.8
$TiN + Cr_2O_3$	3.3	0.0	59.3	40.7	0.0
$MnS + Al_2O_3$	3.1	0.0	30.9	29.0	40.1
TiN + MnS	1.9	7.4	0.0	92.6	0.0
TiN	1.2	0.0	100.0	0.0	0.0
MnS + TiN	0.8	21.2	78.8	0.0	0.0
Other	0.3	100.0	0.0	0.0	0.0
$MnS + TiN + Cr_2O_3$	0.3	0.0	100.0	0.0	0.0
Totals	100.0	20.7	38.9	23.2	17.2

FIG. 3 Sample Report for Method 3

Average Composition (%)

Classes	#	Al	Si	S	Cr	Mn	Ca	Ti
MnS + Cr_2O_3	168	0	0	48	10	42	0	0
MnS	84	0	0	56	3	41	0	0
TiN + Cr_2O_3	7	0	0	0	8	0	0	92
MnS + Al_2O_3	5	9	0	54	2	35	0	0
TiN + MnS	3	0	0	30	3	19	0	49
TiN	2	0	0	0	1	0	0	99
MnS + TiN	3	0	0	50	0	40	0	10
Other	2	0	0	80	20	0	0	0
MnS + TiN + Cr_2O_3	1	0	0	26	7	22	0	45
Totals	275	0	0	48	6	38	0	8

Post Acquisition Analysis Rules used to define above chemical classes:

(Numbers indicate a measure of the amount of the various elements in an inclusion for it to be placed in the corresponding class; in this example, atomic % is used. "True" means that the inclusion does not belong to any defined class).

MnS + Cr_2O_3 = Cr>=0.05 and Mn>0.10 and S>0.20 and Mn + Cr + S>0.90 and Cr>Al

MnS = Mn>0.15 and S >0.20 and Mn + S>0.90 and Cr< 0.05

TiN + Cr_2O_3 = Cr>0.10 and Ti>0.30 and Cr + Ti>0.75 and Ti>S

MnS + Al_2O_3 = Al>0.10 and Mn>0.10 and S>0.20 and Al + Mn + S>0.90

TiN + MnS = S>0.20 and Mn>0.10 and Ti>=0.05 and Mn + S + Ti>0.90

TiN = Ti>0.50

MnS + TiN = S>0.20 and Mn>0.10 and Ti>=0.05 and Mn + S + Ti>0.90

MnS + TiN + Cr_2O_3 = Ti>0.05 and Cr>0.05 and Mn>0.07 and S>0.07 and Ti + Cr + Mn + S>0.90

Other = true

FIG. 3 (continued)

APPENDIXES

(Nonmandatory Information)

X1. Typical Acquisition Analysis Rules

X2. X-Ray Counting and Chemical Classification Statistics

TABLE X1.1 Typical Acquisition Analysis Rules To Be Set For SEM Analysis

Parameter	Explanation
Magnification	Magnification to be used for overall scan. Magnification may be automatically increased to measure identified particles.
Minimum inclusion size	Minimum acceptable particle size (avg Feret's diameter).
Maximum inclusion size	Maximum acceptable particle size (avg Feret's diameter).
Maximum number of inclusions	An analysis termination criterion. The analysis will terminate after this number of inclusions has been identified and accepted.
Maximum number of fields	An analysis termination criterion. The analysis will terminate after this number of fields has been analyzed.
Maximum total time	A termination criterion for the entire inclusion analysis task.
Minimum Aspect Ratio	The minimum aspect ratio necessary to consider an inclusion elongated. Aspect ratio is defined as the ratio of the maximum diameter (length) to the diameter which is perpendicular to the maximum diameter.
Minimum spectrum counts	The sum of the counts in all peaks in the EDS spectrum that a particle must meet or exceed for the particle to be accepted. May be used as a threshold for rejecting ill-defined spectra.
Minimum counts necessary for an element to be recognized	Minimum acceptable count for each element. One accepted criterion for a peak to be considered significant is that the number of net counts in the peak (P) must exceed the Background counts (B) by three times the square root of B, or $P > 3(B^{1/2})$.
Maximum counting timer per inclusion	Maximum spectrum accumulation time for accepted particles. Other criteria may be used to terminate the analysis sooner.
Maximum particles per field	A termination criterion. The analysis of a field stops after this number of particles have been analyzed and accepted.
X-ray acquisition mode	Point—Place the beam at the estimated particle centroid. Chord—Repeatedly redraw the measurement chords. Raster—Scan a rectangular area which fits inside the particle's perimeter. Perimeter—Repeatedly trace the particle perimeter.
Size of search grid	The grid of points to be used when searching for particles: 256 × 256 or 512 × 512, for example.
Search dwell time	In particle detection, the amount of time, in milliseconds, for which the beam will dwell on each point.
Measure dwell time	For particle size measurement, the amount of time, in microseconds, for which the beam will dwell on each point when measuring particles. The higher the value, the less the measurement will be subject to image noise, at the expense of analysis speed.
Image dwell time	Dwell time per pixel used to collect a digital image.
Chemistry format	The format used to store the chemistry for each particle, for example, as concentration, counts, intensity ratios, etc.

X2.1 X-Ray Counting Statistics

X2.1.1 The generation and measurement of X-rays is a statistical process. In an SEM, electrons impinge on the sample, and if they have sufficient energy, there is a finite probability that X-rays will be generated. Further, there are finite probabilities that it will be an X-ray we are set to measure, that it will exit the sample, hit the detector, and finally, that it will be detected and counted. Therefore, even with an ideal sample in a perfect instrument, the measured intensity of a given X-ray will vary from measurement to measurement.

X2.1.2 The distribution of an infinite number of measurements will be Gaussian, and the true number of counts will be given by $\sqrt{I°}$, the mean value of those measurements; the standard deviation s, will be given by $\sqrt{I°}$. The Gaussian distribution predicts that 95.4% of the measurements will have values between $I° \pm 2\sqrt{I°}$, and 99.7% of the measurements will have values between $I° \pm 3\sqrt{I°}$. Stated differently, any given measurement will have a better than 95% chance of falling within the values bounded by $I° \pm 2\sqrt{I°}$, and a better than 99% chance of falling within the values bounded by $I° \pm 3\sqrt{I°}$.

X2.1.2.1 Relating this to an actual measurement of an X-ray line, one can state that a peak is present if the counts in the peak exceed the background counts, B, by $3\sqrt{B}$. This can be stated with a high degree of certainty, because there is only a 0.3% chance that the count represents a statistical fluctuation of the background. The 3σ criterion is often used in the definition of the minimum detection limit.

X2.1.2.2 A single measurement of the peak of interest approaches its true value as more counts are accumulated, i.e., as counting time increases. If a given peak measurement, I, is 10000 counts, and it is assumed that $I = I°$, then it can be predicted that more than 99% of subsequent counts will fall between 9700 and 10,300 ($I \pm 3\sqrt{10000}$). The $\pm$ 3% variation expected in this example would likely not have any effect on the accuracy of inclusion classification. However, if the value of I were only 36 counts, for example, the same reasoning would predict that subsequent measurements would vary by up to 50%, and misclassification would be more likely. However,

as discussed in section X2.2 below, even this low number of counts may be sufficient for accurate classification if the classes are distinct enough. Standard text books on X-ray spectroscopy[4] will provide a more detailed discussion of X-ray counting statistics.

X2.2 Stopping Criteria for X-Ray Analysis of Single Inclusion

X2.2.1 The desired result of an SEM-based inclusion analysis is an accurate histogram representing the distribution of inclusions within predefined classes. This measurement is dominated by two competing sources of error: (*1*) the misclassification of individual inclusions due to insufficient EDX statistics (see section X2.1 above) and (*2*) the statistical variation in the number of members of each classification due to insufficient number of inclusion members.

X2.2.2 To be useful, an analysis must be both accurate and timely. Thus there exists the necessity to optimize the time spent analyzing each inclusion and the total number of inclusions analyzed. The character of this optimization depends heavily on the character of the sample. For samples with a small number of equi-populous and distinct inclusion classes, it is possible to get accurate results quickly. Because the classes are distinct, it is not necessary to collect a high number of x-ray events to unambiguously classify the inclusion; because the inclusion classes are of similar population size, the counting statistics in each class accumulate quickly. Alternatively, if the inclusion class of interest is very similar to one or more other classes, it is necessary to collect a larger number of x-ray counts to accurately distinguish the classification. Fortunately, most inclusion classes are distinct enough to be easily distinguished with relatively poor EDX counting statistics.

X2.2.3 If there are only a few members of a given inclusion class, it is necessary to analyze many inclusions to get adequate distribution statistics. In addition, it may also be necessary to collect more EDX counts in each spectrum to ensure that a member of a more populous class is not misclassified in the rarer one. From a percentage error point of view, it becomes significant when a member of a populous class is misclassified in the rarer class.

X2.2.4 There are various mechanisms commonly used for stopping criteria for EDX acquisition. They include: (*1*) stopping after a fixed time (real or live); (*2*) stopping after a fixed total number of counts; (*3*) stopping after a fixed number of counts summed from all the regions-of-interest for the elements-of-interest.

X2.2.4.1 For inclusion analysis, a combination of 3 and 1 is optimal. Some fixed time is selected to act as a failsafe stopping criteria to reject features in the image that will never meet a counting criterion. These features might consist of surface contamination such as dust or lint. Analyses that stop as a result of the time criterion are rejected immediately. Most frequently, however, the EDX acquisition is halted by meeting the fixed counts requirement.

X2.2.4.2 Thus the problem of selecting a stopping criterion comes down to selecting a counting criterion. This number can be arrived at empirically by manually evaluating a number of inclusions in each of the classes of interest. The operator observes how many counts it takes to accurately classify the inclusion. The counts criteria is set to the largest of these values.

X2.2.4.3 Experience has shown that 1500 counts (summed from all the regions of interest) are usually more than sufficient to perform an accurate classification on a steel sample. On an optimized SEM/EDX system, it is possible to collect the 1500 counts from a 2 μm inclusion in 2 seconds or less. Typically, the time rejection criterion would be set approximately 3 times longer, or 6 seconds in this example.

X2.3 Procedure for Empirically Determining the Optimal Counting Criterion

X2.3.1 Analyze a sample N times at each of M different counting criteria (analysis configurations). The analysis with the longest/largest stopping criteria is assumed to be closest to an accurate representation of the sample.

X2.3.2 For each class defined, plot the number of members for each of the N analyses for each of the M analysis configurations. The width of the distribution is taken as a representation of the relative accuracy of the analysis.

X2.3.3 Select an analysis configuration that provides the optimal balance of speed and accuracy.