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Standard Guide for Testing and Certification of Metal, Ore, and Metal-Related Reference Materials[1]

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1. Scope

1.1 This guide describes procedures to be considered for the testing and certification of metal, ore, and metal-related reference materials in the form of blocks, disks, rods, wires, chips, and powders.

1.2 *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]

E32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition

E34 Test Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

E59 Practice for Sampling Steel and Iron for Determination of Chemical Composition[2]

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E178 Practice for Dealing With Outlying Observations

E255 Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition

E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E351 Test Methods for Chemical Analysis of Cast Iron—All Types

E716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

2.2 *ISO Standards:*

ISO Guide 30 Terms and Definitions Used in Connection With Reference Materials[3]

ISO Guide 31 Contents of Certificates of Reference Materials[3]

ISO Guide 33 Uses of Certified Reference Materials[3]

ISO Guide 35 Certification of Reference Materials—General and Statistical Principles[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *certification report*—a document giving detailed information, supplementary to that contained in a certificate, on the preparation of the material and the methods of measurement used in obtaining the certified value(s) for a given reference material. It includes a summary of the results obtained (including a description of all factors affecting accuracy) and a description of the way in which the results were treated statistically.

3.2.2 *certified reference material (CRM)*—reference material accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes

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

its traceability to an accurate realization of the unit in which the property values are expressed; each certified value is accompanied by an uncertainty at a stated level of confidence (from ISO Guide 30).

3.2.3 *certifying body*—a technically competent body (organization or firm, public or private) that issues a reference material certificate which provides the information detailed in ISO Guide 31.

3.2.4 *comparative analytical method*—an analytical procedure that requires the use of CRMs, reference materials (RMs), or, in certain instances, primary chemical standards for calibration. Methods vary widely in the number of such CRMs required and the degree to which such CRMs must match unknown samples.

3.2.5 *definitive analytical method*—an analytical procedure that does not require the use of CRMs, RMs, or primary chemical standards to achieve accurate results. Examples include gravimetry, coulometry, specific titrimetric methods, and isotope dilution mass spectrometry. Each individual laboratory should validate its performance of such methods with CRMs, RMs, or primary chemical standards.

3.2.6 *method of demonstrated accuracy*—a test method for which proof of accuracy has been published, even though it may not fall within the category of a reference method.

3.2.7 *primary chemical standard*—a pure metal or a compound of sufficient high purity to permit its use in the calibration or validation of analytical methods.

3.2.8 *reference material (RM)*—a material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

3.2.9 *reference material certificate*—a document stating one or more property values and their uncertainties and confirming that the necessary procedures have been carried out to establish their validity and traceability. A reference material certificate is an essential attribute of CRM.

3.2.10 *reference method*—a thoroughly investigated method, clearly and exactly describing the necessary conditions and procedures for the measurement of one or more property values that has been shown to have accuracy and precision commensurate with its intended use and can be used to assess the accuracy of other methods for the same measurement, particularly in permitting the characterization of an RM. This includes all national or international standard methods, which may not be classified as definitive methods because they are calibrated against standard solutions of pure chemical substances.

3.2.11 *traceability*—property of a result of a measurement whereby it can be related, with stated uncertainty, to stated references, usually national or international standards, through an unbroken chain of comparisons (ISO Guide 30).

3.2.12 *uncertainty of a certified value*—the range of values within which the "true" value is asserted to lie with a stated confidence.

3.2.13 *validation (of an analytical method)*—evidence that a method yields accurate results on a test sample because it yields accurate results on a CRM of similar composition which was analyzed at the same time.

4. Significance and Use

4.1 This guide describes the suggested procedures for the preparation, testing, and certification of reference materials (RMs) to be used in the calibration, verification, and control of methods used to characterize the chemical composition of metals, ores, and related materials.

4.2 Certified reference materials are frequently rare and valuable commodities requiring investment of considerable cost and production time. They are frequently available for only a limited portion of a user's range of interest.

4.3 When comparative analytical methods are employed, appropriate CRMs are often unavailable for calibration. In this case, the use of RMs is indicated as the alternative choice.

4.4 The use of uncertified homogeneous materials is appropriate for control chart programs where relative data consistency is being monitored. The use of CRMs for such purposes is often a misuse of valuable CRM stocks, especially when uncertified materials of suitable homogeneity are available. For information on the use and misuse of CRMs, see ISO Guide 33 and NBS Special Publication 260-100.[4]

4.5 Use CRMs and RMs with caution in the validation of analytical methods. The danger involves a potential for undetected systematic error, since the same methodology being validated may have been used to establish the values for the CRMs or RMs. For more information on the use of CRMs in the validation of analytical methods, see NIST Special Publication 829.[5]

5. Hazards

5.1 The preparation of reference materials involves hazards associated with the melting, annealing, casting, sampling, forging, rolling, atomizing, pickling, shot blasting, and machining of metal. Similarly, hazards are encountered in crushing, grinding, and sieving particulate and powdered materials.

5.2 For precautions related to the analysis of reference materials, see Practices E50.

6. Justification of Production

6.1 Reference materials are needed to calibrate, verify, and control instrument methods when sufficient certified reference materials of the required composition or form, or both, are not available from certifying bodies.

6.2 Alloy types or grades not available from any certifying body are often needed to match the composition to be tested.

6.3 A study should be made to estimate the cost of RM production and testing. It is important that users remain aware that the preparation of RMs has an associated cost based on factors such as material cost, facility usage charges, personnel labor rates, outside laboratory fees, and so forth, in which the

[4] NIST Special Publication 260-100, *Handbook for SRM Users* (1993 ed.). Available from NIST, U.S. Department of Commerce, Gaithersburg, MD 20899.

[5] NIST Special Publication 829, *Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories*. Available from NIST.

material cost is, in general, the lowest. For complex compositions, the cost of preparing RMs to match the composition of test samples can exceed that of available CRMs. In these cases, the use of CRMs is recommended.

6.4 A study of the costs associated with the RM production should take into account the amount of usable material compared to the total amount produced. It may be necessary to produce twice as much raw material in order to obtain the target amount of usable RM.

7. Types of Reference Materials and Reference Material Forms Covered in This Guide

7.1 *Reference Materials*:

7.1.1 *Multielement Reference Material*— Certified for a complete composition (may or may not include trace element composition).

7.1.1.1 *Grade-Specific Reference Material*— Meets or is close to the compositional specification for all elements of a particular grade of material.

7.1.1.2 *Drift-Correction Reference Material*—A cast or wrought material evaluated for an array of elements, useful for drift correction of instrumental methods. A drift-correction RM may conform to a compositional specification.

7.1.2 *Element-Specific Reference Material*—Certified for a small number of elements. A common type of element-specific RM consists of chips or pins certified for carbon, sulfur, nitrogen, oxygen, or hydrogen, or a combination thereof.

7.2 *Reference Material Forms*:

7.2.1 *Monolithic Solids*:

7.2.1.1 Castings,

7.2.1.2 Wrought material finished to bar form, and

7.2.1.3 Rod and wire material.

7.2.2 *Particulates*:

7.2.2.1 Chips, and

7.2.2.2 Powders.

NOTE 1—In many cases, full composition data, although not necessarily certified, will be needed to permit corrections for interferences in various instrumental methods, especially for critical elements at low concentrations.

8. Specifications for the Finished Reference Material

8.1 If a composition is to be made by a melting process, a realistic approach should be taken when determining the number of elements and their concentrations in each RM. If a composition is to be made by a melting process, a detailed understanding of the metallurgical interactions between the added constituents and the matrix metal is necessary. In most cases, the more elements specified, the greater the difficulty in achieving the specification. Even the creation of a single element RM, such as sodium in an aluminum matrix, may be very difficult to produce.

8.2 The finished composition may be available in semi-finished form, such as an ingot or a larger-than-specified bar or slab form. A study should be made to determine the requirements for processing to the final form.

8.3 The material may be available in finished form, meeting the physical size requirement, within the plant, within the corporation, or from commercial sources.

8.4 Metallurgical condition is an important consideration. Most instrumental techniques such as X-ray fluorescence, spark optical emission, and glow-discharge optical emission use RMs in their solid metallic form. One or more of these methods may be subject to analytical bias due to the sample's metallurgical history. In order to minimize the influence of metallurgical effects, the unknown sample being analyzed should have the same metallurgical structure as the RM being used to calibrate the instrument response. Some instruments may require separate calibrations for cast, chill cast, and wrought materials. For example, in the analysis of iron base metals, X-ray fluorescence is subject to thermal history effects if the heat treatment causes the precipitation of a second phase and affects the homogeneity of the material.

9. Production to Final Form

9.1 This guide will not specify the procedures used for melting or production of the RM into the final form. Methods will vary in accordance with composition requirements.

9.2 Some portions of the candidate material may need to be discarded if homogeneity testing indicates the material is not uniform.

10. Sample Identification and Recordkeeping

10.1 Material identification is required *at all times* during RM production, especially during random sampling for the homogeneity testing. Proper sample identification will ensure that unacceptable portions of a candidate material may be isolated from the usable portion.

10.2 Proper recordkeeping is vital during the entire process of the RM production, from the initial determination of the need to produce an RM, to the preparation of the analysis report.

11. Homogeneity Check

11.1 Estimate the amount of acceptable inhomogeneity prior to the production of the material. The homogeneity testing procedure shall be designed to test at least the minimum sample size or test area required for its intended use.

11.2 Homogeneity testing is a crucial part of RM evaluation. Costs can be held to a minimum if a preliminary homogeneity test, as described in Practice E826, is performed before expensive fabrication and extensive testing is undertaken. It may be necessary to design special test methods to evaluate homogeneity of the bulk material prior to beginning serial production methods such as ingot to bar, billet to bar, and bar to chips.

11.3 Perform the homogeneity test on the candidate material after it is produced to its final form and all physically unacceptable portions (containing visible inclusions, "pipe," scale, and so forth) have been discarded.

11.4 A test for trend inhomogeneity should be done before random inhomogeneity is evaluated. The samples, however randomly selected, must keep traceability to their original location to avoid loss of trend inhomogeneity information. When trend inhomogeneity is detected, appropriate measures should be performed with the candidate material, such as discarding the extreme parts and subdividing the bulk.