



Standard Guide for Preparing Certificates for Reference Materials Relating to Chemical Composition of Metals, Ores, and Related Materials¹

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1. Scope

1.1 This guide describes the minimum information that should be included or referenced in a certificate for a reference material relating to the chemical compositions of metals, ores, and related materials. The order of headings and the wording may be changed to suit the reference materials or their intended applications. It should not be used to disallow the use of reference materials produced prior to the publication of this guide.

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 ISO Documents:

ISO Guide 31—Contents of Certificates of Reference Materials²

ISO 3207, Statistical Interpretation of Data—Determination of Statistical Tolerance Interval²

3. Significance and Use

3.1 This guide describes the essential contents of a certificate prepared to accompany a reference material to be used for the standardization or control of an analytical test method, or both, or the calibration of an instrument used in carrying out analytical testing of metals, ores, and related materials. It is intended for use by suppliers or certified reference materials for the metals industry.

3.2 This guide is based on criteria described in ISO Guide 31.

4. Certificate Headings

4.1 The order of headings and their wording may be changed to suit the reference materials or their intended application.

4.1.1 *Name and Address of the Certifying Organization*—Print the name and address of the body or organization that accepts responsibility for the information on the certificate.

4.1.2 *Title of the Document*—Use a distinct title, such as “Certified Reference Material,” “Certificate of Analysis,” or “Certificate of a Reference Material.”

4.1.3 *Status of the Certificate*—Clearly state the status of the certificate, such as “Provisional” or “Revised.”

4.1.4 *Name of Material*—Name the reference material to identify its type. For example, “Carbon Steel, 0.14 %” is preferable to “Steel” and “Labrador Iron Ore” is preferable to “Iron Ore.”

4.1.5 *Sample Number (and Batch Number)*—Assign a unique number to each reference material, preferably accompanied by the initials of the certifying organization, for example “BCS No. 24A.” Identify renewals by following the number with a letter. Increment the letter upward through the alphabet with each renewal.

4.1.6 *Date of Certification*—Provide the date of issue of the certificate and the dates of all revisions.

4.1.7 *Other Forms or Sizes of Reference Material*—List other forms or sizes of the material named on the certificate. For example, indicate if one or more packaged units are supplied, such as both chips and disks.

4.1.8 *Origin of Reference Material*—Provide the origin of the reference material that may be helpful to the user in interpreting its characteristic, that is, a BOF Heat continuously cast into 4 in. by 4 in. billets.

4.1.9 *Supplier of Reference Material*—If the certifying organization is not the supplier, list the name and address of the organization from whom the reference material can be obtained.

4.1.10 *Preparer of Reference Material*—If the material was prepared by an organization other than that which undertook the testing and certification, identify that organization.

¹ This guide is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.22 on Statistics and Quality Control.

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² Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

4.1.11 *Description of Reference Material*—Provide a general description of the material, including a detailed explanation of the name and the physical character (that is, disks 31 mm in diameter) of the material. Mention toxic or hazardous ingredients in the material with a warning in bold type if warranted. Supply a MSDS when required by law or regulation.

4.1.12 *Intended Use*—Clearly state the purpose for which the material was issued so that it is not misused, for example, “for calibration of instruments for determining the chemical composition of ore samples.”

4.1.13 *Stability, Transportation, and Storage Instructions*—Describe the conditions of transportation, handling, and storage that are most favorable to the maximum lifespan of the reference material and the dependability of its certified values.

4.1.13.1 Whenever possible, state the date after which the reference material is no longer valid. When the stability is *suspect*, but not known *exactly*, a statement such as the following is appropriate: “Tests have shown that, in a normal laboratory atmosphere (absence of direct sunlight and temperatures of 10 to 30°C), the sample is stable up to a year. The purchaser will be notified if tests show that the sample is stable for more than a year.”

4.1.14 *Instructions for the Correct Use of Reference Material*:

4.1.14.1 Provide all necessary instructions for use to avoid conditions which invalidate the certified values, for example, “Dry the sample at 105°C for 2 h before use.”

4.1.14.2 Include blending and subsampling instructions to be followed by the user under this heading, including warnings, such as, “trace-element values might be affected by contamination products from the grinding and blending equipment.”

4.1.14.3 For some reference materials, the instructions are so detailed that they may be issued in an annex to the certificate. In that case, the entry under this heading should refer the reader to an annex of the certificate.

4.1.14.4 The method of measurement is sometimes an inseparable part of the reference material. This applies particularly to empirical measurements of “technological” properties, such as hardness values. In this case the complete details comprising such methods should be given in an annex to the certificate.

4.1.15 *Method of Preparation of Reference Material*—A summary of this information enables the user of the reference material to form a judgment on the amount of planning and care that has been exercised by the producer, and thus, to make some assessment of the quality of the reference material, including its possible state of homogeneity and stability. Details should be provided in the report.

4.1.16 *State of Homogeneity*—Provide a brief description of the tests that were conducted to test the degree of inhomogeneity. Since inhomogeneity may only become apparent in small specimens, state the mass of the specimen used for the homogeneity tests.

4.1.16.1 A test for homogeneity of powdered materials, such as ores and rocks, is particularly sensitive to specimen size since even if the components of a powder are well distributed, each particle may not be representative of the

whole sample. A minimum specimen size shall be such that repeated analyses of a specific size will show no more of significant standard deviation of the analyte than a larger specimen should show.

4.1.16.2 Solid samples may show inhomogeneity. Specimen sizes taken for homogeneity testing and certification analyses may be mandated by the analytical method and must be described adequately so that the user can use the material appropriately. Notation should be made when certain components are found not to be distributed evenly or if certain sections of a sample should be avoided. For materials that are cast from a molten state, the method of chilling should be noted.

4.1.17 *Certified Values*—List the property, its certified value, its confidence limits, and the method of analysis in a table similar to the example given in Table 1. Use footnotes and keys, as needed, to direct the reader to separate sections on the statistical treatment that were used for the calculation of the certified value and its confidence limits.

4.1.18 *Uncertified Values*—Values for constituents of a reference material that do not qualify as certified values must not be included in Table 1. List them in a second table with an appropriate heading, such as “Uncertified Values.”

4.1.19 *Values Obtained by Individual Laboratories or Methods*—When certified values are obtained as a mean or some other estimator of central tendency of the results obtained by more than one method of measurement by the same analyst or by more than one participating laboratory, give the separate results used in the estimation of the certified value in a separate table, together with an identification of the analytical techniques used. Describe any subjective interpretations on the certified value.

4.1.20 *Estimation and Confidence Limits of the Certified Value*—Provide the meaning and nature of the certified value, such as the unweighted mean, the median, or the mode of several measurements. Where the estimate is more complex, give an unambiguous mathematical expression.

4.1.20.1 When subjective considerations have entered into the assignment of the certified value, for example, where several laboratories have used different methods of analysis and the producer has given greater weight to those techniques that he has felt to be more accurate, briefly explain the nature of this reasoning. Refer the user to the report on the reference material where there must be a full description of the mathematical treatment of the measurement values.

4.1.20.2 Describe how the confidence limits (see 4.1.17) were calculated to make their meaning clear. Whenever possible, give the mathematical formulas used to calculate the

TABLE 1 Example of a Data Table

Constituent, Report Formula	Certified Value, % ^A	95 % Confidence Limit (95 % Level)		Test Method ^B
		High	Low	
Aluminum, Al	0.73	0.77	0.69	B
Chromium, Cr	0.94	0.96	0.92	C

^A The meaning of the certified value and the confidence value are given in _____. (Refer to the appropriate section of the certificate.)

^B The keys to the methods of analysis are given in _____. (Refer to the appropriate section of the certificate.)

confidence limits. Avoid ambiguous statements, such as “the total error does not exceed $\pm 8\%$.”

4.1.20.3 In some cases, it is impossible or inappropriate to give rigorously calculated confidence limits, such as when the distribution of the measurement values does not approximate a Gaussian distribution. In these cases, place subjective confidence limits on the certified value, based on knowledge of the measurement techniques used for the certification. State when subjective decisions have been made and, if possible, provide supplementary information, such as a measure of the spread of the values.

4.1.20.4 For further discussion on appropriate statements of confidence of the certified value, see Monograph 148³ and NBS Handbook 91.⁴

4.1.21 *Measurement Techniques Used for the Certification*—Give a short description of each analytical method in the certificate, and include details of the measuring techniques or analytical methods in the report on the reference material.

³ Cali, J. P., et al., U.S. Department of Commerce, National Bureau of Standards, *Monograph 148*, 1975.

⁴ Natrella, M. C., “Experimental Statistics,” U.S. Department of Commerce, *National Bureau of Standards Handbook 91*, 1963.

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4.1.22 *Names of Participating Laboratories*—List the name of laboratories that participated in the certification program on the certificate.

4.1.23 *Legal Notice*—Give legal restrictions in connection with the procurement, transportation, storage, and handling of the material along with an indemnity clause, if needed.

4.1.24 *References*—The certificate must refer to the report on the reference material, which should include a full account of the procedures used for the preparation and certification of the reference material.

4.1.25 *Signature or Name of Certifying Officer*—Include on the certificate the name of at least one officer of the certifying body signifying that this person accepts responsibility for the contents.

4.1.26 *Annex*—Add an annex to the certificate, if needed, to supplement the information on the certificate. Use of an annex will preserve the synoptic character of the certificate.

5. Keywords

5.1 certificate; contents; reference materials