



Designation: E3330 – 22

# Standard Guide for Contents of Documentation and Statistical Treatments for Reference Materials for Metals, Ores, and Related Materials<sup>1</sup>

This standard is issued under the fixed designation E3330; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide is designed to explain and to clarify documentation that accompanies an RM or a certified reference material (CRM). It explains the contents of certificates of analysis for CRMs and product information documents for RMs, based on existing international standards and guides. It briefly touches on the minimum requirements for a label attached to the CRM/RM unit or unit container and to the package containing the unit or unit container.

1.2 This guide provides some basic guidance on calculation of consensus values and uncertainty estimates for CRMs and RMs with examples of approaches commonly used by national metrology institutes and suggestions for sources of information.

1.3 *Units*—The values stated in SI units are to be regarded as the standard, whenever applicable. Values can be traceable to other higher-order reference systems, including Rockwell Hardness, pH, and other systems defined by an international standard or peer-reviewed publication.

1.4 *Contents*—Sections and topics within this guide are enumerated below:

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<sup>1</sup> 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 Laboratory Quality.

Current edition approved April 1, 2022. Published April 2022. DOI: 10.1520/E3330-22.

## Appendix X4 Examples of Language for Sections of a Certificate of Analysis

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:<sup>2</sup>

E18 Test Methods for Rockwell Hardness of Metallic Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E382 Test Method for Determination of Crushing Strength of Iron Ore Pellets and Direct-Reduced Iron

E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method

E2972 Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

### 2.2 ISO/ANSI Standards:<sup>3</sup>

ANSI/NCSL Z540-2-1997 (R2012) American National Standard for Expressing Uncertainty – U.S. Guide to the Expression of Uncertainty in Measurement

ISO Guide 30:2015 Reference Materials – Selected Terms and Definitions

ISO Guide 31:2015(E) Reference Materials – Contents of Certificates, Labels and Accompanying Documentation

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

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

ISO Guide 33:2015 Reference Materials – Good Practice in Using Reference Materials

ISO Guide 35:2017 Reference Materials – Guidance for Characterization and Assessment of Homogeneity and Stability

ISO Standard 17034:2016(E) General Requirements for the Competence of Reference Material Producers

2.3 BIPM Standards:<sup>4</sup>

JCGM 100:2008 Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections)

JCGM 101:2008 Evaluation of Measurement Data – Supplement 1 to the “Guide to the Expression of Uncertainty in Measurement” - Propagation of Distributions Using a Monte Carlo Method

JCGM 200:2012 International Vocabulary of Metrology – Basic and General Concepts and Associated Terms; (VIM), 3rd Edition, 2008 Version with Minor Corrections.

### 3. Terminology

3.1 *Definitions*—For the purposes of this guide, the terms and definitions given in ISO Guide 30, ISO Guide 31, and ISO Guide 35 are normative. Definitions in JCGM 200:2012 are informative.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *censored data, n*—censoring of data is a condition in which the value of a measurement result is only partially known, because the value either (1) is less than a limit of quantification or detection, (2) exceeds the upper limit of observation of the instrument, (3) has been rounded severely by the analyst, or (4) has been intentionally reported as falling within one of a set of predefined intervals.

3.2.1.1 *Discussion*—Case (1) refers to results that are not quantitative and may be reported as less than either the limit of quantification or the limit of detection. Case (2) is exemplified by using a balance with a capacity of 200 g to weigh a mass of 210 g. The result would be reported as > 200 g. More common in analytical chemistry is case (3), the act of censoring in which the laboratory deliberately rounds the result to an extreme in an effort to be conservative with clients. For example, a test method may be able to give quantitative results to 0.001 %, but the lab routinely reports values rounded to 0.01 %. This causes all values between 0.005 % and 0.014 % to be given the same value of 0.01 %. The true variance of the measured property is obscured. Case (4) is nearly the same as case (3), except the method of reporting is predefined as placing results into a set of categories, such as 1 to 10, 10 to 20, 20 to 30, etc. The problem of censored data, in which the observed value of some variable is partially known, is related to the problem of missing data, where the observed value of some variable is unknown. Censoring should not be confused with the related idea of truncation. With censoring, observations result either in knowing the exact value that applies, or in knowing that the value lies within an interval. With truncation, observations never result in values outside a given range. Values outside the range

are never seen or never recorded. Truncation is not the same as rounding (Appendix X3, X3.3). Types of censoring important to RM development include the following:

*Left censoring*—a result is below a certain value, but it is unknown by how much.

*Right censoring*—a result is above a certain value, but it is unknown by how much.

*Interval censoring*—a result is somewhere in an interval between two values, which includes both a set of intervals predefined prior to measurement and cases of rounding after measurement.

3.2.2 *non-operationally defined measurand, n*—measurand defined independently of any specific measurement procedure.

3.2.3 *operationally defined measurand, n*—measurand that is defined by reference to a documented and widely accepted measurement procedure to which only results obtained by the same procedure can be compared. (See ISO 17034:2016(E), p. 3.)

### 4. Summary of Guide

4.1 This guide discusses steps for implementing a procedure to document and label RMs and CRMs. Specifications for RM product information sheets, CRM certificates of analysis and labels for unit containers discussed in this guide include those mentioned in technical clauses of ISO Guide 30, Guide 31, and Guide 33.

4.2 This guide includes guidance and suggestions for processes for critical evaluation of data created in development of RMs and in calculations of consensus values and uncertainty estimates and their presentation in certificates of analysis for CRMs and product information documents for RMs.

### 5. Significance and Use

5.1 This guide is intended for use by developers of RMs and CRMs for the metals and mining industries.

5.2 The guidance is related to uniform procedures and requirements and is intended to prevent the proliferation of widely varying documentation practices, definitions, and terminology. Where the statements in this guide are made as imperatives, it is because the stated practices are fundamental to chemical metrology, not to CRM/RM development.

5.3 The material in this guide is intended to supplement and to clarify the contents of ISO Guide 31 and to provide guidance specific to the needs of the metals and mining industries.

5.4 The documents described in this guide are intended to contain the minimum amount of information required for a user to understand the material, to help a user judge the quality of the product, and to help a user employ it in appropriate ways. Neither this guide nor resultant documents are meant to be encyclopedic.

5.5 Because this document is a standard guide, it is intended to educate those who are involved in laboratory operation, quality system development and maintenance, reference material development, and accreditation of laboratory operations within the scope of a quality system. It must be understood by all parties that the elements of this guide discuss optional

<sup>4</sup> Available from International Bureau of Weights and Measures (BIPM), <https://www.bipm.org>.