Designation: E 2165 – 01

# Standard Practice for Establishing an Uncertainty Budget for the Chemical Analysis of Metals, Ores, and Related Materials[1]

This standard is issued under the fixed designation E 2165; 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 practice describes a model for establishing ISO 17025-compliant uncertainty budgets for the chemical analysis of metals, ores, and related materials. It is based on applying the Horwitz[2] function to widely accepted, diverse interlaboratory test programs, such as interlaboratory testing of standard test methods and proficiency testing programs. This function expresses the interlaboratory standard deviations that can be expected for any concentration level as competent laboratories use optimized test procedures to analyze any matrix for any analyte. It may be used to set aim uncertainties against which to plan new standard test methods and to assess the performance of existing test methods.

1.2 An optimized test procedure is one in which the final test results are at least equivalent to alternative, state-of-the-art procedures. In the analytical chemistry community, this means that calibrations are carried out, verified, and controlled such that the final test results have no systematic, detectable bias. The elimination of sources of bias is a key responsibility of any person who designs analytical test methods. Hence, an analytical test method that contains systematic, measurable sources of bias would probably not be accepted as an ASTM test method and its performance data would probably not be in compliance with the procedures described in this practice.

1.3 The uncertainty budget model described in this practice is based on the assumption that, in a normally distributed, bias-free environment, measurement uncertainty will improve by the square root of two with each removal of a significant source of variation. Conversely, it is assumed that measurement uncertainty will worsen by the same amount with each addition of a significant source of variation. Furthermore, this model assumes that the hierarchy of increasing variation in any composition-based measurement system begins with calibration and progresses through control to intralaboratory standard deviation to interlaboratory standard deviation to product sampling for conformity assessment. Therefore, aim values for the expected uncertainties at any process step can be predicted using this model.

1.4 When using this model, the aim values generated using this model must then be validated, verified, and documented as part of the development and interlaboratory testing of any new test method, sampling practice, and product specification, as appropriate. It is also expected that each laboratory that elects to use that standard test method will generate data to show that the standard test method complies with the published uncertainties developed during interlaboratory testing of the standard test method. The principles in this practice can also be applied to the development of test methods used to determine the composition of other materials.

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:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores and Related Materials[3]

E 1282 Guide for Specifying the Chemical Compositions and Selecting Sampling Practices and Quantitative Analysis Methods for Metals, Ores, and Related Materials[3]

E 1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis[3]

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[3]

E 2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials[4]

E 2053 Guide for Planning, Carrying Out, and Reporting Traceable Chemical Analyses of Metals, Ores, and Related Materials[4]

E 2093 Guide for Optimizing, Controlling and Reporting Test Method Uncertainties from Multiple Workstations in the Same Laboratory Organization[4]

2.2 *ISO Standards:*

[1] This practice 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] Horwitz, W., *Analytical Chemistry*, Vol 54, pp. 67A-76A, 1982.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

ISO 17025 (1999) General Requirements for the Competence of Calibration and Testing Laboratories[5]

ISO 9000: 2000 Quality Management and Quality System Elements[5]

ISO/TS 16949 (2002) Quality Systems—Automotive Suppliers—Particular Requirements for the Application of ISO 9001:1994[5]

ISO TC/17 SC 1 Steel—Methods and Determination of Chemical Composition[5]

2.3 *Other Document:*

QS9000, 3rd Edition Quality System Requirements, Chrysler Corporation, Ford Motor Company, and General Motors Corporation[6]

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology E 135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aim calibration uncertainty*—the maximum deviation (95 % confidence) to be allowed between an assumed true value and the measured value during the design of the calibration segment of an analytical test method, based on an aim uncertainty budget. In order to ensure that the calibration function does not contribute distinguishable bias to the report value, all individual calibration deviations shall be randomly distributed above and below the assumed true value. It is the method-developer's responsibility to develop and test appropriate protocols for detecting and controlling calibration bias consistent with the intended purpose of the test method and the measuring technology being utilized.

3.2.2 *aim control uncertainty*—the maximum deviation (95 % confidence) to be allowed in the design of the control part of an analytical test method, based on an aim uncertainty budget and including variation due to calibration. Since most control charts are created with three sigma control limits, users must design and control measurement processes to be effective at the 95 % confidence level. To help meet this requirement, it is recommended that control charts used in this model be interpreted using the Westgard rules in accordance with Practice E 1329.

3.2.3 *aim total intralaboratory uncertainty*—the maximum deviation (95 % confidence) to be allowed in the design of the total intralaboratory uncertainty of a test method, beginning with the preparation of a homogeneous sample and ending with a final report value to the client.

3.2.4 *aim total interlaboratory uncertainty*—the maximum deviation (95 % confidence) to be allowed in interlaboratory studies of a test method, based on multipurpose interlaboratory studies of the type carried out in proficiency tests and national and international standard test development studies.

3.2.5 *aim lot uncertainty*—the maximum deviation (95 % confidence) to be allowed when optimized, standardized sampling practices are used to take samples from a specified lot of material and the samples are distributed among several competent laboratories for testing.

3.2.6 *uncertainty budget*—the allocation of total measurement uncertainty among specific components of a measurement process that contribute significantly to the overall deviation.

## 4. Significance and Use

4.1 Knowing and controlling the uncertainty of measurements are important to laboratories as they comply with internal and external needs. For example:

4.1.1 There is a need to know when calibration curves drift so that corrections can be made before time is wasted generating faulty data and, more importantly, to prevent reporting of faulty data. The control of laboratory performance against internally established criteria is usually met with good statistical control programs, such as described in Practice E 1329.

4.1.2 There is a need to demonstrate state-of-the-art performance to customers and accreditation bodies, especially those accrediting laboratories to ISO 17025. One widely accepted way to demonstrate compliance is to participate in proficiency test programs, such as described in Practice E 2027, as available.

4.1.3 There is a need for laboratory management personnel to know, in advance, how tightly to control existing processes, how to set data quality expectations for new work, and how to build uncertainty statements and budgets to comply with ISO 17025. This practice gives one approach for meeting those needs.

4.1.4 There is a need for users of test results to understand the origin of measurement uncertainties and how to apply them in using data for process control or product conformity decisions in order to comply with ISO 9000, ISO/TS 16949, and QS 9000. This practice gives a relatively simple model for use in developing strategies to meet those needs, utilizing the information available from the analytical testing laboratory.

4.2 ISO 17025 accepts laboratory compliance with uncertainty budgets in standard test methods, provided that all of the significant sources of variation are identified and quantified in the standard test method. This practice offers a consensus-based approach to meeting that need, based on the most widely available sources of comparative data available, namely interlaboratory standard deviations.

4.3 Building the model used in this practice on the available interlaboratory standard deviations is convenient because they are in the "middle" of the steps between calibration and final data usage and are at the interface between the laboratories and their clients. Hence, any inaccuracies in the model, either in the laboratory or in the user environment, will be correctable within either community without disturbing the foundation of the model.

4.4 Having allowed for the fact that this model is based on probabilities at the 95 % confidence level, any task group that considers promulgating a standard test method, practice, or specification that exceeds the boundaries set by this practice should seek opportunities to improve the procedure or be prepared to accept uncertainties that exceed normally accepted levels.

4.5 This model is based on 95 % confidence intervals (two

[5] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

[6] Available from Automotive Industry Action Group (AIAG), 26200 Lahser Rd., Southfield, MI 48034.