



Designation: E2437 – 05

Standard Practice for Designing and Validating Performance-Based Test Methods for the Analysis of Metals, Ores, and Related Materials¹

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1. Scope

1.1 This practice covers a model for designing and validating performance-based, ISO 17025-compliant, standard test methods for the instrumental chemical analysis of metals, ores, and related materials. The principles in this practice can also be applied to the development of test methods used to determine the composition of other materials.

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

- E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- E1621 Guide for X-Ray Emission Spectrometric Analysis
- E2027 Practice for Conducting Proficiency Tests in the Chemical Analysis of Metals, Ores, and Related Materials
- E2165 Practice for Establishing an Uncertainty Budget for the Chemical Analysis of Metals, Ores, and Related Materials (Withdrawn 2007)³

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 Laboratory Quality.

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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 The last approved version of this historical standard is referenced on www.astm.org.

2.2 ISO Standards⁴

- ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories
- ISO Guide 31 Reference Materials—Contents of Certificates and Labels
- ISO Guide 32 Calibration in Analytical Chemistry and Use of Certified Reference Materials
- ISO Guide 34 General Requirements for the Competence of Reference Material Producers

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.2 *aim uncertainty budget, n*—during the development of a standard performance-based test method, the target allocation of interlaboratory measurement uncertainty among specific components of a measurement process that contribute significantly to the overall deviation.

3.2.2.1 *Discussion*—The target allocation is made by the task group as described in Practice E2165 and serves as guidance for interlaboratory test participants during method testing.

3.2.3 *interlaboratory uncertainty, n*—in a performance based standard test method, the precision (95 % confidence) that participating laboratories achieved during interlaboratory studies, beginning with the preparation of a homogeneous sample and ending with a final report value to the client.

3.2.4 *intralaboratory uncertainty, n*—in a performance based standard test method, the precision (95 % confidence) that a laboratory achieves when the method is used by more than one operator. In test methods that establish maximum allowable intralaboratory uncertainties, users must be able to

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

demonstrate compliance with those uncertainties in order to report that a given test result was produced using the named method.

3.2.5 performance based method, *n*—a test method that defines (1) the general approaches for sampling, sample preparation, and making measurements on a specified type of material, and (2) defines maximum allowable uncertainties for each measured constituent over its validated concentration range.

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

4. Significance and Use

4.1 This practice provides guidance for methods-writing task group leaders and members in planning, drafting, and testing performance-based test methods. It also guides users of performance-based methods in interpreting and applying them properly.

4.2 Standard test methods, particularly those that cover the instrumental analysis of commercial materials by specific measurement techniques, such as point-to-plane atomic emission, x-ray fluorescence, inductively-coupled plasma, and atomic absorption spectroscopy, need sufficient flexibility to accommodate the various applicable makes and models of instrumentation. At the same time, these standard test methods must be capable of generating commercially-useful results that are sufficiently accurate and precise. These needs can be met by the use of performance-based test methods that rely more on the demonstrated quality of the test results than on strict adherence to specific procedural steps. One model for developing these methods is described in this practice.

4.3 It is expected that laboratories using performance-based standard test methods developed in accordance with this practice will prepare their own detailed work instructions. These work instructions will include detailed operating instructions for the specific laboratory, the specific reference materials (RMs) employed, and performance acceptance criteria to be applied in that laboratory. It is also expected that those laboratories will document their own performance data using their work instruction to show that their data are consistent with the standard test method's Precision and Bias statement. Over time, it is also expected that, when applicable, an individual laboratory's proficiency test results will also be consistent with its documented performance.

4.4 Traditional ASTM Precision and Bias statements, developed during interlaboratory testing in accordance with Practices E691 and E1601, provide information on the performance achieved by participating laboratories. Intralaboratory precision can be estimated by dividing the Repeatability index, *r*, in the Precision and Bias Table by the square root of 2, at any tested concentration. Proficiency test programs, such as those following Practice E2027, periodically provide interlaboratory performance data from larger numbers of laboratories, using a variety of test methods, at many different concentrations over an extended time period. Since interlaboratory precision ob-

tained during both method development and proficiency testing using existing methods is a function of concentration, per Practice E2165, it follows that the interlaboratory testing of a new test method should perform no worse than its predecessors. Practice E2165 describes how historical interlaboratory test results can be used to set aim data quality objectives for new test methods. This practice incorporates those aim data quality objectives in developing uncertainty budgets used in the design, development, and testing of performance-based methods. This approach ensures that interlaboratory test data included in Precision and Bias statements are consistent with that associated with the other standard test methods.

5. Summary of Practice

5.1 The remaining parts of this practice provide instructions for planning a performance-based method development project, conducting the necessary validation and interlaboratory precision tests, and documenting the results.

5.2 The organization of the rest of the practice guides the task group through the following steps:

5.2.1 Section 6—Sets overall objectives for the project, detailed only to the extent needed to convince the task group that the project can be accomplished as planned.

5.2.2 Section 7—Sets the technical parameters to be incorporated in the new test method. Establishes details needed to facilitate organizing and drafting a test method.

5.2.3 Section 8—Addresses writing the first draft of the test method.

5.2.4 Section 9—Describes how to verify the draft method before entering into more extensive interlaboratory testing.

5.2.5 Section 10—Discusses writing the interlaboratory protocol plan so that all of the needed data is available in a form that is easily processed.

5.2.6 Section 11—Describes task group work in conducting the interlaboratory test.

5.2.7 Section 12—Describes performance-based method information to be included in the Research Report.

5.2.8 Section 13—Addresses finalizing the text of the test method after interlaboratory testing is satisfactorily completed.

6. Setting Objectives

6.1 For the material to be tested, identify and list applicable national and international product specifications, sampling practices, and standard test methods. Be sure to reference these documents appropriately in the new test method.

6.2 List the elements and concentration ranges to be included in the materials to be analyzed using the method and list the subset of those elements and concentration ranges to be incorporated as analytes in the new test method.

6.3 Determine how measurement uncertainty and data quality objectives will be handled.

6.3.1 To comply with ASTM requirements, plan to prepare a precision and bias statement. To comply with ISO 17025 requirements, plan to identify all significant sources of measurement uncertainty and to calculate the intralaboratory uncertainty (3.2.3) from the information in the precision and bias statement.

6.3.2 To help laboratories use the new performance-based test method more effectively, the task group should consider developing an uncertainty budget (3.2.5). That budget may be developed as suggested in this practice, or in a different way, provided that the minimum requirements in 6.3.1 are met.

6.3.3 If the task group elects to develop an uncertainty budget as described in this practice, it should attempt to establish each component of its aim uncertainty budget as defined in Practice E2165. In some cases, the method of choice may not be optimum for each analyte to be covered. In those cases, if the anticipated performance meets applicable specification requirements, the task group should set its data quality objectives in a way that is compliant with specifications (commercial needs) and attainable performance. The components of uncertainty will be useful in planning and developing the performance-based test method.

7. Setting Technical Parameters

7.1 *Select the Approach to Make the Measurements:*

7.1.1 Select a measurement technique that represents an optimum choice based on: (1) wide acceptance and use throughout the industry, (2) anticipated ability to measure all elements and concentration ranges in conformance with the aim uncertainty budget, (3) anticipated sustained availability of Certified Reference Materials (CRMs) for achieving traceable calibrations (Note 1) of all elements and concentration ranges, and (4) anticipated sustained availability of homogeneous materials needed for statistical control of the method.

NOTE 1—This practice assumes the use of a Type III calibration as defined in ISO Guide 32. Types I and II do not require the use of CRMs.

7.1.2 Select the sampling equipment and procedure. Resolve any questions about the adequacy of the sampling process. Ensure its acceptability to users. If a new or revised sampling practice is needed, arrange for the development of that practice and establish acceptability with those who are responsible for performing the sampling.

7.1.3 Select a sample preparation technique that is compatible with the selected sampling and measurement technique and current industry practices. If a new or revised sample preparation practice is needed, arrange for the development of that practice as part of the new test method.

7.2 *Design the Measurement Approach:*

7.2.1 *Calibration Approach*—Based on the measurement technique selected in 7.1.1, determine if calibration is to be achieved by matrix-matched CRMs or by laboratory-prepared RMs that are traceable to CRMs. Matrix-matched CRMs are preferred, when available, and are most frequently used when solid samples are analyzed. RMs that are traceable to CRMs are often used in test methods calibrated with solutions. In these cases, materials of known purity are added to CRM solutions in order to match the overall composition of sample solutions. Refer to ISO Guide 32 for recommended approaches to calibration using Certified Reference Materials.

7.2.2 *Data Quality Objectives*—Set the aim intralaboratory uncertainty (3.2.2) to be achieved in the new test method. Complete the aim uncertainty budget for all elements and concentration ranges in accordance with the decisions made in Section 6. Refer to X1.2 and Practice E2165 for one approach.

7.2.3 *Calibration and Calibrants:*

7.2.3.1 For each element and concentration range, ensure the availability of sufficient numbers of acceptable calibrants. Sufficient numbers of calibrants must be available to define the shape of each calibration curve over the concentration range of interest and to allow compensation for interelement effects. Refer to Practice E305 or Guide E1621 for additional background information. An acceptable calibrant is a CRM that is compliant with ISO Guide 34, or an RM that is traceable to a CRM or the appropriate SI unit, and has an estimated uncertainty small enough to make it possible to meet the aim data quality objectives of the method. Some methods may be calibrated directly with CRMs used in the as-received condition. Others may require the dissolution of chip or solid CRMs or the use of CRM spectrometric solutions that must be combined with other reagents or materials in order to provide suitable calibrants. In cases where CRMs are combined with other reagents to make calibrants, the standard test method must provide strict specifications on those reagents to ensure that the calibrants' assigned quantity values are accurate and have correct uncertainty estimates.

7.2.3.2 Set aim uncertainty limits for all calibration curves and calibrants. See X1.2.2.2 and X1.2.2.3 for a suggested approach.

7.2.4 *Verifiers*—Ensure the availability of traceable reference materials that comply with the requirements of 7.2.3, but are reserved for use to independently verify the calibration function.

7.2.5 *Quality Control*—For each element and concentration range to be determined, ensure the availability of material that can be used for control in accordance with ISO 17025, clause 5.9. For planning purposes, consider the availability of at least two concentrations for each calibration range, one at about 20 % and one at about 80 % of the maximum values. Availability means that a typical laboratory can acquire the needed materials in a reasonable way, either commercially or in-house. Establish aim uncertainty limits for each case. See X1.2.2.1 for a suggested approach.

8. Drafting the Method

8.1 Draft the standard test method in accordance with the ASTM Form and Style Manual, Part A, making sure to include the following information which is essential to a performance-based method. Except for the intralaboratory performance requirement, the draft will include estimated uncertainty information consistent with the decisions made in Section 6.

8.2 *Significance and Use*—Emphasize that this is a performance-based method and that each user is expected to create specific work instructions that describe how the performance-based method is applied in that laboratory. Also, emphasize that the user laboratory is expected to have performance data taken in accordance with its work instructions to demonstrate that it meets the minimum data quality objectives specified in the standard test method.

8.3 *Interferences*—In a performance-based method, it is not practical to identify all possible interferences that may cause bias in the test results. In drafting this section of the test method, the task group may identify common interferences and