Designation: E 2438 – 05

Standard Practice for Implementing Standard Performance Based Test Methods for the Analysis of Metals, Ores, and Related Materials[1]

This standard is issued under the fixed designation E 2438; 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 use by analytical chemistry laboratories in implementing standard performance-based test methods, such as those developed in accordance with Practice E 2437.

1.2 The principles in these practices can also be used by ISO 17025 compliant laboratories that need to implement other performance-based test methods or need to document and validate extensions of standard test methods, or non-standard test methods.

1.3 *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]

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

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

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

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

E 2093 Guide for Optimizing, Controlling and Reporting Test method uncertainties from Multiple Workstations in the Same Laboratory Organization

E 2165 Practice for Establishing an Uncertainty Budget for the Chemical Analysis of Metals, Ores, and Related Materials

E 2437 Practice for Designing and Validating Performance Based Test Methods for the analysis of Metals, Ores, and Related Materials

2.2 *ISO Standards*[3]

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ISO Guide 32 Calibration in Analytical Chemistry and Use of Certified Reference Materials

ISO 5725 Precision of Test Results – Determination of repeatability and reproducibility by interlaboratory tests

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aim total intralaboratory uncertainty*, *n*—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.

3.2.2 *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.

3.2.3 *total 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.

3.2.3.1 *Discussion*—In test methods that establish maximum allowable intralaboratory uncertainties, users must be able to demonstrate compliance with those uncertainties in order to report that a given test result was produced using the named method.

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

[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.

Current edition approved May 1, 2005. Published July 2005.

[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 (www.ansi.org).

4. Significance and Use

4.1 This practice is aimed at guiding analytical chemistry laboratories through the implementation of ASTM standard performance-based test methods in a manner that complies with ISO 17025. The principles incorporated in this practice can also be applied in laboratories that wish to extend the application of standard test methods beyond their published scopes or to validate and document non-standard methods.

4.2 The major responsibilities described in this practice include (1) writing an in-house standard operating procedure that describes how the laboratory carries out the test method, (2) developing and documenting data to demonstrate that the laboratory can meet the data quality requirements specified in the standard test method, and, (3) monitoring the quality control and proficiency test data, where available, over time, to ensure sustained data quality performance.

4.3 Some performance-based standard methods specify only intralaboratory precision, while others may include either full or partial uncertainty budgets, with required performance levels at each step in the procedure. Users of the standard performance-based methods are expected to comply with all performance requirements in each method.

4.4 Proficiency test programs, such as those carried out under Practice E 2027, provide interlaboratory performance data from large numbers of laboratories, using a variety of test methods, at many different concentrations over an extended time period. Methods-development interlaboratory test programs, such as those carried out under ISO 5725 and Practices E 691 and E 1601, also give very similar interlaboratory precision data, even though they have very different objectives and test protocols. Since interlaboratory precision obtained during both method development and proficiency testing of existing methods is a function of concentration, per Practice E 2165, it follows that a new test method should perform at least as good as its predecessors.

4.5 Practice E 2165 describes how historical interlaboratory test results can be used to set aim data quality objectives for new test methods. Practice E 2437 describes how to apply those aim data quality objectives in the development and validation testing of performance-based methods. This approach ensures that methods standardized under Practice E 2437 will be capable of providing test results comparable in quality to other standard test methods. It is also true that, if a user laboratory meets the data quality objectives specified in the standard method, it should expect to achieve satisfactory results in future proficiency tests.

5. Develop In-House Standard Operating Procedure (SOP).

5.1 This section identifies key information to be included in the in-house SOP that describes the implementation of an ASTM standard performance-based test method. In order to comply with the standard test method, the instructions in the SOP or referenced documents shall include all documentation required by the standard. In order for the laboratory to report test results based on the standard test method in accordance with ISO 17025, it shall not violate any of the requirements in the standard test method. It may elect to determine fewer analytes specified in the method and/or to calibrate its method over more-limited concentration ranges specified in the test method. However, if the laboratory elects to extend determined elements or concentration ranges, or both, not included in the standard method, it must validate those extensions and report a statement of variation in any test reports that refer to the standard test method. The form and style of the SOP is not prescribed; each laboratory is encouraged to incorporate the required documentation within its existing structure.

5.2 *Scope*—List the elements and concentration ranges, or grade specifications, to be included in the laboratory's implementation of the standard. Also, list the elements and concentration ranges to be determined by the laboratory using this method. If a laboratory includes more than one similar workstation in an SOP, it may coordinate them as described in Practice E 2093, provided that all data quality objectives described in the standard test method are complied with. Be sure to reference the standard test method under implementation.

5.3 *Apparatus*—List all pieces of equipment used in the testing that might influence the quality of test results. Make sure that items on the list are uniquely identified and traceable to other related documentation such as maintenance records. If multiple workstations are used, such as described in Practice E 2093, list each workstation separately with its apparatus.

5.4 *Reagents*—Give specific instructions for preparing all reagents. Include the identity and purity specifications of starting materials. Calculate and record all analyte concentrations for each reagent and estimate the uncertainty of those values. Make sure that the analyte concentrations and uncertainties are consistent with the performance requirements of the standard method.

5.5 *Calibrants and Verifiers*—List, or refer to a related document, all CRMs, RMs and other materials used for calibration and verification. Refer to the Certificates and related documentation that describe the materials, their composition, uncertainties, homogeneity, and traceability, as required by ISO 17025. Specify the number of calibrants, the analyte concentration of each, and the uncertainty of each value. If calibrants and verifiers are prepared in-house, include the preparation procedure or a reference to that information. For calibrants and verifiers, be sure to calculate and record the analyte concentrations and associated uncertainties. If calibrants or verifiers, or both, are prepared from CRMs combined with other chemicals, such as pure metals and dissolving acids, make sure that the total analyte content, with uncertainty, is calculated for each material.

5.6 *Restandardization and Control Materials*—If the method requires that standardization and control materials be procured or prepared, or both, , give complete instructions for preparing them and ensuring their homogeneity. Note that exact analyte concentrations, with estimated uncertainties, are not required for these materials, but that some procedure must be in place to detect any change in concentration of analytes in those materials.

5.7 *Sample Preparation*—Starting with a description of the material as received by the laboratory, write specific instructions on how to prepare it for analysis. Include instructions for

subdividing the sample, if required, to perform various specified tests that ensures that all test results represent the submitted material. If the laboratory uses the test method on materials from different sources, be sure that to document a procedure for each.

5.8 *Calibration*:

5.8.1 Give specific instructions on how to perform the calibration function. Be sure to include statistically important instructions such as the sequence of taking measurements and the number of readings to be taken on each calibrant, and the criteria of acceptability for each calibration data point. Information on various calibration approaches that use CRMs is given in ISO Guide 32.

5.8.2 Give specific instructions on the type of calibration records to be retained and how and where to store them. If the calibration function and curve fitting are carried out under computer control, make sure that the version of the software is recorded, and that the calibration records are securely maintained.

5.9 *Restandardization*:

5.9.1 Give specific instructions on the conditions under which the instrument is to be restandardized, including such statistically and performance sensitive details as when to restandardize, how many exposures of the restandardization materials to be taken, and their criteria of acceptance.

5.9.2 Give instructions regarding the record creation and retention of restandardization information.

5.10 *Control:*

5.10.1 Give specific instructions relating to the use of control materials to monitor and control the performance of the test method. Include such statistically and performance sensitive details as when to analyze the samples, and their criteria of acceptance.

5.10.2 The control protocols may include the use of statistical process control techniques applied to test materials of known homogeneity analyzed as unknowns and evaluated in accordance with Practice E 1329.

5.11 *Procedure*:

5.11.1 Give specific procedures for converting the prepared sample into a form suitable for measurement.

5.11.2 Specify how to order the calibrants, verifiers, and controls, as appropriate, along with the samples to be analyzed.

5.11.3 Give instructions for carrying the above materials through the analysis process.

5.11.4 Give instructions for evaluating the acceptability of controls (5.10) and repeat measurements on samples, as appropriate.

5.11.5 Give instructions for releasing the analysis results to the next step in the laboratory.

6. Develop and Record Performance Data.

6.1 Following the draft in-house procedure and the primary ASTM test method, carry out each of the steps and document the results along with their evaluated uncertainties. Maintain and update these records as needed throughout the lifetime of the test method in the laboratory and after it is discontinued, for the time specified in the laboratory's quality system. Participate in a recognized proficiency test program, when available. If no proficiency test program is available, attempt to organize an informal interlaboratory test program among similar laboratories. Guidance for creating such a program can be found in Practice E 2027.

7. Keywords

7.1 analytical chemistry; laboratory standard operating procedures; performance based test methods