Designation: E 2410 – 04

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

This standard is issued under the fixed designation E 2410; 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 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 requirements 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 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves

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 1621 Guide for X-Ray Emission Spectrometric Analysis

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

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

2.2 *ISO Standards:*

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories[3]

ISO Guide 31 Reference Materials—Contents of Certificates and Labels[3]

ISO Guide 32 Calibration in Analytical Chemistry and Use of Certified Reference Materials[3]

ISO Guide 34 General Requirements for the Competence of Reference Material Producers[3]

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 *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.

3.2.2 *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.

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

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

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

3.2.6 *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. The target allocation is made by the task group as described in Practice E 2165 and serves as guidance for interlaboratory test participants during method testing.

4. Summary of Practice

4.1 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.

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

4.2.1 *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.

4.2.2 *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.

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

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

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

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

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

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

5. Significance and Use

5.1 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.

5.2 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 spectrometry, need sufficient flexibility to accommodate the various applicable makes and models of instrumentation. Also, 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.

5.3 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.

5.4 Traditional ASTM Precision and Bias statements, developed during interlaboratory testing in accordance with Practices E 691 and E 1601, 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 E 2027, 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 obtained during both method development and proficiency testing using existing methods is a function of concentration, per Practice E 2165, it follows that the interlaboratory testing of a new test method should perform no worse than its predecessors. Practice E 2165 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.

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 (3.2.4) 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 E 2165. 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 (3.2.4) 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 E 2165 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 E 305 or Guide E 1621 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 advise laboratories to take steps to avoid them. Likewise, this section shall include a statement to the effect that the user laboratory is responsible for ensuring the absence of, or correcting for, interferences that may bias test results generated while following its specific work instructions.

8.4 *Apparatus*—Identify the generic types of equipment covered in the test method. For example, "sequential and simultaneous X-ray fluorescence spectrometers capable of covering the elements and concentration ranges included in laboratory's documentation and capable of meeting the performance requirements specified in the method." Also, include the functionality required of sampling and sample preparation equipment, as appropriate, taking care not to specify more prescriptively than necessary to remain within the scope of the method.

8.5 *Reference Materials, Reagents, and Related Materials*

8.5.1 *Calibrants*:

8.5.1.1 List the requirements for calibrants and give instructions for selecting them. When defining the requirements for calibrants, refer to the decisions made in 7.2, while trying to avoid, if possible, specifying the use of specific CRMs.

8.5.1.2 For a comparative method to be calibrated using CRMs, identify the qualities needed in the calibrants, such as: "Select sufficient numbers of certified reference materials that are supplied by National Metrology Institutes or are certified to comply with ISO Guides 31 and 34. Cover all elements and concentration ranges, and allow for the correction of interferences, as needed". The standard test method should not specify which specific CRMs to use because the test method will become invalid when those specific CRMs no longer exist. Similarly, new CRMs that become available after the method is published would not be included. If, for some reason the use of a specific CRM is unavoidable, include language that allows the use of its replacement.

8.5.1.3 For a method to be calibrated using solutions or pure chemicals, describe how to obtain or prepare calibrants that meet the traceability requirements without creating bias by the addition or loss of analyte concentration.

8.5.1.4 If the task group elects to develop a full uncertainty budget in accordance with Appendix X1, or an alternative approach, refer to the maximum aim uncertainty to be allowed for the calibrants as a function of concentration.

8.5.2 *Verifiers*—Specify the requirements for verifiers that meet the requirements of 8.5.1 but specify that they not be used as calibrants. Require that verifiers be analyzed as unknowns immediately after the calibration is complete. Since verifiers are used to confirm the accuracy of the calibration process, the limits of uncertainty for the verifiers and the verification process are the same as for the calibrants and the calibration process.

8.5.3 *Standardization Materials*—If the method requires standardization (drift correction), give requirements for the selection and management of the materials. Consider the effect of standardization on the final report value and establish criteria that are consistent with the acceptable intralaboratory precision. It is not necessary to establish specific limits of acceptability for the standardization procedure because these sources of variation are included in the control process.

8.5.4 *Quality Control*—Specify the requirements for establishing the number and concentration of control samples, as needed. Concentrations need not be certified, but homogeneity should be established and should be about the same level as the calibrants. If the use of control charts is to be specified, refer to Practice E 1329.

8.5.5 *Reagents*:

8.5.5.1 *Water*—If water is used in the test method and the purity of the water might influence the quality of the test results, specify the purity requirements. Pay particular attention to setting analyte concentrations that, if exceeded, and not corrected for, could cause bias in the test results.

8.5.5.2 *Chemicals*—If specific chemicals, liquids, solids, or gases, are to be used in the test method, list them and their required purities. If the laboratory has the option to select its own chemicals, give generally applicable specifications. If no chemicals are to be used in the test method, skip this section.

8.6 *Procedure*:

8.6.1 Sampling of lots of materials, whether conducted by laboratory-supervised personnel or not, is usually considered outside the scope of an ASTM test method. However, for some materials, laboratories may be required to subdivide the as-received sample in order to perform the required tests. Such sub-divisions should be considered and described in the test method.

8.6.2 Indicate the major steps that must be accomplished to generate a report value. Refer to manufacturer's instructions and individual user's work instructions for details.

8.6.3 Discuss and explain the criteria of acceptance that must be met if a report value is to be supplied in association with the Standard Test Method. See X1.2.1 for a suggested approach. State the intralaboratory data quality objectives plus any measurement uncertainty requirements that must be met and documented by the laboratory, whether reported to the client or not.

8.7 *Precision and Bias*:

8.7.1 Write this section after completion of the interlaboratory test program, as required by standard ASTM protocols, including Practices E 1601 and E 691.

8.7.2 Add a section that summarizes the derivation of the intralaboratory test data and any detailed measurement uncertainty budget requirements the task group elected to include in the method. Use Appendices and Annexes as appropriate.

8.8 *Report*:

8.8.1 Require that a laboratory comply with all requirements, including data quality of the standard test method in order to state on a test report that the test result was generated using this test method. Require the laboratory to comply with all reporting requirements of ISO 17025.

8.9 *Annex and/or Appendices*:

8.9.1 Provide sufficient detail on how the uncertainty budget data was generated during the interlaboratory study and how the requirements summarized in the Precision and Bias statement shall be interpreted by laboratories that use the standard method. Use either an Annex or an Appendix, as appropriate.

9. Verifying the Drafted Method

9.1 *Verification Laboratory*—Select a competent laboratory (one that complies with the applicable clauses of ISO 17025) which utilizes the measurement technique to analyze the material to be covered in the new standard test method. Obtain sufficient performance data from that laboratory to demonstrate

that the technique of choice can achieve the expected data quality objectives for all elements and concentration ranges, making sure to cover any which might be difficult to achieve. If the results from the selected laboratory cast doubt on the ability to achieve ultimate success, consider redesigning the proposed test method. This step is primarily intended to provide design information to help the task group effectively plan its work program. Therefore, design details are left to the task group.

9.2 If the test materials and interlaboratory test protocol are available as described in Section 10, the task group may elect to have the verifying laboratory perform the interlaboratory study protocol as a test case. If successful, the results of the verifying laboratory may be used as a participating laboratory in the final calculations.

10. Drafting the Interlaboratory Test Protocol

10.1 *Select Test Materials*—Select test materials that cover the elements and concentrations to be included in the new method. Ensure that suspected interferences are also covered. To the fullest extent possible, use CRMs that have reliable uncertainty estimates available for use in demonstrating the absence of measurable and correctable bias. If the test method includes sample preparation steps that might influence the quality of the results, be sure to include some test materials that need to be prepared by the participating laboratories and be prepared to evaluate the precision obtained on these materials. This will allow the task group to evaluate the effects of sample preparation on the precision. Ensure that the homogeneity of all test materials is known, including CRMs.

10.2 *Introduction*:

10.2.1 Explain that this test protocol covers the development of a performance-based method and that the performance of participating laboratories will be used to establish the data quality that all laboratories using the new method will need to achieve in order to claim that they followed the test method. Explain that the Interlaboratory Study is based on Plan A in Practice E 1601 and that all test materials are known to be homogeneous.

10.2.2 Explain that the aim data quality objectives included in this work plan have been achieved by competent laboratories using similar test methods over time and by at least one competent testing laboratory using the draft method under test.

10.2.3 Advise the participating laboratory that the aim data quality objectives should be met during the formal interlaboratory test. If the participating laboratory cannot meet the aim data quality objectives, it should check the function of its equipment, and, if that fails, it should contact the task group chairman immediately.

10.3 *Qualification of Participating Laboratories*:

10.3.1 In order to avoid receiving inferior data, it is suggested that the task group ask the participating laboratory to provide data that shows that it is capable of obtaining acceptable data prior to actually conducting the test. The task group shall design the qualification test and request that the qualification data be generated before conducting the interlaboratory test. The qualification data shall be submitted to the coordinator no later than with the final data package.

10.3.2 Typical qualification packages might include demonstration of ability to achieve adequate signal/noise ratios, demonstrated ability to calibrate over the concentration ranges of interest, and sufficient measurement precision over the anticipated concentration range to achieve the desired analytical performance. In some cases these requirements might be fulfilled by asking the participating laboratory to analyze specified CRMs a given number of times, back to back, on one day, using the participating laboratory's existing in-house procedure.

10.3.3 In designing the qualification and final tests, the task group should carefully give specific directions so that a laboratory will know immediately whether or not its measurement performance meets expectations. In the event that the laboratory does not meet expectations, it has time to resolve the problem before spending time and resources on the official test.

10.4 *Instructions for Carrying Out the Interlaboratory Study*:

10.4.1 The instructions for conducting the interlaboratory test should be limited to the test itself and should not alter any instructions in the draft test method. Give instructions to assist the task group with evaluation of data, for example, by providing data sheets. Data sheets should define the number of significant figures to be supplied by participating laboratories. Without such instructions, the task group may receive data in a form that is not optimum for statistical analysis. Refer to Practice E 1601 for further instructions regarding statistical calculations.

10.4.2 The following types of information might be routinely required by the task group: *(1)* all pre-qualification data; *(2)* make and model of sample preparation and measuring equipment, the use of which might affect data quality; *(3)* list of CRMs used for calibration, with copies of certificates for each showing measurement uncertainties; *(4)* calibration records showing the degree of curve fit and calibration measurement uncertainty; *(5)* list of RMs used for control and standardization, as appropriate, with documentation showing their homogeneity; *(6)* all test data used to calculate each report value included in the interlaboratory test; and *(7)* statements relating to compliance with aim quality objectives.

10.4.3 Provide information on the measurement uncertainty expectations required of the cooperating laboratories and provide resources to contact with questions.

10.4.4 Ask for comments from the participating laboratory.

10.4.5 Set a deadline for submission of all test results to the task group chair.

11. Conducting the Interlaboratory Test

11.1 For each participating laboratory, prepare a packet containing: *(1)* a cover letter (optional, but recommended); *(2)* the drafted method; *(3)* instructions for the interlaboratory test; *(4)* report forms, including data sheets; and *(5)* test samples. Refer to Practice E 1601 for background on conducting an interlaboratory test.

11.2 Receive data sets and comments from all participating laboratories.

11.2.1 Review the qualification data from each laboratory to ensure that the laboratory was able to meet the general competency requirements associated with the test. If the

qualification data is not acceptable, contact the laboratory and resolve the issues before evaluating the test method data. The availability of qualification data helps ensure that measurement uncertainties calculated as a result of the interlaboratory test are representative of competent laboratories. If the qualification data is satisfactory, but the quality of the test data is not, there may be good reason to question the draft test method.

11.3 Prepare a Precision and Bias table and statement in accordance with ASTM requirements. Refer to Practice E 1601 or Practice E 691. Add a brief statement that defines the maximum intralaboratory precision that a laboratory can achieve and still be able to report results in accordance with the new standard test method.

11.4 If the task group elected to include a measurement uncertainty budget as part of the test method, add an additional section to the Precision and Bias statement that describes the maximum uncertainty that a laboratory can achieve at each step and refer to the appropriate appendix/annex for details.

12. Writing the Research Report

12.1 Prepare the Research Report, covering all ASTM requirements and discussing fully the derivation of measurement uncertainty data, as described below.

12.2 Summarize the pre-qualification data so that the official record of the test method will contain evidence that the participating laboratories were capable of performing the test method. Exclude the data from any non-qualified laboratories from further evaluation.

12.3 For each element in each test sample, calculate the parameters required in Practice E 1601. Prepare the usual Precision and Bias table.

12.4 For each element in each test sample, calculate the interlaboratory precision, 95 % confidence, as described in Practice E 1601. Divide by the square root of 2 to estimate the intralaboratory precision. Compare the intralaboratory precision values with the aim data quality objectives. If acceptable, draw a best fit line through the points on a log-log plot, and create a smoothed data table for each element, or, preferably, for all elements, if appropriate. This relationship defines the required intralaboratory performance (95 % confidence) that must be demonstrated in order to report that a set of test results were generated using the new standard test method.

12.5 If the task group wrote the test method including an uncertainty budget, perform calculations as described in 6.3.3 for control, calibration, and reference materials.

13. Finalizing the Method

13.1 Review and revise the draft method as needed for accuracy and completeness according to the usual ASTM procedures as supplemented by this practice.

13.2 Provide the Precision and Bias section. Add a section to the Precision and Bias section defining the intralaboratory performance requirements as described in 6.3.3. Add a sentence indicating that a laboratory that uses this method must meet these performance criteria in order to claim that a report was generated using this test method.

13.3 If an uncertainty budget is included in the method, provide a summary of those requirements either as a separate section or as an appendix or annex, as appropriate. One acceptable way to present the numerical data is in the form of a table, similar to that shown in Practice E 2165.

14. Keywords

14.1 analytical chemistry; measurement uncertainty; measurement uncertainty budget; performance-based test method

APPENDIX

(Nonmandatory Information)

X1. SUGGESTED MEANS FOR ESTABLISHING AIM MEASUREMENT UNCERTAINTIES FOR PERFORMANCE-BASED METHODS AND CONFIRMING THEM DURING INTERLABORATORY TESTING

X1.1 Introduction

X1.1.1 As described throughout this practice, performance-based methods rely much more heavily on the quality of the test results than on obedience to prescriptive experimental procedures. It follows that those who write performance-based standard test methods must be able to clearly define acceptable performance. This practice defines acceptable performance as the intralaboratory precision (95 % confidence) obtained during interlaboratory testing. It then provides one approach for task groups to incorporate model uncertainty budgets in standard test methods, also in harmony with Practice E 2165.

X1.1.2 A primary advantage associated with the use of this uncertainty practice in conjunction with the aim uncertainties given in Practice E 2165 is that the task group will be assured that its aim uncertainty objectives are comparable to other standard test methods and to proficiency test performance in general. This means that laboratories that participate in the interlaboratory testing can be reasonably confident that the aim uncertainties set by the task group are reasonable and achievable. It also means that test results achieved by the laboratories that use the test method will comply with general good laboratory practice and that the laboratory's results submitted to proficiency test programs will be consistent with other participants and other test methods.

X1.1.3 If the task group sets data quality objectives that are less stringent than those identified in Practice E 2165, then there is a lower probability that laboratories that use the

method and contribute to proficiency tests that include other test methods, will perform as predicted by the model.

X1.2 A Model for Establishing Aim Data Quality Objectives

X1.2.1 *Intralaboratory Precision*—The task group should establish acceptability criteria for intralaboratory precision as guidance for the cooperators in the interlaboratory test. For any concentration, this value can be calculated as described in Practice E 2165. Be sure to report the results as 95 % confidence in order to comply with ISO 17025.

X1.2.2 *Uncertainty Budget*—It has been shown that the performance (precision without bias) obtainable by competent laboratories performing optimized, state-of-the-art methods can be described by a straight line on a log-log plot of performance versus concentration.[4] This model has been verified using both proficiency test data and interlaboratory testing of new standard methods of analysis. A scheme for applying these principles to uncertainty budgets is provided in Practice E 2165. This document applies those principles to writing performance-based standard test methods.

X1.2.2.1 *Control*—Divide the intralaboratory precision by the square root of 2 to find the aim uncertainty for the use of control materials when performing the test method. Note that the aim control uncertainty is expressed as 2 sigma or 95 % confidence. It may be helpful to interpret control charts using the Westgard Rules as described in Practice E 1329.

X1.2.2.2 *Calibration*—Divide the aim uncertainty for the control function by the square root of 2 to find the aim uncertainty for the calibration function. This may be interpreted as the maximum difference between the assumed true value of any calibrant and the calculated curve fit through that point. For purposes of assessing the calibration uncertainty, the task group may assume that, because the curve fit is a calculated function that averages responses from several reference materials, all points along the calibration curve should comply with the 95 % confidence.

X1.2.2.3 *Calibrants*—Divide the aim uncertainty for the calibration function by the square root of 2 to find the aim uncertainty at the assumed true concentration value for each calibrant.

X1.3 Confirming the Measurement Uncertainty Budget During Interlaboratory Testing

X1.3.1 If the task group has determined that a full uncertainty budget is to be included in the standard test method, each aim uncertainty budget item shall be included in the Interlaboratory Study work plan so that each laboratory knows that it is expected to meet or exceed these uncertainties during its testing of the method.

X1.3.2 When all test results are in, the task group will compare the aim uncertainties with the experimentally determined values and then adjust the uncertainty budget values as necessary. However, if the experimentally obtained uncertainties are significantly worse than the aim values (confirmed during verification testing), there is reason to believe that the new test method may not be fully optimized and that further revision is needed.

X1.3.3 The data, the findings, and an explanation of decisions made shall be included in the Research Report. A summary shall be included in the test methods for use by laboratories in implementing the new test method.

[4] Flinchbaugh, D. A., Crawford, L. F., and Bradley, D., "A Model to Set Measurement Quality Objectives and to Establish Measurement Uncertainty Expectations in Analytical Chemistry Laboratories Using ASTM Proficiency Test Data," *Accreditation and Quality Assurance*, 6, 2001, pp. 493-500. (www.springeronline.com)

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