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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials[1]

This standard is issued under the fixed designation C 1156; 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.

ϵ^1 NOTE—Figure 1 was corrected editorially in February 1997.

1. Scope

1.1 This guide provides the basis for establishing calibration for a measurement method typically used in an analytical chemistry laboratory analyzing nuclear materials. Guidance is included for such activities as preparing a calibration procedure, selecting a calibration standard, controlling calibrated equipment, and documenting calibration. The guide is generic and any required technical information specific for a given method must be obtained from other sources.

1.2 The guidance information is provided in the following sections:

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General Considerations	5
Calibration Procedure	6
Calibration Standard	7
Control of Calibrated Equipment	8
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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:*

C 859 Terminology Relating to Nuclear Materials[2]

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry[2]

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials[2]

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry[2]

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration, n*—the determination of the values of the significant parameters by comparison with values indicated by a reference instrument or by a set of reference standards. **C 859**

3.1.2 *calibration curve, n*—the graphical or mathematical representation of a relation between a measured parameter and a property of the standard for the substance under consideration. **C 859**

3.1.3 *calibration factor, n*—the slope of the calibration curve, or its inverse. **C 859**

3.1.4 *calibration result, n*—the result obtained in an analysis of a calibration standard. The expected value of the calibration result is the reference value of the calibration standard. **C 859**

3.1.5 *calibration standard, n*—any of the standards of various types having accepted parameters. The calibration standard may be used to adjust the sensitivity setting of test instruments at some predetermined level and for periodic checks of the sensitivity. **C 859**

3.1.6 *calibration verification, n*—the action taken to verify the continued validity of calibration during a time period between calibration and required recalibration (see 5.3). Verification involves less rigor and effort than full calibration and involves analyzing a standard at a specified frequency during the calibration period. Verification could involve using a standard that is lower than the calibration standard in the metrological hierarchy of standards (see 7.1).

4. Significance and Use

4.1 Calibration is a fundamental part of making measurements and its effect on the quality of measurement data is significant. Thus, sufficient attention must be given to calibration when it is established for a measurement method so that

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[2] *Annual Book of ASTM Standards*, Vol 12.01.

the data produced will be acceptable. The use of an inappropriate calibration standard, inadequate instructions for calibration, and poor documentation of the calibration process are examples of circumstances that can adversely affect the validity of a calibration. Thus, the calibration process must conform to criteria established to ensure the validity of calibration results. Such criteria are given in Guide C 1009, in which calibration is identified as a component of laboratory quality assurance (see Fig. 1). This guide expands upon those criteria to provide more comprehensive guidance for establishing calibration.

4.2 The manner of calibration and other technical requirements for calibrating a measurement method are usually established when a method is first introduced into a laboratory, which may be through validation and qualification as defined by Guide C 1068 (see Fig. 1). However, calibration involves more than the technical aspects of the calibration process. The other dimension of the process is the operational requirements that are necessary to ensure that calibration results are valid and that they are documented and verifiable should their integrity be questioned. The provisions of this guide provide those operational requirements and should be considered whenever calibration is planned and established.

5. General Considerations

5.1 The degree of attention and effort given to calibration should depend on how the measurement data are to be used. In the analysis of nuclear materials, for example, measurement data produced for the control and accountability of nuclear material would normally require more attention than data produced for process control during the processing of that material. The areas in which the level of attention and effort could vary are: the calibration standard, number of calibration points, frequency of calibration, and frequency of calibration verification.

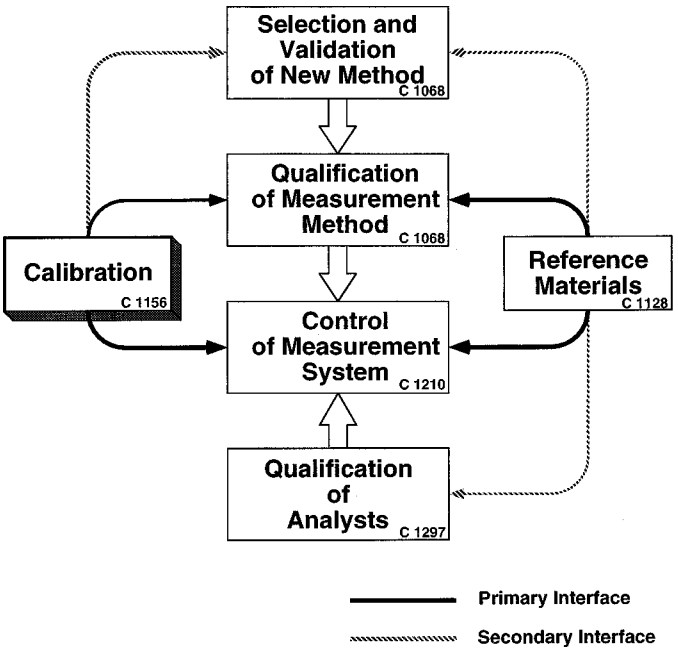

FIG. 1 Quality Assurance of Analytical Laboratory Data

5.2 Many of the provisions of this guide would not apply to the calibration of certain instruments when their calibration is an integral part of the analysis procedure involving a simple one- or two-step adjustment of a meter or gage. The pH meter is an example when a buffer is used to adjust the meter just before a pH reading is taken for a sample solution.

5.3 There are generally two approaches regarding frequency of calibration. In one case, the method is calibrated each time it is used. In the other, calibration is established for a specified period of time, and the method must be recalibrated before that time period elapses to retain calibration. When a calibration period is used, calibration verification should be used. A calibration period might be defined in terms of weeks or months, or defined as a run of a series of samples over a relatively short period of time. In the latter case, calibration verification could involve analyzing a standard periodically during the sample run, for example, after every fifth sample.

5.4 When calibration is being planned and established, a statistician should be consulted regarding the treatment of calibration data, the frequency of calibration, the frequency of calibration verification, and the criteria that determine when calibration has been achieved (see Guide C 1215).

5.5 The organizational responsibility and authority for calibration should be defined and documented. Normally, responsibility for calibrating an individual method rests with the analyst using the method. If the responsibility for calibrating an instrument or class of instruments is contracted to another organization, the laboratory is still responsible for ensuring that calibration requirements are being met by the organization doing the calibration.

6. Calibration Procedure

6.1 Calibration should be established as a written procedure. The procedure should provide instructions for those doing the calibration, and it should document the basis for calibration, which can be used to substantiate the validity of the calibration process, should that be required (see 9.2).

6.2 *Preparation*—The calibration procedure can be prepared as a separate procedure from the one written for the measurement method or it can be a section of the method's procedure as long as the provisions given in 6.3 are addressed. If the former approach is used, the applicable measurement method should be clearly identified in the calibration procedure. The calibration procedure should be reviewed for technical adequacy and approved by management. The provisions contained in the Procedure section of Guide C 1009 regarding the preparation, review, and approval of procedures should be considered. Also, calibration procedures should be revised, distributed, and controlled according to the provisions in the Procedure section of Guide C 1009.

6.3 *Content*—The following subjects should be addressed in the procedure:

6.3.1 Identification of the equipment or portion of the measurement apparatus that requires calibration,

6.3.2 Identification of the calibration standard or standards that will be used and inclusion of instructions for the preparation, pretreatment, and use of the standard(s) as appropriate (see 7.3 and 7.4),

6.3.3 A statement of the required frequencies of calibration