Designation: C 1156 – 95ε1

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

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:

	Section
General Considerations	5
Calibration Procedure	6
Calibration Standard	7
Control of Calibrated Equipment	8
Documentation	9
Keywords	10

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

[1] This guide is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.08 on Quality Assurance Applications.

Current edition approved Nov. 10, 1995. Published January 1996. Originally published as C 1156 – 90. Last previous edition C 1156 – 90.

[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.

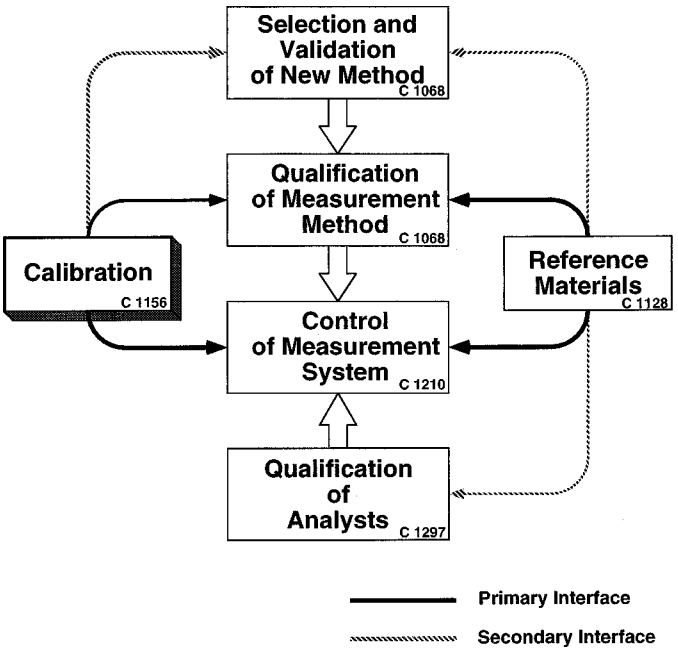

FIG. 1 Quality Assurance of Analytical Laboratory Data

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

and calibration verification as appropriate and a description of any situations or conditions that would alter the frequencies (see 5.3),

6.3.4 Instructions, in a step-by-step format, for performing the calibration, including applicable instructions for calculation of a calibration factor, preparation of a calibration curve, or other treatment of the calibration data required to finalize the calibration process. If calibration verification is required, include instructions for verification (see 8.3).

6.3.5 Criteria that establish when the method or equipment is in- or out-of-calibration, and

6.3.6 A description of actions required to bring the method or equipment back into calibration should calibration be lost.

7. Calibration Standard

7.1 Calibration standards are classified as reference materials. A certified reference material (CRM) is the highest level of standard in the metrological hierarchy of reference materials, followed by a working reference material (WRM). Lower levels of reference materials can exist, depending upon the requirements for a particular application. The level of reference material is governed by the rigor, care, and overall effort put into the preparation and characterization of the material. Guide C 1128 provides a definition of CRM and WRM and addresses the various factors that affect the quality of reference materials.

7.2 *Selection*—The level of a standard required for calibration depends on the importance of the measurement data to be produced and the degree of uncertainty required for the data (see 5.1). Selection should be based on these requirements; for example, a CRM should not be selected when a lower level standard would suffice. Availability, stability, traceability to the national measurement base, and cost are other considerations that could affect selection (see Guide C 1128).

7.3 *Preparation*—Preparation may vary from making a simple dilution of a stock (master) solution to a major preparation and characterization effort as described in Guide C 1128. In some situations, a pretreatment of the standard might be required before use. Instructions for the preparation or pretreatment of the standard should be included in the calibration procedure, or at least a reference to such instructions should be given (see 6.3.2). If the standard is a pre-prepared standard that requires simply taking a packaged unit for a one-time use, or if it is a physical standard (as opposed to a chemical standard) that is used repeatedly, then the source and description of the standard should be included in the calibration procedure.

7.4 *Use*—If special storage or handling practices are required to protect the integrity of the standard, those practices should be provided in the calibration procedure (see 6.3.2). Protecting the integrity of standards in terms of packaging and storage is addressed in Guide C 1128.

8. Control of Calibrated Equipment

8.1 *Identification*—Equipment requiring calibration should be identified in the calibration procedure (see 6.3.1). In addition, there should be a mechanism on the equipment to indicate that it is calibrated equipment. This could be done with a tag or label.

8.2 *Calibration Status*—There should be a mechanism for keeping a current indication of calibration status when an item of equipment is calibrated for an extended period of time (see 5.3). This should not be necessary if the item is calibrated each time it is used. Status can be indicated on the calibrated equipment with a label stating when calibration was done and when it elapses. An alternative would be to document status in the data record system used to record calibration data and results (see 9.3). Those records should be readily available to show current status. In addition, an up-to-date master calibration record for all calibrated equipment should be kept at a central location in the laboratory.

8.3 *Use*—If an item of equipment is calibrated for a specified time period, it should be used and handled in a manner to help ensure that calibration will remain valid during that period (see 5.3). If there is a reason to believe that calibration has become invalid during the calibration period, the equipment should not be used until the situation has been evaluated and corrected (if necessary). A calibration verification is one way to check on the validity of calibration during the calibration period (see 6.3.3 and 6.3.4). Should any measurement data be called into question because of an uncertainty in calibration status, that data should be evaluated for acceptability and not used if judged unacceptable. Actions taken should be documented and justified, particularly if the data are judged acceptable.

8.4 *Out-of-Calibration*—If a calibration time period elapses without recalibration, the item of equipment involved should be considered as being out-of-calibration, and it should not be used to generate measurement data. If the item is not to be recalibrated, it should be labeled or tagged to indicate its status to avoid inadvertent use as calibrated equipment.

9. Documentation

9.1 Without assurance that measurement data were obtained under calibrated conditions, their validity can be challenged. Thus, calibration should be documented to provide substantiation that measurement data were generated while the method was in-calibration and that an established and approved technique was used for calibration. Documentation should provide the evidence and support for judgments regarding the quality of measurement data and should provide historical evidence needed for future reviews and evaluations, particularly if regulatory or legal issues are raised about the data (see the Control of Records section of Guide C 1009).

9.2 *Calibration Procedure*—The calibration procedure should document the technical aspects of calibration. By doing so, it establishes the approved way in which calibration is done, describes the calibration standard or standards used, and defines the output from calibration, for example, calibration factor, calibration curve, or meter adjustment. The procedure should document the statistical treatment used for the calibration data and the criteria that govern calibration status, that is, in-calibration or out-of-calibration.

9.3 *Data Record*—The laboratory data record system should be used to document calibration: who did it, what was done, and when it was done (see the Laboratory Records section of Guide C 1009). The calibration data obtained, who generated the data and when, the calibration calculations or treatment of the data, special observations made during calibration, and a

summary of actions taken to regain calibration if lost are the types of calibration information that should be recorded in the data record system. Through the data record, it should be possible to determine the status of calibration at any point in time when measurement data were generated. An overall discussion of a laboratory record system is found in the Laboratory Records section of Guide C 1009.

10. Keywords

10.1 calibration; calibration procedure; calibration standard; certified reference material (CRM); laboratory equipment