



Standard Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials¹

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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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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 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry²

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry²

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials²

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry²

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry²

C 1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials²

2.2 ISO Standard:

ISO 17025 General Requirements for the Competence of Calibration and Testing Laboratories³

3. Significance and Use

3.1 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 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.

3.2 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

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² Annual Book of ASTM Standards, Vol 12.01.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

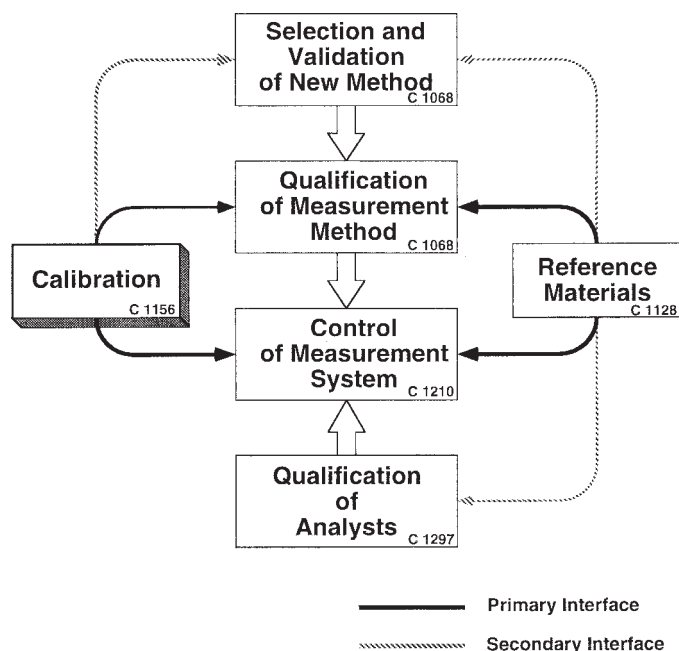


FIG. 1 Quality Assurance of Analytical Laboratory Data

integrity be questioned. The provisions of this guide provide those operational requirements and should be considered whenever calibration is planned and established.

4. General Considerations

4.1 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.

4.2 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.

4.3 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.

4.4 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 checks, and the criteria that determine when calibration has been achieved (see Guide C 1215).

4.5 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.

5. Calibration Procedure

5.1 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.

5.2 *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 5.3 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.

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

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

5.3.2 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;

5.3.3 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;

5.3.4 Instructions, in a step-by-step format, for performing the calibration, including applicable instructions for calculation of the slope of the calibration curve, preparation of a calibration curve, or other treatment of the calibration data (for example, corrections for environmental conditions) required to finalize the calibration process.

5.3.5 Criteria that establish when the method or equipment needs recalibration.

6. Calibration Standard

6.1 Calibration standards are 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). 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.

6.2 Selection—The level of a standard required for calibration depends on the requirements for the measurement data to be produced. 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 a national measurement base, and other considerations that could affect selection (see Guide C 1128).

6.3 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. 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.

6.4 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. Protecting the integrity of standards in terms of packaging and storage is addressed in Guide C 1128.

7. Control of Calibrated Equipment

7.1 Identification—Equipment requiring calibration should be uniquely identified. The identification should be marked on the equipment (for example, with tags, labels, or markings) and on related calibration records.

7.2 Calibration Status—There should be a process for keeping a current indication of calibration status. Status can be indicated on the calibrated equipment with a label stating when

calibration was done and when it expires. An alternative would be to document status in the data record system used to record calibration data and results. Those records should be readily available to show current status.

7.3 Use—Calibration equipment should be used and handled in a manner to help ensure that calibration will remain valid during the calibration period. 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).

7.4 Out-of-Calibration—If a calibration time period expires, the equipment involved should be recalibrated or removed from service. It should not be used to generate measurement data.

8. Documentation

8.1 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 of the data (see the Control of Records section of Guide C 1009).

8.2 Data Record—The laboratory data record system should be used to document calibration (see the Laboratory Records section of Guide C 1009). For each piece of equipment, calibration records should identify the person performing the calibration, calibration date, calibration procedure, standards used, calibration data, special observations during calibration, and a description of actions taken as a result of any out-of-calibration conditions.

9. Keywords

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

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