Designation: C1210 – 21

Standard Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within Nuclear Industry[1]

This standard is issued under the fixed designation C1210; 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 guide provides guidance for establishing and maintaining a measurement system quality control program. Guidance is provided for general program considerations, preparation of quality control samples, analysis of quality control samples, quality control data analysis, analyst qualification, measurement system calibration, measurement method qualification, and measurement system maintenance.

1.2 This guidance is provided in the following sections:

	Section
General Quality Control Program Considerations	5
Quality Control Samples	6
Analysis of Quality Control Samples	7
Quality Control Data Analysis	8
Analyst Qualification	9
Measurement System Calibration	10
Qualification of Measurement Methods and Systems	11
Measurement System Maintenance	12

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C859 Terminology Relating to Nuclear Materials

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1108 Test Method for Plutonium by Controlled-Potential Coulometry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

2.2 *ANSI Standards:*[3]

ANSI/ASQ B1 Guide for Quality Control Charts

ANSI/ASQ B2 Control Chart Method of Analyzing Data

ANSI/ASQ B3 Control Chart Method of Controlling Quality During Production

2.3 *ISO Standard:*[4]

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories

ISO/IEC 17043 Conformity assessment — General requirements for proficiency testing

ISO Guide 80 Guidance for the in-house preparation of quality control materials (QCMs)

3. Terminology

3.1 For definitions of terms used in this guide but not defined herein, see Terminology C859.

3.2 *Definitions:*

3.2.1 *calibration, n*—the set of operations that establishes, under specified conditions, a metrologically traceable relationship between a value measured or indicated by an instrument or system to a corresponding known value, typically derived from appropriate reference standards or established physical constants

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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, http://www.ansi.org.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

3.2.1.1 *Discussion*—The calibration relationship can be expressed by a statement, function, diagram or table.

3.2.1.2 *Discussion*—Test Method C1108 is an example of calibration using established physical constants.

3.2.1.3 *Discussion*—Additional details on calibration requirements for measurement methods used for the nuclear fuel cycle can be found in Guide C1156.

3.2.2 *measurement method, n*—Technique for determination of the presence, or quantity, or both, of one or more analytes in a sample.

3.2.2.1 *Discussion*—A measurement method may utilize chemical reactions (such as titrations), instrumentation (such as a spectrometer), or both. Any sample preparation required prior to the analysis is part of the measurement method.

3.2.3 *measurement system, n*—set of all components used to produce an analytical result.

3.2.3.1 *Discussion*—Components may include, for example, equipment or instrumentation used to prepare samples, perform the analytical measurement, perform any needed data manipulations, and record results in printed or electronic form, or both.

3.2.4 *metrological traceability, n*—property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty.

3.2.5 *qualification, n*—of a measurement method, a formal process to provide a desired level of confidence that measurement methods used will produce data suitable for their intended use. **C1068**

3.2.5.1 *Discussion*—Qualification follows verification and validation, and ensures that methods meet established criteria prior to use and when placed into use.

3.2.6 *quality control program, n*—documented system of activities utilizing quality control samples and appropriate acceptance criteria to ensure that measurement systems are producing results that are suitable for their intended purpose.

3.2.7 *quality control sample, n*—a sample used to verify and monitor measurement system performance and reliability.

3.2.7.1 *Discussion*—Quality control samples may include knowns, unknowns, blinds, blanks, duplicates, matrix spikes, and so forth.

3.2.7.2 *Discussion*—Quality control samples may be used to estimate bias in the measurement system or analytical method.

3.2.8 *validation, n*—of a measurement method, provision of objective evidence that a given item fulfils specified requirements where the requirements are adequate for an intended use. **ISO/IEC 17025**

3.2.9 *verification, n*—of a measurement method, provision of objective evidence that a given item fulfils specified requirements. **ISO/IEC 17025**

4. Significance and Use

4.1 A laboratory quality assurance program is an essential program for laboratories within the nuclear industry. Guide C1009 provides guidance for establishing a quality assurance program for an analytical laboratory within the nuclear industry. This guide deals with the control of measurements aspect of the laboratory quality assurance program. Fig. 1 shows the relationship of measurement control with other essential aspects of a laboratory quality assurance program.

4.2 The fundamental purposes of a measurement control program are to provide the *with-use* assurance (real-time control) that a measurement system is performing satisfactorily and to provide the data necessary to quantify measurement system performance. The *with-use* assurance is usually provided through the satisfactory analysis of quality control samples (reference value either known or unknown to the analyst). The data necessary to quantify measurement system performance is usually provided through the analysis of quality control samples or the duplicate analysis of process samples, or both. In addition to the analyses of quality control samples, the laboratory quality control program should address (*1*) the preparation and verification of standards and reagents, (*2*) data analysis procedures and documentation, (*3*) calibration and calibration procedures, (*4*) measurement method qualification, (*5*) analyst qualification, and (*6*) other general program considerations. Other elements of laboratory quality assurance also impact the laboratory quality control program. These elements or requirements include (*1*) chemical analysis procedures and procedure control, (*2*) records storage and retrieval requirements, (*3*) internal audit requirements, (*4*) organizational considerations, and (*5*) training/qualification requirements. To the extent possible, this standard will deal primarily with quality control requirements rather than overall quality assurance requirements, which are addressed in Guide C1009.

4.3 Although this guide uses suggestive rather than prescriptive language (for example, "should" as opposed to "shall"), the elements being addressed should not be interpreted as optional. An effective and comprehensive laboratory quality control program should, at minimum, completely and adequately consider and include all elements listed in Section 1 and in the corresponding referenced sections of this guide.

5. General Quality Control Program Considerations

5.1 The quality control activities described in this guide are intended for a quality control function which is internal to an analytical chemistry laboratory. The quality control program should have an administrator or manager working in concert with laboratory managers to produce cost effective measurements of demonstrated and acceptable quality. The program manager should have the authority based on quality control sample performance to disqualify analysts or measurement systems, or to request or require additional quality control sample analyses. It is desirable for the quality control program to have periodic internal assessments on a pre-determined schedule. These assessments should involve laboratory managers, the quality control manager, and laboratory customers. The quality control program should be audited for procedure compliance at periodic intervals by the quality assurance organization.

5.2 *Documentation*—The analytical laboratory's quality control program should be described in laboratory procedures and all measurement system quality control activities should be

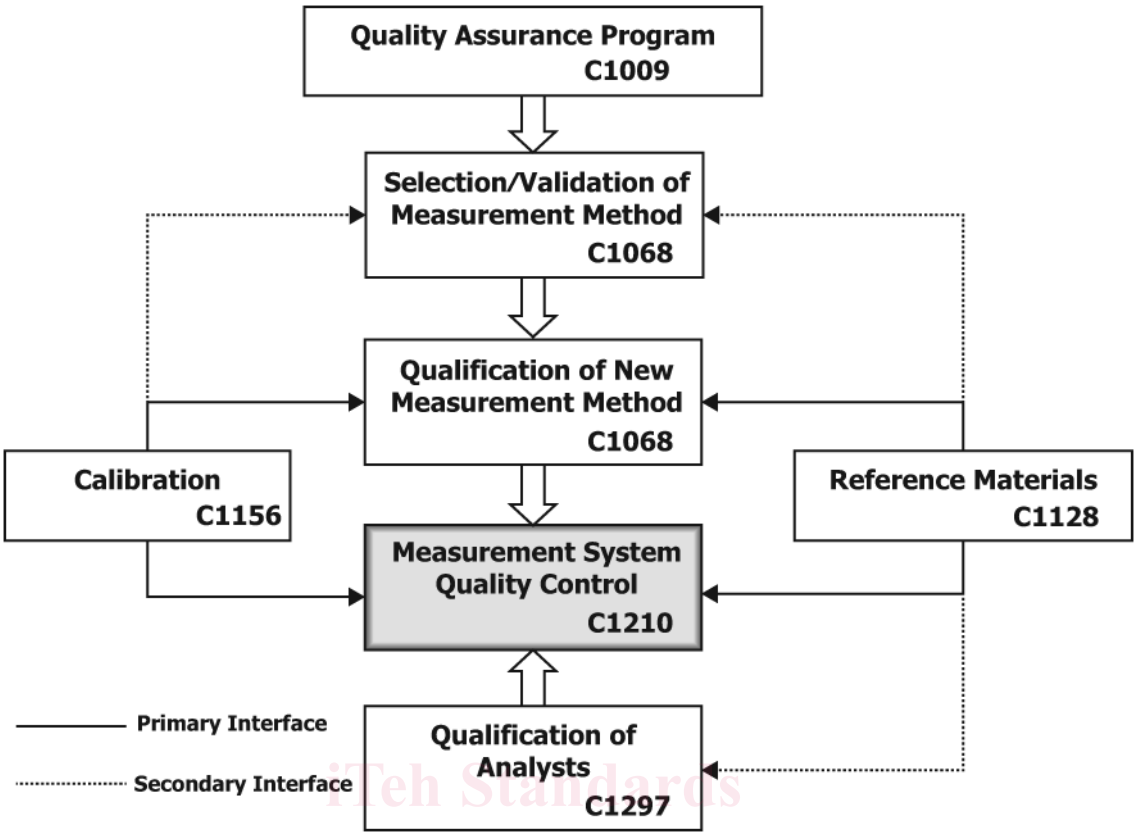

FIG. 1 Quality Assurance of Analytical Laboratory Data

documented. The retention period for the documentation should be described in laboratory procedures and consistent with other laboratory storage requirements and any applicable contractual or regulatory requirements.

5.3 *External Assessment:*

5.3.1 External quality control program ascsessment should be conducted by an outside organization or agency at a frequency dictated by company or facility policy, contract, or other applicable regulations or requirements.

5.3.2 To the extent possible, the laboratory should participate in applicable proficiency testing programs:

5.3.2.1 Use of a proficiency testing provider that conforms to ISO/IEC 17043 is strongly preferred but if not available, the laboratory should use the program which is most fit for purpose. This could mean a consensus-based proficiency testing program.

5.3.2.2 A consensus-based proficiency testing program is one where the results from each laboratory are compared to the aggregate results from all participating laboratories.

5.3.2.3 A proficiency testing program that provides an evaluation for each participating laboratory against a sample having a known value is preferred. However, for proficiency testing programs involving nuclear material measurements, this may not always be feasible.

5.3.3 Laboratory and quality control management should consider participation in external exchange programs, especially in the absence of a formal proficiency testing program. These may include interlaboratory exchange programs, sample exchange programs, sample or standard round robins, and referee analyses programs. These programs provide some degree of external validation of the measurement system quality control program.

6. Quality Control Samples

6.1 *Preparation:*

6.1.1 Quality control samples should be prepared or purchased over the measurement range of interest and have an impurity content and matrix composition that approximates the process samples, unless the measurement method has been shown to be free from sample matrix effects.

6.1.2 Quality control sample preparation procedures, specific requirements (purity of source materials and solvents; storage requirement; shelf life; and so forth), and the preparation should be documented.

6.1.3 Quality control samples may be prepared from the following:

6.1.3.1 Certified Reference Materials (CRMs);

6.1.3.2 Working Reference Materials (WRMs), prepared as described in Guide C1128;

6.1.3.3 Pure elements or compounds with vendor supplied assay;

6.1.3.4 Reagent grade (or better) chemicals with assay; and

6.1.3.5 Well-characterized process materials.

6.2 *Quality Control Samples:*

6.2.1 When used for comparison to a reference value (such as for calibration), exhibit metrological traceability to the Système International d'Unités (SI).

6.2.2 When used for statistical control of a measurement system, instrument performance checks, or determination of operator variability, need not be metrologically traceable to the SI. For these applications, quality control materials (QCM), as defined by ISO Guide 80, may be used.

6.3 When quality control samples are prepared, the preparation procedure and data (mass, volume, etc.) should be documented. Further, appropriate measurements should be performed to verify the prepared value.

6.4 The quality control samples should be characterized to establish their reference values when prepared from materials with uncertain assays, or from process material, or when smaller uncertainties are required on the samples than can be obtained from the source materials. A record of the preparation procedure and data should be maintained. The characterization method or procedure, complete with calibration data and the characterization analysis results, should be referenced or included in the preparation data.

6.5 Quality control samples should be prepared from a different supplier lot from the standards used for calibration (see Guide C1156).

6.6 All quality control samples should be labeled with (*1*) the concentration, activity, abundance, etc. of the species of interest, (*2*) solvent if other than water, (*3*) matrix, (*4*) date prepared, (*5*) identification of preparer, and (*6*) storage requirements or limitations. Alternately, QC samples should be coded in such a manner as to uniquely identify this same information.

6.7 All incoming chemicals and RMs should be labeled with a shelf life, acceptance date, or expiration date, if applicable.

7. Analysis of Quality Control Samples

7.1 The analysis of data from quality control samples provides a demonstration of measurement system performance and provides the information necessary to quantify that performance over the portion of the system covered by the quality control samples. The reference value of the quality control samples may be either known or unknown to the analyst.

7.1.1 The analysis of known quality control samples can provide a satisfactory bench demonstration of whether a system is in- or out-of-control without the need for a computer based quality control program. In general, the data resulting from the analysis of known quality control samples is not recommended for quantifying measurement system performance.

7.1.2 In general, the analysis of unknown quality control samples provides the data necessary to quantify measurement system performance. The data resulting from the analysis of unknown quality control samples may also be used to provide the *with-use* assurance of method performance, but some form of computer based system would be required in order to provide the real-time, at-bench determination of system performance. The use of unknown quality control samples for both functions can significantly increase the amount of data available to model measurement systems.

7.2 The frequency of analysis of quality control samples should be determined and described in laboratory procedures. The frequency should be a function of the stability of the measurement system.

7.3 To the extent possible, quality control samples should be representative of the samples that will be measured.

7.4 Quality control samples should be subjected to the same analysis conditions as the actual samples. The condition should be the same over the entire analysis sequence from sample aliquoting and preparation to data reduction.

7.4.1 When quality control samples are not subjected to a portion of the sample analysis sequence, sufficient documentation should exist to demonstrate that the portion of the system that is not covered does not contribute significantly to the measurement system bias and precision. The limitations that exists for not covering the entire sequence should be understood and documented.

7.4.2 Even though sample aliquoting by mass or by volume may be included in the analysis of quality control samples, this function is so fundamental and common to nearly all measurement systems that laboratories should maintain calibration and quality control programs on balances and, if applicable, on volume aliquoting and measuring devices. Balance and volume aliquoting devices should be treated as measurement systems or methods and should have calibration and quality control programs that satisfy the information contained in this guide and in Guide C1009.

7.5 The analysis of quality control samples should be documented. The documentation should include, but not necessarily be limited to, date and time of analysis, measurement system identification, analyst identification, quality control sample reference value or code, analysis results, analysis raw data, and whether the analysis passed or failed system performance criteria.

7.6 The data resulting from the analyses of quality control samples should be evaluated against pre-established acceptance criteria, and a determination made as to whether the measurement system is in- or out-of-control.

7.6.1 At a minimum, this should include measurement method control limits (real-time, at-the-bench, by plotting on some form of control chart or by computer assessment).

7.6.2 Acceptance criteria may also be established for repeatability of measurement of the quality control samples. Replicates that differ by more than a pre-determined percentage may indicate an adverse condition, such as instrument drift, that requires further evaluation.

7.7 Corrective actions for an out-of-control measurement system should be defined and documented. The quality control program should define responsibilities for taking corrective actions and should establish reporting requirements to technical and operation management.

7.7.1 If the measurement system is out-of-control, corrective actions should be initiated and measurement system