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

This standard is issued under the fixed designation C 1210; 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—Editorial changes were made throughout in March 1997.

1. Scope

1.1 This standard 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:

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2. Referenced Documents

2.1 *ASTM Standards:*

C 859 Terminology Relating to Nuclear Materials[2]

C 986 Guide for Developing Training Programs in the Nuclear Fuel Cycle[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 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials[2]

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

2.2 *ISO Standard:*

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials[3]

2.3 *ANSI Standards:*

ANSI/ASQC B1 Guide for Quality Control Charts

ANSI/ASQC B2 Control Chart Method of Analyzing Data

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *calibration*—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.

3.1.2 *calibration curve*—the graphical or mathematical representation of a relationship between a measured parameter and a property of the standard for the substance under consideration.

3.1.3 *calibration factor*—the slope of the calibration curve, or its inverse for a linear calibration curve.

3.1.4 *calibration standard*—any of the standards of various types having accepted values for parameters of interest.

3.1.4.1 *Discussion*—The calibration standard may be used to adjust the sensitivity of test instruments at some predetermined level and for periodic checks of the sensitivity.

3.1.5 *calibration verification*—the action taken to verify the continued validity of calibration during a time period between calibrations.

3.1.5.1 *Discussion*—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.

3.1.6 *certified reference material (CRM)*—a reference material one or more of whose property values are certified by a

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

[3] Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body (see ISO Guide 30).

3.1.6.1 *Discussion*—A certifying body is a technically competent body (organization or firm, public or private) that issues a reference material certificate (see ISO Guide 30). Such an organization could be the National Institute of Standards and Technology (NIST) or the New Brunswick Laboratory.

3.1.6.2 *Discussion*—A reference material certificate is a document certifying one or more property values for a certified reference material, stating that the necessary procedures have been carried out to establish their validity (see ISO Guide 30).

3.1.7 *quality control sample*—any sample used to verify or monitor measurement system performance.

3.1.8 *reference material (RM)*—a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (see ISO Guide 30).

3.1.8.1 *Discussion*—A reference material may also be referred to in this guide as a standard (for example, *calibration standard* or *control standard*).

3.1.9 *working reference material (WRM)*—a RM usually prepared by a single laboratory for its own use as a calibration standard, as a control standard, or for the qualification of a measurement method (see Guide C 1068).

3.1.9.1 *Discussion—Working reference material* replaces the definitions for secondary standard and working standard.

4. Significance and Use

4.1 A laboratory quality assurance program is an essential program for laboratories within the nuclear industry. Guide C 1009 provides guidance for establishing a quality assurance program for an analytical laboratory within the nuclear industry. The basic elements of a laboratory quality assurance program are organization, quality assurance program, training and qualification, procedures, laboratory records, control of records, control of procurement, control of measuring equipment and materials, control of measurements, and deficiencies and corrective actions. 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,

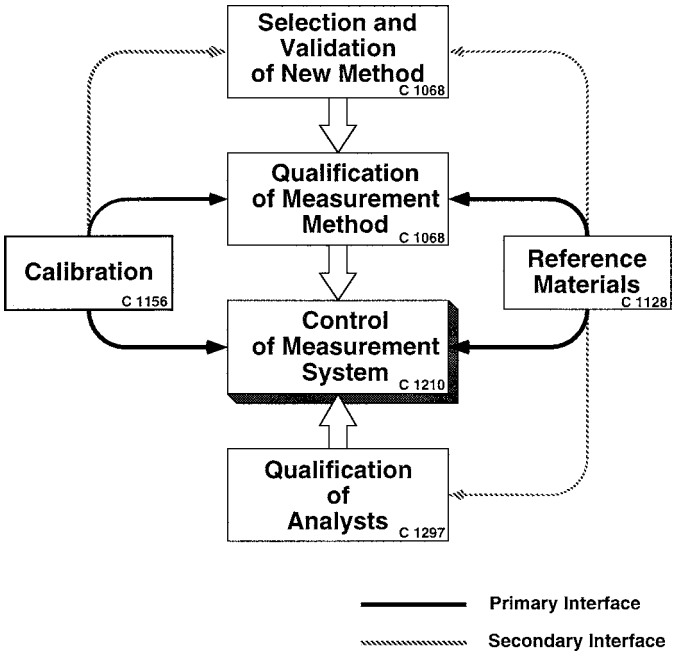

FIG. 1 Quality Assurance of Analytical Laboratory Data

(*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.

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 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. 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 The analytical laboratories quality control program should be described in laboratory procedures and all measurement system quality control activities should be documented. The retention period for the documentation should be described in laboratory procedures and should be consistent with other laboratory storage requirements.

5.3 External quality control program assessment 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. When possible, laboratory and quality control management should involve laboratory measurement systems in external exchange programs, such as: interlaboratory exchange programs, sample exchange programs, sample or standard round robins, and referee analyses programs. The programs provide some degree of external verification or validation of the measurement system quality control program that is desirable.

6. Quality Control Samples

6.1 Quality control samples (knowns, unknowns, blinds, blanks, etc.) are used to verify and monitor measurement system performance. 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. Quality control sample preparation procedures, specific requirements (purity of source materials and solvents; storage requirement; shelf life; etc.), and the preparation should be documented. Quality control samples may be prepared from the following: CRMs, WRMs, RMs, pure elements or compounds with vendor supplied assay, reagent grade (or better) chemicals with assay, and process materials. Guidance on the preparation of WRMs for use in the analysis of nuclear fuel cycle materials is provided in Guide C 1128.

6.2 When quality control samples or quality control sample stock solutions are prepared from CRMs, RMs, WRMs, pure elements or compounds with vendor supplied assay, or reagent grade (or better) chemicals with assay, records of the preparation procedure and sufficient data (mass, volume, etc.) should be maintained to demonstrate that the reference value of the source material was successfully transferred to the standard. Further, a chemical analysis should be performed to verify that the preparation was successful.

6.3 The solution should be characterized to establish its reference value when quality control samples or quality control sample stock solutions are prepared from materials with uncertain assays, or from process material, or when a smaller uncertainty is required on the solution 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.

6.4 When quality control RMs cannot be prepared and verified or characterized by the process described above, then the method of preparation, preparation data, and the basis for the assignment of the reference value should be documented and maintained.

6.5 Traceability (lineage) to the certifying body for quality control reference materials prepared from CRMs is provided by the certificate or report describing the CRM, the preparation data, and the verification data.

6.6 Traceability to the certifying body for any quality control reference material prepared from process materials or materials with uncertain assay is provided by (*1*) establishing the reference value through a measurement system calibrated with a CRM (*2*) the direct use of a CRM as the quantifying reactant (oxidant/reductant, acid/base, etc.), or (*3*) the use of a quantifying reactant which is traceable to the certifying body. When required, a measurement process tested in traceability exercises conducted by a higher level metrology laboratory shall be used.

6.7 All quality control samples and stock solutions 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, if any, or coded in such a manner as to uniquely identify this same information.

6.8 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 can range from once per batch, once per instrument setup, or once per day per analyst to any frequency consistent with the stability of the measurement system and the risk of performing erroneous determinations between quality control sample analyses.

7.3 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.

7.3.1 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 liability that exists for not covering the entire sequence should be understood and documented.

7.3.2 Even though sample aliquoting by mass or by volume may be included in the analysis of quality control samples, this