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

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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.

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, (*5*) analyst qualification, and (*6*) other general program considerations. Other elements of laboratory quality assurance also impact the laboratory quality control program. These elements

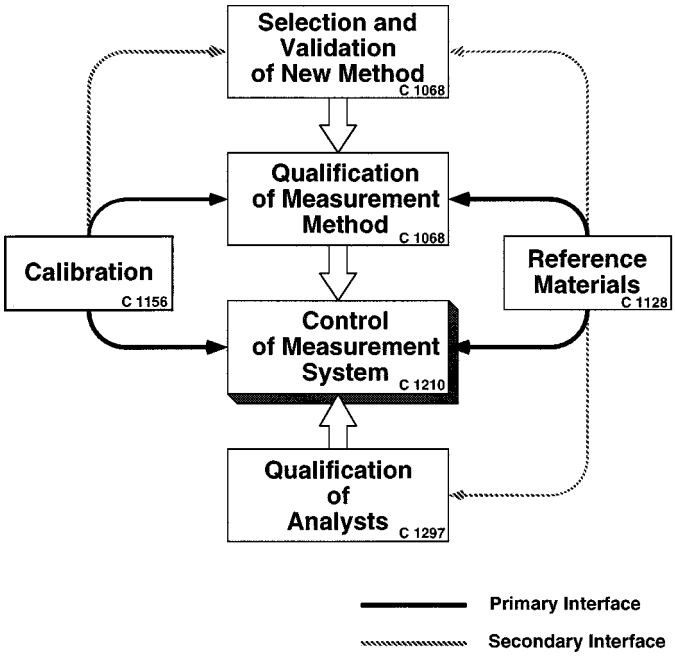

FIG. 1 Quality Assurance of Analytical Laboratory Data

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 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.

7.4 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.

7.5 The data resulting from the analyses of quality control samples should be evaluated against established measurement method control limits immediately (real-time, at-the-bench, by plotting on some form of control chart or by computer assessment) and a determination made as to whether the measurement system is in- or out-of-control.

7.6 Corrective actions for an out-of-control measurement system must 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.

7.6.1 If the measurement system is out-of-control, corrective actions should be initiated and measurement system control should be reestablished before using the measurement system to produce results.

7.6.2 Corrective actions vary with circumstances and systems and may include but not be limited to (*1*) running a number of quality control samples, (*2*) a simple assignment of cause and correction of conditions coupled with the successful analyses of quality control samples, (*3*) assignment of cause that is not readily correctable, that therefore necessitates the generation of a new quality control data base, and (*4*) a complete and detailed evaluation of measurement system performance and suitability to its intended purpose. In all cases, the conditions for reestablishing control need to be defined and control should be reestablished prior to using the measurement system. In general, a single remeasurement of a quality control sample is not adequate to reestablish control.

7.6.3 When a measurement system is out-of-control, an evaluation should be made as to the validity of the results generated since the measurement system was last verified to be in control and the samples should be reanalyzed if possible.

8. Quality Control Data Analysis

8.1 Data from the measurements of quality control samples should be statistically evaluated to assign control limits to measurement systems and to quantify system performance through bias and precision statements. ASTM STP 15D[4] and various references provide guidance on presentation of data and control chart analysis (see Refs **(1-4)**[5] and ANSI/ASQC B1, B2, and B3).

8.1.1 The frequency of measurement system data analysis or the conditions which require data analysis should be documented and described in laboratory procedures.

8.1.2 The data analysis procedures including data transformations (standardizations), used to analyze quality control sample data should be described and documented.

8.1.3 The procedures used for establishing control limits should be described and documented. Control limits, which are based on the statistical analysis of quality control sample results, are generally set at three standard deviations with a warning limit set at two standard deviations. Some situations may dictate control limits based on process or performance criteria separate from those that would arise based solely on the statistical analysis of quality control sample data. An example of a system incorporating both statistical and other control performance criteria limits is described in Ref. **(5)**.

8.2 Data from the measurements of quality control samples should be evaluated to detect problems, patterns, or trends in measurement system performance. Some rules for identification of out-of-control conditions are included in Table 1 (see Ref **(6)**). Other control chart tests and CUSUM procedures are described in various references **(7-11)**.

8.2.1 The frequency or conditions that require analysis should be described in laboratory procedures.

8.2.2 The data analysis procedures used should be defined and documented.

8.2.3 Corrective actions required for measurement systems should be defined and actions taken should be documented.

8.3 Data from the measurement of quality control samples should be evaluated to detect statistically significant differences between analysts, time periods, calibration periods, etc.

8.3.1 The frequency of data analysis or the conditions which require data analysis should be described in laboratory procedures.

8.3.2 The data analysis procedures used and the degree of significance required should be defined and documented.

8.3.3 Corrective actions for significant differences should be defined and documented.

8.4 Data from the measurement of quality control samples should be analyzed to verify measurement system and data base assumptions.

8.4.1 The frequency or condition that requires verification of assumptions should be described in laboratory procedures.

8.4.2 The data analysis procedure used to perform the verification analysis should be defined and documented.

8.4.3 Corrective actions should be defined and documented for situations where assumptions cannot be verified.

8.5 In addition to providing control limits and other parameters that describe or verify measurement system performance, the information that results from quality control samples data

TABLE 1 Some Rules for Identification of Out-of-Control Conditions

Rule Identification	Rule
1	1 point above 3 sigma
2	2 of 3 points above 2 sigma
3	4 of 5 points above 1 sigma
4	8 consecutive points above center line
5	1 point below − 3 sigma
6	2 of 3 points below − 2 sigma
7	4 of 5 points below − 1 sigma
8	8 consecutive points below center line
9	15 points inside ±1 sigma
10	8 points outside ±1 sigma

[4] *Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D,* ASTM, 1976.

[5] The boldface numbers in parentheses refer to the list of references at the end of this guide.

analysis may be used (*1*) to provide measurement system users and customers with confidence levels about measurement system results, (*2*) to test sample analysis results against preestablished limits at various significance levels, (*3*) to routinely verify assumptions of homogeneity with respect to sampling process, (*4*) to qualify and requalify measurement processes, and (*5*) to qualify and requalify analysts.

9. Analyst Qualification

9.1 Training considerations are covered in Guide C 986. Individual laboratory training and qualification practices should be described in laboratory procedures and documented.

9.2 From a quality control viewpoint, new analyst qualification is satisfactorily demonstrated by producing standards data compatible with the existing measurement system data base. The degree of compatibility required, the number of data points required, the measurement conditions, and the statistical procedures used to demonstrate compatibility should be documented and described in laboratory procedures (see Guide C 1297).

9.3 From a measurement system quality control viewpoint, analysts remain qualified or are continually requalified by virtue of satisfactory quality control samples analyses. This condition may require periodic verification, in which case the verification frequency and the statistical tests used should be documented.

9.4 For the initial data base generation for a measurement system, all individuals participating in the data base generation whose data does not differ significantly from the data base should be considered qualified. The minimum number of data points required and the statistical tests should be documented.

10. Measurement System Calibration

10.1 Measurement systems and instruments should be calibrated periodically or with use when such calibration is required to establish, maintain, or normalize response characteristics used for generating measurement results. A calibration program for measurement methods is covered in Guide C 1156.

10.2 For individual measurement systems, calibration may be *with use* or periodic. If the calibration is performed *with use*, then the calibration should be described in the measurement procedure. If the calibration is periodic, then the frequency of calibration or the criteria for recalibration should be specified and the calibration procedure should be described in laboratory procedures and referenced in the measurement procedure.

10.3 If the measurement system calibration is periodic, then the calibration should be verified at a frequency ranging from once per day or batch to any frequency commensurate with the risk of performing erroneous determinations between verifications and the stability of the system. Calibration verification acceptance criteria should be established and documented. Corrective actions should be taken and documented when acceptance performance criteria are not met.

10.4 All measurement system calibrations should be documented. The documentation should include the date, time, analyst, calibration standards used, instrument settings, or system responses before and after calibration, and calibration factors, equations, or curves derived from the systems response to the calibration standards.

10.5 All instruments or measurement systems covered by a periodic calibration program should have calibration labels or equivalent devices affixed to them. The label or other device should indicate or reference, at a minimum, the date the current calibration was performed, the individual that performed the calibration, and the date the next calibration is due.

10.6 If a measurement system calibration procedure cannot be satisfactorily completed, the measurement system is out-of-calibration. If maintenance has been performed which renders the existing calibration questionable, then the measurement system should be taken out of service.

10.7 Quantitative measurement system results should not be reported when a measurement system is out of calibration.

11. Measurement Method Qualification

11.1 Measurement method selection and qualification is addressed in Guide C 1068 which provides guidance from a technical and overall quality assurance point of view.

11.2 The conditions and the minimum number of data points required for data base generation should be established in laboratory procedures in accordance with valid statistical practices.

11.3 The measurement system remains qualified as long as quality control sample analysis data indicates the system is in control or control is lost and then reestablished through documented, defined procedures.

11.3.1 After extended periods without quality control sample analysis activities, the existing quality control data base should be revalidated or a new data base should be generated.

11.3.2 The conditions and time periods for validation of existing data base or the generation of a new data base should be described in laboratory procedures.

12. Measurement System Maintenance

12.1 Any maintenance activities that may have an impact on measurement system performance should be documented and retained. The documentation should include the date, time, the individual that performed the maintenance and a description of the maintenance activities performed. If preventive maintenance is required, then the frequency and the activity should be described in instrument or measurement system procedures.

12.2 Measurement system performance should be verified after any maintenance.

13. Keywords

13.1 calibration; laboratory; measurement; qualification; quality control

REFERENCES

(**1**) Ryan, T. R., *Statistical Methods for Quality Improvement*, John Wiley & Sons, New York, 1989.
(**2**) Bennett, C. A., and Franklin, N. L., *Statistical Analysis in Chemistry and the Chemistry Industry*, John Wiley & Sons, New York, 1954.
(**3**) Deming, W. E., *Quality, Productivity, and Competitive Position*, MIT, Center for Advanced-Engineering Study, Cambridge, MA, 1982.
(**4**) Am. Soc. for Quality Control, *Glossary and Tables for Statistical Quality Control*, Milwaukee, WI, 1983.
(**5**) Bruckner, L. A., "A Measurement Control Program to Include Desired Levels of Precision and Accuracy," *Journal of the Institute of Nuclear Materials Management*, October 1990, pp. 29–34.
(**6**) Western Electric Company, Inc., *Statistical Quality Control Handbook*, Delmar Printing Co., Charlotte, NC, 1989.
(**7**) Nelson, L. S., "The Shewhart Control Chart—Test for Special Causes," *JQT*, October 1984, pp. 237–239.
(**8**) Nelson, L. S., "Interpreting Shewhart $\bar{X}$ Control Charts," *JQT*, April 1984, pp. 114–116.
(**9**) Page, E. S., "Continuous Inspection Schemes," *Biometrika 41*, 1954, pp. 100–115.
(**10**) Lucas, J. M., "Combined Shewhart-CUSUM Quality Control Schemes," *JQT*, April 1982, pp. 51–59.
(**11**) Woodall, William H., "The Design of CUSUM Quality Control Charts," *JQT*, April 1986, pp. 99–102.