



Standard Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry¹

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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:²

- C 1009** Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry
- C 1068** Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry
- C 1128** Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials
- C 1156** Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials
- C 1297** Guide for Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

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

3. Significance and Use

3.1 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.

3.2 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

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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.

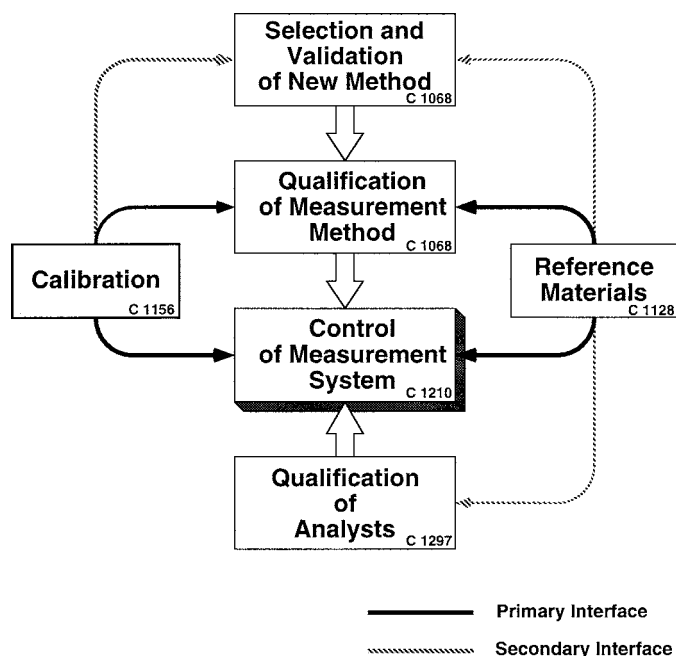


FIG. 1 Quality Assurance of Analytical Laboratory Data

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.

3.3 Although the Standard 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.

4. General Quality Control Program Considerations

4.1 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.

4.2 The analytical laboratories quality control program should be described in laboratory procedures and all measure-

ment system quality control activities should be 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.

4.3 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.

5. Quality Control Samples

5.1 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: Certified Reference Materials (CRMs), Working Reference Materials (WRMs), other reference materials, 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.

5.2 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.

5.3 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 a 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.

5.4 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.

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

6. Analysis of Quality Control Samples

6.1 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.

6.1.1 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.

6.1.2 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.

6.2 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.

6.3 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.

6.3.1 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.

6.3.2 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.

6.4 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.

6.5 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.

6.6 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.

6.6.1 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.

6.6.2 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.

6.6.3 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.

7. Quality Control Data Analysis

7.1 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 7A⁴ 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).

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

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

7.1.3 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).

⁴ *Manual on Presentation of Data and Control Chart Analysis, ASTM 7A, Seventh Edition, 2002*

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

7.2 Data from the measurements of quality control samples should be evaluated to detect problems, patterns, or trends in measurement system performance. Some typical 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).

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

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

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

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

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

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

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

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

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

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

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

7.5 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 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.

8. Analyst Qualification

8.1 Individual laboratory training and qualification practices should be described in laboratory procedures and documented.

8.2 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).

8.3 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.

8.4 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.

9. Measurement System Calibration

9.1 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.

9.2 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.

9.3 If the measurement system calibration is periodic, then operational checks should be performed at a frequency which is a function of the stability of the system. Corrective actions should be taken and documented when acceptance criteria are not met.

9.4 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 equations or curves derived from the systems response to the calibration standards.

9.5 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.

9.6 If a measurement system calibration procedure cannot be satisfactorily completed, or if maintenance that renders the existing calibration questionable has been performed, then the measurement system should be taken out of service. Any measurement system found or suspected to be out of calibration should not be used, pending recalibration.

TABLE 1 Some Typical 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

10. Measurement Method Qualification

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

10.2 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.

10.3 The measurement system remains qualified as long as quality control data indicate the system is in control. When control is lost, it should be reestablished through documented, defined procedures.

10.3.1 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 and associated calibrations repeated.

10.3.2 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.

11. Measurement System Maintenance

11.1 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.

11.2 Measurement system performance should be verified after any maintenance that does not require recalibration.

12. Keywords

12.1 calibration; laboratory; measurement; qualification; quality control

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