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

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ε[1] NOTE—Figure 1 was corrected editorially in February 1997.

1. Scope

1.1 This guide covers the establishment of a quality assurance (QA) program for analytical chemistry laboratories within the nuclear industry. Reference to key elements of ANSI/ISO/ASQC Q9001-1994, Quality Systems, provides guidance to the functional aspects of analytical laboratory operation. When implemented, the recommended practices presented in this guide will provide a comprehensive QA program for the laboratory. The recommended practices are grouped by functions, which are the basic elements of a QA program.

1.2 The basic elements of a QA program appear in the following order:

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Control of Records	10
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2. Referenced Documents

2.1 *ASTM Standards:*

C 986 Guide for Developing Training Programs in the Nuclear Fuel Cycle[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 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry[2]

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

D 1193 Specification for Reagent Water[3]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[4]

E 542 Practice for Calibration of Laboratory Volumetric Apparatus[4]

E 617 Specification for Laboratory Weights and Precision Mass Standards[4]

E 694 Specification for Laboratory Glass Volumetric Apparatus[4]

2.2 *ANSI Standard:*

ANSI/ISO/ASQC Q9001-1994 Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing[5]

2.3 *NIST Standard:*

NIST IR74-461 The Calibration of Small Volumetric Laboratory Glassware (1974)[6]

2.4 *Government Documents:*

10 CFR 830.120 Quality Assurance Requirements[7]

3. Terminology

3.1 *Definitions:*

3.1.1 *laboratory quality assurance, n*—all those planned and systematic actions necessary to provide adequate confidence in each analytical result reported by a laboratory.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *chain of custody, n*—a procedure that documents continuous sample control and security.

3.2.2 *custody, n*—analytical laboratories may prevent tampering and secure possession of a sample by any of the

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

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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

[6] Available from National Institute of Standards and Technology, Gaithersburg, MD 20899.

[7] Available from Superintendent of Documents, P.O. Box 371954, Pittsburgh, PA 15250-7954.

following means: (*1*) the sample is in actual physical possession, (*2*) the sample is in view after being in possession, (*3*) the sample is in a locked area after being in possession, or (*4*) the sample is in a designated secure area.

3.2.3 *laboratory, n*—an organization established to provide analyses of materials for other organizations requiring those analyses. In general, the laboratory exists within a larger, parent organization, although it may exist as an independent organization. It can range from an organization with a small number of employees to a multi-group organization.

3.2.4 *out-of-control, n*—a system that fails to meet preselected performance criteria.

3.2.5 *requester, n*—the person or organization from which samples originate.

3.2.6 *result, n*—a qualitative or quantitative description of a property obtained from an analysis and reported to a requester.

3.2.7 *traveler, n*—a laboratory record used to transmit information and data through the laboratory.

4. Significance and Use

4.1 The mission of an analytical chemistry laboratory is to provide quality analyses on nuclear fuel cycle materials. An analytical chemistry laboratory QA program is comprised of planned and systematic actions needed to provide confidence that this mission is conducted in an acceptable and consistent manner.

4.2 The analytical chemistry laboratories involved in the analysis of nuclear fuel cycle materials are required to use QA in their operations. Regulatory agencies may mandate some form of control requirements for all or a part of a laboratory's operation. When not mandated, laboratory QA programs should be established as a sound and scientific technical practice. This guide provides guidance for establishing a QA program to control those analytical chemistry operations vital to ensuring the quality of chemical analyses.

4.3 Quality assurance programs should be designed to meet the needs of the organization. The quality system is complementary to specific technical requirements. Each laboratory should identify applicable program requirements and use standards to implement a quality program that meets the appropriate requirement. This guide may be used to develop and implement an analytical chemistry laboratory QA program. Other useful implementation standards and documents are listed in Section 2 and Appendix X1.

4.4 The guides for QA in the analytical laboratory within the nuclear fuel cycle have been written to provide guidance for each of the major activities in the laboratory and are displayed in Fig. 1. The applicable standard for each subject is noted in the following sections.

5. Organization

5.1 *Summary*—An organizational structure is the framework within which functional responsibilities, authorities, and interfaces are established. From a QA viewpoint, the subjects included as recommended practices in 5.2 are areas in which administrative controls should be defined. This is particularly true for laboratories having multiple functional groups.

5.2 *Recommended Practices*:

5.2.1 *Organizational Structure*—Each laboratory should define its internal structure and its position within the larger structure when the laboratory exists within a larger organization. For a laboratory having only a few people, defining an internal structure may not be appropriate, but defining its position in a larger organization is relevant.

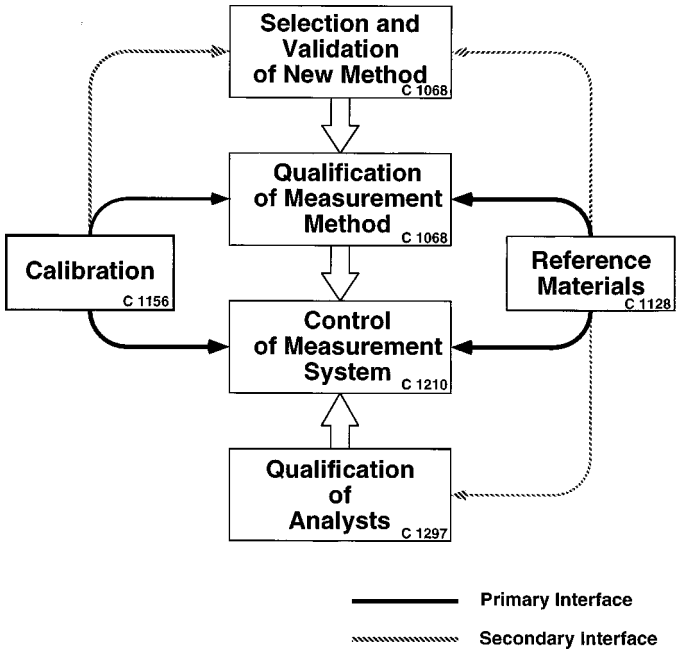

FIG. 1 Quality Assurance of Analytical Laboratory Data

5.2.2 *Functional Responsibilities*—Functional responsibilities should be clearly established for job classifications and functional groups within a laboratory. Functional responsibility defines how work is accomplished in the laboratory in terms of who does it and where it is done. This helps to establish relationships and interfaces within the laboratory.

5.2.3 *Levels of Authority*—Authority to carry out work responsibilities, particularly those involving technical and operational decisions, should be clearly established. Authority includes decision making and approval of actions, extending from the working level up to the manager of the laboratory and beyond if the laboratory is a part of a larger organization. The actions requiring approval and the types of decisions permitted should be established for job classifications at each organizational level.

5.2.4 *Communications*—Methods of communication, both formal and informal, should be clearly established between working groups within a laboratory and, particularly, between the laboratory and outside organizations interacting with the laboratory.

6. Quality Assurance Program

6.1 *Summary*—QA becomes a formal, visible program for a laboratory when a document is prepared and approved that prescribes the QA requirements applicable to operation of the laboratory and that describes how those requirements are implemented.

6.2 *Recommendations*:

6.2.1 *Quality Assurance Program Description*—Once QA requirements have been selected and existing laboratory practices evaluated with respect to those requirements, procedures

should be written that describe how those QA requirements are implemented in laboratory operations. These QA procedures, either added to existing laboratory documents or assembled into a separate laboratory QA manual, define the laboratory QA program.

6.2.2 *Implementation*—Once the QA program documentation has been prepared, reviewed, and approved, new or modified practices should be implemented by training personnel in their use. In addition, personnel should receive general instruction in the overall contents of the QA program.

6.2.3 *Assessment Program*—There should be a procedure established whereby the adequacy of laboratory management and operations is assessed regularly. This procedure should ensure that problems and deficiencies are identified, documented, analyzed, resolved, and followed up. Assessment programs should consist of at least two components: management and independent assessment. Personnel performing assessments must be technically qualified and knowledgeable in the areas assessed.

6.2.3.1 *Management Assessment*—All levels of management should critically assess work under their cognizance and determine whether they are meeting established quality objectives.

6.2.3.2 *Independent Assessment*—QA auditing should foster independent assessment and focus on real issues that affect the organization's performance. Independent assessments should be planned and conducted to measure item and service quality, to measure the adequacy of work performance, and to promote improvement. Independent assessment personnel should have sufficient authority and organizational independence to carry out their responsibility. Independent assessment personnel may act as advisors to senior management to assess quality and process effectiveness.

6.2.3.3 *Reporting*—Assessment procedures should include provisions for reporting the results to those responsible for ensuring correction of the problems identified.

6.2.4 *Quality Improvement*—Processes to detect and prevent quality problems should be established and implemented. The processes should include identification of the causes of problems and work to prevent recurrence. Quality-related information should be reviewed and data analyzed to identify areas of needed improvement, according to the importance of the problem and the work affected.

7. Training and Qualification

7.1 *Summary*:

7.1.1 An important factor affecting all laboratory activities is the training and qualification of those doing the work, including the chemist, technician, and clerical workers. Training can vary from direct, on-the-job training by a more experienced person to a formal program involving both classroom and on-the-job training. The extent of training required depends on the complexity of the work, educational background, demonstrated level of competence, previous work experience, and the requester's requirements. Training should be ongoing and laboratory personnel should be encouraged to attend seminars, courses, and professional meetings as appropriate. Closely associated with training is the concept of qualifying analysts before beginning the analysis of samples.

7.1.2 Qualification includes not only specific training, but also the review and verification of applicable education and experience. A prerequisite for performing all laboratory operations should be the use of adequately trained and qualified people. The requirements for qualification of each person performing analyses should be defined by management (see Fig. 1).

7.2 *Recommendations*:

7.2.1 *Training*—Providing training is a basic management responsibility. The need for training and the type of training used should be a management decision, which is based on the factors mentioned previously. Management should establish a documented training system to ensure that persons are trained adequately and that they remain trained as changes in work practices occur. Such a program should be developed based on job requirements relating to skills, knowledge, and levels of competency required for adequate job performance. Quality assurance training should be included. Guide C 986 provides guidance on developing training programs.

7.2.2 *Qualification*—The concept of qualifying analysts to perform analyses implies that qualification requirements are established for each method. As with training, management is responsible for the qualification process, which can range from a simple practice of stating that an analyst is qualified by reason of education, experience, and job knowledge to a formal system requiring passing tests and routinely demonstrating proficiency in required job skills. Guide C 1297 provides guidance on the qualification of analysts (see Fig. 1).

7.2.3 *Records*—Training and qualification records should be maintained to give visibility to the training program and to show the past and current qualification status of each person trained. The extent of the records required will depend on the scope of the qualification process.

7.2.3.1 The qualification record should consist of at least a single-page form on which the basis of an analyst's qualification is stated, along with those methods for which that analyst is qualified to perform. Management should verify qualification before assigning work.

7.2.3.2 Qualification should be reviewed and updated, if required, on at least a yearly basis.

7.2.3.3 Training and qualification records are QA records, and they should be controlled as prescribed in Section 10.

8. Procedures

8.1 *Summary*:

8.1.1 Analyses are conducted in a planned, systematic, and controlled manner so that the results produced will be valid, that is, based on sound technology. An analysis involves discrete actions taken in a specific order. Any change in an action or in the order without a valid reason probably will produce an unsatisfactory result. To control analyses and avoid errors leading to unsatisfactory results, procedures are written that provide direction for those performing the work. Other purposes for having written procedures are to provide information for training analysts, establish the technical bases of the methods, and document the processes used in the analyses. To be effective and to help provide credibility to the laboratory, procedures must be well written, complete, and correct. Qualification of a procedure (method) may be required. Guide

C 1068 provides guidance on the qualification of measurement methods (see Fig. 1).

8.1.2 Preparation and control of procedures are presented as two functions, although control is not entirely an independent and separate function. Any adequate preparation process has elements of control for assessing the completeness and correctness before a procedure is ready for use. Control as used in this section constitutes those actions taken to ensure that procedures retain their validity as they are used over time.

8.2 *Recommendations*:

8.2.1 *Preparation*—A formal process for writing procedures helps to promote well written, complete, and correct procedures. The following elements should be included in the preparation process:

8.2.1.1 *Format*—Before writing procedures, a format should be established that will help provide consistency across a series of procedures and completeness within each procedure; it will also help simplify the writing process. Formats generally contain such components as purpose or scope, applicability, references, and technical instructions. Technical instructions may include such components as a listing and description of equipment and materials required, applicable safety precautions, tolerances, step-by-step instructions for performing the work, calculations, and expected precision and bias. Instructions for calibration and control charting are sometimes included in the analysis procedures.

8.2.1.2 *Writing*—Writers should, of course, be competent in writing skills. A technical writer, who has a moderate amount of knowledge about the methods involved, can often write adequate procedures from outlines and notes prepared by those with more expertise in the methods. The writing style used should provide clear and concise instructions to avoid confusion and misunderstanding by the users.

8.2.1.3 *Editorial Review*—Someone other than the author should review procedures for conformity to format, consistency in terms and abbreviations, punctuation and spelling, and clarity. An editorial review will help in providing quality documents, which will help enhance the credibility of the laboratory issuing the procedures.

8.2.1.4 *Technical Review*—Procedures should be reviewed for technical adequacy. Such a review would normally be conducted by technically competent persons within the issuing laboratory having no direct responsibility for the procedures. Such a peer review could extend outside of the issuing laboratory to provide a more independent evaluation of technical adequacy.

8.2.1.5 *Approval*—Before procedures are issued and used, management should approve each procedure to certify that the procedures were prepared as prescribed by the established practices of a laboratory. Management approval should also signify that management has accepted responsibility for the adequacy of procedures. Normally, the immediate line manager of the group preparing a procedure makes the approval, with additional upper-level management approval being required as appropriate.

8.2.2 *Control*—Control practices should be established to provide assurance that the adequacy of procedures is not affected adversely with time and use. This includes ensuring that procedures are applied correctly when used. The following actions should be included in the control process:

8.2.2.1 *Distribution*—A controlled distribution should be established to ensure that those requiring procedures will have them where they need them and that all copies are updated when revisions are made. Distribution can be controlled by numbering each copy and establishing a distribution list by number. The distribution list should include the identification of all recipients.

8.2.2.2 *Application*—Someone should be assigned the responsibility of ensuring that each procedure is being applied as intended. That person should be close enough to the work to have knowledge of proper application.

8.2.2.3 *Changes*—Changes in procedures should be controlled to avoid changes that would cause errors in the analyses. All controlled copies of a procedure should be updated when a change is made and approved. Control practices often are established that distinguish, on some basis, between major and minor changes. Although practices often allow minor changes to be made at the work place, such changes should be documented at the time in an established and prescribed manner. Procedure changes shall be reviewed and approved by the same functions/organizations that performed the original reviews and approvals. Differences between minor and major changes should be clearly defined.

9. Laboratory Records

9.1 *Summary*:

9.1.1 Records used to document the work performed in the laboratory serve the following purposes: (*1*) to provide traceability of analytical results back to raw data; (*2*) to provide control of samples as they are processed through the laboratory; and (*3*) to establish who did the work and how it was done. To carry out those purposes, a laboratory record system must provide for five specific activities or functions as follows: (*1*) receive sample information from the requester; (*2*) provide sample identification; (*3*) transmit information and data through the laboratory; (*4*) provide a record of data and information; and (*5*) report results of analyses. Performing those functions usually involves the use of several forms that become laboratory records requiring control actions to prevent loss of data and information. These functions form the basis for the recommended practices that follow. If a computer is used to manage data and information, the five functions should be conducted through the computer program.

9.1.2 The recommended practices are described in the following terms: analysis request, log, traveler, data record, and analytical report. The purposes of each are given, along with recommended distribution and retention time. Purposes can be accomplished using an individual form for each practice or using a combined form that incorporates two or more practices. A combined form should permit all purposes of the individual forms to be fulfilled. The distribution and retention time of a combined form should be governed by the widest distribution and longest retention time represented by the individual forms. A bound laboratory notebook can be used instead of a form for several of the practices. A bound notebook is often used for the data record, for example, using a different

notebook for each analytical method. Notebooks and accumulations of completed forms in loose-leaf notebooks and files must be controlled through distribution lists, retention times, and assigned preparation and custodial responsibilities. The number of record copies is determined by each laboratory.

9.2 *Recommendations*:

9.2.1 *Analysis Request*:

9.2.1.1 *Use*—The two purposes of this function/form are to initiate work in a laboratory and to provide sample information to the laboratory. The following information is typical of that which should be included on the form used: requester, analyses required, date sample submitted, requesters sample identification, and type of material. Each sample submitted should be accompanied by a properly completed form. The requester normally is responsible for initiating this form.

9.2.1.2 *Distribution*—The original should be retained by the laboratory. Copy coverage is optional.

9.2.1.3 *Retention Time*—The original should be kept a specified minimum time period. Copies can be kept at the discretion of the holders.

9.2.2 *Log*:

9.2.2.1 *Use*—The purposes of the log are to provide a source of consecutive serial numbers for the laboratory's sample identification (serial number) and to serve as a record of sample information. The following information is typical of that which should be included in the log for each sample: serial number, requester, requester's sample identification, date sample received, analyses required, type of material, date analyses completed, and sample disposition and date.

9.2.2.2 *Distribution*—The original should be retained by the laboratory.

9.2.2.3 *Retention Time*—Each page of the log should be kept a specified minimum number of years.

9.2.3 *Traveler*:

9.2.3.1 *Use*—The purposes of the traveler are to transmit sample information to the analyst, initiate analyses, and provide analytical results for entry on the analytical report. These purposes may be accomplished by a single form or by a combination of other forms. The following information should be transmitted by this function: serial number, analyses required, results, requester sample identification, and type of material.

9.2.3.2 *Distribution*—If a single-purpose form is used, it should be retained by the laboratory.

9.2.3.3 *Retention Time*—The form can be kept at the discretion of the laboratory.

9.2.4 *Data Record*:

9.2.4.1 *Use*—The purposes of the data record are: (*1*) to provide a record of all data generated during the analyses of samples and standards and a record of the analysts who make the analyses; (*2*) to document activities relating to measurement control; and (*3*) to document unusual or unexpected occurrences taking place during analyses. A data record should be established for each analytical method. The following information should be included in the data record: serial number, requester's sample identification, standard identification, data obtained, results, analyst's signature, date of work, special observations (if any), and a summary of actions taken to regain control when a method goes out of control. A means of associating sample data with formally reported data should be included. All entries in this record system should be in ink and should be legible and neat. There should be no erasures. Incorrect entries should be marked out with a single line through the entry. Marked-out entries should still be legible. Corrected entries should be dated and signed or initialed.

9.2.4.2 *Distribution*—The originals should be retained by the laboratory.

9.2.4.3 *Retention Time*—Data records should be kept for a specified minimum number of years.

9.2.5 *Analytical Report*:

9.2.5.1 *Use*—The purpose of the analytical report is to report analytical results to requesters. The following information should be included in the report as a minimum: serial number, requester's sample identification, and results with reporting uncertainties assigned. The reports should be typed or written in ink. A system of reviewing data and approving reports before issuing should be established. Review should include checking all calculations and transcripts of data for errors. The responsibility for issuing reports after approval should be identified clearly as a part of communications control (see 5.2.4).

9.2.5.2 *Distribution*—The original is sent to the requester and a copy is retained by the laboratory.

9.2.5.3 *Retention Time*—The original is kept at the discretion of the requester. The record copy should be kept by the laboratory for a specified minimum number of years.

10. Control of Records

10.1 *Summary*:

10.1.1 The use and control of records is a key in providing documentary evidence of the technical adequacy of practices. Records provide the direct evidence and support for the technical interpretations, judgments, and decisions regarding the quality of data generated in the laboratory. Records provide the historical evidence needed for future reviews and evaluations, particularly if regulatory or legal questions are raised concerning data generated in the laboratory. Therefore, the control of records should be an integral part of ongoing activities conducted in the laboratory.

10.1.2 There are various ways to control records. The method or system selected must include certain generally accepted features or practices. An effective system of records control must provide records that are identifiable and retrievable. If a computer is used to control records, the computer program used must be verified and validated using established practices for ensuring the quality of computer programs. Recommended practices for an effective control system follow. The record control system is established and documented through the QA program.

10.2 *Recommendations*:

10.2.1 *Identification*—All records to be controlled should be identified. Identification should be by title or type, that is, log, data record, analyst qualification, training records, etc.

10.2.2 *Distribution*—Each type of record included in the record control system should have a distribution plan that identifies recipients of all official copies. The plan should also

identify the individuals or groups responsible for making distribution.

10.2.3 *Storage*—A storage system should be established that provides for safekeeping and physical protection of records. The system should do the following:

10.2.3.1 Identify the individual or the organization responsible for storage,

10.2.3.2 Designate the location and type of storage facilities,

10.2.3.3 Provide a means of protecting records in storage,

10.2.3.4 Provide a method for indexing records, and

10.2.3.5 Provide a method for receiving and handling records while in storage.

10.2.4 *Retrieval*—A method should be included that allows easy retrieval of records. Such a method should be coordinated with the methods used to index and receive records for storage.

10.2.5 *Retention Time*—A retention time should be established for each type of record. The retention times established for various types of records should be coordinated to prevent breaking the traceability of data by a premature discard of one type of record.

11. Control of Procurement

11.1 *Summary*—The quality of procured items and services has an impact on laboratory results. When predetermined control parameters for procurement are established and agreed upon, there is a greater assurance that unknown influences will not affect laboratory results adversely.

11.1.1 Many laboratories are part of a larger organization that has a central procurement organization. That procurement organization is responsible for ensuring that suppliers meet pre-established quality criteria. Quality requirements should be established by the laboratory for items and services used in laboratory operations, with requirements graded based on the use of the item or service. These requirements should be communicated clearly to the procurement organization. Purchase orders and requisitions for selected items should be reviewed to ensure that quality requirements are included. Quality requirements may include certificates of analysis, certificates of conformance, vendor test results, vendor inspection reports, warranties, etc.

11.1.2 Practices that reduce the adverse effect of substandard procured items and services should be identified according to the importance of the item to be purchased, and those practices should be followed. Persons determining the dispositions of substandard items should be authorized and have sufficient knowledge to determine whether the use of substandard material will affect the quality of work. The disposition of nonconforming items should be documented. Dispositions may include the following: use-as-is, repair, return to vendor, etc. Disposition documentation should provide justification for using substandard material when substandard material is used. Some recommended practices for either a procurement organization or laboratory that procures its own items and services that will provide such controls are given in 11.2.

11.2 *Recommendations*:

11.2.1 *Supplier Identification*—Suppliers should be identified based on their predetermined capability to provide materials, services, equipment, and supplies in a timely manner, within a specified cost, and within prescribed control limits. One way for laboratories to establish the criteria is to develop quality and technical specifications. Supplier qualification criteria can be based on historical capability, cost, or a bidding process. Especially when purchasing high-cost equipment, a laboratory should retain the capability to perform quality verification testing both before shipment of the equipment and on-site following installation. A vendor history that provides a documented record evaluating the quality of received items should be established and maintained. Pre-established and agreed-upon hold points allow for the timely performance of such tests.

11.2.1.1 Purchased items and services must meet the expectations of the user. Supplier performance, including sub-tier suppliers, should be re-evaluated periodically. The evaluation should provide data to either support certain suppliers or to question their ability to provide the product satisfactorily. Subcontracting laboratories are usually required to pass a supplier audit and subsequent quality verification tests; the rigor is determined by the requirements for usability of the data produced.

11.2.2 *Procurement Document Control*—Methods of documenting should follow the approach that is taken for controlling other procedures and records.

12. Control of Measuring Equipment and Materials

12.1 *Summary*—The laboratory should maintain adequate measuring equipment and materials to maintain and monitor the performance of analytical instrumentation and methods. Measuring equipment includes all measuring equipment and auxiliary apparatus used to calibrate, measure, gage, test, or inspect. Materials are the reference materials and chemicals necessary for the performance of calibrations and the analytical procedures. Materials are also the items to be tested, that is, the samples. Practices should be followed that ensure and verify that these items are appropriate and acceptable. Control measures may not be required for rulers, tape measures, and other devices if the commercial equipment provides adequate accuracy.

12.2 *Recommendations*:

12.2.1 *Equipment*—Equipment items that are used generally throughout the laboratory, such as analytical balances and volumetric glassware, and that can affect the reliability of measurements should be controlled through a calibration program. The program should identify specific equipment items included, designate calibration standards and the frequency of calibration for each item, identify the calibration status for each on a continuing basis, and control the use of out-of-calibration equipment. Analytical balances should be calibrated using weights meeting the appropriate requirements of Specification E 617. Volumetric glassware should meet the appropriate requirements of Specification E 694, verification of which may be established by the manufacturer's certification. If volumetric glassware requires calibration in the laboratory, the procedures given in Practice E 542 or NIST IR74-461 should be followed.

12.2.2 *Reagents and Standards*—The following requirements are typical of those that should be specified for reagents and standards.

12.2.2.1 *Quality of Chemicals*—Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[8] Water used in the preparation of reagents and in analyses should meet the requirements of Specification D 1193. Requirements for water of special quality should be specified in the appropriate analytical procedure.

12.2.3 *Samples*—Loss of sample integrity can occur from inadequate sampling procedures and from improper control and handling practices once the samples have been taken. Sampling procedures are not included in this guide because they involve technical and statistical considerations that are beyond the scope of this guide. The recommended practices that follow involve controlling and handling samples in a laboratory, beginning with the receipt of a sample and continuing until final disposition is made. Sampling is often the responsibility of the requesters, but in any event sampling responsibility should be established clearly. Proven sampling methods should always be used.

12.2.3.1 *Receipt and Inspection*—Each sample received should be inspected for physical damage of the packaging and container, unexpected condition, and improper identification, all of which can affect integrity adversely. A record that provides all of the sample information needed by the laboratory to perform its work on the sample should accompany each sample (see 9.2.1). Each sample should be labeled clearly by the requester so that it can be distinguished easily from other samples. Labeling should be done in a manner that prevents the loss of identification. If a deficiency is found with a sample, the requester should be contacted and the problem resolved before any work is conducted on the sample.

12.2.3.2 *Handling*—Samples should be handled and stored in the laboratory in ways that do not affect their integrity adversely, which involves preventing contamination from impurities and a change in concentration. If a sample is damaged or its integrity is in any way destroyed, the sample should be disposed of, or, if that is not possible, it should be controlled to prevent its inadvertent use.

12.2.3.3 *Disposition*—A sample should be retained until all analyses have been completed and the results have been accepted by the requester. If a sample is returned to the requester, it should be done in a way that preserves its composition and identification. Disposition actions should be recorded in the record system, giving the date and manner of disposition (see 9.2.2).

12.2.4 *Environment*—The measuring equipment should be maintained in an environment such that the required accuracy is attained and the results are reliable. Storage space, conditions and containers for reference materials, chemicals, and samples should protect materials from deterioration, contamination, and change in concentration.

12.2.4.1 Adequate space and conditions, such as energy sources, lighting, heating, and ventilation, should be provided with instrumentation to monitor the environmental conditions when appropriate.

12.2.4.2 Accommodations should be separated effectively where activities are incompatible.

12.2.4.3 The use and access of all areas affecting the quality of the measuring equipment and materials should be defined and controlled.

12.2.4.4 Materials with special requirements such as avoidance of exposure to light, humidity, and temperature, and shelf-life limitations should be handled and stored appropriately. Proper care and handling of chemicals is also a health and safety concern.

12.2.4.5 Documented procedures should be used to control movement of samples through the laboratory in a manner consistent with their intended use. These procedures should include practices receiving, inspecting, storing, handling, security, and disposition of samples.

12.2.5 *Identification*—Items should be labeled with as much information as is necessary to maintain a complete inventory, to fully identify chemicals and document sample handling procedures.

12.2.5.1 *Measuring Equipment*—These items should be labeled with a name, form of unique identification, and date placed into service. Manufacturers' instructions and maintenance and calibration records should be available in a convenient location.

12.2.5.2 *Chemicals and Reference Materials*—These items should be labeled with the name, concentration, solvent or matrix, date, preparer, expiration date (if appropriate), and any special requirements concerning storage or safety.

12.2.5.3 *Samples*—Sample identification procedures should be developed so that samples are traceable to their origin. A system should be implemented to control and track the location and movement of samples through the laboratory.

12.2.6 *Maintenance*—All equipment should be maintained properly in accordance with documented procedures that may take place either periodically or as needed. Recorded information should include maintenance logs, with descriptions of damage, malfunctions, modifications, or repairs. In those cases in which the laboratory should use measuring equipment and materials outside its permanent control, it should ensure that the appropriate controls are maintained. This is also true when a process should be performed outside a controlled environment.

12.2.7 *Non-Conforming Items*—Any item that has been subjected to overloading or mishandling, that gives suspect results, or has been shown by verification or otherwise defective, should be taken out of service, identified clearly, and stored until it has been repaired and shown by calibration, verification, or testing to perform satisfactorily. Work conducted prior to the discovery of nonconformance shall be evaluated to determine whether the work quality has been compromised. Items that cannot be repaired satisfactorily will be disposed of.

13. Control of Measurements

13.1 *Summary*:

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

13.1.1 Measurements of poor quality will affect programs adversely by causing incorrect decisions, loss of material, rejected product, equipment failure, and operational errors. It is important for analytical chemistry laboratories to control their measurement processes so that reported results will be within required tolerances. Measurement control can vary from simple, manual calibration and control-charting practices to more sophisticated computer programs. The more sophisticated programs usually include provisions for monitoring the qualification of methods and analysts (see Fig. 1).

13.1.2 The recommended practices given in this section define a simplified measurement control program, although the principles involved apply to any measurement control program. If a more sophisticated program involving a computer is required, the computer program used must be verified and validated using established practices for ensuring the quality of computer programs. Also, a statistician should be consulted in designing any sophisticated program.

13.2 *Recommendations*:

13.2.1 *Calibration*—Each calibration procedure written for a method or instrument should specify the standards to be used, any special instructions necessary for obtaining reliable calibration data, the required treatment of data, and the required frequency of calibration. Procedures should be prepared in accordance with Section 8. Guide C 1156 provides guidance for incorporating operational requirements when a calibration procedure is established (see Fig. 1).

13.2.2 *Method Control*—Documented requirements for method control should specify the standard(s) to be used, the required frequency of use, any special instructions necessary for obtaining reliable data, and the required treatment of data. Each laboratory should establish upper and lower limits for acceptance of data. Criteria that indicate when a method is out of control should be given, along with requirements to bring the method back into control. Instructions should also be given for preparing and using control charts when required. Guide C 1210 provides guidance for establishing measurement control over a method or for developing a control program for a laboratory overall measurement system (see Fig. 1).

13.2.3 *Standards*—The calibration and control standards required for a method should be specified in the analytical procedure and in the calibration and control procedures if they are separate from the analytical procedure. Instructions for preparation should be included when appropriate. When possible, standards should be traceable to NIST standards or to other nationally recognized standards. Guide C 1128 provides guidance for the preparation of working reference materials that can be used for standards (see Fig. 1).

13.2.4 *Reporting Significant Numbers*—Consideration should be given for including instructions for reporting significant numbers, based on the capability of the measurement method. Practice E 380 provides guidance for determining significant digits and for rounding values.

13.2.5 *Outlying Observations*—Consideration should be given for including instructions for identifying and treating outlying observations. Also, a statistician should be consulted to select one of the many methods available for treating and identifying suspected outlying observations.

13.2.6 *Tolerances*—Tolerances for all critical parameters and procedure steps made during an analysis should be specified. A tolerance limit can be stated in the procedure, for example, 15 ± 0.1 mL. If a tolerance limit is not stated, then a default tolerance should be specified. For example, unless otherwise specified, values for measurements shall be within ±5 % of the stated value, or "add 20 mL" means that a volume between 19 and 21 mL shall be added.

13.2.7 *Reporting Uncertainties*—Uncertainties should be provided for all reported results. The meaning of the uncertainty value should be clearly defined. For example, an assay value of 0.73 gU/g solution might be accompanied by a statement such as the following: "The analytical method shows no statistically significant bias and has a percent relative standard deviation (% RSD) of 0.2 %." (Guide C 1215 provides guidance on the preparation and interpretation of precision and bias statements.)

13.2.8 *Records*—A record system should be established to document all measurement control actions. Records are QA records, and they should be controlled as prescribed in Section 10.

14. Deficiencies and Corrective Actions

14.1 *Summary*—Deficiencies include failures, defects, errors, deviations from specified requirements, and other conditions considered adverse to management or customer quality objectives. There must be a system established to detect and correct these deficiencies, particularly before they become serious enough to cause significant, unwanted consequences within the operation of a laboratory. Well defined practices are thus required to ensure timely identification and correction. The recommended practices given in 14.2 include identification, evaluation, resolution/disposition (correction), and records. The system established for detecting and correcting deficiencies should be documented through the QA program. Laboratory personnel should be trained in the practices related to deficiencies and corrective actions.

14.2 *Recommendations*:

14.2.1 *Identification*—Deficiencies are often found during the normal performance of work. Other ways are through audits and surveillance activities such as management and independent assessments (see 6.2.3). Laboratories may be subject to outside audits by requesters. If possible, a laboratory should have an internal audit or assessment program that is conducted by a person not directly responsible for work and operations being audited, but who has a good working knowledge of the laboratory's operation. If an internal audit program is used, a reporting system should be established to disseminate results to appropriate management.

14.2.2 *Evaluation*—The system should be established to provide for the technical evaluation of deficiencies. The three components of an evaluation are: (*1*) to determine the cause of deficiencies; (*2*) to identify actions required for correcting deficiencies, including actions that will minimize recurrence; and (*3*) to determine the validity of any analyses that may have been affected by the deficiency. Responsibilities for action should be identified and a schedule for resolution or final disposition established. The assigned actions and schedules should be recorded and reported to responsible technical or

operational management.

14.2.3 *Resolution/Disposition*—After appropriate evaluation, the system should provide for the corrective action needed for problem resolution or the final disposition of defective items. Responsibility for corrective action and a schedule for implementation should be designated. Emphasis should be placed on actions to be taken that will minimize the probability for subsequent failures or deficiencies. The final disposition of an item or condition should be identified, documented, and reported. It is very important that identified changes be reported to the responsible and involved technical and managerial participants. Implementation of corrective action should be verified and documented.

14.2.4 *Records*—All records generated in identifying and correcting deficiencies are QA records, and they should be controlled in accordance with Section 10. Deficiencies found and corrective actions taken during the normal performance of work should be documented.

15. Keywords

15.1 equipment; laboratory; measurement; procedure; procurement; quality assurance; record; training

APPENDIX

(Nonmandatory Information)

X1. LABORATORY QUALITY ASSURANCE AND ANSI/ISO/ASQC GUIDELINES

X1.1 ANSI Standard ANSI/ISO/ASQC Q9001 corresponds to the internationally recognized quality assurance guide 9001 of the International Organization for Standard-ization (ISO). Its 20 elements, the elements of this guide, and regulation 10 CFR 830.120 are shown and matrixed in Table X1.1.

TABLE X1.1 Outline of ANSI/ISO/ASQC Q9001-1994 Elements versus Guide C 1009 and 10 CFR 830.120

Part	ANSI/ISO/ASQC Q9001-94	Part	Guide C 1009	Part	10 CFR 830.120
1	management responsibility	5	organization	(c)(1)(i)	management (program)
2	quality system	6	QA program	(c)(1)(i)	management (program)
3	contract review	11	control of procurement	(c)(2)(iii)	performance (procurement)
4	design control	n/a	not applicable	(c)(2)(ii)	performance (design)
5	document and data control	9	laboratory records	(c)(1)(iv)	management (documents and records)
		10	control records		
6	purchasing	11	control of procurement	(c)(2)(iii)	performance (procurement)
7	control of customer-supplied product	12	control of measuring equipment and materials	(c)(2)(i)	performance (work processes)
8	product identifications and traceability	12	control of measuring equipment and materials	(c)(2)(i)	performance (work processes)
9	process control	8	procedures	(c)(2)(i)	performance (work processes)
10	inspection and testing	13	control of measurement	(c)(2)(iv)	performance (inspection and testing)
11	control of inspecting, measuring, and testing equipment	12	control of measuring equipment and materials	(c)(2)(i)	performance (work processes)
12	inspection and test status	12	control of measuring equipment and materials	(c)(2)(iv)	performance (inspection and testing)
13	control of nonconforming product	12	control of measuring equipment and materials	(c)(1)(iii)	management (quality improvement)
14	corrective and preventive action	14	deficiencies and corrective actions	(c)(1)(iii)	management (quality improvement)
15	handling, storage, packaging preservation, and delivery	8	procedures	(c)(2)(i)	performance (work processes)
16	control of quality records	9	laboratory records	(c)(1)(iv)	management (documents and records)
17	internal quality audits	6	QA program	(c)(3)(i)	assessment (management assessment)
18	training	7	training and qualification	(c)(1)(ii)	management (personnel training and qualification)
19	servicing	8	procedures	(c)(2)(i)	performance (work processes)
20	statistical techniques	13	control of measurements	(c)(2)(i)	performance (work processes)