



Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry¹

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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**, Quality Systems, provides guidance to the functional aspects of analytical laboratory operation. When implemented as recommended, the practices presented in this guide will provide a comprehensive QA program for the laboratory. The practices are grouped by functions, which constitute the basic elements of a laboratory QA program.

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

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2. Referenced Documents

2.1 ASTM Standards:²

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 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

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

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

D 1193 Specification for Reagent Water

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E 542 Practice for Calibration of Laboratory Volumetric Apparatus

E 617 Specification for Laboratory Weights and Precision Mass Standards

E 694 Specification for Laboratory Glass Volumetric Apparatus

2.2 ANSI Standard:

ANSI/ISO/ASQC Q9001 Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing³

2.3 NIST Standard:

NIST IR74-461 The Calibration of Small Volumetric Laboratory Glassware (1974)⁴

2.4 ASME Standard:

ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications⁵

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*—physical possession of a sample by a laboratory.

3.2.3 *laboratory, n*—an organization established to provide analyses of materials.

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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.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

⁵ Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990.

3.2.4 *out-of-control*, *adj*—failing to meet preselected performance criteria.

3.2.5 *requester*, *n*—the person or organization requesting analyses.

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 implement a documented QA program. 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 are 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 XI.

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.

4.5 Although the Standard Guide describes "Recommended Practices" and "Recommendations" and 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 assurance / quality control program should, at minimum, completely and adequately consider and include all elements listed in Section 1 and in the corresponding referenced sections of this guide.

5. 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

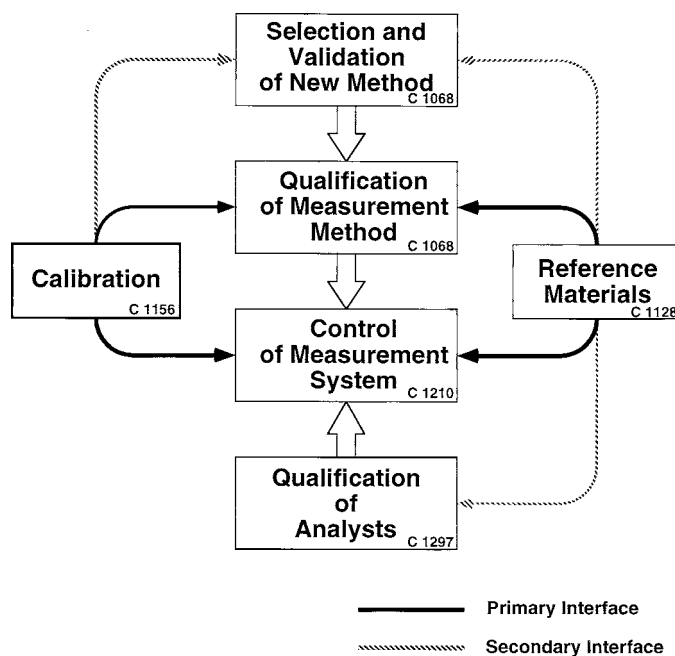


FIG. 1 Quality Assurance of Analytical Laboratory Data

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.

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 that (1) prescribes the QA requirements applicable to operation of the laboratory and (2) describes how those requirements are implemented, is prepared and approved.

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 to 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 an overview of the contents of the QA program and specific instruction in elements applicable to their responsibilities.

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 should 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—Independent assessments should be performed to focus on issues that affect the organization's performance. They 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 chemists, technicians, clerical workers, and other support personnel. 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. Analysts should be qualified 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. All operations should be performed by 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 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.

7.2.2 Qualification—Analysts should be qualified in accordance with qualification requirements 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 identify the basis of the analyst's qualification, and those methods for which the analyst is qualified. 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 should be conducted in a planned, systematic, and controlled manner. Any unauthorized change in the actions or their specified sequence may produce incorrect results. Documented procedures should be implemented to provide direction to those performing the work, provide information for training analysts, and describe the methods to be used and their technical basis. Procedures should be well-written, complete and correct, and should contain criteria for determining whether the prescribed activity has been completed satisfactorily. 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 Measures for the preparation and control of procedures should be established to ensure their completeness and correctness prior to issuance, and 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*—Procedure writers should be competent in technical writing skills, but need not be expert in the analytical methods involved. 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*—Line management should approve each procedure prior to issuance, to certify that the procedure was prepared as prescribed by applicable requirements, and to signify management responsibility for its adequacy. Additional management or customer approvals may also be required.

8.2.2 *Control*—Control practices should be established to provide assurance that the adequacy and effectiveness 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 the correct procedures are available where needed, 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 all recipients of controlled copies.

8.2.2.2 *Application*—Management should ensure that each procedure is being applied as intended.

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 may distinguish between major and minor changes, providing the differences are clearly defined. Where these practices allow minor changes to be made at the work place, the changes should be documented at the time in a prescribed manner, and incorporated in the next revision. Major changes should be reviewed and approved by the same functions that performed the original review and approval.

9. Laboratory Records

9.1 Summary:

9.1.1 Records used to document the work performed in the laboratory provide traceability of analytical results; establish control of samples, and identify how and by whom the work was done. To carry out those purposes, a laboratory record system should 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 should 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 analysis request initiates work in the laboratory and provides sample information. It should identify the requester, submittal date, analyses requested, sample identification, material type and special instructions, as applicable. Each sample submitted should be accompanied by a properly completed analysis request, although the same request may be used for more than one sample. The request should be reviewed by laboratory personnel to ensure that all requirements and other information are clearly understood. Any problems should be resolved with the requester.

9.2.1.2 *Distribution*—The original should be retained by the laboratory and a copy sent to the requester after being logged in.

9.2.2 Log:

9.2.2.1 *Use*—The log provides a source of consecutive serial numbers for the laboratory’s sample identification. For each sample it should identify the serial number, requester, analysis request number, requester’s sample identification, date received, analyses required, type of material, date completed, sample disposition and date.

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

9.2.3 *Traveler:*

9.2.3.1 *Use*—The traveler transmits sample information to the analyst, initiates analyses, and provides sample identification throughout processing. It identifies the sample serial number, analysis request number, and sequence of operations to be performed, and should be signed and dated by the person performing each operation. The traveler may consist of a single form or a combination of forms.

9.2.3.2 *Distribution*—The traveler should be retained by the laboratory.

9.2.4 *Data Record:*

9.2.4.1 *Use*—The data record contains all data generated during the analyses, and documents activities relating to measurement control including unusual or unexpected occurrences during analyses. The data record should maintain traceability between the original sample and the analytical report, and include the sample serial number, requester’s sample identification, data obtained, identification of standards used, analyst’s signature, completion date, special observations (if any) and a summary of actions taken in connection with unusual occurrences.

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

9.2.5 *Analytical Report:*

9.2.5.1 *Use*—The analytical report transmits analytical results to the requester. For each sample it should include the serial number, requester’s sample identification, and analytical results with estimated uncertainties. The report should be typed or written in ink, and should be reviewed for correctness and approved by an authorized person prior to issuance. The responsibility for reviewing, approving and issuing reports should be identified clearly.

9.2.5.2 *Distribution*—The original is sent to the requester and a copy is retained by the laboratory. Additional distribution may also be specified by the requester.

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 An effective records management system should be established as part of the QA program, to ensure that records

are identifiable and retrievable. If a computer program is used it should be verified and validated using established practices.

10.1.3 All records should be in ink, legible and neat, without erasures. Handwritten changes or corrections should be made with a single line through, and signed or initialed and dated by the person making the change. The original information should remain visible after the change.

10.2 *Recommendations:*

10.2.1 *Identification*—All records to be controlled should be identified by title or type, e.g., 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 controlled copies. The plan should also identify the individuals or groups responsible for making distribution.

10.2.3 *Storage*—A storage system that provides for safe-keeping and physical protection of records should be established. 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 that allows easy retrieval of records should be established and coordinated with methods used to index and receive records for storage.

10.2.5 *Retention Time*—A minimum retention time should be established for each type of record. Retention times should be coordinated to ensure that the traceability of data is maintained when records are disposed of. When establishing retention times laboratory needs and customer and regulatory requirements should be considered.

11. Control of Procurement

11.1 *Summary*—The quality of procured items and services may have 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 A procurement organization responsible for ensuring that specified technical, quality, and administrative requirements are imposed on suppliers should be established and identified. Technical and quality requirements should be established by the laboratory for items and services used in laboratory operations, with requirements based on the use of the item or service. These requirements should be communicated clearly to the procurement organization. Procurement documents should be reviewed to ensure that the appropriate technical and quality requirements are included.

11.1.2 Practices for identifying, documenting, and determining the disposition of nonconforming items and services should be implemented. Disposition should be either use-as-is, repair, rework, or reject, and should be documented. The documentation should include the technical justification for use-as-is or repair. Persons determining disposition should be

authorized, and should have sufficient knowledge to determine whether the use of such items and services will affect quality.

11.2 Recommendations:

11.2.1 *Supplier Identification*—Suppliers should be qualified based on their capability to provide products and services that meet applicable quality, technical, and administrative requirements. Qualification criteria should be based on historical performance, a review of the supplier's QA records, a direct evaluation of the supplier's facilities and QA program, or a combination of these. For critical equipment, the laboratory should retain the capability to perform quality verification testing following installation. Pre-established hold points allow for the timely performance of such tests. A supplier history that provides a record of the quality of received items should be established and maintained.

11.2.1.1 Purchased items and services should meet all procurement requirements. Suppliers (including sub-tier suppliers) should be evaluated periodically to verify their continued acceptability. Subcontracting laboratories should be required to pass a QA audit and subsequent quality verification tests, as appropriate.

11.2.2 *Procurement Document Control*—Procurement documents should be controlled using the same practices as are used for other records.

12. Control of Measuring Equipment, Materials and Samples

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 methods. Practices should be followed to ensure and verify that these items are appropriate and acceptable. Control measures may not be required for rulers, tape measures, and similar devices if the commercial equipment provides adequate accuracy.

12.2 Recommendations:

12.2.1 *Equipment*—Equipment 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 should be specified for reagents and standards.

12.2.2.1 *Quality of Chemicals*—Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical

Society where such specifications are available.⁶ 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*—Responsibilities for developing sampling plans and taking samples should be clearly identified. Sampling procedures are outside the scope of this guide. However, once sample have been taken, they should be controlled and handled so as to maintain sample integrity and identification from receipt until final disposition.

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 adversely affect their integrity. This includes preventing contamination from impurities and a change in concentration. If a sample is damaged or its integrity is in any way compromised, 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 Incompatible functions should be separated effectively.

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

⁶ *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 *Anal. 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.

12.2.4.4 Materials with special properties, such as sensitivity to light, humidity, or temperature, should be handled and stored accordingly. Proper care and handling of chemicals is also a health and safety concern. Procedures should be established to ensure that materials having a limited shelf life are not used after their specified expiration dates.

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 for 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, preparation 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 at established, appropriate intervals. The description of any damage, malfunction, modification or repairs should be recorded in maintenance logs.

12.2.7 *Non-Conforming Items*—Any item that has been subjected to overloading or mishandling, gives suspect results, or is suspected of being defective, should be removed from service, checked, repaired if necessary, and recalibrated. Work conducted prior to discovery of the nonconformance should be evaluated to determine whether quality has been compromised. Items that cannot be repaired satisfactorily should be retired.

13. Control of Measurements

13.1 Summary:

13.1.1 Laboratories should 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 should 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 program involving a computer is required, the computer program used should be verified and validated using established practices. 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 for determining 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 Digits*—Consideration should be given for including instructions for reporting significant digits, based on the capability of the measurement method. Practice E 29 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 during an analysis should be specified. A tolerance limit (for example, 15.0 ± 0.1 mL) can be stated in the procedure where applicable, or a default tolerance may be specified (for example, "Unless otherwise specified, values for measurements shall be within $\pm 5\%$ of the stated value.").

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.)

14. Deficiencies and Corrective Actions

14.1 *Summary*—Deficiencies include failures, defects, errors, deviations from specified requirements, and other conditions considered adverse to quality. A system to detect and

correct these deficiencies before they compromise quality, should be established to ensure their timely identification and correction.

14.2 Recommendations:

14.2.1 Identification—Deficiencies may be found either during normal operations, or during audits or assessments. All deficiencies found should be documented and, where warranted, reported to management.

14.2.2 Evaluation—Deficiencies should be technically evaluated. The evaluation should determine probable cause, and verify the validity of any analyses that may have been affected.

14.2.3 Resolution/Disposition—After appropriate evaluation, actions to correct deficiencies and prevent their recurrence should be developed. Responsibilities for these actions should be assigned, and schedules for their completion should be established. The responsibilities and schedules, and the final disposition of deficient items or conditions, should be reported to appropriate management. The implementation of all corrective actions should be verified.

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 **ASME NQA-1** are shown and matrixed in **Table X1.1**.

TABLE X1.1 Outline of ANSI/ISO/ASQC Q9001-1994 Elements versus ASME NQA-1

Part	ANSI/ISO/ASQC Q9001-94	Part	Guide C 1009	Reqmt	ASME NQA-1
1	management responsibility	5	organization	1	organization
2	quality system	6	QA program	2	QA program
3	contract review	9	laboratory records	2	QA program
4	design control	n/a	not applicable	3	design control
5	document and data control	9	laboratory records	4	procurement document control
		10	control records	6	document control
6	purchasing	11	control of procurement	4	procurement document control
				7	control of purchased items and services
7	control of customer-supplied product	12	control of measuring equipment and materials	8	identification and control of items
				12	control of measuring and test equipment
8	product identifications and traceability	12	control of measuring equipment and materials	8	identification and control of items
9	process control	8	procedures	5	procedures, instructions and drawings
				9	control of special processes
				12	control of measuring and test equipment
10	inspection and testing	13	control of measurement	10	inspection
				11	test control
11	control of inspecting, measuring, and testing equipment	12	control of measuring equipment and materials	12	control of measuring and test equipment
12	inspection and test status	12	control of measuring equipment and materials	14	inspection, test and operating status
13	control of nonconforming product	12	control of measuring equipment and materials	15	control of nonconforming items
14	corrective and preventive action	14	deficiencies and corrective actions	16	corrective action
15	handling, storage, packaging preservation, and delivery	8	procedures	13	handling, storage and shipping
16	control of quality records	9	laboratory records	17	quality assurance records
17	internal quality audits	6	QA program	18	audits
18	training	7	training and qualification	2	QA program
19	servicing	8	procedures	5	procedures, instructions and drawings
20	statistical techniques	13	control of measurements	10	inspection
				11	test control

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