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

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1. Scope

1.1 This guide covers the establishment and maintenance of a quality assurance (QA) program for analytical laboratories within the nuclear industry. References to key elements of ASME NQA-1 and ISO 9001 provide guidance to the functional aspects of analytical laboratory operations. 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]

C859 Terminology Relating to Nuclear Materials

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry

C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

D1193 Specification for Reagent Water

D4840 Guide for Sample Chain-of-Custody Procedures

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E178 Practice for Dealing With Outlying Observations

E542 Practice for Calibration of Laboratory Volumetric Apparatus

E617 Specification for Laboratory Weights and Precision Mass Standards

E694 Specification for Laboratory Glass Volumetric Apparatus

E1578 Guide for Laboratory Informatics

2.2 *Other Standards:*

ISO 9001 Quality Management Systems—Requirements

ISO 1042 Laboratory Glassware—One-Mark Volumetric Flasks

ISO/IEC 17020 General Criteria for the Operation of Various Types of Bodies Performing Inspection

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ANSI N15.41 Derivation of Measurement Control Programs—General Principles

ANSI N15.51 Measurement Control Program—Nuclear Materials Analytical Chemistry Laboratory

JCGM 20:2008 International Vocabulary of Metrology—Basic and General Concepts and Associated Terms (VIM)

ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications[3]

3. Terminology

3.1 For definitions of pertinent terms not listed here, see Terminology C859.

[1] This guide is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.08 on Quality Assurance, Statistical Applications, and Reference 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 Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *condition adverse to quality, n*—an all-inclusive term used in reference to any of the following: failures, malfunctions, deficiencies, defective items, and non-conformances. **ASME NQA-1**

3.2.2 *custody, n*—physical possession or control. A sample is under custody if it is in possession or under control so as to prevent tampering or alteration of its characteristics. **D4840**

3.2.3 *customer, n*—the entity requesting analytical services from the laboratory.

3.2.3.1 *Discussion*—A customer may be a person or an organization, and may be internal to the organization of which the laboratory is a part, or may be an external entity.

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

3.2.5 *laboratory quality assurance, n*—all those planned and systematic actions necessary to provide adequate confidence in each analytical result reported by a laboratory (adapted from ASME NQA-1).

3.2.6 *primary measurement standard, n*—measurement standard established using a primary reference measurement procedure, or created as an artifact, chosen by convention. (VIM)

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

3.2.8 *sample, n*—a portion of a process or product matrix that is collected and used to determine the characteristics of that matrix (adapted from Guide D4840).

3.2.9 *sample chain-of-custody, n*—the process whereby a sample is maintained under physical possession or control during its entire life cycle, that is, from collection to disposal. **D4840**

3.2.10 *significant condition adverse to quality*—a condition (see 3.2.1) that, if uncorrected, could have a serious effect on safety or operability. **ASME NQA-1**

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

4. Significance and Use

4.1 The mission of an analytical laboratory is to provide quality analyses on nuclear fuel cycle materials. An analytical 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 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. A documented QA program is also necessary for those laboratory operations required to comply with ASME NQA-1 or ISO/IEC 17025, or the requirements of many accreditation bodies. Even when not mandated, laboratory QA programs should be established as a sound and scientific technical practice. This guide provides guidance for establishing and maintaining a QA program to control those analytical operations vital to ensuring the quality of chemical analyses.

4.3 Quality assurance programs are designed and implemented by organizations to assure that the quality requirements for a product or service will be fulfilled. 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 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.

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 completely and adequately considers and includes all elements listed in Sections 5 – 14 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

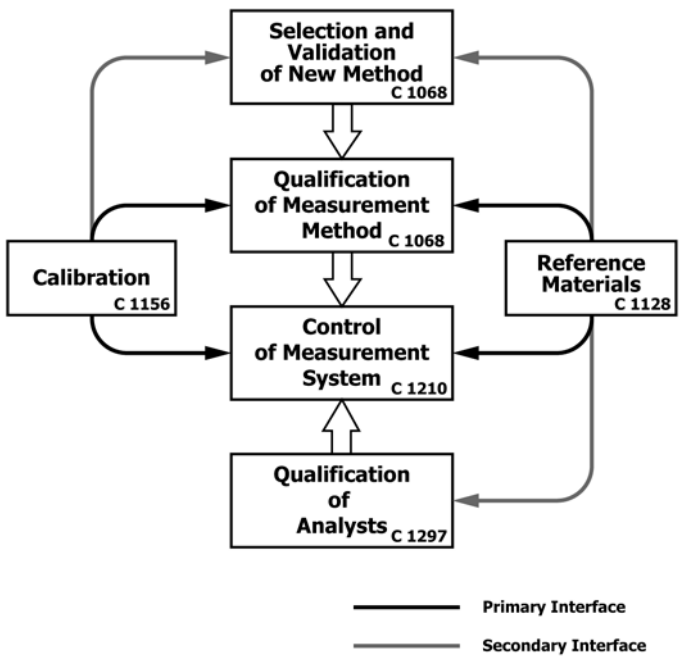

FIG. 1 Quality Assurance of Analytical Laboratory Data

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 small laboratories, defining an internal structure may not be necessary.

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. This document should be reviewed on an established frequency and updated as necessary.

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. Corrective and preventive actions should be identified, evaluated, and resolved as described in Section 14. 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*—Information obtained through QA program implementation, assessments, periodic reviews, corrective and preventive actions should be used to continuously improve the effectiveness of the program.

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 customer'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, or should be working under the direct supervision of a qualified analyst.

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 C1297 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 C1068 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.

8.1.3 Measures should also be established for the preparation and control of instructions or procedures for special or one-analytical processes.

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, terms and definitions, 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 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. 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. Any 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.

8.2.3 *Periodic Review*—Procedures should be reviewed on an established frequency to ensure that they remain effective for their intended use. Changes identified by the periodic review, if any, should be carried out in a timely manner.

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 customer; *(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. the recommended practices that follow. These functions are typically managed electronically through a laboratory information management system (LIMS), with hard copy records generated from the LIMS and controlled as described in Section 10. Additional guidance on the use of a LIMS is found in Guide E1578.

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. and custodial responsibilities. The number of record copies is determined by each laboratory. Electronic notebooks and similar files may be substituted for bound notebooks, provided the users do not have the ability to modify or delete the recorded information once it is entered and transmitted or electronically signed.

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 customer, 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 customer.

NOTE 1—The analysis request may be submitted on chain-of-custody forms. See Guide D4840 for additional information.

9.2.1.2 *Distribution*—The original should be retained by the laboratory and a copy provided to the customer after being logged in. Documentation may be hard copy or electronic, based on established procedures.

9.2.2 *Sample Registration Log:*

9.2.2.1 *Use*—The sample registration log provides a means to register the sample and assign it a unique number for the laboratory's sample identification. For each sample it should identify the unique number, customer, analysis request number, customer's sample identification, date received, analyses required, type of material, date completed, sample disposition and date. The log may be manual or within the LIMS.

9.2.2.2 *Distribution*—The log should be retained by the laboratory. Documentation may be hard copy or electronic, based on established procedures.

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. The traveler may consist of one or more printed forms, or may be incorporated in the LIMS. It identifies the sample registration number, analysis request number, and sequence of operations to be performed, and should be signed and dated, or electronically authenticated, by the person performing each operation.

9.2.3.2 *Distribution*—The traveler should be retained by the laboratory. Documentation may be hard copy or electronic, based on established procedures.

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 unique number, customer'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 customer. For each sample it should include the unique number, customer's sample identification, and analytical results with uncertainties. The report 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 customer and a copy is retained by the laboratory. Additional distribution may also be specified by the customer. Transmission of the report may be hard copy or electronic, based on established procedures.

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, whether in hard copy or electronic form, are identifiable and retrievable for the established retention time.

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, such as log, data record, analyst qualification, or training records.

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 safekeeping 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 may 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. Nonconformance to design requirements dispositioned use-as-is or repair shall be subject to design control measures commensurate with those applied to the original design (ASME NQA-1). 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 E617. Volumetric glassware should meet the appropriate requirements of Specification E694, verification of which may be established by the manufacturer's certification. If volumetric glassware requires calibration in the laboratory, the procedures given in Practice E542 or ISO 1042 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.[4] Water used in the preparation of reagents and in analyses should meet the requirements of Specification D1193. 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 samples have been taken, they should be controlled and handled so as to maintain sample integrity and identification from receipt until final disposition. A formal chain-of-custody program, such as that described in Guide D4840, may 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 customer 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 customer 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, analysis of the sample should be prevented, and the customer should be contacted for further instructions.

NOTE 2—When sample integrity is compromised, disposal of the sample is the preferred action. However, when a replacement sample cannot be obtained, the customer may request the analysis to proceed with the understanding that the results could also be compromised. If the laboratory agrees to proceed with the analysis, the laboratory report should include a data qualifier denoting the compromised nature of the sample and, potentially, of the results.

12.2.3.3 *Disposition*—A sample should be retained until all analyses have been completed and the results have been accepted by the customer. If a sample is returned to the customer, 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 9.2.2.1).

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.

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

[4] *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.

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. Any computer software 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 C1156 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 C1210 provides guidance for establishing measurement control over a method or for developing a control program for a laboratory overall measurement system (see Fig. 1). Additional measurement control program guidance is found in ANSI N15.41 and ANSI N15.51.

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, calibration and control standards should be traceable to national or international standards; for example, in the U.S., standards maintained by the National Institute of Standards and Technology (NIST). Guide C1128 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 E29 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. Additional guidance is found in Practice E178.

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 ±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 g U/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 C1215 provides guidance on the preparation and interpretation of precision and bias statements.)

14. Corrective and Preventive Actions

14.1 *Summary*—A system to detect and address conditions adverse to quality should be established to ensure their timely identification and resolution. The system should include both preventive actions (that is, those identified prior to impacting customer results) and corrective actions (taken to correct problems that have occurred).

14.2 *Recommendations:*

14.2.1 *Identification*—Conditions adverse to quality may be found either during normal operations, during audits or assessments, or as the result of customer complaints. Such conditions, when identified, should be reported to management and corrected as soon as possible.

14.2.2 *Evaluation*—Conditions adverse to quality should be technically evaluated. For significant conditions adverse to quality, the evaluation should determine probable cause, consider the extent of the condition in related areas, and identify actions to prevent recurrence. The evaluation should also verify the validity of any analyses that may have been affected.

14.2.3 *Resolution/Disposition*—After appropriate evaluation, actions to correct conditions adverse to quality 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 GUIDELINES IN ASME NQA-1 AND ISO/IEC 17025

X1.1 The elements of ISO/IEC 17025, this guide, ASME NQA-1, and ISO 9001 are shown and matrixed in Table X1.1.

TABLE X1.1 Cross-Reference of ASME NQA-1, Guide C1009, ISO/IEC 17025, and ISO 9001

Requirement	ASME NQA-1	Part	Guide C1009	Clause	ISO/IEC 17025	Clause	ISO 9001
1	Organization	5	Organization	4.1	Organization	5	Management Responsibility
2	QA program	6	QA program	4.2	Management System	4	Quality Management System
3	Design Control	n/a	Not Applicable	n/a	Not Applicable	7.3	Design and Development
4	Procurement Document Control	11	Control of Procurement	4.6	Purchasing Services and Supplies	7.4	Purchasing
5	Instructions, Procedures, and Drawings	8	Procedures	5.4	Test and Calibration Methods and Method Validation	7.5	Production and Service Provision
6	Document Control	8	Procedures	4.3	Document Control	4.2	Documentation Requirements
7	Control of Purchased Items and Services	11	Control of Procurement	4.6	Purchasing Services and Supplies	7.4	Purchasing
8	Identification and Control of Items	12	Control of Measuring Equipment, Materials, and Samples	5.8	Handling of Test and Calibration Items	7.1	Planning of Product Realization
9	Control of Special Processes	n/a	Not Applicable	n/a	Not Applicable	n/a	Not Applicable
10	Inspection	12	Control of Measuring Equipment, Materials, and Samples	n/a	Not Applicable	7.1	Planning Product of Realization
11	Test Control	n/a	Not Applicable	n/a	Not Applicable	n/a	Not Applicable
12	Control of Measuring Equipment, Materials, and Samples	12	Control of Measuring Equipment, Materials, and Samples	5.5	Equipment	7.6	Control of Monitoring and Measuring Equipment
13	Handling, Storage, and Shipping	12	Control of Measuring Equipment, Materials, and Samples	5.4 5.8	Test and Calibration Methods and Method Validation Handling of Test and Calibration Items	7.5	Production and Service Provision
14	Inspection, Test, and Operating Status	12	Control of Measuring Equipment, Materials, and Samples	5.5	Equipment	7.5	Production and Service Provision
15	Control of Nonconforming Items	11 12	Control of Procurement Control of Measuring Equipment, Materials, and Samples	n/a 4.9	Not Applicable Control of Nonconforming Testing and/or Calibration Work	n/a 8.3	Not Applicable Control of Nonconforming Product
16	Corrective Action	14	Corrective and Preventive Actions	4.11	Corrective Action	8.5.2	Corrective Action
17	Quality Assurance Records	9	Laboratory Records	4.13	Control of Records	4.2.4	Control of Records
18	Audits	6	QA Program	4.11 4.14	Corrective Action Internal Audits	8.5.2 8.2.2	Corrective Action Internal Audit
n/a	Not Applicable	n/a	Not Applicable	4.4	Review of Requests, Tenders, and Contracts	7.2	Customer Related Processes
n/a	Not Applicable	n/a	Not Applicable	4.5	Subcontracting of Tests and Calibrations	n/a	Not Applicable
n/a	Not Applicable	9	Laboratory Records	4.7	Service to the Customer	7.5	Production and Service Provision
n/a	Not Applicable	14	Corrective and Preventive Actions	4.8	Complaints	8.5.2	Corrective Action
n/a	Not Applicable	6	QA Program	4.10	Improvement	8.5	Improvement
n/a	Not Applicable	14	Corrective and Preventive Actions	4.12	Preventive Action	8.5.3	Preventive Action
n/a	Not Applicable	6	QA Program	4.15	Management Reviews	5.6	Management Review
n/a	Not Applicable	7	Training and Qualification	5.2	Personnel	6.2	Human Resources
n/a	Not Applicable	12	Control of Measuring Equipment, Materials, and Samples	5.3	Accommodation and Environmental Conditions	6.4	Work Environment
n/a	Not Applicable	12 13	Control of Measuring Equipment, Materials, and Samples Control of Measurements	5.6	Measurement Traceability	7.6	Control of Monitoring and Measuring Equipment
n/a	Not Applicable	n/a	Not Applicable	5.7	Sampling	n/a	Not Applicable
n/a	Not Applicable	13	Control of Measurements	5.9	Assuring the Quality of Test and Calibration Results	7.6	Control of Monitoring and Measuring Equipment
n/a	Not Applicable	9	Laboratory Records	5.10	Reporting the Results	8.2	Monitoring and Measurement