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Standard Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry[1]

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ε[1] NOTE—Editorial changes were made throughout in March 1997.

[1] This guide is under the jurisdiction of ASTM Committee C-26 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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1. Scope

1.1 This guide provides guidance for selecting, validating, and qualifying measurement methods when qualification is required for a specific program. The recommended practices presented in this guide provide a major part of a quality assurance program for the laboratory data (see Fig. 1). Qualification helps to assure that the data produced will meet established requirements.

1.2 The activities intended to assure the quality of analytical laboratory measurement data are diagrammed in Fig. 1. Discussion and guidance related to some of these activities appear in the following sections:

	Section
Selection of Measurement Methods	5
Validation of Measurement Methods	6
Qualification of Measurement Methods	7
Control	8
Personnel Qualification	9

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

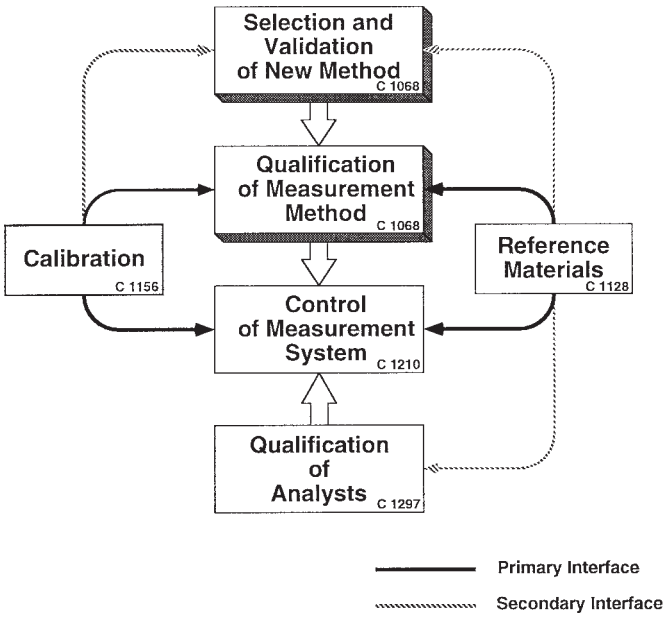

FIG. 1 Quality Assurance of Analytical Laboratory Data

C 100986 Guide for Developing Training Programs in Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Fuel Cycle Industry[2]

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

C 1128 Guide 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials[2]

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials[2]

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry[2]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *qualification*—a formal process to provide a desired level of confidence that measurement methods used will produce data suitable for their intended use. The methods must meet established criteria prior to use and must be used under conditions established for qualifications.

4. Significance and Use

4.1 Because of concerns for safety and the protection of nuclear materials from theft, stringent specifications are placed on chemical processes and the chemical and physical properties of nuclear materials. Strict requirements for the control and accountability of nuclear materials are imposed on the users of those materials. Therefore, when analyses are made by a laboratory to support a project such as the fabrication of nuclear fuel materials, various performance requirements may be imposed on the laboratory. One such requirement is often the use of qualified methods. Their use gives greater assurance that the data produced will be satisfactory for the intended use of those data. A qualified method will help assure that the data produced will be comparable to data produced by the same qualified method in other laboratories.

4.2 This guide provides guidance for qualifying measurement methods and for maintaining qualification. Even though all practices would be used for most qualification programs, there may be situations in which only a selected portion would be required. Care should be taken, however, that the effectiveness of qualification is not reduced when applying these practices selectively. The recommended practices in this guide are generic; based on these practices, specific actions should be developed to establish a qualification program.

5. Selection of Measurement Methods

5.1 *General*:

5.1.1 Before qualifying a method for a specific application, there should be assurance that the method has been properly selected for that application. The guidance given in this section can be used to assess the adequacy of the method's application. The guidance can also be used to select a new method when a new measurement capability is required within a laboratory.

5.1.2 Measurement methods generally can be classified as one of three types as follows:

5.1.2.1 Those published as national or international consensus standards,

5.1.2.2 Those established as acceptable for a specific application based on long-term and wide usage, and

5.1.2.3 Those having limited use, for example, those used only by a few laboratories or those that are relatively new.

5.1.3 For some applications, there is a choice available of two or more acceptable methods. In those cases, one method is usually recognized as the reference method, particularly if it is a published standard or if it is capable of producing the least bias and best precision.

5.1.4 When choosing a method, the four technical factors affecting its application should be evaluated as follows:

5.1.4.1 Is the method actually capable of providing the specific analysis required?

5.1.4.2 Is the method free of adverse effects from the matrix of the particular material requiring analysis?

5.1.4.3 Does the method have the sensitivity and range to cover adequately the concentration levels that will be encountered?

5.1.4.4 Is the method capable of producing data that will meet established bias and precision requirements?

5.1.4.5 Each question must be answered in the affirmative for a method to be acceptable. Although cost is not recognized as a criterion affecting selection, it could be of concern.

5.1.5 The

5.1.4 The selection of a method should be based on the criteria in 5.2. In situations where a reference method and one or more acceptable methods are available, there should be no technical restrictions placed on which method is used.

5.2 *Recommended Practices for Method Selection*:

5.2.1 *Technical Basis*— The method should be based on sound technology. This means that proven laboratory and instrumental techniques are used in ways recognized and accepted by the community of users.

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

5.2.2 *Interferences*— The method should not be adversely affected by components in the matrix of the material to be analyzed. Knowledge about the method's limitations and about the composition of the material should be used to determine if the analysis will be affected by interferences. Other potential interferences such as environmental or electrical/electronic conditions should be considered in the selection process.

5.2.3 *Range*—The method should be capable of responding adequately across the range of concentration levels that will be encountered for the constituent to be measured. This requirement is most often of concern for methods used to measure impurities in materials since impurity concentrations may fluctuate to a greater extent than other constituents. It is important that the measurement technique used discriminates adequately between concentration levels encountered. The lowest concentration level that can be measured reliably should be clearly established (detection limit).

5.2.4 *Reliability of Method*—The method must be capable of producing data that will meet the bias and precision requirements established for the required analysis under the expected conditions of use. The requirements are usually established by the user of the data and they should be based on the concentration levels of the constituents to be measured and on specification limits set for the constituents.

6. Validation of Measurement Methods

6.1 There are occasions when it is desirable to investigate the applicability of a method to a particular use. This may be the case when the method has had limited use or it is being considered for a new or unique application. To provide some confidence that a qualification effort would be successful, it may be desirable to validate the application of the method. Validation is not a mandatory step in the selection and qualification process, but it can prevent wasted effort from attempts to qualify inadequate methods.

6.2 Validation of a method is usually done by an analyst under controlled conditions. Basically, validation involves investigating any or all of the selection criteria in 5.2. The intent is to define method capability and to determine if the method can be properly applied as intended. If modification of the method is required for it to be applicable, validation will provide the technical information needed for modification. Validation also provides the experience and information to write a detailed procedure if necessary. The result of the validation process will be either the rejection of a proposed method or confidence that it is acceptable for use as intended.

7. Qualification of Measurement Methods

7.1 *General*:

7.1.1 Although a method is selected based on the criteria in 5.2 of this guide, there is no assurance that a laboratory can actually obtain the performance expected from the method. In addition, there may not be sufficient assurance that the method is in fact adequate for its intended use. To provide those assurances, demonstration is included in the qualification process.

7.1.2 Qualification requires having a laboratory demonstrate that a method can produce acceptable data under specified conditions of qualification. Demonstration must be done under actual operating conditions and not under ideal test conditions. A specified material is analyzed to produce a specified amount of data. These data are evaluated by the person or organization that is responsible for approving qualification. The procedure established for demonstration should include provisions for handling failures in the demonstration and for repeating the demonstration should the method not be used for a specified period of time. Demonstration could also include producing other evidence such as appropriate literature references that the method is in fact applicable to the material to be analyzed.

7.2 *Recommended Practices*:

7.2.1 *Procedures*—The use of a method to make a laboratory measurement involves taking discrete actions in a specific order. Any change in an action or in the order may produce unsatisfactory data. To minimize potential problems, written, stepwise procedures should be provided within the methods. It is important that procedures are well-written, complete, and correct. They should receive technical and editorial reviews, and should be approved by appropriate management. Approval by the user of the data to be produced also may be required. Procedures prepared in accordance with Guide C 1009 will meet these criteria.

7.2.2 *Method Performance Requirements* —To provide acceptable data, the method must be capable of meeting performance requirements for bias, precision, and range. Before a laboratory demonstrates its capability, these requirements should be clearly established (this should be done even before a method is selected for use; see 5.2). Specifications established for a process or material are the primary source of information on which the performance requirements are based. The performance requirements should be used to establish conditions required for qualification. Such conditions may require a statistically designed experiment to allow for other sources of variability such as the number of analysts or instruments, or both, as well as the concentration range of interest.

7.2.3 *Test Materials*— The material or materials that will be used for demonstration should be specified. The test materials should be as similar as possible to the material that will be analyzed. When possible, the composition or properties of test materials should be defined by measurements traceable to certified reference materials. See Guide C 1128.

7.2.3.1 *Major Constituents*—When the method is to be used to determine a major constituent (for example, uranium in uranium oxide), a single test material may be specified. The concentration of the constituent in this test material should approximate the specification value established for the constituent in the material to be analyzed. The concentration value of the test material should not be given to the laboratory; only those responsible for evaluating the data and approving qualification should know the value

(see 7.2.4.4). The calibration standard should be specified. See Guide C 1156.

7.2.3.2 *Impurities*—When the method is to be used to determine an impurity, at least two test materials should be specified. One should serve as a test standard, meeting the same criteria given in 7.2.3.1 of this guide. Another should be used to demonstrate the detection limit of the method. When possible, the detection limit should be sufficiently below the specification limit to determine whether or not the concentration level of the impurity is within specification. Both test materials would serve to demonstrate the range of the method. When a method requires one or more standards for calibration, the calibration standard(s) that will be used should be specified. See Guide C 1156.

7.2.4 *Qualification Requirements* —A procedure to be followed during demonstration should be established. The procedure that will govern qualification should include the following criteria:

7.2.4.1 *Bias*—A statistical sampling and hypothesis testing plan should be developed such that the risk of qualifying a method is acceptably small when the true bias exceeds the stated requirement and the risk of not qualifying the method is acceptably small when the true bias is zero. The plan would include the number of analyses of a test standard required to control these risks at acceptably small levels and would express the requirement for qualifying based on bias as a statistical hypothesis testing procedure.

7.2.4.2 *Precision*—The precision requirement should state a value of the true standard deviation (larger than zero) that is both desirable and practical to maintain together with an upper limit, above which the true standard deviation would be unacceptable. A statistical sampling and hypothesis testing plan should then be developed such that: the risk of qualifying a method is acceptably small when the true standard deviation exceeds the specified upper limit, and the risk of not qualifying the method is acceptably small when the true standard deviation is less than or equal to the desired value. The plan would include the number of analyses of a test material required to control these risks at acceptably small levels and would express the requirement for qualifying based on precision as a statistical hypothesis testing procedure.

7.2.4.3 *Range*—A requirement, such as the following, should be stated when range is of concern: “Data obtained from the analysis of test materials, including calibration standards, shall be submitted to demonstrate the range of the method under the specific conditions of qualification. The calibration of the method should cover the expected range of concentration.”

7.2.4.4 *Reporting Data*— The agency to whom demonstration data will be submitted should be specified. The agency could be a person or group within or outside of the laboratory, depending upon the program or project requiring qualified methods. The person or persons evaluating the data should be technically competent to do so.

7.2.4.5 *Failure*—Criteria should be established to govern the situation when a laboratory fails one or more of the demonstrations. Based on statistical evaluation, consideration should be given to specifying the following: the number of additional tries that will be allowed and whether or not increased number of analyses will be required for each new try.

7.2.4.6 *Requalification*— Criteria for requalification should be established. For example, requalification may be required if a change is made in a method or if a specific period of time elapsed during which a method was not used and no control standards were analyzed.

7.2.5 *Documentation*— The capability to substantiate the qualification of a method through appropriate records should be available. Actions taken and data generated with each step of qualification should be documented and those records should be easily retrievable.

7.2.5.1 *Laboratory Records*—The records used by the laboratory to record analysis and control data should be based on the appropriate parts of 9.2 in Guide C 1009.

7.2.5.2 *Control of Records*—Records used to document qualification activities should be controlled in accordance with 10.2 of Guide C 1009.

7.2.5.3 *Approval Records*— The agency responsible for approving qualification should document actions that it takes in evaluating and approving qualification (see 7.2.4.4). The records generated should be controlled in accordance with 10.2 of Guide C 1009.

8. Control

8.1 *Measurement Control*:

8.1.1 Control of the measurement system is a major activity in assuring the quality of analytical data and control should be established as described in Guide C 1009. A control system will help to ensure that analytical results are generated by methods that are in control, and under such conditions, those methods remain qualified. This section provides a general description which shows the key points that relate to this guide.

8.1.2 Once a method has been selected and the laboratory has successfully qualified the method, the laboratory is ready to use the method for analysis. However, there should be a continuing effort to assure the acceptability of the data produced as the method is used over time. Acceptability can be assured with the use of a control system that is applied each time the method is used to control the measurements made (Fig. 1). Control can vary from a simple manual calibration and control-charting system to a sophisticated computer program (see Guide C 1210).

8.1.3 Requirements should be specified to prevent one analyst from calibrating a method and producing control data while a second analyst analyzes the samples. When necessary, there should be a specified process for a partially completed analysis to be continued by an incoming analyst. This process should assure that measurement control is maintained.

8.2 *Change Control*— Once a method has been published as a standard or has been written, reviewed, and approved within a laboratory, changes in that method, particularly in the stepwise procedure, should be controlled to avoid introducing errors.

Uncontrolled changes made in a qualified method could become a reason to disqualify that method. A planned, systematic, and controlled system to make changes in a method should be established so that valid and necessary changes can be made while the method is in use. If changes are required in qualified methods, changes should be made in accordance with 8.2.2 of Guide C 1009. Significant changes should be evaluated to determine if requalification of the method is required.

9. Personnel Qualification

9.1 ~~The a~~Analysts producing data ~~during the qualification period~~ should be qualified in accordance with Guide C 1009. Guide C 1297 outlines steps in the qualification of laboratory analysts.

9.2 The adequacy of the laboratory's existing practices for selecting, training, and qualifying analysts should be evaluated. ~~Guide C 986, which provides criteria for developing training programs, should be used as a reference when evaluating the adequacy of a laboratory's existing training program.~~ The results of the evaluation should be included in the criteria used to qualify methods.

10. Keywords

10.1 control; laboratory; measurement(s); personnel; qualification; validation