Designation: C 1068 – 96ε1

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

This standard is issued under the fixed designation C 1068; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

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.*

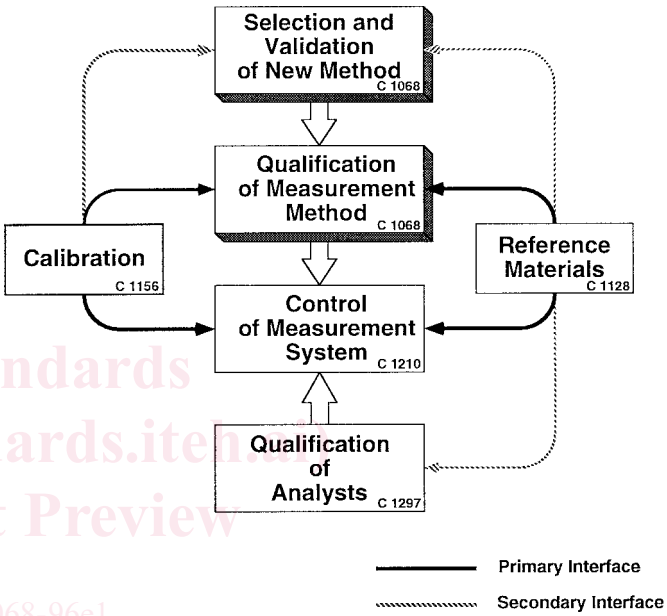

FIG. 1 Quality Assurance of Analytical Laboratory Data

2. Referenced Documents

2.1 *ASTM Standards:*

C 986 Guide for Developing Training Programs in the Nuclear Fuel Cycle[2]

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

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

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

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

C 1297 Guide for 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

[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 Applications.

Current edition approved Dec. 10, 1996. Published January 1997. Originally published as C 1068 – 86. Last previous edition C 1068 – 91.

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

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 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*:

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.

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.