Designation: C 1297 – 03

Standard Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials[1]

This standard is issued under the fixed designation C 1297; 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.

1. Scope

1.1 This guide covers the qualification of analysts to perform chemical analysis or physical measurements of nuclear fuel cycle materials. The guidance is general in that it is applicable to all analytical methods, but must be applied method by method. Also, the guidance is general in that it may be applied to initial qualification or requalification.

1.2 The guidance is provided in the following sections:

	Section
Qualification Considerations	4
Demonstration Process	5
Statistical Tests	6

1.3 This standard does not apply to maintaining qualification during routine use of a method. Maintaining qualification is included in Guide C 1210.

1.4 *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:*

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

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry[2]

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

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

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

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

2.2 *ISO Standard:*

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials[3]

3. Significance and Use

3.1 This is one of a series of guides designed to provide guidance for implementing activities that meet the requirements of a sound laboratory quality assurance program. The first of these, Guide C 1009, is an umbrella guide that provides general criteria for ensuring the quality of analytical laboratory data. Other guides provide expanded criteria in various areas affecting quality, producing a comprehensive set of criteria for controlling data quality. The approach to ensuring the quality of analytical measurements described in these guides is depicted in Fig. 1.

3.2 The training and qualification of analysts is one of the elements of laboratory quality assurance presented in Guide C 1009, which provides some general criteria regarding qualification. This guide expands on those criteria to provide more comprehensive guidance for qualifying analysts. As indicated in Guide C 1009, the qualification process can vary in approach; this guide provides one such approach.

3.3 This guide describes an approach to analyst qualification that is designed to be used in conjunction with a rigorous program for the qualification and control of the analytical measurement system. This requires an existing data base which defines the characteristics (precision and bias) of the system in routine use. The initial development of this data base is described in Guide C 1068. The process described here is intended only to qualify analysts when such a data base exists and the method is in control.

3.4 The qualification activities described in this guide assume that the analyst is already proficient in general laboratory operations. The training or other activities that developed this proficiency are not covered in this guide.

3.5 This guide describes a basic approach and principles for the qualification of laboratory analysts. Users are cautioned to

[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.

Current edition approved July 10, 2003. Published August 2003. Origianlly approved in 1995 as C 1297–95. Last previous edition approved in 1995 as C 1297.

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

[3] Available form American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

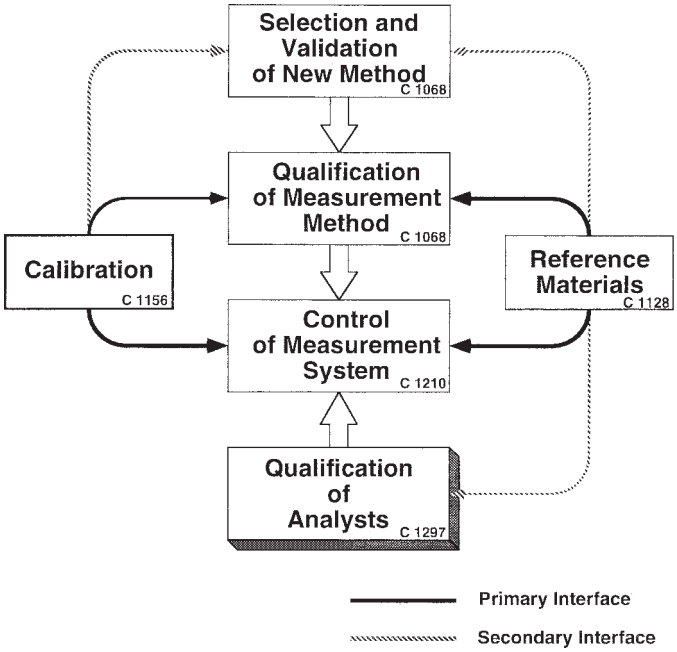

FIG. 1 Quality Assurance of Analytical Laboratory Data

ensure that the qualification program implemented meets the needs and requirements of their laboratory.

4. Qualification Considerations

4.1 When a qualification program is being established, consideration should be given to analyst selection criteria, the training program, and practical demonstration. The criteria that govern when qualification is achieved should be documented along with methods for determining the knowledge and skill of the analyst.

4.1.1 Analyst selection should be based on established criteria that are related to the complexity of the method that analysts are expected to perform. Criteria should include the minimum education required, any prerequisite training, and the overall experience required. The selection criteria should be defined and documented.

4.1.2 The method-specific analyst training program should be an established program with a prescribed training procedure. Some mechanism such as an oral or written test should be used to allow an analyst to demonstrate knowledge and understanding of the chemical, physical, instrumental, and mathematical concepts used to execute the method. It is advisable to monitor progress during training to ensure that the analyst has a reasonable chance of passing the qualification test.

4.1.3 The practical demonstration of the analyst's ability to generate results with the analytical method should be compared to established criteria. The comparison criteria should be defined and documented.

NOTE 1—The qualification of analysts, like many other laboratory processes, has the potential for undetected errors. There are two types of errors that occur. One is to fail an individual who should have been determined to be qualified. The other error is to pass an individual who should not have been determined to be qualified. The potential for these errors to occur and the potential consequences to the laboratory should be carefully considered when determining the laboratory's qualification methodology. A statistical approach includes choosing the significance level at which the determination of qualification will be made. This produces a quantitative value of the two possible risks. This is described further in Appendix X1.

5. Demonstration Process

5.1 The suggested approach to practical demonstration for analyst qualification that is described in the remainder of this guide involves a comparison of the performance of the analyst with the performance of all qualified analysts on a particular analytical method. The performance is measured by the analysis of reference materials (see ISO Guide 30) and comparison of the results to the data base for the analytical method. This approach requires a data base that describes method performance. The comparison described in this guide is statistical in nature and therefore statisticians should be involved early on in the process of defining qualification. Other types of comparisons may serve to qualify equally well; however, such comparisons are not addressed in this guide. If used, they should be defined and documented.

5.2 The data base for a given analytical method is generated by all qualified analysts who run reference material samples on an established schedule or frequency. The data base is used to establish the bias and precision of the method as routinely used in the laboratory. The data base is established through a measurement control program as presented in Guide C 1210. For a new method, a data base should be established according to Guide C 1068 and the analyst should be qualified against that data base.

5.3 If changes in a method occur or changes in the execution of a method occur that render the existing data base representation of the method questionable, the qualification of analysts should be suspended until the data base is verified or a new data base is generated. When a new data base is generated, the old data base should be archived (retained for future reference) as a part of the documentation of the laboratory quality assurance program.

5.4 A predetermined number of reference material samples should be selected for the analyst after training has been completed. The analyst should analyze the samples over several days, and not in a single session, to simulate more realistically the conditions under which the data base was established.

5.5 Since the samples may be at different concentration levels, the analyst's demonstration results are normalized using established parameters from the existing data base for each control standard. The normalized data are used to test for conformity to the data base. Statistical tests for the statistical distribution (normality) as well as precision and bias are suggested in Section 6. These terms are described in Guide C 1215.

5.6 If the results of all three tests are satisfactory, the analyst is qualified on that method. If the analyst does not qualify, retraining should be required before being allowed to retest for qualification. The analyst should be given a different set of reference material samples each time retesting is allowed to maintain the independence of successive tries. That will allow

the same statistical tests to be used on each set of results. See Fig. 2 for a schematic of the qualification process.

6. Statistical Tests

6.1 There are a number of statistical procedures appropriate for performing the statistical tests on the analyst's demonstration data set to determine qualification. The procedures detailed in Appendix X2 are suggested since they have proven to be useful. Further information about these procedures is provided by Snedecor and Cochran[4] and by NUREG/CR-4604.[5]

6.2 The analysts's data set is first tested for statistical normality. If normality is rejected, the data set is rejected and the analyst is determined to have failed the qualification test. If the data set is accepted as normally distributed, bias and precision tests may be performed.

6.3 If these statistical tests indicate that the analyst's data set exhibits bias and precision estimates that are within those of the established data base, the analyst is determined to be qualified. If the precision and/or bias estimates are not acceptable, the data set is rejected and the analysts is determined to have failed the qualification test.

6.4 Examples of statistical tests are presented in Appendix X2.

7. Keywords

7.1 analyst qualification; measurement(s); quality assurance; reference materials

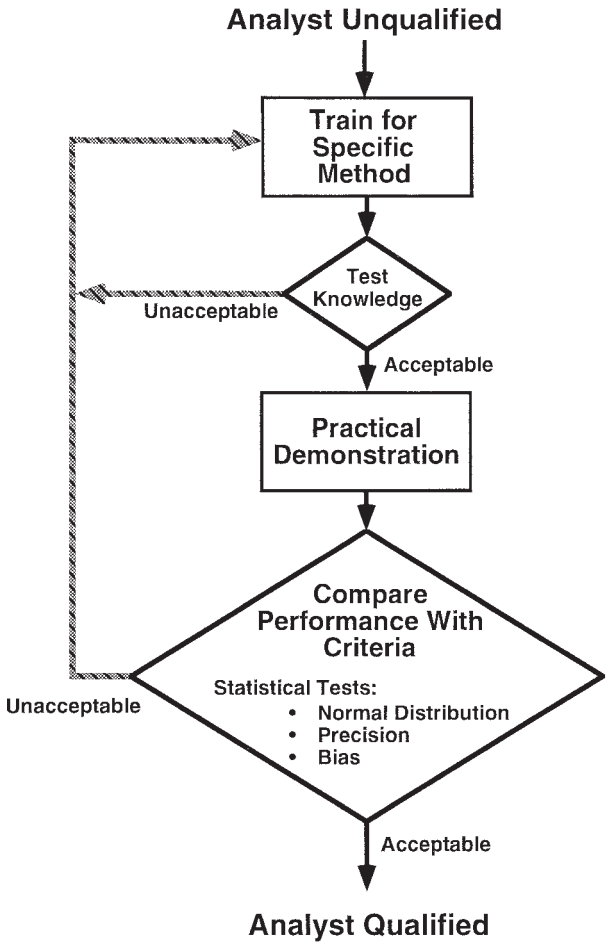

FIG. 2 Steps in the Analyst Qualification Process

[4] Snedecor, G.W., and Cochran, W.G., *Statistical Methods*, 8th Ed., Iowa State University Press, Ames, Iowa, 1989.

[5] NUREG/CR-4604, *Statistical Methods for Nuclear Material Management*, U.S. Nuclear Regulatory Commission, Washington, DC, 1988.

APPENDIXES

(Nonmandatory Information)

X1. STATISTICAL CONSIDERATIONS

X1.1 The significance level, α, for a statistical test is set depending on the desired risk of rejecting a qualified analyst. The smaller the significance level, the smaller the chance that a qualified analyst will be rejected (Type I error). For example, if the significance level is 0.10, then there is a one in ten chance that a qualified analyst will fail the test. However, by using a small α, the chance of accepting an unqualified analyst is large (Type II error). Thus there is a trade-off between accepting an unqualified analyst and rejecting a qualified one. Both types of errors can be controlled at desirable low levels by requiring a sufficiently large number of demonstration tests.[4,5] Practical limitations usually restrict the available number of demonstration tests so that only the risk of rejecting a qualified analyst may be adequately controlled by an appropriately small level of significance.

X1.2 For multiple statistical tests, another factor that should be considered when selecting the significance level of each test is the overall significance level. For example, the overall significance level for three independent tests would be $\alpha' = 1 - (1 - \alpha)^4$. Therefore, if the significance level of each test was 0.05, the overall significance level would be 0.143. In other words, the chance of a qualified analyst failing any one or more of three independent statistical tests when each test has a significance level of 0.05 would be 14.3 %.

X2. SUGGESTED STATISTICAL TESTS

X2.1 *TEST 1—Test for Normality*:

X2.1.1 *Problem Statement*—Test whether the demonstration data set is normally distributed.

NOTE X2.1—This test assumes that the data base itself is normally distributed.

Let,

$$Y_i = \frac{x_i - \mu_i}{\sigma_i} \quad \text{(X2.1)}$$

$$\bar{Y} = \sum_{i=1}^{n} \frac{Y_i}{n} \quad \text{(X2.2)}$$

$$s^2 = \sum_{i=1}^{n} \frac{(Y_i - \bar{Y})^2}{n-1} \quad \text{(X2.3)}$$

where:

x_i = the $_i$th demonstration result,
μ_i = the known mean associated with the $_i$th reference material sample in the data base, and
σ_i = the known standard deviation associated with the $_i$th reference material sample in the data base, and n is the number of demonstration results.

X2.1.2 *Test statistic*:

$$W = \frac{b^2}{(n-1)s^2} \quad \text{(X2.4)}$$

where:

$$b = \sum_{i=1}^{k} a_i (Y_{n-1+1} - Y_i) \quad \text{(X2.5)}$$

Y_i are sorted in ascending order,
k = n/2, rounded down, and
a_i are the Shapiro-Wilks coefficients.[4,5]

X2.1.3 *Acceptance Region*—Use Shapiro-Wilks tables to determine the acceptance region for a desired level of significance.[4,5]

X2.2 *TEST 2—Testing the Variance (Precision)*:

X2.2.1 *Problem Statement*—Test whether the standardized demonstration results have a variance different from the variance of a standard normal distribution.

$$H_o: \sigma^2 = 1 \quad \text{(X2.6)}$$

$$H_a: \sigma^2 \neq 1 \quad \text{(X2.6)}$$

X2.2.2 *Test Statistic*:

$$X^2 = \frac{(n-1)s^2}{\sigma^2} \quad \text{(X2.7)}$$

where:

σ^2 = 1.

X2.2.3 *Acceptance Region*—Use chi-square tables to determine the acceptance region for a desired level of significance and $n-1$ degrees of freedom.[4,5]

X2.3 *TEST 3—Testing the Mean (Bias):*

X2.3.1 *Problem Statement*—Test whether the standardized demonstration results have a mean different from the mean of the standard normal distribution.

$$H_o: \mu = 0 \; H_a: \mu \neq 0 \quad \text{(X2.8)}$$

X2.3.2 *Test Statistic*:

$$Z = \frac{\bar{Y} - \mu}{\sigma/\sqrt{n}} \quad \text{(X2.9)}$$

where:

μ = 0 and σ = 1.

X2.3.3 *Acceptance Region*—Use standard normal tables to determine the acceptance region for a desired level of significance.[4,5]

X2.3.4 The following examples provide data and test results for actual qualification at a particular laboratory.

X2.4 *Example 1*:

Analyst Testing Form

Method: 67015	Log Number: 050416		Analyst: RRR
Demonstration Result	Known Mean[A]	Known Standard Deviation[A]	Standardized Result
0.62616	0.62620	0.01689	−0.002
6.04147	6.14100	0.08341	−1.193
1.74910	1.80680	0.02023	−2.852
3.32222	3.36210	0.03368	−1.184
1.79410	1.80680	0.02023	−0.628
3.32106	3.36210	0.03368	−1.219
5.95575	6.14100	0.08341	−2.221
5.99493	6.14100	0.08341	−1.751
0.60847	0.62620	0.01689	−1.050

[A]From data base.

X2.4.1 All tests performed at the 0.05 level of significance:

X2.4.1.1 The data PASSED the normality test (Shapiro-Wilks value = 0.976).

X2.4.1.2 The calculated chi-square value for precision of 5.673 is not significant (PASSED).

X2.4.1.3 The calculated *Z*-value for bias of −4.790 is significant (FAILED).

X2.4.2 Tests indicate an overall conclusion that Analyst RRR FAILED.

X2.5 *Example 2*:

Analyst Testing Form

Method: 57171	Log Number: 04199		Analyst: QQQ
Demonstration Result	Known Mean[A]	Known Standard Deviation[A]	Standardized Result
169.60333	167.66600	5.27760	0.367
170.62016	167.66600	5.27760	0.560
990.31934	989.90796	15.72945	0.026
178.85460	167.66600	5.27760	2.120
579.69067	571.09302	15.78838	0.545
588.37824	571.09302	15.78838	1.095
32.99648	37.75880	4.71521	−1.010
997.59399	989.90796	15.72945	0.489
35.35918	37.75880	4.71521	−0.513

[A]From data base.

X2.5.1 All tests performed at the 0.05 level of significance:

X2.5.1.1 The data PASSED the normality test (Shapiro-Wilks value = 0.962).

X2.5.1.2 The calculated chi-square value for precision of 7.581 is not significant (PASSED).

X2.5.1.3 The calculated *Z*-value for bias of 1.822 is not significant (PASSED).

X2.5.2 Tests indicate an overall conclusion that Analyst QQQ PASSED.