Designation: D5847 – 02 (Reapproved 2012)

Standard Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis[1]

This standard is issued under the fixed designation D5847; 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 practice provides specific, mandatory requirements for incorporating quality control (QC) procedures into all test methods under the jurisdiction of Committee D-19.

1.2 ASTM has adopted the following:

Policy on implementation of requirements for a quality control section in standard test methods generated by Committee D-19 on Water.

GENERAL—By July 29, 1998, or at the next reapproval or revision, whichever is later, every D-19 Standard Test Method shall contain a QC section that is in full compliance with the requirements of this practice.

NEW COLLABORATIVE TESTING—As of July 29, 1998, each collaborative study design shall include a QC section as part of the method to be tested. Prior to approval of the study design, the Results Advisor shall ascertain the appropriateness of the QC section in meeting the requirements of this Practice and Practice D2777, and shall advise the designer of the study of any changes needed to fulfill the requirements of these practices. Before a collaborative study may be conducted, approval of the study design by the Results Advisor must be obtained.

OLDER VALIDATED METHODS—Standard test methods that were validated using D-2777-77, D-2777-86, or D-2777-94, when balloted for reapproval or revision, shall contain a QC section based upon the best information from the historical record. Where appropriate, information derived from the record of the collaborative study shall be utilized for this purpose. The introduction of the QC section into these standard test methods shall not be construed as a requirement for a new collaborative study, though the Subcommittee may opt for such a study. Any information available regarding QC or precision/bias testing shall be included in the appropriate sections of the published method.

1.3 Required QC sections in all applicable test methods are intended to achieve two goals. First, users of Committee D-19 test methods will be able to demonstrate a minimum competency in the performance of these test methods by comparison with collaborative study data. Second, all users of test methods will be required to perform a minimum level of QC as part of proper implementation of these test methods to ensure ongoing competency.

1.4 This practice contains the primary requirements for QC of a specific test method. In many cases, it may be desirable to implement additional QC requirements to assure the desired quality of data.

1.5 The specific requirements in this practice may not be applicable to all test methods. These requirements may vary depending on the type of test method used as well as the analyte being determined and the sample matrix being analyzed. See Explanation 1 in Appendix X1.

1.5.1 If there are compelling reasons why any of the specific QC requirements listed in this practice are not applicable to a specific test method, these reasons must be documented in the QC section of the test method.

1.5.2 With the approval of Committee D-19 on the recommendation of the D-19 Results Advisor and the Technical Operations section of the Executive Subcommittee, a statement giving the compelling reasons why compliance with all or specific points of this practice cannot be achieved will meet the requirements of both ASTM and this practice.

1.5.3 Test Methods developed prior to the approval of this practice with a QC Section that meet the requirements of Specification D5789 are considered in compliance with this Practice.

1.6 This practice is for use with quantitative methods and may not be applicable to qualitative test methods.

1.7 Presently, this practice is applicable primarily to chemical test methods. It is intended that, in future revisions, the practice will be expanded to include other methods such as microbiological methods.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3648 Practices for the Measurement of Radioactivity

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on Quality Systems, Specification, and Statistics.

Current edition approved June 15, 2012. Published June 2012. Originally approved in 1999. Last previous edition approved in 2012 as D5847 – 02(2007). DOI: 10.1520/D5847-02R12.

[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.

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
D4375 Practice for Basic Statistics in Committee D19 on Water
D5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents[3]
D5810 Guide for Spiking into Aqueous Samples

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice, refer to Terminology D1129 and Terminology D4375.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *batch*—a set (group) of samples analyzed such that results of analysis of the QC samples (laboratory control sample, method blank, matrix spike, and duplicate or matrix spike duplicate) analyzed with the batch are indicative of the quality of the results of analysis of samples in the batch. The number of samples in the batch is defined by the task group responsible for the method. See 6.4 and Explanation 2 in Appendix X1.

3.2.1.1 *Discussion*—When results from tests of any of the QC samples associated with batch the fail to meet the performance criteria, the test method should define the appropriate corrective action. To make such a response valid, the batch must be constructed in such a way as to assure that all variables affecting the batch will affect all samples in the batch in a statistically equivalent manner.

3.2.2 *calibration standard*—a solution containing the analyte of interest at a known concentration either purchased from an external source or prepared in-house from materials of known purity or concentration, or both, and used to calibrate the measurement system.

3.2.3 *detection limit*—the minimum concentration or amount of a substance that can be detected with a known degree of confidence.

3.2.4 *independent reference material (IRM)*—a material of known purity and concentration obtained either from the National Institute of Standards and Technology (NIST) or other reputable supplier. The IRM shall be obtained from a different lot of material than is used for calibration.

3.2.5 *laboratory control sample (LCS)*—a sample of known concentration and composition that is taken through the entire test method to determine whether the analytical system is in control. The LCS must be prepared in the appropriate ASTM-grade water from a material that sufficiently challenges the test. See Explanation 3 in Appendix X1. The LCS can be an IRM obtained from an outside source or prepared in-house from materials of known purity and concentration. Alternatively, the LCS may be a real sample of the matrix that is typically analyzed and which has been fully characterized.

3.2.5.1 *Discussion*—The LCS may also be commonly known as a "quality control sample" or an "ongoing precision and recovery sample" (OPR).

3.2.6 *matrix spike (MS)*—addition of a known concentration of analyte to a routine sample representing a specific matrix for the purpose of evaluating interference from matrix components. (See Guide D5810.)

3.2.7 *method blank (blank)*—reagent water (see Specification D1193) either known to be free of the constituent(s) of interest or containing only a low, known concentration of the constituent(s) of interest not exceeding five times the estimated detection limit.

3.2.7.1 *Discussion*—The purpose of analysis of the method blank is to confirm that the reagents or analytical system, or both, do not contribute a measurable amount of the constituent(s) of interest during analysis of routine samples or, if they do, to determine what the contribution is.

3.2.8 *quantitation limit*—the minimum concentration or amount of a substance that can be measured with a known degree of confidence.

3.2.9 *sample pretreatment (pretreatment)*—any handling, manipulation or treatment of a sample prior to subjecting the sample to the analysis. Examples are filtration, digestion, dilution, pH adjustment and extraction.

4. Summary of Practice

4.1 This practice provides the writer of a test method in Committee D19 specific steps to be included in the QC section of the test method. A QC section is required in all applicable standard test methods that mandates use of the following QC measures:

4.1.1 Periodic calibration or verification of calibration of the measurement system,

4.1.2 Initial demonstration of laboratory capability,

4.1.3 Analysis of at least one blank per batch,

4.1.4 Analysis of at least one LCS per batch,

4.1.5 Analysis of at least one MS per batch, where applicable, and

4.1.6 Periodic analysis of an IRM.

4.2 Duplicate analysis of at least one sample per batch is suggested. The duplicate analysis may be of a sample or of a matrix spike (matrix spike duplicate; MSD). See Explanation 4 in Appendix X1.

4.3 If there are valid reasons why any of the above QC requirements are inapplicable to a specific test method (see Section 1.), these reasons must be documented in the QC section of the test method. See 1.5 and Explanation 1 in Appendix X1.

5. Significance and Use

5.1 In order to be certain that the end user of analytical results obtained from using an ASTM Committee D-19 test method can be confident that the values have been obtained through a competent application of the test method, a demonstration of the proficiency of the analytical system must be performed. Appropriate proficiency is demonstrated by achievement of performance criteria derived from results of the test method collaborative study. The QC measures specified in this practice must be included in each ASTM test method, as applicable, to ensure the quality of measurements.

5.2 In order for users of D-19 test methods to achieve consistently valid results, a minimum level of QC must be performed. This minimum level of QC is stipulated in this practice and by the taskgroups developing D-19 test methods.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

If the specific requirements outlined in this practice are not applicable to the test method, alternative QC must be defined in the test method.

6. Requirements for QC Specifications in Test Methods

6.1 Every test method must have a quality control (QC) section. Listed below are requirements applicable to nearly all chemical test methods and that must be followed to ensure that the test method is in control and to validate the accuracy of data generated for a specific matrix.

6.1.1 The measures that must be specified in the QC section of test methods and the reasons for these measures are as follows:

6.1.1.1 Calibration and calibration verification are necessary to ensure that the analytical system is properly calibrated during the period that the analysis is performed.

6.1.1.2 An initial demonstration of laboratory capability is necessary to prevent errors as a result of unfamiliarity with the test.

6.1.1.3 Analysis of a blank with each batch may indicate that analytes in a test sample are the result of contamination.

6.1.1.4 An LCS is run with each batch to determine that the measurement system is in control at the time samples are being analyzed.

6.1.1.5 An MS (recovery check) provides information on the bias of the test method in a specific matrix.

6.1.1.6 A duplicate analysis (Dup) or duplicate of the MS (matrix spike duplicate; MSD) indicates the repeatability of the method for a specific matrix.

6.1.1.7 An IRM is analyzed periodically to validate the accuracy of the test system and standards used for calibration.

6.1.2 In addition to the QC measures required above, each test method should contain a detection limit and a quantitation limit so that there is an indication of the lowest level at which the substance(s) determined by the test method can be detected and measured.

6.1.3 Statistical tests should be done at a significance level of $\alpha \leq 0.01$, that is, ≥ 99 % confidence level. If other levels are specified, the reason for deviation should be delineated in the method.

6.1.4 The operational principles and characteristics of detectors used for radioactivity measurements are somewhat different from those of instruments used for measurements of chemical and physical properties. Therefore, authors of ASTM test methods for radioactivity measurements should provide specific guidance within each test method, practice or guide relative to applicable QC program requirements. Guidance on the preparation and use of instrument tolerance and control charts can be found in Practices D3648 and D3856, and in ASTM MNL 7.[4]

6.2 *Calibration and Calibration Verification*—For test methods requiring calibration of instrumentation, an appropriate number of calibration standards must be analyzed during day that an analysis is performed to confirm that the instrument is properly set up and required sensitivity is being obtained. The actual number of standards required will depend on the requirements of the test method. For tests run infrequently, analysis of a single calibration standard to verify an existing calibration curve may suffice. For tests run frequently, it may be necessary to intersperse verification standards with test samples. Under these circumstances, it is recommended that a different standard concentration be used each time calibration is verified. Raw data (absorbance, intensity, etc.) should be compared to data generated in the past under the same conditions and should fall within three standard deviations of the mean value found in the past based on the pooled single operator precision. Alternatively, data should be compared to the calibration limits stated in the test method or should be developed from collaborative study data. Refer to Guide D3856 and Practice D3648 for further information on calibration checks.

6.2.1 For titrimetric test methods, titrants must be standardized on a scheduled basis against a standard solution of known concentration in duplicate or triplicate. The average normality/molarity is then used for calculation. The frequency of standardization is left to the judgment of the writer of the test method and should be based on the stability of the titrant.

6.2.2 An alternate calibration procedure, such as an internal standard, external standard, or single-point calibration procedure, must be specified in the test method.

6.2.3 The test method must establish the frequency of calibration and calibration verification.

6.3 *Initial Demonstration of Laboratory Capability*—A test must be included in the test method to confirm that the laboratory is capable of running the test method and generating acceptable data. This test of laboratory capability will vary depending on the test method. Whenever appropriate, a precision and bias (as recovery) test is performed. For most test methods this can be done by analyzing at least seven replicates of a standard solution prepared from a reference material containing the analyte at one of the concentration levels used in the collaborative study. The matrix and chemistry of the solution should be such that, when spiked, results statistically equivalent to results produced in the collaborative study should be produced. Each of the replicates should be presented to the operator as unknowns and should be interspersed with other samples following the procedures used in the collaborative study. For some test methods, fewer replicates may be used, however, the statistical power of the test is dependent on the number of replicates, and the meaningfulness of the study is reduced when fewer than seven replicates are used. (For the examples in this practice, fewer than seven replicates are used for convenience.) Each replicate must be taken through the complete analytical test method including any pretreatment. The mean and standard deviation of these results are then calculated as described in Terminology D4375 and compared to the single operator precision and recovery found in the collaborative study.

NOTE 1—*Initial Demonstration of Laboratory Capability*—The type of test designed to assess the capability of a laboratory or operator is at the discretion of the method writer. It can be designed any way the method writer believes is appropriate for the test method so long as it provides meaningful data to ensure that the laboratory or operator is capable of generating results that are valid and accurate within the confidence limits

[4] ASTM Manual on Presentation of Data and Control Chart Analysis, ASTM MNL 7.