Designation: D5847 – 22

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 D19.

1.2 ASTM International has adopted the following:

Policy on implementation of requirements for a quality control section in standard test methods generated by Committee D19 on Water.

GENERAL—By July 29, 1998, or at the next reapproval or revision, whichever is later, every D19 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 or equivalent 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 or equivalent shall be obtained.

OLDER VALIDATED METHODS—Standard test methods that were validated using Practices D2777 – 77, D2777 – 86, or D2777 – 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 test method.

1.3 Required QC sections in all applicable test methods are intended to achieve two goals. First, users of Committee D19 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.

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 shall be documented in the QC section of the test method.

1.5.2 With the approval of Committee D19 on the recommendation of the D19 Results Advisor or equivalent 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.

1.6 This practice is for use with quantitative test 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 test methods such as microbiological test methods.

1.8 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[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 May 1, 2022. Published July 2022. Originally approved in 1999. Last previous edition approved in 2020 as D5847 – 02 (2020). DOI: 10.1520/D5847-22.

***A Summary of Changes section appears at the end of this standard**

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1141 Practice for Preparation of Substitute Ocean 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
D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
D5810 Guide for Spiking into Aqueous Samples
D6362 Practice for Certificates of Reference Materials for Water Analysis

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *batch, n*—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.

3.2.1.1 *Discussion*—The number of samples in the batch is defined by the task group responsible for the test method. See 6.4 and Explanation 2 in Appendix X1.When results from tests of any of the QC samples associated with the batch fail to meet the performance criteria, the test method should define the appropriate corrective action. To make such a response valid, the batch shall 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, n*—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 *certified reference material (CRM), n*—reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure that established its traceability to an accurate realization of the unit in which the property values are expressed and for which each certified value is accompanied by an uncertainty at a stated level of confidence obtained either from the National Institute of Standards and Technology (NIST) or other national institute or ISO 17034 accredited supplier.

3.2.3.1 *Discussion*—The CRM shall be obtained from a different lot of material than is used for calibration, if possible. There is significant variation in the overall quality of commercially available Certified Reference Materials and caution should be used when choosing Certified Reference Materials. Use Practice D6362 to provide guidance as to what information needs to be included on certificate of a certified reference material.

3.2.4 *detection limit, n*—minimum concentration or amount of a substance that can be discriminated from a blank with a known degree of confidence.

3.2.5 *laboratory control sample, LCS, n*—sample of known concentration and composition that is taken through the entire test method to determine whether the analytical system is in control.

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, n*—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), n*—reagent water (see Specification D1193) free of the constituent(s) of interest at the quantitation 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, n*—minimum concentration or amount of a substance that can be measured with a known degree of confidence.

3.2.9 *sample pretreatment (pretreatment), n*—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 analyst 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 a CRM.

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 shall be documented in the QC section of the test method. See 1.5 and Explanation 1 in Appendix X1.

[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.