Designation: D2777 – 21

# Standard Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water[1]

This standard is issued under the fixed designation D2777; 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 establishes uniform standards for estimating and expressing the precision and bias of applicable test methods for Committee D19 on Water. Statements of precision and bias in test methods are required by the Form and Style for ASTM Standards, "Section A21. Precision and Bias (Mandatory)." In principle, all test methods are covered by this practice. However, the variability equations provided in this standard are applicable only to test methods that yield continuous function values.

1.2 Except as specified in 1.4, 1.5, and 1.6, this practice requires the task group proposing a new test method to carry out a collaborative study from which statements for precision (overall and single-operator standard-deviation estimates) and bias can be developed. This practice provides general guidance to task groups in planning and conducting such determinations of precision and bias.

1.3 This practice requires that a task group making a substantive revision to a test method also perform a limited-scale collaborative study (known as a — comparability study) to evaluate the effect of the revision on the precision and bias statement. This practice provides guidance to task groups for conducting such limited-scale collaborative studies. Examples of substantive modifications may include, but are not limited to, changes in mandatory or allowable instrumentation, reagents, reaction times, etc.

1.3.1 Changes to applicable water matrices in the Scope of a method may constitute a substantive modification under this provision. Only matrices that have been evaluated in an approved collaborative study may be listed in the Scope of a method. It is recognized that the term "matrix" is generally vague. Terms specifying matrix types can cover significantly different chemical constituents, unless the matrix is synthesized to be of a standardized makeup. Substitute Wastewater (Practice D5905) is one such defined matrix. For purposes of this practice, the importance of this requirement is to assist the user of a D19 standard in determining the applicability of the method to their samples. Evaluated matrices should be described with as much detail as possible to minimize misapplication.

1.3.2 A method's concentration-range extension that is deemed to merit additional collaborative testing (even without a method modification that would otherwise be considered substantive) shall require a full collaborative study, as described in 7.1 through 7.5, but only at concentrations representative of the extended range. Note that such a collaborative study could involve as little as a single concentration study in a single reproducible matrix.

1.3.3 Whether a revision to a test method includes substantive modification shall be determined by consensus of the Committee.

1.4 If a full-scale collaborative study is not technically feasible, because of the nature of the test method or instability of samples, the most complete collaborative study that is technically feasible shall be conducted to provide the best possible limited basis for estimating the overall and single-operator standard deviations. In some situations, an intermediate collaborative study as described in Guide D7847 may provide an appropriate approach. It is recognized that there may be circumstances when even a limited collaborative study is not feasible. Any collaborative study plan that does not meet all the requirements spelled out in this practice will require a review and recommendation by the Results Advisor and an approval by the D19 Technical Operations Section of the Executive Subcommittee.

1.4.1 Examples of acceptable studies are the local-area intermediate studies conducted by Subcommittee D19.24 on microbiological methods because of inherent sample perishability. Such intermediate collaborative studies meet the same degrees of freedom and participant requirements as full collaborative studies. They involve six or more completely independent local-area analysts who can begin analysis of uniform samples at an agreed upon time. Guide D7847 can provide guidance to the task group, the Results Advisor, and the Technical Operations Section of the Executive Subcommittee of Committee D19 on the appropriate design of an

[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 Dec. 15, 2021. Published June 2022. Originally approved in 1969. Last previous edition approved in 2013 as D2777 – 13. DOI: 10.1520/D2777-21.

acceptable intermediate collaborative study for test methods that measure highly perishable parameters.

1.4.2 If providing homogenous samples with a sufficiently stable analyte concentration is not feasible under any circumstances, a statement of single-operator precision may meet the requirements of this practice. Whenever possible, this statement should be developed from data generated by multiple independent operators, each doing replicate analyses on independent samples (of a specific matrix type), which generally fall within specified concentration ranges (see 7.2.5.2 (*3*)).

1.5 A collaborative study that satisfied the requirements of the version of this practice in force when the study was conducted will continue to be considered an adequate basis for the precision-and-bias statement required in each test method. If the study does not satisfy the current minimum requirements for a collaborative study, a statement listing the study's deficiencies and a reference to this paragraph shall be included in the precision-and-bias statement as the basis for an exemption from the current requirements.

1.6 Committee D19, through a Main Committee ballot, may approve publication of a "Preliminary" Standard Method for a period not to exceed 5 years. Preliminary Standards must contain a minimum of a single-operator precision-and-bias statement and a Quality Control section based on the single operator data. Publication of a Preliminary Standard is conditional on the approval of a full D2777 collaborative study design for the standard. Precision-and-bias statements authorized by this paragraph shall include the date of approval by Committee D19.

1.7 Per Section A21.2.3 of the ASTM Form and Style Manual the committee may delay an interlaboratory study for a new method and include a temporary statement in the Precision and Bias Section that addresses only single operator precision ("repeatability"). This statement is valid for five years from the initial publication date. In this case, a single laboratory study shall be conducted in accordance with 7.6.

1.8 In Section 12, this practice shows exemplary precision-and-bias-statement formats for: (*1*) test methods yielding a numerical measure, (*2*) test methods yielding a non-numerical report of success or failure based on criteria specified in the procedure, and (*3*) test methods specifying that procedures in another ASTM test method are to be used with only insignificant modifications.

1.9 All studies, even those exempt from some requirements under previous sections, shall receive approval from the Results Advisor before being conducted (see Section 8) and after completion (see Section 13).

1.10 This practice satisfies the QC requirements of Practice D5847.

1.11 It is the intent of this practice that task groups make every effort to retain all the data from their collaborative studies. Values should not be eliminated unless solid evidence exists for their exclusion. The Results Advisor should work closely with the task groups to effect this goal.

1.12 *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.*

## 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
D4375 Practice for Basic Statistics in Committee D19 on Water (Withdrawn 2018)[3]
D5790 Test Method for Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D5905 Practice for the Preparation of Substitute Wastewater
D6091 Practice for 99 %/95 % Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error
D6512 Practice for Interlaboratory Quantitation Estimate
D7847 Guide for Interlaboratory Studies for Microbiological Test Methods
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E178 Practice for Dealing With Outlying Observations
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1169 Practice for Conducting Ruggedness Tests

2.2 *ASTM Adjuncts:*

DQCALC Microsoft Excel-based software for the Interlaboratory Quantitation Estimate (IQE)[4]

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminologies D1129, D4375 and E456, and Practice E177.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accuracy, n*—a measure of the degree of conformity of a single test result generated by a specific procedure to the assumed or accepted true value, and includes both precision and bias.

3.2.2 *bias, n*—the persistent positive or negative deviation of the average value of a test method from the assumed or accepted true value.

3.2.3 *comparability study, n*—a collaborative study that incorporates side-by-side evaluation of the test method before and after a substantive modification to a test method.

[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.

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

[4] Available from ASTM International Headquarters. Order Adjunct No.ADJDQCALC. Original adjunct produced in 2007.