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Standard Guide for Determination of Precision and Bias of Methods of Committee D22[1]

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1. Scope

1.1 This standard provides guidance to task groups of Committee D22 on Sampling and Analysis of Atmospheres in planning and conducting collaborative testing of candidate methods.

1.2 It is intended for use with other ASTM practices for the determination of precision and bias.

1.3 It is applicable to most manual and automated methods and to most components of monitoring systems. It is recognized that the evaluation of monitoring systems may provide special problems. Practice D 3249 should be considered for general guidance in this respect.

1.4 It is directly applicable to chemical methods and in principle to most physical methods, sampling methods, and calibration procedures.

1.5 The processes described are for the general validation of methods of test. A user has the obligation and responsibility to validate any method it uses for a specific application and to demonstrate its own competence in the use of validated methods.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D 3249 Practice for General Ambient Air Analyzer Procedures

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 The terms used in this practice are consistent with those defined in Practices D 2777, E 177, E 180, and E 691.

3.2 *Definitions:*

3.2.1 *accuracy*—the degree of conformity of a value generated by a specific procedure to the assumed or accepted true value. It includes both precision and bias.

3.2.2 *bias*—a systematic (nonrandom) deviation of the method average value or the measured value from an accepted reference value.

3.2.3 *candidate method*—an analytical method or measurement process being considered for standardization. A method is a "candidate" until completion of all phases of the consensus process specified by ASTM regulations for a proposal, an emergency standard, or a standard.

3.2.4 *collaborative test*—an interlaboratory study of a test method wherein the participants analyze or make measurements on sub-samples of the same test material. If the test method includes the sampling of atmospheres, the participants should sample the same test atmosphere, as possible.

3.2.5 *laboratory bias*—systematic differences between the true value and a value reported by a laboratory due to errors of application such as losses, contamination, miscalibration, and faulty manipulations, for example.

3.2.6 *method bias*—systematic departures of the limiting mean from the true value of the parameter measured, caused by physical or chemical phenomena inherent in the methodology.

3.2.7 *over-all precision*—a value including components of within-laboratory and between-user variability.

3.2.8 *precision*—the degree of mutual agreement between individual measurements using an analytical method or measurement process. In practice, the standard deviation of an entire array of reviewed and acceptable data is calculated to provide the value to be stated as the precision of the method.

[1] This guide is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.01 on Quality Control.

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[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.

3.2.9 *ruggedness test*—a factorial test designed to explore the sensitivity of the method to variations in the procedure (see Youden and Steiner, 1975).[3]

3.2.10 *single-operator precision*—a measure of the replication of repeated measurements obtained by a single operator on a given sample.

3.2.10.1 *Discussion*—Other classifications of precision which are useful in evaluating a method, a measurement, or performance within a single laboratory are: multioperator precision, single or multi-apparatus precision, and single or multi-day precision.

3.2.10.2 *Discussion*—The terms "repeatability" and "reproducibility" are not standardized, but have generally come to mean "single-laboratory-operator-material precision" and "multi-laboratory-multi-operator-single material precision," respectively. Such usage is maintained in the text of this practice.

3.2.10.3 *Discussion*—Further classifications of bias which are useful in evaluating performance are: operator bias, apparatus bias, and day bias.

4. Summary of Guide

4.1 Data supporting a statement of single-operator repeatability is the entrance requirement for any candidate method to be considered for standardization by Committee D22. The task group to which a candidate method is assigned will review it for adequacy in this respect, and conduct further tests as necessary to evaluate its precision and bias, as technically feasible. A method may be accepted as a proposed method, provided the repeatability is known or has been ascertained and provided all other criteria for acceptance have been met. Independent tests by at least three laboratories shall be required to substantiate the repeatability of a method before it attains the status of a standard method. Collaborative testing by at least five laboratories to estimate the interlaboratory bias and, if applicable to evaluate the method's inherent bias with respect to the "true" value is needed for all standard methods and must be accomplished within 5 years of its initial issuance as a standard, if such testing has not already been done. Failure to subject such methods to appropriate collaborative testing, constitutes valid grounds for disallowing its reapproval as a standard.

4.2 Procedures that may be used in collecting the required data are given with particular emphasis upon the applicability to analysis of atmospheres. Documentation requirements are established. Terms that are useful in expressing statements of precision and bias are presented.

5. Significance and Use

5.1 The objective of this standard is to provide guidelines to Committee D22 for the evaluation of the precision and bias, or both, of ASTM standard methods and practices at the time of their development. Such an evaluation is necessary to assure that a cross section of interested laboratories could perform the test and achieve satisfactory results, using the method as written. It also provides guidance to the user as to what levels of precision and accuracy may be expected in such usage.

5.2 The write-up of the method describes the media for which the test method is believed to be appropriate. The collaborative test corroborates the write-up within the limitations of the test design. A collaborative test can only use representative media so that universal applicability cannot be implied from the results.

5.3 The fundamental assumption of the collaborative test is that the media tested, the concentrations used, and the participating laboratories are representative and provide a fair evaluation of the scope and applicability of the test method as written.

6. General Policy

6.1 This section describes the general policy to be followed by Committee D22, its subcommittees, and task groups in the development of ASTM standard methods and practices. The objective of Committee D22 is to develop fully evaluated standard methods and practices as far as possible. In cases where this is not expedient, proposed methods, as defined in 6.2, may be developed. In each case, an appropriate task group shall have the responsibility to critically examine the method or practice, conduct evaluation tests by round robins or other techniques including ruggedness tests, and to recommend it, if meritorious, for subcommittee balloting. No method or practice shall be released and recommended for balloting unless the precision or accuracy requirements, or both, as set forth in the following, have been satisfied.

6.1.1 Collaborative testing by D22 is the preferred method of validation. Data obtained by collaborative testing by others may be used in lieu of D22 testing, provided that such testing was equivalent to ASTM approved procedures. In either case, a copy of the test procedures and data must be filed in a research file maintained at ASTM for such purposes.

6.2 *Proposed Method*—A proposed method is one that has found favorable usage in a specific laboratory, or has been used by several laboratories, but has not yet been standardized. In each case, the test method is submitted by its proponents to Committee D22 for standardization.

6.2.1 The minimum requirement for balloting of a proposed method shall be the inclusion in it of a single laboratory's statement of single-operator precision, together with supporting experimental data. Test methods meeting this requirement will be referred to a Task Group, following procedures established by Committee D22.

6.2.2 The experimental data needed to support a proposal must reflect a test of the method as a whole, that is, sampling, apparatus, reagents and, calibration, and must use a procedure that is essentially identical to that described in the proposal. Any significant deviations between the procedure used to gather the data and the proposed procedure shall be clearly identified.

6.2.3 If such data are missing or inadequate, but the method itself is considered by consensus of Committee D22 to be worthy of further study, a task group may be assigned to conduct experimental studies or enlist the services of at least

[3] Youden, W. J. and Steiner, G. H., *Statistical Manual of the Association of Official Analytical Chemists*, AOAC International, 481 North Frederick Ave., Suite 500, Gaithersburg, MD 20877-2417, 1975.