Designation: C802 – 14 (Reapproved 2022)

Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials[1]

This standard is issued under the fixed designation C802; 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 describes techniques for planning, conducting, and analyzing the results of an interlaboratory study (ILS) with the objective of developing the precision statement of a test method. It is designed to be used in conjunction with Practice C670. The methods used in this standard are consistent with those in Practice E691.

1.2 This practice is not intended for use in programs whose purpose is to develop a test method or to assess the relative variability of two or more test methods. Refer to Practice C1067 for procedures to evaluate the ruggedness of a test method.

1.3 The system of units for this practice has not been specified. Dimensional quantities in the practice are presented only in examples of calculations.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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]

C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50 mm] Cube Specimens)

C136 Test Method for Sieve Analysis of Fine and Coarse Aggregates

C311/C311M Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C1067 Practice for Conducting a Ruggedness Evaluation or Screening Program for Test Methods for Construction Materials

E105 Guide for Probability Sampling of Materials

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

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of general statistical terms, refer to Terminology E456.

3.1.2 For definitions of terms associated with precision of test methods for construction materials, refer to Practice C670.

4. Significance and Use

4.1 This practice provides requirements for planning and conducting an interlaboratory study to obtain data to develop single-operator and multilaboratory precision statements for a test method. It includes methods to evaluate data consistency before carrying out the calculations to develop the precision statement. The procedures are compatible with those in Practice E691.

4.2 The ILS data obtained from this practice are intended for developing the precision values for writing single-operator and multilaboratory precision statements in accordance with Practice C670.

4.3 Appendix X1 provides an example to illustrate the calculations to obtain the precision values of the test method from the ILS data. This may be used to check a user-developed electronic spreadsheet for carrying out the calculations.

[1] This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates. This practice was developed jointly by ASTM Committee C01, C09, D04, and D18, and is endorsed by all four committees.

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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.

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

4.4 Appendix X2 discusses the additional calculations required for an interlaboratory study of a test method that includes making test specimens as part of the procedure. In this case, batch-to-batch variability needs to be considered.

4.5 Appendix X3 discusses the use of analysis of variance as an alternative approach to obtain the precision values from the ILS data.

5. General Requirements

5.1 Certain criteria need to be met before undertaking an interlaboratory study to determine the precision of a test method. If some conditions are not met or are met incompletely, the program will become more complicated to administer and require more work and expense, or may result in impaired information. The requirements outlined in this section are intended to ensure that the test method is free of technical difficulties to the greatest extent possible before an expensive and time-consuming interlaboratory study is undertaken.

5.2 The first requirement is the existence of a valid and well-written test method that has been developed in one laboratory and has been subjected to ruggedness evaluation of the testing procedure and conditions as described in Practice C1067. As a result of the screening procedure and some experience with the test method in the sponsoring laboratory and one or two others, a written version of the test method has been developed (but not necessarily published as a standard) that describes the test procedure in terms that can be followed by a competent operator in any properly equipped laboratory. Critical conditions that affect the test results need to be identified and the proper and realistic degree of control of those conditions have to be specified in the description of the test procedure.

5.2.1 The tolerances established for various conditions in a test method provide reasonable ranges for these conditions and recognize that precise values with small tolerances may not be achievable in practice. Variations in test results due to variations in such conditions contribute to the total variation, which determines the precision of the test method. If the resulting variation is so great that uncertainties in average values obtained by the test method are unacceptably high, the test method itself is at fault and it will need to be improved or replaced by a better one. An expensive and time-consuming interlaboratory study is not recommended for such a test method.

5.2.2 Apparatus required for performing the test must be defined clearly and must be available or able to be produced. If alternative apparatus is permitted, criteria need to be provided on the performance requirements of the apparatus, such as by specifying acceptable limits of measurements on standard reference materials.

5.3 Personnel in laboratories participating in the ILS should have adequate experience with routine laboratory procedures so that they are competent to run the test. The importance of this requirement will vary with the complexity of the method and the degree to which it departs from familiar procedures.

5.4 It is helpful to have preliminary knowledge about how changes in materials and conditions affect the test results. There should be a reasonable degree of certainty that the single-operator variances are the same in different laboratories, and that troublesome interactions do not exist. These conditions are investigated in the initial analysis of the data of an interlaboratory study, and are discussed further in 10.4.

5.5 Facilities and procedures for procurement, preparation, and distribution of samples or test specimens must be available.

5.6 Selection of samples or test specimens must be done by a randomization process, and one person who is familiar with randomization procedures needs to be responsible for seeing that an appropriate randomization technique is used. Refer to Practice E105.

5.7 The precision of the test method should be evaluated on different materials with a range of the characteristic being measured that encompasses the typical use of the method in practice. (See 7.1 and 7.2.)

5.8 Adequate numbers of participating laboratories, operators, and materials must be available. Requirements in these areas are specified in Sections 6 and 7.

5.9 The entire interlaboratory test program should be developed from the beginning with the help and advice of persons familiar with statistical procedures and with the materials involved. The ASTM International Interlaboratory Study Program can support subcommittees in the development of precision statements by assisting in the design of an interlaboratory study, distribution of specimens or samples, data analysis, and preparation of a draft research report. Additional information about the ASTM ILS program can be obtained from the ASTM Website.

5.9.1 It may not always be possible to obtain people who are familiar with the materials involved and who have a sufficient knowledge of the proper statistical techniques and their proper use. In this case, the subcommittee should obtain the services of a statistician who has experience in practical work with data from materials testing, and provide that person with an opportunity for learning something about the particular materials and test method involved. Planners of an interlaboratory study need to avoid the pitfall of assuming that the use of statistical analysis software programs necessarily results in special expertise in manipulating the data or interpreting the results.

5.10 It is important to bear in mind that estimates of the precision of a test method are always based on a particular set of data obtained at a particular time and precision values need to be kept up-to-date. As materials, apparatus, and conditions change, and operators change or gain more experience, the characteristic precision of the results obtained may change, especially if the test method is new. In some cases, it may be desirable to conduct more tests at a later date (though not necessarily a repetition of the complete interlaboratory study) in order to provide a check on estimates previously obtained and either verify them or introduce revisions. When a subcommittee revises a test method, it should consider whether the proposed changes might affect the precision of the method. If there is a possibility that precision will be affected, limited

interlaboratory testing is recommended to evaluate whether the existing precision statement is still applicable or if a new ILS needs to be organized to better reflect the precision of the revised method.

6. Laboratories

6.1 Obtaining participating laboratories for an interlaboratory study is often one of the most difficult problems connected with the process. The number of laboratories available is seldom as extensive as one would like, and if the test method is new, complicated, or expensive and time-consuming to run, the problem is further complicated.

6.2 For the purposes of programs using this practice, it is recommended that at least ten competent laboratories be included (**1**, **2**).[3] In cases where it is impossible to obtain ten laboratories, the effect of an increased number may be obtained by repeating the program with the same group of laboratories six months later. If this procedure is followed, it is necessary to be sure that the same materials are used, and that their characteristics have not changed in the interim. This approach, however, may not provide a proper measure of the between-laboratory component of variance, unless different operators or equipment, or both, are used for the repeat testing. In any case, six is the absolute minimum number of laboratories for evaluating the precision of a test method. This means that at least seven to eight laboratories should be in the ILS study in case problems are encountered with the data provided by a participating laboratory.

6.3 In general, it is recommended that any laboratory that is considered qualified to run the test in routine testing situations should be permitted and encouraged to participate. "Qualified" implies proper laboratory facilities and testing equipment, competent operators familiar with routine laboratory techniques, a history of reliable testing work, and sufficient time and interest to do a good job. It does not mean, however, that only a select group of laboratories that are considered to be those best qualified for the interlaboratory study should be picked. Precision estimates for inclusion in a test method must be obtained under conditions and through the efforts of laboratories and personnel that are representative of the situations in which the test method will be used in practice (**2**). If a laboratory satisfies all the other requirements, but its personnel has had insufficient experience with the method, the operators in that laboratory should be given an opportunity to familiarize themselves with the method and to practice its application before the interlaboratory study starts.

7. Materials

7.1 *Number*—The number of materials to be included in an interlaboratory study will depend on the following:

7.1.1 The range of the values of the property that may be measured in practice and how the precision varies over that range;

7.1.2 The types of materials to which the test method is to be applied;

7.1.3 The difficulty and expense involved in obtaining, processing, and distributing samples or specimens;

7.1.4 The difficulty of, length of time required for, and expense of performing the tests; and

7.1.5 The uncertainty of prior information on any of these points. For example, if it is already known that the precision is relatively constant or proportional to the average level over the range of values of interest, a smaller number of materials will be needed than if it is known that the precision changes erratically at different levels. A preliminary pilot or screening program may help to settle some of these questions, and may often result in the saving of considerable time and expense in the full interlaboratory study (**1**).

7.2 In general, at least three materials or three different average values of the measured test characteristic is considered acceptable. The materials need to be selected to obtain as broad a range of the test characteristic as is practicable. If the test method is used to determine properties that are used for acceptance testing in a specification, it is particularly important that materials be included in the ILS whose properties are near the specification limits.

7.3 *Specimen Distribution*—The ILS is based on the assumption that all tests are performed on specimens that are as similar as is possible. Generally, two approaches are used for making and distributing the specimens or materials for the ILS.

7.3.1 For a test method that does not involve production of the test specimens as part of the method, specimens are produced at one location from a homogenous sample and then distributed to the participating laboratories. The specimens need to be assigned to the participating laboratories on a random basis. If the characteristic to be measured changes with age, specific instructions on test age need to be provided.

7.3.2 For a test method that involves fabrication of test specimens as part of the method, the raw materials for making the test specimens are shipped to the participating laboratories. In this case, samples of the constituent materials are taken from homogenous blends of the materials. The samples are selected on a random basis for shipment to the participating laboratories. Facilities are needed that have the proper equipment for blending the materials.

7.3.3 In some cases, it is not possible to distribute materials to participating laboratory because of the nature of the material or effects of transportation or age. This may require operators from participating laboratories to convene at one location to test the materials. This procedure is used commonly in developing precision statements for fresh concrete test methods.

8. Estimates of Precision

8.1 In accordance with Practice C670, the procedure described in this practice is designed to provide data to develop two basic estimates of the precision of a test method: (a) single-operator precision, and (b) multilaboratory precision (**1**). (See Note 1.)

8.2 *Single-operator precision* provides estimates of the inherent variability of the test method and the maximum difference that may be expected between replicate measurements made on the same material in the same laboratory by the

[3] The boldface numbers in parentheses refer to the list of references at the end of this practice.