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Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method with Multi-Valued Measurands[1]

This standard is issued under the fixed designation E2480; 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 the techniques for planning, conducting, and analyzing the results of an interlaboratory study (ILS) conducted for certain test methods within Committee E12.

1.2 This practice does not concern itself with the development of the test method but rather with the gathering of the information needed for the precision and bias statement after the completion of development of the test method. The data obtained in the ILS may indicate, however, that further effort is needed to improve the test method.

1.3 This practice is concerned exclusively with test methods that derive a multi-valued measurand, such as, but not limited to, spectral reflectance, transmittance function, tristimulus values, or RGB values. Variation in measurements of such multi-valued measurands are usually analyzed by reducing the data to a single-valued parameter, such as color difference, ΔE.

1.4 This practice covers methods of dealing with the non-normal distribution of the variation of sets of color-differences. This is done so that the user may derive valid statistics from such non-normal distributions.

1.5 This practice does not cover test methods, even in Committee E12, whose measurands are single-valued, or whose variations are known to be normally distributed. Task groups involved with such test methods are referred to Practice E691 which contains preferable methods of analyzing data with those properties.

1.6 This practice is not intended to establish a method for estimating possible color-difference tolerances.

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E284 Terminology of Appearance
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1345 Practice for Reducing the Effect of Variability of Color Measurement by Use of Multiple Measurements

3. Terminology

3.1 *Definitions*—For color and appearance terms, see Terminology E284.

3.2 *Definitions of Terms Specific to This Standard:*

[1] This practice is under the jurisdiction of ASTM Committee E12 on Color and Appearance and is the direct responsibility of Subcommittee E12.02 on Spectrophotometry and Colorimetry.

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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~~standard's Document Summary page on the ASTM website.

3.2.1 *precision and bias, n*—when a test method is applied to a large number of specimens that are as nearly alike as possible, the test results obtained nevertheless will not all have the same values. A measure of the degree of agreement among these test results describes the precision of the test method for that material. This practice is designed only to estimate the precision of a test method. However, when accepted reference values are known for the materials being tested, the test result data obtained in accordance with this practice may be used to estimate the bias of the test method. For a discussion of bias estimation, see Practice E177.

3.2.2 *repeatability and reproducibility, n*—the term repeatability concerns the variability between independent test results obtained within a single laboratory in the shortest practical period of time by a single operator applying the test method with a specific set of test apparatus using test specimens taken at random from a single quantity of homogeneous material obtained, or prepared, for the ILS. The term reproducibility concerns the variability between single test results obtained in different laboratories, each by a different operator, each of whom has applied the test method to specimens taken at random from a single quantity of homogeneous material obtained, or prepared, for the ILS.

3.2.2.1 *Discussion*—

The above single operator and single apparatus requirement, as specified in 3.2.2, means that for a particular step in the measurement process the same combination of operator and apparatus is used to obtain every test result on every specimen. Thus, one operator could prepare and mount the specimen, another actuate the measurements, and still another record the value of the result.

3.2.2.2 *Discussion*—

The shortest practical period of time means that the test results are obtained in a time not less than normal testing and not so long as to permit significant changes in material, equipment, calibration, or environment.

3.2.2.3 *Discussion*—

The requirement that the measurements be independent means that a single test determination begins with the mounting of the specimen on the sample port or in the transmission compartment, and ends with the removal of the test specimen from the port or compartment. All measurements are made with replacement.

3.2.2.4 *Discussion*—

The requirement for different laboratories does not exclude the case where more than one instrument resides in the same company, laboratory, or room, provided that each has an independent and separate calibration traceability path from each other.

3.2.3 *test method and protocol, n*—in this practice, the term test method applies to both the actual measurement process and the written description of the process, while the term protocol refers to the written instructions given the participants for conducting the ILS.

3.2.4 *test specimens, n*—the portion of the material being tested needed for obtaining a single test determination is called a test specimen. A single test specimen may be measured more than once and the results combined to produce a test result if the protocol or test method so specifies.

4. Summary of Practice

4.1 The procedure presented in this practice consists of three steps: planning the interlaboratory study, guiding the testing phase of the study, and analyzing the test result data. The analysis includes the calculation of the numerical measures of precision of the test method applying to both within-laboratory repeatability and between-laboratory reproducibility.

5. Significance and Use

5.1 ASTM regulations require precision statements for all test methods in terms of repeatability and reproducibility. This practice may be used in obtaining the information needed to prepare a precision statement in accordance with Practice E177 and the “Blue Book.”

PLANNING THE INTERLABORATORY STUDY (ILS)

6. ILS Membership

6.1 *Task Group*—Either the task group that developed the test method or a special task group formed specifically for the purpose must have overall responsibility for the funding, staffing, design, and decision-making with regard to data in the ILS. The task

group should decide on the number of laboratories, materials, and test results for the ILS. The task group should obtain a statement of willingness to participate from each of the participating laboratories. In addition, the task group should obtain, randomize, and distribute the specimens to be tested.

6.2 *ILS Coordinator*—The task group must appoint one individual to act as overall coordinator of the ILS. This person has responsibility for distributing the materials and protocols to the laboratories, and for receiving the test result reports from the laboratories.

6.3 *Statistician:*

6.3.1 The test method task group should obtain the assistance of a person familiar with the statistical procedures of this practice and with the materials being tested. When no such person is available, the task group should obtain the assistance of a statistician who has experience in practical work with data from materials. Task group members need not be members of ASTM.

6.3.2 The calculation of the statistics for each material may be readily done by persons not having knowledge of statistics, but having basic knowledge of calculating and computers.

6.4 *Laboratory ILS Supervisor*—Each participating laboratory must have an ILS supervisor to oversee the conduct of the ILS within the laboratory and to communicate with the ILS Coordinator. This supervisor's name should be obtained at the time that the laboratory states its willingness to participate.

7. Basic Design

7.1 Keep the design as simple as possible in order to obtain estimates of within- and between-laboratory variability that are free of secondary effects. The basic design is represented by a two-way classification table, in which the rows represent the laboratories, and the columns represent the materials, and each cell (the intersection of a row and a column) contains a test result made by a particular laboratory on a particular material.

8. Test Method

8.1 A written version of the test method (but not one necessarily as yet published as an ASTM standard) must have been developed and be distributed with the protocol if otherwise unavailable to the participating laboratories.

8.2 The test method should have been subjected to a screening procedure, in order that some experience with the test method has been obtained before an ILS is conducted. Test conditions that affect the test results, if any, should be identified and a statement of the needed degree of control of these conditions should be provided. In addition, the test method, or the protocol, should specify to how many digits of precision each test result is to be measured.

8.3 The test method should specify the calibration procedure and the frequency of calibration.

9. Laboratories

9.1 *Number of Laboratories*—An ILS should be run with no fewer than 8 laboratories. It is recommended that the number of laboratories be set at 10, and it is desirable that more laboratories be included if available in order that the ILS is conducted with a reasonable cross-section of competent laboratories. Under no circumstances, allowing for attrition, should the final statement of precision of a test method be based on fewer than 6 laboratories when the requisite three materials are employed.

9.1.1 Under some circumstances and with some test methods, it may be impossible to obtain the necessary six laboratories. Under these conditions, it is permissible to proceed with the supplementation of additional materials to make up for the loss of degrees of freedom using the following schedule of materials and laboratories:

Number of Labs	Required Minimum Number of Materials
6	3
5	4
4	5

9.2 The ILS should not be restricted to a group of laboratories judged to be exceptionally qualified and equipped for the ILS. Precision estimates for a test method should be obtained through conditions where laboratories are competent and personnel are operating under conditions that will prevail when the test method is used in practice.

10. Materials

10.1 The term material means anything with a property that can be measured. Different materials having the same property may be expected to have different levels of the property, meaning higher or lower levels of the property.

10.2 The ILS should include a minimum of three different materials each with a different levels of the property under test, and to be broadly applicable more than three materials of differing levels should be assessed.

10.3 The materials involved in any one ILS should differ primarily in the differing levels of the property being assessed by the test method. When it is known, or suspected, that different classes of materials will exhibit different levels of precision when tested by the test method, consideration should be given to conducting separate interlaboratory studies for each class of material.

10.4 The ILS should not be restricted to materials that are judged to be exceptionally qualified for the ILS. Precision estimates for a test method should be obtained through conditions where materials are competent for measurement of the property-under-test under conditions that will prevail when the test method is used in practice.

10.5 If more precise information is required about materials that are not so competent for measurement of the property being tested, those laboratories directly involved with the material in question must conduct interlaboratory studies specifically aimed at the material of interest.

11. Number of Test Results per Material

11.1 The minimum number of test results per laboratory on each material shall be four. The number may rise to as many as ten when test results are apt to vary considerably. The number of test results in any one ILS will be determined by the Task Group, based upon the desired level of tolerance and the anticipated variation of test results from the test method.

12. Protocol

12.1 Prepare a written protocol containing instructions for the participating laboratories to follow. Clearly identify the specific version of the test method being studied. If the test method allows options in apparatus or procedure, clearly specify which option has been selected for conducting the ILS.

12.2 Cite the name, address, telephone number, and E-mail address of the ILS Coordinator. Urge the participants to call the coordinator with any questions that may arise as to the conduct of the ILS.

12.3 Request that the participating laboratory keep a record (or log) of any special events that arise during any phase of the testing. This record should include any specific aspects of the apparatus, calibration, or procedure that ought to be communicated to the task group to allow them to prepare the final research report on the ILS.

12.4 Supply data sheets for each material for recording the raw data as observations are made, or if it would be more convenient for the participating laboratory, specify the format, including the number of significant digits to be recorded, of the data to be returned to the coordinator.

CONDUCTING THE TESTING PHASE OF THE ILS

13. Full Scale Run

13.1 *Material Preparation and Distribution:*

13.1.1 *Sample Preparation and Labeling*—Prepare enough material to supply 50 % more than needed by the number of laboratories committed to the ILS. Label each test specimen with the laboratory number and a letter designator referring to the material. Thus, if 8 laboratories were participating in a test concerning 6 levels of material (perhaps different colors of the same material), then specimens would be labeled from 1A through 1F, to 8A through 8F with the other laboratories similarly labeled between these limits.

13.1.2 *Randomization*—Prepare a table for each laboratory that randomizes the order in which that laboratory is to test its set of specimens. Using the above example, the random table for the first laboratory would include the 6 specimens from 1A to 1F in random order. Each of the 8 tables for the 8 participating laboratories would be different from each other. Use a random number table, or suitable computer randomization to prepare these tables.

13.1.3 *Shipping*—Ensure that the specimens are packaged properly and address the package explicitly to the person at the participating laboratory who is the ILS Supervisor.

13.1.4 *Follow-up*—Once the test units have been shipped, the ILS Coordinator should call each ILS Supervisor to confirm that all units have arrived safely and on-time.

13.2 *Checking Progress*—From time to time at appropriate intervals, the ILS Coordinator should call the ILS Supervisors to assure that progress is bring made in testing.

13.3 *Data Inspection*—The completed data sheets should be examined by the ILS Coordinator immediately upon receipt from the participating laboratory in order to detect missing, unusual, or obviously erroneous data while there is time remaining to correct it, if necessary.

CALCULATION OF THE STATISTICS

14. Calculation of the Statistics

14.1 For repeatability studies, a convenient way of handling the data is to prepare an exhaustive list of color difference components, for instance ΔL^*, Δa^*, Δb^*, for each possible combination of differences. In this data set, the first replication shall be differenced with the second through the last replication. Then the second replication shall be differenced with the third through the last, and so on, until all differing combinations have been treated. As an example, for 30 measurements a list of 435 differences