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An American National Standard

Standard Practice for Conducting an Interlaboratory Study to Determine Precision Estimates for a Test Method with Fewer Than Six Participating Laboratories[1]

This standard is issued under the fixed designation E2653; 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, analyzing, and treating results of an interlaboratory study (ILS) for estimating the precision of a test method when fewer than six laboratories are available to meet the recommended minimum requirements of Practice E691. Data obtained from an interlaboratory study are useful in identifying variables that require modifications for improving test method performance and precision.

1.2 Precision estimates developed using this practice will not be statistically equivalent to precision estimates produced by Practice E691 because a small number of laboratories are used. The smaller number of participating laboratories will seriously reduce the value of precision estimates reported by this practice. However, under circumstances where only a limited number of laboratories are available to participate in an ILS, precision estimates developed by this practice will provide the user with useful information concerning precision for a test method.

1.3 A minimum of three qualified laboratories is required for conducting an ILS using this practice. If six or more laboratories are available to participate in an ILS for a given test method, Practice E691 shall be used for conducting the ILS.

1.4 Since the primary purpose of this practice is the development of the information needed for a precision statement, the experimental design in this practice will not be optimum for evaluating all materials, test methods, or as a tool for individual laboratory analysis.

1.5 Because of the reduced number of participating laboratories, a Laboratory Monitor shall be used in the ILS. See Guide E2335.

1.6 *Field of Application*—This practice is concerned with test methods that yield numerical values or a series of numerical values for different properties associated with the test method. The numerical values mentioned above are typically the result of calculations from a set of measurements.

1.7 This practice includes design information suitable for use with the development of interlaboratory studies for test methods that have categorization (go-no-go) allocation test results. However, it does not provide a recommended statistical practice for evaluating the go-no-go data.

1.8 This practice cannot be used to provide quantitative measures.

1.9 This practice is issued under Committee E05, but it is generic in its statistical approach such that it is applicable to any other method.

1.10 *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.*

1.11 *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]

E176 Terminology of Fire Standards

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

[1] This practice is under the jurisdiction of ASTM Committee E05 on Fire Standards and is the direct responsibility of Subcommittee E05.31 on Terminology and Services / Functions.

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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.

Determine the Precision of a Test Method
E1169 Practice for Conducting Ruggedness Tests
E2335 Guide for Laboratory Monitors

3. Terminology

3.1 *Definitions*—For formal definitions of statistical terms, see Terminology E456. For formal definitions of fire terms, see Terminology E176.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *protocol, n—in this practice,* directions given to the laboratories for conducting the interlaboratory study (ILS).

3.2.2 *repeatability (of results and measurements), n*—quantitative expression of the random variability associated with successive measurements of the same measurand carried out subject to all of the following conditions: the same measurement procedure, the same observer, the same measuring instrument, used under the same conditions, the same location, and repetition over a short period of time.

3.2.2.1 *Discussion*—Repeatability deals with results in a single laboratory while reproducibility deals with results obtained in different laboratories.

3.2.3 *reproducibility (of results of measurements), n*—quantitative expression of the random variability associated with successive measurements of the same measurand carried out by operators working in different laboratories, each obtaining single results on identical test material when applying the same method.

3.2.3.1 *Discussion*—Repeatability deals with results in a single laboratory while reproducibility deals with results obtained in different laboratories.

3.2.4 *test method, n—in this practice,* description of the actual measurement process as well as written description of the process.

3.3 For further discussion of the terms discussed above, see Practice E177 and the formal definitions in Terminology E456.

4. Summary of Practice

4.1 The procedure presented in this practice consists of three basic steps: planning the interlaboratory study, guiding the testing phase of the study, and analyzing the test result data. The analysis evaluates the consistency of the data through the use of numerical estimates of precision of the test method pertaining to both within-laboratory repeatability and between-laboratory reproducibility.

4.2 Planning of the interlaboratory study will include a review of the test procedure to be used in the interlaboratory study. This review will identify portions of the test method that appear to contribute to a loss in precision. Special interlaboratory instructions or modifications to the test method wording are made as needed to clarify these sections and often result in a modification to the test method following the interlaboratory study.

4.3 A manager for the interlaboratory study and an interlaboratory test monitor shall be selected. The same person is allowed to conduct both functions.

4.4 Parties conducting an interlaboratory precision study of a test method will acquire participation agreements with as many laboratories as possible that are willing to take part in the interlaboratory study and have the capability to run the test method of interest. A minimum of three laboratories shall participate in the precision study. Precision results will increase in quality with a larger number of participating laboratories.

4.5 The types of materials and number of test specimens shall be selected for the interlaboratory study. No less than three test specimens shall be selected for the interlaboratory study, and they shall be selected to reflect the range of performance of test specimens normally evaluated by the test method. A minimum of three replicates shall be tested for each test material selected. If a standard reference material is available for the test method, the material shall be included as a specimen in the interlaboratory study. If a standard reference material is not available, a test specimen that consistently produces low variability test results shall be selected as a reference material for the interlaboratory study.

5. Significance and Use

5.1 ASTM regulations require precision statements in all test methods in terms of repeatability and reproducibility. This practice is used when the number of participating laboratories or materials being tested, or both, in a precision study is less than the number specified by Practice E691. When possible, it is strongly recommended that a full Practice E691 standard protocol be followed to determine test method precision. Precision results produced by the procedures presented in this standard will not have the same degree of accuracy as results generated by a full Practice E691 protocol. This procedure will allow for the development of useful precision results when a full complement of laboratories is not available for interlaboratory testing.

5.2 This practice is based on recommendations for interlaboratory studies and data analysis presented in Practice E691. This practice does not concern itself with the development of test methods but with a standard means for gathering information and treating the data needed for developing a precision statement for a test method when a complete Practice E691 interlaboratory study and data analysis are not possible.

PLANNING THE ILS

6. Planning

6.1 *Task Group*—Either the task group that developed the test method or a special task group appointed for the purpose must have overall responsibility for the ILS, including funding where appropriate, staffing, the design of the ILS, and decision-making with regard to questionable data. The task group shall decide on the number of laboratories, materials, and test results for each material. In addition, it shall specify any special calibration procedures and the repeatability conditions to be specified in the protocol.

6.2 *ILS Coordinator*—The task group must appoint one individual to act as overall coordinator for conducting the ILS. The coordinator will supervise the distribution of materials and protocols to the laboratories and receive the test result reports

from the laboratories. Scanning the reports for gross errors and checking with the laboratories, when such errors are found, will also be the responsibility of the coordinator. The coordinator will consult as needed with a statistician in questionable cases.

6.3 *Laboratory Monitor*—The task group must appoint one individual to act as a laboratory monitor for the ILS. The laboratory monitor will develop an ILS checklist specific to the test method, inspect the test laboratories for equipment conformity and operator training, verify compatibility of the data acquisition system, and based on the Checklist and inspection results report to the sponsoring ASTM Subcommittee. Complete details for the function of a laboratory monitor are located in Guide E2335.

6.4 *Statistician*—The task group shall obtain the assistance of a person skilled in the use of statistical procedures, the test method being studied, and with the materials being tested in order to ensure that the requirements in this practice are met in an efficient and effective manner. This person will conduct the data analysis using procedures given in this standard and will assist the task group in interpreting results from the data analysis.

7. Basic Design

7.1 Keep the ILS design simple 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, the columns represent the materials, and the cell (the intersection of a row and column) contains the test results made by a particular laboratory on a particular material (see Table 1).

7.2 An ILS using this practice shall include enough laboratories to represent a reasonable cross-section of the population of qualified laboratories. A minimum of three laboratories is necessary for carrying out an ILS using this practice.

8. Test Method

8.1 Of prime importance is the existence of a valid, well-written test method that has been developed in one or more competent laboratories, and had been subjected to a ruggedness test prior to the ILS.

TABLE 1 Example, ILS Test Result Data

Laboratory	A	B	C	D	E
1	35.3	31.2	38.9	34.0	27.2
	34.0	31.0	35.0	35.5	31.1
	35.5	35.1	50.8	63.1	27.3
2	10.7	12.9	20.6	19.9	15.0
	12.7	15.0	8.0	16.2	8.2
	13.3	12.2	16.2	8.1	12.3
3	36.0	28.0	32.1	32.1	25.1
	36.0	32.1	36.0	32.0	25.0
	29.0	28.0	32.2	32.0	21.2
4	40.9	36.8	32.8	36.7	24.5
	36.7	32.7	28.6	32.7	24.4
	28.6	32.7	32.6	32.7	28.5
5	41.6	37.6	33.2	41.6	29.0
	41.7	25.1	29.2	37.5	29.1
	46.0	29.3	29.0	37.3	29.2

8.2 The ruggedness test is a screening procedure for investigating the effects of variations in environmental and other conditions in order to determine how control of such test conditions shall be specified in the written description of the method. Details for ruggedness testing are found in Guide E1169.

8.3 A written version of the test method must be developed for the ILS (but not necessarily published as a standard method). This draft shall describe the test apparatus and procedure in terms that are easily understood and followed in any properly equipped laboratory by competent personnel with knowledge of the materials and the property to be tested. The method shall contain safety and calibration procedures, details on control related limits that potentially cause test result variability, and specify how test results are to be reported.

ILS TESTING

9. Pilot Run

9.1 Prior to beginning testing for the formal ILS a preliminary laboratory evaluation study shall be carried out using a well characterized test material of known performance. This preliminary study is managed by the ILS Coordinator and Laboratory Monitor and is used to determine if each of the participating laboratories are capable of conducting tests as specified by the written ILS test method. These preliminary tests conducted in the participating laboratories are typically observed by the Laboratory Monitor as a part of the laboratory qualification process.

9.2 The pilot run results give the task group an indication of how well each laboratory will perform in terms of promptness and following the protocol. Laboratories with poor performance are encouraged and helped to take corrective action.

9.3 All steps of the procedures described in this practice shall be followed in detail to ensure that these directions are understood, to disclose any weakness in the protocol or test method.

10. Full Scale Run

10.1 *Materials Preparation and Distribution:*

10.1.1 *Sample Preparation and Labeling*—Prepare enough of each material to supply at least 50 % more than needed by the number of laboratories committed to the ILS. Label each test unit or specimen with a letter for the material and a sequential number. Thus, for three laboratories and three results for each laboratory the test units for materials B would be numbered from B1 to B14.

10.1.2 *Randomization*—For each material independently, allocate the specified number of test units or test specimens to each laboratory, using a random number table, or a suitable computerized random number based program.

10.1.3 *Shipping*—Ensure that the test specimens are packaged properly to arrive in the desired condition. Clearly indicate the name of the person who has been designated as ILS supervisor at the laboratory on the address of each package. Follow shipping directions provided by each laboratory to ensure prompt delivery of the package.

10.1.4 *Follow-up*—Once the test specimens have been shipped, the ILS coordinator shall call each laboratory ILS supervisor to confirm that all test specimens have arrived safely. If the task group has decided to intermingle test specimens from different materials in the order of testing, the testing shall not start until all the test specimens have arrived at the laboratory so they will be tested in the specified order.

10.1.5 *Replacement Sets of Test Specimens*—As the ILS progresses, it is possible that a laboratory will discover that the test method was not used properly on some test specimens. The laboratory ILS supervisor shall discuss this with the ILS coordinator, who has the option to send a replacement set of test specimens, replace the misused test units, or do nothing.

10.1.6 *Checking Progress*—From time to time, at intervals appropriate to the magnitude of the ILS, the coordinator shall call each ILS supervisor to determine how the testing is progressing. Laboratories found to be lagging behind shall be informed.

10.1.7 *Data Inspection*—The completed data sheets shall be examined by the coordinator immediately upon receipt in order to detect unusual values or other deficiencies that shall be questioned. With agreement between the ILS coordinator and the specific laboratory ILS supervisor, replacement sets of test specimens or specific test specimens shall be sent when there is missing or obviously erroneous data. The task group shall decide later whether or not the additional data shall be used in the estimation of precision of the test method.

CALCULATION AND DISPLAY OF STATISTICS

11. Calculation of Statistics

11.1 The statistical analysis of the data for estimates of precision is a simple one-way analysis of variance (within and between laboratories) carried out separately for each material type tested in the ILS. Severe outliers will invalidate this analysis; therefore, it is necessary to first examine the consistency of data gathered from the laboratories. If data suggest that outliers exist, use Practice E178 for the handling of outliers.

11.1.1 For calculations using these procedures, retain extra significant digits in order to ensure that statistically important information is not lost in calculation by rounding off too soon. As a general rule, retain at least two more digits in the averages than in the reported test results and at least three significant figures in the standard deviations.

11.1.2 While the calculations described in this section are arranged for use with a hand calculator; alternatively, they are also readily programmed for use in a computer. These calculations are also adaptable to spreadsheet operations.

11.1.3 *Nomenclature:*

(CV_r) = coefficient of variation for repeatability (within-laboratory)
(CV_R) = coefficient of variation for reproducibility (between-laboratory)
d_{ij} = cell deviations from average
n_{ij} = number of replicates per cell
p = total number of laboratories
s_{ij} = cell standard deviation
$(s_L)_j$ = component of variance between laboratories
$(s_r)_j$ = pooled standard deviation for repeatability
$(s_R)_j$ = standard deviation for reproducibility
x = individual test result
$\bar{x}_{ij}$ = average for cell $(i, j) = \sum_1^n x/n$ where n = number of test results per cell, i represents the laboratory, and j the material
$\bar{x}_j$ = average for the jth material for all laboratories

11.1.4 Each material/laboratory raw data set is arranged into a table grouping of replicate test results producing laboratory data cells (Table 1). The mean average ($\bar{x}$) is then calculated for each cell (i, j) of data where i represents the laboratory and j represents the material (Table 2).

11.1.5 Data cell standard deviations (s_{ij}) are calculated using Eq 1:

$$s_{ij} = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n_{ij} - 1}} \quad (1)$$

11.1.6 *Repeatability*—The pooled standard deviation for repeatability $(s_i)_j$ for the jth material is calculated using Eq 2. This equation is applicable only when the number of replicates is the same for each laboratory for a given material.

$$(s_r)_j = \sqrt{\frac{1}{p} \sum_i s_{ij}^2} \quad (2)$$

11.1.6.1 The pooled standard deviation for repeatability where there are missing replicates in one or more of the laboratories use Eq 3.

$$(s_r)_j = \sqrt{\frac{\sum_i (n_{ij} - 1)\, s_{ij}^2}{\sum_i (n_{ij} - 1)}} \quad (3)$$

11.1.6.2 *Coefficient of Variation for Repeatability (CV_r)*—The coefficient of variation for repeatability is calculated using Eq 4.

$$(CV_r) = 100 \frac{(s_r)_j}{\bar{x}_j} \quad (4)$$

11.1.6.3 *Repeatability Limit (r)*—The repeatability limit (r) is defined as $2.8 \times (s_r)_j$, the pooled repeatability standard deviation. This provides the value below which the absolute difference between two single tests obtained under repeatability conditions are expected to lie with a probability of approximately 95 %.

TABLE 2 Example, ILS Cell Averages Ordered from Lowest to Highest

Laboratory	A	B	C	D	E
1	28.5	32.4	41.6	34.9	44.2
2[A]					
3	23.8	29.4	33.4	33.7	32.0
4	25.8	34.1	31.3	35.4	34.0
5	29.1	30.7	30.5	43.1	38.8
Column Average	26.8	31.7	34.2	36.8	37.3

[A] Using Practice E178, Laboratory 2 is determined to be an outlier and is not included in the above average or further calculations.