



# Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>1</sup>

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*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope

1.1 This practice describes the techniques for planning, conducting, analyzing, and treating the results of an interlaboratory study (ILS) of a test method. The statistical techniques described in this practice provide adequate information for formulating the precision statement of a test method.

1.2 This practice does not concern itself with the development of test methods but rather with gathering the information needed for a test method precision statement after the development stage has been successfully completed. The data obtained in the interlaboratory study may indicate, however, that further effort is needed to improve the test method.

1.3 Since the primary purpose of this practice is the development of the information needed for a precision statement, the experimental design in this practice may not be optimum for evaluating materials, apparatus, or individual laboratories.

1.4 *Field of Application*—This practice is concerned exclusively with test methods which yield a single numerical figure as the test result, although the single figure may be the outcome of a calculation from a set of measurements.

1.4.1 This practice does not cover methods in which the measurement is a categorization; however, for many practical purposes categorical outcomes can be scored, such as zero-one scoring for binary measurements or as integers, ranks for example, for well-ordered categories and then the test result can be defined as an average, or other summary statistic, of several individual scores.

1.5 *This standard may involve hazardous materials, operations, and equipment. 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.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E11 on Quality and Statistics and is the direct responsibility of Subcommittee E11.20 on Test Method Evaluation and Quality Control.

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1.6 *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:<sup>2</sup>

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E1169 Practice for Conducting Ruggedness Tests

E1402 Guide for Sampling Design

E2282 Guide for Defining the Test Result of a Test Method

## 3. Terminology

3.1 *Definitions*—Terminology E456 provides a more extensive list of terms in E11 standards. [730/astm-e691-22](#)

3.1.1 *accuracy, n*—the closeness of agreement between a test result and an accepted reference value. **E177**

3.1.2 *bias, n*—the difference between the expectation of the test results and an accepted reference value. **E177**

3.1.3 *interlaboratory study, (ILS) in ASTM, n*—a designed procedure for obtaining a precision statement for a test method, involving multiple laboratories, each generating replicate test results on one or more materials.

3.1.4 *observation, n*—the process of obtaining information regarding the presence or absence of an attribute of a test specimen, or of making a reading on a characteristic or dimension of a test specimen. **E2282**

3.1.5 *precision, n*—the closeness of agreements between independent test results obtained under stipulated conditions. **E177**

<sup>2</sup> 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.

3.1.6 *repeatability, n—precision* of test results from tests conducted within the shortest practical time period on identical material by the same *test method* in a single laboratory with all known sources of variability conditions controlled at the same levels (see *repeatability conditions*). **E177**

3.1.7 *repeatability conditions, n*—conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time. **E177**

3.1.8 *repeatability limit (r), n*—the value below which the absolute difference between two individual test results obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95 %). **E177**

3.1.9 *repeatability standard deviation, (s<sub>r</sub>), n*—the standard deviation of test result obtained under repeatability conditions. **E177**

3.1.10 *reproducibility, n—precision* of test results from tests conducted on identical material by the same *test method* in different laboratories (see *reproducibility conditions*). **E177**

3.1.11 *reproducibility conditions, n*—conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. **E177**

3.1.12 *reproducibility limit (R), n*—the value below which the absolute difference between two test results obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95 %). **E177**

3.1.13 *reproducibility standard deviation (s<sub>R</sub>), n*—the standard deviation of test results obtained under reproducibility conditions. **E177**

3.1.14 *ruggedness test, n*—a planned experiment in which environmental factors or test conditions are deliberately varied in order to evaluate the effects of such variation. **E1169**

3.1.15 *test determination, n*—the value of a characteristic or dimension of a single test specimen derived from one or more observed values. **E2282**

3.1.16 *test method, n*—a definitive procedure that produces a test result. **E2282**

3.1.17 *test observation, n*—see *observation*. **E2282**

3.1.18 *test result, n*—the value of a characteristic obtained by carrying out a specified test method. **E2282**

3.1.19 *test specimen, n*—the portion of a test unit needed to obtain a single test determination. **E2282**

3.1.20 *test unit, n*—the total quantity of material (containing one or more test specimens) needed to obtain a test result as specified in the test method; see *test result*. **E2282**

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *average of the cell averages,  $\bar{\bar{x}}$ , n*—the average of the cell averages for a particular material.

3.2.2 *between-laboratory consistency statistic, h, n*—the ratio of the cell deviation to the standard deviation of the cell averages.

3.2.2.1 *Discussion*—This statistic is an indicator of how one

laboratory's cell average compares with the average of the other laboratories for a particular material (see A1.2.2).

3.2.3 *between-laboratory standard deviation, s<sub>L</sub>, n*—the sample standard deviation attributable to differences of test result means among laboratories.

3.2.4 *between-laboratory variance, s<sub>L</sub><sup>2</sup>, n*—the sample variance component attributable to differences of test result means among laboratories.

3.2.4.1 *Discussion*—This statistic is estimated indirectly from the variance of cell averages and the repeatability variance. In situations where there is good agreement among laboratories the estimate of this variance component may be close to zero or be negative. In the latter case, the estimate is set to zero. (See Note 2 and A1.1.2.)

3.2.5 *cell, n*—the intersection of a row and column in a two-way classification table, in which the rows represent the laboratories and the columns represent the materials.

3.2.5.1 *Discussion*—The table holds the test results from an interlaboratory study, and each cell contains the test results from a particular laboratory on a particular material (see Section 7 and Table 1).

3.2.6 *cell average,  $\bar{x}$ , n*—the average of the test results in a particular cell.

3.2.7 *cell deviation, d, n*—the cell average minus the average of the cell averages.

3.2.8 *cell standard deviation, s, n*—the standard deviation of the test results in a particular cell.

3.2.9 *repeatability variance, s<sub>r</sub><sup>2</sup>, n*—the sample variance of test results obtained under repeatability conditions.

3.2.9.1 *Discussion*—This statistic is estimated for a material as the pooled within-laboratory variances over all of the laboratories in the ILS.

3.2.10 *reproducibility variance, s<sub>R</sub><sup>2</sup>, n*—the sample variance of test results obtained under reproducibility conditions.

3.2.10.1 *Discussion*—This statistic is estimated as the sum of the two variance components due to between-laboratories, s<sub>L</sub><sup>2</sup>, and within-laboratories, s<sub>r</sub><sup>2</sup>.

3.2.11 *standard deviation of the cell averages, s <sub>$\bar{x}$</sub> , n*—the standard deviation of the cell averages for a particular material.

3.2.12 *variance of the cell averages, s <sub>$\bar{x}$</sub> <sup>2</sup>, n*—the sample variance of the cell averages for a particular material.

3.2.13 *within-laboratory consistency statistic, k, n*—the ratio of the cell standard deviation to the repeatability standard deviation.

3.2.13.1 *Discussion*—This statistic is an indicator of how one laboratory's cell standard deviation under repeatability conditions compares with the repeatability standard deviation estimated from all laboratories for a particular material (see A1.2.3).

## 4. Significance and Use

4.1 ASTM regulations require precision statements in all test methods in terms of repeatability and reproducibility. This practice may be used in obtaining the needed information as simply as possible. This information may then be used to

prepare a precision statement in accordance with Practice E177. Knowledge of the test method precision is useful in commerce and in technical work when comparing test results against standard values (such as specification limits) or between data sources (different laboratories, instruments, etc.).

4.1.1 When a test method is applied to a large number of portions of a material that are as nearly alike as possible, the test results obtained will not all have the same value. A measure of the degree of agreement among these test results describes the precision of the test method for that material. Numerical measures of the variability between such test results provide inverse measures of the precision of the test method. Greater variability implies smaller (that is, poorer) precision and larger imprecision.

4.1.2 Precision is reported as a standard deviation, coefficient of variation (relative standard deviation), variance, or a precision limit (a data range indicating no statistically significant difference between test results).

4.1.3 This practice is designed only to estimate the precision of a test method. However, when accepted reference values are available for the property levels, the test result data obtained according to this practice may be used in estimating the bias of the test method. For a discussion of bias estimation and the relationships between precision, bias, and accuracy, see Practice E177.

4.2 The procedures presented in this practice consist of three basic steps: planning the interlaboratory study, guiding the testing phase of the study, and analyzing the test result data.

4.2.1 The planning phase includes forming the ILS task group, the study design, selection, and number of participating laboratories, selection of test materials, material certifications if applicable, and writing the ILS protocol. A well-developed test method is essential, so including a ruggedness test to determine control of test method conditions is highly recommended.

NOTE 1—In this practice, the term *test method* is used both for the actual measurement process and for the written description of the process, while the term *protocol* is used for the directions given to the laboratories for conducting the ILS.

4.2.2 The testing phase includes material preparation and distribution, liaison with the participating laboratories, and handling of test result data received from the laboratories.

4.2.3 The data analysis utilizes tabular, graphical, and statistical diagnostic tools for evaluating the consistency of the data so that unusual values may be detected and investigated, and also includes the calculation of the numerical measures of precision of the test method pertaining to repeatability and reproducibility.

4.3 The information in this practice is arranged as follows:

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## 5. Concepts of Test Method Precision

5.1 *Repeatability and Reproducibility*—These two terms deal with the variability of test results obtained under specified laboratory conditions and represent the two extremes of test method precision. Repeatability concerns the variability between independent test results obtained within a single laboratory in the shortest practical period of time by a single operator with a specific set of test apparatus using test specimens (or test units) taken at random from a single quantity of homogeneous material obtained or prepared for the ILS. Reproducibility deals with the variability between single test results obtained in different laboratories, each of which has applied the test method to test specimens (or test units) taken at random from a single quantity of homogeneous material obtained or prepared for the ILS.

5.1.1 *Repeatability Conditions*—The single-operator, single-set-of-apparatus requirement means that for a particular step in the measurement process the same combination of operator and apparatus is used for every test result and on every material. Thus, one operator may prepare the test specimens, a second measure the dimensions and a third measure the breaking force. “Shortest practical period of time” means that the test results, at least for one material, are obtained in a time not less than in normal testing and not so long as to permit significant changes in test material, equipment or environment.

5.1.2 *Reproducibility Conditions*—The factors that contribute to variability in a single laboratory, such as operator, equipment used, calibration of the equipment, and environment

(for example, temperature, humidity, air pollution) will generally have different effects in other laboratories, and the variability among laboratories will be greater.

**5.2 Observations, Test Determinations, and Test Results**—A test method often has three distinct stages: the direct observation of dimensions or properties, the arithmetic combination of the observed values to obtain a test determination, and the arithmetic combination of a number of test determinations to obtain the test result of the test method.

**5.2.1** In the simplest of test methods a single direct observation is both the test determination and the test result. For example, the test method may require the measurement of the length of a test specimen dimension, which then becomes the test result.

**5.2.2** A test determination may involve a combination of two or more observations. For example, a test method may require the measurement of the mass and the volume of the test specimen, and then direct that the mass be divided by the volume to obtain the density of the specimen. The whole process of measuring the mass and the volume, and calculating the density, is a test determination.

**5.2.2.1** If the test method specifies that only one test determination is to be made, then the test determination value is the test result of the test method. Some test methods require that several determinations be made and the values obtained be averaged or otherwise combined to obtain the test result of the test method. Averaging of several determinations is often used to reduce the effect of local variations of the property within the material.

**5.2.2.2** In this practice, the term *test determination* is used both for the process and for the value obtained by the process, except when *test determination value* is needed for clarity.

**5.2.3** The test result is the final reportable value of the test method. The precision of a test method is determined from test results, not from test determinations or observations.

**5.2.3.1** The number of test results conducted by each laboratory on a material that is required for an interlaboratory study of a test method is specified in the protocol of that study.

**5.2.4 Test Specimens and Test Units**—In this practice a test unit is the total quantity of material needed for obtaining a test result as specified by the test method. The portion of the test unit needed for obtaining a single test determination is called a test specimen. Usually a separate test specimen is required for each test determination.

## PLANNING THE INTERLABORATORY STUDY (ILS)

### 6. ILS Membership

**6.1 Task Group**<sup>3</sup>—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 should decide on the number of laboratories, materials, and test

results for each material. In addition, it should specify any special calibration procedures and the repeatability conditions to be specified in the protocol (see 12.3 and 12.4).

**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 may wish to consult with the statistician in questionable cases.

#### 6.3 Statistician:

**6.3.1** The test method task group should obtain the assistance of a person familiar with the statistical procedures in this practice and with the materials being tested in order to ensure that the requirements outlined in this practice are met in an efficient and effective manner. This person should also assist the task group in interpreting the results of the data analysis.

**6.3.2** When a person having adequate knowledge of both the materials and the proper statistical techniques is not available, the task group should obtain the services of a statistician who has experience in practical work with data from materials testing. The task group should provide the statistician with an opportunity to become familiar with the statistical procedures of this practice and with both the materials and the test method involved. The statistician should become a member of the task group conducting the ILS (task group members need not be members of ASTM).

**6.3.3** The calculations of the statistics (see Section 15) for each material can be readily done by persons not having statistical knowledge (see 15.1.3 and 15.4.2).

**6.4 Data Analyst**—This individual should be someone who is careful in making calculations and can follow the directions in Sections 15 through 17.

**6.5 Laboratory ILS Supervisor**—Each laboratory must have an ILS supervisor to oversee the conduct of the ILS within the laboratory and to communicate with the ILS Coordinator. The name of the supervisor should be obtained on the response form to the “invitation to participate” (see 9.4).

### 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, the columns represent the materials, and each cell (that is, the intersection of a row with a column) contains the test results made by a particular laboratory on a particular material (see Table 1).

### 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. It is highly recommended that the test method be subjected to a ruggedness test prior to the ILS.

<sup>3</sup> To facilitate the preparation of the final report on the ILS, the task group can obtain the Research Report format guide from ASTM Headquarters.

8.2 A ruggedness test is a screening procedure for investigating the effects of variations in environmental or other conditions in order to determine how control of such test conditions should be specified in the written description of the method. For example, the temperature of the laboratory or of a heating device used in the test may have an effect that cannot be ignored in some cases but may be much less in others. In a ruggedness test, deliberate variations in temperature would be introduced to establish the allowable limits on control of temperature. This subject is discussed more fully in Practice E1169.

8.3 As a result of carrying out the screening procedure, and of some experience with the test method in the sponsoring laboratory and one or two other laboratories, a written version of the test method must have been developed (but not necessarily published as a standard method). This draft should describe the test procedure in terms that can be easily followed in any properly equipped laboratory by competent personnel with knowledge of the materials and the property to be tested. The test conditions that affect the test results appreciably should have been identified and the proper degree of control of the test conditions specified in the description of the test procedure. In addition, the test method should specify how closely (that is, to how many digits) each observation in the test method is to be measured.

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

## 9. Laboratories

### 9.1 *Number of Laboratories:*

9.1.1 An ILS should include 30 or more laboratories but this may not be practical and some ILS have been run with fewer. It is important, that enough laboratories be included in the ILS to be a reasonable cross-section of the population of qualified laboratories; that the loss or poor performance of a few will not be fatal to the study, and to provide a reasonably satisfactory estimate of the reproducibility.

9.1.2 **Under no circumstances should the final statement of precision of a test method be based on acceptable test results for each material from fewer than 6 laboratories.** This would require that the ILS begin with 8 or more laboratories in order to allow for attrition.

9.1.3 The examples given in this practice include only 8 and 7 laboratories, respectively. These numbers are smaller than ordinarily considered acceptable, but they are convenient for illustrating the calculations and treatment of the data.

9.2 Any laboratory considered qualified to run the test routinely (including laboratories that may not be members of ASTM) should be encouraged to participate in the ILS, if the preparatory work is not excessive and enough suitably homogeneous material is available. In order to obtain an adequate number of participating laboratories, advertise the proposed ILS in where appropriate (for example, trade magazines, meetings, circulars, etc.).

9.3 “Qualified” implies proper laboratory facilities and testing equipment, competent operators, familiarity with the test method, a reputation for reliable testing work, and sufficient

time and interest to do a good job. If a laboratory meets all the other requirements, but has had insufficient experience with the test method, the operator in that laboratory should be given an opportunity to familiarize himself with the test method and practice its application before the ILS starts. For example, this experience can be obtained by a pilot run (see Section 13) using one or two trial samples provided by the task group and returning the raw data and the test results to the task group. **The importance of this familiarization step cannot be overemphasized.** Many interlaboratory studies have turned out to be essentially worthless due to lack of familiarization.

9.4 Obtain written assurance from each potential participating laboratory that it is properly equipped to follow all the details of the procedure and is willing to assign the work to a skilled operator in a timely manner. The decision of a laboratory to participate should be recorded on a response form to a written invitation. The invitation should include information covering the required time for calibrating the apparatus and for testing all of the materials, and other possible costs. The response form should include the name, address, and telephone number of the person supervising the ILS work within the laboratory, the address and other markings required to ensure the ILS sample material will be promptly delivered to the ILS supervisor, answers to brief questions concerning equipment, environment, and personnel, including previous use of the test method, upon which the apparent competence of the laboratory may be judged, and an affirmation that the laboratory understands what is involved and agrees to carry out its responsibilities with diligence.

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

## 10. Materials

10.1 Material designates anything with a property that can be measured. Different materials having the same property may be expected to have different property levels, meaning higher or lower values of the property. Different dilutions of the same material or compound to be assayed are considered “different materials” for the purpose of this practice. The terminology “different levels of material” may be used, if appropriate.

10.2 The number and type of materials to be included in an ILS will depend on the range of the levels in the class of materials to be tested and likely relation of precision to level over that range, the number of different types of materials to which the test method is to be applied, the difficulty and expense involved in obtaining, processing, and distributing samples, the difficulty of, length of time required for, and expense of performing the test, the commercial or legal need for obtaining a reliable and comprehensive estimate of precision, and the uncertainty of prior information on any of these points.

10.2.1 For example, if it is already known that the precision is either 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 merely known that the precision is different at different levels. The ruggedness test (see 8.2) and the preliminary pilot program (see Section 13) help to settle some of these questions, and may often result in the saving of considerable time and expense in the full ILS.

10.2.2 An ILS of a test method should include at least three materials representing different test levels, and for development of broadly applicable precision statements, six or more materials should be included in the study.

10.2.3 The materials involved in any one ILS should differ primarily only in the level of the property measured 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.

10.3 Each material in an ILS should be made to be or selected to be as homogeneous as possible prior to its subdivision into test units or test specimens. If the randomization and distribution of individual test specimens (rather than test units) does not conflict with the procedure for preparing the sample for test, as specified in the test method, greater homogeneity between test units can be achieved by randomizing test specimens. Then each test unit would be composed of the required number of randomized test specimens. (See Section 11 and 14.1 for the quantity of each material needed, its preparation and distribution.)

NOTE 2—It may be convenient to use established reference materials, since their homogeneity has been demonstrated.

## 11. Number of Test Results per Material

11.1 In the design of an ILS a sufficient total number of test results on each material must be specified to obtain a good estimate of the measure of repeatability, generally the repeatability standard deviation. In many cases, the standard deviation in question will be a function of the property level being measured. When this occurs, the standard deviation should be determined separately for each level. It is generally sound to limit the number of test results on each material in each laboratory to a small number, such as three or four. The minimum number of test results per laboratory will normally be three for a chemical test and three or four for a physical or optical test. The number may be as small as two when there is little danger that a test unit will be lost or questionable test results obtained, or as many as ten when test results are apt to vary considerably. Generally, the time and effort invested in an ILS is better spent on examining more materials across more laboratories than on recording a large number of test results per material within a few laboratories.

## 12. Protocol

12.1 In the protocol, cite the name, address, and telephone number of the person who has been designated ILS coordinator (see 6.2). Urge the laboratories to call the coordinator when any questions arise as to the conduct of the ILS.

12.2 Clearly identify the specific version of the test method being studied. If the test method allows several options in

apparatus or procedure, the protocol should specify which option or options have been selected for the ILS. Test units and test data sheets must be provided for each option.

12.3 When special calibration procedures are required before every determination or every test result, they should be described specifically in the test method. If the test method specifies calibration only daily or less frequently, the ILS task group must decide whether to require recalibration before obtaining each test result. While doing so will eliminate calibration drift and help ensure relative independence of the test results, changes in calibration may increase the variability between test results.

12.4 Describe any special circumstances that must be addressed in implementing the repeatability conditions, such as the period of time between obtaining the test results for the same material; that is, not less than in normal testing and not so long as to likely permit significant changes in test material, equipment or environment.

12.5 Specify the requirements for acquisition, shipment, documentation (including any material certifications), care, handling, and conditioning of the materials to be tested. Explain the coding system used in identifying the materials and the distinction between test units and test specimens, where appropriate.

12.6 Supply data sheets for each material for recording the raw data as observations are made. Give instructions on the number of significant digits to be recorded, usually one more, if possible, than required by the test method. Also, supply test result sheets on which test results can be calculated and reported. In many instances this can be combined with the raw data sheet. Specify the number of significant digits to be reported, usually two more than required by the test method. Request the laboratories send raw data and test result sheets as soon as the testing is completed, and at least weekly if testing will continue over several weeks. For guidance on the number of significant digits needed for data reporting see Practice E29.

12.7 Request that each laboratory keep a record (or log) of any special events that arise during any phase of the testing. This record, to be sent to the ILS coordinator, will provide a valuable source of information both in dealing with unusual data and in making improvements in the test method in future revisions.

12.7.1 Instruct the laboratories to notify the ILS coordinator promptly whenever an error in test procedure arises, so that a decision can be made as to whether a new set of test units should be sent to the laboratory for a complete retest of the material.

12.8 Enclose with the protocol a questionnaire requesting information on specific aspects of the apparatus, reagents, calibration, or procedure, as well as any other information that might assist in dealing with data inconsistencies, or ensure the task group that the laboratory complied with the current requirements of the test method. Also obtain any other information that may be needed in preparing the final research report on the ILS.

## CONDUCTING THE TESTING PHASE OF THE ILS

### 13. Pilot Run

13.1 Before investing laboratory time in the full scale ILS, it is usually wise to conduct a pilot run with only one, or perhaps two, material(s) to determine whether the test method as well as the protocol and all the ILS procedures are clear, and to serve as a familiarization procedure for those without sufficient experience with the method (see 9.3). The results of this pilot run also 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 should be encouraged and helped to take corrective action.

13.2 All steps of the procedures described in this practice should be followed in detail to ensure that these directions are understood, and to disclose any weaknesses in the protocol or the test method.

### 14. Full Scale Run

#### 14.1 *Material Preparation and Distribution:*

14.1.1 *Sample Preparation and Labelling*—Prepare enough of each material to supply at least 10 % more than needed by the number of laboratories committed to the ILS. Label each test unit or test specimen with a letter for the material and a sequential number. Thus, for ten laboratories and two test results for each laboratory the test units for Material *B* would be numbered from B1 to B22, or, if five test specimens per test unit are required, the test specimens may be numbered B1 to B110.

14.1.2 *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 randomization based on random numbers. See Guide E1402 for a discussion of randomization.

14.1.3 *Shipping*—Ensure that the test units are appropriately packaged to arrive in the desired condition. When the material is sensitive to the conditions to which it is exposed (vibrations, light, heat, humidity, etc.), place special directions for opening the package on a label outside the package. If needed, have the shipper monitor any specified factors deemed to be important from the point of origin to the final destination of any packages. The monitoring of shipments can be done as well by including ILS's own electronic shipment monitoring device. Clearly indicate the name of the person who has been designated as ILS supervisor at the laboratory on the address of each package. Follow each laboratory's instructions for ensuring prompt delivery of the package. Include any Material Certifications required with the materials shipped to the laboratories.

14.1.4 *Follow-Up*—Once the test units have been shipped, the ILS coordinator should call each laboratory ILS supervisor within a week to ten days to confirm that all test units have arrived safely. It is important for the ILS coordinator to express the need for the laboratory ILS supervisor to ensure that only the correct number of replicates are tested and that the test results are reported to the number of decimal places as required in the protocol.

14.1.5 *Replacement Sets of Test Units*—As the ILS progresses, a laboratory may discover that the test method was not used properly on some test units. The laboratory ILS supervisor should discuss this with the ILS coordinator, who may send a replacement set of test units, replace the misused test units, or do nothing, as may seem desirable.

14.2 *Checking Progress*—From time to time, at intervals appropriate to the magnitude of the ILS, the coordinator should call each ILS supervisor to ascertain how the testing is progressing. By comparing the progress of all laboratories, the coordinator can determine whether some laboratories are lagging considerably behind the others and so advise these laboratories.

14.3 *Data Inspection*—The completed data sheets should be examined by the coordinator immediately upon receipt in order to detect unusual values or other deficiencies that should be questioned. Replacement sets of test units or of specific test units may be sent when there is missing or obviously erroneous data. The task group can decide later whether or not the additional data should be used in the estimation of the precision of the test method.

## CALCULATION AND DISPLAY OF STATISTICS

### 15. Calculation of the Statistics

15.1 *Overview*—The analysis and treatment of the ILS test results have three purposes, to determine whether the collected data are adequately consistent to form the basis for a test method precision statement, to investigate and act on any data considered to be inconsistent, and to obtain the precision statistics on which the precision statement can be based. The statistical analysis of the data for estimates of the precision statistics is simply a one-way analysis of variance (within- and between-laboratories) carried out separately for each level (material). Since such an analysis can be invalidated by the presence of severe outliers, it is necessary to first examine the consistency of the data. Annex A1 gives background theory on these procedures. The following paragraphs show, in terms of a numerical example, how the entire program is carried out:

15.1.1 The calculations are illustrated with test results from an ILS in which the concentration of glucose in serum (see Table 1) was measured at five different concentration levels by eight laboratories. Each laboratory obtained three test results at each concentration level. A second example of an ILS is given in Appendix X1 for a test method measuring pentosans in wood pulp that involved seven laboratories and nine materials.

15.1.2 For extended calculations it is usually necessary to 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.

15.1.3 While the calculations described in this section are arranged for use of a hand calculator, they also can be readily programmed for the computer. A spreadsheet can be easily adapted to these calculations, and Appendix X2 illustrates an example spreadsheet for the glucose in serum ILS.

**TABLE 1 Glucose in Serum ILS Test Result Data**

Laboratory	Material				
	A	B	C	D	E
1	41.03	78.28	132.66	193.71	292.78
	41.45	78.18	133.83	193.59	294.09
	41.37	78.49	133.10	193.65	292.89
2	41.17	77.78	132.92	190.88	292.27
	42.00	80.38	136.90	200.14	309.40
	41.15	79.54	136.40	194.30	295.08
3	41.01	79.18	132.61	192.71	295.53
	40.68	79.72	135.80	193.28	290.14
	42.66	80.81	135.36	190.28	292.34
4	39.37	84.08	138.50	195.85	295.19
	42.37	78.60	148.30	196.36	295.44
	42.63	81.92	135.69	199.43	296.83
5	41.88	78.16	131.90	192.59	293.93
	41.19	79.58	134.14	191.44	292.48
	41.32	78.33	133.76	195.12	294.28
6	43.28	78.66	137.21	195.34	297.74
	40.50	79.27	135.14	198.26	296.80
	42.28	81.75	137.50	198.13	290.33
7	41.08	79.76	130.97	194.66	287.29
	41.27	81.45	131.59	191.99	293.76
	39.02	77.35	134.92	187.13	289.36
8	43.36	80.44	135.46	197.56	298.46
	42.65	80.80	135.14	195.99	295.28
	41.72	79.80	133.63	200.82	296.12

15.1.4 If laboratory data contains either missing or an excessive number of test results than required by the protocol, this will result in an unbalanced data set for that material. In

this situation, the calculations in this section cannot be used, but a methodology for calculating the precision statistics is given in **Annex A2**. The consistency statistics must be adjusted for the data imbalance. A highly unbalanced data set, with a deviation of 10 % or greater from the targeted number of required test results, can lead to much greater variability in the estimates of precision.

15.2 *Table of ILS Test Results*—The test results received from the laboratories are usually best arranged in rows and columns as in **Table 1**. Each column contains the data obtained from all laboratories for one material, and each row contains the data from one laboratory for all materials. The test results from one laboratory on one material constitute a cell. Thus, the cell for Laboratory 2 and Material C contains the test results 132.92, 136.90, and 136.40. This cell is called C2, by material and laboratory. It helps in the interpretation of the data to arrange the materials in increasing order of the measured values.

15.3 *Worksheets*—Generally, it facilitates the calculations to prepare a separate calculation worksheet for each material, using **Table 2** as a model but making appropriate changes for different numbers of laboratories, and test results per material. Enter the test result data for one material (from one column of **Table 1**) on a worksheet. Also enter the results of the following calculations for that material on the same worksheet, as illustrated in **Table 2**. Work on only one material at a time.

#### 15.4 Cell Statistics:

15.4.1 *Cell Average,  $\bar{x}$* —Calculate the cell average for each laboratory using the following equation:

**TABLE 2 Interlaboratory Study Worksheet for Glucose in Serum Initial Preparation of Test Result Data for Material C**

Laboratory Number	Test Results, $x$					$\bar{x}$	$s$	$d$	$h$	$k$
	1	2	3							
1	132.66	133.83	133.10	133.197	0.591	-1.946	-0.73	0.22		
2	132.92	136.90	136.40	135.407	2.168	0.264	0.10	0.79		
3	132.61	135.80	135.36	134.590	1.729	-0.553	-0.21	0.63		
4	138.50	148.30	135.69	140.830	6.620	5.687	2.14	2.41		
5	131.90	134.14	133.76	133.267	1.199	-1.876	-0.71	0.44		
6	137.21	135.14	137.50	136.617	1.287	1.474	0.55	0.47		
7	130.97	131.59	134.92	132.493	2.124	-2.650	-1.00	0.77		
8	135.46	135.14	133.63	134.743	0.977	-0.400	-0.15	0.36		

Average of cell averages,  $\bar{\bar{x}} = 135.1429$

Standard deviation of cell averages,  $s_{\bar{x}} = 2.6559$

Repeatability standard deviation,  $s_r = 2.7483$

Between-laboratory standard deviation,  $s_L = 2.1298$

Reproducibility standard deviation,  $s_R = 3.4770$

where:

$x$  = individual test result (see 15.3),

$\bar{x}$  = cell average (see 15.4.1),

$s$  = cell standard deviation (see 15.4.2),

$\bar{\bar{x}}$  = average of cell averages (see 15.5.1),

$d$  = cell deviation (see 15.5.2),

$s_{\bar{x}}$  = standard deviation of cell averages (see 15.5.3),

$s_r$  = repeatability standard deviation (see 15.6.1),

$s_L$  = between-laboratory standard deviation (see 15.6.2),

$s_R$  = reproducibility standard deviation (see 15.6.3),

$h$  = between-laboratory consistency (see 15.7.1), and

$k$  = within-laboratory consistency (see 15.7.2).



$$\bar{x} = \sum_{i=1}^n x/n \tag{1}$$

where:

- $\bar{x}$  = the average of the test results in one cell,
- $x$  = the individual test results in one cell, and
- $n$  = the number of test results in one cell.

Thus, from Table 2 for Material C, Laboratory 2 (that is, for Cell C2):

$$\bar{x} = \frac{(132.92 + 136.90 + 136.40)}{3} = 135.407$$

15.4.2 Cell Standard Deviation,  $s$ —Calculate the standard deviation of the test results in each cell using the following equation:

$$s = \sqrt{\sum_{i=1}^n (x - \bar{x})^2 / (n - 1)} \tag{2}$$

The symbols have the same meaning as for Eq 1. Thus, for Cell C2:

$$s = \sqrt{\frac{[(-2.487)^2 + (1.493)^2 + (0.994)^2]}{(3 - 1)}} = \sqrt{\frac{9.400448}{2}} = 2.168$$

While Eq 2 shows the underlying calculation of the cell standard deviation, inexpensive pocket calculators are available that calculate both the average and the standard deviation directly. Check to be sure the calculator uses  $(n - 1)$  as the divisor in Eq 2, not  $n$ , and has adequate precision of calculation.

15.5 Intermediate Statistics:

15.5.1 Average of the Cell Averages,  $\bar{\bar{x}}$ —Calculate the average of all the cell averages for the one material using Eq 3.

$$\bar{\bar{x}} = \sum_{i=1}^p \bar{x}/p \tag{3}$$

where:

- $\bar{\bar{x}}$  = the average of the cell averages for one material,
- $\bar{x}$  = the individual cell averages, and
- $p$  = the number of laboratories in the ILS.

Thus, for Material C:

$$\bar{\bar{x}} = \frac{1081.1432}{8} = 135.1429$$

15.5.2 Cell Deviation,  $d$ —For each laboratory calculate the cell deviation by subtracting the average of the cell averages from the cell average using the following equation:

$$d = \bar{x} - \bar{\bar{x}} \tag{4}$$

Thus, for Cell C2:

$$d = 135.407 - 135.143 = 0.264$$

15.5.3 Standard Deviation of the Cell Averages,  $s_{\bar{x}}$ —Calculate this statistic using the following equation:

$$s_{\bar{x}} = \sqrt{\sum_{i=1}^p d^2 / (p - 1)} \tag{5}$$

Thus, for Material C:

$$s_{\bar{x}} = \sqrt{\frac{49.376634}{(8 - 1)}} = \sqrt{7.053805} = 2.6559$$

15.6 Precision Statistics—While there are other precision statistics, introduced later in this practice, the fundamental precision statistics of the ILS are the repeatability standard deviation and the reproducibility standard deviation. The other statistics are calculated from these standard deviations.

15.6.1 Repeatability Standard Deviation,  $s_r$ —Calculate this statistic using the following equation:

$$s_r = \sqrt{\sum_{i=1}^p s^2 / p} \tag{6}$$

where:

- $s_r$  = the repeatability standard deviation,
- $s$  = the cell standard deviation ( $p$  of them from Eq 2), and
- $p$  = the number of laboratories.

Thus, for Material C:

$$s_r = \sqrt{\frac{60.425223}{8}} = \sqrt{7.553153} = 2.7483$$

15.6.2 Between Laboratory Variance,  $s_L^2$ , and Standard Deviation  $s_L$ —Calculate this variance and standard deviation using the following equations:

$$s_L^2 = s_{\bar{x}}^2 - s_r^2 / n \tag{7}$$

$$s_L = \sqrt{s_L^2} \tag{8}$$

If  $s_L^2$  is negative, set  $s_L^2 = 0$  and  $s_L = 0$ .

Thus, for Material C:

$$s_L^2 = 2.6559^2 - 2.7483^2 / 3 = 7.053805 - 2.517718 = 4.536087$$

$$s_L = \sqrt{4.536087} = 2.1298$$

15.6.2.1 The data for Material A illustrate the case of negative estimate for  $s_L^2$  (see Table 8 for the required statistics  $s_{\bar{x}}$  and  $s_r$  for Material A).

Thus, for Material A:

$$s_L^2 = 0.6061^2 - 1.0632^2 / 3 = -0.009441, \\ \text{set } s_L^2 = 0, \\ \text{and set } s_L = 0.$$

NOTE 3—This situation may occur when the laboratories are in excellent agreement, in which case both  $s_{\bar{x}}$  and  $s_r^2 / n$  in Eq 7 tend to become estimates of the variance of laboratory averages, and their difference will fluctuate around zero, causing the estimate  $s_L^2$  to take on negative values at times. Because variances cannot be negative (being

TABLE 3 Glucose in Serum-h<sup>A</sup>

Laboratory	Material				
	A	B	C	D	E
1	-0.39	-1.36	-0.73	-0.41	-0.46
2	-0.13	-0.45	0.10	0.15	1.64
3	-0.11	0.22	-0.21	-1.01	-0.68
4	-0.10	1.85	2.14	0.96	0.49
5	-0.09	-0.99	-0.71	-0.64	-0.34
6	0.83	0.21	0.55	0.97	0.17
7	-1.75	-0.16	-1.00	-1.33	-1.62
8	1.75	0.67	-0.15	1.31	0.79

<sup>A</sup> Critical value = 2.15.

TABLE 4 Glucose in Serum-*k*<sup>A</sup>

Laboratory	Material				
	A	B	C	D	E
1	0.21	0.11	0.22	0.02	0.18
2	0.46	0.89	0.79	1.78	2.33
3	1.00	0.56	0.63	0.61	0.69
4	1.70	1.85	2.41	0.74	0.22
5	0.34	0.52	0.44	0.72	0.24
6	1.32	1.09	0.47	0.63	1.03
7	1.17	1.38	0.77	1.45	0.84
8	0.77	0.34	0.36	0.94	0.42

<sup>A</sup> Critical value = 2.06.

proportional to a sum of squared deviations from an average), any negative estimate of the between laboratory variance must be set to zero.

15.6.3 *Reproducibility Standard Deviation, s<sub>R</sub>*—Calculate this statistic using the following equation:

$$s_R = \sqrt{s_L^2 + s_r^2} \quad (9)$$

Thus, for Material C:

$$s_R = \sqrt{4.536087 + 2.7483^2} = 3.4770$$

Thus, for Material A:

$$s_R = \sqrt{0 + 1.0632^2} = 1.0632, \text{ thus } s_R = s_r$$

15.7 *Consistency Statistics, h and k:*

15.7.1 For each cell, calculate a value of *h* using the following equation:

$$h = d/s_{\bar{x}} \quad (10)$$

where:

- h* = the between-laboratory consistency statistic,
- d* = the cell deviation (that is, the deviation of the cell average from the average of the cell averages, from 15.5.2), and
- s<sub>̄x</sub>* = the standard deviation of the cell averages (from 15.5.3).

Thus, for Cell C2:

$$h = \frac{0.264}{2.6559} = 0.10$$

Retain two decimal places in the computed values of *h*.

15.7.2 For each cell, use the following equation to calculate a value of *k*.

$$k = s/s_r \quad (11)$$

where:

- k* = the within-laboratory consistency statistic,
- s* = the cell standard deviation for one laboratory (from 15.4.2), and
- s<sub>r</sub>* = the repeatability standard deviation of the material (from 15.6.1).

Thus, for Cell C2:

$$k = \frac{2.168}{2.7483} = 0.79$$

Retain two decimal places in the computed values of *k*.

15.8 *Other Materials*—Repeat the steps described in 15.4 through 15.7 for each material, entering the calculation results on separate worksheets.

## 16. Tabular and Graphical Display of Statistics

16.1 *Material Order*—It is often useful to arrange the worksheets in order of increasing values of  $\bar{x}$ , the material averages. This order may facilitate interpretation.

16.2 *Tables*—From the Table 2 results for each material, prepare tables of *h* and *k* as shown in Table 3 and Table 4 for the glucose in serum example.

16.3 *Graphs*—Prepare bar graphs for *h* and *k* with materials grouped by laboratory as in Fig. 1 and Fig. 2, respectively. Arrange the laboratories and materials within and between each grouping in the same order as used in Table 1. Thus, the materials will be arranged in order of increasing *x* from left to right, and the laboratories in order of laboratory code number.

## DATA CONSISTENCY

### 17. Flagging Inconsistent Results

17.1 *Critical Values of the Consistency Statistics*—Table 5 lists critical values of the *h* and *k* consistency statistics at the 0.5 % significance level. The critical values for *h* (first column) depend on the number of laboratories (*p*, second column) participating in the ILS and the critical values for *k* (columns headed 2 through 10) depend both on the number of laboratories (*p*) and on the number of replicate test results (*n*) per laboratory per material. The 0.5 % level was chosen based on the judgment and experience that the 1.0 % resulted in too many cells being flagged and the 0.1 % level in too few. For further discussion see Annex A1.

17.1.1 Obtain from Table 5 the appropriate critical values. For the glucose in serum example, the respective critical *h* and *k* values are 2.15 and 2.06. In Table 3 and Table 4 circle those values that exceed the critical values and underline those values that approach the critical values. On Fig. 1, draw horizontal lines for positive and negative values of *h*. On Fig. 2, draw a horizontal line for *k*.

17.1.2 The *h* and *k* graphs and the marked tables give a picture of the overall character of the variability of the test method as well as singling out particular laboratories or cells that should be investigated.

17.2 *Plots by Laboratory*—In order to evaluate the differences between laboratories, use the following guidelines.

17.2.1 *h Graph*—There are three general patterns in these plots. In one, all laboratories have both positive and negative *h* values among the materials. In the second, the individual laboratories tend to be either positive or negative for all materials and the number of negative laboratories equals the number of positive laboratories, more or less. Neither of these patterns is unusual or requires investigation, although they may tell something about the nature of the test method variability. In the third pattern, one laboratory, with all *h* values positive (or negative), is opposed to all the other laboratories, with substantially all the *h* values negative (or positive). Such a pattern calls for an investigation of that laboratory.

**TABLE 5 Critical Values of  $h$  and  $k$  at the 0.5 % Significance Level**

Critical value of $h$	Number of laboratories $p$	Critical values of $k$								
		Number of replicates per laboratories, $n$								
		2	3	4	5	6	7	8	9	10
1.15	3	1.72	1.67	1.61	1.56	1.52	1.49	1.47	1.44	1.42
1.49	4	1.95	1.82	1.73	1.66	1.60	1.56	1.53	1.50	1.47
1.74	5	2.11	1.92	1.79	1.71	1.65	1.60	1.56	1.53	1.50
1.92	6	2.22	1.98	1.84	1.75	1.68	1.63	1.59	1.55	1.52
2.05	7	2.30	2.03	1.87	1.77	1.70	1.65	1.60	1.57	1.54
2.15	8	2.36	2.06	1.90	1.79	1.72	1.66	1.62	1.58	1.55
2.23	9	2.41	2.09	1.92	1.81	1.73	1.67	1.62	1.59	1.56
2.29	10	2.45	2.11	1.93	1.82	1.74	1.68	1.63	1.59	1.56
2.34	11	2.49	2.13	1.94	1.83	1.75	1.69	1.64	1.60	1.57
2.38	12	2.51	2.14	1.96	1.84	1.76	1.69	1.64	1.60	1.57
2.41	13	2.54	2.15	1.96	1.84	1.76	1.70	1.65	1.61	1.58
2.44	14	2.56	2.16	1.97	1.85	1.77	1.70	1.65	1.61	1.58
2.47	15	2.57	2.17	1.98	1.86	1.77	1.71	1.66	1.62	1.58
2.49	16	2.59	2.18	1.98	1.86	1.77	1.71	1.66	1.62	1.58
2.51	17	2.60	2.19	1.99	1.86	1.78	1.71	1.66	1.62	1.59
2.53	18	2.61	2.20	1.99	1.87	1.78	1.72	1.66	1.62	1.59
2.54	19	2.62	2.20	2.00	1.87	1.78	1.72	1.67	1.62	1.59
2.56	20	2.63	2.21	2.00	1.87	1.79	1.72	1.67	1.63	1.59
2.57	21	2.64	2.21	2.00	1.88	1.79	1.72	1.67	1.63	1.59
2.58	22	2.65	2.21	2.01	1.88	1.79	1.72	1.67	1.63	1.59
2.59	23	2.66	2.22	2.01	1.88	1.79	1.72	1.67	1.63	1.59
2.60	24	2.66	2.22	2.01	1.88	1.79	1.73	1.67	1.63	1.60
2.61	25	2.67	2.23	2.01	1.88	1.79	1.73	1.67	1.63	1.60
2.62	26	2.67	2.23	2.02	1.89	1.80	1.73	1.68	1.63	1.60
2.62	27	2.68	2.23	2.02	1.89	1.80	1.73	1.68	1.63	1.60
2.63	28	2.68	2.23	2.02	1.89	1.80	1.73	1.68	1.63	1.60
2.64	29	2.69	2.24	2.02	1.89	1.80	1.73	1.68	1.64	1.60
2.64	30	2.69	2.24	2.02	1.89	1.80	1.73	1.68	1.64	1.60

See Section A1.2 for derivations and calculation formulas for calculation of critical values for the  $h$  and  $k$  consistency statistics.

For calculation of the  $h$  critical values, see Eq A1.5 in A1.2.2.1.

For calculation of the  $k$  critical values, see Eq A1.13 in A1.2.3.2.

**TABLE 6 Glucose in Serum- $h^{A,B}$** 

Laboratory	Material				
	A	B	C	D	E
1	-0.39	-1.36	-0.88	-0.41	-0.46
2	-0.13	-0.45	0.39	0.15	1.64
3	-0.11	0.22	-0.08	-1.01	-0.68
4	-0.10	1.85	1.59	0.96	0.49
5	-0.09	-0.99	-0.84	-0.64	-0.34
6	0.83	0.21	1.09	0.97	0.17
7	-1.75	-0.16	-1.28	-1.33	-1.62
8	1.75	0.67	0.01	1.31	0.79

<sup>A</sup> Recalculated values after correcting Cell C4 (see 20.1.4 and 20.1.5).

<sup>B</sup> Critical value = 2.15.

**TABLE 7 Glucose in Serum- $k^{A,B}$** 

Laboratory	Material				
	A	B	C	D	E
1	0.21	0.11	0.38	0.02	0.18
2	0.46	0.89	1.40	1.78	2.33
3	1.00	0.56	1.12	0.61	0.69
4	1.70	1.85	1.02	0.74	0.22
5	0.34	0.52	0.78	0.72	0.24
6	1.32	1.09	0.83	0.63	1.03
7	1.17	1.38	1.38	1.45	0.84
8	0.77	0.34	0.63	0.94	0.42

<sup>A</sup> Recalculated values after correcting Cell C4 (see 20.1.4 and 20.1.5).

<sup>B</sup> Critical value = 2.06.

17.2.1.1 Another kind of pattern to look for occurs within one laboratory, in which the  $h$  values for low property levels are of one sign, and for high property levels are of the opposite sign. If the values are extreme, this behavior should be investigated.

17.2.2  $k$  Graph—Here the primary pattern to look for is that of one laboratory having large  $k$  values (or very small  $k$  values) for all or most of the materials. High  $k$  values represent

within-laboratory imprecision. Very small  $k$  values may indicate a very insensitive measurement scale or other measurement problem.

## 18. Investigation

18.1 *Clerical and Sampling Errors*—Examine the laboratory report for each flagged cell. Try to locate where each test result in the flagged cell begins to deviate from the others. Is it