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An American National Standard

Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method¹

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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.*

¹ 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:²

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*—Unless otherwise noted in this standard, all terms relating to quality and statistics are defined in E456.

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**

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.6 *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_r), 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_R), 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_L , n —the sample standard deviation attributable to differences of test result means among laboratories.

3.2.4 *between-laboratory variance*, s_L^2 , 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_r^2 , 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_R^2 , 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_L^2 , and within-laboratories, s_r^2 .

3.2.11 *standard deviation of the cell averages*, $s_{\bar{x}}$, n —the standard deviation of the cell averages for a particular material.

3.2.12 *variance of the cell averages*, $s_{\bar{x}}^2$, 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 Group³—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.

³ 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.

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 X1 illustrates an example spreadsheet for the glucose in serum ILS.

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

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

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:

$$\bar{x} = \sum_{i=1}^n x_i/n \quad (1)$$

this situation, the calculations in this section cannot be used, but a methodology for calculating the precision statistics is

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).

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:

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

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.

$$\bar{\bar{x}} = \sum_{i=1}^p \bar{x}_i / p \quad (3)$$

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:

$$d = \bar{x} - \bar{\bar{x}} \quad (4)$$

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:

$$s_{\bar{x}} = \sqrt{\frac{\sum_{i=1}^p d_i^2}{(p-1)}} \quad (5)$$

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:

$$s_r = \sqrt{\sum_{i=1}^p s_i^2 / p} \quad (6)$$

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:

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

$$s_L = \sqrt{s_L^2} \quad (8)$$

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:

$$s_L^2 = 0.6061^2 - 1.0632^2 / 3 = -0.009441,$$

set $s_L^2 = 0$,
and set $s_L = 0$.

NOTE 3—This situation may occur when the laboratories are in excellent agreement, in which case both $s_{\bar{x}}^2$ 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 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_R* —Calculate this statistic using the following equation:

TABLE 3 Glucose in Serum-h^A

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

^A Critical value = 2.15.

TABLE 4 Glucose in Serum- k^A

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

^A Critical value = 2.06.

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

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
- $s_{\bar{x}}$ = 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_r = 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.

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)

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

^A Recalculated values after correcting Cell C4 (see 20.1.4 and 20.1.5).

^B 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

^A Recalculated values after correcting Cell C4 (see 20.1.4 and 20.1.5).

^B Critical value = 2.06.

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.

TABLE 8 Glucose in Serum—Precision Statistics

NOTE 1—This table (with the column for s_x omitted) is a useful format for the presentation of the precision of a test method as required by Section A21 of the *Form and Style of ASTM Standards*.

Material	\bar{x}	s_x	s_r	s_R	r	R
A	41.5183	0.6061	1.0632	1.0632	2.98	2.98
B	79.6796	1.0027	1.4949	1.5796	4.19	4.42
C	134.7264	1.7397	1.5434	2.1482	4.33	6.02
D	194.7170	2.5950	2.6251	3.3657	7.35	9.42
E	294.4920	2.6931	3.9350	4.1923	11.02	11.74

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 in the original observations? Are the data rounded prematurely? Are the calculations correct? Then, look for signs of mislabeling of test units such that the test result for one material was reported as belonging to another material. Check these errors with the laboratories: do not assume them to be so.

18.2 Procedural Errors:

18.2.1 Study the laboratory reports again looking for deviations from either the test method or the protocol. For instance, variations in the number of significant digits reported in the test results may be a sign of incorrect rounding, or that the equipment in one laboratory is different from the rest. Also, study the event log for special comments relating to the flagged cells.

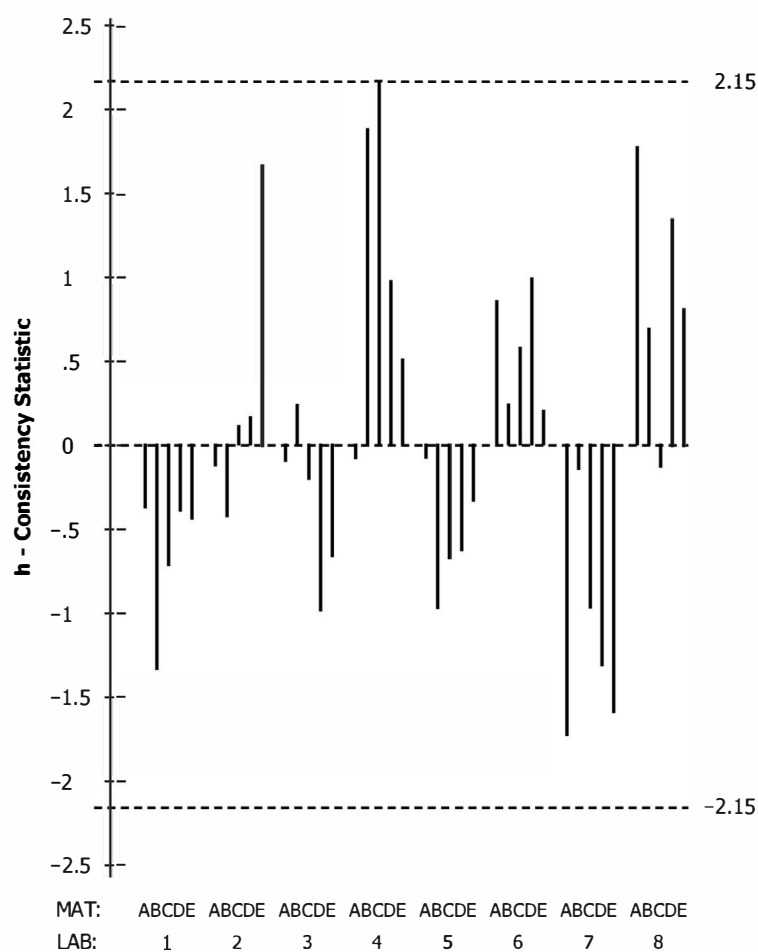


FIG. 1 Glucose in Serum: *h*—Materials within Laboratories

19. Task Group Actions

19.1 General—If the investigation disclosed no clerical, sampling or procedural errors, the unusual data should be retained, and the precision statistics based on them should be published. If, on the other hand, a cause was found during the investigation, the task group has several options to consider. If the laboratory clearly and seriously deviated from the test method, the test results for that laboratory must be removed from the ILS calculations. However, despite the danger of the recalcitrant laboratory having prior knowledge, it may be appropriate to ask the laboratory to retest one or more materials following the correct procedure, and then include the new set of test results in the ILS calculations. Of course, if the data have changed, recalculation of the *h* and *k* values must be made and the data consistency examined again.

19.2 Exception—When a large number of laboratories have participated in the ILS and no causes for some unusual cell values have been found during the investigation, it may be appropriate to delete a cell from the study if all of the other laboratories are in substantial agreement. The number of laboratories that can be considered large enough to support deletion of data without an identified cause cannot be stated exactly. Any action which results in discarding more than ten

percent of the ILS data likely will lead to the presentation of precision data that the test method cannot deliver in routine application.

19.3 Test Method Vagueness—One of the important things to be on the alert for during a laboratory investigation is for vagueness in the test method standard that permits a wide range of interpretation leading to loss of precision. Particular elements to check are lack of measurement tolerances, diversity of apparatus and insufficient direction for operator technique. These problems can be the basis for a revision of the standard.

20. Glucose ILS Consistency

20.1 Glucose in Serum—The ILS is described in 15.1.1.

20.1.1 *h* Statistic—The overall impression given by Fig. 1 and Table 3 is one of reasonable consistency for variation among laboratories. Only Laboratory 4 stands out with large values for Materials B and C.

20.1.2 *k* Statistic—Laboratories 2 and 4 stand out in Fig. 2 and Table 4.

20.1.3 Cells and Test Results—Cells C4 and E2 should be investigated. A look at Table 1 reveals that the second test results of 148.30 in C4 and of 309.40 in E2 are the particular values to be investigated.

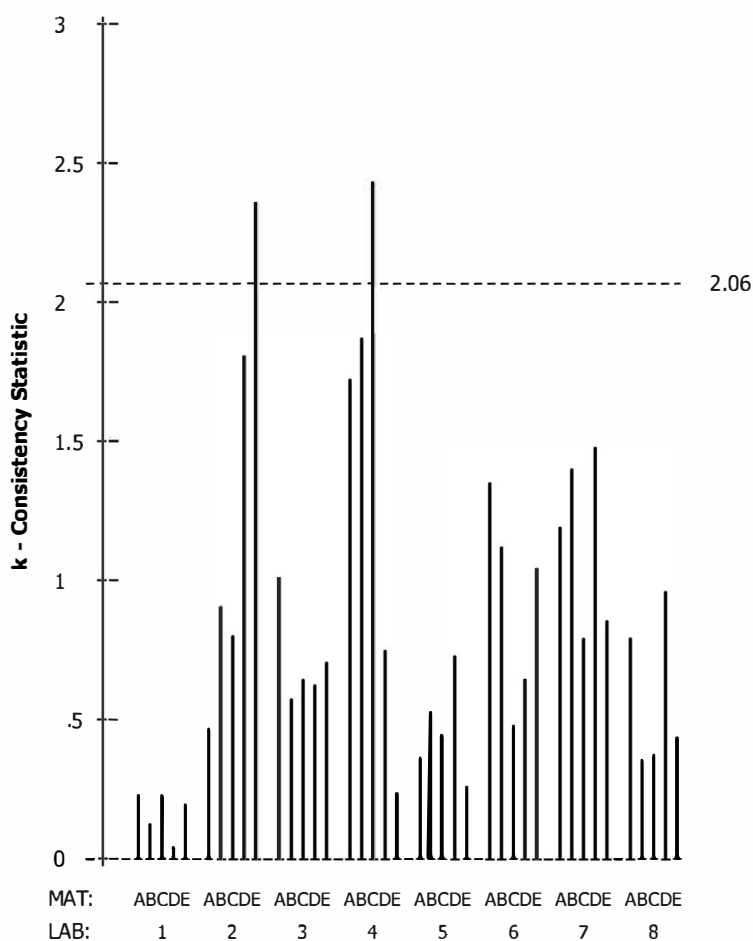


FIG. 2 Glucose in Serum: k—Materials within Laboratories

20.1.4 *Action*—If the data from Laboratory 4 were typed, the result 148.30 in Cell C4 could have been a typographical error. We have no way of knowing this today, many years after this study was made. We will suppose, however, that the task group did indeed call the laboratory and did find that the number should have been 138.30. However, let us suppose that for Cell E2 the task group could find no explanation of the apparently high value of 309.40. In such a case they should retain the value.

20.1.5 *Recalculation*—Table 6 and Table 7 show the recalculated consistency statistics resulting from correcting Cell C4.

PRECISION STATEMENT INFORMATION

21. Repeatability and Reproducibility

21.1 *General*—Once the task group has concluded which cells are sufficiently inconsistent to require action, and action has been taken, the statistics of 15.4 through 15.6 are recalculated (see also 20.1.5). Using the corrected statistics, calculate for each material the 95 % repeatability and reproducibility limits (see Practice E177) according to the following Eq 12 and Eq 13:

$$r = 2.8 s_r \quad (12)$$

$$R = 2.8 s_R \quad (13)$$

21.2 Prepare a table for the corrected precision statistics as shown in Table 8.

21.3 Variation of Precision Statistics with Property Level:

21.3.1 Quite often the values of s_r and s_R will be found to vary with the values of the property level \bar{x} . This type of response can be seen in Fig. 3, that is based on Table 8. The manner in which the statistics vary with the property level should be shown in presenting the precision information in the precision statement of the test method. The statistician should recommend the most appropriate relationship to present, using Practice E177 as a guide.

21.4 *Precision Statement*—Table 8 (with the column for s_R omitted) is a useful format for the presentation of the precision statement of the test method as required by Section A21 of the *Form and Style of ASTM Standards*² (Bluebook). Having obtained the required precision information in accordance with this practice, the final form of the precision statement may be prepared in accordance with Practice E177.

21.5 *Conclusion*—The precision statistics obtained by an ILS such as described in this practice must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The small number of laboratories and of materials included in the usual ILS guarantees that there will

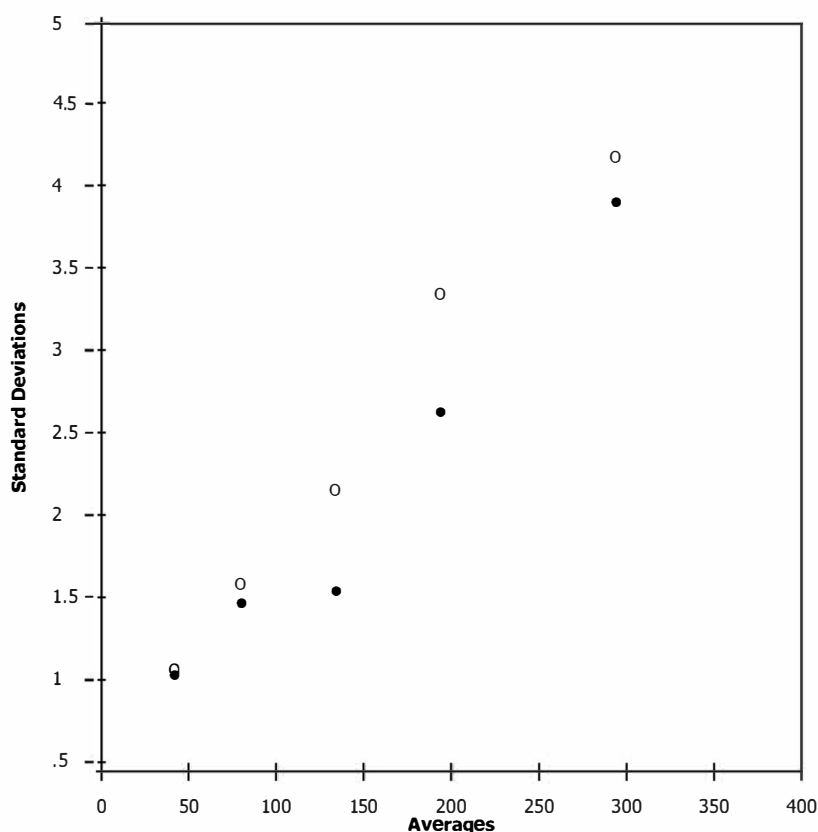


FIG. 3 Glucose in Serum: Standard Deviations of Reproducibility (o) and Repeatability (•) Versus Average

be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected. If more precise information is needed in specific circumstances, those laboratories directly involved in a material comparison must conduct interlaboratory studies specifically aimed at the material of interest.⁴

⁴ Following the ASTM Research Report format guide, prepare a research report on the ILS to be filed at ASTM Headquarters.

22. Keywords

22.1 precision; repeatability; reproducibility; repeatability limit; reproducibility limit

ANNEXES

(Mandatory Information)

A1. THEORETICAL CONSIDERATIONS

A1.1 Underlying Assumptions of ILS

A1.1.1 *Within-Laboratory Variability*—The cell standard deviation is a measure of the within-laboratory variability of each individual laboratory. All laboratories are assumed to have essentially the same level of variability when following

the specified repeatability conditions. This assumption is not always fulfilled. However, the shorter the period of time in which the test results for a particular material are to be obtained by the laboratories the more likely the validity of this assumption. Therefore, the laboratory cell variances can generally be

pooled by averaging the squares of the cell standard deviations. The square root of this average within-laboratory variance is the repeatability standard deviation s_r .

A1.1.2 Between-Laboratory Variability:

A1.1.2.1 Variability of Laboratory Means—The test results obtained on a particular material at any particular laboratory are considered part of a population having a normal distribution with a standard deviation equal to the repeatability standard deviation but with a mean that may be different for each laboratory. The laboratory means are also assumed to vary according to a normal distribution, whose mean is estimated by the average of all ILS test results for a given material, and whose standard deviation is designated by s_L . (The effect of a single outlying laboratory on this assumption will be less if there are enough laboratories.) For the ILS calculations, s_L is estimated from the standard deviation of the cell averages, $s_{\bar{x}}$, and the repeatability standard deviation, s_r as follows:

$$s_L^2 = s_{\bar{x}}^2 - \frac{s_r^2}{n} \quad (\text{A1.1})$$

The term $s_{\bar{x}}^2$ is the observed variance of the cell averages from the ILS data. The term s_r^2 / n estimates the variance of cell averages of n test results assuming that there is no difference among laboratory means. Thus, s_L^2 indirectly estimates the variability of cell averages due to laboratory differences. When s_L^2 calculates to less than zero, s_L is taken equal to zero (see **Note 2**).

A1.1.2.2 Reproducibility Standard Deviation—The variance among individual test results obtained in different laboratories is the sum of the within-laboratory variance and the between-laboratory variance of the laboratory means. Thus, the reproducibility variance is given by **Eq A1.2** as follows:

$$s_R^2 = s_r^2 + s_L^2 \quad (\text{A1.2})$$

Another calculation previously used for estimating the reproducibility standard deviation that does not explicitly use s_L , as seen in previous versions of this practice, is the following.

Substituting **Eq A1.1** into **Eq A1.2** produces **Eq A1.3**:

$$s_R^2 = s_r^2 + s_{\bar{x}}^2 - \frac{s_r^2}{n} \quad (\text{A1.3})$$

Simplifying and taking the square root gives **Eq A1.4** as follows:

$$s_R = \sqrt{s_{\bar{x}}^2 + \frac{(n-1)s_r^2}{n}} \quad (\text{A1.4})$$

When s_R calculates to less than s_r , s_R is set equal to s_r .

A1.2 Consistency Statistics

A1.2.1 Critical Values—The derivation of the equations for calculating critical values of h and k are given in **A1.2.2** and **A1.2.3**. In each case critical values were calculated at three significance levels, 1 %, 0.5 %, and 0.1 %. Of these three only the 0.5 % critical values were chosen for flagging as described in **Section 17**. This choice is based on the judgment from experience that the 1 % values are too sensitive (flag too many)

and the 0.1 % values are not sensitive enough for flagging adequately in the analysis of ILS data.

A1.2.2 Between-Laboratory Consistency:

A1.2.2.1 The consistency statistic h is an indicator of how one laboratory's cell average, for a particular material, compares with the average of the other laboratories. The critical values for the comparison are calculated with an equation derived from an unpaired t -test as given by **Eq A1.5** as follows:

$$t = \frac{(\bar{x}_c - \bar{\bar{x}}^*)}{s_{\bar{x}}^* \sqrt{1 + [1 / (p-1)]}} \quad (\text{A1.5})$$

where:

t = observed Student's t value,

\bar{x}_c = cell average being compared,

$\bar{\bar{x}}^*$ = average of all cell averages except the one being compared,

$s_{\bar{x}}^*$ = standard deviation of all the cell averages except the one being compared, and

p = number of laboratories in the ILS.

In this relationship t has $p - 2$ degrees of freedom. Three further equations are required in order to express h in terms of t from **Eq A1.5**. These follow as **Eq A1.6**, **Eq A1.7**, and **Eq A1.8**:

$$\bar{\bar{x}}^* = (p\bar{\bar{x}} - \bar{x}_c) / (p - 1) \quad (\text{A1.6})$$

$$(s_{\bar{x}}^*)^2 = \frac{(p-1)s_{\bar{x}}^2}{(p-2)} - \frac{p(\bar{\bar{x}} - \bar{x}_c)^2}{[(p-1)(p-2)]} \quad (\text{A1.7})$$

$$h = \frac{d}{s_{\bar{x}}} = \frac{\bar{x}_c - \bar{\bar{x}}^*}{s_{\bar{x}}} \quad (\text{A1.8})$$

Each of these equations is derived by simple algebraic operations from the definitions of symbols accompanying **Eq A1.5** and **Table 2**. Combining them with **Eq A1.5** results in **Eq A1.9** as follows:

$$h = (p-1)t / \sqrt{p(t^2 + p - 2)} \quad (\text{A1.9})$$

A1.2.2.2 The critical values of h were calculated by **Eq A1.9**, where the value of t was the upper 0.9975th percentile of the Student's t distribution with $p - 2$ degrees of freedom. The values obtained are given in **Table 5**. The appropriate spreadsheet function for such values of t is **TINV(0.005, $p-2$)**.

A1.2.3 Within-Laboratory Consistency:

A1.2.3.1 The consistency statistic, k , is an indicator of how one laboratory's within-laboratory variability, under repeatability conditions, on a particular material, compares with all of the laboratories combined. Values of k larger than 1 indicate greater within-laboratory variability than the average for all laboratories. Since such variation among laboratories is expected, critical values of k have been calculated to aid in the decision of whether the cell standard deviation of one laboratory is sufficiently different from the rest of the laboratories as to require investigation.

A1.2.3.2 A valid test for determining whether a particular cell variance is inconsistent relative to the variances of the other laboratories is to calculate the F -ratio of the one cell variance to the pooled variance of all the other laboratories—excluding the variance being tested. This is shown in **Eq A1.10** as follows:

$$F = \frac{s_c^2}{\left[\frac{\sum_{i \neq c} s_i^2}{p-1} \right]} \quad (\text{A1.10})$$

where:

s_c^2 = cell variance of cell being compared,
 $\sum_{i \neq c} s_i^2$ = summation of all variances except the one being compared,
 p = the number of laboratories.

The consistency statistic k is defined by Eq A1.11 and the repeatability variance by Eq A1.12 as follows:

$$k = s_c / s_r \quad (\text{A1.11})$$

$$s_r^2 = \frac{\left[\sum_{i \neq c} s_i^2 \right] + s_c^2}{p} \quad (\text{A1.12})$$

Combining Eq A1.10, Eq A1.11, and Eq A1.12 results in Eq A1.13 as follows:

$$k = \sqrt{\frac{p}{1 + \frac{p-1}{F}}} \quad (\text{A1.13})$$

A1.2.3.3 The upper critical value of k is calculated by Eq A1.13 where the F value is the upper 99.5th percentile of the F distribution with $n-1$ and $(p-1)(n-1)$ degrees of freedom for selected combinations of number of within-laboratory test results (n) and number of laboratories (p). The values obtained are given in Table 5. The spreadsheet function for such values of F is FINV(0.005,(n-1),(n-1)*(p-1)).

A1.3 Consistency Statistics for Unbalanced Data Sets

A1.3.1 The derivations of the consistency statistics, h and k , presented above in Section A1.2 presume a balanced data set. Derivations of h and k for unbalanced data sets are given below in A1.3.2 and A1.3.3. The definitions of h and k given here reduce to those given in Section A1.2 when the data set is balanced.

A1.3.2 Between-Laboratory Consistency:

A1.3.2.1 As in A1.2.2, the critical values for the between-laboratory consistency statistic, h , are based on its relationship to a t -statistic, which in this case is given by Eq A1.14:

$$t = \frac{\zeta}{\sqrt{SS_d^* / (p-2)}} \quad (\text{A1.14})$$

where:

t = observed Student's t value,

$\zeta = (\bar{x}_c - \hat{x}^*) / \sqrt{(1/w_c) + (1/\sum_{j \neq c} w_j)}$,
 c = index of the cell whose average is being compared,
 \bar{x}_c = cell average being compared,
 $\hat{x}^* = \sum_{j \neq c} (w_j \bar{x}_j) / (\sum_{j \neq c} w_j)$, the weighted average of all cell averages except the one being compared,
 w_j = weighting factor for cell j , proportional to the reciprocal of the estimated variance of \bar{x}_j ,
 $SS_d^* = \sum_{j \neq c} w_j (\bar{x}_j - \hat{x}^*)^2$, and
 p = number of laboratories in the ILS.

This t -statistic has $p-2$ degrees of freedom.

A1.3.2.2 Ideally, each weighting factor w_j would equal the reciprocal variance of \bar{x}_j , but since the true variances are unknown, estimated variances are used instead, as shown in Eq A1.15:

$$w_j = \frac{1}{s_L^2 + s_r^2 / n_j} \quad (\text{A1.15})$$

where:

s_L = estimated between-laboratory standard deviation,
 s_r = estimated within-laboratory standard deviation, and
 n_j = number of measurement results in cell j .

A1.3.2.3 In the case of a balanced dataset, where all the cell sizes n_j are equal, all the weighting factors w_j are also equal and can be replaced by 1, in which case the following equations are obtained:

$$\hat{x}^* = \bar{x}^* \quad (\text{A1.16})$$

$$\frac{SS_d^*}{p-2} = s_{\bar{x}}^{*2} \quad (\text{A1.17})$$

$$\zeta = (\bar{x}_c - \bar{x}^*) \sqrt{\frac{p-1}{p}} \quad (\text{A1.18})$$

where:

\bar{x}^* = average of all the cell averages except the one being compared, and
 $s_{\bar{x}}^*$ = standard deviation of all the cell averages except the one being compared.

The equation for t in this case, Eq A1.14, is equivalent to Eq A1.5.

A1.3.2.4 Additional equations are required in order to express h in terms of the t -statistic from Eq A1.14:

$$\hat{x} = \frac{\sum_{j=1}^p (w_j \bar{x}_j)}{\sum_{j=1}^p w_j} \quad (\text{A1.19})$$

and:

$$SS_{\bar{d}} = \sum_{j=1}^p w_j (\bar{x}_j - \hat{x})^2 \quad (\text{A1.20})$$

where:

\hat{x} = weighted average of all the cell averages, and
 $SS_{\bar{d}}$ = weighted sum of squares of all the cell deviations.

A1.3.2.5 Algebraic manipulations yield the following equations:

$$\hat{x}^* = \frac{\bar{x}_c - \bar{x}_c \left(w_c / \sum_{j=1}^p w_j \right)}{1 - \left(w_c / \sum_{j=1}^p w_j \right)} \quad (\text{A1.21})$$

$$\zeta = \frac{\bar{x}_c - \hat{x}}{\sqrt{\frac{1}{w_c} - \frac{1}{\sum_{j=1}^p w_j}}} \quad (\text{A1.22})$$

$$SS_{\bar{d}}^* = SS_{\bar{d}} - \zeta^2 \quad (\text{A1.23})$$

For a balanced data set, with all weighting factors w_j set equal to 1, Eq A1.21 and Eq A1.23 are analogous to Eq A1.6 and Eq A1.7, respectively.

A1.3.2.6 When Eq A1.23 is substituted in Eq A1.14, the result is the following:

$$t = \frac{\zeta}{\sqrt{(SS_{\bar{d}}^* - \zeta^2)/(p - 2)}} \quad (\text{A1.24})$$

A1.3.2.7 The h -statistic is defined as follows:

$$h = \frac{\zeta}{\sqrt{SS_{\bar{d}}^* / (p - 1)}} \sqrt{\frac{p - 1}{p}} \quad (\text{A1.25})$$

$$= \frac{(p - 1)(\bar{x}_c - \hat{x})}{\sqrt{\left(\frac{1}{w_c} - \frac{1}{\sum_{j=1}^p w_j} \right) (p)(SS_{\bar{d}}^*)}}$$

For a balanced data set, Eq A1.25 reduces to the simple form given by Eq A1.8.

A1.3.2.8 In all cases, h and t are related as shown in Eq A1.26 below:

$$h = \frac{(p - 1)t}{\sqrt{p(t^2 + p - 2)}} \quad (\text{A1.26})$$

Since Eq A1.26 is equivalent to Eq A1.9, the same critical values of h calculated for use with balanced data sets are appropriate for use with unbalanced data sets. Critical values of h are presented in Table 5.

A1.3.3 Within-Laboratory Consistency:

A1.3.3.1 As in A1.2.3, the test for determining whether a particular cell variance is inconsistent with the variances of the other laboratories is based on the F -ratio of the one cell variance to the pooled variance of all the other laboratories — excluding the one being tested. However, in the case of an

unbalanced data set, the pooled variance is calculated in a manner that accounts for variations in cell sizes. The F -ratio is defined by Eq A1.27:

$$F = \frac{s_c^2}{s_p^2} \quad (\text{A1.27})$$

where:

c = index number of the cell being compared,
 s_c^2 = cell variance of cell c , and
 s_p^2 = pooled variance of all the cells except cell c .

For a balanced data set, where all the cell sizes n_j are equal, Eq A1.27 is equivalent to Eq A1.10.

A1.3.3.2 The repeatability variance s_r^2 is defined to be the pooled variance of all the cells — including cell c — and is given explicitly by Eq A1.28:

$$s_r^2 = \frac{1}{N - p} \sum_{j=1}^p (n_j - 1) s_j^2 \quad (\text{A1.28})$$

where:

N = total number of test results,
 p = number of laboratories,
 n_j = number of test results in cell j , and
 s_j^2 = cell variance of cell j .

Then the pooled variance s_p^2 without cell c is given by Eq A1.29:

$$s_p^2 = \frac{(N - p)s_r^2 - (n_c - 1)s_c^2}{(N - p) - (n_c - 1)} \quad (\text{A1.29})$$

Defining $p_c = (N - p) / (n_c - 1)$ leads to Eq A1.30 below:

$$s_p^2 = \frac{p_c s_r^2 - s_c^2}{p_c - 1} \quad (\text{A1.30})$$

A1.3.3.3 The consistency statistic k is defined by Eq A1.31:

$$k = \frac{s_c}{s_r} \quad (\text{A1.31})$$

Combining Eq A1.27, Eq A1.30, and Eq A1.31 results in Eq A1.32:

$$F = \frac{k^2(p_c - 1)}{p_c - k^2} \quad (\text{A1.32})$$

A1.3.3.4 Solving Eq A1.32 for k gives Eq A1.33:

$$k = \sqrt{\frac{p_c}{1 + \frac{p_c - 1}{F}}} \quad (\text{A1.33})$$

A1.3.3.5 The upper critical value of k is given by Eq A1.33 with F equal to the upper 99.5th percentile of the F -distribution with $n_c - 1$ and $(N - p) - (n_c - 1)$ degrees of freedom. The spreadsheet function for this percentile of F is FINV(0.005, $n_c - 1$, $(N - p) - (n_c - 1)$).

A1.3.3.6 If the data set is balanced, with all the n_j equal to n , then $N = np$, $n_c = n$, $p_c = p$, and Eq A1.33 is equivalent to Eq A1.13. Critical values of k for this case are given in Table 5. Note that the same tabulated critical values of k must not be used with unbalanced data sets.

A2. CALCULATION OF THE ILS STATISTICS FOR UNBALANCED DATA SETS

A2.1 The protocol for an ILS is designed so that each lab conducts an equal number of replicate test results for a material, resulting in a *balanced* data set. At times, there may be missing values due to labs submitting less than the requested number of replicates, thus giving rise to an *unbalanced* data set. The calculations in Section 15 of this standard are based on a balanced data set. This annex shows how the calculations are carried out for an unbalanced data set.

A2.1.1 This annex deals with the calculations for precision statistics and consistency statistics for an unbalanced data set by allowing for different numbers of replicate test results from the participating laboratories.

A2.1.2 *Example*—The calculations in this annex are again illustrated with test results from the ILS in which the concentration of glucose in serum was measured at five different glucose levels by eight laboratories (see Table 1). Each laboratory obtained three test results at each concentration level. For Material C, an extreme outlier was observed for the second test result from Laboratory 4 as shown graphically on the dot plot in Fig. A2.1. In the discussion of 20.1.4, it was supposed that this result could have occurred because of a typing mistake, so the submitted value was corrected from 148.3 to 138.3 and then the data set was reanalyzed with the correct value. In a different scenario, suppose that a cause for the outlier was found, and that test result was discarded, thus creating a missing value in the data set and rendering the data set to be unbalanced. This outcome will be used as the example of an unbalanced data set in this annex.

A2.2 For an unbalanced data set, the symbols and equations are modified by placing subscripts on the symbols for the test result data, using the letter *i* for the laboratory number and the letter *j* for the replicate number within the laboratory. Thus, the second test result in Laboratory 4 would be designated with the symbol x_{42} . (If the number of laboratories exceeds 9, then the notation $x_{10,3}$ can be used.) Additionally, a new symbol n_i is introduced for the number of replicates in Laboratory *i*.

A2.3 *Worksheets*—As in Section 15, the calculations are facilitated by a separate calculation worksheet for each material, using Table 2 as a model for each material, but making appropriate changes for different numbers of test results per laboratory as shown in Table A2.1. Statistics for each laboratory are placed in columns adjacent to their data columns, and statistics for the entire data set are listed below the rows of laboratory data and statistics.

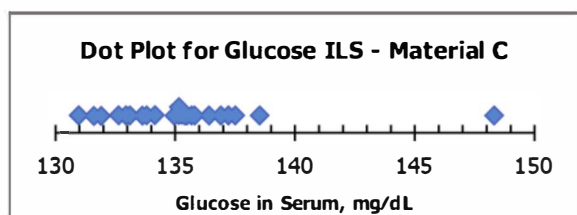


FIG. A2.1 Dot Plot of Submitted Test Results for Material C

A2.3.1 *Data Entry*—Enter the test result data (x_{ij} values) for one material (from one column of Table 1) into the worksheet. The number of replicates (n_i) for each laboratory will also be entered into an adjacent column either manually or by using the worksheet Count function. Note that there is a missing value for Replicate 2 in Laboratory 4. The calculations for the unbalanced data set precision estimates are shown in the following Sections A2.4 – A2.6. The calculations for the consistency statistics are shown in Section A2.7.

A2.4 Laboratory Statistics

A2.4.1 *Laboratory Averages*, \bar{x}_i —Calculate the average for each laboratory using the following equation:

$$\bar{x}_i = \sum_{j=1}^{n_i} x_{ij} / n_i \quad (\text{A2.1})$$

where:

\bar{x}_i = the average of the test results in Laboratory *i*,
 x_{ij} = the *j*th individual test result value from Laboratory *i*, and
 n_i = the number of test results submitted from Laboratory *i*.

Thus, from Table A2.1 for Material C, Laboratory 1, $n_1 = 3$:

$$\bar{x}_2 = \frac{(132.66 + 133.83 + 133.10)}{3} = 133.197$$

A2.4.2 *Laboratory Standard Deviations*, s_i —Calculate the standard deviation of the test results in each laboratory using the following equation:

$$s_i = \sqrt{\frac{\sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2}{(n_i - 1)}} \quad (\text{A2.2})$$

If $n_i = 1$, s_i is set to zero.

The symbols have the same meaning as for Eq A2.1. Thus, for Laboratory 1:

$$s_1 = \sqrt{\frac{[(-0.537)^2 + (0.633)^2 + (-0.097)^2]}{(3 - 1)}} = \sqrt{\frac{0.698467}{2}} = 0.591$$

A2.5 Intermediate Statistics

A2.5.1 *Total Number of Data*, N —Calculate the total number of test result data from all *p* laboratories using the following equation:

$$N = \sum_{i=1}^p n_i \quad (\text{A2.3})$$

where:

p = the number of laboratories in the data set,
 N = the total number of test result data for the material, and
 n_i = the number of test results conducted by Laboratory *i*.

Thus, for Material C with missing value in Laboratory 4:

$$N = 3 + 3 + 3 + 2 + 3 + 3 + 3 + 3 = 23$$

A2.5.2 *Grand Average*, $\bar{\bar{x}}$ —Calculate the grand average as the weighted average of all the laboratory averages, weighted

TABLE A2.1 Interlaboratory Study Worksheet for Glucose in Serum Unbalanced Data for Material C

Laboratory Number, i	Test Results, x_{ij}			n_i	\bar{x}_i	s_i	d_i
	1	2	3				
1	132.66	133.83	133.10	3	133.197	0.591	–1.374
2	132.92	136.90	136.40	3	135.407	2.168	0.836
3	132.61	135.80	135.36	3	134.590	1.729	0.019
4	138.50		135.69	2	137.095	1.987	2.524
5	131.90	134.14	133.76	3	133.267	1.199	–1.304
6	137.21	135.14	137.50	3	136.617	1.287	2.046
7	130.97	131.59	134.92	3	132.493	2.124	–2.078
8	135.46	135.14	133.63	3	134.743	0.977	0.172

Average of laboratory averages, $\bar{\bar{x}} = 134.5709$
Standard deviation of laboratory averages, $s_{\bar{x}} = 1.5965$
Repeatability standard deviation, $s_r = 1.5737$
Between-laboratory standard deviation, $s_L = 1.2984$
Reproducibility standard deviation, $s_R = 2.0402$

where:

x_{ij} = individual test result (see A2.3),
 \bar{x}_i = laboratory average (see A2.4.1),
 s_i = laboratory standard deviation (see A2.4.2),
 $\bar{\bar{x}}$ = average of laboratory averages (see A2.5.2),
 d_i = laboratory deviation (see A2.5.3),
 $s_{\bar{x}}$ = standard deviation of laboratory averages (see A2.5.5),
 s_r = repeatability standard deviation (see A2.6.1),
 s_L = between-laboratory standard deviation (see A2.6.2), and
 s_R = reproducibility standard deviation (see A2.6.3).

by n_i/N , using the following equation:

$$\bar{\bar{x}} = \sum_{i=1}^p n_i \bar{x}_i / N \quad (\text{A } 2.4)$$

where:

$\bar{\bar{x}}$ = the grand average,
 \bar{x}_i = the individual laboratory averages, and
 N = the number of data in the data set.

Thus, for Material C with missing value:

$$\bar{\bar{x}} = \frac{3(133.197) + \dots + 2(137.095) + \dots + 3(134.743)}{N} = \frac{3095.13}{23} = 134.5709$$

A2.5.3 Laboratory Deviation, d_i —For each laboratory, calculate its laboratory deviation by subtracting the grand average from its laboratory average using the following equation:

$$d_i = \bar{x}_i - \bar{\bar{x}} \quad (\text{A } 2.5)$$

Thus, for Laboratory 1:

$$d_1 = 133.197 - 134.5709 = -1.374$$

Note that a laboratory submitting only one test result can have a laboratory deviation because its average is the single test result.

A2.5.4 Operational Number of Replicates, n^* —For Material C, calculate the operational number of replicates⁵ using the following equation:

$$n^* = \left[N - \left(\sum_{i=1}^p n_i^2 / N \right) \right] / (p - 1) \quad (\text{A } 2.6)$$

$$n^* = \frac{\left[23 - \left(\frac{67}{23} \right) \right]}{(8 - 1)} = 2.87$$

For moderate numbers of missing values, n^* will usually be close, but not exactly equal, to the average number of replicates per lab.

A2.5.5 Standard Deviation of the Laboratory Averages, $s_{\bar{x}}$ —Calculate this statistic using the following equation:

$$s_{\bar{x}} = \sqrt{\frac{\sum_{i=1}^p n_i d_i^2}{n^*(p - 1)}} \quad (\text{A } 2.7)$$

Thus, for Material C:

$$s_{\bar{x}} = \sqrt{\frac{3(-1.374)^2 + \dots + 3(0.172)^2}{2.87(8 - 1)}} = \sqrt{\frac{51.2009}{2.87(7)}} = \sqrt{2.54896} = 1.5965$$

Note that a laboratory having only one test result can contribute to an estimate of laboratory variation although it only has a small weight.

A2.6 Precision Statistics

A2.6.1 Repeatability Standard Deviation, s_r —Calculate this statistic using the following equation:

$$s_r = \sqrt{\frac{\sum_{i=1}^p (n_i - 1) s_i^2}{(N - p)}} \quad (\text{A } 2.8)$$

⁵ Searle, S. R., Casella, G., and McCulloch, C., E., *Variance Components*, John Wiley & Sons, Inc., New York, 1992.

where:

s_r = the repeatability standard deviation,
 s_i = the laboratory standard deviation for Laboratory i , and
 p = the number of laboratories.

Thus, for Material C:

$$s_r = \sqrt{\frac{(3-1)(0.591)^2 + \dots + (3-1)(0.977)^2}{(23-8)}} = \sqrt{\frac{37.1467}{15}} = 1.5737$$

Note that a laboratory having only one test result will have a standard deviation of zero with a weight of zero, and thus will not contribute to an estimate of repeatability.

A2.6.2 Between Laboratory Variance, s_L^2 , and Standard Deviation s_L —Calculate this variance and standard deviation using the following equations:

$$s_L^2 = s_i^2 - (s_r^2 / n^*) \quad (\text{A2.9})$$

$$s_L = \sqrt{s_L^2} \quad (\text{A2.10})$$

If s_L^2 is negative, s_L^2 and s_L are set to zero.

Thus, for Material C:

$$s_L^2 = (1.5965)^2 - \frac{(1.5737)^2}{2.87} = 2.5490 - 0.8630 = 1.6860$$

$$s_L = \sqrt{1.6860} = 1.2984$$

A2.6.3 Reproducibility Standard Deviation, s_R —Calculate this statistic using the following equation:

$$s_R = \sqrt{s_L^2 + s_r^2} \quad (\text{A2.11})$$

Thus, for Material C:

$$s_R = \sqrt{1.2984^2 + 1.5737^2} = \sqrt{4.1624} = 2.0402$$

A2.7 Evaluating Laboratory Consistency with Unbalanced Data Sets

A2.7.1 The methodology presented in 15.7 for calculating and using the consistency statistics, h and k , depends on a balanced data set to achieve the required data distributions for the correct analysis of data consistency. When data sets are unbalanced, that methodology must be revised. A more general methodology for the consistency statistics is given, using the numerical calculations for the glucose example with one missing value as an example. Table A2.2 contains the example data and calculated statistics for this section.

A2.7.2 Compute the h Statistics—The between-laboratory consistency statistic, h , checks the consistency of the cell averages for different laboratories. Since the effect of the cell size on the variance of the cell average depends on the partitioning of the total variance into within-laboratory and between-laboratory components, which is not known exactly, this practice calculates the h -statistics for all cells using the estimated variance components s_r^2 and s_L^2 described in A2.6. Section 15 must not be used for the precision estimates with unbalanced data, since these estimates will be biased.

A2.7.2.1 Cell Weighting Factors, w_i —Calculate the cell weighting factors:

$$w_i = \frac{1}{s_L^2 + (s_r^2 / n_i)} \quad (\text{A2.12})$$

TABLE A2.2 Interlaboratory Study Worksheet for Consistency of Glucose in Serum Unbalanced Data for Material C

Laboratory	Test Results, x_{ij}				Cell Statistics						
	1	2	3	n_i	\bar{x}_i	s_i	w_i	\bar{d}_i	h_i	k_i	$k_{C,i}$
1	132.66	133.83	133.10	3	133.197	0.591	0.39819	-1.436	-0.89	0.38	2.04
2	132.92	136.90	136.40	3	135.407	2.168	0.39819	0.774	0.48	1.38	2.04
3	132.61	135.80	135.36	3	134.590	1.729	0.39819	-0.043	-0.03	1.10	2.04
4	138.50	...	135.69	2	137.095	1.987	0.34198	2.462	1.40	1.26	2.57
5	131.90	134.14	133.76	3	133.267	1.199	0.39819	-1.366	-0.85	0.76	2.04
6	137.21	135.14	137.50	3	136.617	1.287	0.39819	1.984	1.23	0.82	2.04
7	130.97	131.59	134.92	3	132.493	2.124	0.39819	-2.140	-1.33	1.35	2.04
8	135.46	135.14	133.63	3	134.743	0.977	0.39819	0.110	0.07	0.62	2.04

Weighted grand average, $\bar{x} = 134.633$

Sum of squares of laboratory deviations, $SS_d = 7.272$

Repeatability standard deviation, $s_r = 1.5737$

Between-laboratory standard deviation, $s_L = 1.2984$

Reproducibility standard deviation, $s_R = 2.0402$

where:

x_{ij} = individual test result (see A2.3),
 \bar{x}_i = laboratory average (see A2.4.1),
 s_i = laboratory standard deviation (see A2.4.2),
 \bar{x} = weighted grand average (see A2.7.2.2),
 w_i = cell weighting factor (see A2.7.2.1),
 \bar{d}_i = laboratory deviation from the weighted grand average (see A2.7.2.3),
 SS_d = weighted sum of squares of laboratory deviations (see A2.7.2.4),
 s_r = repeatability standard deviation (see A2.6.1),
 s_L = between-laboratory standard deviation (see A2.6.2),
 s_R = reproducibility standard deviation (see A2.6.3),
 h_i = between-laboratory consistency (see A2.7.2.5),
 k_i = within-laboratory consistency (see A2.7.3.1), and
 $k_{C,i}$ = critical value for k_i (see A2.7.3.2).

where:

- w_i = the weighting factor for the i^{th} cell,
- s_L = the estimated between-laboratory standard deviation (see Eq A2.10),
- s_r = the estimated within-laboratory standard deviation (see Eq A2.8), and
- n_i = the number of test results from Laboratory i .

Thus, for the unbalanced data set of Table A2.2:

$$w_4 = \frac{1}{1.2984^2 + (1.5737^2 / 2)} = 0.34198$$

and:

$$w_i = \frac{1}{1.2984^2 + (1.5737^2 / 3)} = 0.39819, \text{ for } i \neq 4$$

A2.7.2.2 *Weighted Grand Average, \hat{x}* —Calculate the weighted grand average using Eq A2.13.

$$\hat{x} = \frac{\sum_{i=1}^p w_i \bar{x}_i}{\sum_{i=1}^p w_i} \quad (\text{A2.13})$$

where:

- \hat{x} = the weighted grand average,
- w_i = the cell weighting factors,
- \bar{x}_i = the individual laboratory averages calculated by Eq A2.1, and
- p = the number of laboratories.

In general, \hat{x} will differ from the grand average \bar{x} calculated by Eq A2.4, since different weighting factors are used.

Thus, for the unbalanced data set:

$$\begin{aligned} \hat{x} &= \frac{(0.39819)(133.197) + \dots + (0.34198)(137.095) + \dots + (0.39819)(134.743)}{0.39819 + \dots + 0.34198 + \dots + 0.39819} \\ &= \frac{421.307}{3.12931} = 134.633 \end{aligned}$$

A2.7.2.3 *Laboratory Deviation, \tilde{d}_i* —Recalculate each laboratory deviation by subtracting the weighted grand average, \hat{x} , from the laboratory average using Eq A2.14:

$$\tilde{d}_i = \bar{x}_i - \hat{x} \quad (\text{A2.14})$$

In general, \tilde{d}_i will differ from the laboratory deviation d_i calculated by Eq A2.5.

Thus, for Laboratory 4 of the unbalanced data set:

$$\tilde{d}_4 = 137.095 - 134.633 = 2.462$$

A2.7.2.4 *Weighted Sum of Squares of Deviations, $SS_{\tilde{d}}$* —Calculate the weighted sum of squares of the laboratory deviations using Eq A2.15.

$$SS_{\tilde{d}} = \sum_{i=1}^p w_i \tilde{d}_i^2 \quad (\text{A2.15})$$

Thus, for the unbalanced data set:

$$\begin{aligned} SS_{\tilde{d}} &= 0.39819 \times (-1.436)^2 + \dots + 0.34198 \times (2.462)^2 + \dots \\ &\quad + 0.39819 \times (0.110)^2 = 7.272 \end{aligned}$$

A2.7.2.5 For each laboratory, calculate a value of h using Eq A2.16:

$$h_i = \frac{\tilde{d}_i \times (p - 1)}{\sqrt{\left(\frac{1}{w_i} - \frac{1}{\sum w_j}\right) \times SS_{\tilde{d}} \times p}} \quad (\text{A2.16})$$

where:

- h_i = the between-laboratory consistency statistic for Laboratory i ,
- \tilde{d}_i = the cell deviation for Laboratory i ,
- p = the number of laboratories in the data set,
- w_i = the weighting factor for the i^{th} cell,
- $\sum w_j$ = the sum of all the weighting factors, and
- $SS_{\tilde{d}}$ = the weighted sum of squares of deviations.

Retain two decimal places in the computed values of h_i . To assess data consistency, compare the absolute value $|h_i|$ to the appropriate critical value from Table 5 for the number of laboratories, p .

Thus, for Laboratory 4:

$$h_4 = \frac{2.462 \times (8 - 1)}{\sqrt{\left(\frac{1}{0.34198} - \frac{1}{3.12931}\right) \times 7.272 \times 8}} = 1.40$$

Table 5 gives the critical value 2.15 for h (8 laboratories). Since $|1.40| < 2.15$, there is no apparent data inconsistency.

A2.7.3 *Compute the k Statistics*—The within-laboratory consistency statistic, k , checks the consistency of the cell variances for different laboratories. Since these variances represent only within-laboratory variance, which under the null hypothesis is the same for all laboratories, the methodology for k -statistics with unequal cell sizes does not depend on how the total variance is partitioned into between-laboratory and within-laboratory components.

A2.7.3.1 For each laboratory, calculate a value of k_i using Eq A2.17.

$$k_i = \frac{s_i}{s_r} \quad (\text{A2.17})$$

where:

- s_i = the cell standard deviation calculated by Eq A2.2, and
- s_r = the repeatability standard deviation calculated by Eq A2.8.

Retain two decimal places in the computed values of k_i . These are listed in Table A2.2.

Thus, for Laboratory 4:

$$k_4 = \frac{s_4}{s_r} = \frac{1.987}{1.5737} = 1.26$$

A2.7.3.2 *Critical Values of k* —Do not use Table 5 for critical values of k_i . Instead, calculate a critical value for each k_i using Eq A2.18.

$$k_{C,i} = \sqrt{\frac{p_i}{1 + \frac{p_i - 1}{F}}} \quad (\text{A2.18})$$

where:

- $k_{C,i}$ = the critical value for k_i ,
- p_i = $(N - p) / (n_i - 1)$,

$F = F_{0.995}(n_i - 1, N - p - n_i + 1)$, the 99.5th percentile of the F -distribution with $n_i - 1$ and $N - p - n_i + 1$ degrees of freedom.

Compare the value of k_i to $k_{C,i}$ to decide whether the cell standard deviation for Laboratory i is inconsistent with the rest. Thus, for Laboratory 4:

$$p_4 = \frac{23 - 8}{2 - 1} = 15$$

$$F = F_{0.995}(2 - 1, 23 - 8 - 2 + 1) = F_{0.995}(1, 14) = 11.060$$

$$k_{C,4} = \sqrt{\frac{p_4}{1 + \frac{p_4 - 1}{F}}} = \sqrt{\frac{15}{1 + \frac{15 - 1}{11.060}}} = \sqrt{6.6202} = 2.57$$

Since $1.26 \leq 2.57$, k_4 does not exceed the critical value, indicating no issue with data inconsistency.

A2.7.4 Data Consistency Evaluation—Table A2.2 contains the data and calculations of the h and k consistency statistics for the glucose in serum example. Table 5 gives the (same) critical value 2.15 for each h_i (8 laboratories). The critical value for each k_i is given in the last column of Table A2.2. None of the h_i statistics were outside the two-sided range 0 ± 2.15 , and none of the k_i statistics exceeded their respective critical values, $k_{C,i}$, indicating no issues with data inconsistency.

APPENDIX

(Nonmandatory Information)

X1. SPREADSHEET FOR E691 CALCULATIONS

X1.1 ILS Calculations Using a Spreadsheet

X1.1.1 A spreadsheet for performing Practice E691 calculations is shown in Fig. X1.1 and uses the glucose in serum Material C example in Section 15. This ILS had eight laboratories with each laboratory conducting three replicate test results on each of five materials.

X1.1.2 Data Entry—Test results are entered in cells B3 – D10, one row for each lab. The number of laboratories is entered in B12 and the number of replicates per laboratory is entered in B13, as these values are used in subsequent calculations.

X1.1.2.1 Different numbers of laboratories and replicates per laboratory are accommodated by expanding or contracting the area of the test result entry. This should be done by inserting or deleting the middle rows and columns of the data entry area to preserve the calculation formulas.

X1.1.3 Calculation of Cell Statistics—Five cell statistics are calculated for each laboratory and appear in Columns E – I for this example. The symbols, columns, and spreadsheet formulas for Laboratory 1 are listed in Table X1.1 below. For the other laboratories these formulas can be dragged down through the rows.

	A	B	C	D	E	F	G	H	I
1		Test Results, x			Cell Statistics				
2	Lab	1	2	3	\bar{x}	s	d	h	k
3	1	132.66	133.83	133.10	133.197	0.591	−1.946	−0.73	0.22
4	2	132.92	136.90	136.40	135.407	2.168	0.264	0.10	0.79
5	3	132.61	135.80	135.36	134.590	1.729	−0.553	−0.21	0.63
6	4	138.50	148.30	135.69	140.830	6.620	5.687	2.14	2.41
7	5	131.90	134.14	133.76	133.267	1.199	−1.876	−0.71	0.44
8	6	137.21	135.14	137.50	136.617	1.287	1.474	0.55	0.47
9	7	130.97	131.59	134.92	132.493	2.124	−2.650	−1.00	0.77
10	8	135.46	135.14	133.63	134.743	0.977	−0.400	−0.15	0.36
11									
12	# Labs	8		\bar{x}	135.1429	E12=AVERAGE(E3:E10)			
13	# Reps	3		$s_{\bar{x}}$	2.6559	E13=STDEV(E3:E10)			
14				s_r	2.7483	E14=SQRT(SUMSQ(F3:F10)/B12)			
15				s_L	2.1299	E15=IF((E13^2>(E14^2)/B13),SQRT(E13^2-(E14^2)/B13),0)			
16				s_R	3.4770	E16=SQRT((E14^2)+(E15^2))			
17									

FIG. X1.1 Spreadsheet Example for ILS Calculations

TABLE X1.1 Cell Statistic Symbols and Formulas

Cell Statistic	Symbol	Col	Formula for Laboratory 1
Average	\bar{x}	E	E3=AVERAGE(B3:D3)
Standard deviation	s	F	F3=STDEV(B3:D3)
Deviation from material average	d	G	G3=E3-E\$12
Between-laboratory consistency statistic	h	H	H3=G3/E\$13
Within-laboratory consistency statistic	k	I	I3=F3/E\$14

X1.1.4 Calculation of Statistics for the Material—These calculations are made in Cells E12 – E16 in the spreadsheet example below, and comprise the following:

- E12 Average of cell averages, $\bar{\bar{x}}$
- E13 Standard deviation of the cell averages, $s_{\bar{x}}$
- E14 Repeatability standard deviation, s_r
- E15 Between-laboratory standard deviation, s_L

- E16 Reproducibility standard deviation, s_R

X1.1.5 Disclaimer—This spreadsheet example is not supported by ASTM, and the user of this standard is responsible for its use. For questions pertaining to use of this spreadsheet example, please contact Subcommittee E11.20.

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