Designation: E1601 – 10

StandardPractice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[1]

This standard is issued under the fixed designation E1601; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures and statistics for an interlaboratory study (ILS) of the performance of an analytical method. The study provides statistical values which are useful in determining if a method is satisfactory for the purposes for which it was developed. These statistical values may be incorporated in the method's precision and bias section. This practice discusses the meaning of the statistics and what users of analytical methods may learn from them.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1169 Practice for Conducting Ruggedness Tests

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *interlaboratory test*—measures the variability of results when a test method is applied many times in a number of laboratories.

3.2.2 *replicate results*—results obtained by applying a test method a specified number of times to a material.

3.2.3 *test protocol*—gives instructions to each participating laboratory, detailing the way it is to conduct its part of the interlaboratory test program.

4. Summary of Practice

4.1 Instructions are provided for planning and conducting a cooperative evaluation of a proposed analytical method.

4.2 The following list describes the organization of this practice:

4.2.1 Sections 1-5 define the scope, significance and use, referenced documents, and terms used in this practice.

4.2.2 Section 6 helps users of analytical methods understand and use the statistics found in the Precision and Bias section of methods.

4.2.3 Sections 7 and 8 instruct the ILS coordinator and members of the task group on how to plan and conduct the experimental phase of the study.

4.2.4 Section 9 discusses the procedures for collecting, evaluating, and disseminating the data from the interlaboratory test.

4.2.5 Section 10 presents the statistical calculations.

4.2.6 Sections 11 and 12 discuss the use of statistics to evaluate a test method and the means of incorporating the ILS statistics into Precision and Bias statements.

4.2.7 The Annex A1 gives the rationale for the calculations in Section 10.

5. Significance and Use

5.1 Ideally, interlaboratory testing of a method is conducted by a randomly chosen group of laboratories that typifies the kind of laboratory that is likely to use the method. In actuality, this ideal is only approximated by the laboratories that are available and willing to undertake the test work. The coordinator of the program must ensure that every participating laboratory has appropriate facilities and personnel and performs the method exactly as written. If this goal is achieved, the statistics developed during the ILS will be adequate for

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

determining if the method is capable of producing satisfactory precision in actual use. If the program includes certified reference materials, the test data also provide information concerning the accuracy of the method. The statistics provide a general guide to the expected performance of the method.

6. Statistical Guide for the Users of Analytical Methods Evaluated in Accordance With This Practice

6.1 *Standard Deviations* (for formal definitions, refer to Terminology E135):

6.1.1 *Minimum Standard Deviation of Method, s_M*—This statistic measures the precision of test results under conditions of minimum variability. Because it is improbable that a method in ordinary use will exhibit precision this good, no predictive index is calculated for s_M. Users adept in statistics may wish to compare s_M and the short-term standard deviation of the method measured in their laboratory. For most methods, short-term variability refers to results obtained within several minutes by the same operator using the same equipment. (**Warning—**The standard deviation of results obtained on different occasions, even in the same laboratory, probably will exceed s_M.)

6.1.2 *Between-Laboratory Standard Deviation, s_R*—This statistic is a measure of the precision expected for results obtained in different laboratories. It reflects all sources of variability that operate during the interlaboratory test (except test material inhomogeneity in tests designed to eliminate that effect). It is used to calculate the reproducibility index, *R*. Use s_R for evaluating the precision of methods. It represents the expected variability of results when a method is used in different laboratories.

6.1.3 *Within-Laboratory Standard Deviation, s_r*—This statistic cannot be calculated in a normal interlaboratory test. It is determined only in tests designed to measure variability within laboratories. When this statistic is given in a method, it reflects all variability that may occur from day-to-day within a laboratory (for example, from calibration, standardization, or environmental changes). It is used to calculate the repeatability index, *r*. The user is cautioned that additional sources of variation may affect results obtained in other laboratories.

6.2 *Predictive Indexes*—For the following indexes to apply, these conditions must be met: (*1*) the test materials must be homogeneous; (*2*) analysts must be competent and diligent; (*3*) analytical instruments and equipment must be in good condition; and (*4*) the method must be performed exactly as written (for formal definitions, refer to Terminology E135).

6.2.1 *Reproducibility Index, R*—This statistic estimates the expected range of differences in results reported from two laboratories, a range that is not exceeded in more than 5 % of such comparisons. Use *R* to predict how well your results should agree with those from another laboratory: First, obtain a result under the conditions stated in 6.2, then add *R* to, and subtract *R* from, this result to form a concentration confidence interval. Such an interval has a 95 % probability of including a result obtainable by the method should another laboratory analyze the same sample. For example, a result of 46.57 % was obtained. If *R* for the method at about 45 % is 0.543, the 95 % confidence interval for the result (that is, one expected to include the result obtained in another laboratory 19 times out of 20) extends from 46.03 % to 47.11 %.

NOTE 1—For those not conversant with statistical concepts, it is important to realize that in most such comparisons, the differences will be much smaller than the confidence interval implies. The 50 % confidence interval is only about one third (34.6 %) as wide. Thus, the "average" interval for the above result (one expected to include the result obtained by another laboratory half the time) extends from 46.4 % to 46.8 %. The obvious implication is that, although half the differences will be more than 0.2 %, half will be less than 0.2 %.

6.2.2 *Repeatability Index, r*—This statistic is given in the method only if the interlaboratory test was designed to measure s_r. It estimates the expected range of results reported in the same laboratory on different days, a range that is not exceeded in more than 5 % of such comparisons.

7. Interlaboratory Test Planning

7.1 Analytical test methods start from a perceived need to support one or more material specifications.

7.1.1 Develop a performance requirement for a method from the material specification(s). Include the following factors: expected ranges of chemical compositions of the materials to be covered (method's general scope); specified elements and their concentrations (determination concentration ranges); and the precision required.

7.1.2 Prepare a table of the elements and concentration ranges to cover the critical values in the material specifications. Use this information together with knowledge of the characteristics of the candidate analytical method to select test materials for the interlaboratory program.

7.2 *Draft Method*—The process of developing methods and testing them in a preliminary way is beyond the scope of this practice. All analytical skill and experience available to the task group must be exerted to ensure that the method will meet the project requirements in 7.1 and that it is free of technical faults. A preliminary, informal test of a method must be carried out in several laboratories before the final draft is prepared. Individuals responsible for selecting the method may find helpful information in Practice E691 and Practice E1169. The formal interlaboratory test must not start until the task group reaches consensus on a clearly written, explicitly stated, and unambiguously worded draft of the method in ASTM format, which has completed editorial review.

7.3 *Test Materials*—Appropriate test materials are essential for a successful ILS. The larger the number of test materials included in the test program, the better the statistical information generated. Conversely, the burden of running a very large number of materials may reduce the number of laboratories willing to participate. A method must cover a concentration range extending both above and below the specified value(s). If possible, provide test materials near each limit. Concentration ranges covering several orders of magnitude should be tested with three or more materials.

7.3.1 Material composition and form must be within the general scope of the method. If possible, include all material types the scope is expected to cover. Often, only limited numbers of certified reference materials are available. Use

those that best meet the criteria for the test. If they do not cover all concentration levels, find or prepare other materials to fill in missing values.

7.3.2 The quantity of the material must be sufficient to distribute to all laboratories participating in the test with about 50 % held in reserve to cover unforeseen eventualities.

7.3.3 Materials should be homogeneous on the scale of the test portion consumed in each determination as well as among the portions sent to different laboratories. Usually certified reference materials have been tested for homogeneity, but test materials from other sources may have had only a minimal examination. The use of laboratory-scale melting and casting to produce test materials can sometimes lead to segregation of one or more components in an alloy. Unless specially gathered or prepared materials have been subjected to a thorough homogeneity test, they require the use of Test Plan B. It statistically removes the effect of moderate test material inhomogeneity from the estimates of the ILS statistics.

7.3.4 Test material sent to each laboratory must be permanently marked with its identity in such a manner that the identification is not likely to be lost or obliterated.

7.3.5 If the test program is to evaluate the accuracy of the method, at least one test material must be certified for the concentration of each element in the scope of the method. More certified materials provide more complete information on accuracy.

7.3.6 Prepare a list of the test materials, their identifying numbers, a brief description of material type (for example, low-carbon steel), and approximate concentration of the elements to be determined. This table becomes part of the documentation sent to participating laboratories and provides information needed for the research report and the precision and bias statement.

7.4 *Number of Cooperating Laboratories*—Conventional wisdom holds that the more laboratories participating in an ILS, the better. Further, the laboratory types included in the study task group should consist of typical users' laboratories. There is wide agreement that estimates of precision based upon fewer than six laboratories become increasingly unreliable as the number decreases. A test program involving fewer than six laboratories does not comply with the requirements of this practice (Note 2). An effort should be made to enlist at least seven qualified laboratories before beginning a test program, to allow for attrition. To be qualified to participate, a laboratory must have proper equipment and personnel with sufficient training and experience to enable them to perform the method exactly as it is written.

NOTE 2—If all reasonable efforts fail to recruit at least six cooperating laboratories, up to two of the recruited laboratories may each volunteer to submit two independent sets of test data as an expedient to provide a total of at least six sets of data. Minimum requirements for independence are that two typical analysts, who do not consult with each other about the method, perform the test protocol on different days. They should use separate equipment if possible and must not share calibration solutions or calibration curves.

8. Conducting the Interlaboratory Study (ILS)

8.1 *Program Coordinator*—One individual (presumably the task group chairman) will coordinate the entire ILS, if practical. A prospective ILS program coordinator will find helpful information on conducting the program in Practice E691. One way to organize the work to provide close control while moving the program steadily to its conclusion is as follows:

8.1.1 Prepare a draft of the method to be tested.

8.1.2 Recruit a task group of participating laboratories.

8.1.3 Select a set of test materials and assemble them into kits, one for each laboratory.

8.1.4 Write the test protocol to instruct each laboratory how to run the test.

8.1.5 Prepare a report form.

8.1.6 Establish a realistic time schedule for each part of the test program.

8.1.7 Assemble and deliver to each participating laboratory everything needed to run the test: the draft method; the test materials and a document which describes them; the test protocol; the report forms; a cover letter which includes the deadline for return of results; and the name, address, telephone and fax numbers, and email address of the person who will handle problems and receive the completed report forms. The program coordinator is strongly encouraged to request that all information be returned in electronic format, as most support documentation must be provided to ASTM headquarters in the research report. Refer to the ASTM website for specific requirements regarding the support information that must be provided in the research report. The program coordinator is also strongly encouraged to familiarize himself with the format required for data entry into the program being used for statistical calculations and request that cooperating labs report data in a format amenable to the tool selected for these calculations. For instance, Committee E01 maintains an Excel spreadsheet macro for calculation of Practice E1601 statistics on the ASTM Committee E01 website. The macro program requires that the data for each lab be compiled and entered into a single column. Requiring ILS cooperators to report data in a similar format greatly simplifies use of this statistical tool. If the ASTM Headquarters Statistics support group is used, then they may have specific requirements for data submission.

8.1.8 Expedite the laboratory testing. Follow up to ensure that the laboratories receive the test materials and understand what is expected of them. Encourage laboratories to complete the work.

8.1.9 Inspect results on each report form as it is received. Resolve omissions and apparent clerical errors at once. Obtain missing values. If obvious erroneous data are submitted, determine the cause, if possible, and help the laboratory eliminate the problem. Encourage the laboratory to submit a replacement set of data, if circumstances permit. (The final decision about replacing data will be made by the task group after the testing is complete.)

8.1.10 Perform a preliminary statistical analysis. Summarize the comments from laboratories to explain questionable results. Present this information to the task group.

8.1.11 As approved by the task group, prepare the final statistical evaluation and the research report. Obtain the task group's approval for the completed study.

8.1.12 Modify the scope of the method, if necessary, and prepare the precision and bias statement. Submit the completed

method to the technical subcommittee chairman for editorial review, followed by subcommittee ballot.

8.2 *Task Group*—The task group usually consists of one representative from each participating laboratory. The laboratory representative's name, address, telephone and fax numbers, and email address should be given to the task group chairman when a laboratory agrees to participate.

8.2.1 The laboratory representative shall be fully cognizant of the laboratory's capabilities and be in a position to ensure the following:

8.2.1.1 The laboratory is capable of performing the method properly,

8.2.1.2 Appropriate personnel are assigned to perform the work and the method is followed exactly as written,

8.2.1.3 Test materials are handled properly,

8.2.1.4 The test protocol is complied with in all details,

8.2.1.5 The results are recorded accurately on the report form, and

8.2.1.6 The laboratory adheres to the program time schedule.

8.2.2 As a member of the task group, the laboratory representative must be familiar enough with the analytical techniques used in the method to be able to understand the significance of the test statistics and render considered judgment on how well the method's performance meets the original analytical requirements.

8.3 *Test Protocol*—Preparation of the test protocol is the responsibility of the coordinator. The protocol gives instructions to the participating laboratories such as the following:

8.3.1 *Test Pattern*—Practice E691 requires estimates of the performance of a method under two extreme conditions of variability, minimum variability, and variability among different laboratories. Minimum variability requires that replicate results be obtained with as little elapsed time as possible. For a material of proven homogeneity, specify Test Plan A: three or more sequential replicate results on one portion of the material (Note 3). Direct each laboratory to analyze test materials in random order, but to complete measurements for the replicate results (number specified in the protocol) on one test material before proceeding to another. For a test material of unknown homogeneity, specify Test Plan B (Note 4): sequential duplicate results on at least three portions of the material. Direct each laboratory to obtain the measurements for duplicate results on one test portion, followed by the specified number of other portions of the same material before proceeding to another material. Give explicit instructions to the analyst for each test material, especially if the study uses Test Plan A for some materials and Test Plan B for others.

NOTE 3—In some methods, the test portion is completely consumed in obtaining one result. In these cases, select the sequential test portions to minimize variation in composition, if possible. Any variation that does occur will increase the method's minimum standard deviation.

NOTE 4—Test Plan B is effective only when duplicate results can be taken on a relatively homogeneous test portion. Ideal methods for this approach are those in which replicate test portions can be put into solution and duplicate results obtained on each solution. If determinations are made directly on solid specimens, Test Plan B should be attempted only if each laboratory can be provided with at least three portions of the test material and there is reason to expect that duplicate results on each portion will show less variability than results obtained from different portions.

8.3.2 A third test pattern may be used if the task group wishes to measure the within-laboratory standard deviation, s_r, and calculate the repeatability index, r. Obtain sequential duplicate results on a test material of proven homogeneity on each of at least three days. Direct each laboratory to obtain duplicate results on one test portion of a material on the specified number of (not necessarily sequential) days. Several conditions must be explicitly spelled out in the protocol, as follows:

8.3.2.1 For methods in which samples are dissolved, prepare a single test solution each day. For solid specimens, prepare them each day in the manner specified by the method.

8.3.2.2 Each day the method must be performed in its entirety, including instrument setup, preparation of the calibration solutions and calibration (for methods in which samples are dissolved), and other steps necessary for each day's work in accordance with the method. If the method includes standardization, it must be performed before each day's work whether or not need for it is indicated.

8.3.2.3 Determine the duplicate results on a single test solution. For solid samples, determine the duplicate results with as little disturbance of the specimen as the method permits.

8.3.3 The test protocol specifies analysis requirements incumbent upon the task group lab (see Note 5).

NOTE 5—The following is an illustrative rather than exhaustive example of additional requirements specified in a test protocol: (*1*) specify the number of significant digits with which results are to be recorded for each concentration level (this should be at least one more digit than is expected from the test method in its final form to allow for greater flexibility in statistical review); (*2*) show how to complete the report forms; (*3*) emphasize the importance of keeping written observations that might reveal the cause of unexpected results; (*4*) emphasize the necessity for immediate communication with the coordinator when a problem is encountered; and (*5*) ask for information that might prove useful in the task group's evaluation of the test data, such as a description of test equipment, which is required for the research report.

8.4 *Report Forms*—Provide official report forms to each laboratory. Data forms should be convenient to complete and simple to use when transcribing the data for statistical analysis. Provide spaces for the laboratory to identify itself and the date the test was performed. It is strongly suggested that these report forms be in electronic format (see comments in 8.1.7).

9. Evaluating Data

9.1 The task group must ensure that data are handled properly both in the laboratory and during statistical analysis. Laboratory representatives should be cautioned against submitting "selected" data. For example, a laboratory might be tempted to take extra readings and submit only those that agree well with each other. Such practices or other deviations from the test protocol must not be tolerated because they destroy the integrity of the test design and make correct interpretation of the test results impossible. No result may be rejected just because it does not look good or exceeds a statistical rejection limit. Results may be rejected only when an assignable cause has been documented. Assignable cause is evidence that the

method was not performed as written or that standard laboratory practice was not followed. This may involve human error or equipment malfunction, or both. In this event, the laboratory should correct the problem and, if possible, rerun the test or the portion of the test affected by it. However, laboratory personnel must not make changes in the method. Problems that are perceived as stemming from the method must be discussed with the coordinator. Any unauthorized deviation from the written method, no matter how trivial it may seem to the analyst, may render the laboratory's results unusable.

9.1.1 In the event that a laboratory is unwilling to respond to the task group's request for additional information on how questionable data was obtained, the task group may elect to discard all results from that laboratory. If the task group takes this approach, the reasons must be clearly stated in the research report.

9.2 When test data are received from a laboratory, the coordinator immediately reviews it for consistency and adherence to the test protocol.

9.2.1 The coordinator discusses questionable values with the laboratory representative and clarifies the reasons for rerun data (if any). He transfers the original data to test material tables, marking any values that were questioned or warranted a rerun and recording substitute values (if any) as footnotes. The reasons for proposed deletions or substitutions are documented, observations on the method reported by the laboratories are summarized, and a preliminary statistical evaluation to flag inconsistent data by the *h* and *k* statistics is performed. The coordinator questions laboratories that submitted flagged data to see if assignable causes can be found.

9.3 When all data have been received and the tables and comments have been assembled, the coordinator presents this information to the task group. The task group must decide whether or not the evidence supplied by the contributing laboratory supports rejecting questionable data. When rerun data are presented, it should also consider whether or not the integrity of the test is jeopardized by substitution of the rerun data for the rejected data. If a misunderstanding of the method contributed to a problem, the task group may wish to edit the language of the method (Note 6) to ensure that it will not continue to trouble future users.

NOTE 6—An editorial change to a method, proposed after testing is completed, must be examined carefully to ensure that it does not make or imply a change in the technical substance of the method nor that such a change can be inferred from the edited wording.

9.4 The coordinator performs a final statistical analysis using the data authorized by the task group in the previous step and prepares the research report and the precision and bias section of the method. If the method meets the original project requirements, the task group authorizes its chairman to submit the method to the technical subcommittee chairman for final editorial review and subcommittee ballot. If the task group decides that the method does not meet the requirements, it should examine the test data (with the help of someone who is both adept at using statistics and experienced in analytical chemistry) in order to change the method to improve its performance. Proposed changes to the method should be tested by a small group of laboratories before attempting a full-scale retest. Because such changes affect the technical substance of the method, the revised method must undergo another ILS.

10. Calculation

10.1 The ILS test program measures the variability of the test method in typical laboratories. The between-laboratory standard deviation, s_R, and reproducibility index, *R*, are calculated for this purpose. If the calculated values of these statistics are to reflect the expected future performance of the method, the test data should not contain extraneous results. The *h* and *k* statistics are provided to aid the task group in its search for extraneous data, but the task group is cautioned that statistics alone cannot provide sufficient cause for excluding data. For the relatively small data set produced in a typical ILS using this practice, a result is truly extraneous only if it is caused by errors in chemical manipulations, improper operation of equipment, or failure to follow generally accepted procedures or specific instructions of the method. The task group must use principles of chemistry and physics as well as its analytical experience to show that flagged data are inconsistent with reasonable interpretation and execution of the instructions provided in the method and test protocol. Failing that, the task group must retain the data.

10.2 The equations are arranged for manual calculation of the statistics, but the coordinator is encouraged to use a computer version to save time and avoid errors. A separate statistical analysis is performed for each test material.

10.3 The data for an ILS run according to Test Plan A are shown in Table 1. Each column represents a test material with each laboratory's replicate results in rows.

10.4 *Test Plan A Calculations*—The results of the statistical calculations on the data in Table 1 are displayed in Table 2. (In these equations, *x* represents the replicate results reported by a laboratory, *n* equals the number of replicate results per laboratory, and *p* equals the number of laboratories which provided the data used for this material.)

10.4.1 For each laboratory, calculate the mean $(\bar{x})$, standard deviation (s), and the square of the standard deviation (s^2):

$$\bar{x} = (\sum X/n);$$

$$s = \sqrt{\sum (X - \bar{x})^2/(n-1)};$$

and s^2

10.4.2 Calculate the overall mean result $(\bar{\bar{x}})$for the material:

$$\bar{\bar{x}} = (\sum \bar{x})/p$$

10.4.3 For each laboratory, calculate its laboratory difference (d) and the square of the difference (d^2):

$$d = \bar{x} - \bar{\bar{x}}; \quad \text{and} \quad d^2$$

10.4.4 Calculate the standard deviation of laboratory differences:

$$s_{\bar{x}} = \sqrt{\sum (d^2)/(p-1)}$$

TABLE 1 Nickel ILS Data (% Nickel)

Laboratory Number	Test Materials				
	A	B	C	D	E
1	0.0053	0.053	0.122	0.217	1.08
	0.0053	0.052	0.120	0.215	1.07
	0.0054	0.053	0.120	0.215	1.07
2	0.0057	0.052	0.124	0.207	1.07
	0.0077	0.054	0.124	0.204	1.06
	0.0059	0.053	0.119	0.195	1.05
3	0.0060	0.053	0.120	0.221	1.08
	0.0057	0.055	0.113	0.213	1.05
	0.0060	0.053	0.119	0.220	1.07
4	0.0058	0.057	0.121	0.219	1.06
	0.0053	0.056	0.123	0.225	1.08
	0.0065	0.058	0.130	0.230	1.14
5	0.0058	0.054	0.125	0.220	1.06
	0.0050	0.054	0.123	0.220	1.06
	0.0057	0.053	0.126	0.219	1.08
6	0.0060	0.054	0.120	0.215	1.05
	0.0059	0.054	0.115	0.215	1.05
	0.0060	0.054	0.120	0.210	1.05
7	0.0055	0.056	0.120	0.221	1.05
	0.0060	0.057	0.125	0.221	1.07
	0.0050	0.057	0.125	0.215	1.05
8	0.0069	0.058	0.118	0.218	1.07
	0.0069	0.058	0.121	0.216	1.06
	0.0063	0.057	0.118	0.217	1.08
9	0.0066	0.056	0.117	0.213	1.10
	0.0060	0.057	0.130	0.220	1.05
	0.0062	0.054	0.123	0.225	1.05
10	0.0058	0.055	0.122	0.221	1.08
	0.0056	0.053	0.124	0.223	1.06
	0.0055	0.055	0.120	0.220	1.08
11	0.0049	0.055	0.127	0.220	1.03
	0.0043	0.057	0.132	0.216	1.06
	0.0053	0.054	0.125	0.214	1.05

10.4.5 Calculate the method's minimum standard deviation:

$$s_M = \sqrt{\sum(s^2)/p}$$

10.4.6 Calculate a trial value for the reproducibility standard deviation:

$$s_t = \sqrt{(s_{\bar{x}})^2 + [(s_M)^2 (n-1)/n]}$$

10.4.7 Select the final value for the reproducibility standard deviation:

$$s_R = \text{the larger of } s_t \text{ or } s_M$$

10.4.8 Calculate the reproducibility index and percent relative reproducibility index:

$$R = 2.8(s_R); \quad \text{and} \quad R_{rel} = 100R/\bar{\bar{x}}$$

NOTE 7—The factor of 2.8 (2*sqrt 2) used to calculate R in 10.4.8 and r in 10.6.12 conforms to the calculations for R and r found in Practice E691, 21.1, and originates in Practice E177. For a more complete discussion, see Practice E177, 3 and 27.3.3.

10.4.9 For each laboratory, calculate its between-laboratory consistency statistic:

$$h = d/s_{\bar{x}}$$

10.4.10 For each laboratory, calculate its within-laboratory consistency statistic:

$$k = s/s_M$$

10.5 *Test Plan B Calculations*—Data for a single material obtained in accordance with Test Plan B are shown in Table 3. It is arranged like Table 1, except that space is provided for duplicate results on each replicate portion analyzed by a laboratory. Other test materials in the iron method test are not shown. The results of the statistical calculations start in the last two columns of Table 3 and continue in Table 4. For a test including data for day-to-day within-laboratory variability (replicates analyzed in duplicate on different days in the same laboratory), proceed in accordance with 10.6. For a test including data for material variability (replicates are separate portions analyzed on the one day), proceed in accordance with 10.7.

NOTE 8—In the following equations, x_1 and x_2 represent the duplicate results from one replicate in one laboratory, X represents their mean, n equals the number of replicates per laboratory, and p equals the number of laboratories providing data used in the calculations for one material.

10.6 *Test Plan B—Day-to-Day Variability (see Note 8)*—The replicates are portions of the test material that are analyzed in duplicate on each of several days in each laboratory (see 8.3.2).

10.6.1 For each test portion, calculate the mean of the duplicate results, their difference, and the square of the difference:

$$X = (x_1 + x_2)/2$$
$$D = x_1 - x_2; \quad \text{and} \quad D^2$$

10.6.2 Calculate the method's minimum standard deviation:

$$s_M = \sqrt{\sum D^2/2pn}$$

10.6.3 For each laboratory, calculate the laboratory mean, the standard deviation of the replicate means, and the square of the standard deviation:

$$\bar{x} = (\sum X/n);$$

$$s = \sqrt{\sum(X-\bar{x})^2/(n-1)};$$

and s^2

10.6.4 Calculate the overall mean result for the material:

$$\bar{\bar{x}} = \sum \bar{x}/p$$

10.6.5 For each laboratory, calculate its laboratory difference and the square of the difference:

$$d = \bar{x} - \bar{\bar{x}}; \quad \text{and} \quad d^2$$

10.6.6 Calculate the pooled standard deviation of the replicate means and its square:

$$s_x = \sqrt{\sum s^2/p}; \text{ and } s_x^2$$

10.6.7 Calculate the standard deviation of the laboratory means and its square:

$$s_{\bar{x}} = \sqrt{\sum d^2/(p-1)}; \quad \text{and} \quad s_{\bar{x}}^2$$