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Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials[1]

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1. Scope

1.1 This practice describes techniques for planning, conducting, and analyzing the results of an interlaboratory study of a test method. It is designed to be used in conjunction with Practice C 670. Thus, the procedures recommended in this practice have the limited purpose of providing reliable information on which precision statements of the type described in Practice C 670 can be based. It is not appropriate for use in programs whose purpose is to develop a test method or to assess the relative merits of two or more test methods.

2. Referenced Documents

2.1 *ASTM Standards:*

C 109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)[2]

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[3]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[3]

C 1067 Practice for Conducting a Ruggedness or Screening Program for Test Methods for Construction Materials[3]

E 105 Practice for Probability Sampling of Materials[4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[4]

E 178 Practice for Dealing with Outlying Observations[4]

3. Significance and Use

3.1 Certain criteria need to be met before undertaking an interlaboratory study to determine the precision of a test method. It is not necessary that all of the following conditions described be completely fulfilled in every case; however, if some conditions are not met or are met incompletely, the program will become more complicated and require more work and expense, or may result in impaired information. The recommendations outlined in this section are intended to ensure that the test method is free of technical difficulties to the greatest extent possible before an expensive and time-consuming interlaboratory study is undertaken.

3.1.1 The first requirement is the existence of a valid and well-written test method that has been developed in one competent laboratory (or by cooperative work in a small number of laboratories), and has been subjected to a screening procedure, or to ruggedness testing as described in Practice C 1067. As a result of the screening procedure and some experience with the test method in the sponsoring laboratory and one or two others, a written version of the test method has been developed (but not necessarily published as a standard method) that describes the test procedure in terms that can easily be followed in any properly equipped laboratory. Conditions that affect the test results should be identified and the proper degree of control of those conditions should be specified in the description of the test procedure (see Note 1).

NOTE 1—The desired degree of control of conditions that affect test results may not always be practically achievable, and tolerances in the test method should recognize this fact. Variations in test results due to variations in such conditions contribute to the total variation which determines the precision of the test method. If the resulting variation is so great that uncertainties in average values obtained by the test method are unacceptably high, then the test method itself is at fault, and efforts should be made to improve it or to replace it by a better one. An expensive and time-consuming interlaboratory study should not be undertaken on such a test method.

3.1.2 Any apparatus required for performing the test should be appropriately designed and available at reasonable cost.

3.1.3 Personnel in participating laboratories should have enough experience with the test method so that they are competent to run the test. The importance of this requirement will vary with the complexity of the method and the degree to which it departs from familiar procedures.

3.1.4 Preliminary knowledge should exist about how changes in materials and conditions affect the test results. There should be a reasonable degree of certainty that the within-laboratory variances are the same in different laboratories, and that troublesome interactions do not exist. These

[1] This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates . This practice was developed jointly by ASTM Committee C01, C09, D04, and D18, and is endorsed by all four committees.

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[2] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

conditions are investigated in the analysis of the data of an interlaboratory study, and are discussed further in 8.2.2, 8.2.3, and Appendix X1.

3.1.5 Facilities and procedures for procurement, preparation, and distribution of samples must be available and should be as simple and free of difficulties as practicable.

3.1.6 Selection of samples must be done by a randomization process, and one person who is familiar with randomization procedures should be responsible for seeing that the procedure is carried out. Refer to Recommended Practice E 105.

3.1.7 Adequate numbers of participating laboratories, operators, and materials must be available. Requirements in these areas are specified in Sections 4 and 5.

3.1.8 The entire interlaboratory test program should be developed from the beginning with the help and advice of persons familiar with statistical procedures and with the materials involved (see Note 2). The same persons who design the experiment should also carry out, or at least have control over, the process of analysis of the data.

NOTE 2—It may not always be possible to obtain people who are familiar with the materials involved who have a sufficient knowledge of the proper statistical techniques and their proper use. In this case, the committee should obtain the services of a competent statistician who has experience in practical work with data from materials testing, and provide him with an opportunity for learning something about the particular materials and test method involved. Planners of an interlaboratory study should also be warned to avoid the pitfall of assuming that the use of a large computer necessarily results in special expertise in the handling of data or the interpretation of results.

3.2 It is important to bear in mind that estimates of the precision of a test method are always based on a particular set of data obtained at a particular time and they need to be kept up-to-date. As materials, apparatus, and conditions change, and operators change or gain more experience, the characteristic precision of the results obtained may change, especially if the test method is new. In some cases, it may even be desirable to conduct more tests at a later date (though not necessarily a repetition of the complete interlaboratory study) in order to provide a check on estimates previously obtained and either verify them or introduce revisions.

4. Laboratories

4.1 The problem of obtaining competent participating laboratories for an interlaboratory study is often one of the most difficult ones connected with the process. The number of laboratories available is seldom as extensive as one would like, and if the test method is new, complicated, or expensive and time-consuming to run, the problem is further complicated. The problem usually becomes one of finding and obtaining the cooperation of enough qualified laboratories to obtain meaningful estimates of precision, rather than that of selection among a group of available laboratories. If there is great difficulty in obtaining a sufficient number of competent and willing laboratories, then the possibility exists that the test method should not be subjected to a formal interlaboratory study.

4.2 For the purposes of programs using this recommended practice, it is recommended that at least ten participating laboratories be included **(1, 2).**[5] In cases where it is impossible to obtain ten laboratories, the effect of an increased number may be obtained by repeating the program with the same group of laboratories six months later. Usually, results obtained from the same laboratory after a time lapse of approximately six months display most of the characteristics of results from a different laboratory, especially if a different operator and apparatus can be used. If this procedure is followed, it is necessary to be sure that the same materials are used, and that their characteristics have not changed in the interim.

4.3 In general, it is recommended that any laboratory that is considered qualified to run the test in routine testing situations should be permitted and encouraged to participate. "Qualified" implies proper laboratory facilities and testing equipment, competent operators familiar with the test method, a reputation for reliable testing work, and sufficient time and interest to do a good job. It does not mean, however, that only a select group of laboratories that are considered to be those best qualified for the interlaboratory study should be picked. Precision estimates for inclusion in a test method must be obtained under conditions and through the efforts of laboratories and personnel that are representative of the situations in which the test method will be used in practice **(3).** If a laboratory has all the other requirements, but its personnel has had insufficient experience with the method, the operators in that laboratory should be given an opportunity to familiarize themselves with the method and to practice its application before the interlaboratory study starts.

5. Materials

5.1 The number and type of materials to be included in an interlaboratory study will depend on the following:

5.1.1 The range of the values of the property being measured on a given material and how the precision varies over that range,

5.1.2 The number of different materials to which the test method is to be applied,

5.1.3 The difficulty and expense involved in obtaining, processing, and distributing samples,

5.1.4 The difficulty of, length of time required for, and expense of performing the tests, and

5.1.5 The uncertainty of prior information on any of these points. For example, if it is already known that the precision is 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 known that the precision changes erratically at different levels. A preliminary pilot or screening program may help to settle some of these questions, and may often result in the saving of considerable time and expense in the full interlaboratory study **(4).**

5.2 In general, a minimum of three materials should be considered acceptable.

6. Estimates of Precision

6.1 In accordance with Recommended Practice C 670, the procedure described in this practice is designed to provide two

[5] The boldface numbers in parentheses refer to the list of references at the end of this practice.

basic estimates of the precision of a test method: (*a*) single-operator precision, and (*b*) multilaboratory precision. If other estimates of precision are desired, other references should be consulted (see Practice E 177) **(5).**

6.2 *Single-operator precision* provides an estimate of the difference that may be expected between duplicate measurements made on the same material in the same laboratory by the same operator using the same apparatus within a time span of a few days.

6.3 *Multilaboratory precision* provides an estimate of the difference that may be expected between measurements made on the same material in two different laboratories.

7. Collection of Data

7.1 In order to minimize the problems concerned with analysis of data, a definite form and instructions for obtaining and recording the data should be developed and distributed to all participating laboratories.

7.2 *Directions to Laboratories*—The directions to the laboratories should deal mainly with reporting of data. No special instructions for performing the tests that differ from those given in the description of the test method should be included. The laboratories should be cautioned to conduct tests and report results exactly as specified in the test method, with the one exception as noted in 7.2.2.

7.2.1 *Averaging Test Results*—Laboratories should particularly be cautioned against practices such as running a number of tests and selecting the "best" ones or reporting the average of several determinations, except as such averaging is specified in the test method. For example, Test Method C 109/C 109M specifies three or more test specimens, and requires that the strength of all acceptable test specimens made from the same sample and tested at the same period shall be averaged and reported. In this case, the directions for the interlaboratory test should specify the number of determinations to be obtained and reported. Whenever a test result is defined, either in the test method or in the instructions to laboratories participating in an interlaboratory test program, as the average of a particular number of determinations, the individual determinations should be reported, in addition to the averages. When two or more measurements are averaged to obtain a test result, the data from the interlaboratory test program may be used to develop an estimate of the precision of these individual measurements. See 3.3.3 of Practice C 670.

7.2.2 *Rounding of Data*:

7.2.2.1 Generally, laboratories should be required to report all figures obtained in making the measurements, rather than rounding the results before recording them. In some cases, this may result in recording of more digits than is customary or even more than the test method calls for in the section on Reporting (see X1.3.1). This is necessary because the variation from which information about the precision of the test method comes is contained in the least significant digits, which are often discarded in reporting the results of routine testing **(6).** For example, Method C 136 calls for reporting of percentages to the nearest whole number. This is adequate for the usual reporting purposes, but for purposes of determining precision, at least one decimal place is needed. It is better to require the reporting of too many decimal places than too few, since a decision about rounding all data can be made when the analysis is done. If too few places are reported, however, valuable information may be irretrievably lost, and the result might well be the impairment of the entire program.

7.2.2.2 In cases where a test result is the result of a calculation based on two or more measured quantities, the basic measurements should be used in the calculations without any rounding. The planners of the interlaboratory program will then have to determine how many places need to be reported in order to retain the essential information for determining variability. Sometimes it is advisable to ask the laboratories to report the basic quantities measured instead of, or in addition to, the final calculated result. This enables the final result to be checked, or changes in decisions about the test results to be made, when the data are analyzed. This procedure is especially appropriate if the results are to be analyzed by computer, and the program can be utilized to perform the basic calculations and analyze the calculated results.

7.3 *The Data Sheet*—This practice is based on the following assumptions: p laboratories each have made n replicate measurements on each of q materials (see Ref **7**). Table 1 and Table 2 are sample data sheets for an individual laboratory and for a summary of data for the entire program for a program with: p = ten laboratories, n = four replicates, (test results on each material in each laboratory), and, q = five materials. These data sheets suggest the forms to be used when an individual measurement constitutes the basic test result. In cases where individual measurements are averaged or otherwise subjected to calculation to produce a test result, the form of the individual laboratory sheet may be altered or a secondary sheet provided to permit recording of the fundamental measurements and the test results.

7.4 *Number of Replicates*:

7.4.1 The number of replicate determinations to be made on each material in each laboratory depends largely on the number of laboratories participating, on the homogeneity of the material, and on the expense, difficulty, and time involved in increasing the number of determinations. It should be recognized that in order to obtain the necessary information to write a meaningful precision statement, it is often necessary to use more replicates in the interlaboratory study than is required for

TABLE 1 Data Sheet for an Interlaboratory Test Program for an ASTM Test Method

Laboratory:					
Replicate	Material				
	A	B	C	D	E
a	_____	_____	_____	_____	_____
b	_____	_____	_____	_____	_____
c	_____	_____	_____	_____	_____
d	_____	_____	_____	_____	_____

TABLE 2 Summary Data Sheet for an Interlaboratory Test Program for an ASTM Test Method

Laboratory	Replicate	Material				
		A	B	C	D	E
1	a					
	b					
	c					
	d					
2	a					
	b					
	c					
	d					
3	a					
	b					
	c					
	d					
4	a					
	b					
	c					
	d					
5	a					
	b					
	c					
	d					
6	a					
	b					
	c					
	d					
7	a					
	b					
	c					
	d					
8	a					
	b					
	c					
	d					
9	a					
	b					
	c					
	d					
10	a					
	b					
	c					
	d					

routine use of the test method. An increase in the number of replicates improves the estimates of within-laboratory precision but has no effect on between-laboratory precision **(8).** It is recommended that, for 10 to 15 participating laboratories, at least three replicates should be required. In cases where it is not possible to obtain 10 participating laboratories, the number of replicates, n, should be equal to or greater than $(30/p) + 1$. For more than 15 laboratories, the number of replicates may be reduced to two. (If 30 is not a multiple of p, $30/p$ is rounded to the next higher integer.) This will give an adequte estimate of within-laboratory precision, but information about between-laboratory precision is not as good as desired with fewer than 10 laboratories.

7.4.2 The variation among replicate measurements is supposed to be representative of the irreducible error variance characteristic of the test method. In some cases, it is possible to take supposedly replicate measurements in such a manner that there is little or no opportunity for chance variation, and the measurements are in effect simply repetitions of the same measurement. For example, in making a chemical analysis by atomic absorption or some other kind of automatic measuring device, laboratories have been known to take three readings of the meter on the same sample in quick succession. The three readings so taken were almost identical, but were still reported as replicate readings. In cases such as this, three separate readings with different portions of the sample should be tested possibly on different days, with the same operator and apparatus in order to provide meaningful replicate measurements.

7.5 *Outliers*—In general, the practice of discarding individual test results that appear to differ by suspiciously large amounts from the others, should be avoided unless there is clear evidence that there was some physical reason to consider

the result faulty. It is recommended that no purely statistical criterion be used for the purpose. In particular, laboratories should be asked to report all results in their proper place and include notes describing the conditions surrounding those results that are suspected of being faulty. Sometimes if a test really went wrong, a laboratory should discard the results and repeat the test. Tests should not be repeated, however, just because the results don't look good. Further discussion of the problems of outliers is given in Appendix X2, Practice E 178, and in Refs (**9** and **10**).

7.6 *Missing Data*—Sometimes individual items of data are missing from the summary because they were discarded, failed to be supplied by a laboratory, or for other reasons. In general, if the number of missing data items from all laboratories constitutes no more than 1 % of the total number of items, the analysis may be conducted as though the missing items were present. For example, if one result out of four replicates on a given material from a given laboratory is missing, the three remaining results should be added and then divided by 3 to get the average, $\bar{x}_i$. The within-laboratory variance, s_i^2, should also be calculated using 3 for the number of results. From then on, both results should be used as though they were based on four measurements. If the number of missing results exceeds 1 % of the total, some of the tests should be repeated in order to obtain proper measurements for the missing values. Missing values handled in this way must be individual values distributed throughout the mass of data, and should not be concentrated as a group in one laboratory-material cell. If the latter occurs, the laboratory should provide another group of measurements on the material in question. Analysis-of-variance procedures exist for the analysis of such unbalanced sets of data. The advice of a statistical consultant should be obtained when such practices are used.

8. Analysis of Data

8.1 The procedure described herein is simplified, and statistical terms are avoided to the greatest extent possible in order to make the recommended practice easily usable by persons with little statistical background. This exposes the recommended practice to the danger that, although the technique recommended is widely applicable to many situations using many kinds of data, it may be used mechanically in situations in which it is not applicable by persons who are not familiar with the statistical background of the recommended procedures. For this reason, it is recommended to seek the advice of a person who is familiar with the statistical procedures before undertaking analysis of an interlaboratory study by this or any other published procedure. An example of the procedure is given in Appendix X1. For further description of the method, see Ref (**5**).

8.2 *Between-Laboratory and Within-Laboratory Analysis for Each Material*—The first step in the analysis is to obtain estimates of between-laboratory and within-laboratory variances for each material. This may be done by using the form shown in Table 3. Table 3 is set up as an example, using material A in ten laboratories with four replicate test results per laboratory to correspond with the sample summary data sheet in Table 2. Similar tables should be set up for each material in the study. The subscript i is used to designate a particular laboratory in the analysis and goes from 1 to p, the total number of laboratories. Capital letter subscripts, A, B, etc., are used to designate quantities calculated for the different materials. The averages, $\bar{x}_i$, and variances, s_i^2, in the last two columns are the within-laboratory averages and variances for the given material, and are calculated from the n replicate test results for each of the p laboratories as follows:

$\bar{x}_i = \Sigma x_i/n$ = sum of n replicate test results for laboratory i divided by n.

$s_i^2 = (\Sigma x_i^2 - n\,\bar{x}_i^2)/(n-1)$ = sum of squares of n replicate test results for laboratory i less n times the square of the average for laboratory i, divided by one less than the number of replicate test results.

NOTE 3—The results of the calculations described here may be subject to a rounding error if the numbers involved are large. See Appendix X1 and Note X1.1 for an example of this and a discussion of how to deal with this problem.

From the p individual within-laboratory averages and variances, four quantities for the given material; namely, the overall average, pooled

TABLE 3 Between and Within Analysis for Material A[A]

Laboratory	Data				Average $\bar{x}_1$	Within-Laboratory Variance s_1^2
	a	b	c	d		
1	_____	_____	_____	_____	$\bar{x}_1$	s_1^2
2	_____	_____	_____	_____	$\bar{x}_2$	s_2^2
3	_____	_____	_____	_____	$\bar{x}_3$	s_3^2
4	_____	_____	_____	_____	$\bar{x}_4$	s_4^2
5	_____	_____	_____	_____	$\bar{x}_5$	s_5^2
6	_____	_____	_____	_____	$\bar{x}_6$	s_6^2
7	_____	_____	_____	_____	$\bar{x}_7$	s_7^2
8	_____	_____	_____	_____	$\bar{x}_8$	s_8^2
9	_____	_____	_____	_____	$\bar{x}_9$	s_9^2
10	_____	_____	_____	_____	$\bar{x}_{10}$	s_{10}^2

[A] p = 10 laboratories.
n = 4 replicate test results on each material in each laboratory.
Overall average $\bar{x}_A$ =
Pooled within-laboratory variance s_A^2 (pooled) =
Variance of laboratory averages $s_{\bar{x}_A}^2$ =
Between-laboratory component of variance $s_{L_A}^2$ =

within-laboratory variance, variance of laboratory averages, and between-laboratory component of variance, are calculated and entered on Table 3 as follows:

$\bar{x}_A$ = $\Sigma\ \bar{x}_i/p$ = sum of p averages for the laboratories divided by p

s_A^2 (pooled) = $\Sigma s_i^2/p$ = average of within-laboratory variances (see Note 5).

$s_{\bar{x}_A}^2$ = $[\Sigma\ \bar{x}_i^2 - p\ (\bar{x}_A)^2]/(p - 1)$ = sum of squares of p within-laboratory averages less p times the overall average squared, divided by $p - 1$.

$s_{L_A}^2$ = $s_{\bar{x}_A}^2 - [s_A^2\ (\text{pooled})/n]$ = the variance of laboratory averages less $1/n$ times the pooled variance.

A sample work sheet showing exactly how these calculations are made appears in Appendix X1.

NOTE 4—The method of pooling variances used here applied only when all the individual variances being pooled are based on the same number of measurements. In general, a pooled estimate of a variance is not obtained by averaging individual variances.

8.2.1 Before proceeding with the analysis, it is necessary to investigate agreement of the data with the following two assumptions: (*a*) the variances are the same in different laboratories (homogeneity of variance), and (*b*) the results show the same pattern of change from one material to another in different laboratories (lack of interactions). These two aspects of the analysis are discussed in 8.2.2 and 8.2.3.

8.2.2 *Investigation of Agreement of Variances*—This method is based on the assumption that the within-laboratory variances in different laboratories (of which the s_i^2 in Table 3 and its variations, are estimates) are the same. This does not mean that the s_i^2 have to be very close together, since an individual variance can be about four times the average variance (for $p = 10$ and $n = 4$) when all the calculated variances are really estimates of the same variance. In order to check for agreement among variances, it is helpful to plot the individual variances against the laboratories, draw a horizontal line across the plot at the level of the average variance, and examine the lowest and highest individual variances. A variance that is very low compared to the others may indicate that the laboratory is not permitting the normal causes for variation between results to show up, while a high variance indicates the lack of proper control of the testing process.

8.2.2.1 Table 4 gives approximate values (upper 5 % level) for the ratio of the largest variance to the sum of the variances that should not be exceeded **(11)**.

8.2.2.2 The case of a small variance is not usually as troublesome as that of a variance that is too large. However, when one laboratory performs its tests in such a way that the normal causes of variation do not affect the results, an unrealistically low variance may occur. If no significantly high variance is present, as judged by the criterion given above, the following method may be used to examine a suspiciously low variance. The statistic used is the ratio of highest to lowest variance in the group. Table 5 gives the approximate values (upper 5 % level) for this ratio that should not be exceeded (**12**).

TABLE 4 Approximate Values (Upper 5 % Level) for the Ratio of the Largest Variance to the Sum of the Variances

No. of Laboratories	No. of Replicates				
	2	3	4	5	6
5	0.8412	0.6838	0.5981	0.5441	0.5065
6	0.7808	0.6161	0.5321	0.4803	0.4447
7	0.7271	0.5612	0.4800	0.4307	0.3974
8	0.6798	0.5157	0.4377	0.3910	0.3595
9	0.6385	0.4775	0.4027	0.3584	0.3286
10	0.6020	0.4450	0.3733	0.3311	0.3029
11	0.5700[A]	0.4140[A]	0.3480[A]	0.3070[A]	0.2810[A]
12	0.5410	0.3924	0.3264	0.2880	0.2624
13	0.5140[A]	0.3630[A]	0.3080[A]	0.2690[A]	0.2470[A]
14	0.4920[A]	0.3450[A]	0.2910[A]	0.2530[A]	0.2320[A]
15	0.4709	0.3346	0.2758	0.2419	0.2195
20	0.3894	0.2705	0.2205	0.1921	0.1735
30	0.2929	0.1980	0.1593	0.1377	0.1237

[A] Values obtained by graphic interpolation.

TABLE 5 Approximate Values (Upper 5 % Level) for the Ratio of Highest to Lowest Variance

No. of Laboratories	No. of Replicates				
	2	3	4	5	6
5	[A]	202	51	25	16
6	[A]	266	62	30	19
7	[A]	333	73	34	21
8	[A]	403	84	38	23
9	[A]	475	94	41	25
10	[A]	550	104	45	26
11	[A]	626	114	48	28
12	[A]	704	124	51	30
13	[A]	790[B]	135[B]	54[B]	31[B]
14	[A]	885[B]	145[B]	57[B]	32[B]
15	[A]	995[B]	155[B]	59[B]	33[B]

[A] Although it may be possible to calculate this value, it is suggested that all values be included in the analysis when only 2 replicates are used.
[B] Value obtained by graphic extrapolation.

8.2.2.3 Often the data from one laboratory may indicate a high or low variance compared to the others, and elimination of that laboratory from the analysis results in a set of data with similar variances for the remaining laboratories (see Appendix X1). If all the variances are erratic, however, the test method is in trouble. Efforts to develop precision statements from the data should be suspended and further study of the test method should be undertaken to determine the causes for such erratic behavior. The advice of a statistical consultant should be obtained whenever there is doubt about eliminating a high or low variance.

8.2.3 *Interactions*—A common problem with test results obtained from an interlaboratory study is the presence of interactions between laboratories and materials. This means that the pattern of change of the results obtained on a given group of materials in one laboratory differs from the pattern obtained in another laboratory. In extreme cases, different laboratories may even fail to rate materials in the same order. The accepted statistical technique for finding significant interactions is an analysis of variance. A reasonably reliable method for checking to see if troublesome interactions may exist, however, is to make a plot of the averages obtained on the materials by each laboratory (see X1.3.5). These plots should show similar patterns of change from material to material for all laboratories. One laboratory may show a noticeably different pattern from the others and may be eliminated. However, if