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Standard Practice for Interlaboratory Testing of a Textile Test Method that Produces Normally Distributed Data[1]

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1. Scope

1.1 This practice serves as a guide for planning interlaboratory tests in preparation for the calculation of the number of tests to determine the average quality of a textile material as discussed in Practice D 2905 and for the development of statements on precision as required in Practice D 2906.

1.2 The planning of interlaboratory tests requires a general knowledge of statistical principles including the use of variance components estimated from an analysis of variance. Interlaboratory tests should be planned, conducted, and analyzed after consultation with statisticians who are experienced in the design and analysis of experiments and who have some knowledge of the nature of the variability likely to be encountered in the test method.

1.3 The instructions in this practice are specifically applicable to design and analysis of:

1.3.1 Single laboratory preliminary trial,

1.3.2 Pilot-scale interlaboratory tests, and

1.3.3 Full-scale interlaboratory tests.

1.4 Guides for decisions pertaining to data transformations prior to analysis, the handling of missing data, and handling of outlying observations are provided.

1.5 Procedures given in this practice are applicable to test methods based on the measurement of continuous variates from normal distributions or from distributions which can be made normal by a transformation. Get qualified statistical help to (*1*) decide if the data are from another known distribution, such as the Poisson distribution, (*2*) make a judgment on normality, (*3*) transform data to a more nearly normal distribution, or (*4*) use Practice D 4467. Use the procedures in Practice D 4467 for test methods that produce data that are (*1*) continuous data that are not normally distributed or (*2*) discrete data, such as ratings on an arbitrary scale, counts that may be modelled by use of the Poisson distribution, or proportions or counts of successes in a specified number of trials that may be modelled by the binomial distribution.

NOTE 1—Additional information on interlaboratory testing and on statistical treatment of data can be found in Practice D 1749, D 3040, E 173, E 177, E 691, and Terminology E 456.

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

D 1749 Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products[3]

D 2905 Practice for Statements on Number of Specimens for Textiles[2]

D 2906 Practice for Statements on Precision and Bias for Textiles[2]

D 3025 Practice for Standardizing Cotton Fiber Test Results by Use of Calibration Cotton Standards[2]

D 3040 Practice for Preparing Statements for Standards Related to Rubber and Rubber Testing[4]

D 4270 Guide for Using Existing Practices in Developing and Writing Test Methods[5]

D 4467 Practice for Interlaboratory Testing of a Textile Test Method that Produces Non-Normally Distributed Data[5]

D 4853 Guide for Reducing Test Variability[5,6]

E 173 Practices for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[7]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[8]

E 178 Practice for Dealing with Outlying Observations[8]

E 456 Terminology Relating to Quality and Statistics[8]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[8]

2.2 *ASTM Adjuncts:*

TEX-PAC[9]

NOTE 2—Tex-Pac is a group of PC programs on floppy disks, available through ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428, USA. The calculations required by the Annexes of this practice can be performed using this adjunct and the ouput is printed in a format suitable for direct insertion in the Research Report required when

[1] This practice is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.93 on Statistics.

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] *Annual Book of ASTM Standards*, Vol 15.09.

[4] Discontinued. See *1988 Annual Book of ASTM Standards*, Vol 09.01.

[5] *Annual Book of ASTM Standards*, Vol 07.02.

[6] Discontinued. See *1993 Annual Book of ASTM Standards*, Vol 07.02.

[7] *Annual Book of ASTM Standards*, Vol 03.05.

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

[9] PC programs on floppy disks are available through ASTM. Request ADJD2904.

an interlaboratory evaluation is conducted for the purpose of establishing the precision of a Test Method.

3. Terminology

3.1 For definitions of textile and statistical terms used in this practice, and discussions of their use, refer to Terminologies D 123, and E 456 and appropriate textbooks on statistics (**1-9**).[10]

4. Summary of Practice

4.1 Planning and running an interlaboratory test program presumes that the test method has been adequately developed as directed in Sections 1–7 of Guide D 4270.

4.2 In this practice, directions are given on how to run a pilot-scale interlaboratory test to validate the state of control for a test procedure. A pilot-scale test is run to decide whether (*1*) the procedures for the test method and for the interlaboratory test program are adequate or (*2*) more development work needs to be done on one or both of the procedures.

4.3 Directions are given on how to run a full-scale interlaboratory test.

4.4 Directions are given on making data transformations, handling missing data, testing outlying observations, and running auxiliary tests.

4.5 In Annex A1, the following steps are described on how to examine the data from either the pilot-scale or full-scale interlaboratory tests.

4.5.1 Analyze the data by materials by preparing an analysis of variance table for each material.

4.5.2 Validate a state of statistical control by testing the mean squares in the analysis of variance tables for significant effects. If significant effects are found, a decision must be made on whether to (*1*) return to further development of the test procedure or the instructions for the interlaboratory test, or both, or (*2*) continue with the analysis of the data from the interlaboratory test.

4.5.3 Make a decision on whether to (*1*) combine the data from all materials into a single analysis of variance, (*2*) combine the data into a single analysis of variance with variability expressed as a transformation such as coefficients of variation or (*3*) stop the analysis and write separate statements on precision for each material.

4.5.4 If a decision is made to combine the data from all materials, analyze the data from all materials as a single analysis of variance and validate a state of control by testing for significant effects. If significant effects are found, a decision must be made on whether to (*1*) return to further development of the test procedure or the instructions for the interlaboratory test, or both, or (*2*) continue with the analysis of the data from the interlaboratory test.

4.5.5 Calculate the necessary components of variance for use as directed in Practice D 2905 and Practice D 2906.

5. Significance and Use

5.1 Interlaboratory testing is a means of securing estimates of the variability in results obtained by different laboratories, operators, equipment, and environments when following procedures prescribed in a specific test method and of determining that the method produces results of essentially uniform variability and at a consistent level when the same materials are tested in a number of laboratories.

5.2 The estimates of the components of variance from the interlaboratory test provide the information needed for the preparation of statements on the number of specimens and on precision as directed in Practices D 2905 and D 2906.

6. Basic Statistical Design

6.1 It is desirable to keep the design as simple as possible, yet to obtain estimates of within and between-laboratory variability unconfounded with secondary effects. Provisions also should be made for estimates of the variability due to: materials times laboratories, operators times materials interactions, and instruments within laboratories where two or more instruments may be used in one laboratory.

NOTE 3—Generally, for a test method, there are only a limited or fixed number of laboratories or operators in each laboratory who participate in the interlaboratory tests. Since all do not participate, one assumes that the sampling of laboratories, and operators within laboratories are drawn from a larger population of such laboratories or operators. For this reason, an analysis of variance (ANOVA) model based on random effects is used **(1, 3, 4,** and **8)**. Since specimens are always a random effect, a fixed ANOVA model does not normally apply.

6.2 The basic statistical design should include: a minimum of two or more materials spanning the range of interest for the property being measured, a minimum of five laboratories, and a minimum of two operators per laboratory with each operator testing at least two specimens of each material in a designated order. There is, generally, no major advantage in having the degrees of freedom for error exceeding 40, but it is desirable for the degrees of freedom for all other mean squares to be as large as practical. This basic design may be expanded according to the experience of the task group, the number of laboratories available to perform the specified tests and the degree of heterogeneity (or homogeneity) of the test materials.

6.3 The Laboratory Report Format is represented in Fig. 1 by a two-way classification table in which the rows represent the materials and the columns represent the operators in the laboratory. Each cell contains the replicate observations per operator.

6.4 A basic analysis of variance (ANOVA) design should be a randomized complete-block design or other more suitable factorial, having the following successive subsets:

6.4.1 Materials, *M*,

6.4.2 Laboratories, *L*,

6.4.3 Operators in laboratories, *O*(*L*), and

6.4.4 Specimens per operator within laboratories and materials, *S* (*MLO*).

6.5 The basic statistical design outlined in 6.2-6.4 will provide the following estimated components of variance:

6.5.1 Specimens within operators, laboratories, and materials, *S·MLO*,

6.5.2 Operator times materials interactions within laboratories, *MO·L*,

6.5.3 Operators within laboratories, *O·L*,

6.5.4 Materials times laboratories interactions, *ML*,

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

Material Number	Laboratory Number____						
	Operator Number						
	1	2	3	...	*j*	...	*q*
1	...	...	...		...		...
	...	...	...		...		...
							...
	...	...	...		...		
2	...	...	...		...		...
	...	...	...		...		...
	...	...	...		...		...
3	...	...	...		...		...
	...	...	...		...		...
	...	...	...		...		...
...					*ij* cell		
i	...	...	...				...
	...	...	...		...		...
					...	*n*	
	...	...	...		...		...
P	...	...	...		...		...
	...	...	...		...		...
	...	...	...		...		...

FIG. 1 Laboratory Report Format for Interlaboratory Test Data Involving *p* Materials, *q* Operators, and *n*Tests or Replications

6.5.5 Laboratories, *L*, and

6.5.6 Materials, *M*.

6.6 A range of materials should be intentionally chosen so that the component of variance for materials will be significant. Since this component is not used in estimating the precision of the test method, it is normally not calculated from the associated mean square in the analysis of variance (ANOVA) table.

6.7 The estimates of the components of variance provided for in 6.5 provide the basic data for estimating the number of tests required for a specified allowable variation as directed in Practice D 2905 and for the preparation of statements on precision as directed in Practice D 2906.

6.8 An illustrative example of a full-scale interlaboratory design and its analysis is shown in Annex A1.

7. General Considerations

7.1 *Sampling of Materials*—It is desirable that any one subsample of the material, within which laboratories, operators, days, or other factors are to be compared, be as homogeneous as possible with respect to the property being measured. Otherwise, increased replication will be required to reduce the size of the random error.

7.2 *Complete Randomization*—Divide all the randomized specimens of a specific material, after labeling, into the required number of groups, each group corresponding to a specific laboratory (see 1.2).

NOTE 4—Guides for selection of samples may be found in standard tests (for example, **4, 8**).

7.3 *Partial Randomization*—In some cases, it is advantageous to follow a systematic pattern in the allocation of the specimens to laboratories. For example, if the specimens are bobbins of yarn from different spinning frames, it is often desirable to allocate to each laboratory equal numbers of specimens from each spinning frame. In such cases only the specimens within each spinning frame are randomized, rather than all of the specimens from all frames (see 1.2).

7.4 *Number of Replicate Specimens*— The number of specimens tested by each operator in each laboratory for each material may be calculated from previous information or from a pilot run. This number of specimens or replications (minimum of two) depends on the relative size of the random error and the smallest systematic effect it is desired to be able to detect. A replication consists of one specimen of each condition and material to be tested in the statistical design.

NOTE 5—It is desirable to test a larger number of materials in more laboratories with the number of operators per laboratory and the number of tests per operator at a minimum. When the established sampling error for material exceeds 5 % of the material mean, (when tested by one skilled operator in one laboratory), increased test replication may be necessary.

7.5 *Order of Tests*—In many situations, variability among replicate tests is greater when measurements are made at different times than when they are made together, as part of a group. Sometimes trends are apparent among results obtained consecutively. Furthermore, some materials undergo measurable changes within relatively short storage periods. For these reasons, the dates of testing, as well as the order of tests carried out in a group shall, wherever possible, be treated as controlled systematic variables.

7.6 *Alternative Methods*—When possible, values for each material should be established by alternative test methods to determine if there is a variable bias between the proposed method and the referee method at different levels of the property.

7.7 *Gain of Statistical Information*— More statistical information can be obtained from a small number of determinations on each of a large number of materials than from the same total number of determinations distributed over fewer materials. In the same way, a specific number of determinations per material will yield more information if they are spread over the largest number of laboratories possible. The task group should consider a minimum starting design having two specimens (replications) per material for each operator, two operators per

laboratory, as many laboratories as possible, and at least two materials. If experience with pilot-scale interlaboratory tests casts doubt on the adequacy of this starting design, estimate the number of determinations needed to detect the smallest systematic difference of practical importance (see also Note 5).

7.8 *Multiple Equipment (Instruments)*— When multiple instruments within a laboratory are used, tests must be made on all equipment to establish the presence or absence of the equipment effect. If equipment effect is present and cannot be eliminated by use of pertinent scientific principles, known standards should be run and appropriate within-laboratory quality control procedures should be used.

8. Single-Laboratory Preliminary Trial

8.1 The subcommittee with responsibility must establish an acceptable level of precision desired for the test method under evaluation.

8.2 The test method to be evaluated should be reviewed to determine if there are any variables that need to be controlled to obtain acceptable precision under the optimum conditions of a single operator. If there is any doubt as to the existence of any such conditions a single laboratory preliminary trial or ruggedness test should be conducted as directed in Guide D 4853.

8.3 If the analysis of the ruggedness test shows that there are method variables that need to be controlled to obtain acceptable precision then the method should be modified and the method subjected to another ruggedness test in accordance with Guide D 4853. This procedure should be repeated until the acceptable level of precision is achieved.

8.4 Failure to conduct a ruggedness evaluation may result in (1) a poorly written test method being used in the full-scale interlaboratory tests, or (2) excessive between-laboratory variances, or both.

9. Pilot-Scale Interlaboratory Test

9.1 If the method is new or represents major modifications of an existing method, it may be desirable to conduct a pilot study utilizing two or three materials of established values (low, medium, and high values of the property under evaluation) and preferably three to four laboratories. A minimum of two operators per laboratory should make a minimum of two tests each per material. Misleading or ambiguous directions in the procedure should be detected in the pilot-scale interlaboratory test.

9.2 Prepare a definitive statement of the type of information the task group expects to obtain from the interlaboratory study, including the analysis of variance.

9.3 Based on the data of a single-laboratory preliminary trial, prepare the basic analysis of variance (ANOVA) design plan and circulate it to all task group members and other competent authorities for review and criticism. Also include suggested materials that cover the range of the property to be measured and that represent all classes of the material for which the method will be used.

9.4 Conduct the pilot-scale interlaboratory test using the analysis of variance (ANOVA) design plan.

9.5 Analyze the data using the plan described in 9.3.

9.6 On the basis of the data analysis from the pilot run and comments from the cooperating laboratories, revise instructions and procedures to minimize operator and instrument variances to the extent practicable.

10. Full-Scale Interlaboratory Test

10.1 After a thorough review of procedural instructions and evaluation of pilot run data as specified in Section 9, canvass the potential participating laboratories to ascertain the number and extent of participation in a full-scale test. If practicable, secure a reasonably large number of laboratories (minimum of five suggested), each testing a series of materials, using two or three operators per laboratory and two or more specimens per operator per material. If fewer than five laboratories participate in the interlaboratory test, the statement on precision should include a cautionary note as directed in Practice D 2906.

10.2 Obtain adequate quantities of a series of homogeneous materials covering the general range of values expected to normally be encountered for the test method. Subsample each of these materials so as to ensure subsample homogeneity. Select subsamples of each material for distribution to each participating laboratory. From each material, allocate enough specimens to provide for all participating laboratories and a sufficient number of additional specimens for replacement of lost or spoiled specimens. Label each specimen by means of a code symbol and record the coded identification of the specimens for further reference. Store and maintain reserve specimens in such a manner that the characteristic being studied does not change with time.

11. Data Transformation

11.1 In the analysis of variance, there is an assumption of uniformity of error variances. Departure from this assumption may result in actual probabilities being different from those given by the significance tables. Some of the more common data transformations are given in the following:

11.1.1 In cases where the range in mean values for the material utilized in an interlaboratory study exceeds 100 % of the smaller mean, consideration should be given to some type of data transformation prior to subjection of the data to an analysis of variance. Bartlett **(2)** gives many of the principal transformations that have been found useful.

11.1.2 If sample means are proportional to variances of the respective samples, or the data has a Poisson distribution, use the square root transformation.

11.1.3 If sample means are proportional to the range or standard deviation of the samples, the replacement of each measurement with its logarithm frequently results in variances which are more nearly equal. In many applications and logarithmic transformation tends to normalize the distribution. The utilization of graph paper having a logarithmic scale on one axis and normal probability scale on the other axis simplifies examination of transformed data for normality.[11] Transformed data having a normal distribution will result in an approximately straight line cumulative distribution polygon.

[11] Normal Probability Graph Paper may be bought from most suppliers. The equivalent of Keuffel and Esser Co. Style 46-8000 or of Codex Book Co., Inc., Norwood, MA 02062, Style 3127, is acceptable.

11.1.4 In tests for percent defective where the distribution tends to be binomial in form, the data generally transformed utilizing the arcsin transformation **(9)**.

11.1.5 In cases of grades or scores, the square root transformation is frequently useful. Where the minimum score is one, use the square root of the score. When the minimum score is zero, use the square root of the score plus one.

11.1.6 Other transformations based on knowledge of the scalar values frequently are suggested by the units of measurement, the character of the measurement, and the scientific principles involved in the measurement.

11.2 Decoding of transformed data shall be done after completion of the analysis of variance and mean separation of the analysis of variance and mean separation by procedures such as the Duncan Multiple Range **(5, 6)** procedure. Conclusions should be stated in terms of the decoded data.

12. Missing Data

12.1 Occasionally, in the conduction of interlaboratory tests, accidents may result in the loss of data in one or more cells of a two-way table. Missing items nullify the addition theorem for sums of squares. Correct analysis of data with missing items can be made by use of the theory upon which the methods of calculation are based, that is least squares. Procedures for missing data calculations and their treatment in the analysis of variance can be found in standard statistical texts **(1, 3, 7,** and **8).**

13. Outlying Observations

13.1 In laboratory testing it is generally advisable to retain all test data. Exceptions to this general policy should be made only when assignable causes for deletion of a test value are present. Examples of assignable cause would be; the operator observed some instrument malfunction, sample preparation error, or other circumstance that should logically result in the termination of the test procedure at that specific point. In cases where there is no assignable cause for a test value being out of line, the test value should be retained and reported.

NOTE 6—Although this practice recommends deletion of data only for assignable cause, other procedures for detecting and dealing with outlying observations are specified in Practice E 178.

14. Interpretation of Data

14.1 If the mean square for laboratories is significant, examine and determine which laboratory mean contributed to the significant laboratory mean square (see 15.2). On the basis of this information, ascertain actual test conditions and instrument setups that may have contributed to this significant laboratory mean square. Where significant differences in laboratory level exist and cannot be eliminated, the task group should consider and evaluate the effect of adjustment of laboratory levels to a standard level by use of a correction factor based on the ratio of the level of results in each laboratory to the established value for the materials.

14.2 Where a significant laboratory times materials interaction occurs, reevaluate procedure instructions and instrument setups. Two different instruments, although calibrated and adjusted in accordance with manufacturer's instructions may give different values for a series of materials.

14.3 Where significant operator within-laboratory differences occur, reevaluate procedural instructions and examine operator techniques to find differences in preparation, in procedures, or both. If between-operator differences cannot be eliminated, consider the use of standard calibration materials and adjust the operator data by comparisons of standard values to operator values utilizing appropriate quality control procedures. An example of how this adjustment may be done is found in Practice D 3025. (For more detailed information refer to **(9)**.)

15. Auxiliary Tests

15.1 *Plotting Data to Facilitate Interpretation*:

15.1.1 Plot the averages by laboratories for each material. Utilize two- and three-sigma limits centered on the mean for a given material and sigma equal to the standard error of the mean of all specimens of the material tested in a laboratory.

15.1.2 Plot, within each laboratory, the ranges among operators for each material. Utilize two- and three-sigma limits centered on the average range for all laboratories (for each material) and the sigma equal to standard error for ranges based on the mean of all specimens of the material tested in a laboratory.

15.1.3 Plot the ranges among operators from a single laboratory for each material, with two- and three-sigma limits centered on the average range for all laboratories for a single material and the standard error for ranges based on the mean of all specimens of the material tested in a laboratory.

15.1.4 Plot, by laboratories, the averages for each operator within each laboratory for each material. Utilize two- and three-sigma limits centered on the average for each material and the standard error based on the mean of all specimens of the material tested in all laboratories.

15.1.5 Plot the ranges of values for each operator within each laboratory for each material. Utilize two- and three-sigma limits centered on the average range for each material by laboratory and the standard error of ranges based on the mean of all specimens of the material tested in all laboratories.

15.1.6 Plot the residual (material mean − specimen value) by specimen for each operator by laboratory for each material.

15.1.7 Plot the effect of a possible *ML* interaction. The plot would have the average response of a material in a laboratory as the ordinate and the materials as the abscissa. A series of curves, each showing the average response of a single laboratory by materials, should be plotted with the points indicated by laboratory number.

15.1.8 Plot of the effect of a possible *MO* (*L*) interaction. There would be a series of plots for each laboratory with the average response by operator as the ordinate, the materials as the abscissa, and a curve for each operator within the laboratory showing the response of the operator for each material and with the points plotted using the operator number.

15.2 *Duncan's Multiple Range Test*—The Duncan's Multiple Range Test **(5, 6)** is a procedure for mean separation. The procedure may be useful for separation of both operator and laboratory means.

16. Keywords

16.1 interlaboratory testing; precision; statistics

ANNEXES

(Mandatory Information)

A1. ANALYSIS OF DATA AND REPORTING USING THE STANDARD DESIGN

A1.1 The methods described in this annex apply to data from either a pilot-scale or full-scale interlaboratory test using the design specified in Section 6. Such a design involves *M*, materials; *L*, laboratories; *O*(*L*), operators within a laboratory; and *S* (*MLO*), specimens within a material, laboratory, and operator. If another design is used in the interlaboratory test, qualified statistical help is essential in performing the analysis.

A1.2 Use of this annex requires some experience with the analysis of variance. Information on the subject can be obtained from most standard statistical texts (**1-10**, **13**). Qualified statistical help is essential in performing the analysis. A hand held calculator capable of summing the squares of numbers is adequate although more sophisticated equipment may be used, including "canned" computer programs for the analysis of variance.

A1.3 Record the raw data in a Table like Table A1.1. Note that the table lists totals rather than averages when two or more observations are combined.

Analysis by Materials

A1.4 Prepare an analysis of variance (ANOVA) table for each material using raw data from a table like Table A1.1 and using Table A1.2, Fig. A1.1, and Fig. A1.2 as guides.

A1.5 Using the F-test as specified in standard statistical texts **(1, 3, 4, 7, 8, 10)**, test the mean squares for significant effects. Since significant effects mean that the test method procedure or the interlaboratory test procedure, or both, show a lack of statistical control, make a decision on whether to (*1*) return to further development of the test method or the interlaboratory test procedure or (*2*) continue the analysis of the data.

A1.6 Estimate the components of variance associated with each material using the equations in the right-hand column of Table A1.2 and the mean squares obtained as illustrated in Figs. A1.1 and A1.2. Calculate the components of variance even when the mean square with which it is associated is not significant. Calculate the components of variance by (*1*) starting at the bottom of an analysis of variance table, (*2*) equating each mean square with the corresponding equation, (*3*) substituting the components of variance already calculated, and (*4*) solving the equation for the remaining component of variance. Since components of variance cannot be negative, proceed as directed in A1.6.1, if the calculated value of a component of variance is negative.

A1.6.1 If the calculated value of a component of variance is negative, check all the arithmetic involved. If the value is correctly negative, set the value of that component of variance at zero and strike that component of variance from the equations for calculating components of variance from observed mean squares. This will normally yield two or more mean squares which are equated with identical equations for expected mean squares. These mean squares should be pooled as illustrated in Annex A2.

A1.7 Prepare a table listing the components of variance by materials. The data in Table A1.1 produce the following components of variance:

	Components of Variance	
	Material 1	Material 2
$V(L)$	0.0541	0.0619
$V(O.L)$	0.0075	0.0045
$V(S.LO)$	0.0053	0.0035

A1.8 Make a decision on whether the data for the individual materials seem consistent enough so that the data for all the materials can be combined into a single analysis of variance. A possible theoretical basis for such a decision is discussed in A1.8.2. From a practical standpoint, a decision can be made by (*1*) developing a table as directed in Practice D 2906 showing the critical differences that apply for each material under the conditions of single-operator precision, within-laboratory precision, and between-laboratory precision and (*2*) making an engineering decision on the *practical* importance of the observed variation in the critical differences for the individual materials under the various conditions for precision. Before making a final decision on the decision to pool estimates from more than one material, perform those auxiliary tests specified in Section 15 that seem appropriate. An example of such a decision is given in A1.8.1.

A1.8.1 Using the data tabulated in A1.7 and the methods specified in Practice D 2906, develop the following table of components of variance expressed as standard deviations, if preferred:

	Components of Variance as Standard Deviations	
	Material 1	Material 2
Single-operator precision	0.073	0.059
Within-laboratory precision	0.087	0.067
Between-laboratory precision	0.233	0.249

From the above components of variance expressed as standard deviations, use the procedures in Practice D 2906 to prepare the following critical differences for comparing two single test results at the 95 % probability level:

TABLE A1.1 Raw Data from Interlaboratory Test

		Material 1			Material 2			
Lab.	Opr.	Test 1	Test 2	Sum	Test 1	Test 2	Sum	Totals
1	1	1.02	1.23	2.25	2.35	2.45	4.80	7.05
	2	0.86	0.90	1.76	2.40	2.42	4.82	6.58
	3	1.06	1.14	2.20	2.40	2.47	4.87	7.07
	4	1.05	1.05	2.10	2.47	2.56	5.03	7.13
	Sum			8.31			19.52	27.83
2	1	1.30	1.15	2.45	2.82	2.75	5.57	8.02
	2	1.30	1.27	2.57	2.77	2.77	5.54	8.11
	3	1.34	1.28	2.62	2.70	2.65	5.35	7.97
	4	1.19	1.25	2.44	2.69	2.72	5.41	7.85
	Sum			10.08			21.87	31.95
3	1	1.16	1.01	2.17	2.60	2.62	5.22	7.39
	2	1.12	0.97	2.09	2.68	2.57	5.25	7.34
	3	1.36	1.31	2.67	2.84	2.91	5.75	8.42
	4	1.28	1.39	2.67	2.71	2.61	5.32	7.99
	Sum			9.60			21.54	31.14
4	1	1.40	1.27	2.67	2.79	2.77	5.56	8.23
	2	1.14	1.12	2.26	2.71	2.81	5.52	7.78
	3	1.27	1.15	2.42	2.74	2.84	5.58	.80.00
	4	1.21	1.06	2.27	2.62	2.76	5.38	7.65
	Sum			9.62			22.04	31.66
5	1	1.39	1.33	2.72	2.77	2.73	5.50	8.22
	2	1.43	1.38	2.81	2.82	2.79	5.61	8.42
	3	1.34	1.27	2.61	2.80	2.62	5.42	8.03
	4	1.30	1.35	2.65	2.62	2.56	5.18	7.83
	Sum			10.79			21.71	32.50
6	1	0.95	1.09	2.04	2.44	2.50	4.94	6.98
	2	1.03	1.07	2.10	2.48	2.48	4.96	7.06
	3	0.91	0.89	1.80	2.36	2.36	4.72	6.52
	4	0.84	0.84	1.68	2.55	2.49	5.04	6.72
	Sum			7.62			19.66	27.28
7	1	1.07	1.02	2.09	2.60	2.70	5.30	7.39
	2	1.23	1.16	2.39	2.76	2.79	5.55	7.94
	3	1.05	1.04	2.09	2.59	2.59	5.18	7.27
	4	1.28	1.10	2.38	2.78	2.77	5.55	7.93
	Sum			8.95			21.58	30.53
8	1	0.85	0.77	1.62	2.33	2.20	4.53	6.15
	2	0.70	0.53	1.23	2.19	2.32	4.51	5.74
	3	0.81	0.85	1.66	2.38	2.41	4.79	6.45
	4	0.70	0.70	1.40	2.37	2.20	4.57	5.97
	Sum			5.91			18.40	24.31
9	1	0.86	0.75	1.61	1.94	2.02	3.96	5.57
	2	0.61	0.67	1.28	1.91	2.00	3.91	5.19
	3	0.66	0.59	1.25	2.10	2.19	4.29	5.54
	4	0.40	0.63	1.03	1.95	2.04	3.99	5.02
	Sum			5.17			16.15	21.32
Totals				76.05			182.47	258.52

	Critical Differences	
	Material 1	Material 2
Single-operator precision	0.20	0.16
Within-laboratory precision	0.31	0.25
Between-laboratory precision	0.72	0.73

Since the above critical differences differ so little from material to material, a decision was made to analyze the data for all materials as a single analysis of variance.

A1.8.2 The theoretical basis for comparing the components of variance from the individual materials is too complex for a full explanation in this annex and involves procedures that require statistical judgment. If this approach is used, competent statistical help is essential. The components of variance of each line in a table like that in A1.7 may be compared using a technique such as Bartlett's test **(2, 13)**. If Bartlett's or a similar test is used, care must be taken to use the degrees of freedom associated with the components of variance rather than the degrees of freedom associated with the mean squares from

TABLE A1.2 ANOVA Table for One Material from Basic Design[A]

Sources of Variation	Sums of Squares	Degrees of Freedom	Components of Variance Estimated from Observed Mean Squares
L	(4) − (2)	*L* − *1*	*V(S.LO) + SV(O.L) + OSV(L)*
O(L)	(3) − (4)	*L(O* − *1)*	*V(S.LO) + SV(O.L)*
S(LO)	(1) − (3)	*LO(S* − *1*)	*V(S.LO*)
Totals	(1) − (2)	*LOS* − 1	

[A] Where the names of the sources of variation are read as:

L = number of laboratories,
O(*L*) = number of operators within laboratories, and
S(*LO*) = number of specimens within laboratories and operators,

where:

(1) = Σ (individual observations)^2= ΣX^2 (A11)
(2) = (grand total)^2/*LOS* = $(\Sigma X)^2$/*LOS* (A12)
(3) = Σ (operator totals)^2/*S* (A13)
(4) = Σ (laboratory totals)^2/*OS* (A14)

Where the letters *L*, *O*, and *S* are respectively the number of laboratories, operators within a laboratory, and specimens within laboratories and operators, and

Where the components of variance are identified as:
V(*L*) = component of variance for laboratories,
V(*O.L*) = component of variance for operators within a laboratory, and
V(*S.L.O*)= component of variance for specimens within laboratories and operators.

(1) = $\Sigma X^2 = 1.02^2 + 1.23^2 + \ldots . . + 0.40^2 + 0.63^2$ = 84.6903
(2) = $(\Sigma X)^2$/LOS = $(76.05)^2$/(9)(4)(2) = 80.3278
(3) = Σ (operator totals)^2/S
= $(2.25^2 + 1.76^2 + \ldots . . + 1.25^2 + 1.03^2)/2$ = 84.4994
(4) = Σ (laboratory totals)^2/OS
= $8.31^2 + 10.08^2 + \ldots . . + 5.91^2 + 5.17^2)/(4)(2)$ = 83.9519

Sources of Variation	Sums of Squares	Degrees of Freedom	Mean Squares
Laboratories, L	3.6241	8	0.4530
Operators in Laboratories, O(L)	0.5475	27	0.0203
Specimens in Labs. and Operators, S(LO)	0.1909	36	0.0053
Total	4.3625	71	

Estimates of Components of Variance
MS(S.LO) = V(S.LO) = 0.0053
MS(O.L) = V(S.LO) + 2V(O.L)
0.0203 = 0.0053 + 2V(O.L)
pV(O.L) = (0.0203 − 0.0053)/2 = 0.0075
MS(L) = V(S.LO) + 2V(O.L) + 8V(L)
0.4530 = (0.0203) + 8V(L)
V(L) = (0.4530 − 0.0203)/8 = 0.0541

FIG. A1.1 Analysis of Material 1 Using Table A1.2

(1) = $\Sigma X^2 = 2.35^2 + 2.45... ... + 2.04^2$
(2) = $(\Sigma X)^2$/LOS = $(182.47)^2$/(9)(4)(2) = 466.9577
(3) = (operator totals)^2/S
= $(4.80^2 + 4.82^2 + \ldots . . + 4.29^2 + 3.99^2)/2$ = 466.8327
(4) = (laboratory totals)^2/OS
= $(19.52^2 + 21.87^2 + \ldots . . + 18.40^2 + 16.15^2)(4)(2$ = 466.49741

Sources of Variation	Sums of Squares	Degrees of Freedom	Mean Squares
Laboratories, L	4.0627	8	0.5078
Operators in Laboratories, O(L)	0.3353	27	0.0124
Specimens in Labs and Operators, S(LO)	0.1250	36	0.0035
Total	4.5230	71	

Estimates of Components of Variance
MS(S.LO) = V(S.LO) = 0.0035
MS(O.L) = V(S.LO) + 2V(O.L)
0.0124 = 0.0035 + 2V(O.L)
V(O.L) = (0.0124 - 0.0035)/2 = 0.0045
MS(L) = V(S.LO) + 2V(O.L) + 8V(L)
0.5078 = (0.0124) + 8V(L)
V(L) = (0.5078 − 0.0124)/8 = 0.0619

FIG. A1.2 Analysis of Material 2 Using Table A1.2

which the components of variance were calculated. Satterthwaite's approximation **(1)** may be used to estimate the degrees of freedom associated with the components of variance.

NOTE A1.1—Since there are three lines of components of variance in a table like that in A1.7, the Bartlett's test for testing a single line should be at a probability level of $0.95^{1/3}$ = 0.9830 or 98.30 % in order to have an overall probability level of 95 %.

Analysis of All Materials Together

A1.9 If a decision is made to combine the data from all materials into a single analysis of variance, much of the work already will have been done.

A1.10 Prepare an analysis of variance (ANOVA) table using the raw data in a table like Table A1.1 and using Table A1.3 and Fig. A1.3 as guides. See Table A1.4 for the ANOVA table for the example.

NOTE A1.2—See Fig. A1.3 for an alternate method of obtaining quantities (1), (3), (5), and (6) when the data for individual materials has already been analyzed.

A1.11 Using the F-test as specified in standard statistical texts **(1, 3, 4, 7, 8, 10)** test the mean squares for significant effects. Since significant effects mean that the test method procedure or the interlaboratory test procedure, or both, show a lack of statistical control, make a decision on whether to (*1*) return to further development of the test method or the interlaboratory test procedure or (*2*) continue the analysis of the data.

NOTE A1.3—Significantly large mean squares for the interactions *ML* and *MO* (*L*) are especially important and indicate that the test method, as used by the laboratories in the interlaboratory test, evaluates materials differently when the materials are tested in different laboratories or when tested by different operators in the laboratories. Special efforts should be made to detect and to eliminate or minimize the cause of such interactions.

A1.12 Using the information in a table like Table A1.4, calculate the components of variance by (*1*) starting at the bottom of the table, (*2*) equating each mean square with the corresponding equation, (*3*) substituting the components of variance already calculated, and (*4*) solving it for the remaining component of variance. Calculate the components of variance even when the mean square with which it is associated is not significant. If the calculated value of a component of variance is correctly zero or negative, assign zero as the value of the component of variance and do the necessary pooling of mean squares (see A1.6.1 and Annex A2). For the data in Table A1.4, the calculations are:

$V(S.MLO) = 0.0044$
$V(MO.L) = (0.0099 - 0.0044)/2 = 0.00275$
$V(O.L) = (0.0228 - 0.0044 - (2 \times 0.00275))/4 = 0.00323$
$V(ML) = (0.0267 - 0.0044 - (2 \times 0.00275))/8 = 0.00211$
$V(L) = (0.9341 - 0.0044 - (2 \times 0.00275) - (4 \times 0.00323) - (8 \times 0.00211))/16 = 0.0559$

NOTE A1.4—Normally, *V(M)*, the component of variance for materials, is not of interest because the materials were deliberately chosen to illustrate differences in level of the property of interest. For this reason *V*(*M*) is not usually calculated.

A1.13 Before making a final decision on the adequacy of the estimates of the components of variance, review those auxiliary tests specified in Section 15 that seem appropriate.

TABLE A1.3 ANOVA Table for All Materials from Basic Design[A]

Sources of Variation	Sums of Squares	Degrees of Freedom	Components of Variance Estimated from Observed Mean Squares
M	(3) − (2)	*M* − 1	not normally calculated
L	(4) − (2)	*L* − 1	*V(S.MLO) + SV(MO.L) + MSV(O.L) + OSV(ML) + MOSV(L)*
ML	(5) + (2) − (3) − (4)	*(M − 1) (L − 1)*	*V(S.MLO) + SV(MO.L) + OSV(ML)*
O(L)	(7) − (4)	*L(O − 1)*	*V(S.MLO) + SV(MO.L) + MSV(O.L)*
MO(L)	(4) + (6) −(5) − (7)	*L(M − 1) (O − 1)*	*V(S.MLO) + SV(MO.L)*
S(MLO)	(1) − (6)	*MLO(S − 1)*	*V(S.MLO)*
Totals	(1) − (2)	*MLOS* − 1	

[A] Where the names of the sources of variation are read as:

M = materials
L = laboratories,
ML = material times laboratory interaction,
O(*L*) = operators within laboratories,
MO(*L*) = material times operator interaction within laboratories, and
S(*MLO*) = specimens within materials, laboratories and operators,

where the quantities for calculating the sum of squares are:

(1) = Σ (individual observations)^2= ΣX^2 (A15)
(2) = (grand total)^2/*MLOS* = $(\Sigma X)^2$/*MLOS* (A16)
(3) = Σ (matrial totals)^2/*LOS* (A17)
(4) = Σ (laboratory totals)^2/*MOS* (A18)
(5) = Σ (matrial totals in laboratories)^2/*OS* (A19)
(6) = Σ (operator totals in materials)^2/*S* (A20)
(7) = Σ operator totals in laboratories)^2/*MS* (A21)

where the letters *M*, *L*, *O* and *S* are respectively the number of materials, laboratories, operators within a laboratory, and specimens within materials, laboratories, and operators, and

where the components of variance are identified as;
V(*L*) = component of variance for laboratories,
V(*ML*) = component of variance for material times laboratory interaction,
V(*O.L*) = component of variance for operators within laboratories, and
V(*MO.L*) = component of variance for material times operator interaction within laboratories, and
V(*S.MLO*) = component of variance for specimens within materials, laboratories, and operators.

(1) = $\Sigma X^2 = 1.02^2 + 1.23^2 + \ldots . . + 1.95^2 + 2.04^2 = 551.6480$
(2) = $(\Sigma X)^2/MLOS = (258.52)^2/(2)(9)(4)(2) = 464.1152$
(3) = Σ (material totals)^2/LOS
= $(76.05^2 + 182.47^2)/(9)(4)(2) = 542.7625$
(4) = Σ (laboratory totals)^2/MOS
= $(27.83^2 + 31.95^2 + \ldots . . + 24.31^2 + 21.32^2)/(2)(4)(2) = 471.5884$
(5) = Σ (material totals in labs)^2/OS
= $(8.31^2 + 19.52^2 + \ldots . . + 5.17^2 + 16.15)^2/(4)(2) = 550.4493$
(6) = Σ (operator totals in materials)^2/S
= $(2.25^2 + 1.76^2 + \ldots . . + 4.29^2 + 3.99^2)/2 = 551.3320$
(7) = Σ (operator totals in labs)^2/MS
= $7.05^2 + 6.58^2 + \ldots . . + 5.54^2 + 5.02^2)/(2)(2) = 472.2030$

Sources of Variation	Sums of Squares		Degrees of Freedom	Mean Squares
M	(3) − (2)	= 78.6473	1	78.6473
L	(4) − (2)	= 7.4732	8	0.9342
ML	(5) + (2) − (3) − (4)	= 0.2136	8	0.0267
O(L)	(7) − (4)	= 0.6146	27	0.0228
MO(L)	(4) + (6) − (5) − (7)	= 0.2681	27	0.0099
S(MLO)	(1) − (6) =	= 0.3160	72	0.0044
Totals	(1) − (2)	= 87.5328	143	

NOTE 1—If the data for individual materials were analyzed using Figs. A1.1 and A1.2, the more time consuming tasks have already been done and can be combined as follows:

(1) = sum of quantity (1) for each material = 84.6903 + 466.9577 = 551.6480
(3) = sum of quantity (2) for each material = 80.3278 + 462.4347 = 542.7625
(5) = sum of quantity (4) for each material = 83.9519 + 466.4974 = 550.4493
(6) = sum of quantity (3) for each material = 84.4994 + 466.8327 = 551.3321

Values obtained in this way will reflect earlier roundings.

FIG. A1.3 Analysis of Full Experiment Using Table A1.3

A1.14 For reporting purposes, relabel the components of variance by the generic terms of "single-operator component," "within-laboratory-component," and "between-laboratory component." If it is preferred, express them as the square roots of the components of variance in order to state them in the appropriate units of measure, rather than as the squares of those units of measure. When appropriate, the square roots of the components of variance may be converted to the corresponding coefficient of variation.

A1.14.1 *No Significant Interactions*—If neither of the mean squares associated with the interactions *ML* or *MO* (*L*) is significant, disregard the components of variance *V* (*ML*) and *V* (*MO.L*) and calculate the components of variance to be reported as standard deviations using Eq A1.1-A1.3:

$$s_s = V(S.MLO)^{1/2} \quad \text{(A1.1)}$$

$$s_w = V(O.L)^{1/2} \quad \text{(A1.2)}$$

$$s_b = V(L)^{1/2} \quad \text{(A1.3)}$$

where:
s_s = single-operator component of variance,
s_w = within-laboratory component of variance, and
s_b = between-laboratory component of variance.

A1.14.2 *Significant Interaction(s)*—If either or both of the mean squares associated with the interactions *ML* or *MO* (*L*) is significant, different components of variance apply to the situations where (*1*) specimens of the same material are being compared and (*2*) specimens of different materials are being compared.

A1.14.2.1 *Single-Material Comparisons*—Use the components of variance calculated as directed in A1.14.1, but label them with the notation "(single-material)"; for example, as "s_s (single-material)."

A1.14.2.2 *Multi-Material Comparisons*—Calculate the components of variance to be reported as standard deviations for multi-material comparisons using Eq A1.4-A1.6:

$$s_s\,(\text{multi}-\text{material}) = s_s\,(\text{single}-\text{material}) + V(MO.L)^{1/2} \quad \text{(A1.4)}$$

$$s_w\,(\text{multi}-\text{material}) = V(O.L)^{1/2} \quad \text{(A1.5)}$$

$$s_b\,(\text{multi}-\text{material}) = [(V(ML) + V(L)]^{1/2} \quad \text{(A1.6)}$$

where the symbols for the components of variance are defined in A1.14.1 and where only s_s (single-material) in Eq A1.4 is affected by the number of specimens when used in equations like those in Practice D 2906.

A1.15 Using the procedures in A1.14-A1.14.2.2, the components of variance in A1.12 can be expressed as standard deviations, as follows (Note A1.5):

Single-Material Comparisons:	
Single-operator component	0.0663 units
Within-laboratory component	0.0568 units
Between-laboratory component	0.236 units
Multi-Material Comparisons:	
Single-operator component	0.0663 + 0.0524 units
Within-laboratory component	0.0568 units
Between-laboratory component	0.241 units

NOTE A1.5—If it is preferred, report the square roots of the components of variance in order to express the variability in the appropriate units of measure rather than as the squares of those units of measure.

A1.16 Using the components of variance listed in A1.12 (as variances), or A1.15 (as standard deviations,) and the procedures in Practice D 2906, the following critical differences are obtained:

Number of Observations in Average	Critical Differences Between Two Averages, Units of Measure[A]		
	Single-Operator Precision	Within-Laboratory Precision	Between-Laboratory Precision
	Single-Material Comparisons		
1	0.18	0.24	0.70
2	0.13	0.20	0.69
4	0.09	0.18	0.68
8	0.06	0.17	0.68
	Multi-Material Comparisons		
1	0.23	0.28	0.73
2	0.19	0.25	0.71
4	0.17	0.23	0.71
8	0.16	0.22	0.70

[A] The confidence limits were calculated using $z = 1.960$.

TABLE A1.4 Equations for Estimating Components of Variance[A]

Line No.	Source of Variation	Degrees of Freedom	Mean Squares	Components of Variance Estimated from Observed Mean Squares
1	*M*	1	78.6473	not normally calculated
2	*L*	8	0.9342	*V(S·MLO) + 2V(MO·L) + 4V(O·L) + 8V(ML) + 16V(L)*
3	*ML*	8	0.0267	*V(S·MLO) + 2V(MO·L) + 8V(ML)*
4	*O(L)*	27	0.0228	*V(S·MLO) + 2V(MO·L) + 4V(O·L)*
5	*MO(L)*	27	0.0099	*V(S·MLO) + 2V(MO·L)*
6	*S(MLO)*	72	0.0044	*V(S·MLO)*

[A] The sources of variance are:

M = materials
L = laboratories,
ML = material times laboratory interaction,
O(L) = operators within laboratories,
MO(L) = material times operator interaction within laboratories, and
S(MLO) = specimens within materials, laboratories and operators,

The letters *M*, *L*, *O*, and S in the equations for degrees of freedom and as coefficients in the equations for components of variance are respectively the number of materials, laboratories, operators within laboratories, and specimens within materials, laboratories, and operators.

The components of variance are:

V(L) = component of variance for laboratories,
V(ML) = component of variance for material times laboratory interaction,
V(O·L) = component of variance for operators within laboratories, and
V(MO·L) = component of variance for material times operator interaction within laboratories, and
V(S·MLO) = component of variance for specimens within materials, laboratories, and operators.

A2. EXAMPLE OF POOLING MEAN SQUARES

A2.1 In the following example, the table of analysis of variance (ANOVA) is based on synthetic data. In this table, the mean squares in the higher lines are small enough to require pooling. The column headings are abbreviations of "Sources of Variation," "Sums of Squares," "Degrees of Freedom," "Mean Squares," and "Components of Variance Estimated from Observed Mean Squares."

Sources	SS	DF	MS	Expected Mean Squares
L	0.360	8	0.045	*V(S.LO) + 2V(O.L) + 8V(L)*
O(L)	1.080	27	0.040	*V(S.LO) + 2V(O.L)*
S(LO)	2.160	36	0.060	*V(S.LO)*
Totals	3.600	71		

where:

L = laboratories,
O(L) = operators within laboratories,
S(LO) = specimens within laboratories and operators,
V(S.LO) = the component of variance for *S* (*LO*),
V(O.L) = the component of variance for *O* (*L*), and
V(L) = the component of variance for *L*.

A2.2 Since the mean square for *O*(*L*) is smaller than the mean square for *S*(*LO*), the calculated value of *V*(*O.L*) = −0.010 and the value is set at zero. Dropping *V* (*O.L*) from the equations for *L* and *O* (*L*) gives the following table in which the expected mean squares for both *O* (*L*) and *S* (*LO*) estimate *V*(*S.LO*):

Sources	SS	DF	MS	Expected Mean Squares
L	0.360	8	0.045	*V(S.LO) + 8V(L)*
O(L)	1.080	27	0.040	*V(S.LO)*
S(LO)	2.160	36	0.060	*V(S.LO)*
Totals	3.600	71		

A2.3 The best estimate of the mean square for *S* (*LO*) is obtained by combining or pooling the mean squares for *O*(*L*) and *S*(*LO*). This is done by dividing the total for the appropriate sums of squares by the total for the appropriate degrees of freedom, giving (1.080 + 2.160)/(27 + 36) = 3.240/63 = 0.0514 or 0.051. In turn *V*(*S.LO*) = 0.051. This results in the following new table of the analysis of variance:

Sources	SS	DF	MS	Expected Mean Squares
L	0.360	8	0.045	*V(S.LO) + 8V(L)*
S(LO)	3.240	63	0.051	*V(S.LO)*
Totals	3.600	71		

A2.4 Substituting *V*(*S.LO*) = 0.051 into the revised equation for the expected mean square for *L* gives *V* (*L*) = −0.0008. Setting *V*(*L*) = 0 and dropping it from the equation for the expected mean square for *L* gives two equations for estimating *V*(*S.LO*) as shown below:

Sources	SS	DF	MS	Expected Mean Squares
L	0.360	8	0.045	*V(S.LO)*
S(LO)	3.240	63	0.060	*V(S.LO)*
Totals	3.600	71		

A2.5 Under these conditions, the best estimate of *V* (*S.LO*) is obtained from the totals for both sums of squares and degrees of freedom, giving *V*(*S.LO*) = 3.600/71 = 0.0507 or 0.051. Thus the final estimates are:

$$V(S.LO) = 0.051 \quad \text{(A2.1)}$$
$$V(O.L) = 0$$
$$V(L) = 0$$

A2.6 When pooling the mean squares for a table such as that in A2.1, anywhere from none to two of the estimates of components of variance may be set zero. When pooling the mean squares for a table of analysis of variance such as Table A1.4, anywhere from zero to four of the components of variance may be set at zero.

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