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Designation: C 1067 – 87 (Reapproved 1993)



Designation: C 1067 – 00

Standard Practice for Conducting A Ruggedness or Screening Program for Test Methods for Construction Materials¹

This standard is issued under the fixed designation C 1067; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

¹ This practice is under the jurisdiction of ASTM Committee C-9 C09 on Concrete and Concrete Aggregates. This practice was developed jointly by ASTM Committees C-1, C-9, D-4, and D-18, and is endorsed by all four committees.

Current edition approved June 15, 1987; 10, 2000. Published August 2000. Originally published as C 19067 – 87. Last previous edition C 1067 – 87 (1993).

1. Scope

1.1 This practice covers a procedure for detecting ~~and reducing~~ sources of variation in a test method. ~~The procedure should be used during the development of a test method, before the interlaboratory study is executed, such as those in Practices C 670, C 802, and E 691. Interlaboratory studies can be expensive to execute. Resources will probably be more efficiently used if sources of variation in a test method are eliminated prior to performing the interlaboratory study. One of the main purposes of the procedure, by means of a ruggedness or screening program, The procedure also is the elimination useful for determining sources of variation in an existing test methods that exhibit poor precision even after vigorous efforts has been found to reduce their variability. have poor precision.~~

1.2 This practice covers, in very general terms, techniques for planning, ~~conducting~~, collecting data, and analyzing results from a few laboratories to establish whether a well written test method is good enough to justify an interlaboratory study. laboratories. Annex A1 provides the details of the procedure with an example and Annex A2 gives the theoretical background.

1.3 This practice is intended ~~does not give information pertinent to be used before spending the time and money required to conduct a full scale interlaboratory study, such as those in Practices C 670, C 802, estimating within- or E 691. between-laboratory precision.~~

1.4 ~~This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems concerns, if any, associated with its use. It is the responsibility of whoever uses the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.~~

2. Referenced Documents

2.1 ASTM Standards:

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials²

C 802 Practice for Conducting ~~An~~ an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials²

E 456 Terminology Relating to Quality and Statistics³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

E 1169 Guide for Conducting Ruggedness Tests³

3. Terminology

3.1 Definitions:

3.1.1 *determination value, n*—numerical quantity calculated as directed in the test method using direct measurements obtained in accordance with the procedures given in the test method.

3.1.2 *observation, n*—a direct measurement on a specimen that produces an attribute datum or a variate used to obtain the value of a determination.

3.1.3 *replication, n*—the act of obtaining two or more determination values under specified conditions. The number of replications must be finite and the scope of the replication operation may be narrow or broad, but must be specified.

3.1.4₃ For definitions of other statistical terms used in this standard, refer to Terminology E 456.

² Annual Book of ASTM Standards, Vol 04.02.

³ Annual Book of ASTM Standards, Vol 14.02.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *factor, n*—an element in the test procedure or laboratory environment that is a potential source of variation in test results.

3.2.2 *ruggedness, adj*—the characteristic of a test method that produces test results that are not influenced by small differences in the testing procedure or environment.

3.2.3 *screening, n*—the detection of significant sources of variation as compared to chance variation.

3.2.4 *variable, n*—a number or quantity that varies.

4. Summary of Practice

4.1 This practice has as its purpose requires that the detection and control user develop, from theoretical or practical knowledge, or both, a list of sources of testing factors that plausibly would cause significant variation in test results if the factors were not controlled. The technique is limited to programming an interlaboratory study.

4.2 One of the most productive uses analysis of a ruggedness or screening evaluation is the effects seven factors and requires considerably less effort than would be required to collected data for seven factors in a full factorial study. Procedures exist for analysis of smaller and larger numbers of factors (see Guide E 1169), but seven is a convenient number for many test methods for construction materials. The seven-factor analysis requires 16 determinations by each laboratory. The procedure can be usefully executed by a single laboratory, but sometimes additional information can be obtained if it is repeated in one or two additional laboratories.

4.2 The procedure requires that two levels of each factor be identified, then 16 determinations be done on a prescribed combinations of factor levels. The levels assigned to have poor precision even after making vigorous efforts to reduce a factor may be quantitative or qualitative (for example, brass versus steel).

4.3 The disadvantage of this type of analysis is that the variation. Such test methods can create uncertainty method only estimates simple effects of each factor and confusion and may does not be suitable to become ASTM standards. detect interactive effects among factors.

5. Significance and Use

5.1 Prior to starting an interlaboratory study that involves at least ten laboratories and represents much expense and work, it is necessary to determine if

5.1 The purpose of a test method ruggedness evaluation is ready for such an interlaboratory study. The recommended method for to determining how sensitive the suitability of a test method for an interlaboratory study is the screening or ruggedness test in which a few laboratories introduce known variations in pertinent variables related to testing techniques and environment changes in order to judge the magnitude levels of their effect on the test results. This information is used to determine the controls necessary pertinent operating factors. Normally, operating conditions for these variables in the test method. Examples of such variables are temperature, force, load, time of loading, rate of loading, rate of deformation, humidity, acceleration of moving parts, time of stirring, and concentration of solution.

5.1.1 Before any screening is possible, a valid, well-written test method must exist. The originating laboratory, as part are defined along with an allowable tolerance. A ruggedness analysis determines that effect of its effort worst-case variation in developing the test operating conditions within this tolerance range. The method shall determine the pertinent variables. It shall then can be part of the screening process revised with smaller tolerances on operating conditions to review, study, and possibly identify additional pertinent variables. A improve the precision.

5.2 A major reason for poor precision in test methods is the lack of adequate control over the sources of variation in testing procedures or in the testing environment. In turn, these environments. These sources of variation often are not adequately controlled adequately because they were not identified during the development of the test procedures. I

5.3 All new test methods must be subjected to an interlaboratory program for purposes of developing a precision and bias statement. These programs can be expensive and lengthy, and the result may be that the determination is essential made that the method is too variable to be published without further revision. Interlaboratory studies typically give the subcommittee an indication that the method is too variable, but they do not usually give a clear picture of what is causing the variation. Application of this ruggedness practice using one or a few laboratories may be a much more economical way to determine these causes.

5.4 Many existing test methods were published before there was a requirement that precision and bias statements be developed. Since this became a requirement, most of these test methods have developed precision and bias statements, and the result is that many have been found to suffer from relatively large amount of variation. Use of this practice represents a relatively simple way to reduce investigate the variability causes of variation in test results methods, so that a subcommittee will have some guidance as to which parts of the test method need to be studied further for revision.

5.5 The procedure can be used for a program within a single laboratory, but involvement of at least three laboratories is recommended, particularly if the single laboratory were to be the one in which the test method was developed. This is particularly important for new test methods. The originating laboratory is so much a part of pertinent variables, because such methods the development of the test method that give highly variable results create uncertainty it is difficult for it to be objective in spotting any problems in the clarity of the test method directions. Two additional laboratories will probably contribute fresh critical review of the validity of the test method and confusion, and consequently should not become standards. provide assistance in clarifying the instructions of the test method when needed.

6. Materials

6.1 The number and types of material shall cover the range of material properties to which the test method is applicable. The test method does not apply to material types or property values outside the range evaluated. Three to five materials will usually be sufficient.

6.1.1 Some preliminary testing may help the laboratories involved determine the materials that shall be used in the screening program.

7. Procedure

~~7.1 Three laboratories are considered~~

~~7.1 Determine the ideal number for a screening study.~~

~~7.2 The originating laboratory is so much a part of the development of the test method laboratories that it is difficult for it to be objective will participate in spotting any problems in the clarity of the test method directions. The two other laboratories are to contribute fresh critical review of the validity of the test method program and to provide assistance which materials each will use in clarifying the instruction of the test method when needed: program. The review and study maximum amount of the test method will help the two additional information is obtained if all laboratories to familiarize themselves with the test method before starting the screening process. During this review and study, the participating laboratories shall vigilantly search for additional large or pertinent sources include all materials in their part of variation so they the program, however cost can be included in the evaluation program.~~

~~7.3 The purpose of reduced by each laboratory using a ruggedness evaluation is to determine how sensitive the test method is to level changes different material. Caution must be exercised in interpreting the results since laboratory-dependent cannot be separated from material-dependent effects.~~

~~7.2 Factors that are within likely to have the limits specified greatest effect on the variability in the test method. This information is needed to ensure that final written instructions results are selected for study. Levels of these factors are determined, selecting the test method specify the proper range of such factors. It is obtained by determining the relationship between a change minimum and maximum levels that would plausibly occur in the level execution of a pertinent factor and the effect on test results.~~

~~7.4 A ruggedness procedure, credited method if there were no particular efforts to Youden (1)⁴, has been chosen control them. Only two levels are allowed. Levels often represent quantitative properties, such as temperature, pressure, etc, but they may also represent nonquantitative values-f, such as old vs new, wet vs dry, etc. In this practice. The major departure from Youden's recommendations is standard, factors are assigned letter designations, A – G, and the use two levels of three laboratories rather than one. The use each factor are designated with upper and lower cases of three laboratories is required because experience has shown the importance these letters, as in Table 1.~~

~~7.3 Assign combinations of the fresh insight that additional laboratories can bring factor levels to experimental determinations according to Table 1. The 8 determinations will be done in duplicate, therefore, the clarification of instructions and procedures for full study on each material will require 16 determinations.~~

~~7.4 Construct a 16 row by 16 column results matrix from the test method.~~

~~7.5 The analysis and evaluation of the significance of the variables is carried out using multiple orthogonal comparisons or contrasts 16 determinations values (d_1 (2, 3, 4) d_{16}) as shown in Table 2. The theory absolute values of the procedure is discussed determinations in Annex A2.~~

~~7.6 The factors that each row are likely to have identical, only the greatest effect on the variability signs vary. Calculate Z and W statistics as shown in the test results are selected equations below.~~

$$Z_r = \sum_1^{16} d_i, \text{ where } d_i \text{ s are the 16 results in each row (r).} \quad (1)$$

$$W_r = \frac{Z_r^2}{16} \quad (2)$$

~~7.5 The W statistic for study. They row 1 represents the simple sum of the determinations and are tested at two levels not used in a fractional factorial that evaluates this analysis. Statistics for rows 2–8 ($W_2 - W_8$) represent the effects of each factor, the seven factors. The fractional factorial experiment statistic for row 9 (W_9) represent the total variation between the two replicate sets and~~

TABLE 1 Pattern of Assigning Levels to Seven Factors

Factor	Determination Number							
	1	2	3	4	5	6	7	8
A	a	a	a	a	A	A	A	A
B	b	b	B	B	b	b	B	B
C	C	c	C	c	C	c	C	c
D	D	D	d	d	D	d	D	d
E	e	E	e	E	e	E	e	E
F	F	f	F	f	F	f	F	f
G	G	g	G	g	G	g	G	g

TABLE 2 Results Matrix of 16 Determinations ($d_1 - d_{16}$)

row	Eight Determinations for Replicate Set 1								Eight Determinations for Replicate Set 2									
	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8	Z	W
1	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	d_9	d_{10}	d_{11}	d_{12}	d_{13}	d_{14}	d_{15}	d_{16}	Z_1	W_1
2	d_1	d_2	d_3	d_4	$-d_5$	$-d_6$	$-d_7$	$-d_8$	d_9	d_{10}	d_{11}	d_{12}	$-d_{13}$	$-d_{14}$	$-d_{15}$	$-d_{16}$	Z_2	W_2
3	d_1	d_2	$-d_3$	$-d_4$	d_5	d_6	$-d_7$	$-d_8$	d_9	d_{10}	$-d_{11}$	$-d_{12}$	d_{13}	d_{14}	$-d_{15}$	$-d_{16}$	Z_3	W_3
4	d_1	$-d_2$	d_3	$-d_4$	d_5	$-d_6$	d_7	$-d_8$	d_9	$-d_{10}$	d_{11}	$-d_{12}$	d_{13}	$-d_{14}$	d_{15}	$-d_{16}$	Z_4	W_4
5	d_1	d_2	$-d_3$	$-d_4$	$-d_5$	$-d_6$	d_7	d_8	d_9	d_{10}	$-d_{11}$	$-d_{12}$	$-d_{13}$	$-d_{14}$	d_{15}	d_{16}	Z_5	W_5
6	d_1	$-d_2$	d_3	$-d_4$	$-d_5$	d_6	$-d_7$	d_8	d_9	$-d_{10}$	d_{11}	$-d_{12}$	$-d_{13}$	d_{14}	$-d_{15}$	d_{16}	Z_6	W_6
7	d_1	$-d_2$	$-d_3$	d_4	d_5	$-d_6$	$-d_7$	d_8	d_9	$-d_{10}$	$-d_{11}$	d_{12}	d_{13}	$-d_{14}$	$-d_{15}$	d_{16}	Z_7	W_7
8	d_1	$-d_2$	$-d_3$	d_4	$-d_5$	d_6	$-d_7$	$-d_8$	d_9	$-d_{10}$	$-d_{11}$	d_{12}	$-d_{13}$	d_{14}	d_{15}	$-d_{16}$	Z_8	W_8
9	d_1	d_2	d_3	d_4	d_5	d_6	d_7	d_8	$-d_9$	$-d_{10}$	$-d_{11}$	$-d_{12}$	$-d_{13}$	$-d_{14}$	$-d_{15}$	$-d_{16}$	Z_9	W_9
10	d_1	d_2	d_3	d_4	$-d_5$	$-d_6$	$-d_7$	$-d_8$	$-d_9$	$-d_{10}$	$-d_{11}$	$-d_{12}$	d_{13}	d_{14}	d_{15}	d_{16}	Z_{10}	W_{10}
11	d_1	d_2	$-d_3$	$-d_4$	d_5	d_6	$-d_7$	$-d_8$	$-d_9$	$-d_{10}$	d_{11}	d_{12}	$-d_{13}$	$-d_{14}$	d_{15}	d_{16}	Z_{11}	W_{11}
12	d_1	$-d_2$	d_3	$-d_4$	d_5	$-d_6$	$-d_7$	$-d_8$	$-d_9$	d_{10}	$-d_{11}$	d_{12}	$-d_{13}$	$-d_{14}$	$-d_{15}$	d_{16}	Z_{12}	W_{12}
13	d_1	d_2	$-d_3$	$-d_4$	$-d_5$	$-d_6$	d_7	d_8	$-d_9$	$-d_{10}$	d_{11}	d_{12}	d_{13}	d_{14}	$-d_{15}$	$-d_{16}$	Z_{13}	W_{13}
14	d_1	$-d_2$	d_3	$-d_4$	$-d_5$	d_6	$-d_7$	d_8	$-d_9$	d_{10}	$-d_{11}$	d_{12}	d_{13}	$-d_{14}$	d_{15}	$-d_{16}$	Z_{14}	W_{14}
15	d_1	$-d_2$	$-d_3$	d_4	d_5	$-d_6$	$-d_7$	d_8	$-d_9$	d_{10}	d_{11}	$-d_{12}$	$-d_{13}$	d_{14}	d_{15}	$-d_{16}$	Z_{15}	W_{15}
16	d_1	$-d_2$	$-d_3$	d_4	$-d_5$	d_6	d_7	$-d_8$	$-d_9$	d_{10}	d_{11}	$-d_{12}$	d_{13}	$-d_{14}$	$-d_{15}$	d_{16}	Z_{16}	W_{16}

is replicated not used in order, then an estimate of the component of variance due this analysis. Statistics for rows 10 through 16 ($W_{10} - W_{16}$) are used to calculate the error can be obtained. The component of variance due to error can be (X), which then is used to test calculate the significance of each of the factors in a ratio test using the criterion (F distribution).

7.7 Factors) for each factor, as shown to have significant effects shall be examined to see if it is practical to minimize their effect by controlling them more closely. Alternatively, such factors might be reduced or eliminated by changes the equations below. Calculations are summarized in Table 3.

TABLE 3 Summary of Statistics for Seven Factors and Random Error

Factor	W	F
A	W_2	W_2^2/X
B	W_3	W_3^2/X
C	W_4	W_4^2/X
E	W_5	W_5^2/X
F	W_6	W_6^2/X
G	W_7	W_7^2/X
H	W_8	W_8^2/X
	W_{10}	
	W_{11}	
	W_{12}	
	W_{13}	
	W_{14}	
	W_{15}	
	W_{16}	

$X = \Sigma(W^2)/7, \text{ for } W_{10-16}$

$$X = \left(\sum_{r=10}^{16} W_r^2 \right) / 7 \tag{3}$$

$F_f = \frac{W_f^2}{X}$, where F_f is the test procedure.

7.8 The details of F statistic for the pr effect of factor f (1-7, represented by $W_2 - W_8$, respectively)

7.6 A F value of ≥ 5.59 represents a significant effect for conducting factor f at a probability of 5 % for drawing an erroneous conclusion.

7.7 An example of an analysis of data representing t results on 4 marte grivals from 3 laboratories is shown in Annex A1 with an example: A1.

8. Keywords

8.1 precision; ruggedness; test method; variation

ANNEXES
(Mandatory Information)
A1. EXAMPLE OF A RUGGEDNESS PROGRAM

A1.1 This annex describes the procedure for conducting a ruggedness evaluation using as an example a description of the ruggedness evaluation on a test method for the measurement of the viscosity of asphalt.

A1.2 As the first step in the ruggedness evaluation, each of the laboratories critically examined the procedure in the proposed test method. The objectives of the examination were as follows:

A1.2.1 To determine if the instructions are clear, concise, and complete,

A1.2.2 To decide which factors are likely to influence test results and therefore should be included in the study,

A1.2.3 To pick materials that cover the range of the property of interest for the range of physical forms of the materials to be tested, and

A1.2.4 To determine the proper levels to be evaluated for each of the chosen variables.

A1.3 In this example, representatives of the three laboratories, after familiarizing themselves with the test method as specified in A1.2, met and tried to improve the instructions for the viscosity method. They selected variables, materials, and levels that showed the effect of the variation. One of the laboratories measured viscosity at 24°C, 25°C, and 26°C and found that there was about a 10 % variation with a change of 1°C. This was considered too large so 24.6 and 25.4°C were selected as the lower and upper temperature levels for the ruggedness test. In the same manner, the effect of the other variables were evaluated and the two levels to be evaluated were determined.

NOTE A1.1—Seven variables were selected and placed in a systematic procedure called an incomplete Latin Square or a Youden Square (1). The variables are listed below and shown in a Youden Square in Table A1.1. This plan can evaluate the seven variables with eight determinations. Table A1.2 shows the variables and the levels selected for this example.

A1.4 Four materials were selected to cover the range of the test method and the viscosities were determined by each of the three laboratories with one replication. The results are displayed in Table A1.3. This plan required 16 determinations by each laboratory on each material or 64 determinations by each laboratory.

A1.5 Table A1.4 specifies the experimental plan for a Youden Square for seven factors. The theory of its use is covered in Annex A2. Table A1.4 consists of 16 rows and 16 columns of coefficients each equal to ± 1 and arranged in a definite pattern.

A1.6 To obtain Table A1.5, first copy one row from Table A1.3 16 times in the general format of Table A1.5 and then multiply each entry in the new table by the corresponding entry in Table A1.4. Table A1.5 is just such a table derived from the data for Material 1 and Laboratory 1 in Table A1.3.

TABLE A1.1 Pattern for Assigning Levels to Seven Factors

Variable	Determination Number							
	1	2	3	4	5	6	7	8
A or a	a	a	a	a	A	A	A	A
B or b	b	b	B	B	b	b	B	B
C or c	C	c	C	c	C	c	C	c
D or d	D	D	d	d	d	d	D	D
E or e	e	E	e	E	E	e	E	e
F or f	F	f	f	F	F	f	F	f
G or g	G	g	g	G	g	G	G	g

a = 24.6°, the lower level of temperature.

A = 25.4°, the higher level of temperature.

b = New viscometer tube.

B = Worn viscometer tube.

C = 290-mm Hg, lower vacuum.

c = 310-mm Hg, higher vacuum.

d = Charge viscometer without stirring sample.

D = Charge viscometer after stirring for 1 min.

e = Mount the viscometer vertically.

E = Mount the viscometer 3° from vertical.

f = Charge with meniscus 1 mm above line.

F = Charge with meniscus 1 mm below line.

g = Hold viscometer in bath 10 min less than normal before testing.

G = Hold viscometer in bath 10 min longer than normal before testing.

TABLE A1.2 Conditions for Each Determination in, Experiment with Seven Factors, Asphalt Viscosity

Variable	Determination Number							
	1	2	3	4	5	6	7	8
Temperature °C	24.6	24.6	24.6	24.6	25.4	25.4	25.4	25.4
Age of Tube	New	New	Old	Old	New	New	Old	Old
Vacuum, mm Hg	310	290	310	290	310	290	310	290
Stirring	Yes	Yes	No	No	No	No	Yes	Yes
Angle with Horizontal Degree	90	87	90	87	87	90	87	90
Fill line, mm	4	6	6	4	4	6	6	4
Time in Bath, min	40	20	20	40	20	40	40	20

TABLE A1.3 Raw Data for Viscosity Example Seven Factors With Replication

Material	Viscosity															
	First Replicate Determination Number								Second Replicate Determination Number							
	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
Laboratory 1																
1	2370	2258	2355	2185	1825	1845	1820	1830	2320	2275	2350	2380	1840	1850	1825	1820
2	520	495	519	480	401	404	398	402	492	516	490	522	390	408	402	395
3	4205	4006	4191	3846	3212	3284	3185	3221	4200	4160	4130	4020	3218	3180	3280	3280
4	1075	1061	1060	961	803	793	801	805	1050	1070	1015	1000	808	790	795	805
Laboratory 2																
1	2350	2240	2335	2165	1805	1825	1800	1810	2280	2310	2400	2120	1825	1806	1809	1812
2	540	515	539	500	421	424	418	422	518	545	524	492	410	425	430	420
3	4235	4036	4121	3876	3242	3314	3117	3250	4250	4142	3960	4205	3310	3112	3240	3117
4	1102	1040	1085	980	820	811	824	828	1110	1125	1040	1050	825	804	816	835
Laboratory 3																
1	2390	2278	2375	2205	1845	1865	1840	1850	2400	2268	2350	2250	1860	1850	1870	1845
2	510	485	509	470	391	394	388	392	505	482	510	480	395	390	385	392
3	4200	3975	4160	3816	3190	3246	3150	3200	4180	3990	4140	3890	3200	3180	3220	3195
4	1050	990	1035	930	786	766	775	780	1040	980	1050	970	780	760	785	782

TABLE A1.4 Pattern for Assigning Levels to Seven Factors with Replication

	Replicate 1								Replicate 2							
	1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}
Z_1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Z_2	1	1	1	1	1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1
Z_3	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1
Z_4	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	-1
Z_5	1	1	-1	-1	-1	-1	1	1	1	1	-1	-1	-1	-1	1	1
Z_6	1	-1	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1
Z_7	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1	1	-1	-1	1
Z_8	1	-1	-1	1	-1	1	1	-1	1	-1	-1	1	-1	1	1	-1
Z_9	1	1	1	1	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1
Z_{10}	1	1	1	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1
Z_{11}	1	1	-1	-1	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1
Z_{12}	1	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1
Z_{13}	1	1	-1	-1	-1	-1	1	1	-1	-1	1	1	1	1	-1	-1
Z_{14}	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1	-1	1	-1
Z_{15}	1	-1	-1	1	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1
Z_{16}	1	-1	-1	1	-1	1	1	-1	-1	1	1	-1	1	-1	-1	1

A1.7 To obtain Table A1.6, add, with due regard to sign, each row of Table A1.5 to obtain the first column of Table A1.6 containing Z_1 – Z_{16} . Next, square each entry in column one of Table A1.6 to obtain the corresponding entry in column two and then divide each entry in column two of Table A1.6 by 16 to obtain the corresponding entry in column three. The first row, Z_1 , represents the sum of all viscosities for the first row in Table A1.5 and will not be used in this analysis. The second row, Z_2 , is the algebraic addition of the second row in Table A1.5 and measures the effect of temperature. In the same manner, the third row, Z_3 , measures the effect of the age of the viscometer. The fourth row, Z_4 , measures the effect of vacuum level. The fifth row, Z_5 , measures the effect of stirring. The sixth row, Z_6 , measures the effect of the viscometer being slanted. The seventh row, Z_7 , measures the effect of variation in meniscus level. The eighth row, Z_8 measures the effect of variation in time in the bath of the viscometer prior to testing. The ninth row, Z_9 , measures the variation between the first and second replication. Rows 10 through 16 (Z_{10} – Z_{16}) measure

TABLE A1.5 Matrix Based on Table A1.4 for Laboratory I, Material 1

NOTE 1—The data contained in Tables A1.7-A1.17 is derived from matrices constructed as illustrated by this table for each of the remaining eleven laboratory-material combinations from Table A1.3.

		Replicate 1								Replicate 2					
X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	X_{16}
2370	2258	2355	2185	1825	1845	1820	1830	2320	2275	2350	2380	1840	1850	1825	1820
2370	2258	2355	2185	-1825	-1845	-1820	-1830	2320	2275	2350	2380	-1840	-1850	-1825	-1820
2370	2258	-2355	-2185	1825	1845	-1820	-1830	2320	2275	-2350	-2380	1840	1850	-1825	-1820
2370	-2258	2355	-2185	1825	-1845	1820	-1830	2320	-2275	2350	-2380	1840	-1850	1825	-1820
2370	2258	-2355	-2185	-1825	-1845	1820	1830	2320	2275	-2350	-2380	-1840	-1850	1825	1820
2370	-2258	2355	-2185	-1825	1845	-1820	1830	2320	-2275	2350	-2380	-1840	1850	-1825	1820
2370	-2258	-2355	2185	1825	-1845	-1820	1830	2320	-2275	-2350	2380	1840	-1850	-1825	1820
2370	-2258	-2355	2185	-1825	1845	1820	-1830	2320	-2275	-2350	2380	-1840	1850	1825	-1820
2370	2258	2355	2185	1825	1845	1820	1830	-2320	-2275	-2350	-2380	-1840	-1850	-1825	-1820
2370	2258	2355	2185	-1825	-1845	-1820	-1830	-2320	-2275	-2350	-2380	1840	1850	1825	1820
2370	2258	-2355	-2185	1825	1845	-1820	-1830	-2320	-2275	2350	2380	-1840	-1850	1825	1820
2370	-2258	2355	-2185	1825	-1845	1820	-1830	-2320	2275	-2350	2380	-1840	1850	-1825	1820
2370	-2258	-2355	-2185	-1825	-1845	1820	1830	-2320	-2275	2350	2380	1840	1850	-1825	-1820
2370	-2258	2355	-2185	-1825	1845	-1820	1830	-2320	2275	-2350	2380	1840	-1850	1825	-1820
2370	-2258	-2355	2185	1825	-1845	-1820	1830	-2320	2275	-2350	-2380	-1840	1850	1825	-1820
2370	-2258	-2355	2185	-1825	1845	1820	-1830	-2320	2275	-2350	-2380	1840	-1850	-1825	1820

TABLE A1.6 Results of Calculations for Matrix Due to Laboratory 1 and Material 1

$Z_1 = 33\ 148$	$Z_1^2 = 1\ 098\ 789\ 904$	$W_1^2 = 68\ 674\ 369$
$Z_2 = 3\ 838$	$Z_2^2 = 14\ 730\ 244$	$W_2^2 = 920\ 640.25$
$Z_3 = 18$	$Z_3^2 = 324$	$W_3^2 = 20.25$
$Z_4 = 262$	$Z_4^2 = 68\ 644$	$W_4^2 = 4\ 290.25$
$Z_5 = -112$	$Z_5^2 = 12\ 544$	$W_5^2 = 784$
$Z_6 = 332$	$Z_6^2 = 110\ 224$	$W_6^2 = 6\ 889$
$Z_7 = -8$	$Z_7^2 = 64$	$W_7^2 = 4$
$Z_8 = 42$	$Z_8^2 = 1\ 764$	$W_8^2 = 110.25$
$Z_9 = -172$	$Z_9^2 = 29\ 584$	$W_9^2 = 1\ 849$
$Z_{10} = -142$	$Z_{10}^2 = 20\ 164$	$W_{10}^2 = 1\ 260.25$
$Z_{11} = 198$	$Z_{11}^2 = 39\ 204$	$W_{11}^2 = 2\ 450.25$
$Z_{12} = 242$	$Z_{12}^2 = 58\ 564$	$W_{12}^2 = 3\ 660.25$
$Z_{13} = 248$	$Z_{13}^2 = 61\ 504$	$W_{13}^2 = 3\ 844$
$Z_{14} = 292$	$Z_{14}^2 = 85\ 264$	$W_{14}^2 = 5\ 329$
$Z_{15} = -128$	$Z_{15}^2 = 16\ 384$	$W_{15}^2 = 1\ 024$
$Z_{16} = -138$	$Z_{16}^2 = 19\ 044$	$W_{16}^2 = 1\ 190.25$

the factor differences that yield the estimate of error variance. By adding W_{10} through W_{16} we can estimate the error variance with seven degrees of freedom using Eq A1.1:

$$\chi = \sum_{i=10}^{16} W_i^2 / 7\sigma^2 \quad (\text{A1.1})$$

where:

- χ = pooled sum of squares for error,
- W_i^2 = sum of squares for error in i th row, and
- σ^2 = true, but unknown error variance.

A1.7.1 By dividing χ into W_j^2/σ^2 , representing the sums of squares for the main factors, we can test for the significance of the j th factor difference as shown in Eq A1.2:

$$F_j = W_j^2 / \sigma^2 / \sum_{i=10}^{16} W_i^2 / 7\sigma^2 = W_j^2 / \sum_{i=10}^{16} W_i^2 / 7 \quad (\text{A1.2})$$

A1.7.2 Eq A1.2 will have an F -distribution with 1 and 7 degrees of freedom.

A1.8 The pooled sum of squares for error was determined and compared with the sums of squares for each of the main factors or treatments. The ratio that is significant at the 0.05 level is 5.59.

A1.9 F values for each of the main factors were calculated for Tables A1.6-A1.17. The results of these calculations are shown for all factors in Table A1.18. All ratios that were less than 5.59 are shown in the table as NS to show that they are not significant. Z_2 or the effect of temperature was found highly significant for every material and every laboratory indicating the importance of improved control of temperature. Z_4 or the effect of variation in vacuum showed five significant values indicating a need for

TABLE A1.7 Results of Calculations for Matrix due to Laboratory 1 and Material 2

$Z_1 = 7\ 234$	$Z_1^2 = 52\ 330\ 756$	$W_1^2 = 3\ 270\ 672.25$
$Z_2 = 834$	$Z_2^2 = 695\ 556$	$W_2^2 = 43\ 472.25$
$Z_3 = 18$	$Z_3^2 = 324$	$W_3^2 = 20.25$
$Z_4 = -10$	$Z_4^2 = 100$	$W_4^2 = 6.25$
$Z_5 = 6$	$Z_5^2 = 36$	$W_5^2 = 2.25$
$Z_6 = 26$	$Z_6^2 = 676$	$W_6^2 = 42.25$
$Z_7 = -30$	$Z_7^2 = 900$	$W_7^2 = 56.25$
$Z_8 = 18$	$Z_8^2 = 324$	$W_8^2 = 20.25$
$Z_9 = 4$	$Z_9^2 = 16$	$W_9^2 = 1.00$
$Z_{10} = -16$	$Z_{10}^2 = 256$	$W_{10}^2 = 16$
$Z_{11} = 24$	$Z_{11}^2 = 576$	$W_{11}^2 = 36$
$Z_{12} = 124$	$Z_{12}^2 = 15\ 376$	$W_{12}^2 = 961$
$Z_{13} = 16$	$Z_{13}^2 = 256$	$W_{13}^2 = 16$
$Z_{14} = 116$	$Z_{14}^2 = 13\ 456$	$W_{14}^2 = 841$
$Z_{15} = 4$	$Z_{15}^2 = 16$	$W_{15}^2 = 1$
$Z_{16} = -48$	$Z_{16}^2 = 2\ 304$	$W_{16}^2 = 144$

TABLE A1.8 Results of Calculations for Matrix due to Laboratory 1 and Material 3

$Z_1 = 58\ 618$	$Z_1^2 = 3\ 436\ 069\ 924$	$W_1^2 = 214\ 754\ 370.3$
$Z_2 = 6\ 898$	$Z_2^2 = 47\ 582\ 404$	$W_2^2 = 2\ 973\ 900.25$
$Z_3 = 312$	$Z_3^2 = 97\ 344$	$W_3^2 = 6\ 084$
$Z_4 = 624$	$Z_4^2 = 389\ 376$	$W_4^2 = 24\ 336$
$Z_5 = 456$	$Z_5^2 = 207\ 936$	$W_5^2 = 12\ 996$
$Z_6 = 764$	$Z_6^2 = 583\ 696$	$W_6^2 = 36\ 481$
$Z_7 = -214$	$Z_7^2 = 45\ 796$	$W_7^2 = 2\ 862.25$
$Z_8 = -218$	$Z_8^2 = 47\ 524$	$W_8^2 = 2\ 970.25$
$Z_9 = -318$	$Z_9^2 = 101\ 124$	$W_9^2 = 6\ 320.25$
$Z_{10} = -206$	$Z_{10}^2 = 42\ 436$	$W_{10}^2 = 2\ 652.25$
$Z_{11} = 216$	$Z_{11}^2 = 46\ 656$	$W_{11}^2 = 2\ 916$
$Z_{12} = 248$	$Z_{12}^2 = 61\ 504$	$W_{12}^2 = 3\ 844$
$Z_{13} = -288$	$Z_{13}^2 = 82\ 944$	$W_{13}^2 = 5\ 184$
$Z_{14} = 540$	$Z_{14}^2 = 291\ 600$	$W_{14}^2 = 18\ 225$
$Z_{15} = -150$	$Z_{15}^2 = 22\ 500$	$W_{15}^2 = 1\ 406.25$
$Z_{16} = -2$	$Z_{16}^2 = 4$	$W_{16}^2 = 0.25$

TABLE A1.9 Results of Calculations for Matrix due to Laboratory 1 and Material 4

$Z_1 = 14\ 692$	$Z_1^2 = 215\ 854\ 864$	$W_1^2 = 13\ 490\ 929$
$Z_2 = 1\ 892$	$Z_2^2 = 3\ 579\ 664$	$W_2^2 = 223\ 729$
$Z_3 = 208$	$Z_3^2 = 43\ 264$	$W_3^2 = 2\ 704$
$Z_4 = 122$	$Z_4^2 = 14\ 884$	$W_4^2 = 930.25$
$Z_5 = 232$	$Z_5^2 = 53\ 824$	$W_5^2 = 3\ 364$
$Z_6 = 94$	$Z_6^2 = 8\ 836$	$W_6^2 = 552.25$
$Z_7 = -78$	$Z_7^2 = 6\ 084$	$W_7^2 = 380.25$
$Z_8 = -162$	$Z_8^2 = 26\ 244$	$W_8^2 = 1\ 640.25$
$Z_9 = 26$	$Z_9^2 = 676$	$W_9^2 = 42.25$
$Z_{10} = 18$	$Z_{10}^2 = 324$	$W_{10}^2 = 20.25$
$Z_{11} = 2$	$Z_{11}^2 = 4$	$W_{11}^2 = .25$
$Z_{12} = 116$	$Z_{12}^2 = 13\ 456$	$W_{12}^2 = 841$
$Z_{13} = 18$	$Z_{13}^2 = 324$	$W_{13}^2 = 20.25$
$Z_{14} = 120$	$Z_{14}^2 = 14\ 400$	$W_{14}^2 = 900$
$Z_{15} = -64$	$Z_{15}^2 = 4\ 096$	$W_{15}^2 = 256$
$Z_{16} = -36$	$Z_{16}^2 = 1\ 296$	$W_{16}^2 = 81$

tightened controls on vacuum. Z_6 or the effect of the viscometer deviating from the vertical position was significant in six of the laboratory-material combinations indicating the need for tightened controls on the position of the viscometer. Z_3 , Z_5 , Z_7 , and Z_8 showed some scattering of barely significant values but these were not judged to be of sufficient importance to require tighter controls.

A1.10 Representatives of the three laboratories met after completion of the laboratory work and the subsequent analysis. After discussion of the results, the decision was made that it was practical and desirable to control temperature, vacuum, and the angle of the viscosity tube to the following limits:

Temperature 25 ± 0.1 , °C
 Vacuum 300 ± 2 , mm (Hg) and
 Angle with Horizontal $90 \pm 1^\circ$

TABLE A1.10 Results of Calculations for Matrix due to Laboratory 2 and Material 1

$Z_1 = 32\ 692$	$Z_1^2 = 1\ 068\ 766\ 864$	$W_1^2 = 66\ 797\ 929$
$Z_2 = 3\ 708$	$Z_2^2 = 13\ 749\ 264$	$W_2^2 = 859\ 329$
$Z_3 = 190$	$Z_3^2 = 36\ 100$	$W_3^2 = 2\ 256.25$
$Z_4 = 516$	$Z_4^2 = 266\ 256$	$W_4^2 = 16\ 641$
$Z_5 = 130$	$Z_5^2 = 16\ 900$	$W_5^2 = 1\ 056.25$
$Z_6 = 544$	$Z_6^2 = 295\ 936$	$W_6^2 = 18\ 496$
$Z_7 = -358$	$Z_7^2 = 128\ 164$	$W_7^2 = 8\ 010.25$
$Z_8 = -382$	$Z_8^2 = 145\ 924$	$W_8^2 = 9\ 120.25$
$Z_9 = -32$	$Z_9^2 = 1\ 024$	$W_9^2 = 64$
$Z_{10} = -8$	$Z_{10}^2 = 64$	$W_{10}^2 = 4$
$Z_{11} = 30$	$Z_{11}^2 = 900$	$W_{11}^2 = 56.25$
$Z_{12} = -16$	$Z_{12}^2 = 256$	$W_{12}^2 = 16$
$Z_{13} = 10$	$Z_{13}^2 = 100$	$W_{13}^2 = 6.25$
$Z_{14} = 76$	$Z_{14}^2 = 5\ 776$	$W_{14}^2 = 361$
$Z_{15} = 218$	$Z_{15}^2 = 47\ 524$	$W_{15}^2 = 2\ 970.25$
$Z_{16} = 282$	$Z_{16}^2 = 79\ 524$	$W_{16}^2 = 4\ 970.25$

TABLE A1.11 Results of Calculations for Matrix due to Laboratory 2 and Material 2

$Z_1 = 7\ 543$	$Z_1^2 = 56\ 896\ 849$	$W_1^2 = 3\ 556\ 053.063$
$Z_2 = 803$	$Z_2^2 = 644\ 809$	$W_2^2 = 40\ 300.5625$
$Z_3 = 53$	$Z_3^2 = 2\ 809$	$W_3^2 = 175.5625$
$Z_4 = 57$	$Z_4^2 = 3\ 249$	$W_4^2 = 203.0625$
$Z_5 = 73$	$Z_5^2 = 5\ 329$	$W_5^2 = 333.0625$
$Z_6 = 81$	$Z_6^2 = 6\ 561$	$W_6^2 = 410.0625$
$Z_7 = -97$	$Z_7^2 = 9\ 409$	$W_7^2 = 588.0625$
$Z_8 = -49$	$Z_8^2 = 2\ 401$	$W_8^2 = 150.0625$
$Z_9 = 15$	$Z_9^2 = 225$	$W_9^2 = 14.0625$
$Z_{10} = 15$	$Z_{10}^2 = 225$	$W_{10}^2 = 14.0625$
$Z_{11} = -11$	$Z_{11}^2 = 121$	$W_{11}^2 = 7.5625$
$Z_{12} = 57$	$Z_{12}^2 = 3\ 249$	$W_{12}^2 = 203.0625$
$Z_{13} = -51$	$Z_{13}^2 = 2\ 601$	$W_{13}^2 = 162.5625$
$Z_{14} = 61$	$Z_{14}^2 = 3\ 721$	$W_{14}^2 = 232.5625$
$Z_{15} = 71$	$Z_{15}^2 = 5\ 041$	$W_{15}^2 = 315.0625$
$Z_{16} = 19$	$Z_{16}^2 = 361$	$W_{16}^2 = 22.5625$

TABLE A1.12 Results of Calculations for Matrix due to Laboratory 2 and Material 3

$Z_1 = 58\ 527$	$Z_1^2 = 3\ 425\ 409\ 729$	$W_1^2 = 214\ 088\ 108.1$
$Z_2 = 7\ 123$	$Z_2^2 = 50\ 737\ 129$	$W_2^2 = 3\ 171\ 070.563$
$Z_3 = 755$	$Z_3^2 = 570\ 025$	$W_3^2 = 35\ 626.5625$
$Z_4 = 423$	$Z_4^2 = 178\ 929$	$W_4^2 = 11\ 183.0625$
$Z_5 = 247$	$Z_5^2 = 61\ 009$	$W_5^2 = 3\ 813.0625$
$Z_6 = 191$	$Z_6^2 = 36\ 481$	$W_6^2 = 2\ 280.0625$
$Z_7 = 443$	$Z_7^2 = 196\ 249$	$W_7^2 = 12\ 265.5625$
$Z_8 = 171$	$Z_8^2 = 29\ 241$	$W_8^2 = 1\ 827.5625$
$Z_9 = -145$	$Z_9^2 = 21\ 025$	$W_9^2 = 1\ 314.0625$
$Z_{10} = -433$	$Z_{10}^2 = 187\ 489$	$W_{10}^2 = 11\ 718.0625$
$Z_{11} = 171$	$Z_{11}^2 = 29\ 241$	$W_{11}^2 = 1\ 827.5625$
$Z_{12} = 55$	$Z_{12}^2 = 3\ 025$	$W_{12}^2 = 189.0625$
$Z_{13} = -77$	$Z_{13}^2 = 5\ 929$	$W_{13}^2 = 370.5625$
$Z_{14} = 1\ 107$	$Z_{14}^2 = 1\ 225\ 449$	$W_{14}^2 = 76\ 590.5625$
$Z_{15} = -413$	$Z_{15}^2 = 170\ 569$	$W_{15}^2 = 10\ 660.5625$
$Z_{16} = -385$	$Z_{16}^2 = 148\ 225$	$W_{16}^2 = 9\ 264.0625$

With these changes an interlaboratory study was made on the method.—

TABLE A1.13 Results of Calculations for Matrix due to Laboratory 2 and Material 4

$Z_1 = 15\ 095$	$Z_1^2 = 227\ 859\ 025$	$W_1^2 = 14\ 241\ 189.06$
$Z_2 = 1\ 969$	$Z_2^2 = 3\ 876\ 961$	$W_2^2 = 242\ 310.0625$
$Z_3 = 179$	$Z_3^2 = 32\ 041$	$W_3^2 = 2\ 002.5625$
$Z_4 = 149$	$Z_4^2 = 22\ 201$	$W_4^2 = 1\ 387.5625$
$Z_5 = 265$	$Z_5^2 = 70\ 225$	$W_5^2 = 4\ 389.0625$
$Z_6 = 135$	$Z_6^2 = 18\ 225$	$W_6^2 = 1\ 139.0625$
$Z_7 = 5$	$Z_7^2 = 25$	$W_7^2 = 1.5625$
$Z_8 = -101$	$Z_8^2 = 10\ 201$	$W_8^2 = 637.5625$
$Z_9 = 115$	$Z_9^2 = 13\ 225$	$W_9^2 = 826.5625$
$Z_{10} = -121$	$Z_{10}^2 = 14\ 641$	$W_{10}^2 = 915.0625$
$Z_{11} = -67$	$Z_{11}^2 = 4\ 489$	$W_{11}^2 = 280.5625$
$Z_{12} = 195$	$Z_{12}^2 = 38\ 025$	$W_{12}^2 = 2\ 376.5625$
$Z_{13} = -69$	$Z_{13}^2 = 4\ 761$	$W_{13}^2 = 297.5625$
$Z_{14} = 189$	$Z_{14}^2 = 35\ 721$	$W_{14}^2 = 2\ 232.5625$
$Z_{15} = -65$	$Z_{15}^2 = 4\ 225$	$W_{15}^2 = 264.0625$
$Z_{16} = -11$	$Z_{16}^2 = 121$	$W_{16}^2 = 7.5625$

TABLE A1.14 Results of Calculations for Matrix due to Laboratory 3 and Material 1

$Z_1 = 33\ 341$	$Z_1^2 = 1\ 111\ 622\ 281$	$W_1^2 = 69\ 476\ 392.56$
$Z_2 = 3\ 691$	$Z_2^2 = 13\ 623\ 481$	$W_2^2 = 851\ 467.5625$
$Z_3 = 171$	$Z_3^2 = 29\ 241$	$W_3^2 = 1\ 827.5625$
$Z_4 = 519$	$Z_4^2 = 269\ 361$	$W_4^2 = 16\ 835.0625$
$Z_5 = 141$	$Z_5^2 = 19\ 881$	$W_5^2 = 1\ 242.5625$
$Z_6 = 509$	$Z_6^2 = 259\ 081$	$W_6^2 = 16\ 192.5625$
$Z_7 = -51$	$Z_7^2 = 2\ 601$	$W_7^2 = 162.5625$
$Z_8 = -1$	$Z_8^2 = 1$	$W_8^2 = 0.0625$
$Z_9 = -45$	$Z_9^2 = 2\ 025$	$W_9^2 = 126.5625$
$Z_{10} = 5$	$Z_{10}^2 = 25$	$W_{10}^2 = 1.5625$
$Z_{11} = 45$	$Z_{11}^2 = 2\ 025$	$W_{11}^2 = 126.5625$
$Z_{12} = -15$	$Z_{12}^2 = 225$	$W_{12}^2 = 14.0625$
$Z_{13} = -5$	$Z_{13}^2 = 25$	$W_{13}^2 = 1.5625$
$Z_{14} = 115$	$Z_{14}^2 = 13\ 225$	$W_{14}^2 = 826.5625$
$Z_{15} = -85$	$Z_{15}^2 = 7\ 225$	$W_{15}^2 = 451.5625$
$Z_{16} = -95$	$Z_{16}^2 = 9\ 025$	$W_{16}^2 = 564.0625$

TABLE A1.15 Results of Calculations for Matrix due to Laboratory 3 and Material 2

$Z_1 = 7\ 078$	$Z_1^2 = 50\ 098\ 084$	$W_1^2 = 3\ 131\ 130.25$
$Z_2 = 824$	$Z_2^2 = 678\ 976$	$W_2^2 = 42\ 436$
$Z_3 = 26$	$Z_3^2 = 676$	$W_3^2 = 42.25$
$Z_4 = 108$	$Z_4^2 = 11\ 664$	$W_4^2 = 729$
$Z_5 = 0$	$Z_5^2 = 0$	$W_5^2 = 0$
$Z_6 = 126$	$Z_6^2 = 15\ 876$	$W_6^2 = 992.25$
$Z_7 = -8$	$Z_7^2 = 64$	$W_7^2 = 4$
$Z_8 = -34$	$Z_8^2 = 1\ 156$	$W_8^2 = 72.25$
$Z_9 = 0$	$Z_9^2 = 0$	$W_9^2 = 0$
$Z_{10} = -6$	$Z_{10}^2 = 36$	$W_{10}^2 = 2.25$
$Z_{11} = 16$	$Z_{11}^2 = 256$	$W_{11}^2 = 16$
$Z_{12} = 6$	$Z_{12}^2 = 36$	$W_{12}^2 = 2.25$
$Z_{13} = 22$	$Z_{13}^2 = 484$	$W_{13}^2 = 30.25$
$Z_{14} = 16$	$Z_{14}^2 = 256$	$W_{14}^2 = 16$
$Z_{15} = -18$	$Z_{15}^2 = 324$	$W_{15}^2 = 20.25$
$Z_{16} = 4$	$Z_{16}^2 = 16$	$W_{16}^2 = 1$

TABLE A1.16 Results of Calculations for Matrix due to Laboratory 3 and Material 3

$Z_1 = 57\ 932$	$Z_1^2 = 3\ 356\ 116\ 624$	$W_1^2 = 209\ 757\ 289$
$Z_2 = 6\ 770$	$Z_2^2 = 45\ 832\ 900$	$W_2^2 = 2\ 864\ 556.25$
$Z_3 = 390$	$Z_3^2 = 152\ 100$	$W_3^2 = 9\ 506.25$
$Z_4 = 948$	$Z_4^2 = 898\ 704$	$W_4^2 = 56\ 169$
$Z_5 = 288$	$Z_5^2 = 82\ 944$	$W_5^2 = 5\ 184$
$Z_6 = 1\ 070$	$Z_6^2 = 1\ 144\ 900$	$W_6^2 = 71\ 556.25$
$Z_7 = -190$	$Z_7^2 = 36\ 100$	$W_7^2 = 2\ 256.25$
$Z_8 = -168$	$Z_8^2 = 28\ 224$	$W_8^2 = 1\ 764$
$Z_9 = -58$	$Z_9^2 = 3\ 364$	$W_9^2 = 210.25$
$Z_{10} = -40$	$Z_{10}^2 = 1\ 600$	$W_{10}^2 = 100$
$Z_{11} = 180$	$Z_{11}^2 = 32\ 400$	$W_{11}^2 = 2\ 025$
$Z_{12} = -22$	$Z_{12}^2 = 484$	$W_{12}^2 = 30.25$
$Z_{13} = -62$	$Z_{13}^2 = 3\ 844$	$W_{13}^2 = 240.25$
$Z_{14} = 280$	$Z_{14}^2 = 78\ 400$	$W_{14}^2 = 4\ 900$
$Z_{15} = -60$	$Z_{15}^2 = 3\ 600$	$W_{15}^2 = 225$
$Z_{16} = -58$	$Z_{16}^2 = 3\ 364$	$W_{16}^2 = 210.25$

TABLE A1.17 Results of Calculations for Matrix due to Laboratory 3 and Material 4

$Z_1 = 14\ 259$	$Z_1^2 = 203\ 319\ 081$	$W_1^2 = 12\ 707\ 442.56$
$Z_2 = 1\ 831$	$Z_2^2 = 3\ 352\ 561$	$W_2^2 = 209\ 535.0625$
$Z_3 = 45$	$Z_3^2 = 2\ 025$	$W_3^2 = 126.5625$
$Z_4 = 343$	$Z_4^2 = 117\ 649$	$W_4^2 = 7\ 353.0625$
$Z_5 = 105$	$Z_5^2 = 11\ 025$	$W_5^2 = 689.0625$
$Z_6 = 267$	$Z_6^2 = 71\ 289$	$W_6^2 = 4\ 455.5625$
$Z_7 = -23$	$Z_7^2 = 529$	$W_7^2 = 33.0625$
$Z_8 = -107$	$Z_8^2 = 11\ 449$	$W_8^2 = 715.5625$
$Z_9 = -35$	$Z_9^2 = 1\ 225$	$W_9^2 = 76.5625$
$Z_{10} = -35$	$Z_{10}^2 = 1\ 225$	$W_{10}^2 = 76.5625$
$Z_{11} = 99$	$Z_{11}^2 = 9\ 801$	$W_{11}^2 = 612.5625$
$Z_{12} = 17$	$Z_{12}^2 = 289$	$W_{12}^2 = 18.0625$
$Z_{13} = 51$	$Z_{13}^2 = 2\ 601$	$W_{13}^2 = 162.5625$
$Z_{14} = 33$	$Z_{14}^2 = 1\ 089$	$W_{14}^2 = 68.0625$
$Z_{15} = -17$	$Z_{15}^2 = 289$	$W_{15}^2 = 18.0625$
$Z_{16} = -33$	$Z_{16}^2 = 1\ 089$	$W_{16}^2 = 68.0625$

TABLE A1.18 Summary of F Values For All Laboratories, All Materials, And All Factors

Laboratory	Material	Table	Temperature of Tube, F_a	Age, F_b	Vacuum, F_c	Stir, F_d	Vertical, F_e	Meniscus, F_f	Bath, F_g
1	1	A1.6	343.56	NS	NS	NS	NS	NS	NS
	2	A1.7	151.02	NS	NS	NS	NS	NS	NS
	3	A1.8	608.20	NS	NS	NS	7.46	NS	NS
	4	A1.9	739.16	8.93	NS	11.11	NS	NS	NS
2	1	A1.10	717.47	NS	13.89	NS	15.44	6.69	7.61
	2	A1.11	294.64	NS	NS	NS	NS	NS	NS
	3	A1.12	200.66	NS	NS	NS	NS	NS	NS
	4	A1.13	266.11	NS	NS	NS	NS	NS	NS
3	1	A1.14	3 001.24	6.44	59.34	NS	57.08	NS	NS
	2	A1.15	3 375.59	NS	57.99	NS	78.93	NS	5.74
	3	A1.16	2 593.81	8.61	50.86	NS	64.79	NS	NS
	4	A1.17	1 432.46	NS	50.26	NS	30.46	NS	NS

■ —

A2. THEORY OF THE RUGGEDNESS ANALYSIS

A2.1 Any mathematical analysis depends on assumptions. It is particularly difficult to make valid assumptions when there is little experience on which to make the assumptions. Since a ruggedness or screening program is usually run on a new test method, there is very little history or experience to validate the necessary assumptions. An extensive study could yield the experience to validate the assumptions, but it would also increase the cost of the ruggedness program to the point that few such programs could be undertaken. This practice seeks to balance these risks to make the practice practical and useful.

A2.2 A ruggedness program attempts to identify the important factors or variables which cause variability of results obtained using the test method. It is important that all of the major factors be included in the study, since, if one is left out, the study will not help in identifying its significance. The example in Annex A1 evaluates seven factors. This is usually sufficient to cover the major sources of variability. Design for both fewer and more factors are given in statistical texts for use when needed (5).

A2.2.1 It is unusual for more than seven or for less than five factors to be required. When only five factors are considered significant, two other factors can nearly always be selected about which there may be some doubt. A seven factor analysis is usually suitable for most ruggedness evaluations.

A2.2.2 A full factorial experiment for seven factors at two levels would require 2^7 or 128 runs or determinations for each laboratory and each material. If this design had been followed in Annex A1, 128 runs times 3 laboratories times 4 materials times 2 replications equals 3072 determinations instead of the 192 determinations actually required in the example.

A2.2.3 A full factorial experiment identifies not only significant effects of the main factors but also those of the interactions. An interaction is a source of variability due to the combination of main factors that cannot be explained by the individual variability of those main factors. There are instances where the effect of an interaction is greater than the sum of the effects of the main factors that create the interaction. However, interactions are usually regarded as being smaller sources of variation; particularly three and four factor interactions. In this standard, the effect of all interactions are assumed to be negligible. This is done to permit testing of the significance of a large number of main effects while holding the size of the experiment down to manageable levels. The effect of interactions will not always be negligible and there are times when an estimate of a main effect will include an interaction.

A2.2.4 The assumption is also made that the random errors are normally distributed. Since a new method is being evaluated, there is no data available to show that the errors are normally distributed. However, the assumption appears reasonable based on experience with other ASTM test methods.

A2.2.5 While the risks inherent in adopting these assumptions are real, it is thought that despite the risks the ruggedness program will serve a useful purpose in improving the precision of most test methods.

A2.3 Two levels have been chosen for this study. Three or more levels could have been used. The higher number of levels would have given information about the shape of the curves that could be derived by plotting the viscosity versus each of the main factors at the levels chosen. Increasing the levels would also increase the number of runs or determinations. For example, seven factors at three levels would require 2187 runs compared with 128 at two levels. Since each of the factors vary by a small amount, the curves would be substantially straight and there would be little gained by measuring at a higher number of levels than two.

A2.4 A linear model or equation for the seven main factor ruggedness program would be:

$$Z = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_5X_5 + B_6X_6 + B_7X_7 + e \quad (\text{A2.1})$$

where:

X_1 = through X_7 are the main effects,
 B_0 = through B_7 are the unknown parameters, and
 e = is random error.

A2.4.1 A Youden Square, which is the basis for this ruggedness program, is in turn part of a Hadamard matrix an example of which is shown in Table A2.1. The Youden Square would be Table A2.1 with the top or Z_1 line removed. Table A2.1 represents the values of B that equate each of the Z 's to the $X_0, X_1, X_2,$ through X_7 in eight linear equations. The fact that Hadamard matrices are orthogonal makes each row of Table A2.1 an independent vector in the transformation matrix which is Table A2.1. This also means that when any line in the table is multiplied by any other line the sum is always zero. The B_0 through B_7 are represented by numbers + 1 or - 1 shown on each line of Table A2.1.

A2.4.2 It should be noted that Z_2 divided by 4 is the difference between average results when temperature is at a high level and the average results when temperature is at a low level. The design of the experiment based on the characteristic of Hadamard matrices, is such that when determinations of temperature are at a high level all other factors are twice at a high level and twice at a low level. When determinations of temperature are at a low level, all other factors are twice at a high level and twice at a low level. Measurement of temperature is freed of the effect of the level of the factors other than temperature leaving only the effect

TABLE A2.1 Values for the coefficients B₀ through B₇

	X ₀	X ₁	X ₂	X ₃	X ₄	X ₅	X ₆	X ₇
Z ₁	1	1	1	1	1	1	1	1
Z ₂	1	1	1	1	-1	-1	-1	-1
Z ₃	1	1	-1	-1	1	1	-1	-1
Z ₄	1	-1	1	-1	1	-1	1	-1
Z ₅	1	1	-1	-1	-1	-1	1	1
Z ₆	1	-1	1	-1	-1	1	-1	1
Z ₇	1	-1	-1	1	1	-1	-1	1
Z ₈	1	-1	-1	1	-1	1	1	-1

of the temperature and of random error, e of the other factors.

A2.4.3 A close examination of Table A2.1 shows that what is true for temperature is also true for each of the other factors. With the experimental design in Table A2.1, separate assessments of each of the effects of seven factors can be made with only eight tests.

A2.4.4 There is no separate estimate of the error variance by which the statistical significance of the factor effects can be tested.

A2.5 An estimate of error variance can be obtained by replicating the experiment. It is important in experiments of this kind to keep the amount of work and therefore the cost as low as possible while obtaining valid results. The amount of work would be halved if the work of each laboratory is regarded as a replication of the experiment. Experience in ASTM interlaboratory studies shows that there usually are significant differences between laboratories. Therefore, the recommendation is that the experiment be replicated in each laboratory according to the experimental design shown in Table A1.4 and used in Annex A1.1.

A2.6 The first eight rows in Table A1.4 are the same as in Table A2.1 except that they are repeated in the second replication. Z₉ shows the differences between replications. The coefficients in Z₁₀ through Z₁₆ are exactly the same as those in Z₂ through Z₈ but in the replication all signs are reversed in Z₁₀ through Z₁₆. It is these differences from replicate to replicate that yield the necessary estimate of error variance. The assumption as already stated is made that there are no real interactions between factor differences and replications.

A2.7 The transformation matrix for the 16 determinations is shown in Table A1.4. A column containing values for $W_i^2 = Z_i^2/D_i$ appears in Table A1.6 and Table A1.7 where: Z_{*i*} equals the sum of the Z's in the *i*th row squared, and D_{*i*} equals the sum of squares of the coefficients in the *i*th row.

In all instances for an Hadamard matrix D_{*i*} equals the number of columns in the matrix since both 1 and $-1^2 = 1$. All rows in Table A1.4 sum to 16 so D_{*i*} = 16 for every row.

If σ^2 is the true, but unknown error variance, then on the assumption that the errors are normally distributed each W_i^2/σ^2 will have an independent χ^2 -distribution with 1° of freedom. The sum $\sum_{i=10}^{16} W_i^2/7\sigma^2$ represents the pooled sums of squares for error divided by the error variance that will have a χ^2 distribution with 7° of freedom. For the *i* th row of Table A1.4, *j* = 2 to 8, the ratio will have an *F*-distribution with 1 and 7° of freedom.

$$F_j = W_j^2/\sigma^2 / \sum_{i=10}^{16} W_i^2/7\sigma^2 = W_j^2 / \sum_{i=10}^{16} W_i^2/7 \quad (\text{A2.2})$$

A2.8 Annex A1 consists of a detailed example using the theory of Annex A2. It is recommended that the test of significance be carried out at the probability level of 0.05 of the *F*-distribution. The ratio that is significant at the 0.05 probability level can be obtained from tables of the *F*-distribution that are in most standard statistical text books.

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