Designation: E 826 – 85 (Reapproved 1996)$^{\epsilon 1}$

Standard Practice for Testing Homogeneity of Materials for Development of Reference Materials[1]

This standard is issued under the fixed designation E 826; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 NOTE—Section 12 was added editorially in June 1996.

1. Scope

1.1 This practice is suitable for testing the homogeneity of metals, either in solid or powdered form, and finely ground oxide materials that are intended for use as reference materials in X-ray emission, or optical emission spectroscopy, or both. The criteria for acceptance of the test specimens as reference materials, however, must be previously determined by the user for meeting his specific requirements.

1.2 The procedure is designed primarily for testing specimens by X-ray emission spectrometry or optical emission spectroscopy, or both. However, the practice could be easily adapted for use with other instrumental techniques such as atomic absorption spectrophotometry.

1.3 This procedure can be applied to one or more elements in a specimen provided the signal-to-background ratio is not a limiting factor.

1.4 This practice includes one method, if desired, to correct for systematic or periodic (sinusoidal) drift in the instrument with time through the use of a control reference material.

NOTE 1—**Caution:** If serious drift occurs (for example, unstable power supply, X-ray tube, etc.) erroneous conclusions may be obtained from the data analysis.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 178 Practice for Dealing with Outlying Observations[3]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E 135, and Practices E 177 and E 178.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *drift*—a gradual systematic or sinusoidal change in instrument readings with time.

3.2.2 *homogeneity—as defined in this practice*, is statistically acceptable differences among means of specimens in the test.

4. Summary of Practice

4.1 This procedure, which is based on statistical methods (**1-6**),[5] consists of stepwise instructions for testing homogeneity of candidate reference materials. The candidate materials are selected as described in Section 6, and then measured by either X-ray emission or optical emission spectroscopy (see Sections 7 and 8). The resultant data are corrected for instrumental drift, if desired (see Section 10), and then tabulated (see Table 1, Table X1.3, and Table X1.4) to facilitate the statistical calculations that are performed according to Section 9. The homogeneity of the material is determined from the results of the data analysis.

4.2 This procedure *requires* that repeated measurements on the same specimen have sufficient precision (that is, repeatability) through appropriate selection of instrumental parameters so that any significant difference among specimens can be detected with confidence.

4.3 This procedure requires that there be an absence of outliers in the data (see Practice E 178).

NOTE 2—**Caution:** The use of Practice E 178 dealing with outliers should be done with extreme care to ensure that values are not discarded that may be valid for the analysis.

5. Significance and Use

5.1 The purpose of this practice is to ensure the quality of

[1] This practice is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.22 on Statistics and Quality Control.

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

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

[4] *Annual Book of ASTM Standards*, Vol 03.06.

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

TABLE 1 Data for Homogeneity Testing

Run No.	Specimen Number							Total
	1	2	3	4	5	...	...t^A	
1								B_1 =
2								B_2 =
3								B_3 =
4								B_4 =
5...								$B_{5...}$ =
...b^B								B_n =
Total	T_1 =	T_2 =	T_3 =	T_4 =	T_5 =	...	T_n =	$G = (\Sigma B_1...B_n)$
Mean	t'_1 =	t'_2 =	t'_3 =	t'_4 =	t'_5 =	...	t'_n =	

[A] t = number of specimens.
[B] b = number of runs.

materials selected in order that they can serve as a supplement to primary standard reference materials.

5.2 This procedure is applicable to the testing of samples taken at various stages during production. For example, continuous cast materials, ingots, rolled bars, wire, etc., could be sampled at various stages during the production process and tested.

6. Selection of Test Specimens from a Large Batch

6.1 If the candidate material consists of 15 specimens or less, then all specimens should be tested.

6.2 If the candidate material is in a form or quantity that prohibits testing all specimens, then a minimum of 8 % but not less than 15 specimens shall be tested according to the random sample selection scheme described in 6.3.

6.3 Label all specimens consecutively (that is, 01, 02, 03,⋯). From a table of random numbers **(3)** (see Table 2 and Note 3), pick an arbitrary starting place and select any direction for reading the numbers, provided the direction is fixed in advance and is independent of the numbers occurring. Select those specimens for testing which match the numbers read from the tables.

NOTE 3—**Caution:** Table 2 included herein is for example, only. Use the more complete tables in **(3)** when actually using this test procedure.

7. X-Ray Emission Spectroscopy Test Procedure

7.1 Select optimum instrumental conditions to assure adequate count rates from each element to be tested in the specimens.

7.2 Select a counting time that is long enough to minimize the random error due to counting. Also, avoid counting rates greater than 70 000 c/s to minimize dead-time corrections in the detection system.

7.3 Measure the element(s) of interest on the specimens selected in Section 6. Repeat the measurements of X-ray intensity until a minimum of four sets have been made. For each set, the specimens shall be taken in random order.

7.4 If correction of instrumental drift is desired, a control reference material shall be measured along with the specimens.

7.5 Examine the data and discard any values which have been determined to be outliers according to Practice E 178. In Table 1, enter either the raw or normalized values for each element, or values corrected for drift.

7.6 If any outliers occur, repeat the complete test, as provision is not made for missing data in the mathematical treatment.

8. Optical Emission Spectroscopy Test Procedure

8.1 Select optimum instrumental conditions to obtain adequate sensitivity for each element to be tested in the specimens.

8.2 Use excitation conditions appropriate for the element(s) of interest. Select a spectral line(s) that has a minimum of interferences from other elements in the specimen.

8.3 Measure the element(s) of interest in the specimens selected in Section 6. Repeat the measurements until a minimum of four sets have been made. For each set, take the specimens in random order.

8.4 If correction of instrumental drift is desired, measure a control reference material along with the specimens. The control reference material shall be homogeneous with respect to the element(s) being determined, in the specimens.

8.5 Examine the data and discard any values that have been determined to be outliers according to Practice E 178. In Table 1, enter either the raw or normalized values for each element or values corrected for drift.

8.6 If any outliers occur, repeat the complete test, as provision is not made for missing data in the mathematical treatment.

9. Calculations to Determine Homogeneity

9.1 Compute T, B, t', and G, (see Table 1), where: T = sum of each column; B = sum of each row; t' = mean of each column; and G = sum of $B_1 \cdots B_n$; b = number of replicate measurements (that is, runs); and t = number of specimens.

9.2 Choose a significance level (α) for the test.

NOTE 4—A 95 % significance level is recommended for this procedure. See **(1)** for more extensive tables containing values at other significance levels.

9.3 From Table 3, obtain the q value that corresponds to t and ν, where: ν = the number of degrees of freedom:

$$\nu = (b - 1) \times (t - 1) \quad (1)$$

9.4 Compute S_t = sum of squares due to specimens:

$$S_t = [(T_1{}^2 + T_2{}^2 + \cdots T_t^2)/b] - (G^2/tb) \quad (2)$$

9.5 Compute S_b = sum of squares due to runs:

$$S_b = [(B_1{}^2 + B_2{}^2 + \cdots B_b^2)/t] - (G^2/tb) \quad (3)$$

9.6 Compute $\bar{S}$ = sum of the squares of all the measurements in the tables and subtract G^2/tb:

$$\bar{S} = (\sum_{i=1}^{t})(\sum_{j=1}^{b}) Y_{ij}{}^2 - (G^2/tb) \quad (4)$$

TABLE 2 Short Table of Random Numbers[A]

NOTE 1—**Caution:** See Note 3Note 3.

46	96	85	77	27	92	86	26	45	21	89	91	71	42	64	64	58	22	75	81	74	91	48	46	18
44	19	15	32	63	55	87	77	33	29	45	00	31	34	84	05	72	90	44	27	78	22	07	62	17
34	39	80	62	24	33	81	67	28	11	34	79	26	35	34	23	09	94	00	80	55	31	63	27	91
74	97	80	30	65	07	71	30	01	84	47	45	89	70	74	13	04	90	51	27	61	34	63	87	44
22	14	61	60	86	38	33	71	13	33	72	08	16	13	50	56	48	51	29	48	30	93	45	66	29
40	03	96	40	03	47	24	60	09	21	21	18	00	05	86	52	85	40	73	73	57	68	36	33	91
52	33	76	44	56	15	47	75	78	73	78	19	87	06	98	47	48	02	62	03	42	05	32	55	02
37	59	20	40	93	17	82	24	19	90	80	87	32	74	59	84	24	49	79	17	23	75	83	42	00
11	02	55	57	48	84	74	36	22	67	19	20	15	92	53	37	13	75	54	89	56	73	23	39	07
10	33	79	26	34	54	71	33	89	74	68	48	23	17	49	18	81	05	52	85	70	05	73	11	17
67	59	28	25	47	89	11	65	65	20	42	23	96	41	64	20	30	89	87	64	37	93	36	96	35
93	50	75	20	09	18	54	34	68	02	54	87	23	05	43	36	98	29	97	93	87	08	30	92	98
24	43	23	72	80	64	34	27	23	46	15	36	10	63	21	59	69	76	02	62	31	62	47	60	34
39	91	63	18	38	27	10	78	88	84	42	32	00	97	92	00	04	94	50	05	75	82	70	80	35
74	62	19	67	54	18	28	92	33	69	98	96	74	35	72	11	68	25	08	95	31	79	11	79	54
91	03	35	60	81	16	61	97	25	14	78	21	22	05	25	47	26	37	80	39	19	06	41	02	00
42	57	66	76	72	91	03	63	48	46	44	01	33	53	62	28	80	59	55	05	02	16	13	17	54
06	36	63	06	15	03	72	38	01	58	25	37	66	48	56	19	56	41	29	28	76	49	74	39	50
92	70	96	70	89	80	87	14	25	49	25	94	62	78	26	15	41	39	48	75	64	69	61	06	38
91	08	88	53	52	13	04	82	23	00	26	36	47	44	04	08	84	80	07	44	76	51	52	41	59
68	85	97	74	47	53	90	05	90	84	87	48	25	01	11	05	45	11	43	15	60	40	31	84	59
59	54	13	09	13	80	42	29	63	03	24	64	12	43	28	10	01	65	62	07	79	83	05	59	61
39	18	32	69	33	46	58	19	34	03	59	28	97	31	02	65	47	47	70	39	74	17	30	22	65
67	43	31	09	12	60	19	57	63	78	11	80	10	97	15	70	04	89	81	78	54	84	87	83	42
61	75	37	19	56	90	75	39	03	56	49	92	72	95	27	52	87	47	12	52	54	62	43	23	13
78	10	91	11	00	63	19	63	74	58	69	03	51	38	60	36	53	56	77	06	69	03	89	91	24
93	23	71	58	09	78	08	03	07	71	79	32	25	19	61	04	40	33	12	06	78	91	97	88	95
37	55	48	82	63	89	92	59	14	72	19	17	22	51	90	20	03	64	96	60	48	01	95	44	84
62	13	11	71	17	23	29	25	13	85	33	35	07	69	25	68	57	92	57	11	84	44	01	33	66
29	89	97	47	03	13	20	86	22	45	59	98	64	53	89	64	94	81	55	87	73	81	58	46	42
16	94	85	82	89	07	17	30	29	89	89	80	98	36	25	36	53	02	49	14	34	03	52	09	20
04	93	10	59	75	12	98	84	60	93	68	16	87	60	11	50	46	56	58	45	88	72	50	46	11
95	71	43	68	97	18	85	17	13	08	00	50	77	50	46	92	45	26	97	21	48	22	23	08	32
86	05	39	14	35	48	68	18	36	57	09	62	40	28	87	08	74	79	91	08	27	12	43	32	03
59	30	60	10	41	31	00	69	63	77	01	89	94	60	19	02	70	88	72	33	38	88	20	60	86
05	45	35	40	54	03	98	96	76	27	77	84	80	08	64	60	44	34	54	24	85	20	85	77	32
71	85	17	74	66	27	85	19	55	56	51	36	48	92	32	44	40	47	10	38	22	52	42	29	96
80	20	32	80	98	00	40	92	57	51	52	83	14	55	31	99	73	23	40	07	64	54	44	99	21
13	50	78	02	73	39	66	82	01	28	67	51	75	66	33	97	47	58	42	44	88	09	28	58	06
67	92	65	41	45	36	77	96	46	21	14	39	56	36	70	15	74	43	62	69	82	30	77	28	77
72	56	73	44	26	04	62	81	15	35	79	26	99	57	28	22	25	94	80	62	95	48	98	23	86
28	86	85	64	94	11	58	78	45	36	34	45	91	38	51	10	68	36	87	81	16	77	30	19	36
69	57	40	80	44	94	60	82	94	93	98	01	48	50	57	69	60	77	69	60	74	22	05	77	17
71	20	03	30	79	25	74	17	78	34	54	45	04	77	42	59	75	78	64	99	37	03	18	03	36
89	98	55	98	22	45	12	49	82	71	57	33	28	69	50	59	15	09	25	79	39	42	84	18	70
58	74	82	81	14	02	01	05	77	94	65	57	70	39	42	48	56	84	31	59	18	70	41	74	60
50	54	73	81	91	07	81	26	25	45	49	61	22	88	41	20	00	15	59	93	51	60	65	65	63
49	33	72	90	10	20	65	28	44	63	95	86	75	78	69	24	41	65	86	10	34	10	32	00	93
11	85	01	43	65	02	85	69	56	88	34	29	64	35	48	15	70	11	77	83	01	34	82	91	04
34	22	46	41	84	74	27	02	57	77	47	93	72	02	95	63	75	74	69	69	61	34	31	92	13

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where:

Y_{ij} = individual values in the table.

9.7 Compute:

$$s = \sqrt{(\bar{S} - S_b - S_t)/(b - 1)(t - 1)} \quad (5)$$

9.8 Compute:

$$w = qs/\sqrt{b} \quad (6)$$

9.9 If the absolute difference between any two mean values (that is, $t'_1 \cdots t'_n$) exceeds w, then there is strong evidence, at the 95 % confidence level, that the specimens are not homogeneous. If the absolute difference between any two mean values does *not* exceed w, then the specimens shall be considered homogeneous.

10. Test for Instrumental Drift

10.1 This test for drift is made on repeat analyses of the control reference material (C) measured along with the specimens (see Practice E 876). The control reference material is measured at the beginning of each test set and repeated after

TABLE 3 Values of q for Various Combinations of t and ν at 95 % Significance Level (1)

ν ↓ / t→	2	3	4	5	6	7	8	9	10
1	17.97	26.98	32.82	37.08	40.41	43.12	45.40	47.36	49.07
2	6.08	8.33	9.80	10.88	11.74	12.44	13.03	13.54	13.99
3	4.50	5.91	6.82	7.50	8.04	8.48	8.85	9.18	9.46
4	3.93	5.04	5.76	6.29	6.71	7.05	7.35	7.60	7.83
5	3.64	4.60	5.22	5.67	6.03	6.33	6.58	6.80	6.99
6	3.46	4.34	4.90	5.30	5.63	5.90	6.12	6.32	6.49
7	3.34	4.16	4.68	5.06	5.36	5.61	5.82	6.00	6.16
8	3.26	4.04	4.53	4.89	5.17	5.40	5.60	5.77	5.92
9	3.20	3.95	4.41	4.76	5.02	5.24	5.43	5.59	5.74
10	3.15	3.88	4.33	4.65	4.91	5.12	5.30	5.46	5.60
11	3.11	3.82	4.26	4.57	4.82	5.03	5.20	5.35	5.49
12	3.08	3.77	4.20	4.51	4.75	4.95	5.12	5.27	5.39
13	3.06	3.73	4.15	4.45	4.69	4.88	5.05	5.19	5.32
14	3.03	3.70	4.11	4.41	4.64	4.83	4.99	5.13	5.25
15	3.01	3.67	4.08	4.37	4.59	4.78	4.94	5.08	5.20
16	3.00	3.65	4.05	4.33	4.56	4.74	4.90	5.03	5.15
17	2.98	3.63	4.02	4.30	4.52	4.70	4.86	4.99	5.11
18	2.97	3.61	4.00	4.28	4.49	4.67	4.82	4.96	5.07
19	2.96	3.59	3.98	4.25	4.47	4.65	4.79	4.92	5.04
20	2.95	3.58	3.96	4.23	4.45	4.62	4.77	4.90	5.01
24	2.92	3.53	3.90	4.17	4.37	4.54	4.68	4.81	4.92
30	2.89	3.49	3.85	4.10	4.30	4.46	4.60	4.72	4.82
40	2.86	3.44	3.79	4.04	4.23	4.39	4.52	4.63	4.73
60	2.83	3.40	3.74	3.98	4.16	4.31	4.44	4.55	4.65
120	2.80	3.36	3.68	3.92	4.10	4.24	4.36	4.47	4.56
∞	2.77	3.31	3.63	3.86	4.03	4.17	4.29	4.39	4.47

every three, five, or ten specimens have been run.

10.1.1 Select the control reference material frequency (such as, three, five, or ten) and maintain this measurement sequence throughout the entire test.

10.1.2 Arrange the measurements on the control reference material in the exact sequence in which they were made. For example: $C_1, C_2, C_3, \cdots C_n$.

10.1.3 Obtain the differences (Δ) between immediate successive measurements as follows:

$$\Delta_1 = C_1 - C_2; \Delta_2 = C_2 - C_3, \cdots \quad (7)$$

10.1.4 Calculate an estimate of variance S_1^2 as follows:

$$S_1^2 = \Sigma\Delta^2/(n-1) \quad (8)$$

where:

Δ = difference between successive measurements, and
n = number of times the control reference material was run.

10.1.5 Calculate a second estimate of variance S_2^2 as follows:

$$S_2^2 = \Sigma d^2/(n-1) \quad (9)$$

where:

d = difference of each measurement on the control reference material from the overall average of the measurements on the control.

10.1.6 Calculate the ratio:

$$S_1^2/S_2^2 (\mathbf{4\text{-}6}) \quad (10)$$

If the ratio S_1^2/S_2^2 is *larger* than the values listed in Table 4, for example, for the number of times the control reference material was measured, there is not sufficient evidence at the 95 % confidence level to indicate that drift has occurred. If no drift has occurred, the values obtained on the specimens should be tabulated in Table 1 in the order in which they were made, and then proceed to perform the calculations to determine homogeneity. However, if the ratio is *smaller* than the value listed in Table 4, for example, for the appropriate number of runs, there is strong evidence, with 95 % confidence, that drift has occurred. When drift has occurred, make corrections as specified in 10.2.

TABLE 4 Critical Values for Determining Occurrence of Drift from S_1^2/S_2^2 Ratio[A]

Number of Measurements on Control Reference Material, n	Ratio
4	0.78
5	0.82
6	0.89
7	0.94
8	0.98
9	1.02
10	1.06
11	1.10
12	1.13
15	1.21
20	1.30
25	1.37

[A] This table (from (**3**) is shown as an example. For more complete tables, see (**4 and 5**). However, the values in the latter references are half the values shown in this table because of a slightly different method of dermination.

10.2 *Calculation of Drift Factors*—Correct the measured values obtained on the specimens by calculating drift factors as follows:

10.2.1 Arrange the data obtained on the control reference material in chronological order (for example, $C_1, C_2, C_3, \cdots C_n$.

10.2.2 Compute the drift factors (F) as follows:

$$F_1 = (C_1 + C_2)/2C_1 \quad (11)$$

$$F_2 = (C_2 + C_3)/2C_1 \quad (12)$$

$$F_3 = (C_4 + C_5)/2C_1 \quad (13)$$

10.2.3 Divide the measured values by the *appropriate* drift factor to obtain corrected values, for example, observed value/F = corrected value (see example X1.3.5.1 and Table X1.2).

10.2.4 Enter drift-corrected values in Table 1.

10.3 Other valid drift correction methods may be used in this procedure.

11. Precision and Bias

11.1 Precision data for this practice will be developed in the future if they are determined necessary.

12. Keywords

12.1 homogeneity; optical emission spectroscopy; reference materials; X-ray emission spectroscopy

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES TO ILLUSTRATE THIS PROCEDURE

X1.1 Specimen Selection

X1.1.1 Suppose the material to be tested contains 50 specimens. Each of the 50 specimens are labeled consecutively. For ease of presentation in this example, only six specimens from the set of 50 will be considered instead of 15. Six specimens are selected from the set of 50 using a table of random numbers (see Table 2). From the table, select an arbitrary starting point and read in any direction using only numbers less than 50. For example, look across the top row of Table 2 to number 71, then down to number 22. From this starting point, reading down that column the numbers would be 22, 33, 47, 25, 12, and 10. The specimens having these numbers would be used for testing.

X1.2 Test Procedure A (Drift Monitor)

X1.2.1 The six specimens are measured in random order. The measurements are repeated five times, taking care to select the specimens in random order each time. Suppose a drift monitor is desired, then a control reference material (C) is measured with the specimens. Selecting a control frequency of three, for example, the complete measurement scheme can be represented as follows (reading across each row):

Run No.	Sample No. →
↓ 1	C_1, 22, 33, 47, C_2, 25, 10, 12, C_3
2	C_4, 25, 47, 10, C_5, 33, 12, 22, C_6
3	C_7, 10, 22, 12, C_8, 47, 33, 25, C_9
4	C_{10}, 12, 25, 22, C_{11}, 33, 47, 10, C_{12}
5	C_{13}, 47, 33, 25, C_{14}, 10, 12, 22, C_{15}
6	C_{16}, 33, 12, 47, C_{17}, 25, 22, 10, C_{18}

where C_1, C_2, $C_3 \cdots C_{18}$ are the control reference material run in chronological order.

X1.3 Test for Instrumental Drift

X1.3.1 The data from the control reference material are arranged in chronological order and Δ, and d, are calculated as tabulated in Table X1.1.

X1.3.2 Calculate: $S_1^2 = \Sigma\Delta^2/(n - 1) = 15.53/(18 - 1) = 0.914$

X1.3.3 Calculate: $S_2^2 = \Sigma d^2/(n - 1) = 17.920/(18 - 1) = 1.0541$

X1.3.4 Calculate: $S_1^2/S_2^2 = 0.914/1.0541 = 0.867$

X1.3.5 Since the ratio 0.867 is smaller than the ratio for n = 18 from Table 4 (for example, interpolated value = 1.26), there is sufficient evidence at the 95 % confidence level to indicate that drift has indeed occurred.

TABLE X1.1 Tabulation of Control, Δ, and *d* Values

Control	Δ	Δ^2	d	d^2
C_1 = 62.0	0.600	0.360	1.0333	1.0678
C_2 = 61.4	0.600	0.360	1.6333	2.6678
C_3 = 62.0	1.200	1.44	1.0333	1.0678
C_4 = 63.2	1.800	3.24	0.1666	0.0278
C_5 = 61.4	0.600	0.360	1.6333	2.6678
C_6 = 62.0	1.200	1.44	1.0333	1.0678
C_7 = 63.2	0.600	0.360	0.1666	0.0278
C_8 = 62.6	1.300	1.69	0.4333	0.1878
C_9 = 63.9	1.300	1.69	0.8666	0.7511
C_{10} = 62.6	0.00	0.00	0.4333	0.1878
C_{11} = 62.6	0.600	0.360	0.4333	0.1878
C_{12} = 63.2	1.300	1.69	0.1666	0.0278
C_{13} = 64.5	1.300	1.69	1.4666	2.1511
C_{14} = 63.2	0.700	0.490	0.1666	0.0278
C_{15} = 63.9	0.00	0.00	0.8666	0.7511
C_{16} = 63.9	0.600	0.360	0.8666	0.7511
C_{17} = 64.5	0.00	0.00	1.4666	2.1511
C_{18} = 64.5	...	...	1.4666	2.1511

Average = 63.0333; $\Sigma\Delta^2$ = 15.53;Σ d^2 = 17.920

X1.3.5.1 *Calculation of Drift Factors*—The sum of $C_1 + C_2$ is computed and divided by $2C_1$, etc., as shown below: F

$F_1 = C_1 + C_2/2C_1 = 0.9952$
$F_2 = C_2 + C_3/2C_1 = 0.9952$
$F_3 = C_4 + C_5/2C_1 = 1.0048$
$F_4 = C_5 + C_6/2C_1 = 0.9952$
$F_5 = C_7 + C_8/2C_1 = 1.0145$
$F_6 = C_8 + C_9/2C_1 = 1.0202$
$F_7 = C_{10} + C_{11}/2C_1 = 1.0097$
$F_8 = C_{11} + C_{12}/2C_1 = 1.0145$
$F_9 = C_{13} + C_{14}/2C_1 = 1.0298$
$F_{10} = C_{14} + C_{15}/2C_1 = 1.0250$
$F_{11} = C_{16} + C_{17}/2C_1 = 1.0355$
$F_{12} = C_{17} + C_{18}/2C_1 = 1.0403$

X1.3.5.2 The observed measurements are divided by the appropriate drift factors calculated in X1.3.5.1 to obtain corrected measurements as shown in Table X1.2.

TABLE X1.2 Drift—Corrected Measurements

Run No.	Specimen	Observed Measurement	Drift Factor	Corrected Measurements
1	22	48.8	0.9952	49.0
	33	48.7	0.9952	48.9
	47	50.9	0.9952	51.1
	25	49.7	0.9952	49.9
	10	48.8	0.9952	49.0
	12	48.7	0.9952	48.9
2	25	50.2	1.0048	49.9
	47	49.1	1.0048	48.9
	10	49.3	1.0048	49.0
	33	49.8	0.9952	50.0
	12	48.7	0.9952	48.9
	22	49.7	0.9952	49.9
3	10	49.7	1.0145	48.9
	22	50.8	1.0145	50.0
	12	50.9	1.0145	50.2
	47	51.0	1.0202	49.9
	33	51.2	1.0202	50.2
	25	52.1	1.0202	51.1
4	12	49.5	1.0097	49.0
	25	49.4	1.0097	48.9
	22	51.6	1.0097	51.1
	33	50.7	1.0145	49.9
	47	50.6	1.0145	49.9
	10	49.8	1.0145	49.0
5	47	52.6	1.0298	51.1
	33	52.5	1.0298	50.9
	25	52.4	1.0298	50.9
	10	51.3	1.0250	50.0
	12	51.2	1.0250	49.9
	22	53.1	1.0250	51.8
6	33	50.7	1.0355	48.9
	12	53.8	1.0355	51.9
	47	54.9	1.0355	53.0
	25	53.1	1.0403	51.0
	22	53.0	1.0403	50.9
	10	55.0	1.0403	52.9

X1.3.5.3 The corrected measurements are entered in Table X1.3.

NOTE X1.1—To facilitate the calculations in this example, the corrected values are rounded to the nearest whole number.

X1.4 Test for Homogeneity Utilizing Procedure A

X1.4.1 Compute T, B, t', and G.

X1.4.1.1 *Example:* See Table X1.3.

X1.4.2 Choose α, that is, the significance level of this test.

NOTE X1.2—A 95 % significance level is recommended for this procedure.

X1.4.2.1 *Example:* $\alpha = 0.05$ (that is, 95 % confidence level)

X1.4.3 Calculate ν, the degrees of freedom where b = the number of runs, and t = the number of specimens.

X1.4.3.1 *Example:* $\nu = (b - 1)(t - 1) = (6 - 1)(6 - 1) = 25$

X1.4.4 The appropriate q value is obtained from Table 3.

X1.4.4.1 *Example:* Look across Column 6 down to 24 (that is, the closest value to 25) and interpolate for the value for $\nu = 25$ which is 4.36.

X1.4.5 Compute S_t, where: $S_t = [(T_1^2 + T_2^2 + \cdots T_t^2)/b] - G^2/tb$.

X1.4.5.1 *Example:*

$$S_t = [(303^2 + 299^2 + 304^2 + 302^2 + 299^2 + 299^2)/6] - 1806^2/6 \times 6 = 4.333$$

X1.4.6 Compute S_b, where: $S_b = [(B_1^2 + B_2^2 + \cdots B_b^2)/t] - G^2/tb$.

X1.4.6.1 *Example:*

$$S_b = [(297^2 + 297^2 + 300^2 + 298^2 + 305^2 + 309^2)/6] - 1806^2/36 = 20.333$$

X1.4.7 Compute $\bar{S}$, where $\bar{S}$ = sum of squares of all the measured values minus G^2/tb.

X1.4.7.1 *Example*:

$$\bar{S} = [49^2 + 49^2 + 51^2 + 50^2 + 49^2 + 49^2 + 50^2 + 50^2 + 49^2 + 50^2 + 49^2 + 49^2 + 50^2 + 50^2 + 50^2 + 51^2 + 50^2 + 49^2 + 51^2 + 50^2 + 50^2 + 49^2 + 49^2 + 49^2 + 52^2 + 51^2 + 51^2 + 51^2 + 50^2 + 50^2 + 51^2 + 49^2 + 53^2 + 51^2 + 52^2 + 53^2] - 1806^2/36 = 45.000$$

X1.4.8 Compute s, where:

$$s = \sqrt{(\bar{S} - S_b - S_t)/(b - 1)(t - 1)}$$

X1.4.8.1 *Example*:

$$s = \sqrt{(45.000 - 20.333 - 4.333)/5 \times 5} = 0.90186$$

X1.4.9

TABLE X1.3 Example for Homogeneity Testing (Procedure A)

Run No.	Specimen Number						Total
	22	33	47	25	10	12	
1	49	49	51	50	49	49	$B_1 = 297$
2	50	50	49	50	49	49	$B_2 = 297$
3	50	50	50	51	50	49	$B_3 = 300$
4	51	50	50	49	49	49	$B_4 = 298$
5	52	51	51	51	50	50	$B_5 = 305$
6	51	49	53	51	53	52	$B_6 = 309$
Total	$T_1 = 303$	$T_2 = 299$	$T_3 = 304$	$T_4 = 302$	$T_5 = 299$	$T_6 = 299$	$G = 1806.0$
Mean	$t'_1 = 50.5$	$t'_2 = 49.8$	$t'_3 = 50.7$	$t'_4 = 50.3$	$t'_5 = 49.8$	$t'_6 = 49.8$	

t = number of specimens
b = number of runs

Compute w, where: $w = qs/\sqrt{b}$

X1.4.9.1 *Example*:

$$q = 4.36, s = 0.90186, \text{ and } b = 6$$

$$|mCw = (4.36)(0.90186)/2.449 = 1.605$$

X1.4.10 The maximum difference between any of the mean t' values in Table X1.3 (Procedure A) equals 0.87. Since 0.87 does *not* exceed the w computed value of 1.605, there is evidence at the 95 % confidence level that the specimens are homogeneous.

X1.4.11 An evaluation of the overall quality of the data in Table X1.3 (Procedure A) can be made by computing the relative standard deviation (RSD) as follows: $RSD = s/t' \times 100$ where s is computed in X1.4.8. For the above example, $RSD = 0.90186/50.16 \times 100 = 1.80$ %.

X1.5 Test Procedure B (No Drift Monitor)

X1.5.1 A measurement is made on each of the six specimens. The measurements are repeated five times, taking care to choose the specimens in random order each time. For example, the complete measurement scheme can be represented as follows (reading across row):

Run No.	Sample No. →
↓ 1	22, 33, 47, 25, 10, 12
2	25, 47, 10, 33, 12, 22
3	10, 22, 12, 47, 33, 25
4	12, 25, 22, 33, 47, 10
5	47, 33, 25, 10, 12, 22
6	33, 12, 47, 25, 22, 10

X1.5.1.1 The measurements for each element are entered in Table X1.4.

X1.6 Test for Homogeneity Utilizing Procedure B

X1.6.1 Compute T, B, t', and G.

X1.6.1.1 *Example:* See Table X1.4.

X1.6.2 Choose α, that is, significance level of this test.

NOTE X1.3—95 % confidence level is recommended for this procedure.

X1.6.2.1 *Example*:

$$\alpha = 0.05 \text{ (that is, 95 \% confidence level)}$$

X1.6.3 Calculate ν, the degrees of freedom, where b = the number of runs, and t = the number of specimens.

X1.6.3.1 *Example*:

$$\nu = (b - 1)(t - 1) = (6 - 1)(6 - 1) = 25$$

X1.6.4 The appropriate q value is obtained from Table 3.

X1.6.4.1 *Example:* Look across Column 6 down to 24 (that is, the closest value to 25) and interpolate for the value for $\nu = 25$ which is 4.36.

X1.6.5 Compute S_t, where: $S_t = [(T_1^2 + T_2^2 + \cdots T_t^2)/b] - G^2/tb$

X1.6.5.1 *Example*:

$$S_t = [(8.615^2 + 8.685^2 + 8.719^2 + 8.798^2 + 8.699^2 + 8.691^2)/6] - 52.207^2/36 = 0.00291$$

X1.6.6 Compute S_b, where: $S_b = [(B_1^2 + B_2^2 + \cdots B_6^2)/t] - G^2/tb$

X1.6.6.1 *Example*:

$$S_b = [(8.82^2 + 8.722^2 + 8.706^2 + 8.673^2 + 8.781^2 + 8.505^2)/6] - 52.207^2/36 = 0.01004$$

X1.6.7 Compute $\bar{S}$, where: $\bar{S}$ = *sum of squares of all the measured values minus* G^2/tb.

X1.6.7.1 *Example*:

$$\begin{aligned}\bar{S} = [&1.47^2 + 1.461^2 + 1.502^2 + 1.482^2 + 1.447^2 \\ &+ 1.458^2 + 1.417^2 + 1.445^2 + 1.391^2 \\ &+ 1.480^2 + 1.486^2 + 1.503^2 + 1.426^2 \\ &+ 1.468^2 + 1.431^2 + 1.508^2 + 1.470^2 \\ &+ 1.403^2 + 1.428^2 + 1.485^2 + 1.445^2 \\ &+ 1.438^2 + 1.440^2 + 1.437^2 + 1.469^2 \\ &+ 1.446^2 + 1.491^2 + 1.459^2 + 1.457^2 \\ &+ 1.459^2 + 1.405^2 + 1.380^2 + 1.459^2 \\ &+ 1.431^2 + 1.399^2 + 1.431^2] \\ &- 52.207^2/36 \\ &= 0.03589\end{aligned}$$

X1.6.8 Compute s, where:

$$s = \sqrt{(\bar{S} - S_b - S_t)/(b - 1)(t - 1)}$$

X1.6.8.1 *Example*:

$$s = \sqrt{(0.035899 - 0.01004 - 0.00291)/25} = 0.03029$$

X1.6.9 Compute w, where: $w = qs/\sqrt{b}$

X1.6.9.1 *Example*:

$$q = 4.36, s = 0.030298, b = 6$$

$$|mCw = (4.36)(0.030298)/2.449 = 0.0539$$

X1.6.10 The maximum difference between any of the mean t' values in Table X1.4 (Procedure B) equals 0.0305. Since 0.0305 does *not* exceed the w computed value of 0.0539, there

TABLE X1.4 Data for Homogeneity Testing (Procedure B)

Run No.	Specimen Number						Total
	22	33	47	25	10	12	
1	1.470	1.461	1.502	1.482	1.447	1.458	$B_1 = 8.820$
2	1.417	1.445	1.391	1.480	1.486	1.503	$B_2 = 8.722$
3	1.426	1.468	1.431	1.508	1.470	1.403	$B_3 = 8.706$
4	1.428	1.485	1.445	1.438	1.440	1.437	$B_4 = 8.673$
5	1.469	1.446	1.491	1.459	1.457	1.459	$B_5 = 8.781$
6	1.405	1.380	1.459	1.431	1.399	1.431	$B_6 = 8.505$
Total	$T_1 = 8.615$	$T_2 = 8.685$	$T_3 = 8.719$	$T_4 = 8.798$	$T_5 = 8.699$	$T_6 = 8.691$	$G = 52.207$ ($\Sigma B_1 ... B_6$)
Mean	$t'_1 = 1.436$	$t'_2 = 1.448$	$t'_3 = 1.453$	$t'_4 = 1.466$	$t'_5 = 1.450$	$t'_6 = 1.449$	

t = number of specimens.
b = number of runs.

is evidence at the 95 % confidence level that the test specimens are homogeneous.

X1.6.11 An evaluation of the overall quality of the data in Table X1.4 (Procedure B) can be made by computing the relative standard deviation (RSD) as follows:

$$RSD = 0.03029/t'_{mean} \times 100$$

where s is computed in 10.1.4. For the above example,

$$RSD = 0.03029/1.450 \times 100 = 2.09\ \%.$$

X2. ADDITIONAL INFORMATION CONCERNING USE OF TABLE 2 IN STATISTICAL ANALYSIS OF DATA

X2.1 It is often desirable in some solid specimens to examine the homogeneity from front to back, corner-to-corner, etc. to find compositional differences caused by metallurgical structure or segregation. This statistical procedure allows one to obtain such information. For example, to determine if there is a significant difference between the front and back sides of a solid disk specimen, the two sides of the one specimen are treated as though they were two separate specimens. Then the data in Tables X1.3 and X1.4 would reflect the difference between the sides of the specimen. To ensure that the sides of the specimen are taken at random for measurement, the following sequence is recommended:

	Specimen	
↓Side	A	B
a	1	3
b	4	2

That is, for two specimens *A* and *B*, each having sides *a* and *b*, the recommended sequence is *aA, bB, aB, bA*.

X2.2 The recommended sequences for three to six specimens are given in Table X2.1.

TABLE X2.1 Recommended Sequences for Three to Six Specimens

Side	Specimens					
	A	B	C	D	E	F
a	1	5	4	...	...	...
b	6	2	3	...	...	...
a	1	7	6	4	...	...
b	8	2	3	5	...	...
a	1	8	9	5	4	...
b	10	3	2	6	7	...
a	1	11	3	9	8	7
b	12	2	10	4	5	6

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