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Standard Guide for Maintaining Test Methods in the User's Laboratory[1]

This standard is issued under the fixed designation D 4697; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide is intended to assist laboratories in maintaining precision and controlling bias in testing. It includes statistical procedures for detecting lack of control, changes in calibration constants, and in operator technique. Suggestions are given for the correction of some out of control situations.

1.2 This guide includes the following topics:

1.3 This standard does not give all of the details for preparing statistical quality control charts or the statistical tests used to interpret them, but such information may be found easily in a number of publications.[2]

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[3]

D 1578 Test Method for Breaking Strength of Yarn in Skein Form[3]

E 456 Terminology Relating to Quality and Statistics[4]

2.2 ASTM Adjuncts:

TEX-PAC[5]

NOTE 1—Tex-Pac is a group of PC programs on floppy disks, available through ASTM Headquarters, 100 Barr Harbor Drive, Conshohocken, PA 19428, USA. The control chart parameters described in the annexes of this guide can be calculated with one of these programs and the control charts screen plotted.

3. Terminology

3.1 *Definitions:*

3.1.1 *accuracy, n—of a test method*, the degree of agreement between the true value of the property being tested (or an accepted standard value) and the average of many observations made according to the test method, preferably by many observers. (See also *bias* and *precision*.)

3.1.2 *attribute data, n*—observed values or determinations which indicate the presence or absence of specific characteristics.

3.1.3 *bias, n—in statistics*, a constant or systematic error in test results.

3.1.3.1 *Discussion*—Bias can exist between the true value and a test result obtained from one method; between test results from two methods; or between two test results obtained from a single method, for example, between operators or between laboratories.

3.1.4 *calibrate, vt*—to determine and record the relationship between a set of standard units of measure and the output of an instrument or test procedure.

3.1.5 *calibration, n*—the act or process of calibrating; the recorded relationship resulting from calibrating.

3.1.6 *measurement value, n*—the numerical result of quantifying a particular property or dimension. (*Syn.* measurement, measurement datum.)

3.1.7 *moving range, MR, n*—the difference without regard to sign between two successive observations.

3.1.8 *precision, n*—the degree of agreement within a set of observations or test results obtained as directed in a test method.

3.1.8.1 *Discussion*—The term "precision" delimited in various ways is used to describe different aspects of precision. This usage was chosen in preference to the use of "repeatability" and "reproducibility" which have been assigned conflicting meanings by various authors and standardizing bodies.

3.1.9 *test method, n*—a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result.

3.1.10 *verification, n*—the act or process of verifying.

3.1.11 *verify, vt*—(*1*) to determine whether a previously calibrated instrument, standard solution, or other standard is

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[1] This guide 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] For information see: Juran, J. M., ed., *Quality Control Handbook*, McGraw Hill, New York, 4th ed., 1988; *Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D*, ASTM, 1976; and *Statistical Quality Control Handbook*, Western Electric Co., Inc., 2nd ed., 1985 (Inquiries may be made to: AT& T Technologies, Commercial Sales Clerk, Select Code 700-444, P.O. Box 19901, Indianapolis, IN 44219).

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

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

[5] PC programs on floppy disks are available through ASTM. For a 3½ inch disk request PCN:12-429040-18, for a 5¼ inch disk request PCN:12-429041-18.

still properly calibrated; (*2*) to establish that an operation has been completed correctly.

3.1.12 For definitions of textile terms in this standard, refer to Terminology D 123. For definitions of statistical terms in this standard, refer to Terminology D 123 or Terminology E 456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *maintain*—to monitor the usage of test methods and to take remedial steps when necessary.

4. Significance and Use

4.1 Following this guide will aid the user in maintaining control of both the bias and the precision of any test method.

4.1.1 It is necessary to control bias so that, if a change in level of an unknown material under test occurs, the user can be confident that the change was not due to the execution of the test method.

4.1.2 It is also necessary to control precision so that the established confidence limits and critical differences will be maintained.

4.2 Should the use of the test method be out of control, this guide will detect it, and may give an indication of where the problem lies.

5. Calibration Control

5.1 *Calibration Procedure*—To be completely useful, the test method must contain a calibration procedure if it makes use of a reagent, apparatus, or standard that is subject to change with age or use. If it does not have a calibration procedure, then one should be obtained or written and incorporated in the method.

5.2 *Calibration Records*—Establish for each instrument and each test method the following calibration records:

5.2.1 Establish a schedule for calibration and verification of calibration. The choice of frequency depends on the cost of verification and the consequences of failing to detect a shift in calibration. Always verify at least once every six months.

5.2.2 Keep a log book of calibrations and verifications of calibrations. Include the date when the check was made, the results of the check, what adjustments were made, if any, and who did the checking and the work.

5.2.3 Attach a permanent tag to the calibrated equipment, standard, or reagent. Put on the tag the date of calibration or verification, the results, and the name of the person performing the work.

5.3 *Quality Control Chart*—Establish and maintain a statistical quality control chart for examining the results of calibration verification of each piece of equipment. An example is given in Annex A1. If the control chart indicates a change in the equipment, recalibrate it.

5.3.1 When the test results are measurement data, use the method of moving ranges to estimate the standard deviation of average verification results. When the test results are attribute data, use the standard methods for such data (see STP 15D).[2]

5.3.2 In the case of calibration control, it is seldom necessary to plot a control chart for the range of specimens. This aspect of test method maintenance will usually be well controlled when the directions in 6.1, 6.2, and 6.5 are followed.

6. Test Performance Control

6.1 *Standard Sample*—Reserve an adequate supply of a stable standard sample of material for maintaining test performance (Note 2). Each day, before making a test on unknown laboratory samples, have each analyst run an analysis on the standard sample. Have the analyst use the machine he will use later to test the unknown samples.

NOTE 2—It is essential to obtain a new supply of a standard material well before the old supply is exhausted, and to run the old supply and the new supply in parallel for a short length of time.

6.1.1 If a supply of a stable standard sample cannot be obtained, there are techniques available for overcoming this problem. For example, two analysts could analyze a specimen from the same sample at the same time, and the difference between the two results could be plotted. For problems of this kind, competent statistical help is required in the planning of the program, the presentation, and analysis of the data.

6.2 *Quality Control Chart*—Using the standard sample test data, establish statistical quality control charts for the test results produced by each analyst-instrument pair. If there is more than one analyst-instrument pair, plot a separate average and range chart for each pair, using the same control lines (for an example, see Annex A2).

6.3 *Average Center Line*—Use the established expected test value of the standard sample as the center line on the chart for averages. This value may have been obtained with a standard material procured from an outside source, or it may have been the average of a series of test results from various analyst-instrument pairs over a suitable length of time.

6.4 *Control Limits for Averages*—When the test results are measurement data, use the method of moving ranges to estimate the standard deviation of successive test results. When the test results are attribute data, use the standard methods for such data (see STP 15D[2]). Base the control limits for averages on this standard deviation.

6.4.1 If there is more than one analyst-instrument pair, calculate the average moving range for all pairs, using moving ranges within each pair, and excluding moving ranges spanning more than five days (for an example, see A2.1).

6.5 *Range of Specimen Test Results*—For measurement data, obtain the center line and limits for the specimen range control chart by using the average range of specimens within each reported test result by the various analyst-instrument pairs. For attribute data, do not prepare range charts.

6.6 *Control Chart Follow-up*—If a control chart indicates a lack of control, investigate and take corrective action immediately. For instructions on detecting instability in the testing process or changes in level, or interpreting patterns on control charts, see Juran, pages 24-15 through 24-18.[2]

ANNEXES

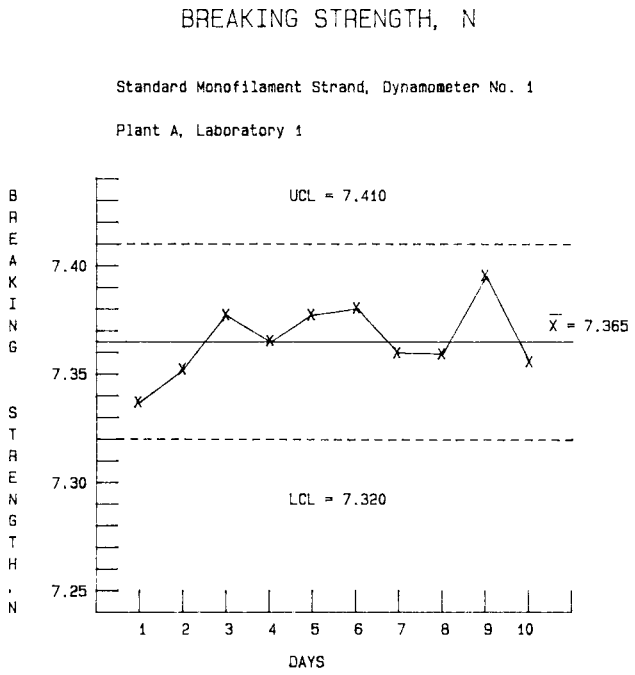
(Mandatory Information)

A1. CALIBRATION CONTROL

A1.1 *Example*—A standard monofilament strand was tested once each day, making two breaks by the same operator on a dynamometer to verify the calibration of the machine. The resulting data are shown in Fig. A1.1, and in Table A1.1 which also shows the details of some of the calculations for control chart methods as described in A1.1.3.

A1.1.1 Control charts were prepared for these data as directed in 5.3.

A1.1.2 The manufacturer certified that the average strength of the monofilament is 7.365 N. This value is used as the center line, *T*, on the calibration control chart for averages of two breaks in Fig. A1.1.



Note—Certified Average = 7.365 N
3s = 0.045 based on average MR = 0.017
Points represent average of 2 breaks
Limits based on days 1-10

FIG. A1.1 Calibration Control

TABLE A1.1 Calibration Control for Dynamometer

Day	Breaking Strength, N			Range
	Specimen	Average	Moving Range	
1	7.335			
	7.345	7.340	...	0.010
2	7.365			
	7.345	7.345	0.005	0.020
3	7.384			
	7.375	7.380	0.035	0.009
4	7.394			
	7.345	7.370	0.010	0.049
5	7.404			
	7.355	7.380	0.010	0.049
6	7.394			
	7.375	7.384	0.004	0.019
7	7.384			
	7.345	7.365	0.019	0.039
8	7.355			
	7.375	7.365	0.000	0.020
9	7.404			
	7.394	7.399	0.034	0.010
10	7.345			
	7.375	7.360	0.039	0.03
Avg.			0.017	0.026

A1.1.3 Control limits for averages were calculated as follows:

$$T \pm E_2\overline{MR} \quad \text{(A1.1)}$$

where:

T = average value of standard,
E_2 = 2.660 (from STP 15D), and
MR = average moving range.

A1.1.3.1 When $T = 7.365$ and $\overline{MR} = 0.017$, the control limits are 7.320 and 7.410 N. These values are used on the control chart in Fig. A1.1.

A1.1.4 The control charts in Fig. A1.1 give no indication of lack of control. Therefore, draw the conclusion that currently the dynamometer needs no adjustment.

A2. TEST PERFORMANCE CONTROL

A2.1 *Example*—A cone of 30s single polyester yarn was reserved as a standard material for use in controlling the measuring of break factor, using Test Method D 1578. Each day before making any break factor tests, each analyst made one break factor determination on each dynamometer he was to use that day. The resulting data are shown in Fig. A2.1 and Table A2.1 which also shows some of the calculations described in A2.1.3. In order to save space, Fig. A2.1 is shown in four sections. Each section and its continuation should be plotted on four separate graphs.

A2.1.1 Control charts were prepared as directed in 6.2.

A2.1.2 Extensive testing of the standard material has shown its average break factor to be 3788, a dimensionless value. This value is used as the center line, *T*, on the control chart for break factor in Fig. A2.1.

A2.1.3 Control limits for averages were calculated using Eq A1.1. In the present case, the average moving range of the averages is 688/13 = 52.92.

A2.1.4 These figures, using Eq A1.1, produce control limits of 3629 and 3947 for break factor.

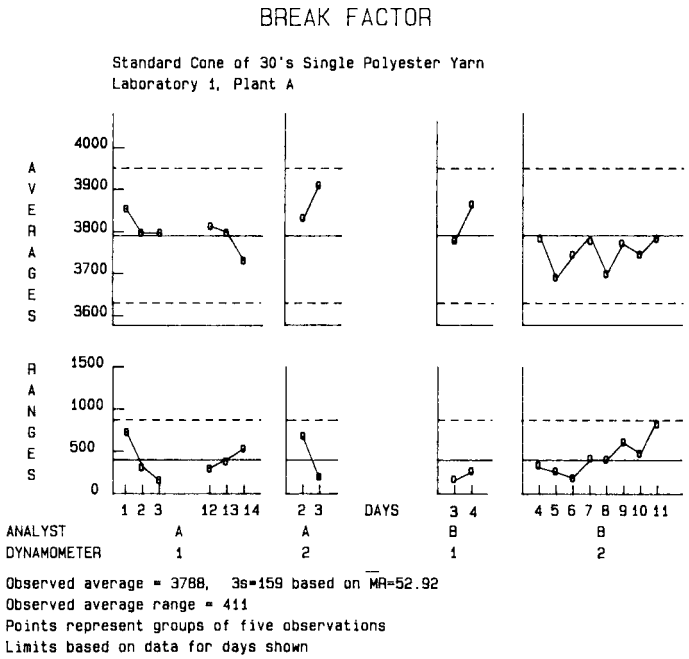


FIG. A2.1 Control Charts

A2.1.5 Control limits for ranges of specimen test results were calculated as follows:

$$LCL = 0 \quad \text{(A2.1)}$$

$$UCL = D_4\bar{R} \quad \text{(A2.2)}$$

where

LCL = lower control limit for range of five,
UCL = upper control limit for range of five,
D_4 = 2.115 (from STP 15D), and
$\bar{R}$ = average range of five determinations.

A2.1.5.1 When $\bar{R} = 7397/18 = 410.94$, $LCL = 0$ and $UCL = 869$.

A2.1.6 These charts indicate that the control of the use of this test method is in need of close attention. While the data are not conclusive, the average results obtained by operator B when using machine 2 may very well be too low.

TABLE A2.1 Performance Control for Test Method D 1578

Day	Specimen	Break Factor Average Factor	Moving Range of Avg.	Range of Specimens
		Analyst A Machine 1		
1	3789	...	...	...
	4219	...	...	...
	3909	...	...	...
	3814	...	...	...
	3497	3846	...	722
2	3926	...	...	...
	3660	...	...	...
	3788	...	...	...
	3636	...	...	...
	3947	3791	55	311
3	D[A]	3796	5	171
12	D	3816	...	367
13	D	3791	25	402
14	D	3731	60	511
		Analyst A Machine 2		
2	D	3838	...	697
3	D	3916	78	200
		Analyst B Machine 1		
3	D	3781	...	173
4	D	3863	82	267
		Analyst B Machine 2		
4	D	3779	...	361
5	D	3688	91	286
6	D	3744	56	191
7	D	3779	35	416
8	D	3703	76	404
9	D	3776	73	608
10	D	3754	22	470
11	D	3784	30	840
Total		68176	688	7397

[A] D indicates single breaks omitted to save space.