Designation: D 5591 – 02

Standard Test Method for Thermal Shrinkage Force of Yarn and Cord With a Thermal Shrinkage Force Tester[1]

This standard is issued under the fixed designation D 5591; 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 test method covers preparation and procedures to measure the thermal shrinkage force of yarns and cords in air.

1.2 This test method is applicable to measurement of the thermal shrinkage force of yarns and cords whose shrinkage force at 180 ± 2°C (355 ± 4°F) in air does not exceed 20 N (4 lbf). This test method is applicable to nylon, polyester, and aramid yarns and cords within the applicable range of thermal shrinkage force, as well as to comparable yarns and cords from other polymers.

1.2.1 Test specimens may be taken from yarn or cord packages, or retrieved from fabrics.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

D 885 Methods for Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns Made from Manufactured Organic-Base Fibers[2]

D 2258 Practice for Sampling Yarn for Testing[2]

D 6477 Terminology Relating to Tire Cord and Fabrics[3]

3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms relating to tire cord and fabrics, refer to Terminology D 6477.

3.2 For definitions of textile terms used in this test method, refer to Terminology D 123.

4. Summary of Test Method

4.1 A specified length of yarn or cord is conditioned in a relaxed state, mounted with a pretension of 5 ± 1 mN/tex (0.05 ± 0.01 gf/den), then exposed to dry heat at a temperature of 180 ± 2°C (355 ± 4°F) for 120 ± 5 s.

4.2 The shrinkage force induced in the specimen is read from the tester.

5. Significance and Use

5.1 This test method may be used for the acceptance testing of commercial shipments of yarns and cords.

5.1.1 If there are differences of practical significance between reported test results for two laboratories (or more), comparative tests should be performed to determine if there is a statistical bias between them, using competent statistical assistance. As a minimum, test samples should be used that are as homogeneous as possible, that are drawn from the material from which the disparate test results were obtained, and that are randomly assigned in equal numbers to each laboratory for testing. Other materials with established test values may be used for this purpose. The test results from the two laboratories should be compared using a statistical test for unpaired data, at a probability level chosen prior to the testing series. If a bias is found, either its cause must be found and corrected, or future test results for that material must be adjusted in consideration of the known bias.

5.2 Experience shows that yarns or cords on would packages, usually being under tension, exhibit a contraction in length (and a resulting increase in linear density) when removed from the package and allowed to relax over a period of time at room temperature. Consequently, it they are tested without being allowed to relax, they will register higher thermal shrinkage force values as the relaxation shrinkage will be incorrectly included as the thermal shrinkage force.

5.2.1 Retractive forces vary widely by polymer type, being almost nil within aramids and significant within most nylons. For example, the exposure of untensioned skeins of nylon yarn or cord to 95 to 100 % relative humidity at room temperature for two days and reconditioning under standard laboratory conditions will cause most of the length change that is possible at room temperature to occur within a sample. This reduction in length is accompanied by some lowering of thermal shrinkage force.

[1] This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.19 on Tire Cord and Fabrics.

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

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

5.3 The thermal shrinkage force of nylon, polyester, and aramid fiber is related to the polymer of origin and its manipulation in processing. Thermal shrinkage force measurement can be used to control product uniformity.

5.4 The level of thermal shrinkage force is critical in the user's subsequent operations, such as the drum-set (original length of cord) required to build a tire of a particular size.

5.5 The thermal shrinkage force is critical to the final shape and size of fiber-reinforced articles. For example, thermal shrinkage force affects the final size of V-belts and their ability to maintain tension during their operation.

5.6 This test method is in agreement with the nominal procedures of Methods D 885 for the determination of thermal shrinkage force in yarns and cords.

5.6.1 Shrinkage force is measured while the specimen is within an oven at a specified temperature and after a specified length of time.

6. Interferences

6.1 If the chamber in which the specimen is heated is open on three sides, air drafts can effectively shorten the length of specimen experiencing the prescribed temperature environment. The results obtained without a shield are generally lower than those obtained with a shield.

6.2 The accurate control of temperature at any prescribed setting is of utmost importance. Differences between the set point temperature and the temperature experienced by the specimen are a major cause of the bias of test results. The temperature that the specimen experiences may be checked by attaching a small calibrated thermocouple to a piece of cord and suspending it in the specimen position such that the tip of the thermocouple is in the center of the oven cavity. The thermocouple must not touch the oven walls. Either correct any set point/sample temperature bias or determine the proper set point to give the specified specimen temperature. An intralaboratory comparison is the preferred method to determine whether a bias exists.

6.3 The differences in the amount of pre-relaxation of yarns can cause differences in thermal shrinkage force, as noted in 5.2.1.

6.4 Shrinkage force is a combination of pretension force and the force that is developed in the specimen as a result of the specimen being heated.

6.5 Shrinkage force can be affected by the pretension, the length of specimen exposure, improper location of the specimen within the oven, and oven-surface contact of any part of the specimen. Specimens that are spun, textured, or crimped (such as those removed from a fabric) may allow filaments to come into contact with interior surfaces of the thermal shrinkage force oven. Such physical contact will cause inaccurate readings of the thermal shrinkage force.

7. Apparatus

7.1 *Thermal Shrinkage Oven*, consisting of a specimen heating cavity capable of heating up to 250°C (480°F), a means of accurately controlling the temperature of the cavity ± 2°C (4°F), and a means for measuring and displaying the shrinkage force up to 0.1 N (0.02 lbf).

7.2 *Stopwatch or Time*, capable of reading to ± 1.0 s.

7.3 *Clip-On Tensioning Masses.*

7.4 *Draft Shield for Shrinkage Oven*, if the oven does not have one provided.

8. Hazards

8.1 Do not touch the oven while it is in operation because it can reach temperatures up to 200°C (390°F).

8.2 Do not leave the oven unattended if a specimen is installed.

9. Sampling

9.1 *Lot Sample*—As a lot sample for acceptance testing, randomly select the number of shipping containers directed in an applicable material specification or other agreement between the purchaser and the supplier. In the absence of such an agreement or material specification, proceed as directed in Practice D 2258. Consider shipping containers of yarn, cord and rolls of fabric to be the lot sampling units.

NOTE 1—An adequate specification or other agreement between the purchaser and the supplier requires taking into account the variability between shipping containers, between laboratory sampling units within a shipping container, and between test specimens within a laboratory sampling unit to produce a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

9.2 *Laboratory Sample*—As a laboratory sample for acceptance testing, proceed as follows:

9.2.1 For yarn or cord, take at random the number of packages per shipping container in the lot sample as directed in an applicable material specification or other agreement between the purchaser and the supplier. In the absence of such an agreement or material specification, proceed as directed in Practice D 2258.

9.2.2 For fabric, take a full-width swatch at least 1-m (1-yd) long from the outside of each roll of fabric in the lot sample, after first discarding all fabric from the outside of the rolls that contains creases, fold marks, disturbed weave, or contamination by foreign material.

9.3 *Test Specimens*:

9.3.1 For yarns and cords, strip at least 50 m (55 yd) from the outside of each package in the laboratory sample. Inspect the outside of the package after stripping off the yarn. If there is visible damage, continue to strip off units of 50 m (55 yd) and reinspect until there is no visible damage. Take at least three specimens, 600-mm (24-in.) long, from each package in the laboratory sample. Discard and replace specimen lengths that are visibly damaged.

9.3.2 For tire cord fabrics, remove a minimum of five lengths of warp yarn or cord 600-mm (24-in.) long from each swatch in the laboratory sample, with the specimens being taken at least 75 mm (3 in.) from the selvage of the swatch. For fabrics other than tire cord fabric, such as square-woven fabrics, also take from each swatch in the laboratory sample a minimum of five lengths of filling yarn or cord 600-mm (24-in.) long after discarding those portions within 75 mm (3 in.) of the selvage of the swatch.

9.3.2.1 The instructions on number of test specimens given in 9.3.2 assume that the mean value of three thermal shrinkage force results will characterize adequately the thermal shrinkage force of the laboratory sample from which the specimens were taken. The extra two specimens from fabric are taken to ensure that a specimen free of handling damage is available after conditioning. If the applicable material specification or other agreement between the purchaser and the supplier specifies testing more than three specimens per laboratory sample, an additional two specimens above the number specified should be taken from the laboratory sample and conditioned.

9.4 Exercise caution that the specimens do not change twist in handling.

10. Preparation of Apparatus

10.1 Preheat the oven 45 min prior to testing with the draft shield covering the three open sides of the heating chamber.

10.2 Test in the standard atmosphere for testing industrial yarns (see 3.1).

10.3 Adjust the oven temperature controller set point to 180°C (355°F).

11. Conditioning

11.1 Condition unrestrained specimens or segments of untensioned fabric in the atmosphere for testing industrial yarns (see 3.1). Ensure that no change in twist occurs while conducting this procedure.

11.1.1 Condition and relax the yarn and greige cord specimens 12 to 28 h.

11.1.2 Condition and relax the adhesive-treated cord samples 16 to 28 h, unless immediate testing (5 to 20 min after processing) is agreed upon between the purchaser and the supplier. Immediate testing must be reported as an exception to this test method (see Section 13).

12. Procedure

12.1 For yarns or cords, use a pretension load of 5 ± 1 mN/tex (0.05 ± 0.01 gf/den).

12.2 Pull the specimen transport carriage assembly forward against the front stops.

12.3 Insert one end of the specimen through the open right hand clamp and guide the end through to the opposite clamp atop the load cell post.

12.4 Zero the load cell.

12.5 Close the right hand clamp, firmly securing the right hand end of the specimen.

12.6 Apply the prescribed pretensioning mass to the free end of the specimen (see Table 1) outside the left hand post.

12.7 Close the left hand clamp, securing the specimen atop the load cell post.

NOTE 2—Take care that during the closing of the clamp on top of the load cell, the reading stays on zero.

12.8 Remove the pretensioning mass.

12.9 Push the carriage assembly back into the oven. Ensure that the specimen is centered in the oven and that no part of it is in contact with oven surfaces.

12.10 Start the timer at the moment the carriage assembly is in the oven, if the apparatus does not have an automatic start feature.

12.11 At the end of 120 ± 5 s, read the maximum shrinkage force on the instrument scale to the nearest 0.1 N (0.02 lbf).

TABLE 1 Tensioning Masses[A,B]

Dtex	Single Strand Yarns Denier	Tensioning Mass, g
235	210	12
940	840	47
1100	1000	55
1170	1050	60
1400	1260	70
1440	1300	72
1880	1680	94
2100	1890	105
Multiple Strands or Cords of Multiple Strands		
Construction, Dtex	Construction, Denier	Tensioning Mass, g
940 × 2	840 × 2	94
1100 × 2	1000 × 2	110
1400 × 2	1260 × 2	140
1440 × 2	1300 × 2	144
1880 × 2	1680 × 2	188
2100 × 2	1890 × 2	210

[A] For yarns or cords not shown in Table 1, calculate clip-on tensioning mass (g) required by multiplying the total dtex of the specimen by 0.05 or total denier by 0.055 g.

[B] Specified tensioning masses are for the nominal dtex specified. The denier column is for information only.

12.12 Pull the carriage to the front and discard the specimen.

13. Report

13.1 State that the specimens were tested as directed in D 5591. Describe the material(s) or product(s) tested and the method of sampling used. Report the following information:

13.1.1 Individual thermal shrinkage force results as read from the indicator dial to the nearest tenth of a unit. Exercise caution that the final shrinkage force is reported. Some computerized data printout options available may show the average force for a defined time interval.

13.1.2 Pretension force used.

13.1.3 Measurements as "immediate testing" if not conditioned for the standard period (see 11.1.1 and 11.1.2).

13.1.4 Measurements as "package testing" if the specimens taken from packages are not relaxed prior to testing.

14. Precision and Bias

14.1 *Interlaboratory Test Data*—An interlaboratory test was run in 1995 in which randomly drawn samples of three materials, 1260/2 denier nylon cord, 1000/1 denier polyester yarn, and 1500/2 denier polyester cord, were tested in each of five laboratories. Two operators in each laboratory tested three specimens of each material on each of two days. For each material, the components of variance for thermal shrinkage force expressed as variances were calculated and are listed in Table 2.

TABLE 2 Components of Variance (Variances)

Material	Single Operator	Within Laboratory	Between Laboratory
1260/2 Denier Nylon Cord	0.0133	...	1.3547
1000/1 Denier Polyester Yarn	0.0018	0.0004	0.0415
1500/2 Denier Polyester Cord	0.0047	0.0057	0.0264

14.2 *Critical Differences*—For each material two averages should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed in Table 3.

TABLE 3 Critical Differences for Two Averages, 95 % Probability Level, Newtons

Number of Test Results in Each Average	Single Operator Precision	Within Laboratory Precision	Between Laboratory Precision
	1260/Denier Nylon Cord		
1	0.32	0.32	3.24
2	0.23	0.23	3.23
3	0.18	0.18	3.23
4	0.16	0.16	3.23
5	0.14	0.14	3.23
	1000/1 Denier Polyester Yarn		
1	0.12	0.13	0.58
2	0.08	0.10	0.57
3	0.07	0.09	0.57
4	0.06	0.08	0.57
5	0.05	0.08	0.57
	1500/2 Denier Polyester Yarn		
1	0.19	0.28	0.53
2	0.13	0.25	0.51
3	0.11	0.24	0.51
4	0.09	0.23	0.51
5	0.08	0.22	0.50

14.3 *Bias*—The procedure in this test method for measuring thermal shrinkage force has no bias because the value of this property can be defined only in terms of a test method.

15. Keywords

15.1 thermal shrinkage force; tire cords; yarn