



## Standard Test Method for Thermal Shrinkage Force of Yarn and Cord With the Testrite Thermal Shrinkage Force Tester<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers preparation and procedures for use of the Testrite Shrinkage-Force Tester 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  $177 \pm 2^\circ\text{C}$  in air does not exceed 20 N. 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 Yarns or cords for testing 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<sup>2</sup>

D 885 Methods for Testing Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns Made from Manufactured Organic-Base Fibers<sup>2</sup>

D 2258 Practice for Sampling Yarn for Testing<sup>2</sup>

D 2906 Practice for Statements on Precision and Bias for Textiles<sup>2</sup>

### 3. Terminology

3.1 *Definitions:*—For definitions of textile terms used in this test method, refer to Terminology D 123.

#### 3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adhesive-treated tire cord, n*—a tire cord whose adhe-

sion to rubber or other elastomer has been improved by the application of a dip followed by rapid drying and (normally) additional heat treatment.

3.2.2 *atmosphere for testing textiles, n*—for tire cords and industrial yarns, air maintained at a relative humidity of  $55 \pm 2\%$  and at a temperature of  $24 \pm 1^\circ\text{C}$  ( $75 \pm 2^\circ\text{F}$ ).

3.2.3 *cord, n*—a twisted or formed structure composed of one or more single or plied filaments, strands, or yarns of organic polymer or inorganic materials.

3.2.3.1 *Discussion—*for the manufacture of pneumatic tires or other industrial fabrics, the direction of twist used to combine single or plied yarn elements into a cord construction is in a direction opposite to that used in the yarns. Tire and other reinforcing cords frequently consist of a single yarn strand having little or no twist. These cords, as well as single monofilaments, are used synonymously with twisted and plied cords in this test method.

3.2.4 *greige cord, , n*—in tire cords, a cord that has not received adhesive treatment, heat treatment, or other finishing treatment.

3.2.5 *greige tire cord, n*—a tire cord that has not been dip treated or heat treated before use (see *tire cord*).

3.2.6 *pneumatic tire, n*—a hollow tire that becomes load-bearing upon inflation with air, or other gas, to a pressure above atmosphere.

3.2.7 *retraction, n*—in yarns and cords, the reduction in length when previous restraint is removed and relaxation is allowed, thus causing a directionally proportional increase in linear density.

3.2.8 *thermal shrinkage force, n*—that force induced when a restrained material is restricted from shrinking upon exposure to heat.

3.2.9 *thermal shrinkage force tester, n*—an apparatus that measures the force achieved when a yarn or similar specimen, held at constant (fixed) length, is subjected to a temperature above that at which the specimen was mounted in the apparatus.

3.2.10 *tire, n*—a load-bearing, ground-contacting circumferential attachment to a vehicle wheel.

3.2.11 *yarn, n*—a generic term for a continuous strand of textile fibers, filaments, or material in a form suitable for knitting, weaving, or otherwise intertwining to form a textile fabric.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 07.01.

## 4. Summary of Test Method

4.1 A specified length of yarn or cord is relaxed, conditioned, and pretensioned with a specific mass to induce a specified pretension force in the yarn or cord.

4.2 A conditioned specimen is exposed for  $120 \pm 5$  s to dry heat at a temperature of  $177 \pm 2^\circ\text{C}$ .

4.3 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, but caution is advised since information on between-laboratory precision is incomplete.

5.1.1 In case of a dispute arising from differences in reported test results when using this test method for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine whether there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens that are as homogeneous as possible and that are from a lot of material of the type in question. The test specimens should then be assigned randomly in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using the Student's *t*-test for unpaired data and an acceptable probability level chosen by the two parties before the testing is begun. If a bias is found, either its cause must be found and corrected or the purchaser and the supplier must agree to interpret future test results in view of the known bias.

5.1.2 Yarn and cord may contract in length over a period of time due to room temperature retraction. Thermal shrinkage force values are reduced proportionately by the amount of room temperature retraction.

5.1.3 Experience shows that retractive forces are present in most wound packages. This may be observed directly as a shortening of length (or indirectly as an increase in linear density) in unrestrained yarn or cord that is not relaxed fully. After retraction, such relaxed yarns exhibit lower thermal shrinkage force values.

5.1.4 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.

5.2 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.

5.3 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.

5.4 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.

5.5 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.<sup>3</sup>

5.5.1 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 Because 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 one of the manufacturer's strongest claims. Nevertheless, for a number of reasons, it is possible for a difference to exist from tester to tester in the actual temperature developed in the specimen. An intralaboratory comparison is the preferred method to determine whether a bias exists between the results from one tester to another. If a bias is found, its cause may be related to any number of factors. To ensure that either the temperature induced in the specimen is the same between instruments, or the temperature on the specimen matches the set-point temperature of a given instrument, or both, use the procedures given in the Annex to calibrate Mark III Testrite ovens.

6.3 The differences in the amount of pre-relaxation of yarns can cause differences in thermal shrinkage force, as noted in 5.1.2.

6.4 Shrinkage force as measured by the Testrite Shrinkage Force Tester 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 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 *Testrite Thermal Shrinkage Force Tester Mark III and Mark V*,<sup>4</sup> software version 5.12 and later (Fig. 1).<sup>3</sup>

7.2 *Stopwatch or Time*, capable of reading to  $\pm 1.0$  s.<sup>5</sup>

<sup>3</sup> Besides the apparatus specified in Section 7, there is a Mark IV and Mark V with software version 5.01 that do not fit the requirements of this test method. These models eliminate the pretension force via electronic circuitry. Thermal shrinkage force readings that include pretension differ from readings that do not include pretension.

<sup>4</sup> "Testrite" Thermal Shrinkage Force Tester and clip-on tensioning masses are available from Testrite, Ltd. Woodfield Works, Old Lane, Halifax, England, HX3 6TF.

<sup>5</sup> Stopwatch manufactured by Galco, Jules Racine & Co., Dept 2, 85 Executive Boulevard, Elmsford, NY 10523, has been found to be satisfactory for this purpose.



### 9.3 Test Specimens:

9.3.1 For yarns and cords, strip at least 15 m (16 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 15 m and reinspect until there is no visible damage. Take one specimen, 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 fabrics, remove a minimum of three 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 three 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. Take warp specimens that are free of filling material and filling specimens that are free of warp material in all cases.

9.3.2.1 The instructions on number of test specimens given in 9.3.2 assume that a single valid thermal shrinkage force result 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 (see Section 11). If the applicable material specification or other agreement between the purchaser and the supplier specifies testing more than one specimen per laboratory sample, an additional two specimens above the number to be tested 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 3.2.2).

10.3 Adjust the oven temperature controller set point to 177°C.

10.4 Consider use of the procedure described in the Annex if the calibration of the oven temperature is suspected to be in error.

## 11. Conditioning

11.1 Condition unrestrained specimens or segments of un-tensioned fabric in the atmosphere for testing industrial yarns (see 3.2.2). 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 buyer and the seller. Immediate testing must be reported as an exception to this test method (see Section 13).

## 12. Procedure

12.1 Raise the draft shield.

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

12.3 Lower the draft shield.

12.4 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.

12.5 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.

12.8 Remove the pretensioning mass.

12.9 Raise the draft shield.

12.10 Push the carriage assembly to the rear of the tester against the back stops. Ensure that the specimen is centered in the oven and that no part of it is in contact with oven surfaces or will be in contact with the draft shield after it is lowered.

12.11 Lower the draft shield.

12.12 Start the timer at the moment that the draft shield is lowered if the apparatus does not have an automatic start feature.

12.13 At the end of  $120 \pm 5$  s, read the maximum shrinkage force on the instrument scale to the nearest 0.1 N.

12.14 Raise the draft shield, pull the carriage to the front, lower the draft shield, and discard the specimen.

## 13. Report

13.1 State that the specimens were tested as directed in this test method. 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

**TABLE 1 Tensioning Masses<sup>A,B</sup>**

dtex	Single Strand Yarns	
	Denier	Tensioning Mass, g
235	210	12
940	840	48
1100	1000	56
1170	1050	60
1400	1260	71
1440	1300	73
1880	1680	96
2100	1890	107
Multiple Strands or Cords of Multiple Strands		
Construction	Tensioning Mass, g	
940 × 2	840 × 2	96
1100 × 2	1000 × 2	112
1400 × 2	1260 × 2	143
1440 × 2	1300 × 2	147
1880 × 2	1680 × 2	192
2100 × 2	1890 × 2	214

<sup>A</sup>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.

<sup>B</sup>Specified tensioning masses are for the nominal dtex specified. The Denier column is for information only.

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 are not removed from the package and relaxed prior to testing.

#### 14. Precision and Bias

14.1 *Summary*—The following precision and bias statements have been prepared in accordance with Practice D 2906. In comparing two single observations (single operator precision) for the materials tested, the difference should not exceed the values shown in Table 2 in 95 out of 100 cases when both observations are taken by the same well trained operator using the same piece of test equipment and specimens randomly drawn from the same sample of material. Larger differences are likely to occur under all other circumstances. The true value of thermal shrinkage force can be defined only in terms of a specific test method. Within this limitation, this test method has no known bias. Paragraphs 14.2-14.4 explain the basis for this summary and for evaluations made under other conditions.

14.2 *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 testrite thermal shrinkage force expressed as variances were calculated and are listed in Table 3.

14.3 *Critical Differences*—For each material and for the components of variance listed in Table 3, two averages should be considered significantly different at the 95 % probability

**TABLE 2 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

**TABLE 3 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

level if the difference equals or exceeds the critical differences listed in Table 2.

14.4 *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

### ANNEX

#### (Mandatory Information)

#### A1. CALIBRATION OF OVEN TEMPERATURE ON TESTRITE TESTER

##### A1.1 Objective

A1.1.1 The objective is to operate at a true temperature level, as measured by a thin wire thermocouple tied to a threadline of 177°C at the center of the oven.

##### A1.2 Apparatus

A1.2.1 *Type “J” Certified Wire Thermocouple and Certified Indicator Device*, 12-in. long, 1/16-in. diameter.

A1.2.2 *Hook Mass*, approximately 10 N.

A1.2.3 *Strand of Yarn or Cord*, 65 to 75-cm long.

##### A1.3 Procedure

A1.3.1 Tie the wire thermocouple (probe) to a yarn or cord specimen to be inserted into the oven. Tie the probe near the tip and at one or two other locations to prevent the thermocouple from touching the oven.

A1.3.2 Attach one end of the yarn strand and the thermocouple wire in the Testrite specimen clamp so that the yarn and probe are held firmly. Adjust the yarn and probe assembly so that the tip of the probe will be in the center of the oven.

A1.3.3 Bring the other end of the yarn or cord specimen through the other (open) clamp and attach approximately 10 N mass to the end of the strand to hold the specimen/probe assembly taut in the oven.

A1.3.4 Raise the oven cover and push the carriage assembly toward the oven so that the specimen/probe assembly comes to rest at the normal testing position.

A1.3.5 Check to ensure that the threadline and wire thermocouple probe are positioned correctly in the normal threadline testing path. The thermocouple probe should be collinear with the thread path used during testing. The probe should also be centered between the oven platens. The probe must not

touch either platen. Close the cover during this process.

A1.3.6 Allow the above system to reach thermal equilibrium (approximately 5 min).

A1.3.7 Record and compare the temperature indicated on the Testrite digital readout and temperature measured by the certified wire thermocouple device. These temperatures must agree within 2.0°C.

A1.3.8 If necessary, the controller may be calibrated so that the Testrite controller indication agrees with the certified wire calibration check probe. This procedure is described in A1.4.

#### **A1.4 Procedure for Re-Calibration of Controller for Mark III and Earlier Testrite Models (Use a Qualified Electrician)**

A1.4.1 If the Testrite controller is found to be miscalibrated as determined by the procedure described above, proceed as follows. Switch the machine off (tag, lock, and so forth), and then pull the flap open on the bottom front of the controller.

A1.4.2 Release the controller from the Testrite unit by turning the screw fastener on the right side of the controller. The controller slides out of the Testrite as a complete unit from front of the tester.

A1.4.3 On the bottom side of the controller are three holes. Located through the two holes closest to the front of the controller are potentiometers; the SPAN (closest to the front of the tester) and the ZERO (middle opening). The SPAN should be used to adjust temperatures above 150°C. By turning the SPAN pot clockwise or counterclockwise, the temperature in the oven will fall or rise accordingly for a given setpoint. Only the slightest movement is necessary.

A1.4.4 Replace the controller. Switch the instrument on. Leave for 10 to 15 min until the set temperature is indicated by the controller. Recheck the calibration by the threadline method and readjust as necessary until the controller indication agrees with the certified device.

#### **A1.5 Procedure for Re-Calibration of Controller for Mark IV and Later Testrite Models**

A1.5.1 The procedure for re-calibration of the Mark IV and V Testrite can be supplied upon request from Testrite Ltd, Halifax, England, HX36TF, according to a letter to D13.19 Subcommittee in 1992. The user should contact Testrite Ltd. directly for this information as needed.

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