



Designation: D 4974 – 042

# Standard Test Method for Hot Air Thermal Shrinkage of Yarn and Cord Using a Thermal Shrinkage Oven<sup>1</sup>

This standard is issued under the fixed designation D 4974; 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.

<sup>1</sup> 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. Current edition approved Sept. 10, 2004<sup>2</sup>. Published ~~December 2004~~, November 2002. Originally published as D4974-99. Last previous edition D4974-9901.

## 1. Scope

- 1.1 This test method covers the measurement of shrinkage of yarns and cords when exposed in a thermal shrinkage oven.
- 1.2 This test method is applicable to yarns and cords made of nylon, polyester, and other polymers not detrimentally affected by the temperature used and with linear densities in the range from 20 to 700 tex (180 to 6300 denier).
  - 1.2.1 Yarns or cords for testing may be taken from yarn or cord packages or from fabrics.
- 1.3 This test method shows values in both SI and inch-pound units. SI is the technically correct name for the system of units known as the International System of Units. Inch-pound units is the technically correct name for the customary units used in the United States. The values stated in either acceptable metric units or other units shall be regarded separately as standard. The values expressed in each system may not be exact equivalents; therefore, each system must be used independently of each other, without combining values in any way. Referee decisions are to use SI units.
- 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 hazard 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 of Testing Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns Made from Manufactured Organic-Base Fibers<sup>2</sup>
  - D 1776 Practice afor Conditioning Textiles for Testing<sup>2</sup>
  - D 6477 Conditioning Textiles for Testing<sup>3</sup>

## 3. Terminology

- 3.1 ~~Definitions of Terms Specific to This Standard:~~
  - 3.1.1 ~~greige cord, Definitions:  $n$ —in tire cords, a cord that has been adhesive-treated, heat-treated, or otherwise treated before use (see cord).~~
  - 3.1.2 ~~standard atmosphere for testing textiles,  $n$ —laboratory conditions for testing fibers, yarns, and fabrics in which air temperature and relative humidity are maintained at specific levels with established tolerances:~~
    - 3.1.2.1 ~~Discussion—for tire cord and yarns, a relative humidity~~
  - 3.2 ~~For definitions of  $55 \pm 2\%$  and at a temperature of  $24 \pm 1^\circ\text{C}$  ( $75 \pm 2^\circ\text{F}$ ).~~
  - 3.1.3 ~~thermal shrinkage,  $n$ —of textile yarns and cords, contraction in length caused by heat.~~
  - 3.1.4 ~~tire cord,  $n$ —a twisted or formed structure composed of two or more single or plied industrial yarn elements having the same nominal twist, direction of twist, length, and tension:~~
    - 3.1.4.1 ~~Discussion—The direction of twist used terms relating to—combine the single or plied yarn elements into a cord construction is in the direction opposite to that used in the yarns. Frequently, tire cord, bead wire, hose wire, and other reinforcing cords consist of a single yarn strand having little or no twist. These cords are used synonymously with twisted and plied cords in this test method.~~

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

<sup>3</sup> Commercial thermal shrinkage ovens are available. Clip-on masses also are available from the manufacturer.

<sup>3</sup> Annual Book of ASTM Standards, Vol 07.02.

~~3.1.5 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 tire cord fabrics, refer to form a textile fabric.~~

~~3.2 Definitions—For the Terminology D 6477.~~

~~3.3 For definitions of other textile terms used in this test method, related to textiles, refer to Terminology D 123.~~

#### **4. Summary of Test Method**

4.1 A relaxed, conditioned specimen of yarn or cord is subjected to dry heat for a specified time while under a specified tension. The percent shrinkage is read directly from a scale or display on the instrument while the specimen is still under tension and exposed to heat.

#### **5. Significance and Use**

5.1 This test method may be used for the acceptance testing of commercial shipments of yarns and cords. Caution is advised because yarn and cord may contract in length over a period of time due to room temperature retraction. Thermal shrinkage values are reduced proportionately by the amount of room temperature retraction.

NOTE 1—Experience, especially with nylon, shows that yarn retraction, which may be observed directly as shortening of length (or indirectly as denier increase), will occur in unrestrained yarn or cord that is not at equilibrium (equilibrium in this case being defined as essentially zero thermal shrinkage yarn or fully relaxed yarn). Normally, retractive forces are present in most wound packages of yarn and cord; thus, unrestrained yarn near the surface is likely, with time, to undergo some retraction. After retraction, such yarns exhibit lower thermal shrinkage values than yarn or cord deeper within the package. The opposite condition of yarn on the surface exists with yarn or cord wound against or near a rigid package core, such as a metal or hardwood wind-up spool. Such core yarn or cord cannot move against this restraint, and thus, will exhibit thermal shrinkage values even several weeks later near to those which were measured immediately from the surface of the freshly wound package. Elevated humidity will accelerate retraction of unrestrained yarn, but moisture content in itself will have little influence on thermal shrinkage. Exposure of untensioned skeins of 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 room temperature retraction that is possible within a sample to occur.

5.1.1 In case of differences of practical significance in reported test results from two or more laboratories conduct comparative tests to determine if there is a statistical bias between them. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the 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 then should be assigned randomly in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using Student's *t*-test for unpaired data and a probability level chosen by the parties before the testing is begun. If a bias is found, either its cause must be found and corrected, or future test results must be adjusted in consideration of the known bias.

5.2 Thermal shrinkage of nylon, polyester, and other fibers is related to the polymer of origin and its manipulation in processing. Thermal shrinkage measurement can be used to control product uniformity.

5.3 The level of thermal shrinkage is critical in the user's subsequent operations. For example it can affect the drumset (original length of cord) required in tire building to produce a finished, final tire of a particular size.

5.4 Thermal shrinkage is critical to final shape and size of fiber reinforced articles. For example, thermal shrinkage affects final size of V-belts and their ability to maintain tension while running.

5.5 This test method is similar to the procedures of Methods D 885 for the determination of thermal shrinkage of yarns and cords. Shrinkage is measured while the specimen is within an oven and tensioned as specified in Methods D 885; however, there are enough vagaries among different units of apparatus for measurement of thermal shrinkage that numerical equivalence between units of different design should not be assumed, even under the same nominal conditions.

#### **6. Interferences**

6.1 An effective draft shield on the thermal oven is critical. 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. Results obtained without a shield generally are lower than with a shield.

6.2 The accurate control of temperature at any prescribed setting is of utmost importance. Bias between the set point temperature and the temperature that the specimen sees is a major cause of instrument bias. The temperature that the specimen sees 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.

6.3 The degree of room temperature length retraction, (for example, that which occurs in unrestrained skeins of yarn over time in the testing laboratory), of the laboratory sample or specimen affects the measurement of thermal shrinkage. Unrelaxed nylon, for example, shrinks much more than relaxed nylon. The amount of relaxation (retraction) occurring prior to testing of thermal shrinkage can affect the result.

6.4 Frictional forces against the pulley/indicator assembly cause measurement errors. Ensure that the indicator needle does not rub against the scale. Maintain the pulley bearings in good condition. The pulley wheel must be centered in the bearings. A force of 10 mN or less, applied to the tip of the indicator needle, should cause pulley rotation when the tester is in proper operating condition.

6.5 Thermal shrinkage increases as the exposure time increases. Check the exposure time with a stopwatch. Some thermal oven models have a timer installed. Compare this timer to a stopwatch and calibrate as necessary.

6.6 Center the threadline between the heating plates of the oven to obtain a correct measurement.

6.7 Specimens that are spun, textured, or crimped (as those removed from a fabric) may allow filaments to come in contact with interior surfaces of the thermal shrinkage oven. Such physical contact will cause inaccurate readings of thermal shrinkage.

6.8 Yarn or cords that are sufficiently sticky or tacky to prevent their free release from the pulley surface as it rotates will cause inaccurate readings of thermal shrinkage.

## 7. Apparatus

7.1 *Thermal Shrinkage Oven*<sup>4</sup>, 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  $\pm 2^\circ\text{C}$  ( $\pm 4^\circ\text{F}$ ), and a means for measuring and displaying the amount of specimen shrinkage to the nearest 0.1 %. Fig. 1 shows the principle of operation of commercial thermal shrinkage ovens.

7.2 *Stopwatch or Timer.*

7.3 *Clip-On Masses.*

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

## 8. Safety Hazards

8.1 The oven portion of the tester can reach temperatures over 200°C (390° F). Do not touch the oven.

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

## 9. Sampling and Test Specimens

9.1 *Primary Sampling Unit*—Consider shipping containers of cords or rolls of fabric to be the primary sampling unit, as applicable.

9.2 *Laboratory Sampling Unit*—As a laboratory sampling unit for acceptance testing, take material randomly from the primary sampling unit as follows:

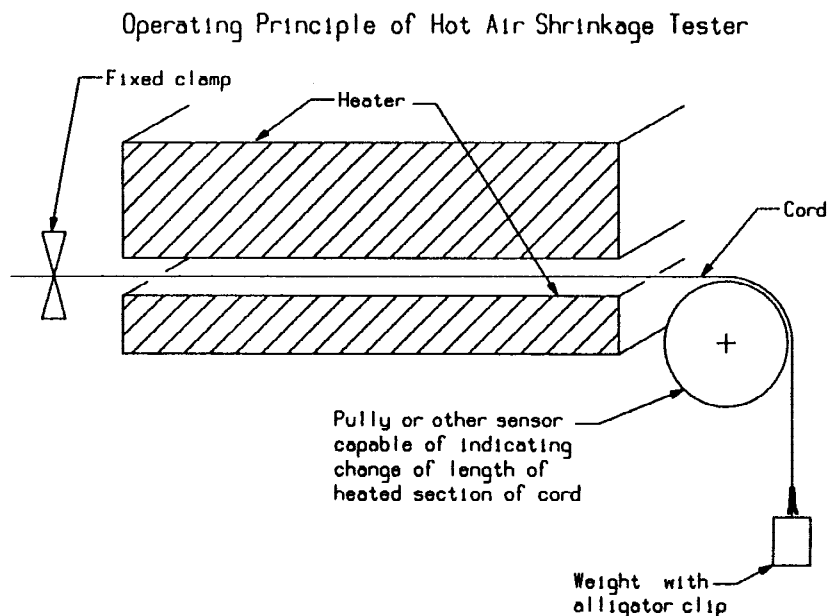
9.2.1 For cords, take spools or packages per carton using the applicable procedure in Practice D 2258.

9.2.2 For fabric, take from rolls at least one full-width piece of fabric that is at least 1 m (1 yd) along the selvage (machine direction), after first discarding all fabric from the outside of the roll 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 15 m (16 yd) from the outside of each package in the laboratory sampling unit. Inspect the outside of the package after stripping off the yarn. If there is visible damage, continue to strip off units of 15 m (16 yd) and reinspect until there is no visible damage. Take one specimen, 600 mm (24 in.) long, from each package in the laboratory sampling unit. Discard and replace specimens that are visibly damaged.

<sup>4</sup> Commercial thermal shrinkage ovens are available. Clip-on masses also are available from the manufacturer.



**FIG. 1 Principle of Operation of a Thermal Shrinkage Oven**

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 sampling unit, 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 tire fabrics, also take from each swatch in the laboratory sampling unit 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. In all cases, take warp specimens that are free of filling material and filling specimens that are free of warp material.

9.3.2.1 Instructions on the number of test specimens in 9.3.2 assume that a single valid thermal shrinkage result will adequately characterize the thermal shrinkage of the laboratory sampling unit from which the specimens are taken. The extra two specimens are taken to assure that a specimen free of handling damage is available after conditioning (see 10.1). If the applicable material specification or other agreement between the purchaser and the supplier specifies testing more than one specimen per laboratory sampling unit, an additional two specimens above the number to be tested should be taken from the laboratory sampling unit and conditioned.

## 10. Conditioning Specimens and Preparation for Testing

### 10.1 Conditioning:

10.1.1 Condition specimens as relaxed skeins or segments of untensioned fabric as directed in Practice D 1776 using the atmosphere specified for industrial yarns (see ~~3.1.2~~, Section 3). Ensure that no change in yarn/cord twist occurs while carrying out this procedure.

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

10.1.3 Condition and relax 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 supplier. Immediate testing must be reported as an exception to this method (see Section 12).

### 10.2 Preparation for Testing:

10.2.1 With the draft shield in place, preheat the oven until the chamber has attained the specified temperature for a minimum of 5 min.

10.2.2 Test in standard atmosphere for testing industrial yarns (see ~~3.1.2~~, Section 3).

10.2.3 Adjust the oven temperature controller set point to give a specimen temperature of  $177 \pm 2^\circ\text{C}$  ( $350 \pm 4^\circ\text{F}$ ). (See 6.2).

10.2.4 One may consult with the instrument vendor if calibration of the oven temperature is suspected to be in error.

## 11. Procedure

### 11.1 Test Conditions (Table 1):

11.1.1 For yarns or cords possessing low levels of shrinkage force, a tension loading of  $1.0 \pm 0.2$  mN/tex ( $0.010 \pm 0.002$  gf/den) may be used. Use of this lower tension load should be noted in the report.

11.2 Install one end of the specimen in the fixed clamp.

11.3 Bring the other end of the specimen over the pulley.

11.4 Set the indicator to zero and hold it on zero while performing the next step.

11.5 Attach a clip-on mass to the unclamped end of the specimen so that twist is not lost. Use a mass that creates a tension load as specified in 11.1. Table 2 shows the total mass needed to effect such tensions for typical yarns and cords.

NOTE 2—If the specimen is likely to untwist in the portion below the pulley, attach a toothpick or the equivalent (approximately 7.5 cm long) to the mass or clamp, perpendicular to the vertical threadline, so it will bear against the instrument case and prevent rotation of the mass.

11.6 Load specimen(s) into the oven. The specimen is centered automatically in the oven.

NOTE 3—In the case of highly-crimped yarns, typically those removed from tightly woven fabric, the initial dimensional change that occurs is usually growth, that is, an immediate elongating of the specimen as the specimen loses crimp and straightens. If the instrument allows, set the indicator to a shrinkage level in 11.4 sufficient to prevent the indicator from going below zero. Record positive shrinkage as the interval between the lowest and highest reading of the indicator during the test of that specimen. Note for the report (Section 12) the percent change in length due to crimp relaxation in the tester.

11.7 Lower the draft shield immediately after introducing the specimen.

11.8 Start the timer at the moment the draft shield is lowered.

11.9 At the end of  $120 \pm 10$  s ( $240 \pm 10$  s for specimens equal to or greater than 400 tex or 3600 denier), read the percent shrinkage as indicated on the instrument output to the nearest 0.1 %.

11.10 Remove and discard the specimen.

**TABLE 1 Test Conditions**

Temperature	Tension Load	Time, s	
		Specimen with Linear Mass up to 400 tex (3600 denier)	Specimen with Linear Mass Greater than 400 tex (3600 denier)
$177 \pm 2^\circ\text{C}$ ( $350 \pm 4^\circ\text{F}$ )	$5.0 \pm 1.0$ mN/tex (or $0.05 \pm 0.01$ gf/den)	$120 \pm 10$	$240 \pm 10$

**TABLE 2 Tensioning Masses<sup>A,B</sup>**

A. Single-Strand Yarns		
<i>d</i> Tex	Denier	Tensioning Mass, g
235	210	10
940	840	42
1100	1000	50
1170	1050	53
1400	1260	63
1440	1300	65
1870	1680	84
2100	1890	95
B. Multiple Strands or Cords of Multiple Strands		
Construction		Tensioning Mass, g
940 × 2	840 × 2	84
1100 × 2	1000 × 2	100
1400 × 2	1260 × 2	126
1440 × 2	1300 × 2	130
1870 × 2	1680 × 2	168
2100 × 2	1890 × 2	189

<sup>A</sup>For yarns or cords not shown in Table 2, calculate clip-on mass required by multiplying total *d* Tex of specimen by 0.50 mN or total denier by 0.05 gf (for specimens expected to have low shrinkage tensions, the factors are 0.090 mN and 0.01 gf, respectively).

<sup>B</sup>If yarns or cords outside the linear density specified in this test method are tested on this type equipment, it may be necessary to use more mass in tensioning light yarns (to ensure sufficient friction to accurately move the pulley) or less mass in tensioning very heavy yarns or cords (to prevent overstressing of the pulley bearings). In such an event, show, in addition to the mass in grams in 12.1.2, the words *modified procedure*, immediately followed in parentheses by the calculated tension per unit of linear density, such as millinewtons per denier or grams per denier. For example, testing of a 10 000-denier cord with 100 g of tensioning mass will be reported in 12.1.2 as “100-g modified procedure (0.01 gf/denier).”

## 12. Report

12.1 State that the specimens have been tested as directed in Test Method D 4974. Describe the material(s) or product(s) tested and the method of sampling used. Report the following information:

12.1.1 Individual thermal shrinkage results as indicated on the instrument output to the nearest tenth of a unit.

12.1.2 Mass, in g, used to apply tension force.

12.1.3 Measurements as “immediate testing” if not conditioned for standard period (see 10.1.2 and 10.1.3).

12.1.4 Measurements as “package testing” if specimens are not removed from package and relaxed prior to testing.

12.1.5 “Percent length increase prior to shrinkage” if such occurred during testing (see Note 3).

## 13. Precision and Bias

13.1 *Interlaboratory Test Data*—An interlaboratory test was conducted in 2000 using commercially available testers from Lawson-Hemphill, Lenzing, and Testrite. Six tire cord materials were included in the study, varying in polymeric structure (nylon and polyester), form (yarn, greige cord, and dipped cord), and linear density. The type of materials chosen and their processing conditions produced an intentional thermal shrinkage among the specimens from less than 1 % to greater than 7 %. Thirteen laboratories participated in the study. Three of the laboratories used two instruments for a total of sixteen instruments. In each laboratory two operators made triplicate shrinkage measurements on each of four test occasions. Variance components were computed for individual thermal shrinkage determinations and are summarized in Table 3.

13.2 *Precision*—Repeatability and reproducibility deal with the variability of test results obtained under specified laboratory conditions. Repeatability concerns the variability between independent test results obtained within a single laboratory in the shortest practical period. Those results are obtained by a single operator with a specific set of test apparatus using test specimens (or test units) taken at random from a single quantity of homogeneous material obtained or prepared for the interlaboratory study (ILS). Reproducibility deals with the variability between single test results obtained in different laboratories, each of which has applied the test method to test specimens (or test units) taken at random from a single quantity of homogeneous material obtained or prepared for the ILS.

Method repeatability is defined as the “maximum difference” that can “reasonably” be expected between two test results obtained on the same material when the test results are obtained in the same laboratory. Repeatability standard deviation is taken to be the square root of the “determination” variance component, and represents within-operator precision. Method reproducibility is defined as the “maximum difference” that can “reasonably” be expected between two test results obtained on the same material when the test results are obtained from different laboratories. The total, or reproducibility, standard deviation, is formed by taking the square root of the sum of intra- and inter-laboratory variance components.

The values in Table 4 show maximum critical differences for single determinations specified averages of determinations for the

**TABLE 3 Interlaboratory Study Variance Components**

Material	Average Shrinkage	Variance Lab	Variance Instrument	Variance Operator	Variance Occasion	Variance Determination	Total Variance	Within-Lab Variance	s (repeatability)	s (within-lab)	s (reproducibility)
Polyester Yarn 1400 dtex	5.2	0.043665	0.015556	-0.00076	0.003951	0.010685	0.073097	0.029432	0.10	0.17	0.27
Nylon Yarn 930 dtex	7.6	0.40272	0.002372	0.001638	0.010809	0.019147	0.436686	0.033966	0.14	0.18	0.66
Nylon Yarn 1880 dtex	6.5	0.167416	0.009564	0.001451	0.004993	0.014463	0.197887	0.030471	0.12	0.17	0.44
Polyester Cord 1400/1/2	0.94	0.004404	0.000046	-0.00011	0.002657	0.002752	0.009749	0.005345	0.05	0.07	0.10
Nylon Cord 930/1/2	7.7	0.580907	-0.00434	0.001816	0.030175	0.016896	0.625454	0.044547	0.13	0.21	0.79
Nylon Cord 1800/1/2	5.4	0.126382	0.006947	0.000544	0.010189	0.018177	0.162239	0.035857	0.13	0.19	0.40



Material	Number of Observations	Single Operator Precision	Within-Laboratory Precision	Between Laboratory Precision
Polyester Yarn 1400 dtex	1	0.287	0.476	0.749
	2	0.203	0.336	0.530
	4	0.143	0.238	0.375
	8	0.101	0.168	0.265
	16	0.072	0.119	0.187
Nylon Yarn 930 dtex	1	0.384	0.511	1.832
	2	0.271	0.361	1.295
	4	0.192	0.255	0.916
	8	0.136	0.181	0.648
	16	0.096	0.128	0.458
Nylon Yarn 1880 dtex	1	0.333	0.484	1.233
	2	0.236	0.342	0.872
	4	0.167	0.242	0.617
	8	0.118	0.171	0.436
	16	0.083	0.121	0.308
Polyester Cord 1400/1/2	1	0.145	0.203	0.274
	2	0.103	0.143	0.194
	4	0.073	0.101	0.137
	8	0.051	0.072	0.097
	16	0.036	0.051	0.068
Nylon Cord 930/1/2	1	0.360	0.585	2.192
	2	0.255	0.414	1.550
	4	0.180	0.293	1.096
	8	0.127	0.207	0.775
	16	0.090	0.146	0.548
Nylon Cord 1880/1/2	1	0.374	0.525	1.116
	2	0.264	0.371	0.789
	4	0.187	0.262	0.558
	8	0.132	0.186	0.395
	16	0.093	0.131	0.279

single operator case (“repeatability”), within-laboratory case, and between-laboratory case (“reproducibility”). Two values or averages of observed values are considered significantly different at the 95 % probability level if the difference between them exceeds the appropriate critical difference in the table.

13.3 *Bias*—The procedure in this test method produces a test value that can be defined only in terms of a test method. There is no independent referee method by which bias may be determined. This test method has no known bias.

## 14. Keywords

### 14.1 shrinkage; thermal; tire cord

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