Designation: D 4776 – 98

Standard Test Method for Adhesion of Tire Cords and Other Reinforcing Cords to Rubber Compounds by H-Test Procedure[1]

This standard is issued under the fixed designation D 4776; 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 the measurement of adhesion of reinforcing cords that are bonded to rubber compounds. This test method is applicable to textile cord structures from both natural and manmade fibers, other than steel. For adhesion testing of steel tire cords, refer to Test Method D 2229.

1.2 This test method is primarily used to evaluate tire cords, using a suitable tire cord adhesive and a suitable rubber compound. This test method is also used to evaluate (*1*) tire cord adhesives, and (*2*) the process of adhesive reaction on the cord using one consistent form of tire cord and one consistent rubber compound. This test method may be used to evaluate cords in industrial hose and belting products and other cord reinforced rubber products.

1.3 This test method is written in SI units. The inch-pound units which are provided in this test method are not necessarily exact equivalents of the SI units. Either system may be used in this test method.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 76 Specification for Tensile Testing Machines for Textiles[2]

D 123 Terminology Relating to Textiles[2]

D 1566 Terminology Relating to Rubber[3]

D 2138 Test Methods for Rubber Property—Adhesion to Textile Cord[4]

D 2229 Test Method for Adhesion Between Steel Tire Cords and Rubber[2]

D 4393 Test Method for Strap Peel Adhesion of Reinforcing Cords or Fabrics to Rubber Compounds[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *adhesion*, *n*—the property denoting the ability of a material to resist delamination or separation into two or more layers.

3.1.2 *adhesion*, *n—in tire fabrics*, the force required to separate a textile material from rubber or other elastomer by a definite prescribed method.

3.1.3 *curing*, *n*—see the preferred term *vulcanization*.

3.1.4 *industrial yarn*, *n*—a yarn composed of continuous filaments, usually of high breaking tenacity, produced with or without twist, and intended for applications in which functional properties are of primary importance; for example, in reinforcing material in elastomeric products (tires, hose, belting), in protective coverings, and in cordage and webbing, etc.

3.1.4.1 *Discussion*—For example, in reinforcing material in elastomeric products (tires, hose, belting), in protective coverings, and in cordage and webbing, etc.

3.1.5 *reinforcing cord*, *n*—a cord made from industrial yarns and used to provide added support to other materials, such as tires, hose, belting, protective coverings, webbings, etc.

3.1.6 *rubber*, *n*—a material that is capable of recovering from large deformations quickly and forcibly, and can be, or already is, modified to a state in which it is essentially insoluble (but can swell) in boiling solvent, such as benzene, methylethyl ketone, and ethanol-toluene azeotrope.

3.1.7 *rubber compound*, *n—as used in the manufacture of rubber articles*, an intimate mixture of elastomer(s) with all the materials necessary for the finished article.

3.1.8 *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.

3.1.8.1 *Discussion*—For the manufacture of pneumatic tires or other industrial fabrics, the direction of twist used 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 and other reinforcing cords 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.

3.1.9 *vulcanization*, *n*—an irreversible process, usually accomplished through the application of heat, during which a rubber compound through a change in its chemical structure (for example, cross-linking) becomes less plastic and more

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[1] This test method is under the jurisdiction of ASTM Committee D-13 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 09.01.

[4] *Discontinued 1990—Replaced by D 4776, D 4777.*

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

resistant to swelling by organic liquids, and elastic properties are conferred, improved, or extended over a greater range of temperature.

3.1.10 For definitions of other textile terms used in this standard, refer to Terminology D 123. For definitions of other terms relating to rubber, refer to Terminology D 1566.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *H-test adhesion—as used in this test method,* the force to extract either end of a textile cord structure that is embedded in a rubber compound under specified conditions.

4. Summary of Test Method

4.1 A cord specimen is sandwiched between two layers of rubber compound test stock in a form resembling an "H," placed in a heated mold, and cured at a specified temperature and pressure. The test specimen sandwich is then cut to create an H-test specimen consisting of a single cord with each end embedded in the center of a tab end of the rubber test block (Fig. 1). The test specimen is placed in the grips of the tensile tester, and then the grips are separated. The maximum force obtained is the H-test adhesion force.

5. Significance and Use

5.1 Test Method D 4776 for the determination of the H-test adhesion of reinforcing cords to rubber compounds may be used for the acceptance testing of commercial shipments of reinforcing cords but caution is advised since information about between-laboratory precision is incomplete. Comparative tests as directed in 5.1.1 may be advisable.

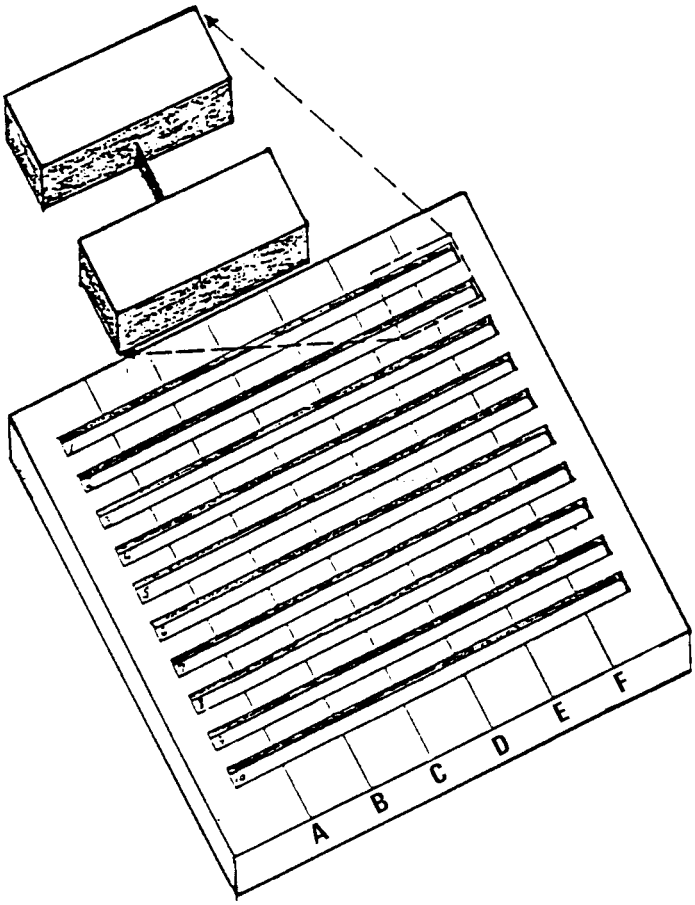


FIG. 1 H-Test Specimen

5.1.1 In cases of dispute arising from the differences in reported test results when using Test Method D 4776 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is 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 which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimens should then be randomly assigned 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 an acceptable probability level chosen by the two parties before testing begins. 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 light of the known bias.

5.2 This test method is used to measure the force required to extract the cord from a rubber compound test block.

5.3 This test method is designed to test the adhesion of textiles that are bonded to rubber compounds. Variables that may contribute to differences in results of this test method include adhesive type, adhesive application procedure, adhesive cure, fiber type, construction of cords, rubber type, rubber cure, and rubber thickness.

5.3.1 The deleterious effect of ozone in combination with atmospheric moisture on the ability of adhesives to bond with rubber requires assiduous protection of cords prior to embedment.

5.4 The expected range of values which characterize acceptable adhesion can be determined in any cord-rubber combination with experience. For this reason, the purchaser normally establishes a minimum level of adhesion to be obtained by the supplier in either the supplier's laboratory or the purchaser's laboratory using either the supplier's standard rubber compound or the purchaser's rubber compound.

5.5 Other procedures for testing adhesion of cords to rubber compounds are available, such as Test Methods D 4393 or D 2138. These procedures have been used extensively in the trade for acceptance testing. The decision on which test procedure to use is determined by agreement between the purchaser and supplier based on historical data and experience of the contractual parties. Results obtained by any of these methods cannot be used interchangeably since there is no overall correlation between them.

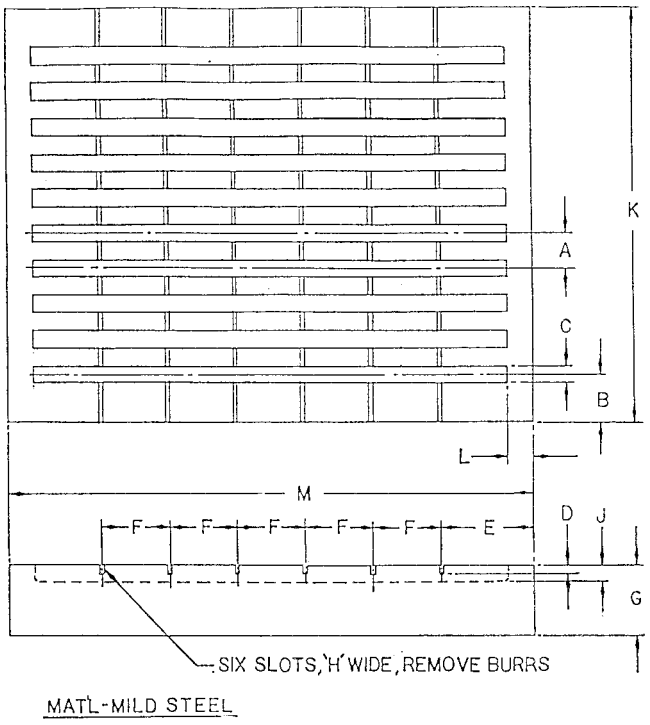
6. Apparatus and Materials

6.1 *Tensile Testing Machine*—Although a constant-rate-of-extension (CRE) tensile testing machine is preferred, a constant-rate-of-traverse (CRT type, pendulum type) may be used. The specification and methods of calibration and verification of these machines shall conform to Specification D 76. The testing machine shall be equipped with an autographic recorder (rectilinear coordinates preferred) or an interface computer. There is a distinct difference between the CRE and CRT type testing machines. Consequently, they cannot be used interchangeably unless a mathematical correlation has been

established and agreed upon by the purchaser and supplier.

6.2 *Curing Press*, capable of exerting a ram force of 135 kN (30 000 lbf), equipped with 300 by 300 mm (12 by 12 in.) platens or larger, and capable of a platen temperature control within ±3°C (±5°F) of the temperature specified for curing the rubber compound.

6.3 *Molds*—The design of the molds shall be as shown in Fig. 2. The dimensions of the test specimen are controlled by the specifications and tolerances of the mold. The dimensions of the mold (6.40 mm [0.250 in.] embedment) in Fig. 2a shall be used for cords with a dtex of 1100/2 or less. The dimensions of the mold (9.52 mm [0.375 in.] embedment) in Fig. 2b shall be used for cords with a dtex larger than 1100/2. The slot size for each mold shall be 1.17 mm ± 0.13 mm (0.46 in. ± 0.005 in.).



NOTE 1—The mold as shown will produce 30 specimens. It may be fabricated to produce a larger or smaller number, but the dimensions that govern the specimen size shall not be altered.

NOTE 2—A mold cover plate must be provided. It should be 9.5 mm (.38 in.) thick and the same outside dimensions ("K" and" L") as the mold.

NOTE 3—Mold dimension tolerances are XX.X mm ± .3 mm (XX.XX in. ± .01 in.) or XX.XX mm ± .13 mm (XX.XXX in. ± .005 in.).

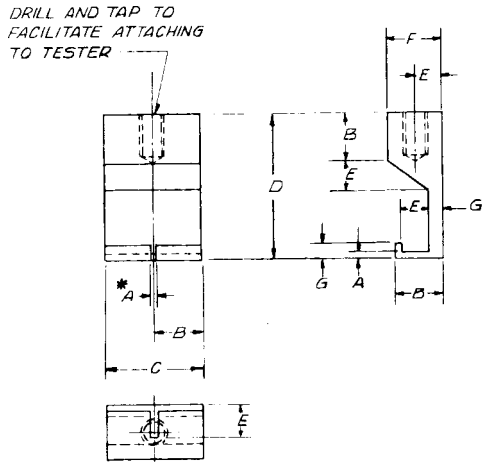
Dimension	2a		2b	
	mm	in.	mm	in.
A	12.70	.500	15.88	.625
B	17.5	.69	17.5	.69
C	6.35	.250	9.52	.375
D	3.17	.125	3.17	.125
E	34.9	1.38	34.9	1.38
F	25.40	1.000	25.40	1.000
G	25.4	1.00	25.4	1.00
H	1.17	1.046	1.17	.046
J	6.40	.250	6.40	.250
K	149.2	5.88	177.8	7.00
L	9.5	.38	9.5	.38
M	196.8	7.75	196.8	7.75

FIG. 2 Mold for H-Test

6.4 *Specimen Grips*—The design of the specimen grips shall be as shown in Fig. 3. Two grips are required.

6.5 *Tensioning Masses*—The masses may be of the hook type, or designed in such a manner that they can be clamped to the cord. In any event, the total mass shall be 50 g.

6.6 *Sheeted Rubber Compound*, (sometimes called skim stock), supported on a non hygroscopic backing, such as a plasticizer-free plastic material. Polyethylene film has been found to be satisfactory. The thickness of the rubber stock required to fill the mold properly shall be determined by experience. As a guide, the thickness of the rubber stock should be not less than 8 % greater than one-half of the mold cavity depth. In any event, the thickness is agreed upon between the purchaser and supplier.



NOTE 1—The important dimension is shown by an asterisk and shall not be altered. All other dimensions are included as guides and may be altered if desired.

Dimension	mm	in.
A	1.6	.06
B	12.5	.50
C	25.0	1.00
D	40.0	1.50
E	7.0	.28
F	14.0	.56
G	4.0	.16

FIG. 3 Specimen Grips for H-Test

6.6.1 Rubber stock properties are best maintained by storage in a cool, dry atmosphere. Excessive rubber stock moisture may lower adhesion of some fiber/rubber composites. Storage at 25 ± 2°C (77 ± 4°F) and 30 to 50 % relative humidity has been found satisfactory.

6.6.2 Rubber compounds exhibit wide variations in shelf life (properties suitable for good adhesion results) dependent upon both composition and storage condition. Rubber compounds are usually replaced after three months; however, some may require replacement within a few weeks. In any event, storage conditions and shelf life should be specified by the supplier of the rubber compound.

6.7 *Timers*, having 60 min capacity, ½ min intervals.

6.8 *Marking Pen*, silver ink for rubber.[6]

6.9 *Shears*, for trimming.

6.10 *Gloves*, of temperature resistant material.

6.11 *Mold Release*,[7] for ease of specimen release from mold.

7. Sampling

7.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of primary sampling units directed in an applicable material specification or other agreement between the purchaser and the supplier. Consider shipping cases of cord to be the primary sampling units. Exercise caution in sampling and handling so that samples receive minimum exposure to ambient atmosphere and light prior to rubber embedment. This can be accomplished by storing the sample in a sealed, black plastic bag. Rayon is particularly sensitive to moisture pick-up (which negatively affects adhesion) and should be handled accordingly.

NOTE 1—A realistic specification or other agreement between the purchaser and the supplier requires taking into account the variability between primary shipping units and within primary shipping units so as to provide a sampling plan with meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

7.2 *Laboratory Sample*—Take at random the number of packages per shipping case in the lot sample directed in an applicable material specification or other agreement between the purchaser and the supplier.

7.3 *Test Specimens*—Prepare 15 specimens, or other number as agreed upon between the purchaser and the supplier, from each sample as directed in Section 8.

8. Preparation of Test Specimens

8.1 Cut the rubber stock in strips 6 mm (0.25 in.) wide and 152 mm (6.0 in.) long, leaving the protective cloth attached. This may be done with shears or with a clicker die.

8.1.1 Some laboratories have fabricated larger molds to accommodate a greater number of test specimens. In those laboratories, the length of the rubber stock must be adjusted accordingly.

8.2 A mold preheated to the curing temperature is preferred.

NOTE 2—When a preheated mold is used, the specimens must be loaded and placed in the curing press within 3 min. The use of a preheated mold will alter the curing conditions (time and temperature) of the rubber.

8.3 Remove the protective film from the rubber test strips, then place the individual rubber test strips in the mold cavities with the protective film side on top.

8.4 Place the cords in the cord slots by cutting cord test specimens allowing sufficient overhang for masses and tie a knot in both ends. Take care to prevent the loss of cord twist. The portion of the cord that is embedded in rubber must not be touched by the bare hand. Attach a 50 g mass on one knotted end of the test cord. Two cords may be tied together and used to fill adjacent slots; a 100 g mass is used in this case (Fig. 4).

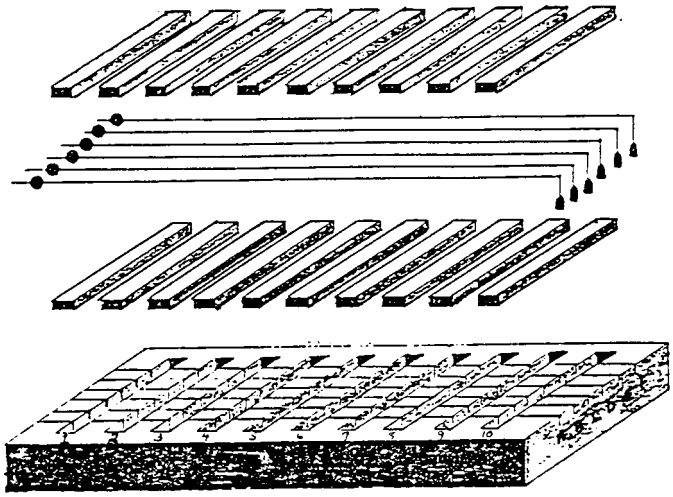


FIG. 4 Mold Loading

Place one end of the knotted test specimen so that it is snubbed firmly against the cord slots on one side of the mold.

8.5 After all the cord slots are filled, remove the protective film from the additional strips of rubber and place them in the mold cavities on top of the cords. The side from which the protective film was removed will be down. Identify each specimen with a silver ink marking pen.

8.6 Cover the mold with a smooth metal plate if the upper press platen is not smooth. Remove the masses and place the mold in a preheated press. Adjust the pressure to a minimum of 3.5 MPa (500 psi) with reference to the mold surface. Vulcanize for the specified time at the specified pressure and temperature. Immediately remove the mold assembly from the hot curing press.

8.7 Remove mold cover, if used. Carefully remove the constructed rubber and cord test pad from the test mold cavity and cool at room temperature. Age prepared specimens at ambient temperature for 4 h minimum, 4 days maximum, unless otherwise agreed between purchaser and supplier. (Immediate testing is sometimes preferred.)

8.8 Cut the cord test pad using shears, a sharp knife, or clicker die to produce individual H-test adhesion specimens consisting of a single cord with each end embedded in the center of a rubber tab approximately 25 mm (1 in.) in length (Fig. 1). Trim off excess rubber flash.

9. Procedure

9.1 Perform test in the standard atmosphere for testing tire cords, which is 24 ± 1°C (75 ± 2°F) and 55 ± 2 % relative humidity.

9.2 Prepare tensile tester with test grips described in Fig. 3; ensure axial alignment. Set the gage distance between the grips to 1.5 mm (0.0625 in.). Set the crosshead speed to 127 ± 10 mm/min (5 ± 0.5 in./min).

9.3 Insert the H-test adhesion specimen in the clamps, having one leg of the "H" in the upper clamp, and the other leg in the lower grip.

9.4 Activate the crosshead. Observe and record the cord pull-out adhesion force to the nearest 0.5 N (0.1 lbf) by means of an automatic recorder, or interfaced computer.

[6] Available from Fisher Pen Co., Forest Park, IL.

[7] "Krylon" No. 1349 Industrial Lube, available from Borden, Inc., Columbus, OH has been found satisfactory for this purpose.

9.5 Repeat, until the number of test specimens, as agreed upon between the purchaser and supplier, have been tested for each laboratory sample.

9.6 When elevated temperature testing is required, test as directed in 9.2-9.5, except enclose the specimen grips in an oven attached to the tensile tester. Heat the specimens in the oven, controlled at 100 ± 2°C (212 ± 4°F) for not less than 15 min and not more than 60 min total elapsed time for testing of any one specimen.

9.6.1 As an alternate procedure, the specimens may be heated in an oven adjacent to the tensile testing machine and then removed, one at a time, and tested within 15 s after removal. The technique for heating and testing the specimens must be agreed upon by the purchaser and the supplier.

10. Report

10.1 Report the following information:

10.1.1 State that the tests were performed as directed in Test Method D 4776. Describe the products sampled and the method of sampling used.

10.1.2 Cord identification and construction.

10.1.3 Adhesive identity, cord percent dip pick-up, and curing conditions (time, temperature, pressure), when known.

10.1.4 Test condition temperature.

10.1.5 Rubber stock identification and gage (thickness).

10.1.6 Type tensile tester used.

10.1.7 Average of the number of specimen determinations per sample, to the nearest 0.5 N (0.1 lbf).

11. Precision and Bias

11.1 *Test Data*—No recent interlaboratory test has been conducted using this method. However, 1500/2 polyester and 1890/2 nylon were tested for the current example. Analysis of variance indicates that most of the variance in test results is variability in the material.

11.1.1 Available information indicates the pullout force for these materials expressed as a coefficient of variation is typically about 1.36 % of the average, but may range from about 0.66 % to 2.49 % of the average.

11.2 *Precision*—For the typical single-operator component of variance reported above, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed below:

Number of Observations In Each Average	Critical Differences Percent of The Grand Average For Single Operator Precision[A]
3	4.08
4	3.53
8	2.50
15	1.82
16	1.77

[A] The critical differences were calculated using t = 1.960 which is based on infinite degrees of freedom.

NOTE 3—To convert the tabulated values of the critical differences to units of measure, multiply the average of the two specific sets of data being compared by the critical differences expressed as decimal fractions.

12. Keywords

12.1 adhesion; reinforcements (textiles); rubber; tire cord