



Designation: D 429 – 99

Standard Test Methods for Rubber Property—Adhesion to Rigid Substrates¹

This standard is issued under the fixed designation D 429; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for testing the static adhesional strength of rubber to rigid materials (in most cases metals).

Method A—Rubber Part Assembled Between Two Parallel Metal Plates.

Method B—90° Stripping Test—Rubber Part Assembled to One Metal Plate.

Method C—Measuring Adhesion of Rubber to Metal with a Conical Specimen.

Method D—Adhesion Test—Post-Vulcanization (PV) Bonding of Rubber to Metal.

Method E—90° Stripping Test—Rubber Tank Lining—Assembled to One Metal Plate.

Method F—Rubber Part Assembled Between Two Parallel Convex-Shaped Metal Plates

1.2 While the test method may be used with a wide variety of rigid materials, use of materials other than metals is the exception. For this reason, we have used the word “metal” in the text rather than “rigid materials.”

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.*

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Practice for Operating Salt Spray (Fog) Apparatus²
- D 395 Test Methods for Rubber Property—Compression Set³

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-Like Materials and are the direct responsibility of Subcommittee D11.25 on Rubber Adhesive Systems.

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² Annual Book of ASTM Standards, Vol 03.02.

³ Annual Book of ASTM Standards, Vol 09.01.

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension³

D 413 Test Methods for Rubber Property—Adhesion to Flexible Substrate³

D 471 Test Method for Rubber Property—Effect of Liquids³

D 572 Test Method for Rubber—Deterioration by Heat and Oxygen³

D 573 Test Method for Rubber—Deterioration in an Air Oven³

D 1149 Test Method for Rubber Deterioration—Surface Ozone Cracking in a Chamber³

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets³

D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products³

D 3491 Test Methods for Testing Vulcanizable Rubber Tank and Pipe Lining⁴

E 4 Practices for Force Verification of Testing Machines⁵

G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials⁶

3. Significance and Use

3.1 These test methods are designed primarily for specimens prepared in a laboratory under standardized conditions such as may be used to provide data for development and control of rubber compounds and methods of manufacture. With slight modifications as indicated, Methods A, B, C, D, and E are also used for obtaining comparative adhesional test values of production parts whenever the design permits preparation of suitable test specimens. Methods A, B, C, and D are applicable in the case of many products in which rubber is used for controlling vibration.

⁴ Annual Book of ASTM Standards, Vol 09.02.

⁵ Annual Book of ASTM Standards, Vol 03.01.

⁶ Annual Book of ASTM Standards, Vol 14.02. This practice is also in the 1978 SAE Handbook.

METHOD A—RUBBER PART ASSEMBLED BETWEEN TWO PARALLEL METAL PLATES

4. Terminology Adhesion Failure Terminology

4.1 *R* indicates the failure is in the rubber.

4.2 *RC* indicates the failure is at the rubber-cover cement interface.

4.3 *CP* indicates the failure is at the cover cement-prime cement interface.

4.4 *M* indicates the failure is at the metal-prime cement interface.

NOTE 1—*Example*—The percentages of the various types of failure may be estimated as in the following examples:

R-50, *RC*-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber cover cement interface.

R-25, *RC*-25, *M*-50 means three types of failure were present with the *M* indicating 50 % failure at the metal-primer interface.

5. Significance and Use

5.1 Method A is used to determine adhesion values in instances where users feel this design is more reflective of an actual product and where control tests have been set up as a result. The sample also lends itself to testing the effectiveness of different processing techniques and various adhesive systems.

6. Apparatus

6.1 *Testing Machine*—A tension testing machine conforming to the requirements of Practices E 4 shall be used for measuring the strength of adhesion. The moving head of the machine shall travel at the rate of 0.40 ± 0.04 mm/s (1.0 ± 0.1 in./min). The machine shall be provided with a recording device to give the total force at the conclusion of the test. A

machine of not more than 40 kN (10 000 lbf) capacity will be found suitable in most cases.

6.2 *Grips*—The fixtures for holding the specimen in the testing machine will depend on the type of specimen but in all cases shall be provided with ball seats to permit proper centering of the load during the test. A typical adhesion test assembly is shown in Fig. 1. Specially molded laboratory specimens shall be attached by means of threaded studs on the metal parts as shown in Fig. 2, but with specimens cut from production parts the clamp illustrated in Fig. 3 may be used.

7. Standard Test Specimens

7.1 The standard test specimen shall consist of a cylinder of rubber 3.2 ± 0.1 mm thick (0.125 ± 0.005 in.) and an end area of 1250 ± 5 mm² or 2.00 ± 0.01 in.² (diameter of 39.9 ± 0.1 mm (1.597 ± 0.005 in.)) attached to the faces of two metal plates each at least 9.5 mm (0.37 in.) in thickness and of the same diameter as the rubber cylinder. The metal plates shall have smooth flat faces that shall be parallel in the finished specimen. The cylindrical shape is used to eliminate sharp corners and to give uniform distribution of the pulling force.

NOTE 2—While a 1250 mm² (2 in.²) area test specimen shall be considered the standard for reference purposes, it shall be permissible to use a specimen of only 625 mm² (1 in.²) in area, when it is desirable to prepare and cure a large number of test specimens at one time. The diameter of this test specimen shall be 28.21 ± 0.02 mm (1.129 ± 0.001 in.) and the unvulcanized piece shall be cut to dimensions of approximately 24 mm ($1\frac{5}{16}$ in.) in diameter and approximately 5 mm ($\frac{3}{16}$ in.) in thickness (approximately 10 % volume excess).

8. Laboratory Preparation of Standard Test Specimens

8.1 Prepare the standard test specimen to be used for development and control purposes in the laboratory as described in 8.2 through 8.6.

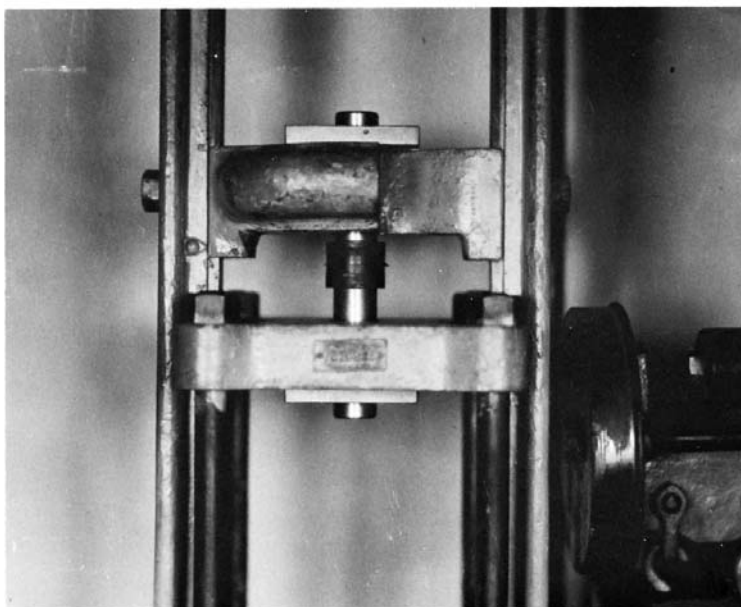


FIG. 1 An Example of an Adhesion Test Specimen Attached in a Testing Machine

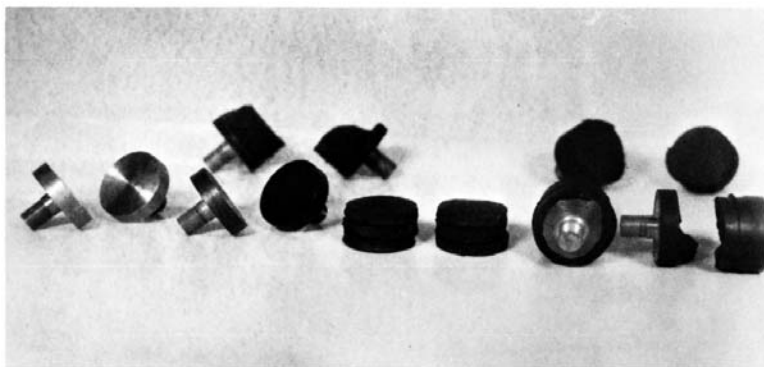


FIG. 2 Views of a Typical Test Specimen Showing Individual Metal Parts and Rubber Cylinders

8.2 Machine circular metal parts of standard dimensions from rolled bar steel SAE No. 1020⁷ for standard reference pieces. However, other metals may be used if in conformity with the essential dimensions. Prepare smoothly machined test faces of the metals in accordance with any test method for securing adhesion that may be under investigation.

8.3 Cut unvulcanized rubber pieces to dimensions of approximately 35 mm (1³/₈ in.) in diameter and approximately 5 mm (³/₁₆ in.) in thickness so as to give maximum pressure of the rubber against the metal surface during vulcanization (see Fig. 2). Clean or treat surfaces to be adhered in accordance with the method being investigated.

8.4 Assemble the metal parts and rubber pieces for vulcanization in the mold. A typical mold and test piece are shown in Fig. 4. The test method of assembly is illustrated in Fig. 2. The mold is so constructed that there is obtained after vulcanization approximately 0.039 mm (0.0015 in.) of rubber over the edge of the metal to eliminate tearing of the rubber during test from the sharp edge of the metal. Prior to vulcanization, take great care to keep the surfaces to be adhered clean and free of dust, moisture, or other foreign material. Do not touch surfaces with the hands.

8.5 Vulcanize by heating the mold for a definite time at a controlled temperature in a hydraulic vulcanization press. Choose the time and temperature of cure according to the rubber compound used. At the conclusion of the cure, take care in removing the specimens from the mold to avoid subjecting the adhered surfaces to stress before the specimens have cooled.

8.6 After vulcanization, store the specimens at a temperature of $23 \pm 2^{\circ}\text{C}$ ($73.4 \pm 3.6^{\circ}\text{F}$) at least 16 h prior to testing.

9. Test Specimens from Production Parts

9.1 For comparative test of adhesion in production parts, it is necessary to select a portion of the part so that a specimen consisting of a rubber piece sandwiched between two parallel plates of metal may be cut. Hollow-mill a specimen out of the production part and finish the piece either by turning or grinding down to size in such a manner that the surfaces of the rubber and metal shall be free of imperfections that would have

any tendency to start a tear in the rubber part during the application of tension while testing.

9.2 In routine production testing, it is sometimes necessary to test specimens of different size and shape than those specified as standard. In such cases, the test method of preparation shall be similar to that given in 9.1, but the test results secured shall not be compared with those obtained using the standard test specimen.

10. Procedure

10.1 Mount the vulcanized standard test specimen or the specimen cut from a production part in the testing machine, as shown in Fig. 1, using care in centering and adjustment so that the tension shall be uniformly distributed. Apply a steady tension load at the specified machine speed until the rubber either separates from the metal surface or ruptures. Record the total force at the time of failure.

10.2 In case of rupture in the rubber, the strength of the adhesion bond is obviously not measured, but is shown to be greater than the strength of the rubber itself. Record the type or types of failure, and express in accordance with the adhesion failure terminology described in Section 5. Estimate and record the percentages of the various types of failures.

11. Calculation

11.1 Express the adhesion value in pascals or pounds-force per square inch and calculate by dividing the tension force causing failure by the original area of the adhered surface that separates. In case of rupture of the rubber, the adhesion value is recognized as being higher than that reported.

12. Report

12.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet as illustrated in Table 1:

12.1.1 The result, calculated in accordance with Section 11,

12.1.2 All observed and recorded data including the number of test pieces involved,

12.1.3 Notation of type or types of failure, and the estimated percentages of the various types of failures (Section 10),

12.1.4 A description of the specimen including a statement of the test method used for determining adhesion, if known, and a notation indicating whether the specimen was molded in the laboratory or prepared from a production part.

⁷ Supporting data are available from ASTM Headquarters. Request RR: D11-1040.

METHOD B—90° STRIPPING TEST—RUBBER PART ASSEMBLED TO ONE METAL PLATE

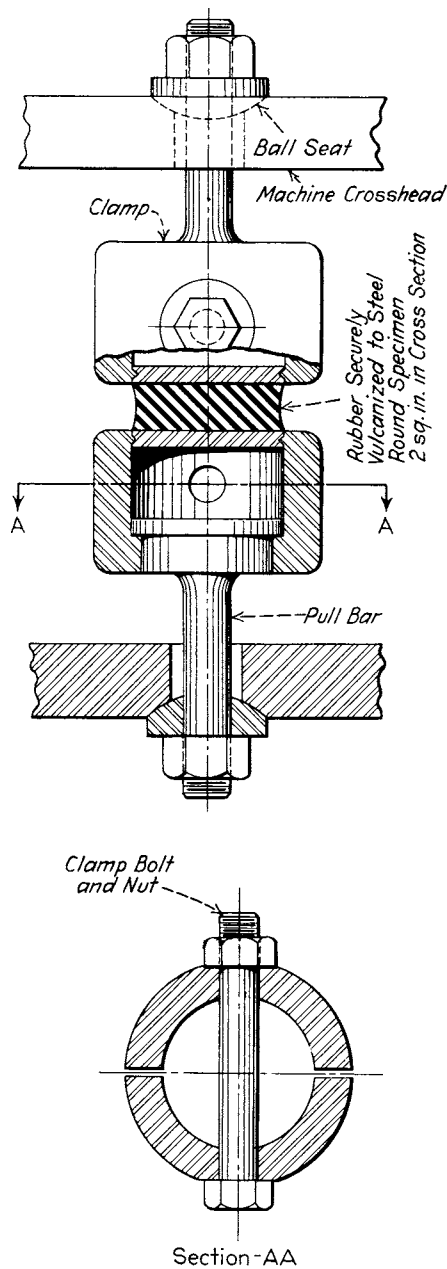


FIG. 3 Method of Testing Adhesion When Sample Section is Taken from Production Parts

- 12.1.5 Dimensions of the test specimen (comparisons may be made only among specimens of the same size and shape),
- 12.1.6 Date of manufacture or vulcanization, if known,
- 12.1.7 Time and temperature of vulcanization, if known,
- 12.1.8 Temperature of test room, and
- 12.1.9 Date of test.

13. Precision and Bias ⁷

13.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.

14. Significance and Use

14.1 This test is intended for determining the adhesive strength of rubber-to-metal bonding agents. The results are obtained by measuring the force necessary to separate a rubber from a metal surface. The data obtained indicate the strength of adhesion along a line across the width of the rubber strip being separated from a metal plate at a 90° angle. The test provides valuable data for development and control of rubber compounds and test methods of bonding, and it also serves as a screening test for the evaluation of various bonding agents or techniques, or both.

NOTE 3—It is permissible to use a 45° angle to bring about separation, but if this is done, the same should be indicated on the form on which the data are recorded. If this notation does not appear, it must be assumed that the 90° angle has been used. The angle of pull, in this case 45°, merely means that the equipment has been so designed that the angle made by that face of the rubber being stripped from the metal and the metal to which it has been adhered is 45°. Experience indicates a lower force is obtained for the 45° angle than for the 90° angle, and also a break significantly closer to the bond interface.

15. Adhesion Failure Terminology

15.1 *R* indicates the failure is in the rubber.

15.2 *RC* indicates the failure is at the rubber-cover cement interface.

15.3 *CP* indicates the failure is at the cover cement-prime cement interface.

15.4 *M* indicates the failure is at the metal-prime cement interface.

NOTE 4—Example—The percentages of the various types of failure may be estimated as in the following examples:

R-50, *RC*-50 means that roughly one half or 50 % of the area showed failure in the rubber and the other 50 % showed failure at the rubber-cover cement interface.

R-25, *RC*-25, *M*-50 means that three types of failure are present with the *M* indicating 50 % failure at the metal-primer interface.

16. Apparatus

16.1 *Testing Machine*— A power driven test machine equipped to produce a uniform rate of grip separation shall be used for measuring the strength of adhesion. The head of the machine shall travel at the uniform rate of 0.83 ± 0.08 mm/s (2 ± 0.2 in./min).

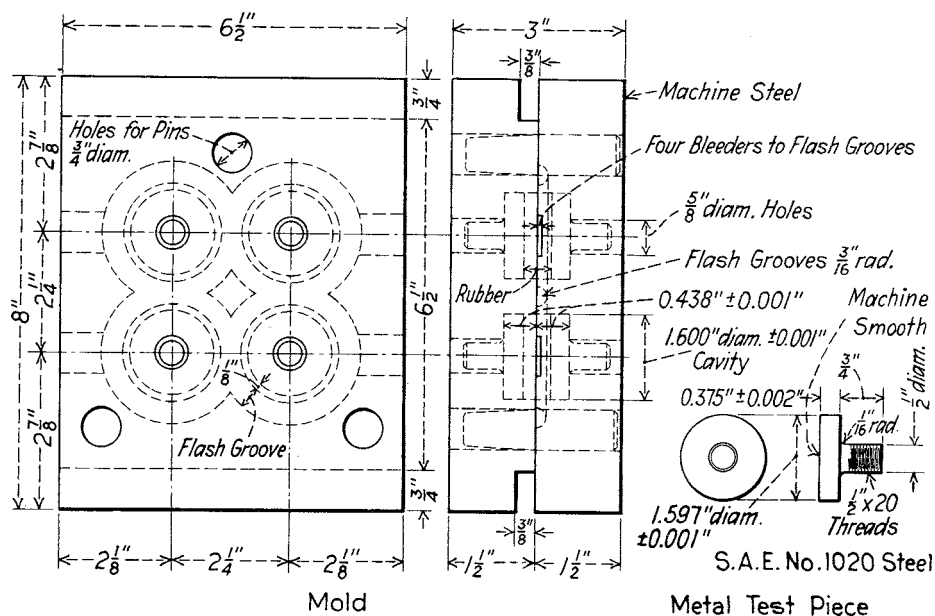
16.2 *Fixture*—Any suitable fixture for holding the test specimen to the upper head of the machine may be used provided the direction of pull to cause separation shall be, at all times during the test, as nearly perpendicular as possible, that is, making a 90° angle with the top holding fixture. The test fixture shown in operation in Fig. 5 complies with the above and provides the ease and simplicity of testing.⁸

16.3 *Grips*—Any suitable grip may be used, provided it does not slip or rupture the rubber strip. The grip shown in Fig. 5 complies and provides ease and simplicity of operation.

⁸ Complete detail drawings of the fixture are available at a nominal cost from ASTM, 100 Barr Harbor Drive, Conshohocken, PA 19428. Request Form 101-1019.



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in.	mm
1/16	2
1/8	3
3/16	5
3/8	10
1/2	13
5/8	16
3/4	19
1 1/2	38
2 1/8	54
2 1/4	57
2 7/8	73
3	76
6 1/2	165
8	203
0.375 ± 0.002	9.53 ± 0.05
0.125 ± 0.005	3.2 ± 0.1
1.597 ± 0.001	15.16 ± 0.03
1.600 ± 0.001	40.64 ± 0.03

FIG. 4 Detailed Drawing of a Mold for Adhesion Test Pieces

TABLE 1 Example of Form for Reporting Adhesion test Results

Stock	Cure	Metal and Metal Finishing Used	Pull		Type of Break, %			
			Sample, lbf/in.	Average, lbf/in.	R	RC	CP	M

Date _____

By _____

17. Standard Test Specimen

17.1 The standard test specimen shall consist of a strip of rubber 6.3 ± 0.1 mm (0.250 ± 0.005 in.) in thickness, 25 ± 0.05 mm (1.000 ± 0.002 in.) in width, and 125 mm (5 in.) in length adhered to the face of a metal strip 1.6 ± 0.1 mm (0.062 ± 0.004 in.) in thickness, 25 ± 0.05 mm (1.000 ± 0.002 in.) in width, and 60 ± 1 mm (2.36 ± 0.04 in.) in length. The test specimen shall be so prepared that the bonded area of 25 by 25 mm (1 by 1 in.) is fixed approximately in the middle of the metal member as shown in Fig. 6.

NOTE 5—In cases where the 1.6-mm (0.062-in.) thick metal is perma-

nently deformed during testing, a thicker metal may be used when agreed upon between the purchaser and the supplier.

18. Preparation of Standard Test Specimen

18.1 Prepare the standard test specimen as described in 18.1.1 to 18.1.6:

18.1.1 Rectangular metal parts of the standard dimensions described in Section 17 shall have the area to be bonded prepared in accordance with the test method for determining adhesion that may be under investigation. Mask both ends with pressure-sensitive tape so that only the area described in Section 17 will be available for adhesion.



Documents

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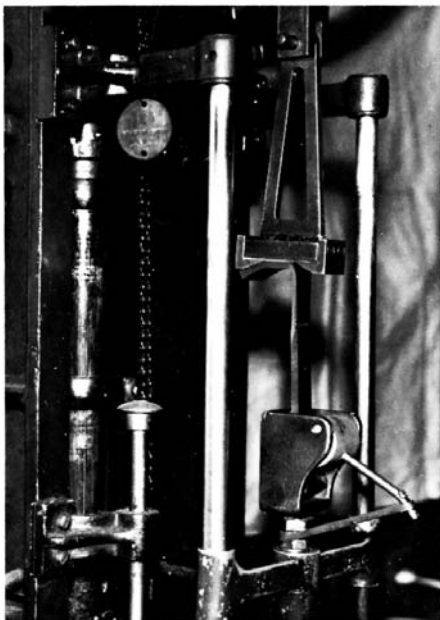


FIG. 5 Adhesion Tensile Testing Fixture in Operation

18.1.2 Cut unvulcanized rubber slabs approximately 8 mm (0.3 in.) in thickness to dimensions of the required size for the mold, so as to provide maximum pressure of the rubber against the metal surfaces during vulcanization. (See Fig. 6 for suitable mold design.) The inside mold dimensions parallel to the longitudinal axis of the metal part shall be 125 mm for nominal 5 in. The dimension along the transverse axis of the metal part can be altered according to the number of pieces required for any one cure. This will leave sufficient length of rubber strip for fastening in the grip shown in Fig. 5. Solvent-wash rubber surface to be adhered or treat in accordance with the method being investigated. Freshly milled rubber need not be solvent wiped.

18.1.3 Assemble the metal parts and rubber pieces for vulcanization as shown in Fig. 6, properly spacing the metal parts along one edge of the rubber slab and then placing the assembly, metal parts down, in the mold cavity. Cure the whole assembly in accordance with the method being investigated. Prior to assembly and vulcanization, take great care to keep the surfaces to be adhered clean and free of dust, moisture, or other foreign material.

18.1.4 Vulcanize by heating the mold under pressure for a definite time at a controlled temperature in a hydraulic vulcanizing press. The time and temperature of cure shall be in accordance with the system being investigated. At the conclusion of the cure, take care in removing the specimens from the mold to avoid subjecting the adhered surfaces to stress before the specimens have cooled.

18.1.5 After vulcanization, store the specimens at a temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) for at least 16 h.

18.1.6 Separate the specimens from each other in preparation for testing. This is done by cutting with a scissors, handknife, or other suitable equipment. The edges of the specimen may then be buffed on a belt sander to bring the edge of the rubber flush with the edge of the metal strips (Note 6).

Take care not to overheat the metal parts or the rubber and not to reduce the width of the sample being tested beyond the tolerances described.

NOTE 6—Present experience indicates negligible differences in results between samples buffed clean and samples tested without buffing, because edges of metal parts are not specifically prepared for adhesion and the edge contact area, given a metal part thickness of 1.6 mm (0.062 in.), is not significant.

19. Procedure

19.1 Place the test specimen (Note 7) symmetrically in the jaws attached to the fixed grip with the separating edge toward the operator, as shown in Fig. 5. Before the load is applied, strip the rubber from the metal plate for a distance of approximately 2 mm (0.08 in.) by using a sharp knife (Note 8). Place the tab in the grip and apply a steady load at the rate of 50 mm (2.0 in.)/min until separation is complete. Use the autographic recorder in order to show graphically the adhesion values over the full length of the test specimen.

NOTE 7—This specimen lends itself to the determination of the relative effect of environmental conditions on possible bond deterioration. Specimens may be exposed unstressed or under a constant, relatively low bond stress. It is recommended that one of the standard ASTM environmental exposure conditions be used. Among those applicable are the following: Test Methods D 471, D 572, D 573, D 1149, Practice G 23, and Method B 117. If environments other than the above are used, they shall be described completely. This should include the conditions of the exposure such as time, temperature, etc.

NOTE 8—Stripping the rubber from the plate initially, and as necessary during the test, is done to observe more clearly adhesion to the substrate rather than the tear strength of the stock.

20. Calculations

20.1 Express the adhesion value in newtons per millimetre or pounds-force per inch of width.

21. Report

21.1 The report shall include the following, preferably reported on an Adhesion Test Data Sheet illustrated in Table 1:

21.1.1 The results calculated in accordance with Section 20.

21.1.2 All observed and recorded data,

21.1.3 Notation of the type or types of failures and the estimated percentages of the various types of failures (Section 15),

21.1.4 Description of the specimen including statement of method of determining adhesion, if known,

21.1.5 Date of vulcanization, if known,

21.1.6 Time and temperature of vulcanization, if known,

21.1.7 Temperature of test room, and

21.1.8 Date of test.

22. Precision and Bias ⁷

22.1 Precision and bias do not exist for this test method because resources necessary for round-robin testing have not been forthcoming.