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Standard Test Methods for Rubber Cements[1]

This standard is issued under the fixed designation D 816; 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.

ε[1] NOTE—Section 36, Keywords, was added editorially in June 1993.

1. Scope

1.1 These test methods describe tests to measure the properties of adhesives, commonly called rubber cements, that may be applied in plastic or fluid form and that are manufactured from natural rubber, reclaimed rubber, synthetic elastomers, or combinations of these materials. All tests are not to be considered as applicable to a particular type of adhesive nor is every test included that may be applicable to a particular type. The tests do provide standard procedures for evaluating the more important properties of the usual adhesives ordinarily classed as rubber cements.

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

1.3 *This standard does not purport to address all of the safety problems, 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 230 Specification for Numbered Cotton Duck and Army Duck[2]

D 413 Test Methods for Rubber Property—Adhesion to Flexible Substrate[3]

D 429 Test Methods for Rubber Property—Adhesion to Rigid Substrates[3]

D 573 Test Method for Rubber—Deterioration in an Air Oven[3]

D 1084 Test Methods for Viscosity of Adhesives[4]

D 3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing[5]

NOTE 1—The specific dated edition of Practice D 3040 that prevails in this document is referenced in the Precision section.

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[3]

2.2 *Other Document:*

SAE Handbook[6]

3. Summary of Test Methods

3.1 The tests described in these test methods may be grouped into two classes, the first including those procedures in which the adhesive is applied to specimens of materials to be bonded together after which the quality of the bond is evaluated, and the second including those procedures applicable to the adhesive itself without consideration of the bonding properties. The classes are as follows:

Class I:	Section
Adhesion strength	8 to 15
Bonding range	16 to 18
Softening point	19 to 21
Cold flow	22 to 24
Class II:	
Viscosity	25
Stability	26 to 27
Cold brittleness	28 to 29
Density	30 to 31
Plastic deformation	32 to 33

4. Significance and Use

4.1 These tests are used as a test method of classifying, evaluating, and controlling cement compositions. Adhesion strength in tension, shear, and in a peeling mode are necessary where rubber materials are used in various engineering applications where forces are encountered both normal to the adhesion plane and parallel to the plane of the adhesive interface.

5. Materials for Bonded Specimens

5.1 The following materials shall be used as specified for the preparation of specimens bonded with the test cement for tests of Class I and shall be reported with the result of the test:

[1] These methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.25 on Rubber Adhesive Systems.

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[2] Discontinued; see *1975 Annual Book of ASTM Standards*, Part 32.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

[4] *Annual Book of ASTM Standards*, Vol 15.06.

[5] Discontinued—see *1986 Annual Book of ASTM Standards*, Vols 09.01 and 09.02.

[6] Available from Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017.

5.1.1 *Aluminum Sheet, Cladded Material*, gage 1.6 mm (0.064 in.),

5.1.2 *Standard Rubber Test Sheets*,

5.1.3 *Cotton Duck*, medium texture 1030 g/m^2, in accordance with Specification D 230, and

5.1.4 *Coated Fabrics*, of which the fabric strength and the adhesion of coating to fabric exceed the strength of the adhesive bond.

5.1.5 *Rolled Sheet or Machined Steel*, SAE No. 1020, sand blasted or sanded with medium/fine abrasive, and

5.1.6 Special materials such as leather, cork, felt, etc.

6. Sampling

6.1 Before sampling, the cement to be tested shall be thoroughly agitated to uniform consistency throughout, after which a sample shall be taken of sufficient quantity for performance of the tests required. The sample shall be kept in a tightly closed container to prevent evaporation and shall be agitated or stirred to uniform consistency immediately before test or use in preparation of test specimens.

7. Test Conditions

7.1 The tests of adhesives shall be conducted at a temperature of 23 ± 2°C (73.4 ± 3.6°F) and relative humidity of 50 ± 5 %. All material and equipment shall be held at these conditions prior to test for a sufficient time to assure equilibrium.

ADHESION TESTS

8. Types of Adhesion Test

8.1 The adhesion strength of a cement used to bond two materials is measured by determining the force required to produce separation by failure of the adhesive. This force may be applied as (*1*) a direct tension pull in the direction normal to the adhered surfaces, (*2*) a shearing force in the same plane as the surfaces, or (*3*) a stripping or peeling force which produces progressive separation over the adhered surfaces. It is obvious that the stripping procedure can be used only when at least one of the adhered materials is flexible. Test methods, designated A, B, and C, are given for preparing and testing bonded specimens by each of these types of adhesion test. The adhesion test may also be applied, when specified, to bonded specimens that have been subjected to deteriorating influences such as accelerated aging or action of oils or solvents after their preparation and before separation, but in this case full details concerning such treatment shall accompany the statement of the result of the adhesion test.

Test Method A—Adhesion Strength in Tension

9. Preparation of Test Specimens

9.1 For testing cements for use in vulcanized applications, the test specimens shall be those specified in Sections 5 and 6 of Test Methods D 429, except that after buffing or grinding the surfaces to be adhered, and washing them free from dirt or oil using trichloroethylene or perchloroethylene, they shall be coated with a uniform film of the test adhesive 0.025 mm (0.001 in.) in thickness or of an optimum thickness suitable to the products, as agreed by the parties concerned.

9.2 For testing cements for use in unvulcanized applications, test specimens similar to those specified in 9.1 shall be prepared except that the rubber cylinders used in their preparation shall be cut from vulcanized rubber and the assembly of the specimen shall be made without vulcanization in a mold. To accomplish this, the circular faces of the rubber cylinders shall be buffed smoothly and evenly to parallel planes and shall be coated with a uniform film of the test cement 0.025 mm (0.001 in.) in thickness or of an optimum thickness suitable to the products, as agreed by the parties concerned. With thin cements, the coating may be done with a brush, applying successive coats and allowing sufficient time between coats for evaporation of the solvent. The faces of the metal plates shall be freshly sand-blasted or ground to a dull finish with fine emery and washed free of dust and oil with trichloroethyene or perchloroethylene. The metal faces shall also be coated with the test cement as in the case of the rubber cylinders. When the cement films have dried to the point where there is still aggressive tackiness but no tendency for the film to lift when touched with a finger, each rubber cylinder shall be bonded firmly by hand between the cemented faces of two metal plates and the assembly shall be clamped in a suitable loading fixture under a compressive load of 44 N (10 lbf) for at least 24 h.

10. Procedure

10.1 Test the prepared specimens as described in Section 8 of Test Methods D 429. Take care to use a tension testing machine of capacity and sensitivity suitable for loads of the magnitude anticipated with the particular adhesive being tested. Express the adhesion strength in kilopascals (or pounds per square inch) calculated by dividing the tension load causing failure by the original area of the adhered surface that separates.

10.2 Test two specimens and report the higher of the results as the adhesion strength in tension provided they check within 10 % of the higher value. Otherwise, test additional specimens until results are obtained consisting of a high value and one that checks this within 10 %. If no check of a high value is obtained when six specimens have been tested, the average of all six results may be reported as the *average* adhesion strength in tension.

Test Method B—Adhesion Strength in Shear

11. Types of Test Specimens

11.1 Specimens for shear tests shall be of two types, namely, Type 1 lap specimens and Type 2 sandwich specimens, as shown in Fig. 1. Lap specimens shall be used when the adhesive is to be tested for bonding materials that are both extensible such as rubber, felt, or cork, or both relatively nonextensible like metal, cloth, and leather. Sandwich-type specimens shall be used when one of the bonded materials is extensible and the other nonextensible. In both types of specimens, two strips of the material 25 mm (1 in.) in width and 125 mm (5 in.) in length shall be bonded over an area 625 mm^2 (1 in.^{2}) with the free ends extending parallel in opposite directions to permit clamping in a testing machine. In the case of the laptype specimens the two strips shall be bonded directly to each other by means of the adhesive being tested, but in the case of the sandwich-type specimen a 625 mm^2 square block of

FIG. 1 Test Specimens for Adhesion Strength in Shear

extensible material having a thickness of not more than 3.2 mm (⅛ in.) shall be bonded between two nonextensible strips by means of the test adhesive.

12. Preparation of Test Specimens

12.1 The faces of the materials that are to be bonded by the test adhesive shall be buffed by hand using a wire brush, fine emery cloth, or similar suitable abrasive to assure a slightly roughened surface which shall then be washed free of dirt and oil by means of oil-free 1,1,1 trichloroethane. The specimens shall be allowed to dry thoroughly and the surfaces shall not be touched after washing. The test adhesive shall then be applied by brushing, spraying, or spreading to form a film 0.025 mm (0.001 in.) in thickness or of an optimum thickness suitable to the products, as agreed by the parties concerned.

NOTE 2—With cements of sufficiently heavy consistency to allow their use, the volumetric or static-mass cement spreaders shown in Fig. 2 and Fig. 3 may be found convenient for applying the adhesive in preparing the required specimens. With thin cements applied by means of brushing, several coats may be necessary, in which case adequate drying time to permit evaporation of the solvent should be allowed between coats. Three such coats will usually be found sufficient.

12.2 When the cement film has dried to the point where there is still aggressive tackiness but no tendency for the film to lift when touched with a finger, the surfaces to be bonded shall be aligned carefully so that 625 mm^2 (1 $in.^2$) shall be adhered and pressed together firmly by hand. The specimens, resting on a smooth hard base, shall then be rolled with five complete strokes using a metal roller 25 mm (1 in.) in diameter applied with a hand force of approximately 90 N (20 lbf). The specimens shall then be allowed to age for at least 24 h under the standard test conditions of temperature and humidity (Section 7) without any applied force.

13. Procedure

13.1 After the required aging period, clamp the prepared specimen in the grips of a tension testing machine conforming to the requirements in Section 7 of Test Methods D 413, using shims in the grips as indicated in Fig. 1 so that the applied force shall be in the plane of the cemented area. Take care to be certain that the specimen is properly centered and is straight in the grips. When testing Type 1 specimens made of extensible materials, provide for maintaining the strip during the test approximately in the plane of the clamps. This may be done by holding the specimen against a plate attached to the stationary

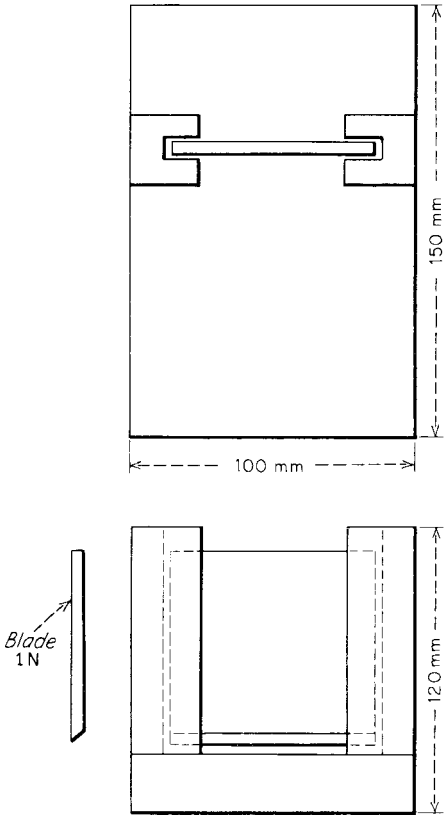

FIG. 3 Static-Mass Cement Spreader

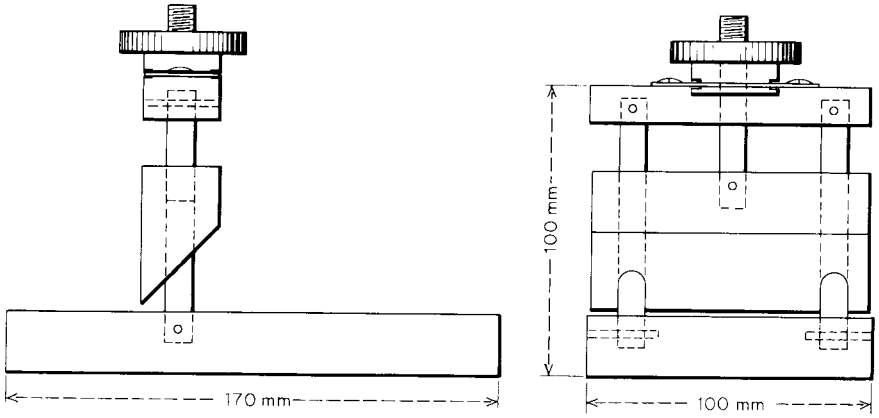

FIG. 2 Volumetric Cement Spreader

clamp. Separate the clamps at the rate of 0.8 mm/s (2 in./min) and record the force causing separation of the specimen in kilopascals (or pounds per square inch) of the adhered surface that separates.

NOTE 3—When shims or plates are added to the clamps, the testing machine must be adjusted for zero force with these additions or the force due to their mass must be deducted from the indicated reading.

13.2 Test two specimens in each required combination and report the higher of the results as the adhesion strength in shear, provided they check within 10 % of the higher value. Otherwise, test additional specimens until results are obtained consisting of a high value and one that checks this within 10 %. If no check of the high value is obtained when six specimens have been tested, the average of all six results may be reported as the *average* adhesion strength in shear.

Test Method C—Adhesion Strength by Stripping Method (Peeling Test)

14. Preparation of Test Specimens

14.1 A strip of flexible material at least 150 mm (6 in.) in length by 25 mm (1 in.) in width shall be bonded by means of the test cement except for 50 mm (2 in.) at one end to a strip 300 mm (12 in.) in length by 50 mm in width of the other material that is to be adhered. The center lines of the two strips shall be superimposed. The surface to be bonded shall be properly prepared, for example, by buffing, washing with 1,1,1 trichloroethane, drying, and coating with a film of the test cement 0.025 mm (0.001 in.) in thickness or of an optimum thickness suitable to the products, as agreed by the parties concerned (Note 4). Uniting, rolling down, and aging shall be done as specified in Section 12 for shear test specimens.

NOTE 4—In the case of porous material, the thickness of the cement film shall not be specified.

15. Procedure

15.1 Place the test specimens in a tension testing machine by clamping the free end of the 50-mm (2-in.) wide strip in one grip and turning back the free end of the flexible 25-mm (1-in.) wide strip and clamping it in the other grip. The testing machine and the procedure used shall conform to that specified under the machine method for strip specimens in Sections 6 and 7 of Methods D 413. The autographic chart obtained constitutes the report of the test but determine the value of the adhesion by drawing on the chart the best average line between the maximum and minimum load values. Consider the load so indicated, expressed in newtons per metre (or pounds-force per inch) of width for separation at 0.4 mm/s (1 in./min), as the adhesion strength by the stripping method. For the evaluation of each cement specimen, prepare and test two specimens and report the higher average load obtained as the adhesion strength of the specimen.

BONDING RANGE

16. Type of Test

16.1 The test for bonding range is for determining the most suitable drying period for use with the test cement after application and before bonding the materials in order to obtain the maximum initial bond strength. A series of Type 2 shear adhesion test specimens (Fig. 1) is prepared using the test cement and allowing it to dry for different known periods before uniting the materials. These specimens are cemented, dried, bonded, and tested for adhesion strength in shear according to a definite time schedule. The drying period that results in the highest shear adhesion value is the optimum bonding range for the cement being tested.

17. Preparation of Test Specimens

17.1 The test specimens shall be Type 2 shear adhesion test specimens each made from two strips of aluminum alloy 25 mm (1 in.) in width by 125 mm (5 in.) in length bonded by the test cement to a block of heavily compounded rubber 25 mm square by 3.2 mm (⅛ in.) in thickness having a durometer hardness of 75 to 85. The specimens shall be prepared in duplicate as specified in Section 12 except that the application of the cement shall be so scheduled that successive pairs of specimens may be dried 1, 3, 5, 10, and 20 min before bonding and each tested exactly 3 min after making the bond. After the first 20 min of drying time, additional intervals of 20 min to a maximum of 2 h shall be used.

18. Procedure

18.1 Test the specimens prepared according to the prearranged schedule as specified in 13.3, 3 min after making the bond and rolling them. Record for each specimen the force in newtons (or pounds-force) required to separate the rubber from the metal and select the maximum value. Report the drying time used for cementing that specimen as the optimum bonding range. If the maximum value is obtained with a specimen dried for 2 h, report the optimum bonding range as 2 h or more.

SOFTENING POINT

19. Type of Test

19.1 The test for softening point is for the determination of the temperature at which the cement softens sufficiently to provide an ineffective bond. It consists of subjecting Type 2 shear adhesion test specimens (Fig. 1) under a definite shearing load for a specified interval to successive increments of temperature until the bond fails. The temperature at which failure occurs is taken as the softening point. Obviously, the test is useful primarily for comparisons between different cements, as the adhesion strength may be seriously reduced at temperatures below the softening point.

20. Preparation of Test Specimens

20.1 Type 2 shear adhesion test specimens (Fig. 1) using the test cement shall be prepared in duplicate as specified in 17.1, except that the drying time shall be that determined as the optimum bonding range and the specimens shall be aged before test at least 24 h under the standard conditions of temperature and humidity (Section 7) without applied pressure.

21. Procedure

21.1 Hang the test specimens with one metal end of each attached to a suitable support in an electric oven complying with the requirements in Section 5 of Test Method D 573.

Apply a force of 4.5 N (1 lbf) by means of a static mass (Note 5) carefully without shock to the lower end of each specimen. In applying the force and suspending the specimens, use suitable clamps and shims to maintain the specimens in a vertical position and to distribute the load uniformly over the test specimens. Maintain the oven temperature at 40°C (104°F) for 15 min and then increase 0.5°C (0.9°F)/min until the rubber separates from the metal. Report the temperature at which the bond fails as the softening point of the test cement used in the specimen.

NOTE 5—With certain special adhesives, it may be desirable to employ very much larger forces than the 4.5 N (1 lbf) specified. In these cases, the method may be modified accordingly, but any such change must be clearly indicated with the reported results.

COLD FLOW

22. Type of Test

22.1 The cold flow test is for evaluating the tendency for surfaces bonded by means of the test cement to slip or be displaced when subjected to difference in stress for a sustained period. This yielding is considered to be due to cold flow of the adhesive. The test consists of subjecting a bonded specimen to a specified load for a prolonged period and measuring the elapsed time for failure of the bond.

23. Preparation of Specimens

23.1 Type 2 shear adhesion test specimens (Fig. 1) using the test cement shall be prepared in duplicate as specified in Section 20.

24. Procedure

24.1 Hang the test specimens with one metal end of each attached to a suitable support and attach a mass of 0.45 kg (1 lb) (Note 5) carefully without shock to the lower end of each specimen. In attaching the mass and suspending the specimens, use suitable clamps and shims to maintain the specimens in a vertical position and to distribute the load uniformly over the test specimens. Maintain the loaded specimens at the standard test conditions of temperature and humidity (Section 7) and observe hourly for 24 h or until the time of failure. Record this time as the bond failure time in the cold flow test. If no failure occurs in 24 h, increase the force to 6.7 N (1.5 lbf) and observe the specimens for an additional 24 h as before. If the bond failure time is over 24 h with the 4.5-N (1-lbf) force and a second period with the larger force is required, the report shall indicate a bond failure time of over 24 h under 4.5 N (1 lbf) and also shall state the added time under the higher force required for failure.

VISCOSITY

25. Procedure

25.1 Determine the viscosity of the cement in accordance with Test Methods D 1084, using a duplicate specimen for checking.

STABILITY

26. Type of Test

26.1 The stability test provides a means for estimating the relative life of cements in usable form prior to application. When stored, even in containers, many cements jell because of slow vulcanization at ordinary temperatures or other physical or chemical changes and become unusable as adhesives. These changes are evidenced by change in viscosity of the cement. The test consists of subjecting portions of the sample of the cement under specified conditions to an elevated temperature and determining the viscosity at intervals until a marked change occurs, which time is taken as a measure of the stability of the cement.

27. Procedure

27.1 Measure twelve 250-cm^3 portions of the sample of test cement into glass test tubes approximately 38 mm in diameter and 300 mm in length so that each tube is approximately two thirds full. Fit the tubes with small reflux condensers and place in a constant-temperature water bath at 60°C (140°F). Determine and record the viscosity of the test cement at the start of the test as specified in Section 25. After 1 week in the water bath, remove two of the tubes, cool to room temperature, and measure for viscosity as at the start of the test. If these samples have jelled or show a marked increase in viscosity, discard all of the samples and start new ones, checking them in pairs daily. If the original samples show no appreciable change in viscosity, continue the heating for 5 additional days, taking out two samples each day for viscosity determinations until marked change in viscosity occurs or the cement jells. Record and report all viscosity results. Report the number of days of heating to produce the change as the stability number of the cement.

NOTE 6—With some special cements made from synthetic rubbers, the viscosity may increase progressively and the cement may become unusable before showing any sudden marked change indicating jelling. In such cases, it may be desirable to test a series of samples daily in the specified manner and plot the viscosity versus time. The resulting curve may be used as the report and no stability number stated. The evaluation of the usability of the cement will depend upon the purpose for which it is intended and the materials to be bonded.

COLD BRITTLENESS

28. Type of Test

28.1 The test for cold brittleness is for evaluating and comparing the resistance of cements to cracking or chipping when bent at low temperatures. A coat of the test cement on an aluminum strip is cooled at specified temperatures for a definite time after which the strip is bent in a prescribed manner and examined visually for cracking or chipping of the cement coat.

29. Procedure

29.1 Clean six strips of aluminum alloy 25 mm (1 in.) in width by 75 mm (3 in.) in length by 1 mm (0.040 in.) in thickness and cement on one side, forming a film of adhesive 0.025 mm (0.001 in.) in thickness or of an optimum thickness suitable to the products, as agreed by the parties concerned and as specified in Section 12. The film shall completely cover the one side of the metal. After aging for at least 3 h under the standard atmospheric conditions (Section 7), place two of the strips in cold atmospheres at each of the following temperatures: − 25°C (−13°F) and − 40°C (−40°F). Maintain

the strips at these temperatures for 30 min and then immediately bend double over a mandrel 10 mm (⅜ in.) in diameter with the cemented side out and without removing them from the cold atmosphere. Examine the coatings for visual signs of cracking or chipping. Report a qualitative statement of the results with the cement at each temperature.

DENSITY, OR MASS PER UNIT VOLUME

30. Apparatus

30.1 The density of a cement shall be determined by use of a calibrated pycnometer having a volume between 50 and 110 cm^3.

NOTE 7—If the volume is 100 cm^3 at 23°C (73.4°F), the density in kg/m^3 is ten times the mass in grams required to fill the pycnometer. If the volume is 83.3 cm^3 at 23°C, the density in lb/U.S. gal is ten times the mass required to fill the pycnometer.

31. Procedure

31.1 Weigh the pycnometer in grams accurately to three significant figures. Fill it with the calibration volume of the test cement which has been stirred until homogeneous and is free from air bubbles. Record the filled mass in grams. Calculate the density or the mass per unit volume as follows: Designate the difference in the mass of the filled pycnometer and empty pycnometer as *X*.

Divide *X* by 0.0833 to obtain density in kg/m^3.
Divide *X* by 10 to obtain density in lbm/U.S. gal.

PLASTIC DEFORMATION

32. Type of Test

32.1 The test for plastic deformation is applicable only to heavy adhesives such as doughs, sealers, putties, etc. It is not suitable nor of use in the case of liquid adhesives.

33. Procedure

33.1 Prepare two small spheres of the heavy adhesive approximately 25 mm (1 in.) in diameter by rolling in the hands. Place these spheres on small watch glasses and heat in an air oven at 125°C (257°F) for 1 h. Remove the spheres and examine for flow or distortion. If the spheres have suffered any marked change in shape, report the adhesive as subject to plastic flow in the deformation test.

34. Report

34.1 The report shall include the following:

34.1.1 Complete identification of the test sample of cement.

34.1.2 Date of test and date of manufacture if known.

34.1.3 Description of special materials used.

34.1.4 Film thicknesses used, whenever they may influence the test results, as the average thickness of adhesive layer after formation of the joint, within 0.025 mm (0.001 in.). (The method of obtaining the thickness of the adhesive layer shall be described including procedure, location of measurements, and range of measurements),

34.1.5 All observed and recorded data.

34.1.6 Statement of the results obtained in each of the tests required.

35. Precision and Bias

35.1 Tests for bonding range, softening point, cold flow, stability, cold brittleness, and plastic deformation are either of a process control character or of a qualitative nature and no precision statement on these is given.

35.2 For adhesion strength, precision statements are in the process of being prepared in the form specified by Practice D 3040-81. They will be added to these test methods when they are completed.

36. Keywords

36.1 cold flow; fabric; rubber cements