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Standard Test Method for Measuring Adhesion of Organic Coatings to Plastic Substrates by Direct Tensile Testing¹

This standard is issued under the fixed designation D 5179; 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 laboratory determination of organic coating adhesion to plastic substrates by mounting and removing an aluminum stud from the surface of the coating and measuring the force required to break the coating/substrate bond with a tensile tester.

1.2 This test method requires that the aluminum stud be adhered directly to the surface of a coated, cured panel (Fig. 1).

1.3 This test method is used to compare the adhesion of coatings to various plastic substrates, thus allowing for a quantitative comparison of various coating/substrate combinations.

1.4 Other tensile test methods are Test Method D 4541 and ISO 4624 (but are not technically equivalent).

1.5The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.6 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: ²

D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers

2.2 Other Standard:

ISO 4624 Paints and Varnishes—Pull-off test for adhesion³

3. Summary of Test Method

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3.1 An aluminum stud is bonded directly to a coated cured panel. The adhesive is allowed to cure for 2 h at room temperature. The specimen is then subjected to test on a tensile tester equipped with an upper coupling adapter (Fig. 2), and a restraining device (Fig. 3).

3.2 If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because the substrate and its surface preparation (or lack of it) has a drastic effect on the adhesion of coatings, a method of evaluation adhesion of a coating to different substrates or surface treatments, or of different coating to the same substrate and treatment, is of considerable usefulness in the industry.

4. Significance and Use

4.1 The pull-off strength (commonly referred to as adhesion) of a coating is an important performance property that has been used in specifications. This test method serves as a means for uniformly preparing and testing organic coatings on plastic substrates.⁴ Further information may be found in Appendix X1.

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¹ This test method is under the jurisdiction of ASTM Committee <u>D-1D01</u> on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films .

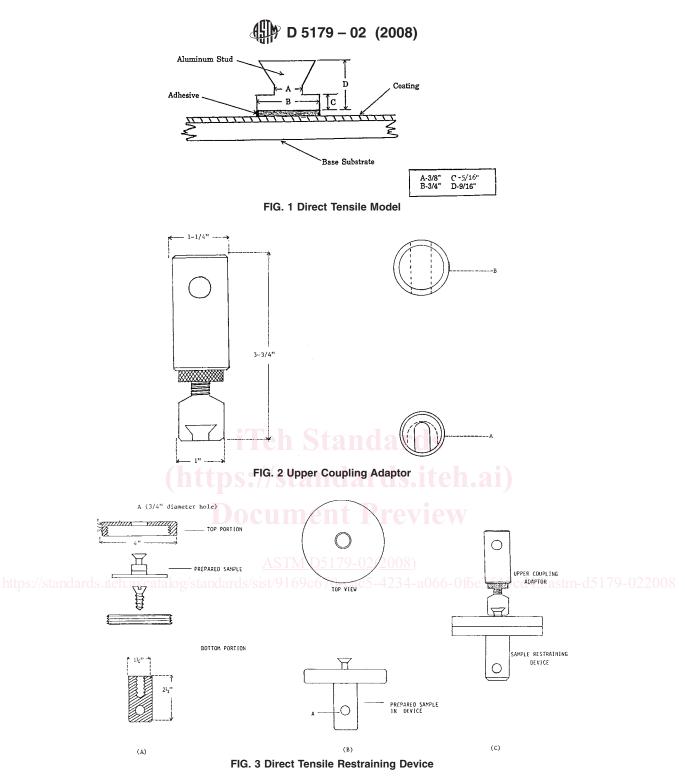
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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards, Vol 06.02.volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁴ Gray, K. N., Buckley, S. E., and Nelson, G. L., "Accessing Measurement Standards for Coating Adhesion to Plastics," *Modern Paint and Coatings Journal*, Vol 75, No. 10, October 1985, p. 160.



5. Apparatus and Materials

5.1 Tensile Tester, commercially available.⁵

5.2 Aluminum Stud—The shape and dimensions of the stud are shown in Fig. 1.

5.3 Upper Coupling Adaptor—The apparatus is shown in Fig. 2. The adaptor is $3\frac{3}{4}$ in. (94 mm) long, $1\frac{1}{4}$ in. (31 mm) in diameter at the top, and 1 in. (25 mm) in diameter at the bottom. The hole, indicated by "*B*," is used to attach the adaptor to the tensile tester load cell. The hole has a $\frac{1}{2}$ -in. (13-mm) diameter. The machined opening indicated by "*A*," is to receive the head of the aluminum stud.

⁵ The sole source of supply of the wooden puritan applicators, in sizes 6 in. (150 mm) in length and 0.007 in. (2 mm) in diameter, known to the committee at this time is Hardwood Products Co., Guilford, ME 04443. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. International. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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5.4 *Restraining Device*—The apparatus is shown in Fig. 3A and 3 *B*. The $\frac{1}{2}$ -in. (13-mm) diameter hole, marked "A," in the 2 $\frac{1}{2}$ -in. (63.5-mm) long, 1 $\frac{1}{4}$ -in. (31-mm) diameter lower coupling adapter, is used to mount the device in the tensile tester. The top portion is 4 in. (100 mm) in diameter, $\frac{3}{4}$ in. (19 mm) high, and is fitted with a hole slightly larger than $\frac{3}{4}$ in. (19 mm) in diameter to allow stud clearance. The bottom screw portion is machined to fit with the top portion and is $\frac{1}{2}$ in. (13 mm) thick. Fig. 3C illustrates the final appearance of the test assembly before it is tested.

5.5 Wooden Applicators,⁵ used to clean the adhesive from around the aluminum stud.

5.6 *Adhesive*, cyanoacrylate adhesive⁶. Since cyanoacrylate adhesives lose adhesive bond strength with time, do not use previously opened containers or lots of adhesive known to be old.

5.7 Two-Kilogram Weight, approximately 2 in. (50 mm) in diameter and 5 in. (130 mm) in height.

6. Preparation and Conditioning of Specimen

6.1 *Sanding Procedure*—Sand the large face of stud with 80-grit sandpaper, making certain that the surface is uniformly roughened. When sanding studs, sand straight up and down; rotate stud one quarter of a turn and continue sanding. Make certain the surface of the stud is flat but rough. While sanding, keep the stud face parallel to the sandpaper. Nonplanar surfaces cannot be used in testing because they lead to nonuniform bonding of the stud to the coated surface. Only flat (planar) studs should be used in testing. Planarity may be assessed by placing a stud on a flat surface and checking to see if the stud wobbles. Careful preparation of studs is essential for good adhesive adhesion.

6.2 *Cleaning Procedure*—Place sanded studs in a large beaker. Pour technical grade acetone over the studs and swirl the beaker to completely wash the studs. Pour out acetone and repeat the cleaning procedure. Soak the stud for at least 15 min, pour out the acetone, and allow the studs to dry.

6.3 Place washed and dried studs and beaker in an oven at 120°C and allow to heat for 1 h. Remove the beaker and studs from the oven and allow to cool. DO NOT TOUCH FACE OF STUDS.

6.4 Spread four drops of cyanoacrylate adhesive on the large face of an aluminum stud. Next, quickly press the stud onto the coated test substrate. Place a 2-kg weight on the stud to ensure good contact between the stud, adhesive, and surface of the coating. Clean the excess adhesive from the edge of the stud with a wooden applicator. Carefully remove the weight after 2 min.

NOTE 1-Substrate panels may be cut to any size that fits the restraining device.

Note 2—When the stud is pressed, excess adhesive should escape from under the stud. Excess adhesive buildup at the edge of the stud is a major source of error if allowed to dry. This excess must be removed from around the stud. The adhesive may be removed using a wooden applicator and an absorbent, creped, low-lint material. Some workers have found cotton swabs also work well when removing the excessive adhesive.

6.5 Allow the sample to cure for 2 h at room temperature (see Note 3).

Note 3—The time specified led to the results reported in the Precision and Bias Section. If other than the recommended adhesive is used, the user should be guided by the cure time recommended by the adhesive manufacturer. A series of preliminary tests at several cure times may be necessary in the selection of a time that ensures cure of the adhesive used $TM D5179_{-}02(2008)$

Note 4—When reusing aluminum studs, the studs must be soaked in acetone to remove the coating and resanded. It has been found that soaking the studs in two separate acetone baths thoroughly removes the adhesive and coating. The first acetone bath is used to dissolve the cyanoacrylate adhesive, and the second is used to further clean and dissolve any coating or adhesive left on the stud. Before the studs are used, they should be resanded and cleaned with acetone as specified in 6.2, 6.3, and 6.4.

6.6 Conditioning—Condition the coated substrates for at least 24 h at $23 \pm 2^{\circ}C$ (73.5 $\pm 3.5^{\circ}F$) and 50 ± 5 % relative humidity, and test in the same environment or immediately on removal therefrom, unless otherwise specified by the purchaser and the seller.

7. Procedure

7.1 Install the restraining device and upper adaptor in the tensile tester.

7.2 Calibrate the tensile tester. Make sure that the chart speed is set at 8 to 20 in./min (20 to 50 cm/min). Make sure that the crosshead speed is set at 2 in./min (5 cm/min). Make sure that the chart full scale is set at 100 kg.

7.3 Place the specimen to be tested in the restraining device (Fig. 3A and 3 B). Pre-position the crosshead and then slowly lower it so the upper coupling adaptor can be attached to the test specimen. Take care to prevent the crosshead from impacting into the top of the specimen. Carefully attach the upper coupling adaptor to the stud (Fig. 3 C).

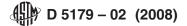
NOTE 5—When testing thin substrates, a piece of plastic may be placed in the restraining device behind the test specimen to prevent the substrate from flexing when the stud is pulled from it.

7.4 Turn on the chart recorder and pen. Start the tensile test. Stop the test when the stress returns to zero on the chart.

7.5 Examine the test area on each specimen to determine the type of coating failure, rating it according to the following:

7.5.1 Adhesive failure of the coating at the substrate, A,

⁶ Elmer's[®] Wonder Bond Plus[®] Super Glue, a cyanoacrylate adhesive sold by Elmer's Products, Inc., 180 E. Broad St., Columbus, OH 43215, was found acceptable in the round-robin study upon which this test method is based. Other adhesives may be used if they provide adequate adhesion between the aluminum stud and the coating being tested; the results reported in the Precision and Bias Section, however, may not apply. From other work it is noted that some cyanoacrylate adhesives may penetrate certain coatings and soften the coating or the plastic substrate. The same is true for other adhesives. Care in observation of unusual results is necessary to ensure against the possibility of error. This latter is to be suspected if an adhesive gives an unexpectedly low result. Adhesives that yield a brittle adhesive layer should not be used.



7.5.2 Cohesive failure in the coating, C,

7.5.3 Combination of adhesive failure at the coating/substrate interface and cohesive failure in the coating, AC,

7.5.4 Adhesive failure at the stud, S, and

7.5.5 Combination of adhesive failure at the stud and cohesive failure in the coating, CS.

7.6 For multilayer coatings, note if the failure is between the layers. If so, label as CM.

7.7 Number and retain all test specimens for adhesion failure calculations. Test five specimens of each coated substrate one day and five on a second day. If one specimen differs significantly from the other four at the same time, fails because of an uneven (nonplanar) stud, or for any other reason performs unlike the other four, test a replacement specimen.

Note 6—Examine the stud and specimen carefully. Adhesive should have been applied uniformly to the entire stud surface. Coating should have pulled off uniformly over the entire stud surface either with adhesive failure from the substrate (A) or cohesive failure in the coating (C). If failure is less than 90 % A or C or (or CM), if the adhesive has failed at the stud, retest exercising particular care in the specimen and stud preparation.

NOTE 7—The percentage of adhesive failure at the coating/substrate interface is determined by inspecting the tested area on the substrate. This may be assessed by overlaying a transparent sheet grid marked in 0.10-in. (2.54-mm) squares and estimating the percentage of adhesive failure in each square that lies over the tested area. These percentages may then be averaged to obtain failure for each specimen.

NOTE 8—Sample conditioning, if any, and humidity and temperature of test room may affect results. Notation of these conditions should be made on the test report.

8. Report

8.1 Report the number of tests, the maximum stress obtained in each test, and the type of failure. Test data sheet is shown in Fig. 4. Fill in data sheet with results obtained from each test. Attach tensile test traces to the data sheets. Calculate and report mean and standard deviation for tensile strength for each coating/substrate combination tested.

9. Precision and Bias ⁷

9.1 The precision and bias are primarily dependent upon the accuracy of the force measurements, the alignment of the device, the care exercised in stud and specimen preparation, and the care in testing.

9.2 A round robin involving five different types of coatings and seven different plastic substrates, and ten different laboratories yielded interlaboratory reproducibility data as shown in Table 1. Within laboratory repeatability data is shown in Table 2 and Table 3.

9.3 Bias—No information can be provided on the bias of this test method for measuring adhesion because no material having an accepted reference value is available.

10. Keywords

10.1 adhesion; bond strength; cyanoacrylate adhesive; plastic substrate; pull-off strength; pull testing; tensile tester

⁷ A complete report with additional data is available. See Nelson, G. L., "Testing of EMI/RFI Coating Adhesion to Plastics by a Tensile Test Method," *Final Report for Computer and Business Manufacturers Association and Society of the Plastics Industry*, University of Southern Mississippi, June, 1989.

-	TESTING PROGRAM DATA SHEET									
NAME : COMPANY :							ADDRESS:			
		DAY	ONE:	Date				DAY TWO: Date		
Material:	Run Tensile Str :	Α	B	C	D	E	F	Run A B C D E F Tensile Str:		
	Type Failure <u>:</u>							Type Failure:		
	% Adhesive Fail <u>:</u>							% Adhesive Fail:		
Material:				C				Run A B C D E F Tensile Str <u>:</u>		
	Type Failur <u>e:</u>							Туре Failu <u>re:</u>		
	% Adhesive Fa <u>il:</u>							% Adhesive F <u>ail:</u>		
Material:	Kun Tensile Str :	A	B	с	D	E	F	Run A B C D E F TensileStr:		
	Type Failure:							Type Failure:		
	% Adhesive Fail:							% Adhesive Fail:		

FIG. 4 Coating Adhesion Testing by Direct Tensile Methods