



Designation: D 4541 – 02

Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers¹

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1. Scope

1.1 This test method covers a procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating on rigid substrates such as metal, concrete or wood. The test determines either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or whether the surface remains intact at a prescribed force (pass/fail). Failure will occur along the weakest plane within the system comprised of the test fixture, adhesive, coating system, and substrate, and will be exposed by the fracture surface. This test method maximizes tensile stress as compared to the shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable.

1.2 Pull-off strength measurements depend upon both material and instrumental parameters. Results obtained by each test method may give different results. Results should only be assessed for each test method and not be compared with other instruments. There are five instrument types, identified as Test Methods A-E. It is imperative to identify the test method used when reporting results.

1.3 This test method uses a class of apparatus known as portable pull-off adhesion testers.² They are capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible. Measurements are limited by the strength of adhesion bonds between the loading fixture and the specimen surface or the cohesive strengths of the adhesive, coating layers, and substrate.

1.4 This test can be destructive and spot repairs may be necessary.

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

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:

D 2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding³

D 3933 Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)³

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials⁴

2.2 ANSI Standard:

N512 Protective Coatings (Paints) for the Nuclear Industry⁵

2.3 ISO Standard:

4624 Paints and Varnish—Pull-Off Test for Adhesion⁵

3. Summary of Test Method

3.1 The general pull-off test is performed by securing a loading fixture (dolly, stud) normal (perpendicular) to the surface of the coating with an adhesive. After the adhesive is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension normal to the test surface. The force applied to the loading fixture is then gradually increased and monitored until either a plug of material is detached, or a specified value is reached. When a plug of material is detached, the exposed surface represents the plane of limiting strength within the system. The nature of the failure is qualified in accordance with the percent of adhesive and cohesive failures, and the actual interfaces and layers involved. The pull-off strength is computed based on the maximum indicated load, the instrument calibration data, and the original surface area stressed. Pull-off strength results obtained using different

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² The term adhesion tester may be somewhat of a misnomer, but its adoption by two manufacturers and at least two patents indicates continued usage.

³ Annual Book of ASTM Standards, Vol 15.06.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

devices may be different because the results depend on instrumental parameters (see [Appendix X1](#)).

4. Significance and Use

4.1 The pull-off strength 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 coated surfaces, and evaluating and reporting the results. This test method is applicable to any portable apparatus meeting the basic requirements for determining the pull-off strength of a coating.

4.2 Variations in results obtained using different devices or different substrates with the same coating are possible (see [Appendix X1](#)). Therefore, it is recommended that the type of apparatus and the substrate be mutually agreed upon between the interested parties.

4.3 The purchaser or specifier shall designate a specific test method, that is, A, B, C, D or E, when calling out this standard.

5. Apparatus

5.1 *Adhesion Tester*, commercially available, or comparable apparatus specific examples of which are listed in [Annex A1-Annex A5](#).

5.1.1 *Loading Fixtures*, having a flat surface on one end that can be adhered to the coating and a means of attachment to the tester on the other end.

5.1.2 *Detaching Assembly* (adhesion tester), having a central grip for engaging the fixture.

5.1.3 *Base*, on the detaching assembly, or an annular bearing ring if needed for uniformly pressing against the coating surface around the fixture either directly, or by way of an intermediate bearing ring. A means of aligning the base is needed so that the resultant force is normal to the surface.

5.1.4 Means of moving the grip away from the base in a smooth and continuous a manner as possible so that a torsion free, co-axial (opposing pull of the grip and push of the base along the same axis) force results between them.

5.1.5 *Timer*, or means of limiting the rate of stress to less than 1 MPa/s (150 psi/s) so that the maximum stress is obtained in less than about 100 s. A timer is the minimum equipment when used by the operator along with the force indicator in [5.1.6](#).

NOTE 1—Obtaining the maximum stress in 100 s or less by keeping the maximum rate of shear to less than 1 MPa/s (150 psi/s) is valid for the levels of pull-off strength measured with these types of apparatuses.

5.1.6 *Force Indicator and Calibration Information*, for determining the actual force delivered to the loading fixture.

5.2 *Solvent*, or other means for cleaning the loading fixture surface. Finger prints, moisture, and oxides tend to be the primary contaminants.

5.3 *Fine Sandpaper*, or other means of cleaning the coating that will not alter its integrity by chemical or solvent attack. If any light sanding is anticipated, choose only a very fine grade abrasive (400 grit or finer) that will not introduce flaws or leave a residue.

5.4 *Adhesive*, for securing the fixture to the coating that does not affect the coating properties. Two component epoxies⁶ and acrylics⁷ have been found to be the most versatile.

5.5 *Magnetic or Mechanical Clamps*, if needed, for holding the fixture in place while the adhesive cures.

5.6 *Cotton Swabs*, or other means for removing excess adhesive and defining the adhered area. Any method for removing excess adhesive that damages the surface, such as scoring (see [6.7](#)), must generally be avoided since induced surface flaws may cause premature failure of the coating.

5.7 *Circular Hole Cutter* (optional), to score through to the substrate around the loading fixture.

6. Test Preparation

6.1 The method for selecting the coating sites to be prepared for testing depends upon the objectives of the test and agreements between the contracting parties. There are, however, a few physical restrictions imposed by the general method and apparatus. The following requirements apply to all sites:

6.1.1 The selected test area must be a flat surface large enough to accommodate the specified number of replicate tests. The surface may have any orientation with reference to gravitational pull. Each test site must be separated by at least the distance needed to accommodate the detaching apparatus. The size of a test site is essentially that of the secured loading fixture. At least three replications are usually required in order to statistically characterize the test area.

6.1.2 The selected test areas must also have enough perpendicular and radial clearance to accommodate the apparatus, be flat enough to permit alignment, and be rigid enough to support the counter force. It should be noted that measurements close to an edge may not be representative of the coating as a whole.

6.2 Since the rigidity of the substrate affects pull-off strength results and is not a controllable test variable in field measurements, some knowledge of the substrate thickness and composition should be reported for subsequent analysis or laboratory comparisons. For example, steel substrate of less than 3.2 mm (1/8-in.) thickness usually reduce pull-off strength results compared to 6.4 mm (1/4-in.) thick steel substrates.

6.3 Subject to the requirements of [6.1](#), select representative test areas and clean the surfaces in a manner that will not affect integrity of the coating or leave a residue. Surface abrasion may introduce flaws and should generally be avoided. A fine abrasive (see [5.3](#)) should only be used if needed to remove loose or weakly adhered surface contaminants.

6.4 Clean the loading fixture surface as indicated by the apparatus manufacturer. Failures at the fixture-adhesive interface can often be avoided by treating the fixture surfaces in accordance with an appropriate ASTM standard practice for preparing metal surfaces for adhesive bonding.

⁶ Araldite Adhesive, available from Ciba-Geigy Plastics, Duxford, Cambridge, CB2 4QA, England, Hysol Epoxy Patch Kit 907, available from Hysol Div., The Dexter Corp., Willow Pass Rd., Pittsburg, CA 94565, and Scotch Weld Adhesive 1838B/A, available from 3M, Adhesives, Coatings and Sealers Div., 3M Center, St. Paul, MN 55144, have been found satisfactory for this purpose.

⁷ Versiloc 201 and 204 with accelerator, available from Lord Corp., Industrial Adhesive Div., 2000 W. Grandview Blvd., P.O. Box 10038, Erie, PA 16514, have been found satisfactory for this purpose.

NOTE 2—Guides D 2651 and D 3933 are typical of well-proven methods for improving adhesive bond strengths to metal surfaces.

6.5 Prepare the adhesive in accordance with the adhesive manufacturer's recommendations. Apply the adhesive to the fixture or the surface to be tested, or both, using a method recommended by the adhesive manufacturer. Be certain to apply the adhesive across the entire surface. Position fixture on the surface to be tested. Carefully remove the excess adhesive from around the fixture. (**Warning**—Movement, especially twisting, can cause tiny bubbles to coalesce into large holidays that constitute stress discontinuities during testing.)

NOTE 3—Adding about 1 percent of #5 glass beads to the adhesive assists in even alignment of the test fixture to the surface.

6.6 Based on the adhesive manufacturer's recommendations and the anticipated environmental conditions, allow enough time for the adhesive to set up and reach the recommended cure. During the adhesive set and early cure stage, a constant contact pressure should be maintained on the fixture. Magnetic or mechanical clamping systems work well, but systems relying on tack, such as masking tape, should be used with care to ensure that they do not relax with time and allow air to intrude between the fixture and the test area.

6.7 Scoring around the fixture violates the fundamental in-situ test criterion that an unaltered coating be tested. If scoring around the test surface is employed, extreme care is required to prevent micro-cracking in the coating, since such cracks may cause reduced adhesion values. Scored samples constitute a different test, and this procedure should be clearly reported with the results.

NOTE 4—It is common to score around the test fixture when performing tests on cementitious substrates where the tensile strength of the substrate is significantly lower than either the pull-off or cohesive strength of the coating system.

6.8 Note the approximate temperature and relative humidity during the time of test.

7. Test Procedure

7.1 Test Methods:

7.1.1 *Test Method A — Fixed Alignment Adhesion Tester Type I:*

7.1.1.1 Operate the instrument in accordance with **Annex A1**.

7.1.2 *Test Method B — Fixed Alignment Adhesion Tester Type II:*

7.1.2.1 Operate the instrument in accordance with **Annex A2**.

7.1.3 *Test Method C — Self-Alignment Adhesion Tester Type III:*

7.1.3.1 Operate the instrument in accordance with **Annex A3**.

7.1.4 *Test Method D — Self-Alignment Adhesion Tester Type IV:*

7.1.4.1 Operate the instrument in accordance with **Annex A4**.

7.1.5 *Test Method E — Self-Alignment Adhesion Tester Type V:*

7.1.5.1 Operate the instrument in accordance with **Annex A5**.

7.2 Select an adhesion-tester with a detaching assembly having a force calibration spanning the range of expected values along with its compatible loading fixture. Mid-range measurements are usually the best, but read the manufacturer's operating instructions before proceeding.

7.3 If a bearing ring or comparable device (5.1.3) is to be used, place it concentrically around the loading fixture on the coating surface. If shims are required when a bearing ring is employed, place them between the tester base and bearing ring rather than on the coating surface.

7.4 Carefully connect the central grip of the detaching assembly to the loading fixture without bumping, bending, or otherwise prestressing the sample and connect the detaching assembly to its control mechanism, if necessary. For nonhorizontal surfaces, support the detaching assembly so that its weight does not contribute to the force exerted in the test.

7.5 Align the device according to the manufacturer's instructions and set the force indicator to zero.

NOTE 5—Proper alignment is critical, see **Appendix X2**. If alignment is required, use the procedure recommended by the manufacturer of the adhesion tester and report the procedure used.

7.6 Increase the load to the fixture in as smooth and continuous a manner as possible, at a rate of less than 1 MPa/s (150 psi/s) so that failure occurs or the maximum stress is reached in about 100 s or less (see **Note 1**).

7.7 Record the force attained at failure or the maximum force applied.

7.8 If a plug of material is detached, label and store the fixture for qualification of the failed surface in accordance with **8.3**.

7.9 Report any departures from the procedure such as possible misalignment, hesitations in the force application, etc.

8. Calculation and Interpretation of Results

8.1 If instructed by the manufacturer, use the instrument calibration factors to convert the indicated force for each test into the actual force applied.

8.2 Either use the calibration chart supplied by the manufacturer or compute the relative stress applied to each coating sample as follows:

$$X = 4F/\pi d^2 \quad (1)$$

where:

X = greatest mean pull-off stress applied during a pass/fail test, or the pull-off strength achieved at failure. Both have units of MPa (psi).

F = actual force applied to the test surface as determined in **8.1**, and

d = equivalent diameter of the original surface area stressed having units of inches (or millimetres). This is usually equal to the diameter of the loading fixture.

8.3 For all tests to failure, estimate the percent of adhesive and cohesive failures in accordance to their respective areas and location within the test system comprised of coating and adhesive layers. A convenient scheme that describes the total test system is outlined in **8.3.1** through **8.3.3**. (See **ISO 4624**.)

NOTE 6—A laboratory tensile testing machine is used in ISO 4624.

8.3.1 Describe the specimen as substrate *A*, upon which successive coating layers *B*, *C*, *D*, etc., have been applied, including the adhesive, *Y*, that secures the fixture, *Z*, to the top coat.

8.3.2 Designate cohesive failures by the layers within which they occur as *A*, *B*, *C*, etc., and the percent of each.

8.3.3 Designate adhesive failures by the interfaces at which they occur as *A/B*, *B/C*, *C/D*, etc., and the percent of each.

8.4 A result that is very different from most of the results may be caused by a mistake in recording or calculating. If either of these is not the cause, then examine the experimental circumstances surrounding this run. If an irregular result can be attributed to an experimental cause, drop this result from the analysis. However, do not discard a result unless there are valid nonstatistical reasons for doing so or unless the result is a statistical outlier. Valid nonstatistical reasons for dropping results include alignment of the apparatus that is not normal to the surface, poor definition of the area stressed due to improper application of the adhesive, poorly defined glue lines and boundaries, holidays in the adhesive caused by voids or inclusions, improperly prepared surfaces, and sliding or twisting the fixture during the initial cure. Scratched or scored samples may contain stress concentrations leading to premature fractures. Dixon’s test, as described in Practice D 3980, may be used to detect outliers.

8.5 Disregard any test where glue failure represents more than 50 % of the area. If a pass/fail criterium is being used and a glue failure occurs at a pull-off strength greater than the criterium, report the result as “pass with a pull-off strength >{value obtained}...”

8.6 Further information relative to the interpretation of the test results is given in Appendix X2.

9. Report

9.1 Report the following information:

9.1.1 Brief description of the general nature of the test, such as, field or laboratory testing, generic type of coating, etc.

9.1.2 Temperature and relative humidity and any other pertinent environmental conditions during the test period.

9.1.3 Description of the apparatus used, including: apparatus manufacturer and model number, loading fixture type and dimensions, and bearing ring type and dimensions.

9.1.4 Description of the test system, if possible, by the indexing scheme outlined in 8.3 including: product identity and generic type for each coat and any other information supplied, the substrate identity (thickness, type, orientation, etc.), and the adhesive used.

9.1.5 Test results.

9.1.5.1 Date, test location, testing agent.

9.1.5.2 For pass/fail tests, stress applied along with the result, for example, pass or fail and note the plane of any failure (see 8.3 and ANSI N512).

9.1.5.3 For tests to failure, report all values computed in 8.2 along with the nature and location of the failures as specified in 8.3, or, if only the average strength is required, report the average strength along with the statistics.

9.1.5.4 If corrections of the results have been made, or if certain values have been omitted such as the lowest or highest values or others, reasons for the adjustments and criteria used.

9.1.5.5 For any test where scoring was employed, indicate it by placing a footnote superscript beside each data point affected and a footnote to that effect at the bottom of each page on which such data appears. Note any other deviations from the procedure.

10. Precision and Bias ⁸

10.1 *Precision*—In an interlaboratory study of Test Methods A–D, operators made measurements, generally in triplicate but in a few cases in duplicate, on coated panels covering a moderate range at the intermediate adhesion level using four different types of instruments (see Annex A1–Annex A5 and Appendix X1). The number of participating laboratories varied with each instrument and in the case of one instrument with the material. Only two laboratories had access to Type I instruments but two operators in each made the triplicate tests. During the statistical analysis of the results three individual results and one set of triplicates, obtained with Type II instruments were rejected as outliers; one single test with Type III instruments and three single results with Type I instruments were rejected. The pooled intra- and inter-laboratory coefficients of variation were found to be those shown in Table 1. Based on these coefficients the following criteria should be used for judging, at the 95 % confidence level, the acceptability of results:

10.1.1 *Replicate Repeatability*—Triplicate results obtained by the same operator using instruments from the same category should be considered suspect if they differ in percent relative by more than the values given in Table 1.

NOTE 7—Difference in percent relative to two results, x_1 and x_2 , is the absolute value of

$$\frac{(x_1 - x_2)}{(x_1 + x_2)/2} \times 100. \tag{2}$$

10.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by operators in different laboratories using

⁸ Supporting data are available from ASTM International Headquarters. Request RR: D01-1094.

TABLE 1 Precision of Adhesion Pull-Off Measurements

Instrument	Coefficient of Variation, v, %	Degrees of Freedom	Maximum Acceptable Difference, %
Intralaboratory Instrument:			
Type IV	8.5	48	29.0
Type I			
Type II			
Type III			
Total		177	
Interlaboratory Instrument:			
Type IV	8.7	20	25.5
Type I			
Type II			
Type III			
Total		78	

instruments of the same category should be considered suspect if they differ in percent relative by more than the values given in **Table 1**.

10.2 *Bias*—This test method has no bias statement since there is no acceptable reference material suitable for determining the bias of this test method.

11. Keywords

11.1 adhesion; coatings; field; paint; portable; pull-off strength; tensile test

ANNEXES

(Mandatory Information)

A1. FIXED-ALIGNMENT ADHESION TESTER, TYPE I

A1.1 Apparatus:

A1.1.1 A fixed-alignment portable tester as shown in **Fig. A1.1**⁹

NOTE A1.1—Precision data for Type I instruments described in **Table 1** were obtained using the devices illustrated in **Fig. A1.1**.

A1.1.2 The tester is comprised of detachable aluminum loading fixtures, 50 mm (1.97 in.) in diameter, screws with spherical heads that are screwed into the center of a fixture, a socket in the testing assembly that holds the head of the screw, pressure gage, dynamometer, wheel and crank.

A1.1.3 The testers are available in four models, with maximum tensile forces of 5, 15, 25, and 50 kN (1125, 3375, 5625, and 11 250 lb) respectively. For a fixture having a 50 mm (1.97 in.) diameter, a 5 kN device corresponds to a range of 2.5 MPa (0 to 360 psi).

⁹ The Dyna Z5 tester is available from PROCEQ SA, Riesbachstrasse 57, CH-8034, Zurich, Switzerland.

A1.2 Procedure:

A1.2.1 Follow the general procedures described in Sections 6 and 7. Procedures specific to this instrument are described in this section.

A1.2.2 Set the pointer on the zero mark by first pressing the push-button located on the left of the indicator. While holding the push-button, turn the little knob located on the upper part of the indicator to set the pointer at zero. Set the zero after testing by pressing the push-button.

A1.2.3 After fixing a loading fixture to a substrate, insert a screw with a spherical head into the center of the fixture. Position the testing equipment on the metal disc. Then by means of the notched wheel, fix the head of the spherical screw into the socket at the base of the equipment. For the first mechanical approach, stop screwing down the wheel when the pointer on the indicator shifts from the ZERO mark. Tests are done by turning the crank. After each test, turn the crank in the opposite direction until it stops.

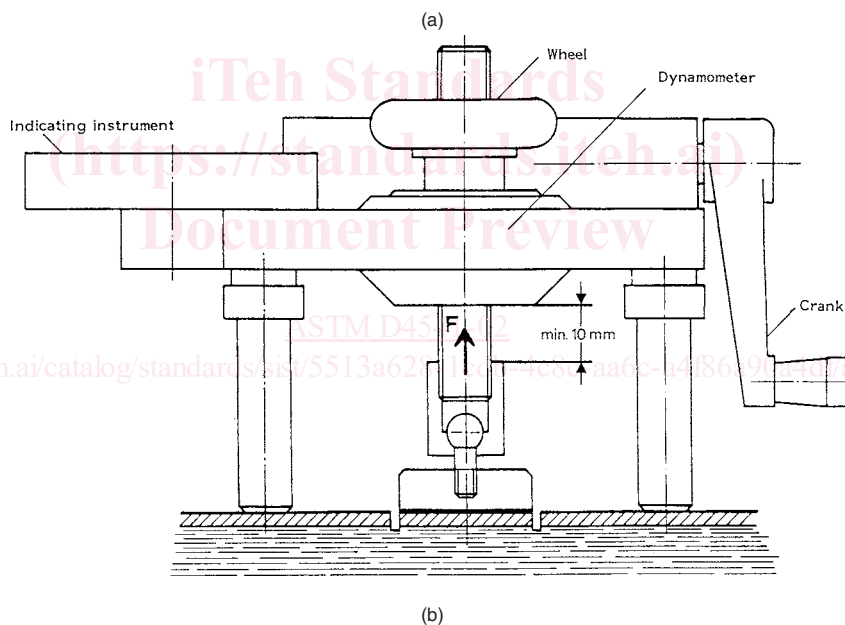


FIG. A1.1 Photograph (a) and sketch (b) of Type I instruments

A2. FIXED-ALIGNMENT ADHESION TESTER TYPE II

A2.1 Apparatus:

A2.1.1 This is a fixed-alignment portable tester, as shown in Fig. A2.1.¹⁰

NOTE A2.1—Precision data for Table 1 were obtained using the devices illustrated in Fig. A2.1.

¹⁰ The Elcometer, Model 106, adhesion tester is available from Elcometer Instruments, Ltd., Edge Lane, Droylston, Manchester M35 6UB, United Kingdom, England.

A2.1.2 The tester is comprised of detachable aluminum loading fixtures having a flat conic base that is 20 mm (0.8 in.) in diameter on one end for securing to the coating, and a circular T-bolt head on the other end, a central grip for engaging the loading fixture that is forced away from a tripod base by the interaction of a handwheel (or nut), and a coaxial bolt connected through a series of belleville washers, or springs in later models, that acts as both a torsion relief and a spring that displaces a dragging indicator with respect to a scale.