

Designation: <del>D7234 - 05</del> D7234 - 12

# Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers<sup>1</sup>

This standard is issued under the fixed designation D7234; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 This test method covers procedures for evaluating the pull-off adhesion strength of a coating on concrete. The test determines the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached. 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.
- 1.2 This test method uses a class of apparatus known as portable pull-off adhesion testers.<sup>2</sup> 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, coating system and the substrate or the cohesive strengths of the adhesive, coating layers, and substrate.
- 1.3 Pull-off adhesion strength measurements depend upon both material and instrumental parameters. There are different instruments used that comply with this test method. The specific instrument used should be identified when reporting results. This test is destructive and spot repairs may be necessary.
  - 1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.5 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:<sup>3</sup>

C1583 Test Method for Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair and Overlay Materials by Direct Tension (Pull-off Method)

D16 Terminology for Paint, Related Coatings, Materials, and Applications 2a-a2d8-1ba935caaa2a/astm-d7234-12

D2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

D3933 Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)

D4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E178 Practice for Dealing With Outlying Observations

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

## 3. Terminology

- 3.1 The terms and definitions in Terminology D16 apply to this test method.
- 3.2 Definitions:
- 3.2.1 *adhesive*, *n*—(with respect to this test method) adhesive refers to the material that bonds the bottom of the loading fixture to the top surface of the coating to be tested.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatingss.

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<sup>&</sup>lt;sup>2</sup> The term adhesion tester may be somewhat of a misnomer, but its adoption by two manufacturers and at least two patents indicates continued usage.

<sup>&</sup>lt;sup>3</sup> 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* volume information, refer to the standard's Document Summary page on the ASTM website.



- 3.2.2 *loading fixture*, *n*—(also referred to as dollies or studs) a metal structure that is flat on one end for bonding to the coating surface and shaped on the other end for attachment to the adhesion tester and is used to determine the pull-off adhesion strength of eoatings coatings.
- 3.2.3 portable pull-off adhesion testers, n—instruments that 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.

## 4. Summary of Test Method

4.1 The general pull-off adhesion test is performed by scoring through the coating down to the surface of the concrete substrate at a diameter equal to the diameter of the loading fixture (dolly, stud), and securing the loading fixture 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 uniformly increased and monitored until a plug of material is detached. 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 adhesion strength is computed based on the maximum indicated load, the instrument calibration data, and the surface area stressed. Pull-off adhesion strength results obtained using different devices may be different because the results depend on instrumental parameters.

### 5. Significance and Use

- 5.1 The pull-off adhesion strength and mode of failure of a coating from a concrete substrate are important performance properties that are used in specifications. This test method serves as a means for uniformly preparing and testing coated surfaces, and evaluating and reporting the results.
- 5.2 Variations in strength results obtained using different instruments, different substrates, or different loading fixtures with the same coating are possible. Therefore, it is recommended that the specific test instrument and loading fixture be mutually agreed upon between the interested parties.
- 5.3 This test method should not be used to determine surface strength of uncoated concrete. Test Method C1583 is suitable for that determination.

# 6. Apparatus

- 6.1 Adhesion Tester, including the components and accessories described in 6.1.1-6.1.5
- 6.1.1 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. The bonding surface may be round, square or rectangular. The round loading fixtures are usually 50 mm (2.0 in) in diameter but may range from 20 mm (0.75 in) to 75 mm (3.0 in) in diameter.
  - 6.1.2 Detaching Assembly, having a central grip for engaging the loading fixture.
- 6.1.3 *Base*, on the detaching assembly, 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.
- 6.1.4 *Force Applicator*, means of moving the grip away from the base in as 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.
- 6.1.5 Force Indicator and Calibration Information, for determining the actual force delivered to the loading fixture. The force indicator shall be verified to be within +/-5 % of the force measured by a calibrated testing machine at a frequency determined by the user, typically once a year.
- 6.2 *Timer*, or means of limiting the rate of stress to less than <u>or equal to 0.2 MPa/s</u> (30 psi/s) so that the maximum stress (failure) is obtained in about 5 to 30 s.
  - 6.3 Solvent, or other means for cleaning the loading fixture surface.
  - 6.4 Fine Sandpaper, or other means of cleaning or preparing the coating that will not alter its integrity.
- 6.5 *Adhesive*, for securing the fixture to the coating that does not affect the coating properties. Two-component epoxies and acrylics<sup>4</sup> have been found to be the most versatile.
  - 6.6 Mechanical Clamps, if needed, for holding the fixture in place while the adhesive cures.
  - 6.7 Cotton Swabs, or other means for removing excess adhesive.
- 6.8 Core Bit with Drill Press or Hand Drill, and means to ensure that the scoring is normal to the coating for the procedures that use a round loading fixture. The core bit inside diameter should equal the diameter of the loading fixture. If a core bit with an inside diameter equal to the diameter of the loading fixture is not available, the closest size available should be used. The core

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the acrylics known to the committee at this time is Versiloc 201 and 204 with accelerator, available from Lord Corp., Industrial Adhesive Div., 2000 W. Grandview Blvd., P.O. Box 10038, Erie, PA 16514. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, <sup>1</sup> which you may attend.

bit or saw blades should be diamond tipped and, when required to minimize heat and suppress dust, supplemented with water lubrication. For the test procedures that use a square or rectangular loading fixture, a circular saw is required instead of a core bit and drill. Alternately, for thin or elastomeric coatings, a sharp knife or hole saw may be sufficient to score around the loading fixture.

# 7. Test Preparation

- 7.1 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:
- 7.1.1 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 required in order to statistically characterize the test area.
- 7.1.2 The selected test areas must also have enough perpendicular and radial clearance to accommodate the apparatus, and be flat enough to permit alignment. It should be noted that measurements close to an edge may not be representative of the coating as a whole.
- 7.2 Scoring the coating down to the surface of the substrate is required for all coatings thicker than 0.5 mm (20 mils) and for all reinforced or elastomeric coatings. While scoring is recommended for coatings thinner than 0.5 mm (20 mils), the test may be performed without scoring, but the results should note this exception. Scoring shall be performed in a manner that ensures the cut is made normal to the coating surface and in a manner that does not twist or torque the test area and minimizes heat generated and edge damage or microcracks to the coating and the concrete substrate. For thick coatings it is recommended to cool the coating and substrate during the cutting process with water lubrication. When using a round loading fixture, scoring is performed before the loading fixture is attached (see Fig. 1). When using square or rectangular loading fixtures, scoring is typically performed after the loading fixture is attached (see Fig. 2).
- 7.3 Clean the surfaces in a manner that will not affect integrity of the coating or leave a residue. 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. Fingerprints, moisture, and oxides tend to be the primary contaminants.

Note 1—Guides D2651 and D3933 are typical of well-proven methods for improving adhesive bond strengths to metal surfaces.

7.4 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

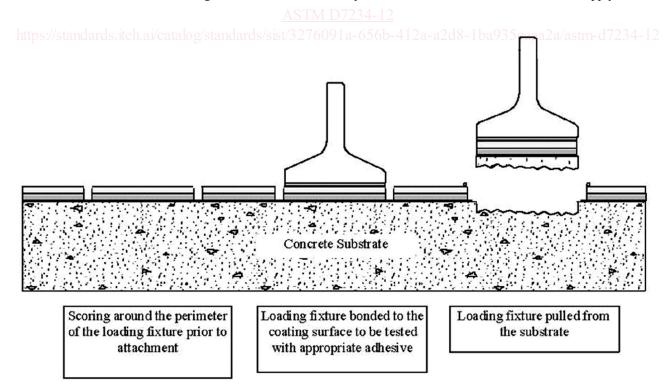


FIG. 1 Scoring Around the Loading Fixture Prior to Attachment of the Fixture (Round Loading Fixtures)

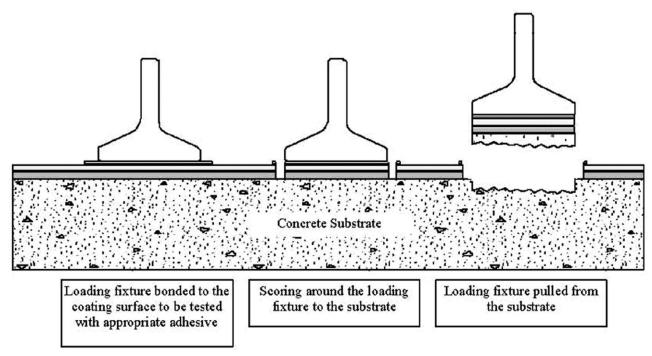


FIG. 2 Scoring Around the Loading Fixture After Attachment of the Fixture (Square or Rectangular Fixtures)

across the entire surface. Position fixture on the surface to be tested centered directly over the scored section with the fixture outer sides lined up with the inside circumference of the scored section. 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 2—Adding about 1 percent of #5 glass beads to the adhesive assists in even alignment of the test fixture to the surface.

7.5 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. 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.

7.6 Note the temperature and relative humidity during the time of test.

## 8. Test Procedure

- 8.1 Select an adhesion-tester 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.
- 8.2 If a bearing ring or comparable device 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.
- 8.3 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.
  - 8.4 Align the device according to the manufacturer's instructions and set the force indicator to zero.
  - Note 3—Proper alignment is critical. If alignment is required, use the procedure recommended by the manufacturer of the adhesion tester.
- 8.5 Increase the load to the fixture in as smooth and continuous a manner as possible, at a uniform rate of less than <u>or equal</u> to 0.2 MPa/s (30 psi/s) so that failure occurs or the maximum stress is reached before 30 s.
  - 8.6 Record the force attained at failure.
- 8.7 When the plug of material is detached, label and store the fixture for qualification of the failed surface in accordance with 9.3.
  - 8.8 Report any departures from the procedure such as possible misalignment, hesitations in the force application, etc.



# 9. Calculation and Interpretation of Results

- 9.1 If provided by the manufacturer, use the instrument calibration factors to convert the indicated force for each test into the actual force applied.
- 9.2 Either use the calibration chart supplied by the manufacturer or compute the relative stress applied to each coating sample as follows:

$$X = \frac{4F}{d^2} \tag{1}$$

where:

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- X = pull-off adhesion strength achieved at failure in MPa (psi).
- $F = \text{Maximum force applied to the test surface at failure and as determined in 9.1 in <math>N(lb_f)$ , and
- $F = Maximum force applied to the test surface at failure and as determined in 9.1 in <math>N(lb_t)$ , and
- d = diameter of the loading fixture in mm (in.).

Note 4—d should be the inside diameter of the scored sample if this does not equal the diameter of the loading fixture.

- 9.3 Estimate the percent of adhesive and cohesive failures in accordance to their respective areas and location within the test system comprised of substrate, coating and adhesive layers. A convenient scheme that describes the total test system is outlined in 9.3.1-9.3.4.
- 9.3.1 Describe the specimen as substrate A, B, C, upon which successive coating layers D, E, F, etc., have been applied, including the adhesive, Y, that secures the fixture, Z, to the top coat.
  - 9.3.2 Designate cohesive substrate failures by the quantity and type of substrate removed (see Fig. 3).
  - 9.3.3 Designate cohesive coating failures by the layers within which they occur as D, E, F, etc., and the percentage of each.
  - 9.3.4 Designate adhesive failures by the interfaces at which they occur as A/B, B/C, C/D, etc., and the percent of each.
- 9.4 A result that is very different from most of the results may be caused by a mistake in recording or calculating, among other things. 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

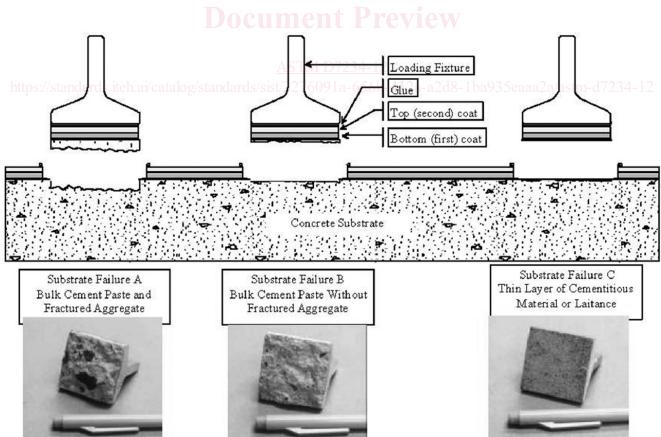


FIG. 3 Substrate Failure Classification

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. Dixon's test, as described in Practice E178, may be used to detect outliers.

9.5 Disregard any test where adhesive failure (between the adhesive, Y, and the loading fixture, Z or the coating surface) represents more than 20 % of the area.

#### 10. Report

- 10.1 Report the following information:
- 10.1.1 Brief description of the general nature of the test, such as, field or laboratory testing, generic type of coating, etc.
- 10.1.2 Temperature and relative humidity and any other pertinent environmental conditions during the test period.
- 10.1.3 Description of the apparatus used, including: apparatus manufacturer and model number, loading fixture type and dimensions, and bearing ring type and dimensions.
- 10.1.4 Description of the test system, if possible, by the indexing scheme outlined in 9.3 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.
  - 10.1.5 Test results, including:
  - 10.1.5.1 Date, test location, testing agent.
- 10.1.5.2 Report all values computed in 9.2 along with the nature and location of the failures as specified in 9.3. Report the average % failure for each mode of failure, and the average pull-off adhesion strength for each predominant mode of failure, rounded to the nearest 0.1 MPa (10 psi).
- 10.1.5.3 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.
- 10.1.5.4 For any test where scoring was not 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.

#### 11. Precision and Bias

an interlaboratory study of Test Method D7234been completed and is awaiting publication. The average coefficient of variation (standard deviation/average pull-off adhesion values) for the pooled data set (different instruments, loading fixture sizes, and loading rates, but same mode of failure) was 15 %. Some instruments have been tested for precision using coatings on steel substrates. These results are reported in Test Method, Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers, conducted in 2011. Six analysts, using seven different instruments, tested samples of two coatings prepared on the same substrate. Every analyst reported three test results for each instrument/coating combination in this study. While the test results are representative of individual determinations, the data was combined for analysis

TABLE 1 Pull-Off Adhesion Measurements for Instruments 1 to 7 Separately

Coating – Instrument	Average <sup>A</sup>	Repeatability Standard	Reproducibility Standard	Repeatability <u>Limit</u>	Reproducibility Limit
	_	Deviation	Deviation	<u>(2σ)</u>	<u>(2σ)</u>
	X	<u>s<sub>r</sub></u>	$\underline{S_{R}}$	<u>r</u>	<u>R</u>
1–1	270.1	44.0	62.5	87.9	125.1
1–2	219.7	44.7	46.9	89.3	93.7
1–3	243.7	32.4	67.0	64.7	133.9
1-4	271.4	60.9	60.9	121.7	<u>121.7</u>
1-2 1-3 1-4 1-5 1-6 1-7	264.4	52.9	<u>63.0</u>	<u>105.9</u>	126.1
1–6	293.3	44.7	50.4	89.4	100.7
1–7	182.5	35.8	46.0	71.6	91.9
2–1	365.3	47.4	61.4	94.9	122.7
2–2	303.9	30.5	37.4	61.0	74.8
2–3	399.8	38.8	61.9	77.5	123.8
2–4	404.4	58.3	58.3	116.6	116.6
2-1 2-2 2-3 2-4 2-5 2-6 2-7	361.4	53.2	57.0	106.4	114.0
2–6	457.8	75.0	75.0	150.0	150.0
2–7	228.3	64.0	87.4	128.1	174.9

<sup>A</sup> The average of the laboratories' calculate averages.