Designation: D4541 – 09$^{\varepsilon 1}$

Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[1]

This standard is issued under the fixed designation D4541; 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.

ε[1] NOTE—Practice D3980 was deleted from Section 2 in August 2010.

1. Scope*

1.1 This test method covers a procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating system from metal substrates. Pull-off strength of coatings from concrete is described in Test Method D7234. 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.

NOTE 1—The procedure in this standard was developed for metal substrates, but may be appropriate for other rigid substrates such as plastic and wood. Factors such as loading rate and flexibility of the substrate must be addressed by the user/specifier.

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 B-F. It is imperative to identify the test method used when reporting results.

NOTE 2—Method A, which appeared in previous versions of this standard, has been eliminated as its main use is for testing on concrete substrates (see Test Method D7234).

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:*[3]

D2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

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

D7234 Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers

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

2.2 *ANSI Standard:*

N512 Protective Coatings (Paints) for the Nuclear Industry[4]

2.3 *ISO Standard:*

ISO 4624 Paints and Varnish—Pull-Off Test for Adhesion[4]

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

[1] 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 Coatings.

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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] 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 standar's Document Summary page on the ASTM website.

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

*A Summary of Changes section appears at the end of this standard 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 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 Section 10). 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, B, C, D, E, or F 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 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.

5.1.5 *Timer,* or means of limiting the loading rate to 1 MPa/s (150 psi/s) or less for a 20 mm loading fixture so that the test is completed in about 100 s or less. A timer is the minimum equipment when used by the operator along with the force indicator in 5.1.6.

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*[5], for securing the fixture to the coating that does not affect the coating properties. Two component epoxies and acrylics 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 reduces 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. To reduce the risk of glue failures, the surface of the coating can be lightly abraded to promote adhesion of the adhesive to the surface. If the

[5] Scotch Weld 420, available from 3M, Adhesives, Coatings and Sealers Div., 3M Center, St. Paul, MN 55144, was used in the round robin.

surface is abraded, care must be taken to prevent damage to the coating or significant loss of coating thickness. Solvent clean the area to remove particulates after abrading. Select a solvent that does not compromise the integrity of the coating.

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.

NOTE 3—Guides D2651 and D3933 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 4—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. Scoring is only recommended for thicker-film coatings, that is, thicknesses greater than 500 µm (20 mils), reinforced coatings and elastomeric coatings. Scoring, if performed, shall be done 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 substrate. For thick coatings it is recommended to cool the coating and substrate during the cutting process with water lubrication.

NOTE 5—A template made from plywood with a hole of the same size drilled through it has been found to be an effective method to limit sideways movement of the drill bit.

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 (discontinued).*

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

7.1.2.1 Operate the instrument in accordance with Annex A1.

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

7.1.3.1 Operate the instrument in accordance with Annex A2.

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

7.1.4.1 Operate the instrument in accordance with Annex A3.

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

7.1.5.1 Operate the instrument in accordance with Annex A4.

7.1.6 *Test Method F — Self-Alignment Adhesion Tester Type VI:*

7.1.6.1 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 6—Proper alignment is critical, see Appendix X1. 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 1 MPa/s (150 psi/s) or less for a 20 mm loading fixture so that the test is completed in about 100 s or less.

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 or 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 7—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.

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 X1.

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[6,7]

10.1 The precision of this test method is based on an interlaboratory study of Test Method D4541 conducted in 2006. Analysts from seven laboratories tested six different coatings applied to ¼ in. thick hot-rolled carbon steel plates using five different adhesion testers. Every "test result" represents an individual determination. In order to standardize and balance the data, any pull which exceeded the tester's upper limit with the available accessories at the time of testing was eliminated from the statistical analysis. Any pull in which there was 50 % or more glue failure was also eliminated from the statistical analysis. If four valid pulls were obtained from one operator for a given material, the fourth was eliminated and the first three valid replicate test results (from one operator) for each material were included in the statistical analysis. Practice E691 was followed for the design and analysis of the data; the details are given in Research Report No. D01–1147.

NOTE 8—The pull-off strength of two of the coatings, identified during the round robin as Coating A and Coating F, exceeded the measurement limits of the testers with the accessories available at the time of testing, and were therefore eliminated from the statistical analysis.

10.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1094. Contact ASTM Customer Service at service@astm.org.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1147. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Adhesion Testing Method B, Pull-Off Strength (*psi*)

Coating	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x̄	sr	sR	r	R
B	1195	278	330	777	925
C	549	109	117	305	326
D	1212	412	483	1155	1351
E	1385	192	276	537	774

Coating	Average	Repeatability Limit		Reproducibility Limit	
	x̄	r	% of average	R	% of average
B	1195	777	69.1	925	77.4
C	549	305	55.6	326	59.0
D	1212	1155	95.3	1351	111.5
E	1385	537	38.8	774	55.9
Avg.			64.7		76.0

TABLE 2 Adhesion Testing Method C, Pull-Off Strength (*psi*)

Coating	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x̄	sr	sR	r	R
B	1974	261	324	732	907
C	1221	136	548	382	1535
D	2110	252	316	706	886
E	2012	239	359	669	1004

Coating	Average	Repeatability Limit		Reproducibility Limit	
	x̄	r	% of average	R	% of average
B	1974	732	37.1	907	45.9
C	1221	382	31.3	1535	125.7
D	2110	706	33.5	886	42.0
E	2012	669	33.3	1004	49.9
Avg.			30.4		70.5

TABLE 3 Adhesion Testing Method D, Pull-Off Strength (*psi*)

Coating	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x̄	sr	sR	r	SR
B	2458	146	270	408	755
C	1232	31	116	87	324
D	2707	155	233	434	651
E	2354	163	273	456	764

Coating	Average	Repeatability Limit		Reproducibility Limit	
	x̄	r	% of average	R	% of average
B	2458	408	16.6	755	30.7
C	1232	87	7.1	324	26.3
D	2707	434	16.0	651	24.0
E	2354	456	19.4	764	32.5
Avg.			14.8		28.4

representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.1.1 Repeatability limits are listed in Tables 1-5.

10.1.2 *Reproducibility*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

TABLE 4 Adhesion Testing Method E, Pull-Off Strength (*psi*)

Coating	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x̄	sr	sR	r	SR
B	2210	173	215	483	601
C	1120	115	155	321	433
D	2481	361	422	1011	1181
E	2449	173	198	485	555

Coating	Average	Repeatability Limit		Reproducibility Limit	
	x̄	r	% of average	R	% of average
B	2210	483	21.9	601	27.2
C	1120	321	28.7	433	38.7
D	2481	1011	40.7	1181	47.6
E	2449	485	19.8	555	22.7
Avg.			27.8		34.1

TABLE 5 Adhesion Testing Method F, Pull-Off Strength (*psi*)

Coating	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	x̄	sr	sR	r	SR
B	2070	102	125	287	351
C	1106	60	108	169	304
D	2368	124	160	347	449
E	2327	217	237	609	664

Coating	Average	Repeatability Limit		Reproducibility Limit	
	x̄	r	% of average	R	% of average
B	2070	287	13.9	351	17.0
C	1106	169	15.3	304	27.5
D	2368	347	14.7	449	19.0
E	2327	609	26.2	664	28.5
Avg.			17.5		23.0

10.1.2.1 Reproducibility limits are listed in Tables 1-5.

10.1.3 Any judgment in accordance with these two statements would have an approximate 95 % probability of being correct.

10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement is being made.

10.3 The precision statement was determined through statistical examination of 394 results, produced by analysts from seven laboratories, on four coatings, using five different instruments. Different coatings were used as a means to achieve a range of pull-off strengths covering the operating range of all the instruments.

10.3.1 Results obtained by the same operator using instruments from the same Method should be considered suspect if they differ in percent relative by more than the Intralaboratory values given in Table 6. Triplicate results obtained by different operators using instruments from the same Method should be considered suspect if they differ in percent relative by more than the Interlaboratory values given in Table 6.

11. Keywords

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

TABLE 6 Precision of Adhesion Pull-Off Measurements (averaged across coating types for each instrument)

Intralaboratory	Maximum Recommended Difference, %	Interlaboratory	Maximum Recommended Difference, %
Method B	64.7	Method B	76.0
Method C	33.8	Method C	65.9
Method D	14.8	Method D	28.4
Method E	27.8	Method E	34.1
Method F	17.5	Method F	23.0

ANNEXES

(Mandatory Information)

A1. FIXED-ALIGNMENT ADHESION TESTER TYPE II (TEST METHOD B)

A1.1 *Apparatus:*

A1.1.1 This is a fixed-alignment portable tester, as shown in Fig. A1.1.[8,9]

NOTE A1.1—Precision data for Type II instruments shown in Table 6 were obtained using the devices described in Fig. A1.1.

A1.1.2 *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 hand wheel (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.*

A1.1.3 The force is indicated by measuring the maximum spring displacement when loaded. Care should be taken to see that substrate bending does not influence its final position or the actual force delivered by the spring arrangement.

A1.1.4 The devices are available in four ranges: From 3.5, 7.0, 14, and 28 MPa (0 to 500, 0 to 1000, 0 to 2000, and 0 to 4000 psi).

A1.2 *Procedure:*

A1.2.1 Center the bearing ring on the coating surface concentric with the loading fixture. Turn the hand wheel or nut of the tester counterclockwise, lowering the grip so that it slips under the head of the loading fixture.

A1.2.2 Align or shim the three instrument swivel pads of the tripod base so that the instrument will pull perpendicularly to the surface at the bearing ring. The annular ring can be used on flexible substrates.

A1.2.3 Take up the slack between the various members and slide the dragging (force) indicator located on the tester to zero.

A1.2.4 Firmly hold the instrument with one hand. Do not allow the base to move or slide during the test. With the other hand, turn the hand wheel clockwise using as smooth and constant motion as possible. Do not jerk or exceed a stress rate of 150 psi/s (1 MPa/s) that is attained by allowing in excess of 7 s/7 MPa (7 s/1000 psi), stress. If the 14 or 28 MPa (2000 or 4000 psi) models are used, the hand wheel is replaced with a nut requiring a wrench for tightening. The wrench must be used in a plane parallel to the substrate so that the loading fixture will not be removed by a shearing force or misalignment, thus negating the results. The maximum stress must be reached within about 100 s.

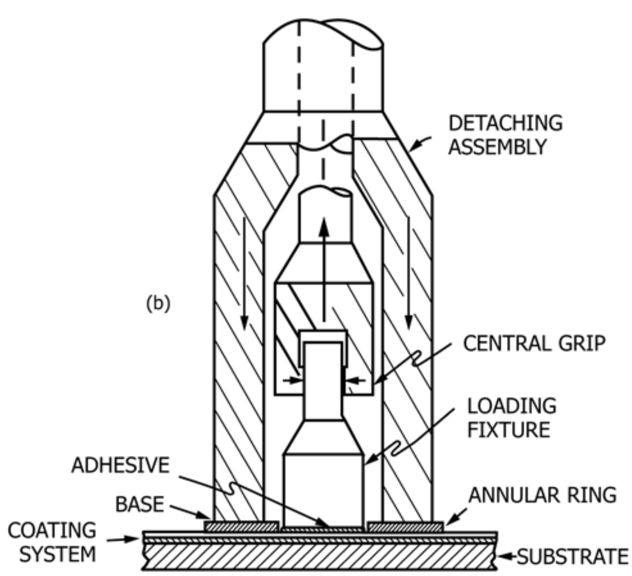
A1.2.5 The pulling force applied to the loading fixture is increased to a maximum or until the system fails at its weakest locus. Upon failure, the scale will rise slightly, while the dragging indicator retains the apparent load. The apparatus scale indicates an approximate stress directly in pounds per square inch, but may be compared to a calibration curve.

A1.2.6 Record the highest value attained by reading along the bottom of the dragging indicator.

[8] The sole source of supply of the Elcometer, Model 106, adhesion tester known to the committee at this time is Elcometer Instruments, Ltd., Edge Lane, Droylston, Manchester M35 6UB, United Kingdom, England.

[9] If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend

(a)



(b)

FIG. A1.1 Photograph (a) and Schematic (b) of Type II, Fixed Alignment Pull-Off Tester

A2. SELF-ALIGNING ADHESION TESTER TYPE III (TEST METHOD C)

A2.1 *Apparatus:*

A2.1.1 This is a self-aligning tester, as shown in Fig. A2.1.[10,9]

NOTE A2.1—Precision data for Type III instruments shown in Table 6 were obtained using the devices described in Fig. A2.1.

A2.1.2 Load is applied through the center of the loading fixture by a hydraulic piston and pin. The diameter of the piston bore is sized so that the area of the bore is equal to the net area of the loading fixture. Therefore, the pressure reacted by the loading fixture is the same as the pressure in the bore and is transmitted directly to a pressure gauge.

A2.1.3 The apparatus is comprised of: a loading fixture, 19 mm (0.75 in.) outside diameter, 3 mm (0.125 in.) inside diameter, hydraulic piston and pin by which load is applied to the loading fixture, hose, pressure gauge, threaded plunger and handle.

A2.1.4 The force is indicated by the maximum hydraulic pressure as displayed on the gauge, since the effective areas of the piston bore and the loading fixture are the same.

A2.1.5 The testers are available in three standard working ranges: 0 to 10 MPa (0 to 1500 psi), 0 to 15 MPa (0 to 2250 psi), 0 to 20 MPa (0 to 3000 psi). Special loading fixtures shaped to test tubular sections are available.

A2.2 *Procedure:*

A2.2.1 Follow the general procedures described in Sections 6 and 7. Procedures specific to this instrument are described in this section.

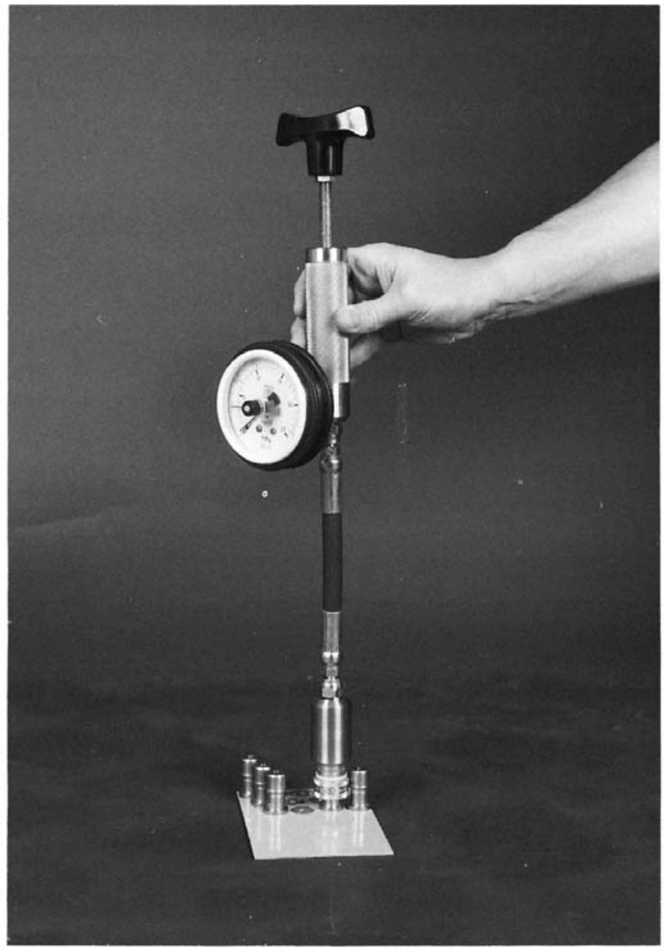
A2.2.2 Insert a decreased TFE-fluorocarbon plug into the loading fixture until the tip protrudes from the surface of the loading fixture. When applying adhesive to the loading fixture, avoid getting adhesive on the plug. Remove plug after holding the loading fixture in place for 10 s.

A2.2.3 Ensure that the black needle of the tester is reading zero. Connect a test loading fixture to the head and increase the pressure by turning the handle clockwise until the pin protrudes from the loading fixture. Decrease pressure to zero and remove the test loading fixture.

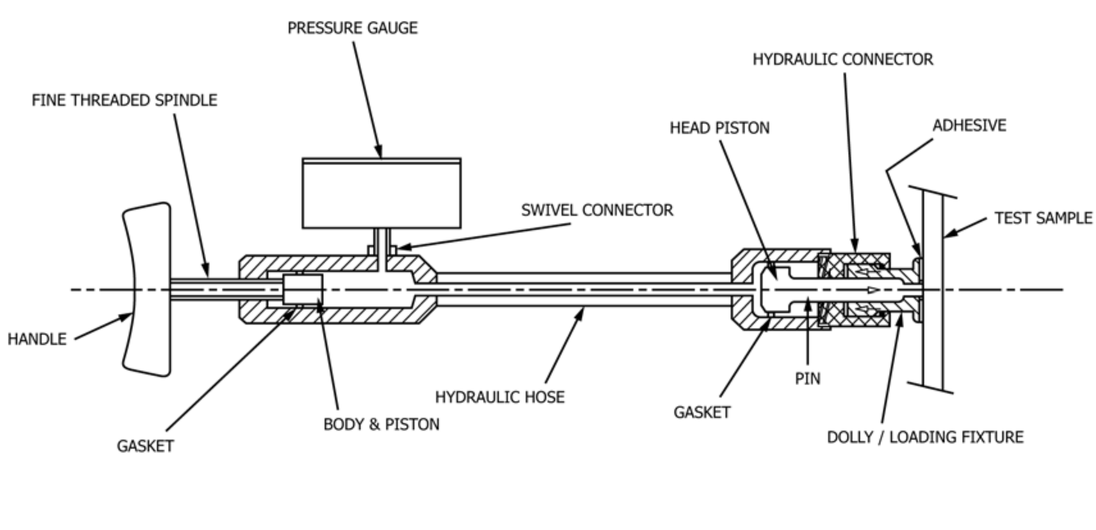
A2.2.4 Connect the head to the loading fixture to be tested, by pulling back the snap-on ring, pushing the head and releasing the snap-on ring. Ensure the tester is held normal to the surface to be tested and that the hose is straight.

A2.2.5 Increase the pressure slowly by turning the handle clockwise until either the maximum stress or failure is reached.

[10] The sole source of supply of the Hate Mark VII adhesion tester known to the committee at this time is Hydraulic Adhesion Test Equipment, Ltd., 629 Inlet Rd., North Palm Beach, FL 33408.



(a)



(b)

FIG. A2.1 Photograph (a) and Schematic (b) of Type III, Self-Alignment Tester

A3. SELF-ALIGNMENT ADHESION TESTER TYPE IV (TEST METHOD D)

A3.1 *Apparatus:*

A3.1.1 This is a self-aligning automated tester, which may have a self-contained pressure source and has a control module that controls a choice of different load range detaching assemblies, or pistons. It is shown in Fig. A3.1.

NOTE A3.1—Precision data for Type IV instruments shown in Table 6 were obtained using the devices described in Fig. A3.1.

A3.1.2 The apparatus is comprised of: (*1*) a loading fixture, (*2*) a detaching assembly, or piston, (*3*) one of several control modules, and (*4*) a pressurized air source.

A3.1.3 The loading fixtures are available on many different sizes (3 to 75 mm) based on the particulars of the system being tested. The standard loading fixture is 12.5 mm (0.5 in) in diameter. The face of the loading fixture can be rough, smooth, curved, machined, etc.

A3.1.4 The pistons are also available in several different sizes, or load ranges. It is recommended that a piston is chosen so that the midpoint of the range is close to the suspected tensile strength of the coating to be tested. This will provide the most forgiveness in errors of assumed coating strength.

A3.1.5 Several models of control modules are available. The digital models may include optional accessories allowing for features such as wireless real-time transmission of pull-tests via Bluetooth and your PC, LabVIEW-created software, USB camera attachment to photo document your pulls, and computer generated reporting capabilities.

A3.1.6 The pressurized air source may be (*1*) a self-contained miniature air cylinder for maximum portability, (*2*) shop (bottled) air, or (*3*) air from an automated pump.

A3.2 *Procedure:*

A3.2.1 Follow the general procedures described in Sections 6 and 7. Procedures specific to Type IV testers are described in the following section.

A3.2.2 Adhere a loading fixture to the coating based on the epoxy manufacturers instructions, employing either a cut-off ring or adhesive mask to reproducibly define the area being tested. On larger sized loading fixtures, simply wipe away excess epoxy with a cotton tipped applicator or rag.

A3.2.3 Place the piston over the loading fixture and gently thread the reaction plate (top of piston) onto the loading fixture.

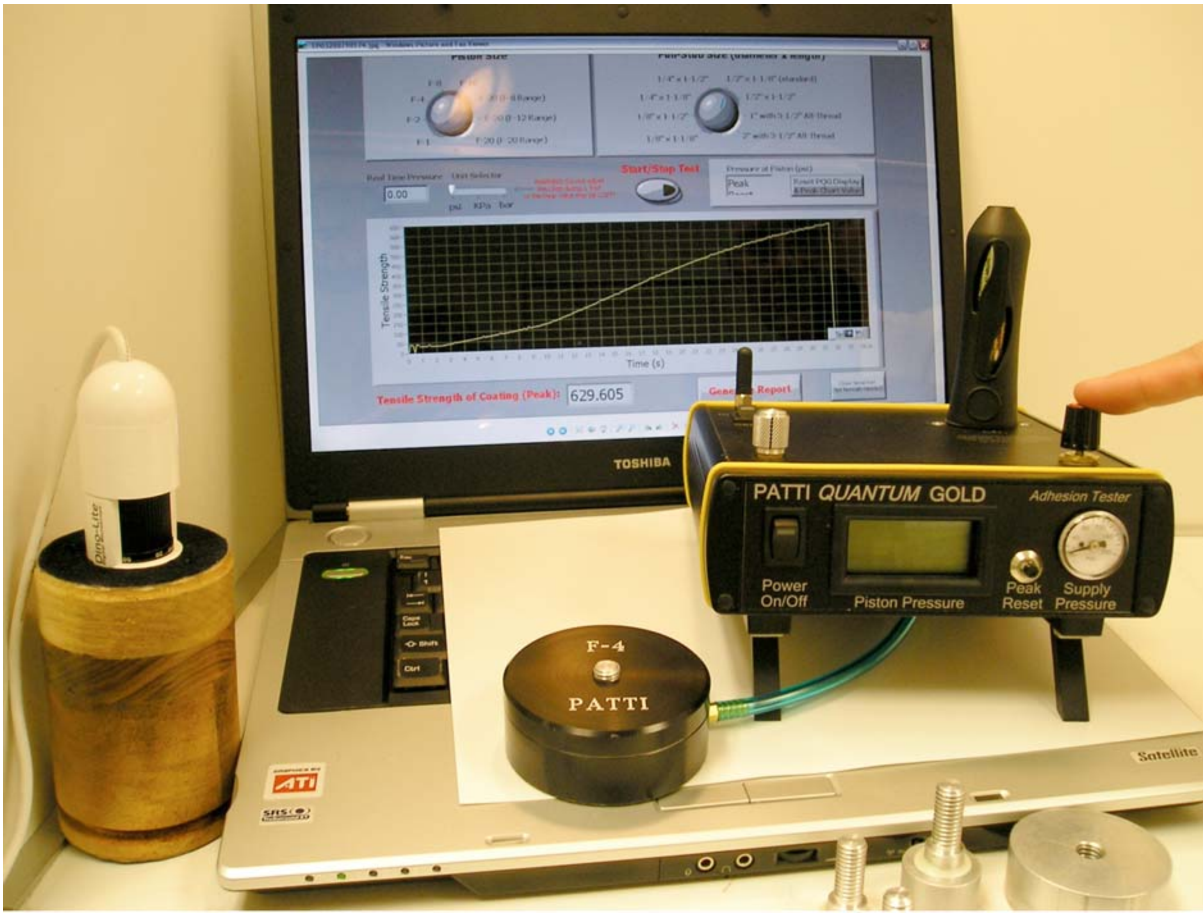
A3.2.4 Attach the appropriate pneumatic hoses and ensure that the control module has an air supply of at least 0.67 Mpa (100 psi) as read on the supply gauge. Zero the Piston Pressure gauge/display.

A3.2.5 Ensure that the Rate Valve is closed (clockwise finger tight) and then press and hold the Run button. Slowly open the Rate Valve (counterclockwise) and monitor the Piston Pressure gauge/display to obtain a rate of pressure increase of less than 1 MPa/s (100 psi/s) yet allowing for the entire test to be complete within 100 s. When the loading fixture detaches from the surface or the required pressure is attained, release the Run button.

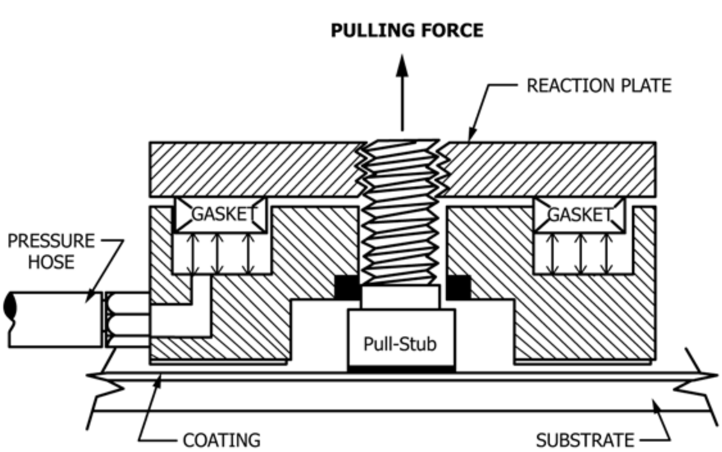
A3.2.6 Open the Rate Valve even further (counterclockwise) to relieve the residual pressure so the loading fixture can be removed from the piston to prepare for the next test.

A3.2.7 Record both the maximum pressure attained and the specific piston used. Convert the maximum Piston Pressure to coating tensile strength using the conversion charts or set the specific testing parameters within the software to have this step completed automatically.

A3.2.8 Photo document the test site if possible/necessary using the optional USB camera.



(a)



CROSS SECTION SCHEMATIC OF PISTON ATTACHED TO PULL-STUB
(b)

FIG. A3.1 Photograph (a) and Schematic of Piston (b) of Type IV Self-Alignment Adhesion Tester

A4. SELF-ALIGNING ADHESION TESTER TYPE V (TEST METHOD E)

A4.1 *Apparatus:*

A4.1.1 This is a self-aligning tester, as shown in Fig. A4.1.[11,9]

NOTE A4.1—Precision data for Type V instruments shown in Table 6 were obtained using the devices described as "Manual" in Fig. A4.1.

A4.1.2 A self-aligning spherical loading fixture head is used by this tester. Load evenly distributes pulling force over the surface being tested, ensuring a perpendicular, balanced pull-off. The diameter of the standard loading fixture 20 mm (0.78 in.) is equal to the area of the position bore in the actuator. Therefore, the pressure reacted by the loading fixture is the same as the pressure in the actuator and is transmitted directly to the pressure gauge. The tester performs automatic conversion calculations for the 50 mm (1.97 in.) loading fixtures and common custom sizes 10 and 14 mm (0.39 in. and 0.55 in. respectively).

A4.1.3 The apparatus is comprised of: a loading fixture, 10 to 50 mm (0.39 and 1.97 in. respectively) diameter, hydraulic actuator by which the load is applied to the loading fixture, pressure gauge with LCD display, and hydraulic pump.

A4.1.4 The display on the pressure gauge indicates the maximum force and the rate of pull.

A4.1.5 The tester is available with accessories for finishes on plastics, metals, and wood. Special loading fixtures, typically 10 mm (0.39 in.) and 14 mm (0.55 in.) are available for use on curved surfaces and when higher pull-off pressures are required.

A4.2 *Procedure:*

A4.2.1 Follow the general procedures described in Sections 6 and 7. Procedures specific to Type V Testers are described in this section.

A4.2.2 Ensure the pressure relief valve on the pump is completely open. Push the actuator handle completely down into the actuator assembly.

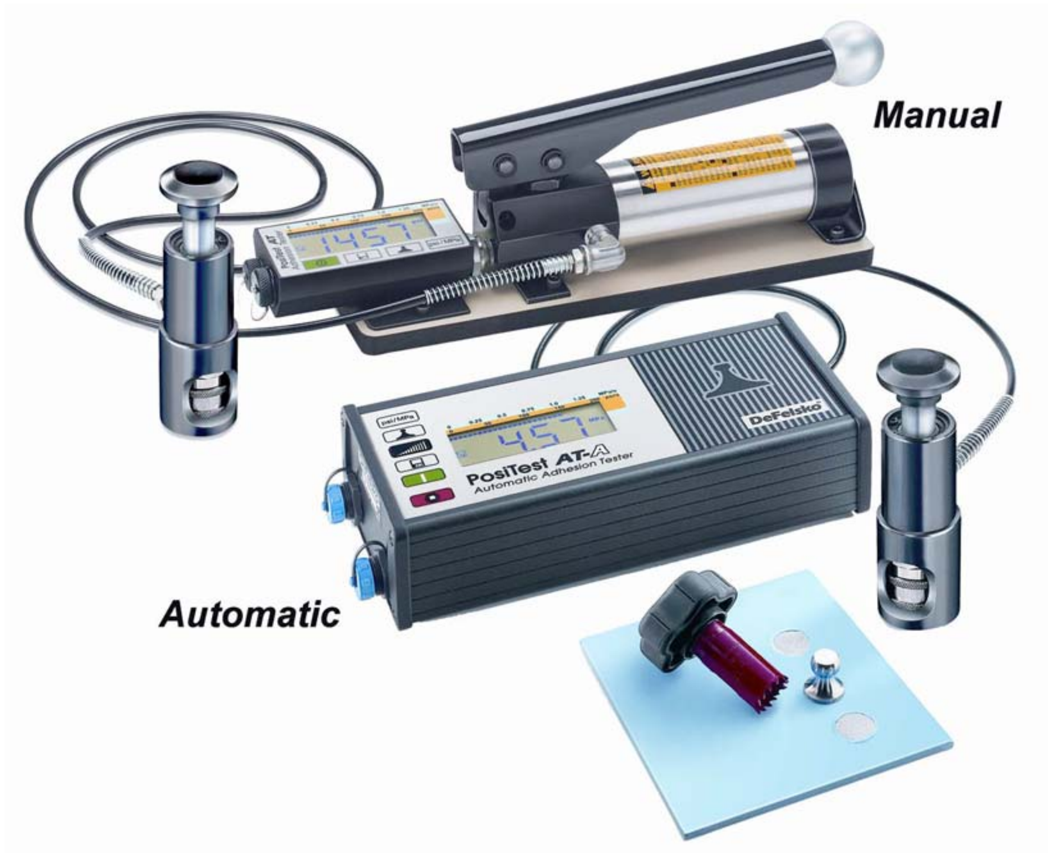
A4.2.3 Place the actuator assembly over the loading fixture head and attach the quick coupling to the loading fixture. Close the pressure relief valve on the pump. Select the appropriate loading fixture size on the display and then press the zero button.

A4.2.4 Prime the pump by pumping the handle until the displayed reading approaches the priming pressure as explained in the instruction manual. Return the pump handle to its full upright position and then complete a single stroke at a uniform rate of no more than 1 MPa/s (150 psi/s) as shown on the display until the actuator pulls the loading fixture from the surface.

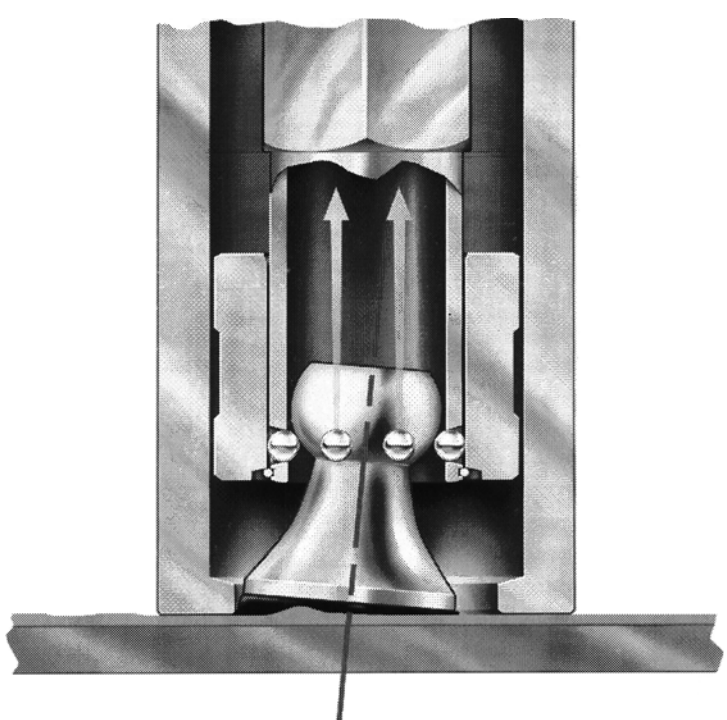
A4.2.5 Immediately following the pull, open the pressure relief valve on the pump to release the pressure. The display will maintain the maximum pressure reading. Record this pull off pressure into the tester's memory and mark the loading fixture for future qualitative analysis.

A4.2.6 A version of this tester is available with an automatic hydraulic pump.

[11] The sole source of supply of the PosiTest Pull-Off Tester known to the committee at this time is DeFelsko Corporation, 802 Proctor Avenue, Ogdensburg, NY 13669 USA.



(a)



(b)

FIG. A4.1 Photograph (a) and Schematic (b) of Type V, Self-Aligning Tester

A5. SELF-ALIGNING ADHESION TESTER TYPE VI (TEST METHOD F)

A5.1 *Apparatus:*

A5.1.1 This is a self-aligning tester, as shown in Fig. A5.1.

NOTE A5.1—Precision data for Type VI instruments shown in Table 6 were obtained using the devices described in Fig. A5.1.

A5.1.2 The self-aligning testing head uses four independently operated feet to ensure that the pull stress on the loading fixture is evenly distributed independently of the shape of the substrate or the angle of the loading fixture to the surface. See Fig. A5.1

A5.1.3 The apparatus comprises a crank handle pull mechanism with a hydraulic cable mechanism, a self-aligning test head rated at 6.3 kN and loading fixtures.

A5.1.4 A range of loading fixtures, from 2.8 to 70 mm diameter is available. The 20 mm diameter loading fixtures are directly connected to the test head by means of a quick release connector. Other loading fixture sizes are supplied with threads machined to allow connection to the self-aligning test head using an adapter. Loading fixtures with diameters in the range 2.8 to 5.7 mm are used with a micro self-aligning test head rated at 1 kN.

A5.1.5 The force applied to the loading fixture is displayed on a hydraulic pressure gauge with a dragging indicator that shows the maximum reading at the point where the loading fixture is removed from the surface. The gauge carries both PSI and MPa values on two scales.

A5.2 *Procedure:*

A5.2.1 Following the general procedures described in Sections 6 and 7, procedures specific to Type VI testers are described in the following section.

A5.2.2 Ensure that the pressure in the pull mechanism is released by opening the valve at the bottom of the cylinder. Turn the dragging indicator to zero in line with the gauge indicator needle.

A5.2.3 Attach the self-aligning test head to the hydraulic cable mechanism using the quick release connector on the side of the test head. Return the crank handle to the start position and ensure that the four pistons of the self-aligning head are level by pushing the head against a flat surface.

A5.2.4 Place the relevant support ring over the loading fixture. A support ring is not required for 25 mm, 50 mm, or 70 mm diameter loading fixtures or for 50 mm square loading fixtures.

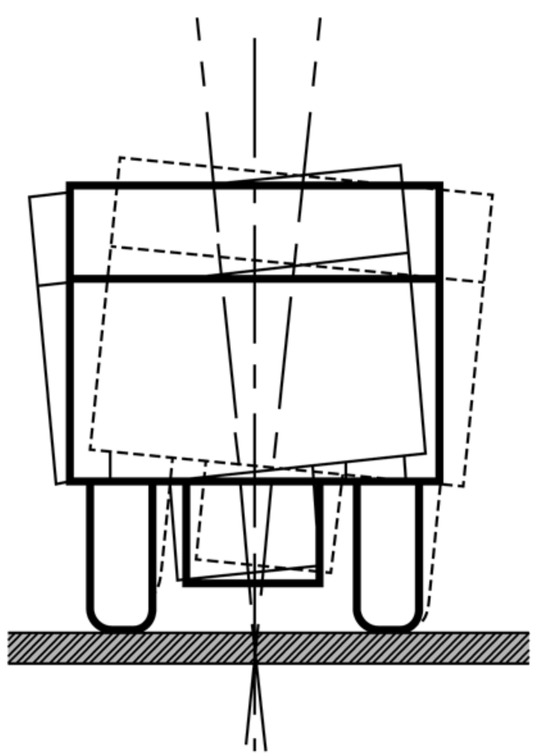
A5.2.5 Attach the test head to the loading fixture either directly or using the adapter, where appropriate. Close the valve.

A5.2.6 Ensure that the hydraulic cable mechanism is not pulled tight. Hold the pull mechanism in one hand and operate the crank with the other using a smooth and regular motion to ensure that the force is applied evenly until the desired value is reached or the fracture occurs.

A5.2.7 Immediately following the completion of the pull, open the valve to release any residual pressure and return the crank handle to the start position. The unit is now ready for the next pull.

A5.2.8 Note the value indicated by the dragging indicator and mark the loading fixture for further analysis as described in Section 8.

(a)



(b)

FIG. A5.1 Photograph (a) and Schematic (b) of Type VI, Self-Aligning Tester

APPENDIX

(Nonmandatory Information)

X1. STRESS CALCULATION

X1.1 The stress computed in 8.2 is equal to the uniform pull-off strength of the analogous rigid coating system if the applied force is distributed uniformly over the critical locus at the instant of failure. For any given continuous stress distribution where the peak-to-mean stress ratio is known, the uniform pull-off strength may be approximated as:

$$U = XR_o \quad \text{(X1.1)}$$

where:

U = uniform pull-off strength, representing the greatest force that could be applied to the given surface area, psi (MPa),
X = measured in situ pull-off strength calculated in 8.2, psi (MPa), and
R_o = peak-to-mean stress ratio for an aligned system.

It is important to note that a difference between these pull-off strengths does not necessarily constitute an error; rather the in-situ measurement simply reflects the actual character of the applied coating system with respect to the analogous ideal rigid system.

X1.2 An error is introduced if the alignment of the apparatus is not normal to the surface. An approximate correction by the peak-to-mean stress ratio is:

$$R = R_o(1 + 0.14\, az/d) \quad \text{(X1.2)}$$

where:

z = distance from the surface to the first gimbal or the point at which the force and counter force are generated by the action of the driving mechanism, in. (mm),
d = diameter of the loading fixture, in. (mm),
a = angle of misalignment, degrees (less than 5), and
R = maximum peak-to-mean stress ratio for the misaligned rigid system.

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D4541 - 02) that may impact the use of this standard. (Approved February 1, 2009.)

(1) The scope was modified to describe the types of substrates covered by the test method.
(2) Test Method A was discontinued. Test Method F and Annex F were added.
(3) Section 10 — The precision and bias statement was revised based on the results of a new round-robin study.
(4) Editorial changes were made throughout the document.