

# Standard Test Method for Durability of Sealants Exposed to Continuous Immersion in Liquids<sup>1</sup>

This standard is issued under the fixed designation C1247; 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 a laboratory procedure that assists in determining the durability of a sealant and its adhesion to a substrate while continuously immersed in a liquid. This test method tests the influence of a liquid on the sealant and its adhesion to a substrate. It does not test the added influence of constant stress from hydrostatic pressure that is often present with sealants used in submerged and below-grade applications, nor does it test the added influence of stress from joint movement while immersed. This test method also does not (in its standard form) test the added influence of acids or caustics or other materials that may be in the liquid, in many applications.
- 1.2 The values stated in SI units are to be regarded as the standard. The inch-pound given in parentheses are provided for information only.
- 1.3 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>2</sup>

C33 Specification for Concrete Aggregates

C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

C150 Specification for Portland Cement

C717 Terminology of Building Seals and Sealants

C719 Test Method for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement (Hockman Cycle)

#### 2.2 ISO Standards

ISO 13638 Building Construction-Sealants-Determination of Resistance of Prolonged Exposure to Water<sup>3</sup>

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 Definitions of the following terms used in this test method are found in Terminology C717: adhesive failure; cohesive failure; compound; cure; hydrostatic pressure; joints; primer; sealant; self-leveling sealant; standard conditions; and substrate.

#### 4. Summary of Test Method

- 4.1 Specimens are prepared and the sealant is cured. The specimens are immersed in a hot liquid at  $50 \pm 2^{\circ}\text{C}$  (122  $\pm$  3.6°F) for six weeks. At the end of the six weeks, the specimens are placed into a compression/extension machine, as described in Test Method C719, and cycled through the movement range appropriate to the test sealant or its application, or both.
- 4.2 If after three movement cycles at 3.2 mm/h (1/8 in./h) adhesive or cohesive failure is not noted, the specimens are again placed in the hot liquid for an additional four weeks and then retested.
- 4.3 This test method consists of six weeks of immersion followed by three extension and compression cycles. This can be followed by four weeks of further immersion and a repeat of the three extension and compression cycles. The four weeks of immersion and subsequent extension and compression testing can be repeated as many times as required for the application.

#### 5. Significance and Use

5.1 This test method uses elevated temperature to accelerate the degradation of a sealant and its adhesion to a substrate. This test method is an accelerated method and will only be a predictor of long-term durability if the actual service temperature is significantly lower than the elevated test temperature.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee C24 on Building Seals and Sealantsand is the direct responsibility of Subcommittee C24.30 on Adhesion.

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<sup>&</sup>lt;sup>2</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.

 $<sup>^3</sup>$  Available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

- 5.2 This test method can be used as an indicator of longevity but direct correlation to actual use will be difficult for many applications.
- 5.3 The correlation of data from this test method to applications where the sealant joint will have wet and dry cycles will be difficult since, with some sealants on some substrates, adhesion that is lost during wet periods is regained during dry periods.
- 5.4 This test method is performed in a hot liquid and may be considered an acceleration of deterioration of the sealant or the sealant's adhesion to a substrate. Compared to how the sealant will be used in some applications, in some cases, this test may be less severe than the actual application. The benefit from the use of this test method will depend on the comparison of the conditions of this test to the actual conditions of use (temperature, duration, nature of substrate, composition of the liquid).
- 5.5 To determine the ability of a sealant to perform in a given application; modification of this procedure will often be required and is permissible, as mutually agreed upon by purchaser and seller.

## 6. Apparatus

- 6.1 Water Bath, equipped with a heater to maintain the test bath at  $50 \pm 2$ °C ( $122 \pm 3.6$ °F).
- 6.2 Compression/Extension Machine, <sup>4</sup>designed to automatically compress the joint width of the test specimen from 12 mm (½ in.) to the maximum extension desired at a constant rate of 3.2 mm/h (½ in./h). The machine shall be equipped with grips of sufficient strength to withstand, without bending, the resistance of high-tensile-strength sealants.
  - 6.3 "C" Clamps, or other devices for specimen preparation.
  - 6.4 Casting Spacers, for specimen preparation.
  - 6.5 Liquid, appropriate to the application for the test bath.

Note 1—The precision and bias statement for this test method is based on the use of distilled water for the test bath.

#### 7. Test Specimens

7.1 Standard substrates for this test method shall be portland cement mortar and aluminum alloy.

Note 2—The precision and bias statement of this test method is based on standard substrates of mortar and aluminum. When requested, only one or two of the standard substrates may be tested with the sealant. Likewise, other substrates such as brick, marble, etc., may be specified by the purchaser in place of or in addition to the standard substrates for test with the sealant sample.

7.1.1 *Mortar Block*<sup>5</sup>—Prepare cement mortar blocks, each 75 by 25 by 25 mm (3 by 1 by 1 in.) in size, using one part of high early strength portland cement conforming to Type III of Specification C150, to two parts by weight of clean uniformly

graded, concrete fine aggregate (sand) conforming to Specification C33. Use sufficient water to produce a flow of  $100 \pm 5$  when tested in accordance with the procedure for the determination of consistency of cement mortar described in Test Method C109/C109M. After curing one day in moist air and six days in saturated lime water at  $23 \pm 2^{\circ}$ C ( $73 \pm 3.6^{\circ}$ F), prepare the surface of one face, 25 by 76 mm (1 by 3 in.) of each block by wet grinding either with a belt sander using No. 60 aluminum carbide sanding belt or using an iron lap with No. 60 silicon carbide (or aluminum oxide) grain, until the aggregate is uniformly exposed. Return blocks to saturated lime water storage until needed.

- 7.1.2 Blocks may be prepared and shipped to other locations for use. The blocks may be shipped dry and returned to lime water storage on arrival until needed.
- 7.1.3 Prior to use, wet grind the previously ground face to remove any laitance, rinse thoroughly under running tap water and dry the blocks overnight at 105 to 110°C (220 to 230°F). Clean the blocks of film or powder by vigorous brushing with a stiff-bristled fiber brush. Condition the blocks at standard conditions for not less than one nor more than seven days.
- 7.1.4 *Aluminum*—The aluminum plates shall be 75 by 25 by 6 mm (3 by 1 by ½ in.) aluminum alloy, 6063-T5 or 6061-T6 with anodizing process AA-MIOC22A31. Prior to use clean the aluminum by wiping the surface with methyl ethyl ketone or similar solvent. Then dip the surface in a detergent solution. This should be a 0.04 % solution of alcohol ethoxy sulfate. An alternative would be a 0.1 % solution of a clear hand dishwashing detergent. These solutions should be made up in distilled water. Rinse the surface (without touching it) in distilled water and allow it to air dry.

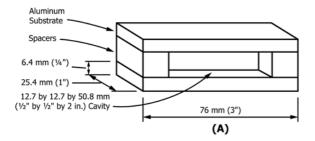
Note 3—Methyl ethyl ketone and similar solvents are both toxic and flammable and should be handled with caution in a well-ventilated hood.

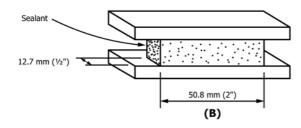
Note 4—At the request of the sealant producer, the detergent cleaning step shall be omitted.

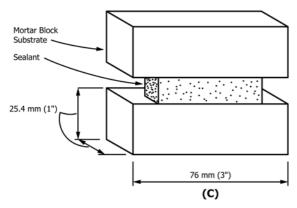
- 7.2 *Primers*—Where the use of a primer(s) is recommended by the sealant manufacturer, prime substrate materials with the recommended primer or primers.
  - 7.3 Preparation of Test Specimens:
- 7.3.1 Multicomponent Sealants—Prepare three test specimens for each substrate that is to be used in the test. After maintaining the unopened sample for at least 24 h at standard conditions, mix thoroughly for 5 min at least 250 g of base compound with the appropriate amount of curing agent. Apply a bead of sealant 12 by 12 by 50 mm ( $\frac{1}{2}$  by  $\frac{1}{2}$  by 2 in.) between parallel 25 by 76 mm (1 by 3 in.) faces of similar blocks or plates (see Fig. 1). Use appropriate casting spacer to form the proper size sealant bead. Use non-adhering casting spacers or apply polyethylene adhesive tape or any other suitable inert release agent to the inside surface of the spacers to prevent adhesion of the spacers to the sealant after cure. Use adhesive tape, rubber bands, or clamps to hold the test assembly together before and after filling it with the sealant. For self-leveling sealants, use masking or any other suitable tape to retain the sealant.

<sup>&</sup>lt;sup>4</sup> Series 520 Sealing Compound Tester, manufactured by Applied Test Systems, Inc., 348 New Castle Road, Butler, PA 16001 has been found suitable for this purpose

<sup>&</sup>lt;sup>5</sup> Prepared concrete blocks are available from Masonry Test Block Company, 6389 Lawdale Road, Saginaw, MI 48604.







# Test Specimen:

- (A) Prepared Specimen Before Placement of Sealant.
- (B) Sealant with Spacers Removed.
- (C) Sealant Between Mortar Block Substrates.

FIG. 1 Test Specimen

7.3.2 *Single-Component Sealants*—Prepare three test specimens as described in 7.3.1 except that no mixing of components is required. Condition the sealed sealant cartridge or bulk container at standard conditions at least 24 h before use.

## 8. Conditioning

- 8.1 Cure specimens made with multicomponent sealants for 14 days at standard conditions. During the second week of the curing period, make attempts to free the sealant from the casting spacers at the ends and bottom without damaging the sealant bead.
- 8.2 Cure specimens made with single-component sealants for a total of 21 days as follows: (a) seven days at standard conditions; (b) seven days at 37.8  $\pm$  2°C (100  $\pm$  3.6°F) and 95  $\pm$  5% relative humidity; and (c) seven days at standard conditions. Separate the casting spacers from the sealant as soon as practical during the curing period without damaging the sealant.

#### 9. Procedure

- 9.1 Remove the spacers and place the cured test specimens into the  $50\pm\,2^{\circ}C$  ( $122\pm\,3.6^{\circ}F$ ) test bath, totally immersing them and maintaining them at this condition for six weeks. Use a  $3.8\,L$  (1 gal) size container containing  $1.9\,L$  (2 qt) of test bath. Mortar and aluminum specimens should be separated and conditioned in separate containers.
- 9.2 After six weeks immersion, remove the specimens from the test bath, place them immediately into the compression and extension machine and start cycling with the compression mode, using the movement range appropriate to the test sealant or its application, or both. The test is to be conducted at standard conditions.
- 9.3 Cycle the specimens through three compression and three extension cycles.
- 9.4 Remove the specimens, hand flex the specimens to check sealant adhesion by rotating them  $60^{\circ}$ , and examine them for adhesive or cohesive failure. Measure the amount of adhesive or cohesive failure in square millimetres (square inches) and record these results on the report form. Note also any apparent swelling or change in appearance or contour of the sealant.
- 9.5 Return the specimens to the  $50 \pm 2^{\circ}\text{C}$  ( $122 \pm 3.6^{\circ}\text{F}$ ) test bath for four weeks. After immersion, remove the specimens from the test bath. Place them immediately into the compression and extension machine and start cycling with the compression mode.
- 9.6 Repeat 9.3 and 9.4. If there is not total adhesive or cohesive failure and, if required for the application, repeat 9.5,9.3, and 9.4 in that order.

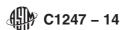
#### 10. Report

- 10.1 Report the test results and observations on the form shown in Fig. 2. Report total square millimetres (square inches) of adhesion and cohesive failure for all three samples of each substrate tested.
  - 10.2 Report the following additional information:
- 10.2.1 Techniques used in substrate preparation including the solvent, primers, or other materials used for cleaning. If other than the standard substrates or different preparation techniques are used, describe them completely,
- 10.2.2 Other pertinent observations on the condition of the sealant or its adhesion or other characteristics, and
  - 10.2.3 Any variation in test procedure or cure cycle used.

## 11. Precision and Bias<sup>6</sup>

- 11.1 Polyurethane Sealant, Six and Ten Weeks of Water Immersion on Aluminum and Concrete Substrates:
- 11.1.1 Repeatability I(r)—The repeatability (within a given laboratory) interval for four test parameters tested by four laboratories is  $0.0 \text{ mm}^2$  ( $0.0 \text{ in.}^2$ ). In future use of this test method, the difference between two test results obtained in the

<sup>&</sup>lt;sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C24-1043.



SUBMITTED BY:			START DATE:		
			COMPLETION DATE:		
SEALANT:			SUBSTRATE:		
PRIMER:			TEST BATH:		
MOVEMENT: EXTENSION					
COMPRESSION					
IMMERSION PERIOD	CYCLES		FAILURE MODE		
	СОМР.	EXT.	ADHESION SQ./IN.	COHESIVE SQ./IN.	COMMENTS
INITIAL @ 6 WEEKS					
ADDITIONAL					
1ST @ 4 WEEKS					
2ND @ 4 WEEKS					
3RD @ 4 WEEKS					

FIG. 2 Report Form

same laboratory on the same material will be expected to exceed  $0.0~\text{mm}^2~(0.0~\text{in.}^2)$  only about 5 % of the time.

COMMENTS:

- 11.1.2 Reproducibility I(R)—The reproducibility (between given laboratories) interval for four test parameters tested by four laboratories is  $0.0 \text{ mm}^2$  ( $0.0 \text{ in.}^2$ ). In future use of this test method, the difference between two test results obtained in a different laboratory on the same material will be expected to exceed  $0.0 \text{ mm}^2$  ( $0.0 \text{ in.}^2$ ) only about 5 % of the time.
- 11.2 Polysulfide Sealant, Six and Ten Weeks of Water Immersion on Aluminum and Concrete Substrates:
- 11.2.1 Repeatability I(r)—The repeatability (within a given laboratory) interval for four test parameters tested by four laboratories is 26.5 cm<sup>2</sup> (4.1 in.<sup>2</sup>). In future use of this test method, the difference between two test results obtained in the same laboratory on the same material will be expected to exceed 26.5 cm<sup>2</sup> only about 5 % of the time.
- 11.2.2 Reproducibility I(R)—The reproducibility (between given laboratories) interval for four test parameters tested by four laboratories is 43.9 cm<sup>2</sup> (6.8 in.<sup>2</sup>). In future use of this test method, the difference between two test results obtained in a

different laboratory on the same material will be expected to exceed  $43.9 \text{ cm}^2$  only about 5% of the time.

- 11.3 Silicone Sealant, Six and Ten Weeks of Water Immersion on Aluminum and Concrete Substrates:
- 11.3.1 Repeatability I(r)—The repeatability (within a given laboratory) interval for four test parameters tested by four laboratories is 23.9 cm<sup>2</sup> (3.7 in.<sup>2</sup>). In future use of this test method, the difference between two test results obtained in the same laboratory on the same material will be expected to exceed 23.9 cm<sup>2</sup> only about 5 % of the time.
- 11.3.2 Reproducibility I(R)—The reproducibility (between given laboratories) interval for four test parameters tested by four laboratories is  $34.2 \text{ cm}^2$  (5.3 in.<sup>2</sup>). In future use of this test method, the difference between two test results obtained in a different laboratory on the same material will be expected to exceed  $34.2 \text{ cm}^2$  only about 5 % of the time.

#### 12. Keywords

12.1 adhesion; liquid immersion; sealant; water immersion

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