



Standard Test Method for Hydrophobic Surface Films by the Water-Break Test¹

This standard is issued under the fixed designation F22; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the detection of the presence of hydrophobic (nonwetting) films on surfaces and the presence of hydrophobic organic materials in processing environments. When properly conducted, the test will enable detection of molecular layers of hydrophobic organic contaminants. On very rough or porous surfaces, the sensitivity of the test may be significantly decreased.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound values given in parentheses are 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:²

- [C813 Test Method for Hydrophobic Contamination on Glass by Contact Angle Measurement](#)
- [D351 Classification for Natural Muscovite Block Mica and Thins Based on Visual Quality](#)
- [D1193 Specification for Reagent Water](#)
- [D2578 Test Method for Wetting Tension of Polyethylene and Polypropylene Films](#)
- [D5946 Test Method for Corona-Treated Polymer Films Using Water Contact Angle Measurements](#)
- [D7334 Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement](#)
- [D7490 Test Method for Measurement of the Surface Tension](#)

¹ This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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

[of Solid Coatings, Substrates and Pigments using Contact Angle Measurements](#)

[F21 Test Method for Hydrophobic Surface Films by the Atomizer Test](#)

3. Terminology

3.1 Definitions:

3.1.1 *contact angle, n*—the interior angle that a drop makes between the substrate and a tangent drawn at the intersection between the drop and the substrate as shown in [Fig. 1](#); this is the angle formed by a liquid at the three phase boundary where a liquid, gas (air) and solid intersect.

3.1.2 *hydrophilic*—having a strong affinity for water, wettable.

3.1.2.1 *Discussion*—Hydrophilic surfaces exhibit zero contact angle with water. A sessile drop of water applied to the surface will immediately spread out to form a film.

3.1.3 *hydrophobic*—having little affinity for water, nonwettable.

3.1.3.1 *Discussion*—Hydrophobic surfaces exhibit contact angles between a sessile drop of water and the surface appreciably greater than zero.

3.1.4 *sessile drop*—a drop of liquid sitting on the upper side of a horizontal surface.

3.1.5 *water-break*—a break in the continuity of a film of water on a surface on removal from an aqueous bath or on removal of a flowing water source from the surface.

4. Summary of Test Method

4.1 The water-break test is performed by withdrawing the surface to be tested, in a vertical position, from a container of purified water and observing the behavior of the water. For large parts, the test surface may be doused with water and the water behavior observed on removal of the water source.

4.2 The interpretation of the test is based upon the pattern of wetting. Contaminated areas having a surface tension lower than water will cause the water to bead up at that location or “break” while draining. Most common film contaminants such as oils, silicones, or fluorocarbon greases have surface tensions significantly lower than water. In the absence of hydrophobic films, the draining water layer will remain as a film over the

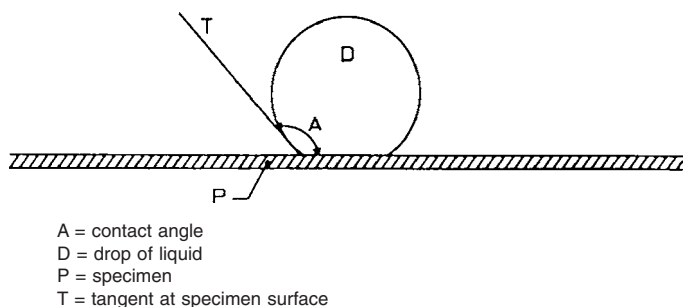


FIG. 1 Contact Angle

surface. In areas where hydrophobic materials are present on the surface, the draining water layer will break up into a discontinuous film within one minute.

NOTE 1—It is possible to use this test method with liquids other than water. Liquids with different wetting tensions will exhibit different contact angles with a given surface and therefore different levels of sensitivity to hydrophobic films. This principle has been applied to develop differential wetting tension tests such as described in Test Method D2578 using standardized liquids commonly referred to as dyne liquids.

5. Significance and Use

5.1 The water-break test as described in this test method is rapid, nondestructive, and may be used for control and evaluation of processes for the removal of hydrophobic contaminants. This test method is commonly used for in-process verification of the absence of surface contaminants on metal surfaces that may interfere with subsequent surface treatments such as priming, conversion coating, anodizing, plating, or adhesive bonding

5.2 This test method is not quantitative and is typically restricted to applications where a go/no go evaluation of cleanliness will suffice.

5.3 The test may also be used for the detection and control of hydrophobic contaminants in processing environments. For this application, a witness surface free of hydrophobic films is exposed to the environment and subsequently tested. The sensitivity of this test will vary with the level of airborne contaminant and the duration of exposure of the witness surface.

5.4 For quantitative measurement of surface wetting, test methods that measure contact angle of a sessile drop of water or other test liquid may be used in some applications. Measurement methods based on contact angle are shown in Test Methods C813, D5946, and D7490; and Practice D7334.

5.4.1 Devices for in situ measurement of contact angle are available. These devices are limited to a small measurement surface area and may not reflect the cleanliness condition of a larger surface. For larger surface areas, localized contact angle measurement, or other quantitative inspection, combined with water-break testing may be useful.

5.5 For surfaces that cannot be immersed or doused with water, or where such immersion or dousing is impractical, Test Method F21 may be useful.

NOTE 2—This test method is not appropriate where line of sight evaluation is not feasible; or for assembled hardware where there is a risk

for entrapment of water in faying surfaces or complex structures where it may not be effectively removed.

6. Interferences

6.1 Loss of sensitivity may result from either of the following factors:

- 6.1.1 The presence of hydrophilic substances on the surface to be tested, in the test equipment, or in the test materials, or
- 6.1.2 An unusually rough or porous surface condition.

6.2 On hot parts or in elevated temperature conditions, water may evaporate before water-break can be observed.

7. Apparatus

7.1 *Overflow Container*, such as a glass beaker for small parts.

7.2 *Purified Water Source*, final rinse tank or dousing hose and drain, for large parts.

7.3 *Low Power Microscope*, (5 to 50×) and light source for observation of small piece parts (optional).

8. Reagents and Materials

8.1 *Purity of Water*—Deionized or distilled water per Specification D1193 Types II, III, or IV is preferred. Water of higher ionic content may render the test destructive. The water used must be free of hydrophobic and hydrophilic substances. When processing large parts in a production environment, the water shall be sampled directly from the rinse tank or at the exit of the dousing water source prior to water-break test. Hydrophilic contaminants such as trace surfactants present on the rinse tank walls or dousing hose may contaminate the test water and invalidate the test. Statistical process control may be used to verify the cleanliness of the test water in lieu of sampling prior to each water-break test.

NOTE 3—Freedom of the water from hydrophobic and hydrophilic contamination may be determined in accordance with Appendix X1.

8.2 *Acetone*, reagent grade, A.C.S.³

8.3 *Mica Blanks*, preferably 25 by 50 by 0.38 mm (1 by 2 by 0.015 in.) or larger.⁴

8.4 *Oleic or Stearic Acid*—A 0.05 % solution in acetone.

9. Procedure

9.1 *Testing of Surfaces*—

9.1.1 Immerse the test surface in clean water. This water may be the final rinse of a multi-step cleaning or surface treatment process. For very large surfaces, water may be applied to the vertical surface with a hose or gentle spray.

9.1.2 Withdraw the test surface, in a vertical position, from the container of water or withdraw the flowing water source.

9.1.3 Immediately upon removal from the water, or removal of the flowing water source, observe the gravity flow of the water from the test surface for 1 min.

³ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC.

⁴ Mica meeting quality level V-6 or better per Classification D351 has been found to be acceptable.

9.1.4 Dry the surface of any remaining water on completion of the test. Corrosion-sensitive surfaces should be dried immediately by a flow of clean, oil-free air or nitrogen. Drying may be enhanced by a rinse with isopropyl alcohol prior to drying with oil-free air or nitrogen.

NOTE 4—The presence of oil or contaminants in the drying gas or the processing environment may recontaminate the surface and invalidate the water-break test results.

9.2 *Testing of Processing Environments*—Expose a smooth test surface, verified clean and water-break-free, to the environment and subsequently continue as described in 10.1. A freshly cleaved mica blank may be used as the test surface.

NOTE 5—For testing of rinse water, exposure may be by immersion of the test surface in the water or by deposition of a sample of the water on the test surface.

10. Interpretation of Results

10.1 Surfaces tested as described in 9.1 shall be considered free of hydrophobic contaminants by this test method if the draining water layer remains as a thin continuous film over the surface for 60 s after withdrawal of the surface from the immersion container or removal of the dousing water source (see Fig. 2). If hydrophobic contaminants are present, as evidenced by formation of a discontinuous water film within 60

s after withdrawal from or of the test fluid, the length of time necessary for the water-break to occur is a rough indication of the degree of contamination.

10.2 A discontinuous film may appear in the form of localized or general beading of the water, or discontinuous flow of the water around discrete locations where contaminants are present (see Fig. 3 and Fig. 4). Significant contamination will be apparent within 2 to 15 s.

11. Precision and Bias

11.1 Precision and bias are intended to be adequate for use as an in-process monitoring method.

11.1.1 *Repeatability*—Repeatability of this test method requires inspectors that have been trained in the observation of surfaces for water breaks.

11.2 Bias:

11.2.1 No absolute standard traceable to the National Institute of Standards and Technology is available, therefore the bias of this test method cannot be determined at this time.

12. Keywords

12.1 contact angle; hydrophilic films; organic contamination; surface contamination; water break



FIG. 2 Clean aluminum panel showing no water break. Water drains in a continuous sheet.



FIG. 3 Aluminum panel showing water-break on withdrawal from immersion. The right side of the panel has been contaminated with a film of grease.



FIG. 4 Contaminated aluminum panel showing water-break on withdrawal from immersion. The water may break in a different pattern after each immersion of the same part.

APPENDIX

X1. CALIBRATION AND STANDARDIZATION

X1.1 Calibration and Standardization

X1.1.1 Freedom of the test equipment and materials from hydrophobic contamination may be determined as described in 9.1 on a mica sheet having both surfaces freshly cleaved. If water-break does not occur within 60 s after withdrawal of the freshly cleaved mica surface from the overflow container, the test equipment and materials can be considered free of hydrophobic contamination for this test.

X1.1.2 Freedom of the test equipment and materials from hydrophilic contamination may be determined as follows:

X1.1.2.1 On a mica sheet having both surfaces freshly cleaved, apply 1 drop (0.05 to 0.10 mL) of a 0.05 % solution of oleic or stearic acid in acetone and allow this drop to evaporate.

X1.1.2.2 This treated mica surface, when tested in accordance with 9.1, should clearly show within 60 s the demarcation between the clean and contaminated areas. The absence of a clear demarcation between the clean and contaminated areas is an indication that hydrophilic contaminants may be present in the test water.



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