



Designation: **F22–02 (Reapproved 2007) F22 – 13**

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 ~~ambients-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 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.

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.

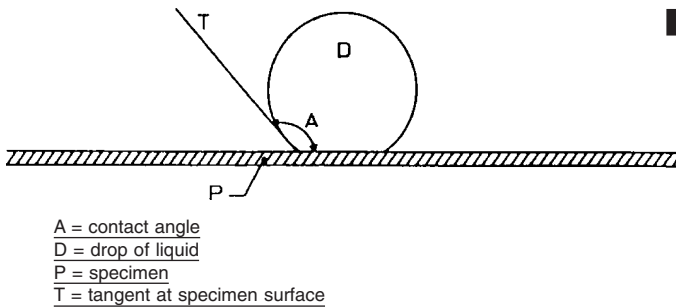
Current edition approved April 1, 2007; Nov. 1, 2013. Published April 2007; November 2013. Originally approved in 1962. Last previous edition approved in 2002 as F22–02; F22–02 (2007). DOI: 10.1520/F0022-02R07-10.1520/F0022-13.

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.

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, nonwetable.


FIG. 1 Contact Angle

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 water-break test is performed by withdrawing the surface to be tested, in a vertical position, from a container overflowing with water. 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 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 water-break test as described in this test method is nondestructive and may be used for control and evaluation of processes for the removal of hydrophobic contaminants. The test may 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 ambient 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-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-parts (optional).

8. Reagents and Materials

8.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,³ where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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-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—The freedom-Freedom of the water from hydrophobic and hydrophilic contamination may be determined in accordance with Section 9Appendix X1.

8.2 Acetone-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, larger, having⁴ minimum ASTM Quality V6 as described in Classification D351.

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

9. Calibration and Standardization

9.1 Freedom of the test equipment and materials from hydrophobic contamination shall be determined as described in 10.1 on a mica sheet having both surfaces freshly cleaved. If water-break does not occur within 1 min after withdrawal of the freshly cleaved mica surface from the overflow container, the test equipment and materials shall be considered free of hydrophobic contamination for this test.

9.2 To ensure the freedom of the test equipment and materials from hydrophilic contamination, a mica sheet having both surfaces freshly cleaved, from which the solvent from 1 drop (0.05 to 0.10 mL) of a 0.05 % solution of oleic or stearic acid in acetone has been allowed to evaporate shall, when tested, clearly show within 1 min the demarcation between the clean and contaminated areas.

9. Procedure

9.1 Testing of Surfaces—Withdraw the test surface, in a vertical position, from the container overflowing with water.

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.

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.

³ Reagent Chemicals, American Chemical Society Specifications⁷, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analabs Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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