



# Standard Test Method for Hydrophobic Surface Films by the Atomizer Test<sup>1</sup>

This standard is issued under the fixed designation F21; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 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 fractional 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:*<sup>2</sup>

- [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](#)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## Angle Measurements

### F22 Test Method for Hydrophobic Surface Films by the Water-Break Test

## 3. Terminology

### 3.1 Definitions:

3.1.1 *hydrophilic, adj*—having a strong affinity for water; wettable.

3.1.1.1 *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.

3.1.2 *hydrophobic, adj*—having little affinity for water; nonwetable.

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

3.1.3 *sessile drop, n*—a drop of liquid sitting on the upper side of a horizontal surface.

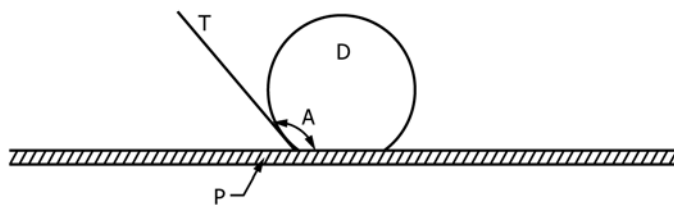
3.1.4 *water-break, n*—a break in the continuity of a film of water on a surface on removal from an aqueous bath or on removal of a flowing water source from the surface.

3.1.5 *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.

## 4. Summary of Test Method

4.1 The atomizer test<sup>3</sup> is performed by subjecting the dry surface to be tested to a fine water spray. The interpretation of the test is based upon the pattern of wetting. In the absence of hydrophobic films, the impinging water droplets will wet the surface and spread immediately to form a continuous water film. Contaminated areas having a surface tension lower than water will cause the water to bead up at that location. Most common film contaminants such as oils, silicones, or fluorocarbon greases have surface tensions significantly lower than water. In areas where these hydrophobic materials are present

<sup>3</sup> Linford, H. B., and Saubestre, E. B., "A New Degreasing Evaluation Test: The Atomizer Test," *ASTM Bulletin*, May 1953, p. 47.



A = contact angle  
D = drop of liquid  
P = specimen  
T = tangent at specimen surface

**FIG. 1 Contact Angle**

on the surface, the sprayed droplets will not wet the surface and spread but will tend to remain as fine droplets.

## 5. Significance and Use

5.1 The atomizer 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. The test may also be used for the detection and control of hydrophobic contaminants in processing environments. For this application, a surface free of hydrophobic films is exposed to the environment and subsequently tested.<sup>4</sup>

5.2 This test method is related to Test Method **F22**. This test method may be suitable as an alternative to the water-break test when the surface of interest should not or cannot be immersed or doused with water, or when such immersion or dousing is impractical.

NOTE 1—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.

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

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 This test method is only for use on test surfaces composed of materials, such as metal surfaces, that are hydrophilic (“wetable”) when clean. Some materials such as gold and many plastics are poorly wetted by water, making contamination difficult to detect by this method.

<sup>4</sup> Feder, D. O., and Koontz, D. E., “Detection, Removal and Control of Organic Contaminants in the Production of Electron Devices,” *ASTM STP 246*, Am. Soc. Testing Mats., p. 40 (1959).

## 6. Interferences

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

6.1.1 The presence of hydrophilic substances, such as trace surfactants, on the surface to be tested, test equipment, or test materials,

6.1.2 An unusually rough or porous surface condition, or

6.1.3 A test surface composed of a material that is significantly hydrophobic (not “wetable”) when clean.

6.2 On hot parts or in elevated temperature conditions, water may evaporate before surface wetting can be accurately observed.

## 7. Apparatus

7.1 *Spray Gun Atomizer*.<sup>5</sup>

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

## 8. Reagents and Materials

8.1 *Acetone*, reagent grade.<sup>6</sup>

8.2 *Mica Blanks*, preferably 25 by 50 by 0.38 mm (1 by 2 by 0.015 in.) or larger.<sup>7</sup>

8.3 *Oleic or Stearic Acid*—A 0.005 to 0.05% solution in acetone.

8.4 *Compressed Air*—Filtered, oil-free, low-pressure 100 kPa (15 psi) min compressed air or other suitable gas that is free of hydrophobic and hydrophilic substances.

NOTE 2—Freedom of the water and the gas from hydrophobic and hydrophilic contamination may be determined in accordance with **Appendix XI**.

8.5 *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 (see **Note 2**).

## 9. Procedure

9.1 *Testing of Surfaces*

9.1.1 Hold the test surface 250 to 500 mm (10 to 20 in.) from the atomizer and spray with a fine mist of water for a period of 1 to 10 s.

9.1.2 Immediately after spraying, observe the behavior of the water on the test surface to detect general or localized beading.

9.1.3 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 3—The presence of oil or contaminants in the drying gas or the

<sup>5</sup> Paasche Air Brush Model VL-1 or its equivalent has been found satisfactory for this purpose.

<sup>6</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC.

<sup>7</sup> Mica meeting quality level V-6 or better per Classification **D351** has been found to be acceptable.

processing environment may recontaminate the surface and invalidate the water-break test results.

9.2 *Testing of Processing Environments—*

9.2.1 Expose a smooth test surface, verified clean and water-break-free, to the environment and subsequently perform the test as described in 9.1. A freshly cleaved mica blank may be used as the test surface.

NOTE 4—For testing of the water itself for background contamination, 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. The procedure described in Appendix X1 may be used to increase the sensitivity of this test.

10. Interpretation of Results

10.1 Surfaces tested as described in 9.1 shall be considered free of hydrophobic contaminants by this test if the impinging water droplets spread immediately to form a thin, continuous uniform water film as shown in Fig. 2. If hydrophobic contaminants are present, as evidenced by discontinuous or nonuniform water films, the degree of contamination may be estimated by comparison of the pattern of wetting with Figs. 3-6.

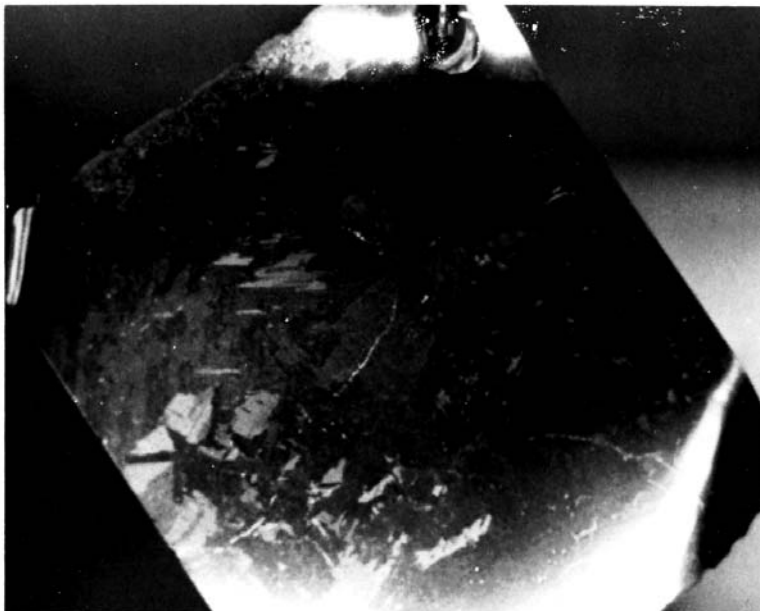


FIG. 2 Atomizer Test of Nickel Specimen with No Detectable Hydrophobic Contaminant (x2 Reduced One-Half in Reproduction)

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 hydrophilic films; organic contamination; surface contamination; water-break

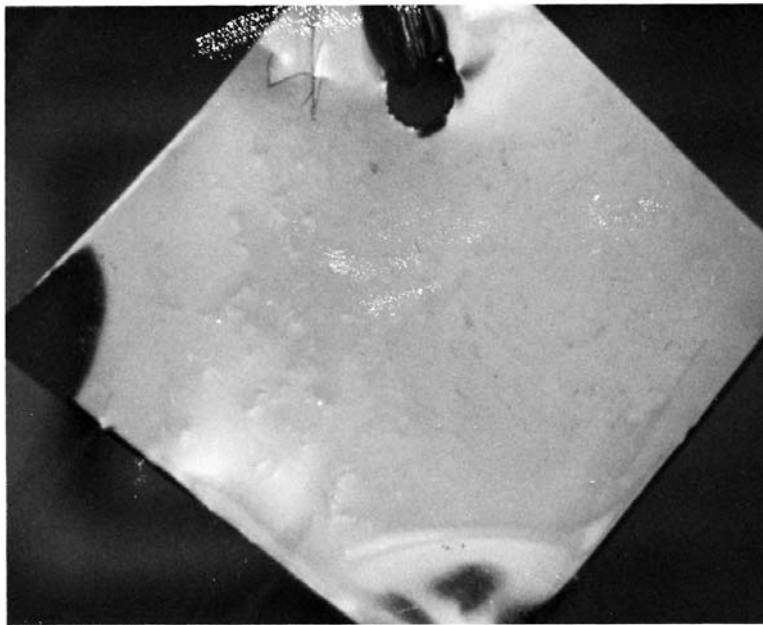


FIG. 3 Atomizer Test of Nickel Specimen Showing Trace of Hydrophobic Contaminant (x2, Reduced One-Half in Reproduction). Note Slight Discontinuities in Otherwise Uniform Water Film

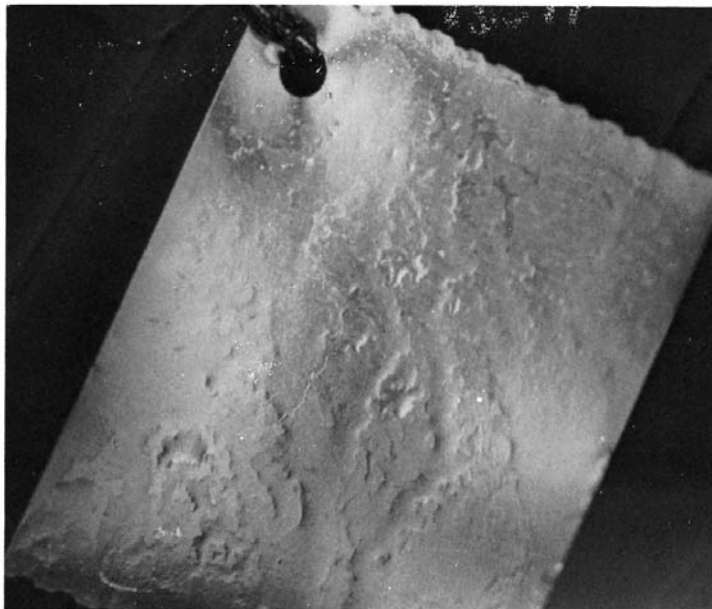


FIG. 4 Atomizer Test of Nickel Specimen Showing Traces of Hydrophobic Contaminant (x2, Reduced One-Half in Reproduction)



FIG. 5 Atomizer Test of Partially Contaminated Nickel specimen (x2, Reduced One-Half in Reproduction)



FIG. 6 Atomizer Test of Nickel Specimen with Hydrophobic Film Covering Surface (x2, Reduced One-Half in Reproduction)



**APPENDIX****(Nonmandatory Information)****X1. CALIBRATION AND STANDARDIZATION****X1.1 Calibration and Standardization**

X1.1.1 The degree of freedom of the test equipment and materials from hydrophobic contamination is proportional to the number of alternate spraying and drying cycles required to produce detectable hydrophobic contamination on a freshly cleaved mica surface. This test may be performed with greater sensitivity by masking a portion of the freshly cleaved surface under test with a mica sheet (having both surfaces freshly cleaved) during the sequential test cycle. After the desired number of sprays, the mask shall be removed and the entire surface retested for a detectable difference in wetting behavior between the two areas.

X1.1.2 To assure that test results are not obscured by flooding or by hydrophilic contaminants, a freshly cleaved mica surface from which the solvent from one drop (0.05 to 0.10 mL) of a 0.005 to 0.05% solution of oleic or stearic acid in acetone has been allowed to evaporate should clearly show 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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