Designation: F 22 – 02

Standard Test Method for
Hydrophobic Surface Films by the Water-Break Test[1]

This standard is issued under the fixed designation F 22; 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.

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. 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:*

D 351 Classification for Natural Muscovite Block Mica and Thins Based on Visual Quality[2]

3. Terminology

3.1 *Definitions:*

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

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

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 overflowing with water. The interpretation of the test is based upon the pattern of wetting. 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.

5. Significance and Use

5.1 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 also be used for the detection and control of hydrophobic contaminants in processing ambients. For this application, a surface free of hydrophobic films is exposed to the ambient and subsequently tested.

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.

7. Apparatus

7.1 *Overflow Container*, such as a glass beaker.

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

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.

8.2 *Purity of Water*—Deionized or distilled water is preferred. Water of higher ionic content may render the test destructive. The water used must be free of hydrophobic and hydrophilic substances.

NOTE 1—The freedom of the water from hydrophobic and hydrophilic contamination may be determined in accordance with Section 9.

8.3 *Acetone*.

8.4 *Mica Blanks*, preferably 25 by 50 by 0.38 mm (1 by 2 by

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[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] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

0.015 in.) or larger, having minimum ASTM Quality V6 as described in Classification D 351.

8.5 *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 demarkation between the clean and contaminated areas.

10. Procedure

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

10.2 *Testing of Ambients*—Expose a freshly cleaved mica surface to the ambient and subsequently continue as described in 11.1.

NOTE 2—Exposure may be by immersion of the mica surface in the ambient or by deposition of a sample of the ambient on the mica surface.

11. Interpretation of Results

11.1 Surfaces tested as described in 10.1 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 1 min after withdrawal of the surface from the overflow container. If hydrophobic contaminants are present, as evidenced by formation of a discontinuous water film within 1 min after withdrawal of the surface from the overflow container, the length of time necessary for the water-break to occur is a rough indication of the degree of contamination.

12. Keywords

12.1 hydrophilic films; organic contamination; surface contamination