Designation: C813 – 20

Standard Test Method for Hydrophobic Contamination on Glass by Contact Angle Measurement[1]

This standard is issued under the fixed designation C813; 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 hydrophobic contamination on glass surfaces by means of contact angle measurements. When properly conducted, this test method will enable detection of fractions of monomolecular layers of hydrophobic organic contaminants. Very rough or porous surfaces may significantly decrease the sensitivity of this test method.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

3. Terminology

3.1 *Definitions:*

3.1.1 *equilibrium contact angle, n*—the angle observable immediately after a liquid droplet is increased in size.

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

3.1.2.1 *Discussion*—Perfectly hydrophilic surfaces exhibit zero contact angles.

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

3.1.3.1 *Discussion*—Hydrophobic surfaces exhibit contact angles appreciably greater than zero (generally greater than 45° for the equilibrium angle).

3.1.4 *receding angle, n*—the smallest angle observable when a liquid droplet is decreased in size.

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

3.1.5.1 *Discussion*—See Fig. 1.

4. Summary of Test Method

4.1 The contact angle test is performed by depositing droplets of permanganate-distilled water or reagent water in accordance with Specification D1193 on the surface to be tested using a mounted hypodermic syringe, said droplets being deposited in such a way, as described herein, as to measure the equilibrium contact angle. The measurements shall be made by either of these two well-known methods: (*1*) viewing the sessile drop through a comparator microscope fitted with a goniometer scale with direct measurement of the angle, or (*2*) photographing the sessile drop and measuring the angle with a protractor. The interpretation of the measurements is based on the fact that organic contamination on surfaces results in contact angles appreciably higher than the near-zero angles measured on clean surfaces or those contaminated by hydrophilic materials.

5. Significance and Use

5.1 The contact angle test 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 conditions and is subsequently tested.

[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.02 on Chemical Properties and Analysis.

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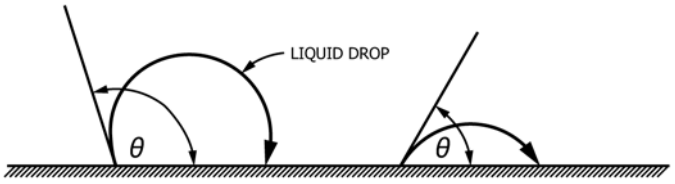
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



NOTE—the indicated angle, θ, is the contact angle. It is measured in the liquid phase, as indicated. Shown are two contact angles, >90° and <90°.

FIG. 1 Schematic Diagram Indicating Measurement of Contact Angle

6. Interferences

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

6.1.1 The presence of hydrophilic substances such as remnant cleaning detergent on the surface to be tested, or in the water used for the test, or

6.1.2 An unusually rough or porous surface.

7. Apparatus

7.1 *Low-power Comparator Microscope* (5 to 50×), with goniometer scale and light source for illumination of small drops.

7.2 *Camera,* that can take photographs of the drop if this alternative method is used.

7.3 *Protractor,* to measure the angle if the alternative method is used.

7.4 *Hypodermic Syringe or Micrometer Buret,* in a fixed mount. The needle of the syringe or the buret tip should be BD gauge 20 to 26 or equivalent and should have a perfectly clean square-cut tip free of any foreign material that may impede drop formation.

7.5 *Movable Platen,* on which to mount the specimen when viewing the drop in profile.

8. Reagents and Materials

8.1 *Purity of Water*—Either Type II reagent water in accordance with Specification D1193, or water distilled from a 3 g/L potassium permanganate solution is usually satisfactory. The water used must be free of hydrophobic and hydrophilic substances. To test the purity of the water for freedom from hydrophobic contamination, a freshly cleaved sheet of mica shall be used as the test surface for the measurement of the equilibrium contact angle of the water as described in Section 10. If the average of two such equilibrium contact angle determinations is not less than 4°, further purification of the water is required.

8.2 *Mica.*

8.3 *Polytetrafluoroethylene Sheet.*

9. Calibration and Standardization

9.1 Contact angles of water on clean polytetrafluoroethylene shall be measured to familiarize the operator with the test and serve as a check on the equipment and techniques. Equilibrium angles on polytetrafluoroethylene vary from 108 to 125°, depending on the particular type of this high polymer used.

10. Procedure

10.1 Randomly select at least two sites where measurements will be made on the test surface. Bring the syringe needle into close proximity to the test surface and, with the test surface in a horizontal position, manipulate the hypodermic syringe so as to force a drop having a volume of 0.02 to 0.05 mL onto the surface to give an equilibrium contact angle (Note 1). The needle should remain immersed in the drop and should be centered in the drop. Care must be taken that when the syringe plunger is released no motion is imparted to the plunger so as to cause any retraction of the drop. Any retraction will cause the contact angle to be less than the true equilibrium contact angle (Note 2). For each site, measure two equilibrium contact angles by measuring the contact angle on the left side and on the right side of the drop after each of two successive incremental increases in the drop size (a total of four measurements for each drop for the equilibrium angle). By making left- and right-side determinations, the effect of a slightly nonlevel surface is alleviated. Where any consistent differences between the left-side and right-side determinations are noted, it is preferable to level the platen before proceeding (Note 3).

NOTE 1—Drops that are not deposited on a surface so as to exhibit equilibrium or receding angles may have any value of the contact angle between this range of values.

NOTE 2—It is important that when making an equilibrium angle measurement, water be added to the drop until the drop advances.

NOTE 3—Because of possible contamination by material present in the atmosphere, it is desirable to make the measurements promptly and to allow samples to sit unprotected no longer than necessary. Storage of samples in plastic containers or in containers with plastic or coated liners should also be avoided. It is also very important to clean syringes and needles carefully before making determinations on different specimens. Silicone grease is a common contaminant that must be avoided.

11. Interpretation of Results

11.1 Consider surfaces tested as described in Section 10 free of hydrophobic contaminants by this test if the equilibrium angle is no greater than 5°. Under these conditions, organic contamination will be no greater than several percent of a monomolecular layer in coverage. It must be kept in mind that very clean surfaces without oxide layers such as silicon and gold are nonwettable by water and exhibit high contact angles.

12. Precision and Bias

12.1 Interlaboratory tests show that between-laboratory variations are about ±10°. Within-laboratory variations can be considerably less if the test is conducted with care.

13. Keywords

13.1 contact angle; contamination; glass; organic