Designation: D7334 – 08 (Reapproved 2013)

Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement[1]

This standard is issued under the fixed designation D7334; 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 practice covers the measurement of the angle of contact when a drop of liquid is applied to a coated surface, substrate, or preformed disk of pigment.

1.2 There are two types of contact angles, advancing and receding. This standard deals only with advancing contact angles.

1.3 This practice is intended to supplement the manufacturer's instructions for the device being used to make the measurements, but is not intended to replace them.

1.4 A common test liquid is water, but many other liquids such as solvents, surfactant and dispersant solutions and even liquid paints can be used.

1.5 This practice is based on goniometry, which involves the observation of a sessile drop of test liquid on a solid substrate.

1.6 Although contact angles are governed by surface tension, this standard cannot be used to measure surface tension directly.

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.8 *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]

D1193 Specification for Reagent Water

D5725 Test Method for Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester (Withdrawn 2010)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *advancing contact angle, n*—the contact angle that is measured immediately after the sessile drop is placed on the surface.

3.1.1.1 *Discussion*—The drop has the maximum volume allowable for the liquid-solid interfacial area: any addition will make the drop expand and increase the liquid-solid interfacial area. This can be thought of as the "wetting angle" because the drop is ready to wet additional area. This is the contact angle used in this method and in most coating measurements.

3.1.2 *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.

3.1.2.1 *Discussion*—This is the angle formed by a liquid at the three phase boundary where a liquid, gas (air) and solid intersect.

3.1.3 *receding contact angle, n*—the contact angle measured when material is removed from the drop so that it contracts.

3.1.3.1 *Discussion*—The liquid-solid interfacial area will decrease. This is the "de-wetting angle."

3.1.4 *sessile drop, n*—drop of liquid on the upper side of a horizontal surface (as in Fig. 1).

3.1.5 *surface tension, n, and surface energy, n*—the terms surface tension and surface energy are often used interchangeably.

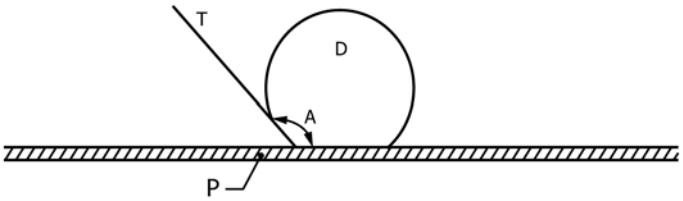
3.1.5.1 *Discussion*—They are the same numerically, although they have different units. They are measures of an effect that arises from unbalanced molecular cohesive forces at a surface that cause the surface to contract and behave like a film or membrane. The surface molecules have higher free energy than those in the bulk material. The excess free energy is what we call surface energy, which has units of energy/unit area such as Joules/cm^2. However, what we measure is surface tension,

[1] This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved June 1, 2013. Published June 2013. Originally approved in 2008. Last previous edition approved in 2008 as D7334 – 08. DOI: 10.1520/D7334-08R13.

[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] The last approved version of this historical standard is referenced on www.astm.org.



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

FIG. 1 Measuring Angle of Contact

the force necessary to break a film of a given length, usually in dynes/cm or Newtons/m.

4. Summary of Practice

4.1 A drop of a specified volume of water or another agreed upon test liquid is applied to a test specimen using a syringe.

4.2 The contact angle is measured by either of two methods:

4.2.1 by viewing the sessile drop through a microscope fitted with a goniometer scale for direct measurement of the angle,

4.2.2 by capturing an image of the drop, then measuring with a protractor or using appropriate software to process the image and measure the angle.

5. Significance and Use

5.1 This standard is useful for characterizing the wettability of surfaces. A surface that is easy to wet is one over which a coating is more likely to give good adhesion and appearance and less likely to suffer surface tension related defects such as crawling, cratering, pinholing and orange peel.

5.2 This standard also can be used to test pigment surfaces for wettability, particularly by potential surfactant- or resin-based dispersants or mill bases. Easily wetted pigments are more likely to be easy to disperse and dispersants/mill bases that wet pigments of interest are more likely to disperse those pigments well.

5.3 Although the contact angle is governed by the surface tensions of the test liquid and test surface, the angle cannot provide a surface tension value directly.

5.4 A low advancing contact angle value (<45°) is indicative of wetting and angles of 10 to 20º are indicative of excellent wetting.

5.5 Water can be used as a test liquid to establish (via the advancing contact angle) whether a surface is hydrophilic (angle <45º), hydrophobic (angle >90º) or somewhere in-between (angle of 45 to 90º). Water contact angles have been used to estimate surface cleanliness before and after cleaning operations, ease of wettability of surfaces by waterborne coatings and the effectiveness of rinsing processes.

5.6 An organic liquid such as a solvent also can be used to characterize a substrate, coating or pigment. The resultant contact angle will depend on the surface tensions of the liquid and the test surface. A low surface tension (energy) test surface will not be wet by a high surface tension liquid.

5.7 In addition to water and solvents, a surfactant dispersion or dispersant solution can be used to test a pigment surface. Any test liquid that is a potential dispersant for a test pigment must wet the pigment well or it will not work as a dispersant.

5.8 Contact angle measurements can be used to map surfaces in terms of hydrophilicity, presence of low surface tension components or contaminants, or variations in composition. Other analytical methods such as infrared microscopy would be needed to identify the chemical moieties that give the contact angle differences.

5.9 This test method can be used on nearly all coatings and substrates and may be extended to pigments by compressing the pigment powder into a solid disk.

6. Interferences

6.1 The following factors may interfere with results:

6.1.1 Dirt or fingerprints on the surface being tested.

6.1.2 A rough or porous test surface such that the drop sinks in rapidly.

6.1.3 A curved test surface such that angles are difficult to measure.

6.1.4 Low humidity (<50 % RH) when water is the test liquid such that the contact angle changes rapidly.

7. Apparatus

7.1 A horizontal stage onto which a flat surface is mounted,

7.2 A device (generally a syringe) to place a droplet on the surface.

7.3 An enclosure (if desired) to prevent interference of vapor-borne impurities and to secure saturation of vapor (particularly useful in measuring water angles).

7.4 A light source to illuminate the three-phase contact region from behind.

7.5 A microscope which magnifies the contact area. It must be able to travel along when the drop advances. Nowadays eyepieces and still cameras usually are replaced by digital video cameras (CCTV) provided with appropriate software to process the image and determine the contact angle. A description of an automated instrument for measuring angle of contact and a method for using it can be found in Test Method D5725.

8. Test Liquids

8.1 The liquid(s) selected for testing purposes are chosen based on the characteristics of the surface that are of interest.

8.2 Water is used to determine wetting characteristics of that liquid on the coating or substrate and/or to characterize the hydrophilicity and hydrophobicity of that surface.

8.3 Organic test liquids such as solvents must be of reagent grade or better.

8.4 Water—Type II reagent water (distilled) in accordance with Specification D1193.

8.5 Paint may be used as a test liquid to evaluate the ability of that paint to wet a substrate or primer.

8.6 A mill base, dispersant solution or surfactant dispersion may be used as a test liquid to evaluate the ability of that material to wet a pigment.

9. Test Specimens

9.1 If the part or panel of interest is too large to fit on the stage, then test specimens should be cut to a size appropriate for the instrument being used. They shall be cut in such a way as to be thoroughly representative of the sample.

9.2 The actual test areas tested shall not contain visible blemishes or defects and shall not be touched with the fingers or contaminated in any other way.

9.3 If contamination or improper handling is suspected, the specimen may be rinsed with water or washed with laboratory detergent and water. However, cleaning may affect the results and must be noted on the report.

9.4 Pigment specimens shall be in the form of disks prepared in a press such as those used to prepare KBr disks for infrared analysis.

10. Procedure

10.1 Test the specimens at a temperature of 23 ± 2°C (73.5 ± 3.5°F) and at a relative humidity ≥50 % unless otherwise agreed upon.

10.2 Set up the goniometer and level the stage in accordance with the manufacturer's instructions.

10.3 Place the test specimen on the instrument, if necessary holding it in close contact with the stage by means of small weights, clips, or whatever fixtures are associated with the instrument.

10.4 Contact angle drop— set the tip of the hypodermic needle at the distance from the surface recommended by the manufacturer of the instrument (3 mm (⅛ in.) has worked well for testing coatings) and deposit a drop of test liquid no greater than 20 µL in size on the specimen. With automated equipment, use the drop size recommended by the manufacturer (usually ~ 5 µL). With manual equipment, 20 µL is commonly used. For best results, drop size should be controlled to ±0.1 µL. The contact angle measured on this drop will be an advancing contact angle.

10.5 Adjust the goniometer eye piece and the internal measuring mechanism so that the interior angle of each of the two points of contact of the drop can be determined (see Fig. 1) or focus the camera or video device so that the image of the drop can be captured.

10.6 Make two angle measurements (one on each drop edge) of each of three drops on the specimen. If the contact angles on two edges are significantly different, the values should be eliminated and the test repeated. The contact angle for the specimen shall be the average of the six angles measured.

10.7 Water contact angles must be measured rapidly (within 30 s of depositing the drop) to avoid changes in angle as the water evaporates.

11. Report

11.1 Report the following information:

11.1.1 Apparatus used.

11.1.2 Identification of the specimen.

11.1.3 Identification of the test liquid.

11.1.4 Average contact angle liquid on the specimen.

11.1.5 If the specimen was cleaned, this must be noted.

11.1.6 Whether or not a humidity chamber was used for a water contact angle measurement.

12. Keywords

12.1 advancing contact angles; contact angles; surface tensions; wettability; wettings