



Designation: D5946 – 17

Standard Test Method for Corona-Treated Polymer Films Using Water Contact Angle Measurements¹

This standard is issued under the fixed designation D5946; 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 (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers measurement of the contact angle of water droplets on corona-treated polymer film surfaces.

NOTE 1—This standard is identical to ISO 15989.

1.2 The values stated in SI units are to be regarded as the standard. The 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.*

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

D618 Practice for Conditioning Plastics for Testing

D724 Test Method for Surface Wettability of Paper (Angle-of-Contact Method) (Withdrawn 2009)³

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*⁴

ISO 15989 Plastics—Film and Sheeting—Measurement of Water-Contact Angle of Corona-Treated Films

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *static contact angle, θ (degrees), n* —the angle between the substrate surface and the tangent line drawn to the droplet surface at the three-phase point, when a liquid drop is resting on a plane solid surface.

3.1.1.1 *Discussion*—Contact angle values can be determined as follows: (1) by analyzing an image of a droplet using various projection or reflective devices and measuring the contact angle directly with a protractor by using tangential alignment of a cursor line or the use of reference alignment procedures (Test Method D724); or (2) by analyzing an image of a droplet using various projection or reflective devices and measuring the height and width on the substrate surface to calculate the contact angle as follows:

$$\theta = 2 \cdot \arctan(H/R) \quad (1)$$

where:

θ = contact angle,

H = height of a droplet's image, and

R = half its width.

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.19 on Film, Sheeting, and Molded Products.

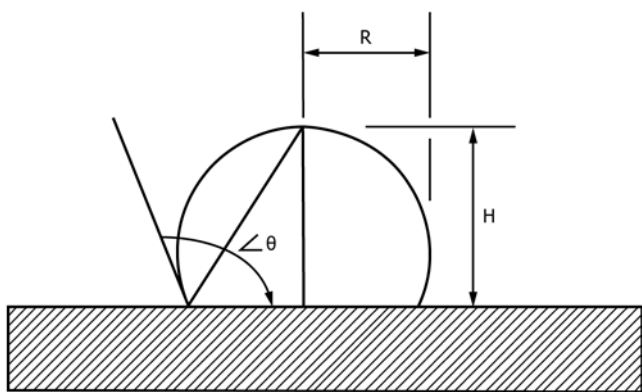
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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

*A Summary of Changes section appears at the end of this standard



3.1.1.2 *Discussion*—Instrumentation is also available that utilizes an image and directly determines the contact angle based on the calculation shown for Method (2) of U.S. Patent No. 5,268,733.

3.1.1.3 *Discussion*—Method (1) may be biased due to the subjective nature of visually finding a tangent to the droplet image at the three-phase point; if Method (1) is to be used, the bias of each operator's measurements shall be determined. Because Method (2) utilizes measured dimensions of the droplet, it would be expected to have less bias. Comparative analyses using these various procedures have not been conducted. Therefore, caution is recommended when comparing data obtained from these different methods.

3.1.1.4 *Discussion*—Method (2) may not be applicable in special cases in which the contact angle is greater than 90°

4. Summary of Test Method

4.1 In this test method, drops of water are placed on the surface of a film sample, and the contact angle values are measured and then averaged.

5. Significance and Use

5.1 The ability of polymer films to retain inks, coatings, adhesives, etc. is primarily dependent on the character of their surfaces and can be improved by one of several surface-treating techniques. The electrical discharge treatment, such as corona treatment, has been found to increase the wetting tension of a polymer film. The stronger the treatment, the more actively the surface reacts with different polar interfaces. It is therefore possible to relate the contact angle of a polymer film surface to its ability to accept and retain inks, coatings, adhesives, etc., if the ink, coating, or adhesive contains the polar functionalities. Contact angle in itself is not a completely acceptable measure of ink, coating, or adhesive adhesion.

5.2 The wetting tension of a polymer film belongs to a group of physical parameters for which no standard of accuracy exists. The wetting tension of a polymer cannot be measured directly because solids do not change shape measurably in reaction to surface energy. Many indirect methods have been proposed.⁵ Different test methods tend to produce differ-

ent results on identical samples. Practical determination of a solid's surface energy uses this interaction of the solid with test liquids.

5.3 Although the level of surface treatment of polymer films has been traditionally defined in the industry in terms of dynes/cm (mN/m), these values are derived from a subjective interpretation of the observed test liquid behavior.

5.4 The following ranges of water contact angle values can be used as a guide for defining the level of surface treatment of polyolefins and many other polymer films with initial low surface energies:

Marginal or no treatment	>90°
Low treatment	85 to 90°
Medium treatment	78 to 84°
High treatment	71 to 77°
Very high treatment	<71°

5.4.1 The suitability of the test for specification acceptance, manufacturing control, and end use of polymer films will have to be established through capability studies for each particular film and treatment.

5.5 Almost all materials have variations in contact angle as one moves from point to point. Nonuniform treatment of film with corona treaters may also add variability to the results. Therefore, multiple measurements are necessary to reflect variation in treatment and surface roughness.

6. Interferences

6.1 The wetting tension of a polymer film in contact with a drop of liquid in the presence of air is a function of the surface energies of both the air-film and film-liquid interfaces; any trace of surface-active impurities in the test liquid or on the film will potentially effect the results. It is therefore important that the portion of the film surface to be tested not be touched or rubbed, that all equipment be scrupulously clean, and that water purity be carefully controlled. Glass apparatus, in particular, is likely to be contaminated with detergents having very low surface tension, unless specific precautions are taken to ensure their absence, such as cleaning with chromic sulfuric acid and rinsing with distilled water.

6.2 The presence of slip, antistatic, antiblocking, or other additives has the potential to change the surface of the film.

6.3 Finely embossed films add more variability to the measurements.

6.4 Polymer films often generate static charges when they are processed. Corona treatment usually exacerbates the problems. The film sample for surface measurement has the potential to carry the static charge generated in processing or may acquire a charge by friction when sliding over the specimen holder surface.

6.4.1 The static charges have the potential to cause errors in contact angle measurements. These errors occur as the electrostatic field generated by the charged sample pulls the water droplet to the film surface stronger than it would be in the absence of charges.

6.4.2 To prevent measurement errors, the film sample shall be kept free of static buildup before and during measurements by placing an air ionizer, also known as a static eliminator, in

⁵ Wu, S., *Polymer Interface and Adhesion*, Marcel Dekker, Inc., New York, NY, 1982.

the vicinity of the specimen holder. These devices produce air ions of positive and negative polarity. The charged object in the vicinity of the ionizer will attract ions of the opposite polarity and will be neutralized.

6.5 The transfer of a droplet onto the measured surface shall be accomplished following the procedure described in Section 11. An improper transfer technique increases the variability of measurements.

6.6 The contact angle measuring method using tangential alignment of the cursor line of a protractor, as described in 3.1.1.1 (I), is biased due to the subjective nature of finding a tangent to the droplet image at the three-phase point. The smaller the measured contact angle, the larger the error. The bias of each operator's measurements shall be determined if this method is to be used.

7. Apparatus

7.1 *Contact Angle Meter, or Goniometer*—The apparatus for measuring contact angles shall contain the following: (1) a liquid dispenser capable of suspending a precise droplet of the specified volume from the tip of the dispenser, (2) if equipped, with a sample holder it needs to stretch the film to provide a flat surface without wrinkles and distortions, if unit does not have a sample holder, care needs to be taken to provide a flat surface without wrinkles and distortions, (3) provisions for bringing the sample in contact with the droplet in a controlled manner to accomplish droplet transfer onto the measured surface, and (4) means for projecting a “silhouette” image of the drop with minimal distortions. The apparatus shall have means for direct angle measurements, such as a protractor, or direct measurements of the droplet's dimensions, such as a grid or scale, or both, or a suitable charge coupled device sensor and analysis hardware and software appropriate for subsequent analysis.

8. Reagents and Materials

8.1 *Purity of Water*—It is suggested to use deionized ultra filtered water should be used for testing purposes. The test liquid shall be kept in scrupulously clean containers.

9. Sampling

9.1 The minimum amount of film required for this test is an approximately 25-mm wide and 300-mm long sample strip. If a sample is taken from a roll, the direction of the sample relative to the machine direction of the web shall be noted.

9.2 Extreme care shall be taken to prevent the surface of the film sample from being touched or handled in the areas upon which the test is to be made.

9.3 The recommended number of readings per sample is ten.

NOTE 2—The number of measurements per film sample can be determined using published tables for sampling plans. Complete tables can be found in most books on quality control.⁶

9.4 For the purpose of determining the contact angle profile across the width of the roll, one contact angle measurement can be taken every 25 mm (1 in.) across the sample.

10. Conditioning

10.1 Conditioning is not generally required for routine quality assurance (QA) or process control measurements. Conditioning will potentially affect the measured value and misrepresent the actual conditions.

10.2 Conditioning is required for interlaboratory measurements intended to compare the results. Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 10\%$ relative humidity for not less than 40 h prior to testing in accordance with Procedure A of Practice D618 unless otherwise specified by agreement or the relevant ASTM material specification. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and $\pm 5\%$ relative humidity.

10.3 *Test Conditions*—Special test conditions are not generally required for routine QA or process control measurements.

10.4 *Test Conditions for Interlaboratory Studies*—Conduct tests in the standard laboratory atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 10\%$ relative humidity, unless otherwise specified by agreement or the relevant ASTM material specification. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and $\pm 5\%$ relative humidity.

11. Procedure

11.1 Place a sample strip onto the specimen holder of the instrument. Make sure the film sample is lying flat without wrinkles and distortions.

11.2 Suspend a 5 to 8- μL droplet at the end of a syringe needle. Bring the surface with the mounted sample upward until it touches the pendant drop. Then lower the surface with the sample to complete the droplet transfer. Do not drop or squirt droplets on the surface. See Fig. 1. Alternatively, the syringe may be lowered to the specimen to release the droplet.

11.3 Follow the instrument manufacturer's instructions for a maximum time between dropping of the water droplet and measurement. Use the measurement techniques listed in 3.1.1.1.

11.4 Advance the sample to place the next droplet onto a previously untouched area.

11.5 Take ten contact angle measurements on the sample.

12. Calculation

12.1 *Calculation:*

12.1.1 Calculate the average of the ten measurements.

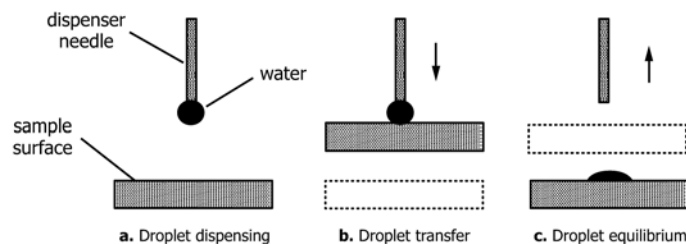


FIG. 1 Water Droplet Transfer Technique

⁶ Duncan, J. A., *Quality Control and Industrial Statistics*, 3d Ed., Irwin, Homewood, IL, 1965.