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An American National Standard

Standard Test Method for Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester[1]

This standard is issued under the fixed designation D 5725; 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.

INTRODUCTION

The property of a liquid to adhere to, or "wet", a sheeted surface, or to be absorbed by that surface, or both, is important in many aspects of paper manufacturing and converting, as well as in the end-use applications of many converted paper products.

Examples include, but are not limited to, the absorption of water or other liquid by an absorbent structure (such as an absorbent tissue or wipe); the adhesion of an ink to a polymer film or a coated or uncoated paper (such as a packaging or wrapping material); the adherence of a polymer film or sizing material to a paper substrate in a laminate or coated structure; the adhesion of a pressure sensitive tape to a release paper; the adhesion of a film to a paper substrate in a composite structure (such as a diaper or other composite structure); and the non-wetting or non-absorbency, or both, of a barrier paper.

The wetting or sorptive behavior between a liquid and a particular sheeted substrate is dependent, at least in part, upon the relationship of the surface energy (tension) of the liquid and the surface energy of the substrate. The theoretical relationship of these energies is complex, and the different mathematical models which have been proposed for adhesion, wettability, and sorption are beyond the scope of this test method, but may be found in standard texts in these areas. In many cases, however, the contact angle of the fluid which will be in contact with the substrate, or the contact angle of a liquid of known surface tension, when placed in contact with a substrate of interest, is used to understand or predict in-process or end-use results of a particular printing, adhesion, or sorptive application.

Contact angle measurements as described in Test Method D 724 or Canadian Pulp and Paper Association CPPA F.3H have been used to study and define the printability relationship between an (aqueous) ink and a paper at the water/paper interface. TAPPI T 552 and Test Method D 2578 use a somewhat different, semi-quantitative approach to provide information regarding the energy relationship between a polymer film and a nonaqueous liquid, the test end-point being the place where the contact angle between a liquid of known surface tension and the test specimen approaches zero under the conditions of the test.

The procedure presented in this test method is a simple, completely automated approach to contact angle measurement applicable to a wide range of sheeted materials and liquids where interfacial contact angles range from near zero to near 180 degrees. The automated procedure shows increased precision and greater ease in use than manual procedures.

1. Scope

1.1 This test method measures the contact angle of a test liquid in contact with a flat specimen of a film or a paper substrate under specified test conditions. This test method may be used with any liquid of interest which is compatible with the equipment used, particularly with regard to liquid viscosity, wetness, and vapor pressure (evaporation). This test method may be used with any substrate of interest, which can be cut to dimensions compatible with the equipment used.

1.2 For materials which sorb the test liquid under the specified test conditions, the rate of change of the contact angle as a function of time may be significant, and may be determined using procedures described in this test method. It is also possible to evaluate the sorptive properties of a surface, as the

[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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remaining liquid volume on top of the specimen surface is measured as a function of time.

1.3 The conditions required in this test method specify reagent water as the test liquid when testing papers designed to be absorbent, such as absorbent tissue grades.

1.4 Conditions are specified for the testing of a wide range of papers considered to be of low absorbance or nonabsorbent, including release papers, sized, coated, or unsized papers designed for printing, writing, wrapping, and similar tasks where the paper surface interaction with aqueous or solvent based inks or other aqueous or nonaqueous liquids is important. In such cases, test liquids other than reagent water, including writing and printing inks, or organic liquids or mixtures of organic liquids may be used as the test liquid upon prior agreement of those involved in the testing, provided the liquid is compatible with the equipment used. Where test liquids other than reagent water are used, the actual liquid used is reported.

1.5 Conditions are also specified for the testing of polymer films, polymer-coated papers, paper laminates, felt, textiles and non-wovens, using water or other fluids compatible with the equipment and important to the end-use applications of the materials tested, including gluing and printing.

1.6 *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]

D 528 Test Method for Machine Direction of Paper and Paperboard

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D 685 Practice for Conditioning Paper and Paper Products for Testing

D 724 Test Method for Surface Wettability of Paper (Angle-of Contact Method)

D 1193 Specification for Reagent Water

D 1968 Terminology Relating to Paper and Paper Products

D 2578 Test Method for Wetting Tension of Polyethylene and Polypropylene Films

D 5039 Test Methods for Identification of Wire Side of Paper

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *TAPPI Standard:*

T 552 Determination of Wetting Tension of Polyolefin Films and Coated Surfaces with the Mayer Rod Technique[3]

[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] Available from the Technical Association of the Pulp and Paper Industry (TAPPI), P.O. Box 105113, Atlanta, GA 30348-5113.

3. Terminology

3.1 *Definitions:* For definitions used in this test method, refer to Terminology D 1968 or the *Dictionary of Paper*.[4]

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *contact time, n*—the length of time the droplet has been in contact with the specimen surface.

3.2.2 *droplet diameter, n*—diameter of the surface of contact between the specimen surface and the droplet, shown as distance "D" in Fig. 1.

3.2.3 *droplet height, n*—height of the droplet in contact with the specimen surface, shown as distance "H" in Fig. 1.

3.2.4 *drop motion time, n*—the time it takes for the droplet to reach the specimen surface after the drop application has been triggered.

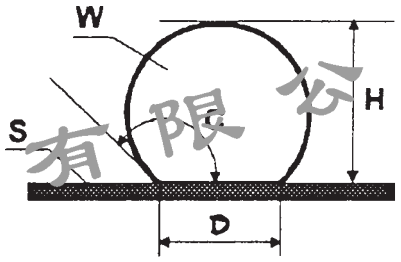
4. Summary of Test Method

4.1 A drop of a specified volume of water or another agreed upon test liquid is applied to a test specimen surface using a liquid delivery system and specified deposition parameters. Images of the drop in contact with the substrate are captured by a video camera at specified time intervals following deposition.

4.2 At a specified time after drop deposition, which is varied based upon the sorptive or barrier properties of the substrate/liquid interface, the test is terminated. The contact angle between the drop and the substrate at various time intervals following drop deposition are determined by image analysis techniques on the captured images, and the contact angle at specified time(s), the rate of change of the contact angle change as a function of time, and changes in droplet height and diameter, as well as other test variables are analyzed, based on specific information requirements for the materials being tested.

4.3 The test method is divided into two parts, Procedures A and B, which vary only in certain procedural aspects and allow the use of the procedure over the wide range of sample types described in the Introduction and Section 1.

[4] Available from TAPPI.



W—water droplet
S—specimen
D—diameter of the surface of contact of the droplet
H—height of droplet
C—contact angle

NOTE 1—For materials exhibiting sorptive properties with respect to the test liquid used, the values for contact angle, droplet diameter, and droplet height may vary as a function of time following drop deposition on the material substrate.

FIG. 1 Principle of Measurement

4.4 To identify the applicable procedure, a drop of the standardized size is formed at the tip of the liquid delivery system. The drop is then slowly lowered towards the specimen surface until contact is initiated between the liquid and the specimen. Use Procedure A if the drop releases immediately from the tip on contact with the specimen surface. Use Procedure B if the drop remains attached to the tip on contact with the specimen surface.

4.5 In order to measure the highest contact angle possible, the drop should be applied as gently as possible. With Procedure A, the drop may be applied with a very short stroke, as the drop will release from the liquid delivery system tip immediately upon contact with the specimen surface. Therefore, Procedure A should be tried as the first option.

4.6 Procedure A gives specific conditions for the testing of sheeted materials having contact angles with water less than about 100°. Materials of this type are generally sorbent papers.

4.7 Procedure B gives specific conditions for testing of sheeted materials having contact angles with water above about 100°. Procedure B is applicable when the drop is not immediately released from the liquid delivery system tip upon contact with the specimen surface.

4.8 In cases where a liquid other than water is used, the specific procedure applied will depend on the contact angle between the liquid and the specimen substrate. For example, where the film side of a paper-film laminate, or a polymer film itself, is tested with a liquid whose surface tension is approximately equal to or below that of the film, the contact angle at the liquid/substrate will approach zero, and Procedure A would be used. If the same film were tested with water as the liquid, Procedure B might be appropriate. The procedure is chosen based on the resulting interfacial wetting properties, not the identity of the liquid or specimen substrate.

5. Significance and Use

5.1 Contact angle measurements may be used to study the relative sorbtive rates of uncoated sorbent papers, or the relative printing or writing characteristics of coated or sized printing and writing papers.

5.2 The complex interaction between a liquid and a surface may be looked upon as a combination of three different processes of wetting, absorption, and adsorption. Wetting is best explained with a drop of water on a plastic film. The liquid volume remains the same, the drop base diameter will increase, and the contact angle will decrease as a function of time. When the liquid volume is reduced as a function of time, the base diameter of the drop is studied. When this diameter remains constant, the absorption is dominating. When the drop is spreading across the surface (increasing base diameter), the interaction is based on adsorption.

5.3 For sized papers, an increase in feathering is likely as the rate of change in the contact angle with time increases, indicating a relative increased degree of liquid transport or penetration (absorption) into the paper.

5.4 For sorbent papers, the change in contact angle with time may be very rapid, with those papers showing the greater relative change per unit time having the fastest rate of sorption.

5.5 For hard sized papers, little change in contact angle with time may be seen, and for laminates or polymer coated and barrier papers, release papers, or other similar specialty grades, there may be no change in contact angle over the time interval of a typical test.

5.6 It is generally found that papers having contact angles with water-based inks in the range 90 to 110° work best in printing and writing applications. Feathering may be expected for contact angles less than 90°. Breaks in the flow of ink onto the paper may occur for contact angles greater than 110°.

5.7 Because of the wide range of paper coating possibilities and ink compositions, further generalizations are difficult. However, contact angle is a precise empirical tool for use in studying specific liquid/substrate combinations for product and process improvements.

5.8 In addition, contact angle measurements on films are used to determine printing and gluing characteristics of films with specific printing inks or adhesives. In such applications, the procedure may use a constant film substrate with various test liquids of significance to a specific end-use application. By measuring substrate surface free energy and then monitoring and controlling any surface treatment of the material using contact angle measurements, improved end-use performance in gluing or printing applications is possible.

6. Apparatus

6.1 *Automated Contact Angle Tester*[5], consisting of the following components, each of which are described in detail: a light source, a video camera, a specimen stage, a liquid delivery system consisting of a pump and micro-syringe and a computer and associated software suitable for video image capture, image analysis, and reporting.

6.1.1 *Light Source*:

6.1.1.1 *Halogen Lamp*, sealed in a separate lamp housing with its own ventilating fan. Room temperature air is circulated inside the lamp housing and the warm air is then returned outside the instrument so it cannot reach the test specimen or the test liquid.

6.1.1.2 Other designs are possible, using heat dissipating filters or similar equipment to eliminate heating of the specimen or test liquid.

6.1.2 *Video Camera*:

6.1.2.1 The video camera is equipped with a lens to achieve an image view of about 10 by 7.5 mm and an electronic shutter. The shutter is set for a 1-ms exposure time for purposes of this test method.

6.1.2.2 The video camera will, depending on the video standard used, send out video images continuously at a rate of 50 (CCIR) or 60 (EIA) images per second. Hence, the time between two consecutive images is 20 ms (CCIR) or 16.7 ms (EIA). Either of these video standards may be used. The CCIR timing has, however, been used throughout this description in the timing examples.

6.1.2.3 When a droplet of a different size than standard is used, other magnifications may be needed.

[5] One contact angle tester which complies with the requirements is the FIBRO 1100 DAT Dynamic Absorption Tester, FIBRO System AB, Stockholm, Sweden. There may be others.

6.1.2.4 The depth of the focal plane must be sufficient. If this is not properly arranged, the base of the droplet will be influenced by the forward edge of the specimen.

6.1.3 *Specimen Stage*:

6.1.3.1 The specimen shall be positioned so the test surface is flat and horizontal.

6.1.3.2 To avoid the influence of capillary forces, the specimen shall be freely suspended across the wetted test area.

6.1.3.3 When the specimen is moved to a new area, avoid the wetted area of the previous drop.

6.1.4 *Liquid Delivery System*:

6.1.4.1 The pump drives a 1 mL micro-syringe. By moving the plunger forward, a droplet containing 4.0 ± 0.1 µL is delivered at the tip of a PTFE tube, with an inner diameter of 0.50 ± 0.05 mm and an outer diameter of 0.70 ± 0.05 mm.

6.1.4.2 When a droplet of different volume is requested, it is possible to use a drop size from 0.2 to 20 ± 0.1 µL.

6.1.4.3 For certain fluids or drop sizes, or both, a PTFE tube with smaller or wider inner diameter may be used.

6.1.4.4 The drop size, tubing material, and tubing dimensions stated in 6.1.4.1 are standard for this test method, and any deviations from those conditions stated shall be included in the report.

6.1.5 *Drop Applicator*:

6.1.5.1 The purpose of the drop applicator is to apply the droplet onto the specimen surface with a down-going motion ("stroke"). The length of this stroke should be as short as possible, in order to minimize the force exerted on the droplet. Depending on the wetting properties between the liquid and the specimen surface, there are two different procedures for the application of the drop. Depending on stroke length and acceleration, there are some timing considerations.

6.1.5.2 To achieve the requested timing, the drop applicator is activated (triggered) by the sync pulse from the video camera. The time difference between the video sync pulse and the activation of the drop application may not vary more than +1.0 ms.

6.1.5.3 The time elapsed from activation of the drop applicator until contact is initiated between the droplet and the specimen surface, is defined as the "drop motion time". The drop motion time shall, for any given drop size, stroke length, and distance above the specimen surface, not vary more than ±1.0 ms.

6.1.5.4 With the video standards described in 6.1.2, there is an interval of 20 ms (CCIR) or 16.7 ms (EIA) between the images captured in one sequence. If the drop applicator is activated slightly ahead (offset) of the video sync pulse (1 to 19 ms), the captured image sequence will display the drop captured somewhat later. This time shift enables capturing of images at any point between two sync pulses. For example, an offset of 5 ms will capture images at 5, 25, 45 ms, and a 12 ms offset will capture images at 12, 32, 52 ms after initial contact between the drop and the specimen surface.

6.1.6 *Video Image Capture System*:

6.1.6.1 The video image capture system shall capture a minimum of 50 video images equally spaced during the first second. After the first second images are captured less frequently, but often enough to follow the dynamic wetting/sorptive process.

6.1.6.2 The digitization grid of the captured image must be at least 512 by 512 pixels to provide sufficient details for the image analysis.

6.1.7 *Software*, which includes functions for light calibration, scale factor adjustments, trigger and capture time settings, data analysis, and reporting described as follows:

6.1.7.1 *Light Calibration*—The image delivered from the video camera depends upon many factors, such as the lamp intensity, light-reflecting system, lens, iris settings, camera sensitivity, and gain. Because of this, the image system shall have a calibration function compensating for all possible system settings. After the light calibration has been performed, there should be no adjustment to the lamp intensity, the light reflecting system, the lens or iris settings, and the camera sensitivity or gain. If such an adjustment is needed, the light calibration shall be repeated before a test is performed.

6.1.7.2 *Scale Factor Adjustment*—To allow calculations based on absolute dimensions of the viewed image, the system shall have an input for adjustment of scale factor depending on camera distance and lens magnification, as well as aspect ratio.

6.1.7.3 *Initial Contact Time Setting*—To achieve accurate contact times reported by the system, the timer has to start within 2 ms upon contact of the liquid drop and the specimen surface. This timing margin will result in a ±10 % timing error.

6.1.7.4 *Image Time Stamp*— Each captured image shall have an assigned time stamp showing elapsed time after initial contact between the liquid drop and the specimen surface. The time stamp should be accurate within ±2 ms.

6.1.7.5 *Data Analysis and Presentation*:

6.1.7.5.1 For each image captured, the base diameter, height, contour, and projected area of the applied drop is observed. From these primary observations, the contact angle, volume, and contact area of the drop are calculated.

6.1.7.5.2 The resulting data from one drop is represented by the contact angle and volume as a function of time. For summary statistics, the contact angle is reported for three selectable specific times (check points).

6.1.7.5.3 When more than one drop is measured, the average contact angle and volume is reported at the three selected check points. The coefficient of variation is used to express the drop-to-drop variation across the specimen surface.

7. Preparation

7.1 Set up the apparatus following the instructions for the equipment used.

8. Reagents and Materials

8.1 *Reagent Water*— Water of any of the types listed in Specification D 1193 may be used in this test method.

8.2 Other liquids and test surfaces may be used as agreed upon by users of this test method, provided they are agreed upon in advance, are compatible with the equipment used, and are stated in the report.

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9. Calibration

9.1 The automated contact angle tester uses a video image. Therefore, the brightness of the image and the light sensitivity of the system is crucial for the image analysis. Calibration is required for thresholding and scale factor. The equipment shall also have an adjustment for the drop application, resetting the timer to zero on contact between the liquid drop and the specimen surface. Consult the manual with regard to the instrument used.

9.2 For verification, an artificial drop may be produced from a steel ball pressed into a flat metal body. When the artificial drop is put into the instrument, it will appear as a drop to the system. If the physical dimensions of the steel ball are accurately measured, the contact angle and the volume of the artificial drop may be calculated as part of a sphere.

10. Maintenance

10.1 Consult the manual with regard to the instrument used.

11. Sampling, Test Specimens, and Test Units

11.1 *Sampling*—Sample the material to be tested as described in Practice D 585. Where testing is for other than acceptance purposes, Practice E 122 is recommended as an alternative.

11.2 *Test Specimens*— Determine and mark the machine direction of each test unit following Test Method D 528. Be careful not to touch the areas to be tested or contaminate them in any other way.

11.2.1 Determine and mark the felt and wire sides of each test unit, if applicable, following Test Methods D 5039. Where the terms“ felt” and “wire side” do not apply, assign arbitrary designations such as “top” and“ bottom” to the principle surfaces of the test unit, based on the side which is intended to be in contact with water or other liquid in the end-use or other application of interest.

11.2.2 When the specimen thickness is not greater than 1.0 mm, cut three 14.9 ± 0.1 mm wide and about 300 mm long clean specimen strips, free of folds, wrinkles, blemishes, water marks, and other defects not normally inherent in the specimen. This width is used because a wider specimen may cause curl on thin paper qualities during the test. On a more narrow specimen strip, a droplet of the standardized size may reach the edge of the specimen during a test, due to wetting or adsorption.

11.2.3 Where the specimen thickness is greater than 1.0 mm, an adapter for thick specimens may be installed as an option. Although thick specimens will not curl during the test, take care that the liquid drop does not reach the edge of the specimen before the test has terminated.

11.2.4 Curled specimen strips that are not penetrated by the test liquid must be mounted on a carrier strip with double-sided adhesive tape to achieve a flat test surface.

11.2.5 If the test surface has different wetting characteristics in the machine and cross directions, specimens are to be cut in the two directions and marked accordingly. Alternatively, one set of specimens cut at a 45° angle relative to the machine direction may be used, provided this has been agreed in advance and is stated in the report.

12. Conditioning

12.1 Condition the test specimens in accordance with Practice D 685.

13. Procedure

13.1 *General*:

13.1.1 Fill the liquid delivery system with the required test liquid following the manufacturer’s instructions.

13.1.2 Load the test specimen into the feed system.

13.1.3 Select the proper settings of drop size, stroke length, and distance between the drop and the specimen surface, depending on Procedure A or B.

13.1.4 Adjust the position of the specimen surface and liquid delivery tip.

13.1.5 Pump out a drop at the end of the liquid delivery system tip.

13.1.6 Apply the drop onto the specimen surface.

13.1.7 Capture a sequence of images until the drop has been absorbed or the testing time has elapsed.

13.1.8 Compile the image data.

13.1.9 Change the specimen position and repeat the sequence from 13.1.5, until the requested number of drops have been evaluated.

13.1.10 Calculate the average results for the drops applied.

13.1.11 Consult the instruction manual of the instrument used for specific operating instructions.

13.2 *Procedure A*— This procedure is applicable to sheeted materials having contact angles with water less than about 100°, and which will cause the water drop to release from the liquid delivery system tip on contact with the specimen surface. The procedure is performed as follows:

13.2.1 Pump out the standardized drop size at the end of the liquid delivery system tip. Move the drop towards the specimen surface until the distance is 0.5 ± 0.1 mm.

13.2.2 Select too short a stroke for the drop applicator, and check that the drop does not reach the specimen surface when the drop applicator is triggered.

13.2.3 Increase the stroke length gradually, until the drop reaches the specimen surface and releases from the tip on contact with the specimen surface.

13.3 *Procedure B*— This procedure is applicable to sheeted materials having a contact angle with water above about 100°, which generally do not cause the immediate release of the water drop from the liquid delivery system tip on contact with the specimen surface. The procedure is performed as follows:

13.3.1 Pump out the standardized drop size at the end of the liquid delivery system tip. Move the drop towards the specimen surface until the distance is 0.5 ± 0.1 mm.

13.3.2 Select too short a stroke for the drop applicator and check that the drop does not reach the specimen surface when the drop applicator is triggered.

13.3.3 Increase the stroke length gradually until the drop reaches the specimen surface and remains attached to the liquid delivery tip on contact with the specimen surface.

13.3.4 Pull the tubing slowly away from the specimen surface, until the drop releases from the liquid delivery tip.

13.3.5 Advance the specimen to a dry test area and pump out a new drop of the standardized size.

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13.3.6 Increase the stroke length gradually until the drop comes in contact with the specimen surface and releases from, or slides off, the tip. If the drop stays attached to the tip on contact with the specimen surface, repeat from 13.3.4.

14. Calculations

14.1 All calculations are made on the two-dimensional images captured from the video camera. It is assumed that the drop is symmetrical around its vertical axis. For a paper surface, the degree of anisotropy will result in an elliptical contact area, and the reported contact angles are higher for cross direction specimens (viewed in the machine direction) compared to machine direction specimens. The reported volumes are higher for machine direction specimens (viewed in the cross direction) compared to cross-direction specimens. Refer to 11.2.5 for details on non-symmetrical surface properties.

14.2 The contour of the drop is traced and the curve is used to calculate the average contact angle and the volume.

14.3 When the specimen surface is rough, the drop contour cannot be traced all the way down to the surface. Instead, a certain distance from the specimen surface is excluded from the analysis, and the corresponding contour is compiled from the remaining drop contour. The standard distance excluded from analysis is 0.1 mm. Other distances may be used provided they are agreed upon in advance by users of this test method, are compatible with the equipment used, and are stated in the report.

14.4 The data compiled from all captured images in one sequence are represented by three selected check points (1, 1.0, and 10 s. Other check points may be used if agreed upon by users of this test method provided they are agreed upon in advance, are compatible with the equipment used, and are stated in the report.

14.5 The contact angle is calculated for each sample by averaging the compiled contact angle values for the selected check points. Calculate the coefficient of variation of the averaged contact angle values.

14.6 The volume is calculated for each sample by averaging the compiled volume values at the selected check points. Calculate the coefficient of variation of the averaged volume values.

15. Interpretation of Results

15.1 *Paper Specimens Tested With Water and Ink*:

15.1.1 Contact angle data may be used as an indication of the writing quality, ruling quality, or both, when a paper substrate is tested with water. Other variables, for example, the type and uniformity of sizing, are quite important in ruling and writing quality, as well.

15.1.2 When papers which are to be ruled are tested with water, papers having a contact angle between 90 and 110° will generally yield excellent ruling. When the contact angle is greater than 110°, breaks in the ruled lines are more likely to occur, while at contact angles less than 90°, feathering is more likely.

15.1.3 For writing papers, paper having an initial contact angle less than 90° may feather immediately when written upon. For writing papers having an initial contact angle of greater than 90°, which show a change of 5 % or greater in contact angle over the interval from 5 to 60 s after drop deposition, feathering upon standing may occur, depending upon the drying time required for the ink used.

15.1.4 For sorbent papers tested with water, those having the lowest initial contact angle, or the greatest change in contact angle with time, or both, will generally have the greatest rate of sorption upon initial contact with water.

15.1.5 In rotogravure printing, the combination of rotogravure ink and paper surface properties may result in bad dot-edge definition. In severe cases a “donut effect” with a less intense color tone in the center of the printed dot may appear. These effects correlate well with differences in contact angle at contact times of about 100 ms. After some 500 ms, however, no difference in contact angle may be detected.

15.1.6 Another common printing problem is print mottle or“ cloudiness”. This effect correlates to the “wetting retardation time,” defined as the time elapsed from initial contact between the fluid and the specimen surface until the contact angle goes below 90°.[6]

15.1.7 In some applications, the “initial contact angle” of a sorptive process is of special interest. As the drop has to stabilize on the surface, it is usually not possible to measure the initial contact angle. When the sorptive process is mainly absorption,[7] however, the contact angle rate of change may be extrapolated down to time equals zero for useful results.

15.2 *Film Specimens*:

15.2.1 Untreated polymer films generally exhibit surface-free energy values in the range of 20 to 50 mJ/m^2, depending upon the chemical formulation. For example, treatment with a corona discharge unit may increase the surface-free energy of the base film. When tested with reagent water having a nominal surface tension of 72 mJ/m^2, the contact angle between reagent water and a film specimen will be greater than zero, indicating that the water does not completely “wet” the film. The greater the difference between the surface tension of the water and the surface-free energy of the film, the farther from zero will be the contact angle.

15.2.2 If a liquid has a surface tension in between the surface-free energy of the film specimen and the surface tension of the test liquid, the contact angle of the test liquid will normally be less than that of the test liquid and the film. Likewise, if a liquid having a surface tension greater than that of reagent water is used in the test, the measured contact angle will be greater than when reagent water is used. Exceptions may, however, occur if the polarity of the used liquid differs strongly from that of reagent water.

15.2.3 Where the test liquid is equal to, or less than the surface-free energy of the specimen, the contact angle will approach zero. By testing the film with liquids of different surface tensions and plotting the contact angle versus liquid surface tension, it is possible to determine the “critical surface tension of wetting” of the film as the surface tension where the

[6] Louman, H. W., “Mottling and Wettability,” *TAPPI Proceedings 1991 Coating Conference*, pp. 505–519, 1991.

[7] Strom, G., “Wettability of kraft pulps-effect of surface composition and oxygen plasma treatment,” *Journal of Adhesion Science Technology*, Vol 6, No. 6, pp. 745–761, 1992.

contact angle becomes zero. The critical surface tension of wetting is a useful empirical parameter, which becomes equal to the surface-free energy of the solid when the interfacial surface tension between the liquid having zero contact angle and the solid is zero. Most often, the interfacial tension is small, and thus the surface-free energy of the solid is approximated by the value of critical surface tension of wetting.

15.2.4 In cases where one liquid exhibits sorption by a substrate or penetrates the substrate to some depth and a second does not (for example, in the case of a sorbent paper tested with water and oil), the relationship between contact angle and the liquid and substrate surface-free energies may not be valid because of the impact of other variables, such as the capillary (pore) radius of the substrate. Adhesive viscosity differences may overshadow the impact of contact angle differences.

16. Report

16.1 Report the following information:

16.1.1 The test liquid used, if other than reagent water.

16.1.2 The droplet size, if other than the standardized size.

16.1.3 The center-to-center distance between two consecutive liquid drops applied on the same specimen surface.

16.1.4 The stroke applied.

16.1.5 The drop distance from the specimen surface, if other than the standard.

16.1.6 The number of drops used for the test.

16.1.7 The average contact angle compiled at the selected check points for all the drops in a test, including the coefficient of variation.

16.1.8 The average volume compiled at the selected check points for all the drops in a test, including the coefficient of variation.

16.1.9 Any other information, as agreed upon in advance between the users of this test method.

17. Precision and Bias

17.1 *Precision*—The following estimates of precision are based upon an interlaboratory trial involving 19 laboratories and six lots of printing papers of different types tested using water as the liquid. The papers used included papers suitable for Procedure A (contact angle with water less than about 100°) and Procedure B (contact angle with water above 100°). Not all laboratories participated in all phases of the study. Data analysis was performed as described in Practice E 691 using ASTM Interlaboratory Data Analysis Software (copyright 1990.)[8]

17.1.1 *Repeatability* (of test results within a single laboratory, each test result being the average of ten determinations):

$$\text{Contact angle: } 7\ \% \tag{1}$$

$$\text{Drop volume: } 11\ \% \tag{2}$$

17.1.2 *Reproducibility* (of test results between laboratories, each test result being the average the ten determinations):

$$\text{Contact angle } 13\ \% \tag{3}$$

$$\text{Drop volume } 20\ \% \tag{4}$$

17.1.3 The repeatability and reproducibility values quoted here are estimates of the maximum difference (95 % confidence limit) which should be expected when comparing replicate measurements. Precision for values of contact angle below 30° and drop volumes below 20 µL may be a higher percentage than that shown here. Further, these values may not apply for all materials or liquids other than water.

17.2 *Bias*—It is not possible to make a statement regarding the bias of this test method for measuring contact angle, as the values reported are based on the automated equipment described in the apparatus section. Other (manual) procedures may give results equal to, higher than, or lower than the results reported here, based in part on the skill of the operator in performing those manual procedures.

18. Keywords

18.1 absorption; adhesion; adsorption; contact angle; paperboard; printing; wettability; wetting

[8] Complete details of the precision study may be found in a research report available from ASTM Headquarters. Request RR: 06-1003.

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