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An American National Standard

Standard Test Method for Surface Wettability of Paper (Angle-of-Contact Method) [1]

This standard is issued under the fixed designation D 724; 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 measures the angle of contact when a drop of liquid is applied to the surface of a specimen of paper.

1.2 The measurement of both the initial angle of contact and the angle of contact after a defined elapsed period of time may be measured.

1.3 A standard liquid (ink) for making measurements is described.

1.4 Other liquids including printing inks, writing inks, adhesives, oils, and coating materials may be used upon agreement of those involved in the testing being done.

1.5 Two types of apparatus for measuring the contact angle using the procedure in this test method are described. Additional instruments that can perform the procedure in this test method may also provide equivalent results. A different procedure using an automated instrument for measuring angle of contact will be found in Test Method D 5725

1.6 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *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 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Products

D 685 Practice for Conditioning Paper and Paper Products for Testing

D 5725 Test Method for Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester

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

2.2 *TAPPI Standard:*

TAPPI Test Method T 458 Surface wettability of paper (angle of contact method)[3]

3. Significance and Use

3.1 This test method is useful in measuring the interaction of paper with liquids in a variety of applications.

3.2 When using the standard ink described in this test method, the contact angle measured initially is often considered a measure of the ruling quality of the paper.

3.3 When using the same standard ink, the change in contact angle between the times specified in this test method, 5 and 60 s, is often considered a measure of writing quality of the paper when an aqueous ink is used.

3.4 In many applications, a contact angle between 90 and 100 degrees, when a paper is tested with the standard ink, indicates that the paper will be suitable for general activities where water-based inks are used.

3.5 When this test method is used with specific non-standard inks of interest, or non-aqueous liquids, the users must agree in advance the data to be collected and how the data will be used.

3.6 Additional suggestions for more specific use of this test method will be found in Section 10.

4. Apparatus

4.1 *Drop Projection Instrument*—An instrument capable of projecting the image of a drop of liquid on the surface of paper onto a screen so that the angle of a line tangent with the drop at the paper surface can be measured. The general concept is shown in Fig. 1 and shall consist of the following:

NOTE 1—While the projection instrument illustrated and described is antiquated, it describes the concept of the measuring procedure. Instruments are available in a more convenient form that serve the same purpose, that is projecting the image of the drop on a screen.

4.1.1 *Angle Measuring Device*—A protractor or similar device for measurement of contact angle.

4.1.2 *Filter*, used to reduce the heat on the specimen and drop.

[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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[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, Technology Park/Atlanta, P.O. Box 105113, Atlanta, GA 30348.

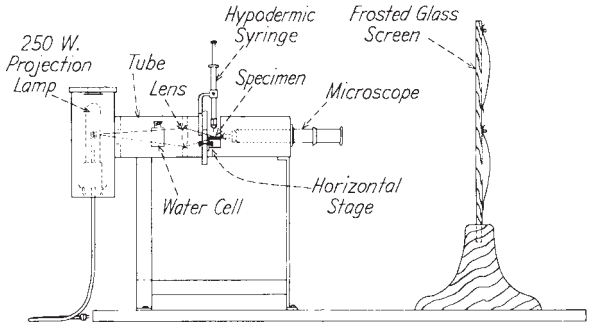


FIG. 1 Projection Apparatus

4.1.3 *Lamp*—A ventilated lamp house containing a light source.

4.1.4 *Microscope*—A microscope draw tube suitable for projecting the image of the drop on a screen with an enlargement of 25 to 30 times.

4.1.5 *Screen*—A frosted glass screen on which the drop is projected is required.

4.1.6 *Stage*—A horizontal stage capable of holding the test specimen, with means for vertical adjustment of the stage.

4.1.7 *Tube and Lens*— A tube capable of containing a lens to concentrate the beam of light.

4.2 *Hypodermic Syringe*—A syringe, such as a 1-mL hypodermic, equipped with a No. 27 stainless steel needle, capable of providing 150 to 200 drops, 1 mL, is suitable for use with water-like liquids. More viscous liquids may require a needle of different size.

4.3 Other instruments suitable for measuring contact angle include a device where the screen is replaced by a camera using instant developing film.

4.4 *Reflective Goniometer*—An instrument consisting of a controlled light source, stage to hold the test specimen, and a microscope for direct viewing of the drop on the paper specimen is required. The microscope shall be fitted with an ocular graduated in degrees and adjustable so that the angle of contact between a line tangent to the drop at the paper surface can be determined.

5. Test Liquids

5.1 The liquid selected for testing purposes is a function of the characteristic to be evaluated. The standard ink (see 5.4) is used to determine writing characteristics of paper. The ink described is typical of that used in fluid ink writing instruments.

5.2 Water is used for determining wetting characteristics of that liquid when paper is expected to be exposed to water only.

5.3 Other liquids may be used using the technique described for special applications. There may be a need under those conditions to modify the test method from the standpoint of size of droplet used and the time at which contact angle is to be measured. Slow wetting liquids may be evaluated over a longer time period.

5.4 *Standard Ink*— A standard ink having the following composition shall be used for determining the writing qualities of paper:

Tannic acid, g	11.5
Gallic acid crystals, g	3.8
Ferrous sulfate 7 hydrate, g	15.0
Hydrochloric acid 1 *N*, mL	12.5
Phenol, g	1.0
Soluble blue dye, Color Index 42755, Acid Blue 22, g	3.5
Water, distilled or deionized to make 1000 mL at 20°C (68°F)	

All chemicals used in preparing the ink shall be of reagent grade or equivalent quality. Some blue dyes react with phenol to cause a film having a metallic appearance and such dyes shall not be used. The ink shall be prepared as follows:

5.4.1 Dissolve the tannic acid and gallic acid in about 400 mL of water at about 50°C (122°F). In a separate vessel, dissolve the ferrous sulfate in about 200 mL of water that contains the hydrochloric acid. In a third vessel dissolve the dye in about 200 mL of water. Mix the three solutions in a 1-L volumetric flask, rinse the vessels with small portions of water, and add the rinsings to the flask. Add the phenol. Dilute the water to 1 L at 20°C (68°F).

5.5 *Water*—Distilled or deionized.

5.6 *Other Liquids*— Other liquids might be either aqueous- or nonaqueous-based. Some examples would be specified inks, adhesive, oils, and coating materials.

6. Sampling

6.1 For acceptance sampling, obtain the sample in accordance with Practice D 585.

6.2 When sampling for other purposes, use Practice E 122 as an alternative.

7. Test Specimens

7.1 The test specimens shall 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. Tests shall be made on both sides of each specimen. The actual test areas tested shall not contain water marks or visible blemishes or defects and shall not be touched with the fingers or contaminated in any other way.

7.2 The test specimens shall be conditioned in accordance with Practice D 685.

8. Procedure

8.1 Test the specimens under standard atmospheric conditions in accordance with Practice D 685.

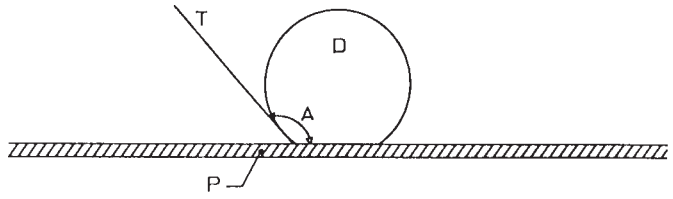
8.2 Place the test specimen in the instrument, holding it in close contact with the stage by means of small weights, clips, or whatever fixtures are associated with the instrument.

8.3 Set the tip of the hypodermic needle 3.2 mm (⅛ in.) from the surface of the specimen and deposit a drop of test liquid 1/150 to 1/200 mL in size on the specimen.

8.4 For initial wettability, used as a measure of ruling quality, make the measurement of the angle of contact after the drop has been in contact with the paper for 5 s. For rate of change of wettability, used as a measure of writing quality, make two measurements, one after 5 s and the other after 60 s have elapsed.

8.5 *Angle of Contact Measurement* (see Fig. 2):

8.5.1 *Projected Image Procedure*—Project the image of the drop enlarged on a glass screen, at the back of which shall be clamped a sheet of transparent onionskin paper. Draw a horizontal line on the transparent paper coinciding with the



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

FIG. 2 Measuring Angle of Contact

image of the base of the drop and as soon as the specified time of contact of the drop with the specimen has elapsed, quickly draw two tangents to the curve at the two points of contact with the base line. Measure the two interior angles between the base line and the tangents with a protractor.

8.5.2 *Reflective Goniometer Procedure*—Adjust the eye piece and the internal measuring mechanism so that the interior angle of each of the two points of contact can be determined.

8.6 Make measurements for 5 drops on each side of the specimen. The contact angle for each drop shall be the average of the angles at the two edges of the drop. If the two contact angles are significantly different, the value should be eliminated and the test repeated.

9. Calculation

9.1 *Calculation*— Calculate the rate of change in wettability as follows:

$$R = (A - a)/55$$

where:

R = rate of change in wettability,°/ s,
A = average angle of contact after 5 s, and
a = average angle of contact after 60 s.

10. Interpretation of Results

10.1 In considering the results of the angle-of-contact test, the following factors that all affect the results in different degrees shall be considered: (*a*) the wetting power of the test liquid used, (*b*) the wettability of the sizing agent used in sizing the paper, and (*c*) the surface texture of finish of the paper.

10.2 The standard ink will give smaller angles of contact than water, indicating that it wets the paper more readily than water. Papers surface-sized with starch will generally show smaller angles than papers of about the same finish tube-sized with glue. A machine-finished paper with a grainy surface would have a greater angle of contact than a plated or calendered paper of equal sizing. It is known that in practice, ruling results will depend on both the surface wettability and finish of the paper. Thus, the angle of contact should give a very good idea of what is to be expected. It has been found that excellent ruling will prevail when the average angle of contact with water lies between 90 and 100°; when the angle of contact is greater than 110°, breaks are likely to occur in the ruled lines; when the angle is smaller than 90°, the ruling fluid is likely to feather.

10.3 In determining the writing qualities of paper by means of the angle-of-contact method, it is necessary to take into account that medium-sized papers will at times show feathering only after the ink has partly penetrated the paper. The tendency of a writing paper to feather will be indicated by the decrease in the angle of contact between measurement after 5 and 60 s. In hard-sized papers, the angle of contact will not change perceptibly between the measurements at 5 and 60 s. If the initial wettability is less than 90°, it is quite likely the paper will feather as soon as it is written upon.

10.4 As the range in contact angle for specific conditions is small, a refined technique is required in performing the test. This is indicated by the above examples as well as by theoretical considerations.

11. Report

11.1 Report the following information:

11.1.1 *Standard Ink*:

11.1.1.1 The initial wettability or ruling quality shall be reported as the average angle of contact after 5-s exposure expressed to the nearest degree,

11.1.1.2 The rate of change of wettability or writing quality is reported as calculated in 9.1,

11.1.1.3 If possible to identify the two sides, the results shall be identified as to the wire side and the felt side. Otherwise, the size may be arbitrarily designated, as for example, *A* and *B*, and

11.1.1.4 Test results shall be expressed as maximum, minimum, and average for each side of the specimen tested, reported to two significant figures.

11.1.2 *Other Liquids*— If liquids other than the standard ink are used, the liquid used along with any modifications of the procedures shall be reported.

12. Precision and Bias

12.1 *Precision*:

12.1.1 When the procedure in this test method is applied by a single operator using the same equipment to paper whose surface characteristics impacting this test method are homogeneous, and where ten tests are run on the same side of two test specimens cut from the same sheet of paper giving a result of 90° or greater using this test method, the repeatability standard deviation is approximately 7° and the 95 % repeatability is 2° (see TAPPI T458).

12.1.2 Using the data reported by Bristow,[4] the reproducibility standard deviation is approximately 2.5° and the 95 % reproducibility limit is approximately 7° for two different operators using the same apparatus.

12.1.3 It is not practicable to specify in greater detail the precision of the procedure for determining contact angle in this test method because of variations in equipment, variations in test liquid, and most specifically in paper coating uniformity which are encountered. It must be left to individual laboratories to determine precision estimates for specific paper grades and testing needs.

12.2 *Bias*—No statement can be made about the bias of the procedure in this test method because contact angle is defined in terms of the test method.

[4] Bristow, J.A., "The Reproducibility of Contact Angle Measurement," *Paperi ja Puu* 50 (4a): 171, 1968.

13. Keywords

13.1 angle-of-contact; contact angle; paper; surface wettability