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T _____ 458 _____

BALLOT NO. _____ 02 - SARG _____

DRAFT NO. _____ 01 _____

DATE _____ May 16, 2024 _____

WORKING GROUP
CHAIR _____ N/A _____

SUBJECT
CATEGORY _____ Physical Properties _____

RELATED
METHODS _____ See "Additional Information" _____

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Surface Wettability of Paper (Angle of Contact Method) *(Ten-year review of Classical Method T 458 cm-14)*

1. Scope

1.1 In this method (1-3), the contact angle between air and liquid on a paper surface is taken as a measure of the resistance of the paper surface to wetting by the liquid.

1.2 The initial angle of contact or initial wettability is considered to be a measure of the *ruling* quality of the paper. The rate of change in the wettability is considered to be a measure of the *writing* quality.

1.3 There are other important purposes for which surface wettability of a paper by contact angle measurement may be of interest, for example, with regard to adhesives. However, considerable modification of the procedure might be required. Hence, this procedure is restricted to ruling and writing purposes.

2. Significance

2.1 The ruling and writing qualities of paper are of importance for papers intended for drafting and writing. These papers must accept ink (ruling quality) without feathering (writing quality). Certain ranges of contact angle are reportedly indicative of these qualities (see Additional Information 12.3.2, 12.3.3).

2.2 The property being measured is an *apparent* contact angle, since paper is not smooth but is porous and rough on a microscopic scale. For this reason, some doubt has been expressed concerning the significance of the contact angle test (4); however, the method is widely used on glassines, printing papers, release papers, film laminates, and other papers, and there are both Canadian and SCAN test methods for contact angle. Contact angle is considered meaningful and measurable “as long as the absorption rate is sufficiently low as to be negligible during the first 10 s after application of the liquid drop on the surface” (5).

3. Summary of method

3.1 A small drop of liquid (water or ink) is placed on the paper surface. The contact angle of the drop is magnified and measured, both initially when the drop is placed on the surface and 60 s later.

3.2 Any methods for magnifying and measuring the contact angle may be used, such as projection, photography, and microscopic goniometry, provided that the contact angle may be determined to the nearest degree.

4. Apparatus¹

4.1 *Horizontal surface*, such as a microscope slide mounted on a test table with leveling screws, for holding the test specimen by means of small weights or two-sided tape in order to prevent distortion of the specimen when it is wetted.

4.2 *Projector, camera, or microscope* for obtaining a magnified image of the liquid drop. See Appendix.

4.3 *Device for measuring contact angle*. See Appendix.

4.3.1 Protractor or goniometer for direct measurement of the contact angle.

4.3.2 Scale for measuring dimensions of drop, from which the contact angle may be calculated.

4.4 *Microburette or syringe* such as a 1-mL hypodermic equipped with a No. 27 stainless steel needle giving 150 to 200 drops per milliliter.

NOTE 1: A smaller drop is needed for the method described in Appendix A.4.

4.5 *Light source* furnished with a filter to protect the test specimen from heat.

4.6 *Timer*.

5. Reagents

5.1 *Distilled water* at 23°C, and having a surface tension of at least 72 mN/m (72 dyn/cm).

5.2 *Ink*, as agreed upon (e.g., the ink with which the paper is to be used).

5.3 Alternate liquids may be used as agreed by manufacturer and user.

6. Sampling and test specimens

6.1 Sample the paper in accordance with TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product.” Be careful not to touch the areas to be tested, or contaminate them in any other way. Identify and mark the wire and felt sides or, if not possible, mark “A” on the same side of all sheets.

¹Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set of TAPPI Test Methods, or may be available from the TAPPI Quality and Standards Department.

6.2 From each test unit of the sample, cut, so as to be representative of the test unit, ten $100 \pm 1 \times 15 \pm 0.1$ mm clean specimen strips, free of folds, wrinkles, blemishes, water marks and other defects not normally inherent in the paper. Repeat the markings to identify each specimen strip.

NOTE 2: If there is a marked fiber orientation, cut and test strips in both directions.

7. Procedure

7.1 Condition and test the paper in the conditioning/testing atmosphere specified in TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products."

7.2 Place a test specimen strip on a slide on the test table. Hold it in close contact with the surface of the slide by means of small weights or clamps placed close enough on each side of the syringe located above the slide to prevent distortion of the paper when it is wetted by the drop. Alternatively, use double-faced pressure sensitive tape to hold the test strip to the slide. Level the table.

7.3 Set the tip of the hypodermic needle 2.5 mm from the surface of the strip and deposit a drop on the paper. As soon as the specified time of contact of the drop with the paper has elapsed, quickly determine both angles of contact (left and right sides of the image of the drop) by one of the procedures specified in the Appendix or by an equivalent procedure. The interior angles (C) between the base line of the drop and the two tangents to the curve at the two points of contact with the base line (Fig. 1) are designated angles of contact.

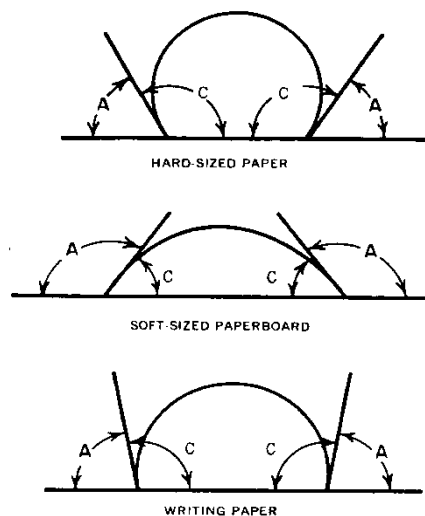


Fig. 1. Measuring angle of contact. **NOTE:** Angle A is the exterior angle and angle C is the interior angle.

7.4 For *initial wettability*, used as a measure of *ruling* quality, measure the angles after the drop has been in contact with the paper for 5 s. For *rate of change in wettability*, used as a measure of *writing* quality, make an additional set of measurements after 60 s.

7.5 During the test, turn off the lamp when not needed, to avoid heating the specimen any more than is necessary.

7.6 Test five specimen strips on one side and five on the other. Carry out the tests on unwetted areas.

8. Calculation

8.1 For each side, calculate the average rate of change of wettability, in degrees per second, as follows:

$$R = \frac{C - C'}{55}$$

where

R = the rate at which the angle of wetting changes

C = the average angle of contact after 5 s

C' = the average angle of contact after 60 s

9. Report

- 9.1 State the liquid used.
- 9.2 State the methods used for magnifying the drop and measuring the contact angles (see Appendix).
- 9.3 For each test unit and each side of the paper report:
 - 9.3.1 Average drop volume if different than specified in 4.4.
 - 9.3.2 The average initial angle of contact in degrees.
 - 9.3.3 The average rate of wettability in degrees per second.

10. Precision

10.1 The values of repeatability and reproducibility provided below have been calculated for test units each of which is an average of 5 replicate test determinations on the same side of the sheet.

10.1.1 *Repeatability* (within a laboratory) = 3% or 4% for angles of 90° or higher.

10.1.2 *Reproducibility* (between operators using the same apparatus) = 7%.

10.2 The above terms are used as defined in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility." The source of the first repeatability information is not known, but essentially the same information was reported in the 1970 and earlier editions of the projection method (Appendix A.1). The second value of repeatability and the value for reproducibility are derived from Bristow (5) who also used a projection method but modified in accordance with Appendix A.4.4. Because of unreliable timing in drawing of the tangents in the projection method A.1.2, the reproducibility may exceed 7%.

11. Keywords

Paper, Paperboard, Sized papers, Writing papers, Contact angle, Wettability, Projection, Printability, Feathering

12. Additional Information

12.1 Effective date of issue: **To be assigned.**

12.2 The 1989 revision provided for a choice of apparatus for magnifying and measuring the contact angle. The revision, completed in 1994, reclassified this method as a Classical Method.

12.3 *Interpretation of test result.*

12.3.1 The following factors affect the results of the angle-of-contact test: (a) the wetting power of the solution used; (b) the wettability of the sizing agent used in sizing the paper; and (c) the surface texture or finish of the paper. For instance, ink will show smaller angles of contact than will water, because ink wets the paper more readily than water. Papers surface-sized with starch will generally show smaller angles than papers of about the same finish surface-sized with glue. A machine-finished paper with a grainy surface will show a greater angle of contact than a plated or calendered paper sized to the same degree.

12.3.2 In practice, ruling results will depend on both the surface wettability and the finish of the paper; the angle of contact gives a good idea of what may be expected. It has been found (2) that excellent ruling will result when the average angle of contact with water lies between 110 and 90°. 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°, it is quite likely that the liquid will feather.

12.3.3 In judging the writing quality of paper, the tendency of a writing paper to feather will be indicated by the decrease in the angle of contact between the 5-s and the 60-s measurement. In hardsized papers, the angle of

contact will not change perceptibly between the 5-s and the 60-s reading. If the initial wettability is less than 90°, it is quite likely that the paper will feather as soon as it is written upon. Medium-sized papers will occasionally show feathering when the ink has only partly penetrated the paper surface.

12.3.4 The range of contact angles for specific conditions is small; consequently, careful testing technique is required as is indicated by the above examples.

12.4 Related methods: Canadian PAPTAC F-3; Scandinavian SCAN-P18. TAPPI T 558 "Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester."

References

1. Lafontaine, G. H., "Surface Wettability of Paper and the Angle of Contact Test," *Paper Trade J.* **113**(6): 29 (1941).
2. Codwise, J. A., "The Determination of the Resistance to Wetting of Paper and Paperboard," *Tech. Assoc. Papers* **22**: 246 (1939).
3. Back, E., and Lundin, B., *Svensk Papperstidning* **58**: 20, 758-763 (1955).
4. Mason, S. G., "How Liquids Wet Paper," *Trend* **23**: 6 (Winter 1974-75).
5. Bristow, J. A., "The Reproducibility of Contact Angle Measurement," *Paperi ja Puu* **50** (4a): 171 (1968).

Appendixes

A.1 Projection method.

A.1.1 Apparatus.

A.1.1.1 *Frosted glass screen*, with clamps to hold a sheet of transparent paper (Fig. 2 or Fig. 3).

A.1.1.2 *Ventilated lamp housing* (1 in Fig. 2), containing a 250-W projection lamp.

A.1.1.3 *Tube* (2 in Fig. 2) containing a lens to concentrate the beam of light.

A.1.1.4 *Microscope draw tube* (3 in Fig. 2), fitted with a 25-mm objective and a 5X ocular, mounted horizontally beside the stage.

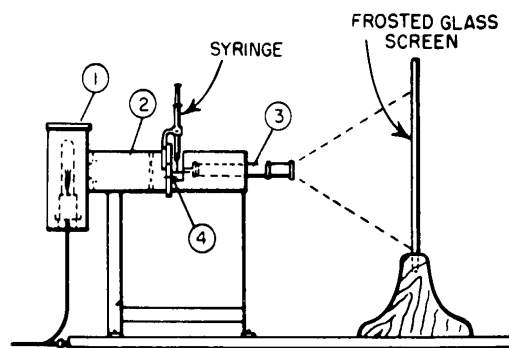


Fig. 2. Projector.

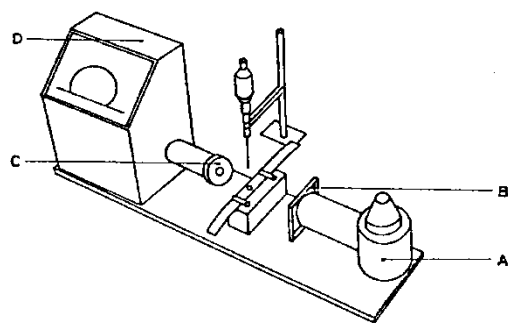


Fig. 3. The apparatus. A: light source; B: filter; C: microscope; D: projection device.

A.1.1.5 *Horizontal stage (or test table)* (4 in Fig. 2), to hold the specimen under test, the stage being horizontally adjustable.

A.1.1.6 *Cell* containing copper sulfate solution inserted between the lens which concentrates the light rays and the stage on which the specimen is placed in order to reduce the heat on the specimen and drop. The concentration of the copper sulfate solution is adjusted by the user for the desired background color.

NOTE A.1. Alternatively a small projection microscope, with a magnification of about 30X, may be used.

A.1.1.7 *Protractor.*

A.1.2 *Procedure.* Project the image of the drop, enlarged 25 to 30 times, on the glass screen at the back of which is clamped a sheet of transparent manifold paper. Draw a horizontal line on the transparent paper tangent to the image of the base of the drop. As soon as the specified time of contact of the drop with the paper has elapsed, quickly draw two tangents to the curve at the two points of contact with the base line (see Fig. 1). Measure the exterior angles A with a protractor. The average of the interior angles [where $C = (180^\circ - A)$] between the base line and each tangent, is designated as the angle of contact.

A.2 *Camera method.*

A.2.1 *Apparatus.*

A.2.1.1 *Polaroid or other "instant" camera*, mounted on a microscope.

A.2.1.2 *Protractor.*

A.2.2 *Procedure:* From enlarged photographs of the drop taken at the specified times, use the protractor to determine the contact angles as in the projection method.

A.2.3 *Alternative:* A TV camera and video cassette may be used instead of a camera. If the TV camera is triggered from the pipette, contact angles can be obtained: (a) very soon after application of the drop, and (b) at any time after application, using the TV monitor in a "stop-motion mode."

A.3 *Microscopic goniometry method.*

A.3.1 *Apparatus:* Microscope with protractor mounted on the eyepiece ("contact angle goniometer").

A.3.2 *Procedure:* Use the eyepiece protractor to determine the contact angles of the image as in the projection method.

A.4 *Microscopic micrometer method.*

A.4.1 *Scope:* This method is suitable for sized paper and paperboard having a contact angle exceeding 90° .

A.4.2 *Apparatus.*

A.4.2.1 *Microscope* with eyepiece micrometer or microcomparator.

A.4.2.2 *Microburette* that can deliver a drop of 4.0 ± 0.4 microliters of liquid, with tip of circular cross section cut perpendicular to the capillary.

A.4.3 *Procedure.*

A.4.3.1 Produce a droplet small enough for the curvature above the surface of contact to be considered spherical.

A.4.3.2 Using the micrometer, determine the height h of the droplet and the diameter a of the surface of contact of the droplet with the paper (Fig. 4).

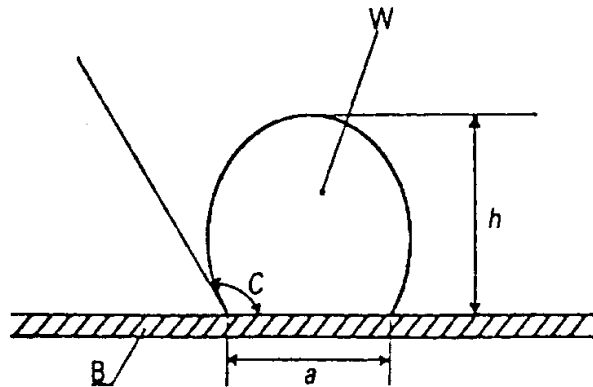


Fig. 4. Principle of measurement. W: water droplet; B: paper; a: diameter of the surface of contact of the droplet; h; height of the droplet; C: contact angle.

A.4.3.3 Then use the formula $\tan (C/2) = 2h/a$ or the nomograph (Fig. 5).

A.4.4 *Alternative:* The apparatus of A.1 or A.2 may be used with the small drops and measurements of h and a specified in this procedure.

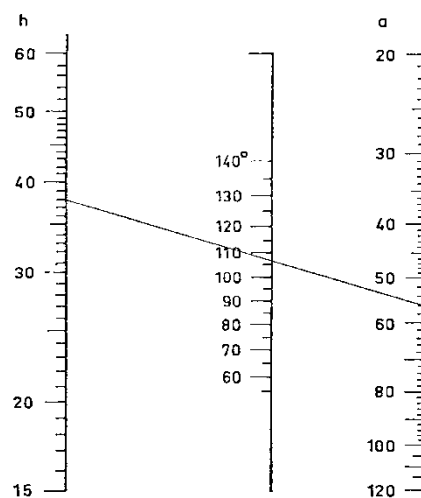


Fig. 5. Nomogram for reading off the contact angle. The transverse line illustrates the use of the nomogram.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■