Designation: D 779 – 03

An American National Standard

Standard Test Method for Water Resistance of Paper, Paperboard, and Other Sheet Materials by the Dry Indicator Method[1]

This standard is issued under the fixed designation D 779; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the time required for water to pass through a specimen of paper. It is based on the dry-indicator method of Carson **(1-4)**[2] and modifications of it developed by Abrams **(5)** and Codwise **(6-8)**.

1.2 It is designed for use with materials that require a relatively short time to test (up to approximately 30 s), and has been found useful in testing slack-sized papers.

NOTE 1—For testing hard-sized paper and paperboard, see TAPPI Test Method T 441.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product[3]

D 685 Practice for Conditioning Paper and Paper Products for Testing[3]

E 122 Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process[4]

2.2 *TAPPI Standards:*

T 441 Water absorptiveness of sized (non-bibulous) paper and paperboard (Cobb test)[5]

T 433 Water resistance of sized paper and paperboard (dry indicator method)[5]

3. Summary of Test Method

3.1 This test method consists of bringing one side of the test specimen in contact with water and finding the time required for the water to pass through the specimen, as indicated by the development of color in an indicator powder consisting of a mixture of water-soluble dye, sugar, and starch. The sugar serves the two-fold purpose of masking the color of the particles of dye as long as they are dry, and of absorbing the transuded moisture and holding it in close proximity to the dye. The starch increases the stability of the mixture.

3.2 Five tests are made on each side of the paper and reported as two separate averages.

4. Significance and Use

4.1 The dry indicator used in this test method is so strongly hygroscopic it will change color in a moderate- to high-humidity atmosphere without contacting liquid water. It will also change in contact with liquid water. This test method, therefore, measures the combined effect of vapor and liquid transmission **(9)**. For test times up to approximately 30 s, liquid transudation rate is dominant and this test method can be considered to measure this property. As test times exceed 30 s, the influence of vapor-transmission rate increases and this test method cannot be regarded as a valid measure of liquid resistance (sizing).

4.2 This test method is of value in cases where paper or a paper container comes into contact with water on one face.

4.3 This test method is sometimes *not* applicable to materials containing large amounts of water-soluble components.

5. Apparatus

5.1 Any form of apparatus for applying the test may be used that fulfills the following conditions:

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[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] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] *Annual Book of ASTM Standards,* Vol 15.09.

[4] *Annual Book of ASTM Standards,* Vol 14.02.

[5] Published in related material section of *Annual Book of ASTM Standards,* Vol 15.09. Also available from the Technical Association of the Pulp and Paper Industry, Technology Park/Atlanta, P.O. Box 105113, Atlanta, GA 30348.

5.1.1 One surface of the specimen is wetted uniformly at the moment the count of time is begun,

5.1.2 The indicator on the opposite surface is continuously visible,

5.1.3 No moisture reaches the indicator except that which passes through the specimen from the wetted surface, and

5.1.4 No moisture that does so reach the indicator escapes from contact with it.

5.2 *Float Arrangement* **(7)**—Fig. 1 shows one apparatus that fulfills the requirements of 5.1. It is made of a thin-walled aluminum pan approximately 5 in. (127 mm) in diameter and 1 in. (25.4 mm) high, with a hole 2 in. (50.8 mm) in diameter cut in the bottom. The surface is coated with paraffin or beeswax to make it more water repellent. The watchglass is clamped over the specimen by means of a wire-frame clamp hinged at one side and fastened under a spring at the opposite side. In the absence of the float the paper can be supported by a hollow cylinder having the upper end barely submerged under the surface of the water in a suitable vessel, while the watchglass is placed over the indicator. Such a cylinder may also be found useful in supporting an occasional paper whose surface is so easily wetted that it would be difficult to float it unsupported when prepared as shown in Fig. 1.

5.3 *Shaker and Desiccator Assembly* (Fig. 2) *for the Indicator*—The shaker is prepared from a 10-mL screw-top vial by cutting away most of the metal of the flat portion of the top, fitting a 70-mesh (27.5-mesh/mm) wire screen inside the top, and screwing it back in place. The desiccator is made of a small wide-mouth bottle containing desiccant covered with a layer of glass wool. A hole is bored in the cork stopper just large enough to admit the shaker. The bottle remains on its side and the vial is inserted through the hole with the screened end inside. The assembly is kept in the usual type of laboratory desiccator when not in use.

5.4 *Watchglass*, 50-mm (2-in.).

5.5 *Stopwatch or Electric Timer*, reading to 0.2 s.

6. Reagents

6.1 *Desiccant*—Anhydrous calcium chloride, activated alumina, or silica gel.

6.2 *Indicator*—The water-transudation indicator is composed of pure, powdered cane sugar (do not use confectioner's sugar, which contains starch), pure soluble starch, and methyl violet dye (Color Index 680). Pass each ingredient separately through a No. 100 screen (39.4 mesh/mm), and completely dry it in a desiccator over a desiccant (see 6.1) before making the mixture. When dry, weigh and mix the following proportions by weight:

Sugar	45
Soluble starch	5
Dye	1

FIG. 1 Cross Section of Float

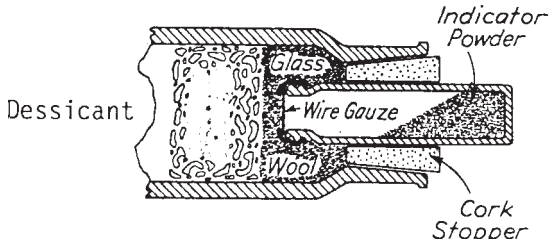

FIG. 2 Longitudinal Section Showing Construction of Desiccator and Shaker

Mix the ingredients by screening repeatedly through a No. 60 screen (23.6 mesh/mm) until the mixture is uniform. Keep the indicator in a desiccator when it is not being used.

6.3 *Water*, distilled or demineralized.

7. Sampling, Test Units, and Test Specimens

7.1 For acceptance purposes, sample the lot of material in accordance with Practice D 585.

7.2 When sampling for other purposes, Practice E 122 may be used for an alternative.

7.3 From each test unit obtained in accordance with 7.1 or 7.2, cut ten test specimens, free from folds, wrinkles, or other blemishes not commonly inherent in the material. A convenient size is approximately 2.5 by 2.5 in. (63 by 63 mm).

7.4 For ordinary papers, designate the sides as wire side and felt side when they can be differentiated; otherwise, use a suitable code designation such as Side I and Side II, or when there is an obvious difference between the sides, such as Side I, coated, and Side II, uncoated. The side designated is that which is to be in contact with the water.

7.5 Condition and test the specimens in a standard atmosphere in accordance with Practice D 685. The water on which the specimens are floated should be 23 ± 0.5°C.

8. Procedure

8.1 Place a test specimen on a level, smooth surface, and sprinkle the indicator on the specimen by gently tapping the inverted shaker until a thin, even layer is formed, avoiding either a very sparse covering or one in which the powder is piled up. In the former case, the color change produced by too few dye particles may be insufficient to attract attention, unless they are exposed for a longer time than the proper end point. When the powder is placed on too thickly, the upper layer may obscure changes in the indicator in contact with the paper. Also, the greater total quantity of powder may require more time to change, since it would necessitate more moisture to affect all the dye. Handle the test specimen with care once the powder is applied, for jarring or excessive tipping may cause rearrangement of the particles of powder, which then tend to form in clusters, thus destroying the intimate contact and uniform thin layer that is desirable. As soon as the application of the layer of the indicator powder is completed, cover it with the watchglass and keep it covered during the test.

8.2 Instead of using the float arrangement described in 5.2, it is also permissible to form a paper boat, by folding up the edges of the specimen, and float it on the surface of the water. Apply indicator to the top surface in accordance with 8.1 just before floating the specimen on the water.

8.3 Place the specimen on the water, making contact with the water at a slight angle to avoid trapping air bubbles that might cause uneven wetting of the specimen. When testing materials having uneven surfaces, wet the bottom surface, using a soft brush, immediately before placing the specimen on the water. Examine each specimen after the test, and if there is evidence of uneven wetting, discard the test result.

8.4 Measure the time interval from the instant of contact of the test specimen with water until the rate of change in the color of the indicator is at a maximum. This time interval is conveniently determined as the mean of the values corresponding to the development of pronounced color in one fourth and in three fourths of the area covered by the indicator. For materials on which the color develops uniformly over the whole area, it may be necessary to tabulate the values of the time interval against the intensity of the color to determine the maximum rate of change. Artificial light, when used for viewing the test specimen, should be reasonably brilliant but completely shaded from the eyes. The light should be applied to one side of the specimen (thus avoiding reflection from the cover glass) and the eye should view from the side next to the illuminant, to avoid interference of shadows cast by the indicator particles with the judgment of the color. Record the test time to the nearest 0.2 s.

8.5 Run five tests on each side of the paper.

9. Report

9.1 Report the average value of the time of transudation from each side of the paper, to the nearest 1 s. See Table 1.

TABLE 1 Repeatability Limits

Sample	Result Range, s	Standard Deviation, s	95 % Repeatability Limits, s
A	5–10	1.1	3.1
B	10–20	0.8	2.3
C	20–40	1.9	5.4
K	5–10	0.45	1.2
L	10–20	0.85	2.4
M	20–40	1.05	2.9

10. Precision and Bias

10.1 *Precision*:

10.1.1 *Repeatability*:

10.1.1.1 Repeatability within a single laboratory appears to vary as a function of the water resistance level of the material. Other aspects of material composition and uniformity can also impact test repeatability.

10.1.1.2 Tests in one laboratory on three different grades of paper resulted in the following estimates of repeatability **(10)**:

10.1.1.3 Tests in a second laboratory on a total of 194 samples gave the following estimates of repeatability **(13)**:

10.1.2 *Reproducibility*—Reproducibility estimates on this procedure are still in progress.

10.2 *Bias*—No information can be presented on the bias of the procedure in this test method, because the water resistance as determined by the dry indicator method is defined only in terms of the test method.

11. Keywords

11.1 dry indicator method; paper; paperboard; water resistance

APPENDIX

(Nonmandatory Information)

X1. ACCELERATED TESTING

X1.1 Investigations of the use of high temperatures (up to the boiling point of water) in the dry-indicator test have shown that, in general, there is good correlation between transudation time and temperature. For products that show such relation, the use of hot water is of value in shortening the test period, and in some instances in making the end point more distinct **(11, 12)**.

REFERENCES

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(**2**) Carson, F. T., U.S. Bureau of Standards, *Technologic*, Paper No. 326, September 1926.

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(**7**) Codwise, P. W., “Water Resistance of Papers,’’ *Technical Association Papers*, TAPAA, Vol 15, No. 234, 1932; *Paper Trade Journal*, Vol 94, No. 5, pp. 42 to 45; TS 46–49, Feb 4, 1932.

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(**9**) Van den Akker, J. A., et al, "The Fluorescence Size Test, An Improved Method and a New Instrument for Measuring the Water Resistance of Paper,'' *Paper Trade Journal*, Vol 109, No. 21, Nov. 23, 1939, pp. 33 to 42.

(**10**) Hruska, R. J. Letter of April 8, 1986 to Aubey, R. A. See ASTM files.

(**11**) Codwise, P. W., "Resistance of Sized Paper & Paperboard to Water at Elevated Temperatures,'' *Technical Association Papers*, TAPAA, Vol 26, No. 165, 1943; *Paper Trade Journal*, Vol 116, No. 9, pp. 30 to 33; TS 90–93, March 4, 1943.

(**12**) Mullen, E. G., "Use of the Dry Indicator Method for Testing the Water Resistance of Asphalted Papers,'' *Paper Trade Journal*, Vol 119, No. 2, pp. 41 to 42; TS 11–12, July 13, 1944.