



Standard Test Method for Sorption of Bibulous Paper Products (Sorptive Rate and Capacity Using Gravimetric Principles)¹

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INTRODUCTION

The interaction of water in either the gaseous or liquid state is an extremely important property of paper and paper products. In addition to being the basis for performance characteristics of various different types of paper, such as paper for printing and writing, paper for wrapping and packaging, and paper for liquid absorbing tasks, measured values for many of the physical properties of paper vary, depending upon the amount of water present in the sheet when it is tested.

The amount of moisture present in paper and paper products may be measured by procedures such as Test Method D 644.

To enable physical measurements on different paper materials to be easily compared, equilibration of specimens to a standard moisture content before testing is done using procedures such as Practice D 685.

Where paper materials are to be used as barriers to the transfer of water vapor in, for example, packaging or wrapping applications, their gaseous moisture barrier properties may be measured by TAPPI T 523. A method for measuring the barrier properties of similar paper materials to liquid water is found in Test Method D 3285.

Where the interaction between a paper material and a liquid is important in an application such as printing, measurements may be made using procedures such as Test Methods D 5455, and Test Methods D 724.

Where paper materials are required which will absorb and retain water or water based fluids, as for example, absorbent tissue or blotting papers, test methods such as Test Methods D 824, D 2177, or D 4250, may be useful. The first two of these methods measure absorbent rate, the third, absorptive capacity. Similar test methods for absorptive rate are found in TAPPI T 431 and T 432. The absorbency of paperboard is measured using TAPPI T 491, which operates on the same gravimetric principles found in Test Method D 4250. Similar standardized testing procedures for absorbent structures are found in, for example, INDA Test Method IST 10, which gives instructions for the determination of absorbency time, capacity, and wicking rate of absorbent structures such as napkins, towels, and diapers.

These procedures are all similar in that they measure either the time (seconds) required for absorption of a specified volume of liquid under specified conditions by a test specimen, or the saturation capacity (grams) of a specimen under specified conditions, or both, in the case of the INDA Test Method IST 10.1.

While such information is of use in setting minimum material performance specifications, procedures which measure both rate and capacity simultaneously may also be used for such purposes, while providing significantly improved understanding of a material's absorbent character by true rate data (volume or weight/time) at points of interest prior to saturation. Numerous workers have published proposals for such testing procedures for paper or paper products, but few have been subjected to the consensus standardization process of ASTM or a similar organization. An excellent summary of such work is contained in the *Handbook of Physical and Mechanical Testing of Paper and Paperboard*.²

This test method is based upon the work of Miller and Tyomkin³ and Painter⁴ and provides a procedure whereby absorptive capacity and rate may be measured simultaneously and dynamically to provide an absorption curve showing the time dependency of the absorption process. This test method

also provides test values comparable to those of existing manual procedures showing the time required for absorption of a specified amount of liquid or the liquid capacity at saturation.

1. Scope

1.1 This test method determines the liquid sorption of bibulous paper, paperboard, and paper products using gravimetric principles.

1.1.1 The procedure in this test method describes testing of absorbent materials such as those in 1.1. Similar testing may be done on composite absorbent products, incorporating cellulose and nonwoven fabrics, polymer films, or other similar materials for use such as absorbent pads, wipes, and so on with slight modification. This test method does not describe the application of this technique to textiles.

1.2 The standard test liquid is distilled water.

1.2.1 Other test liquids of interest may be used if desired, provided they are compatible with the liquid delivery system specified.

1.2.1.1 Examples of such test liquids include, but are not limited to, buffered solutions of distilled water, fruit or vegetable juices, milk, synthetic body liquids such as urine, and organic fluids such as salad oil, cooking oil, motor oil, and others.

1.3 The test liquid is delivered at an effective negative head pressure after initiating liquid flow (wicking). This allows the controlling sorptive properties to be demand wettability, the ability of the specimen to absorb through capillarity and surface wetting.

1.3.1 Using the equipment and principles described, absorption at other instrument configurations and head pressures, as well as desorption may be measured.

1.4 This test method measures both the rate and capacity of liquid sorption through demand wettability or wicking. Liquid capacity is defined as the point in the analysis where the sorption rate falls below 3.0 mg in 5 s. Agreed intermediate points are measured during the analysis to determine rate.

1.5 An analog or digital report of the sorptive behavior of the test specimen may be produced, if desired, to further characterize its sorptive behavior.

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:

D 585 Practice for Sampling and Accepting a Single Lot of

Paper, Paperboard, Fiberboard, or Related Product⁵
 D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying⁵
 D 685 Practice for Conditioning Paper and Paper Products for Testing⁵
 D 724 Test Method for Surface Wettability of Paper (Angle-of-Contact Method)⁵
 D 824 Test Method for Rate of Absorption of Water by Bibulous Papers⁵
 D 828 Test Method for Tensile Properties of Paper and Paperboard Using Constant-Rate-of-Elongation Apparatus⁵
 D 1193 Specification for Reagent Water⁶
 D 1968 Terminology Relating to Paper and Paper Products⁵
 D 2177 Test Method for Ink Absorption of Blotting Paper⁵
 D 3285 Test Method for Water Absorptiveness of Nonbibulous Paper and Paperboard (Cobb Test)⁵
 D 4250 Test Method for Water-Holding Capacity of Bibulous Fibrous Products
 D 5455 Test Method for Short Term Liquid Sorption into Paper (Bristow Test)⁵
 E 122 Practice Relating to Choice of a Sample Size to Estimate a Measure of Quality for a Lot or Process⁷
 2.2 TAPPI Test Methods:
 T 431 Ink Absorbency of Blotting Paper⁸
 T 432 Water Absorbency of Bibulous Papers⁸
 T 491 Water Immersion Test of Paperboard⁸
 T 523 Dynamic Measurement of Water Vapor Transfer Through Sheet Materials⁸
 2.3 INDA Test Method:
 IST 10 Method for Absorption of Water⁹

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1968 or the *Dictionary of Paper*.¹⁰

4. Summary of Test Method

4.1 A specimen of the sample is placed on a horizontal test plate, such that its bottom surface rests on the plate and its upper surface is covered by a test weight. The test plate, in turn, is connected to a liquid reservoir by means of a siphon tube. The spatial relationship between the bottom surface of the test specimen in contact with the effluent of the siphon tube and the top surface of the liquid in the liquid reservoir is variable, and may be continuously adjusted to maintain a constant spatial relationship during the test. The liquid reservoir is placed on a suitable electronic load cell or weighing device, such as an electronic balance.

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 Hollmark, "Absorbency of Tissue and Toweling", *Handbook of Physical and Mechanical Testing of Paper and Paperboard*, Vol 2, Chapter 20, Richard E. Mark, ed., Marcel Dekker, New York, NY.

³ Miller, B., and Tyomkin, I., *Textile Research Journal*, Vol 54, pp. 706–712, November 1984.

⁴ Painter, V., *TAPPI 68(12)*, pp. 54–59, 1985. See also *U.S. Patent* 4,357,827.

⁵ *Annual Book of ASTM Standards*, Vol 15.09.

⁶ *Annual Book of ASTM Standards*, Vol 11.01.

⁷ *Annual Book of ASTM Standards*, Vol 14.02.

⁸ Available from the Technical Association of the Pulp and Paper Industry (TAPPI), Technology Park/Atlanta, P.O. Box 105113, Atlanta, GA 30348.

⁹ Available from INDA.

¹⁰ Available from TAPPI.

4.2 During a test, liquid is absorbed into the specimen. This absorption causes a reduction in the liquid present in the reservoir, which is measured by the weighing device. The decrease in weight in the liquid reservoir is a measure of the liquid absorbed by the test specimen, and may be plotted by a recorder, printed as a table of weights and corresponding times, or transferred to a data storage device for processing at a later time.

4.3 The test conditions described in this test method are a negative liquid head pressure. The surface of the liquid in the liquid reservoir is below the bottom surface of the test specimen in contact with the test plate, a circular fluid orifice of defined size in the center of the test plate, the upper surface of the specimen in contact with a flat weight to ensure proper contact of the bottom surface of the specimen with the test plate, and a test liquid of reagent water.

4.4 Nonstandard variations of this procedure not described here include different test plate designs or orifice sizes, or replacement of the orifice with a porous plate, liquid head pressures of zero or greater than zero, no pressure plate or plates of various sizes and weights placed on the upper surface of the specimen, test liquids other than reagent water, and others.

5. Significance and Use

5.1 The absorptive characteristics, both rate and capacity, are of great importance in creped tissue papers made for use as absorbent sanitary tissue products, such as toweling and tissue.

5.2 The absorptive characteristics, both rate and capacity, are of great importance in creped tissue or similar papers produced for incorporation into composite absorbent structures, such as disposable bed pads, and composite disposable absorbent structures containing an absorbent paper component such as hospital disposable bed pads and related products.

5.3 This test method provides a standardized approach to the dynamic evaluation of both absorptive rate and absorptive capacity of materials such as those in 5.1 and 5.2.

6. Apparatus

6.1 *Load Measuring Device:*

6.1.1 *Electronic Balance*, commercially manufactured capable of measurements to the nearest 0.001 g. The load measuring device capacity will depend upon the total weight of the liquid reservoir when filled with the test liquid. A capacity of 400 g will generally be quite satisfactory.

6.1.1.1 A load measuring device other than a commercially manufactured electronic balance, for example the load cell of a tensile testing machine as described in Test Method D 828, or an independent load measuring device may be used, providing the sensitivity and capacity comply with 6.1.1.

6.2 *Test Plate:*

6.2.1 A smooth, flat plexiglas test plate configured as follows: It shall have a diameter greater than 10 cm and a thickness greater than 2 cm. It shall have an orifice in the center 3 ± 0.02 mm in diameter through which the liquid will flow to wet the specimen.

6.2.2 Other test plates with multiple orifices or made of porous materials may be used, but shall be described as part of the test report and will be considered non-standard.

6.2.3 As described in Section 12, the test specimen shall remain at a constant horizontal position in reference to the top of the fluid level in the fluid reservoir during a test. The standard test condition requires this position to be such that the bottom surface of the specimen in contact with the test plate is 0.5 ± 0.1 cm above the parallel plane represented by the top surface of the liquid in the liquid reservoir. This will prevent flooding of the test plate after saturation of the specimen. Because liquid is constantly removed at a variable rate from the liquid reservoir during the test, if the volume of the liquid used requires it, it may be necessary to maintain the plane of the specimen relative to the plane of the liquid reservoir. This may be done manually, but is better controlled with volume or position sensors and devices. If the flow of liquid continues uninterrupted to endpoint, maintaining these relative positions is unnecessary.

6.3 *Test Weight*, with a smooth, flat bottom surface to contact with the top surface of the specimen, greater than 5 cm in diameter and weighing 50 ± 1 g, used to ensure proper contact of the specimen with the test plate surface.

6.4 *Liquid Reservoir*, a chamber or container made of plexiglas having a capacity of at least 200 mL and a filled weight of about 350 g connected to the load measuring device in 6.1. A flexible silicone tube having an inside diameter greater than 3.2 mm, such that it will not restrict the flow of liquid through the orifice in the test plate, leads from the liquid reservoir to the orifice in the center of the test plate. (See Fig. 1.)

6.5 *Liquid Supply Container*, for a supply 1 L or more, depending upon the amount of testing to be done for use in replenishing the liquid in the liquid reservoir between tests. This may be done manually, but is more conveniently done automatically using automatic volume or position sensors (see 6.2.3).

6.6 *Data Acquisition Equipment*—An analog or digital data collection device to attach to the load measuring device for use in capturing data during a test.

6.6.1 In the simplest configuration of the apparatus, the absorbency data for a test specimen may be acquired manually by recording the liquid weight remaining in the liquid reservoir as measured by the load-measuring device at intervals using a timer or stop watch.

6.6.2 A more useful approach may be to connect the load-measuring device to an analog recorder, providing a continuous record of the progress of the analysis.

6.6.3 A record of the readings of the load-measuring device at specific intervals may be achieved using a printing device attached to the load-measuring device.

6.6.4 For the most comprehensive testing, the load-measuring device may be attached to a microprocessing device for data capture and subsequent data analysis. In this case, it

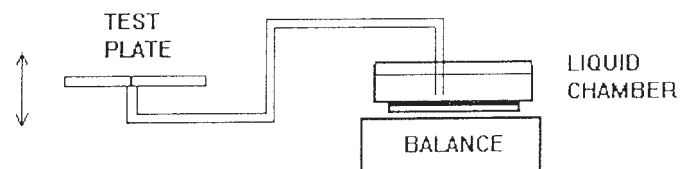


FIG. 1 Liquid Reservoir

will be convenient to control other features of the analysis, such as adjustment of the positioning of the test plate (6.2.3) using the microprocessor as well.

7. Reagents

7.1 *Reagent Water*—Any of the types of reagent water described in Specification D 1193 may be used in this test method.

7.2 Other test liquids compatible with the apparatus may be found suitable for specific testing purposes. When these are used, state in the report that test results do not comply with this test method.

8. Sampling

8.1 Acceptance sampling shall be done in accordance with Practice D 585.

8.2 When testing is being done for purposes other than acceptance of a lot of material, other sampling plans stated in the report may be used. Practice E 122 is recommended.

9. Test Specimens

9.1 For each test specimen, prepare ten replicates 50.0 ± 0.5 mm in diameter and at least 0.3 g in mass.

9.1.1 Multiple layers should be used to reach minimum mass, if necessary.

9.1.2 Specimens of other diameters and thicknesses may be used but shall be described as part of the report and considered nonstandard.

10. Calibration

10.1 Calibrate the load measuring device based on the vendors' procedures or recommendations.

10.1.1 Calibration with standard weights certified and traceable to the National Institute of Standards and Technology (NIST) is recommended.

11. Conditioning

11.1 Condition the samples as required in Practice D 685.

12. Procedure

12.1 Attach the liquid reservoir to the load-measuring device, and adjust the measuring device read-out to 0.000 g with the reservoir in place. Add reagent water to the liquid reservoir. Choose an amount (200 g is recommended) which will be suitable for most testing purposes.

NOTE 1—To minimize evaporation, the reservoir may be covered during the analysis. The weight of the cover, if it touches the reservoir, may also be tared out in 12.1.

12.2 Place a specimen on the test plate.

12.3 Place the test weight on top of the test specimen.

12.4 Initiate the test as required by the equipment used.

12.4.1 Since the specimen bottom surface is above the horizontal liquid level in the reservoir, it will be necessary to initiate liquid flow. This may be done manually or automatically by raising the liquid level in the orifice in the test plate. Pinching the tube connected to the orifice until the liquid raises to touch the specimen is acceptable.

12.5 Record the liquid absorption process using one of the means described in 6.5.

12.6 Continue the test until the absorption process removes less than 3.0 mg of fluid from the liquid reservoir in 5 s (0.6 mg/s).

12.7 Remove the saturated test specimen.

12.8 Dry the test plate and refill the liquid reservoir as in 12.1.

12.9 Repeat the procedure on additional test specimens.

13. Calculation and Interpretation of Results

13.1 Various information may be calculated from the data obtained, as agreed upon by users of this test method.

13.2 A recorder trace of the absorption process as in 6.6.1.1 may be provided. The amount of liquid absorbed by the test specimen is plotted as a function of time. A typical example is shown in Fig. 2.

13.3 Specimens having a steep slope in the initial portion of the absorption curve when tested by this procedure exhibit the fastest initial absorption rate and may show lower absorption times when tested by Test Method D 824 than do samples having less steep slopes in the initial portion of the absorption curve.

13.4 Specimens absorbing a higher amount of test liquid at termination of the test (that is, the moment the absorption rate falls below 0.6 mg/s, as in 12.6) may show higher water holding capacity when tested by Test Method D 4250.

13.5 The absorption process is complex, and dependent upon numerous compositional and design features of absorbent papers. No relationship between the initial absorptive rate (13.3) and the maximum absorptive capacity (13.4) should be assumed.

13.6 The absorption rate in millilitres per second of a specimen may be calculated at any desired time as the slope of the tangent to the absorption process (13.2).

13.7 The maximum absorbent capacity of the specimen in grams of test liquid may be calculated.

14. Report

14.1 Report the following information:

14.1.1 The number of replicates of each specimen tested.

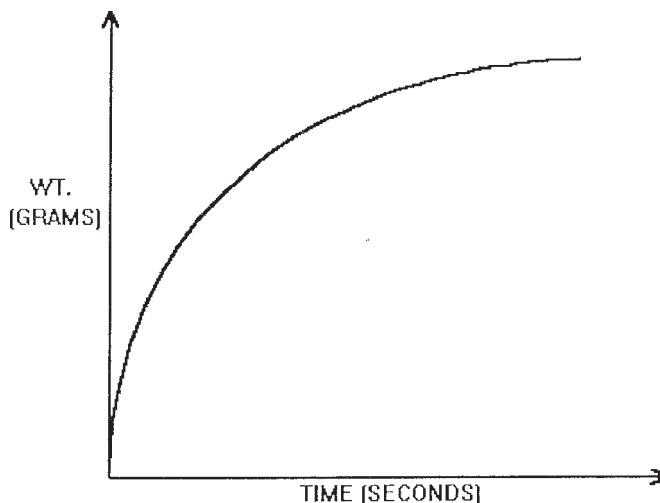


FIG. 2 Example of Recorder Trace

14.1.2 Specimen diameter to the nearest 0.5 mm.

14.1.3 Specimen mass to the nearest milligram.

14.1.4 Amount of liquid absorbed in milligrams at time intervals to be specified.

14.1.5 Total time elapsed when absorption rate first falls below 0.6 mg/s, in seconds.

15. Precision and Bias

15.1 *Precision*—Based on testing of a variety of absorbent paper products in a single laboratory, the repeatability of the standard deviation and the 95 % repeatability limit for samples tested as required in Section 12, (that is samples allowed to

absorb fluid until they reach saturation as described in this test method) are approximately 6.3 % and 17.6 %, respectively of the amount of fluid absorbed. The reproducibility of the test method is being determined.

15.2 *Bias*—No statement is made regarding the bias of the procedure because the results of this test method are dependent upon the specific testing procedures.

16. Keywords

16.1 absorbency; absorptive capacity; absorptive rate; demand wettability; paper; paperboard

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