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T 558

DRAFT NO. 02 - SARG

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WORKING GROUP CHAIRMAN 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 and absorbency of sheeted materials using an automated contact angle tester

(Five-year review of Official Method T 558 om-15)

(no changes from Draft 1; editorial corrections incorporated)

1. Introduction

1.1 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.

1.2 This test method is an 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°.

2. Scope

2.1 This test method measures the contact angle of a test liquid in contact with 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, tackiness, 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.

2.2 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 here after. It is also possible to evaluate the sorptive properties of a surface, as the remaining liquid volume on top of the specimen surface is measured as a function of time.

2.3 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.

3. Applicable documents

- TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product”;
- TAPPI T 402 “Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products,”
- TAPPI T 409 “Machine Direction of Paper and Paperboard;”
- TAPPI T 455 “Identification of Wire Side of Paper;”
- TAPPI T 552 “Determination of Wetting Tension of Polymeric Films and Coated Surfaces via the Mayer Rod Technique;” (*withdrawn*)
- TAPPI T 698 “Determination of Wetting Tension of Polyethylene and Polypropylene Films and Coatings (Modified Visking Analytical Technique);” (*withdrawn*)
- TAPPI T 458 “Surface Wettability of Paper (Angle of Contact Method);”
- TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.”
- ASTM D 1193 “Specification for Reagent Water;”
- CPPA F.3H “Surface Wettability of Paper and Paperboard;”
- ASTM E 122 “Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process.”

4. Definitions

4.1 *Contact angle,* the angle formed by the substrate and the tangent to the surface of the liquid drop in contact with the substrate, shown as "C" in Fig. 1.

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

4.3 *Droplet height,* height of the droplet in contact with the specimen surface, shown as distance "H" in Fig. 1.

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

4.5 *Contact time,* the length of time the droplet has been in contact with the specimen surface.

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.

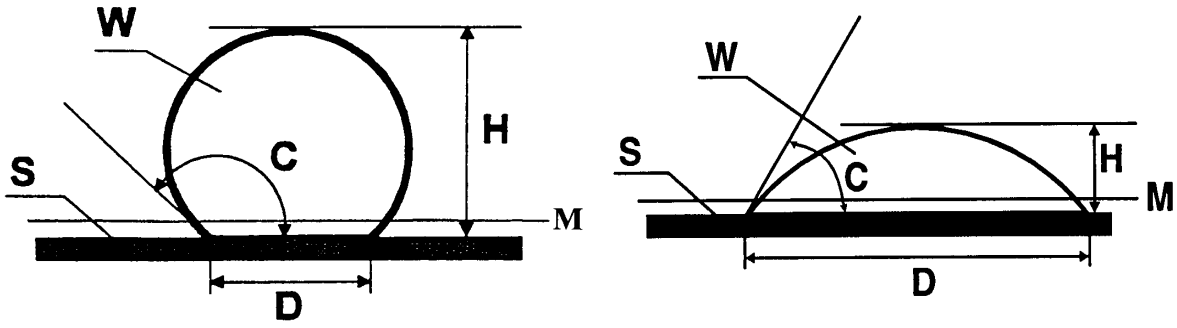

Fig. 1. Principle of measurement: W: water droplet; S: specimen; H: height of droplet; C: contact angle; D: diameter of the surface of contact of the droplet; M: minimum height of drop to be analyzed.

5. Summary

5.1 A drop of a specified volume of water or another agreed test liquid is automatically 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.

5.2 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 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.

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

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

5.5 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 immediately on contact with the specimen surface. Therefore the Procedure A should be tried as the first option.

5.6 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.

5.7 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 on contact with the specimen surface.

5.8 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.

6. Significance

6.1 Contact angle measurements can be used to study the relative sorptive rates of uncoated sorbent papers, or to study the relative printing or writing characteristics of coated or sized printing and writing papers.

6.2 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.

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

6.4 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.

6.5 It is generally found that papers having contact angles with water-based inks in the range 90 to 110° work best (*1*). 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°.

6.6 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.

6.7 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 different 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.

6.8 The complex interaction between a liquid and a surface can be looked upon as a combination of three different processes; wetting, absorption and adsorption. Wetting is best explained with a drop of water on a plastic film. Here 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, absorption is dominating. When the drop is spreading across the surface (increasing base diameter), the interaction is based on adsorption.

7. Apparatus[1]

7.1 An automated contact angle tester is required to perform the testing described in the procedure and consists of the following components, each of which are described in detail below: 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.

7.1.1 The *light source* is described as follows:

7.1.1.1 A halogen lamp is 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.

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

7.1.2 The *video camera* is described as follows:

7.1.2.1 The video camera is equipped with a lens to achieve an image view of about 10 x 7.5 mm.

7.1.2.2 The video camera is equipped with an electronic shutter. The shutter is set for a one millisecond exposure

[1] Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.

time for the purpose of this method.

7.1.2.3 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.

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

7.1.2.5 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.

7.1.3 The *specimen stage* is described as follows:

7.1.3.1 The specimen must be positioned so the test surface appears flat and horizontal to the video camera.

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

7.1.3.3 When the specimen is moved to a new area, the previous drop's wetted area must be avoided.

7.1.4 The *liquid delivery system* is described as follows:

7.1.4.1 The pump drives a 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.

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

7.1.4.3 For certain liquids and/or drop sizes a PTFE tube with different dimensions may be used.

7.1.4.4 The drop size, tubing material and tubing dimensions stated in 7.1.4.1 are standard for this test method, and any deviations from the conditions stated therein must be included in the report.

7.1.5 The *drop applicator* is described as follows:

7.1.5.1 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 to the droplet. Depending on the wetting properties between the liquid and the specimen surface, there are two different procedures, A and B, for the application of the drop. Depending on stroke length and acceleration, there are some timing considerations.

NOTE 2: Stroke, droplet velocity and acceleration are important to the application of the drop. The correct approach is to capture the image of the highest contact angle as early as possible.

7.1.5.2 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 applicator may not vary more than +1.0 ms.

7.1.5.3 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 must, for any given drop size, stroke length and distance above the specimen surface, not vary more than ± 1.0 ms.

7.1.5.4 With the video standards described in 7.1.2, 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 (1-19 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. An offset of 5 ms will for example capture images at 5, 25, and 45 .. ms and a 12 ms offset will capture images at 12, 32, and 52 .. ms after initial contact between the drop and the specimen surface.

7.1.6 The *video image capture system* is described as follows:

7.1.6.1 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.

7.1.6.2 The resolution of the digitized image should be at least 512 by 512 pixels to provide sufficient details for the image analysis.

NOTE 3: It is suggested that the minimum resolution should not exceed a pixel size of 20 microns.

7.1.7 The software functions for light calibration, scale factor adjustments, trigger and capture time settings, data analysis and reporting are described as follows:

7.1.7.1 *Light adjustment.* 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 must 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, camera sensitivity or gain. If such an adjustment is needed, the light calibration must be repeated before a test is performed.

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

7.1.7.3 *Initial contact time setting.* To achieve accurate contact times reported by the system, the timer must start within ± 2 ms upon contact of the liquid drop and the specimen surface. This timing margin will result in a ± 10% timing error.

7.1.7.4 *Image time stamp.* Each captured image must 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.

7.1.7.5 *Data analysis and presentation* described as follows: (1) For each image captured the applied drop's base diameter, height, contour and projected area are observed. From these primary observations the drop's contact angle, volume and contact area are calculated. (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). (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.

8. Calibration

8.1 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 must 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.

8.2 For verification, use artificial drops 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. The physical dimensions of the steel ball should be accurately measured to allow the contact angle and the volume of the artificial drop to be calculated as part of a sphere with tolerances of accuracy as set forth in this test method.

9. Maintenance

Consult the manual with regard to the instrument used.

10. Reagents and materials

10.1 *Reagent water.* Water of any of the types listed in ASTM Specification D1193 for reagent water may be used in this procedure.

10.2 Other liquids and test surfaces may be used as agreed 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.

11. Sampling, test specimens, and test units

11.1 *Sampling:*

11.1.1 Sample the material to be tested as described in TAPPI T 400.

11.2 *Test specimens:*

11.2.1 Determine and mark the machine direction of each test unit following TAPPI T 405. Be careful not to touch the areas to be tested, or contaminate them in any other way.

11.2.2 Determine and mark the felt and wire sides of each test unit, if applicable, following TAPPI T 455. 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.

11.2.3 When the specimen thickness is not greater than 1.0 mm, cut three clean specimen strips 14.9 ± 0.1 mm wide and about 300 mm long, free of folds, wrinkles, blemishes, water marks and other defects not normally inherent in

the specimen. This width is specified to minimize paper curl and to prevent the droplet from reaching the specimen edge during the test.

11.2.4 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, care must be taken that the liquid drop does not reach the edge of the specimen before the test has terminated.

11.2.5 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.

11.2.6 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 can be used, provided this has been agreed in advance and is stated in the report.

12. Conditioning

Condition the test specimens in conformance with TAPPI T 402.

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 below.

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 ten 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 information in following this procedure.

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 the standardized drop size out 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 Select a 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 the standardized drop size out 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 a stroke short enough to assure 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 cannula 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 system tip.

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

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 step 13.3.4 above.

14. Calculations and interpretation of results

14.1 *Calculations.*

14.1.1 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 11.2.6 for details on non-symmetrical surface properties.

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

14.1.3 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 (see Fig. 1). Other distances may be used as agreed by users of this test method provided they are agreed in advance, are compatible with the equipment used, and are stated in the report.

14.1.4 The data compiled from all captured images in one sequence are represented by three selected check points 0.1, 1.0 and 10 s. Other check points may be used as agreed by users of this test method provided they are agreed in advance, are compatible with the equipment used, and are stated in the report.

14.1.5 Contact angle is calculated for each sample by averaging the compiled contact angle values for the selected checkpoints. Using the software included with the instrument, calculate the coefficient of variation of the averaged contact angle values.

14.1.6 Volume is calculated for each sample by averaging the compiled volume values at the selected checkpoints. Using the software included with the instrument, calculate the coefficient of variation of the averaged volume values.

15. Report

15.1 Report the following information:

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

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

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

15.1.4 The stroke applied.

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

15.1.6 The number of drops used for the test.

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

15.1.8 The average volume calculated at the selected check points for all the drops in a test including the coefficient of variation.

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

16. Precision and bias

16.1 Contact Angle:

Repeatability (within a laboratory) 6%

Reproducibility (between laboratories) 13%

16.2 Drop Volumes:

Repeatability (within a laboratory) = 10%

Reproducibility (between laboratories) = 20%

Repeatability and reproducibility are estimates of the maximum difference (at 95% confidence) that should be

expected when comparing test results for materials similar to those described below under similar test conditions. These estimates may not be valid for different materials and testing conditions.

16.3 These estimates of repeatability and reproducibility are based on results from an interlaboratory trial conducted in 1995 using the pm-95 version of the method; testing was conducted using both procedures A and B. The trial was conducted in accordance with TAPPI T 1200 "Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility." Testing was conducted on 6 paper grades: newsprint, SBS coated food packaging board, directory paper, matte coated offset, cast-coated gloss cover and uncoated xerographic. Contact angle and drop volume were determined at three checkpoints (0.1, 1.0 and 2.0 seconds or 0.1, 1.0 and 10.0 seconds). Between 10 and 12 laboratories are included in the calculations for each sample. Reagent water and a 4-µL droplet size were used for all samples.

16.4 For this interlaboratory trial a test result was defined to be the average of 10 determinations at each checkpoint on a single specimen. Repeatability is the variation between test results on 3 specimens tested successively. Reproducibility is the variation between the average of the 3 test results in each laboratory. See Tables 1 and 2.

17. Keywords

Contact angle, Paper, Paperboard, Adsorption, Absorptivity, Wetting, Wettability, Adhesion, Printing.

18. Additional information

18.1 Effective date of issue: To be assigned.

18.2 Revisions in the 2010 edition include a new precision statement and several editorial changes.

Table 1. Contact angle data table.

Material	*Checkpoint*	*Grand Mean*	*Between Lab Stnd Dev*	*Repeatability r and %r*	*Reproducibility R and %R*	*Labs Included*
Newsprint	0.1	88.79	6.47	7.59 8.5%	19.47 21.9%	12
	1.0	64.72	12.38	16.98 26.2%	38.28 59.1%	
	2.0	51.53	14.29	29.86 57.9%	49.58 96.2%	
SBS Coated Board	0.1	67.93	2.24	4.38 6.4%	7.59 11.2%	12
	1.0	60.29	0.95	2.16 3.6%	3.41 5.7%	
	2.0	58.44	0.99	3.57 6.1%	4.52 7.7%	
Directory	0.1	71.12	3.43	5.40 7.6%	10.91 15.3%	12
	1.0	49.37	5.08	5.79 11.7%	15.21 30.8%	
	10.0	28.11	7.03	8.53 30.4%	21.25 75.6%	
Matte Coated Text	0.1	79.88	2.42	5.60 7.0%	8.73 10.9%	12
	1.0	69.00	2.00	5.79 8.4%	8.01 11.6%	
	10.0	57.10	2.86	3.57 6.3%	8.67 15.2%	
Cast Coated Gloss	0.1	93.27	6.50	4.04 4.3%	18.48 19.8%	11
	1.0	91.38	6.29	8.23 9.0%	19.28 21.1%	
	10.0	83.59	4.05	13.49 16.1%	17.53 21.0%	
Xerographic	0.1	108.33	3.06	8.75 8.1%	21.19 11.3%	11
	1.0	108.67	3.00	9.00 8.3%	12.24 11.3%	
	10.0	107.82	2.44	8.97 8.3%	11.25 10.4%	

Results listed in degrees for contact angle and microliters for drop volume

Table 2. Drop volume data table

Material	*Checkpoint*	*Grand Mean*	*Between Lab Stnd Dev*	*Repeatability r and %r*	*Reproducibility R and %R*	*Labs Included*
Newsprint	0.1	3.567	0.474	1.042 29.2%	1.676 47.0%	11
	1.0	2.920	0.285	0.360 12.3%	0.867 29.7%	
	2.0	2.071	0.474	1.042 50.3%	1.676 80.9%	
SBS Coated Board	0.1	3.818	0.211	0.260 6.8%	0.640 16.8%	11
	1.0	3.829	0.210	0.332 8.7%	.0670 17.5%	
	2.0	3.827	0.215	0.269 7.0%	0.654 17.1%	
Directory	0.1	3.634	0.359	0.435 12.0%	1.086 29.9%	11
	1.0	2.749	0.438	0.543 19.7%	1.330 48.4%	
	10.0	1.058	0.379	0.518 49.0%	1.169 110.5%	
Matte Coated Text	0.1	3.543	0.145	0.402 11.3%	0.568 16.0%	11
	1.0	3.552	0.192	0.490 13.8%	0.723 20.4%	
	10.0	3.138	0.236	0.582 18.5%	0.875 27.9%	
Cast Coated Gloss	0.1	3.707	0.276	0.343 9.3%	0.837 22.6%	10
	1.0	3.703	0.274	0.440 11.9%	0.878 23.7%	
	10.0	3.646	0.286	0.515 14.1%	0.945 25.9%	
Xerographic	0.1	3.680	0.225	0.404 11.0%	0.742 20.2%	10
	1.0	3.689	0.218	0.404 11.0%	0.726 19.7%	
	10.0	3.613	0.192	0.413 11.4%	0.673 18.6%	

Results listed in degrees for contact angle and microliters for drop volume

Appendix A

A.1 *Wetting properties*

A.1.1 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 liquid 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.

A.1.2 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); the non-wetting or non-absorbency, or both, of a barrier paper.

A.1.3 TAPPI T 458 "Surface Wettability of Paper (Angle of Contact Method)" (which has been reclassified as a Classical Method) and CPPA F.3H "Surface Wettability of Paper and Paperboard" measure contact angle at 5 and 60 s after contact. These methods provide meaningful information only in cases where the adsorption rate is negligible during the first few seconds after contact and is suitable for predicting the ruling and writing qualities of paper. This automated method is capable of measuring changes in contact angle and drop volume dynamically within 20 to 40 ms of contact and provides information which may be useful in high speed operations such as printing, coating, sizing, and gluing. TAPPI T 552 "Determination of Wetting Tension of Polymeric Films and Coated Surfaces via the Mayer Rod Technique" (*withdrawn*) and TAPPI T 698 "Determination of Wetting Tension of Polyethylene and Polypropylene Films and Coatings (Modified Visking Analytical Technique)" (*withdrawn*) use a somewhat different, semi-quantitative approach to provide information regarding the energy relationship between a polymer film and a non-aqueous 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.

A.2 *Other properties*

A.2.1 This method specifies conditions for the testing of a wide range of papers considered to be of low absorbance or non-absorbent, 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 non-aqueous 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 liquids upon prior agreement of those involved in the testing, provided the agreed liquid is compatible with the equipment used. Where test liquids other than reagent water are used, the actual liquid used is reported.

A.3 *Interpretation of results.*

A.3.1 Paper specimens tested with water and ink.

A.3.1.1 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. It must be understood, however, that other variables, for example the type and uniformity of sizing, are quite important in ruling and writing quality, as well.

A.3.1.2 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.

A.3.1.3 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 s to 60 s after drop deposition, feathering upon standing may occur, depending upon the drying time required for the ink used.

A.3.1.4 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.

A.3.1.5 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 can be detected.

A.3.1.6 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 liquid and the specimen surface until the contact angle goes below 90° (*1*).

A.3.1.7 In some applications the "initial contact angle" of a sorptive process is of special interest. As the drop must stabilize on the surface, it is usually not possible to measure the initial contact angle. When the sorptive process is mainly absorption (*2*), however, the contact angle rate of change can be extrapolated down to time equals zero for useful results.

A.3.2 *Film specimens.*

A.3.2.1 Untreated polymer films generally exhibit surface free energy values in the range of 20 to 50 mJ/m^2, depending upon the chemical formulation. Treatment with, for example, 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.

A.3.2.2 If a liquid has a surface tension between the surface free energy of the film specimen and the surface tension of the test liquid in this method, the contact angle of the 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 from this rule of thumb may, however, occur if the polarity of the used liquid differs greatly from that of reagent water.

A.3.2.3 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 vs. liquid surface tension, it is possible to determine the "critical surface tension of wetting" of the film as the surface tension where the 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.

A.3.2.4 In cases where one liquid exhibits sorption by a substrate or penetrates the substrate to some depth and a second does not, in for example 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, and adhesive viscosity differences may overshadow the impact of contact angle differences (*4*).

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