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Paper and board — Measurement of water contact angle by optical methods

Papier et carton — Mesurage de l'angle de contact de l'eau par des méthodes optiques

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The interaction between the liquid and the solid phases influences the contact angle. Contact angles above 90° define a situation where the liquid is repelled by the solid; below 90° defines a situation of attraction where the liquid wets the surface. The magnitude above or below 90° shows the relative degree of repulsion or attraction between the two phases.

On many surfaces the contact angle varies with duration of contact through a combination of spreading, penetration (in the case of porous substrates) and evaporation. Both manual and automated apparatus are available for optical measurement of contact angle, but automated equipment is preferred for precision and rapid measurement, and because it is often applicable to a wider combination of liquid and paper or board samples.

Contact angle measurement is used to predict how liquids interact with paper surfaces. This document describes the most common form of test, using water as probe liquid, and from the data a probable interaction between the paper surface and another liquid with comparable surface tension and viscosity characteristics is often inferred. The veracity of this inference should be tested wherever possible.

Notwithstanding the above, contact angle measurement is used widely as a predictive tool in several industrial settings, for example:

a) for assessing writing, ruling or printing quality with water-based or solvent based inks (e.g. in inkjet, gravure or flexographic printing);

b) for gluing applications;

c) for wet offset lithographic printing;

d) for hot-foil applications;

e) for barrier or release coatings;

f) for coating applications.

In some cases, measurements of contact angle are used to calculate two or three components of the surface energy of the paper or board, which requires the use of two or three liquids of known surface energy, respectively. In such cases, the values calculated for the components of surface energy will be related to the liquids chosen for the analysis[1]. This is not covered in this document.

The test method described in this document is sometimes known as 'static' or even 'sessile drop', since on many surfaces the droplet remains static and in equilibrium with the paper surface and air. Yet on paper and board surfaces the droplet often changes its dimensions with time, due to sorption and wetting phenomena. This has led some instrument manufacturers and researchers to describe the automated optical technique outlined in this document as measurement of the 'dynamic' contact angle.

This document does not use the term 'dynamic' because this nomenclature confuses a measurement changing with respect to time with one that changes due to a plane of shear. For example, the Willhelmy plate method of contact angle measurement, which measures the force required to push a solid material into and then pull it out from a liquid reservoir, is a true 'dynamic' method[2].

Similarly, this document does not cover the situation where a droplet is placed on a horizontal surface that is subsequently tilted so that gravity causes the droplet to assume an asymmetric shape, then to commence movement.

Paper and board — Measurement of water contact angle by optical methods

1 Scope

This document specifies the method for optical assessment of the contact angle between water and the surface of paper and board, where the process of droplet formation, application to planar substrates, or measurement of the droplet shape in contact with the solid is performed by automated equipment.

The limits of measurement are determined by the capabilities of the instrumentation used. The instrumental capabilities defined by this document use a digital image capturing system operating at a minimum of 50 frames per second and needs the ability to perform the first measurement after no more than 20 ms to 40 ms contact between the droplet and substrate.

The test method is applicable to most kinds of paper or board however it cannot be applicable to structured materials.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 186, *Paper and board — Sampling to determine average quality*

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 13530, *Water quality – Guidance on analytical quality control for chemical and physicochemical water analysis*

3 Terms and references

For the purposes of this document, the following terms and definitions apply:

3.1
baseline
line of contact between the water droplet and paper or board surface, determined by optical means and taken as the plane from which the contact angle is measured

Note 1 to entry: Precise measurement of contact angle requires precise assessment of the baseline. For the optical system to achieve the best estimate of the baseline, it may be necessary to tilt the camera so that it views the droplet slightly from above, at a shallow angle (typically 0-3° relative to the horizontal), rather than directly from the side (0°). Experience shows this small change in viewing angle does not affect the measured contact angle value but can enhance detection of the baseline[3].

3.2
contact angle
Θ
angle to a *baseline* (3.1), formed by means of a tangent on the droplet contour through one of the three-phase points at the specified contact time

Note 1 to entry: The contact angle is expressed in degrees (°).

3.3
contact time
duration of contact(s) between the water droplet and the test piece surface, as measured automatically from the instant at which the timing mechanism is triggered to the instant of measurement

3.4
droplet height
H
distance from the top of the droplet in contact with the test piece surface as measured from the *baseline* (3.1) at the specified *contact time* (3.3)

Note 1 to entry: The droplet height is expressed in millimetres.

3.5
droplet base diameter
D
maximum width of the droplet base in contact with the test piece surface, as measured along the *baseline* (3.1) at the specified *contact time* (3.3)

Note 1 to entry: The droplet base diameter is expressed in millimetres.

3.6
three-phase point
point of intersection of the liquid/solid, liquid/gaseous and solid/gaseous boundary lines

4 Principle

A water droplet is applied to the horizontal planar surface of paper or board, the angle of contact it makes with the material is assessed by optical digital imaging.

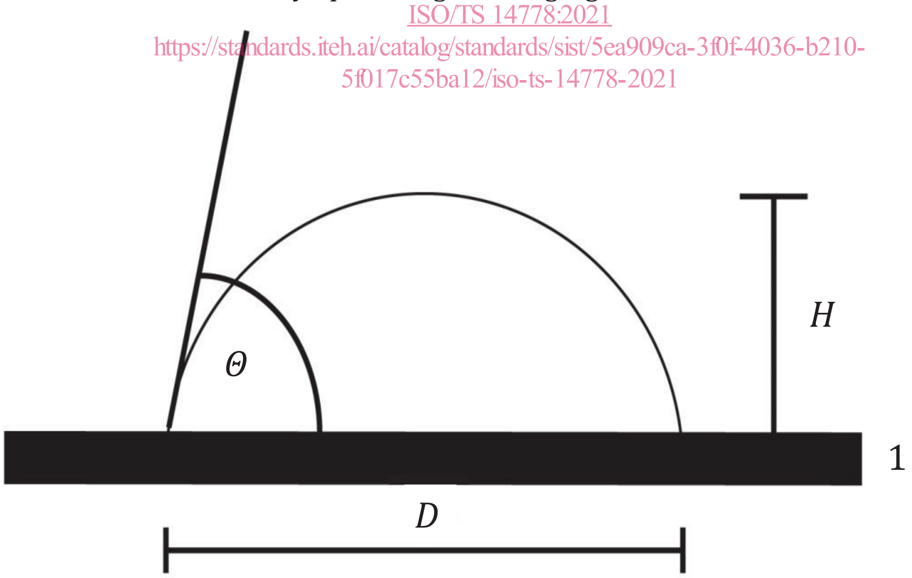


Figure 1 — Principle of measurement

Key

1 substrate

D droplet base diameter

H droplet height

Θ contact angle

5 Reagents

5.1 Distilled water, with a surface tension of at least 72 mN/m at 23 °C, as specified in ISO 13530.

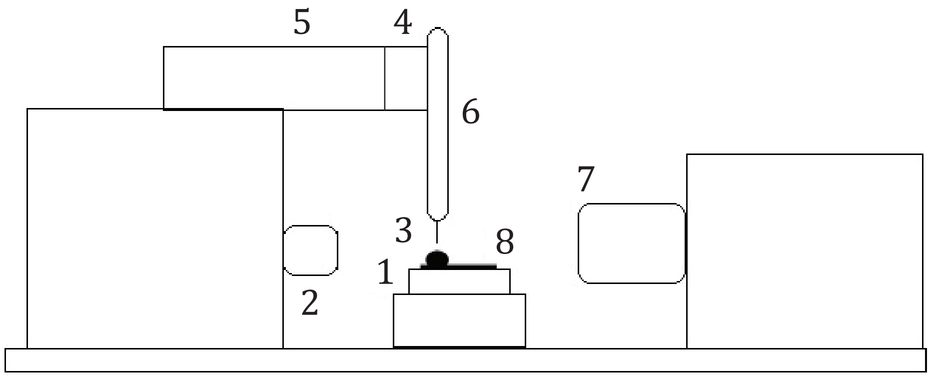
NOTE 1 This method can be used with other liquids providing they can be delivered in the form of a droplet by the liquid delivery system (6.1.3) described in this document, and providing they do not cause substantial swelling of the substrate over the time scale of the measurements such that the baseline can no longer be defined. Also, for liquids with high vapour pressure, the rate of evaporation may be too fast to allow the technique to be used, unless performed in a vapour-saturated atmosphere (e.g. a closed glass cuvette containing a few drops of solvent). Contact angle values produced using other liquids are not comparable with those produced using water.

NOTE 2 Some contact angle measuring apparatus (6.1) have the facility to heat liquids prior to droplet formation. Heating alters both surface tension and viscosity, both of which can affect contact angle. If the liquid is heated, this is a deviation from the standard.

NOTE 3 Many contact angles measuring apparatus (6.1) are also capable of measuring liquid surface tension and can be used to examine water surface tension. It is recommended to periodically check that the water has not been contaminated as it would have a direct effect on the contact angle readings.

6 Apparatus

6.1 Contact angle measuring apparatus.



Key

1 platform

2 light source

3 droplet

4 pump

5 liquid reservoir

6 droplet application system

7 video camera

8 substrate

Figure 2 — General schematic of automated contact angle apparatus

For the optical determination of contact angles on a horizontal, planar substrate, any type of apparatus having the general geometry shown in Figure 2 and further conforming to the requirements in the following sub clauses is used.

NOTE This schematic is not intended to describe all possible instrumental arrangements. Accordingly, instruments without a platform, with separated liquid container, with different kind of construction or those using different viewing arrangements, are also permitted.

6.1.1 Platform with a holder mechanism (key item 1), to secure the test piece, capable of maintaining flatness of the test piece over the duration of the test. The platform shall be horizontal.

NOTE The “platform” can be a table-top and the holder can be the housing of the device.

6.1.2 Light source (key item 2), capable of illuminating the droplet, with suitable heat filters, if needed to eliminate heating of the test piece or water droplet. The illumination system shall produce homogeneous lighting over the entire field of view.

6.1.3 Liquid delivery system (key item 5), consisting of a water reservoir and a dosing mechanism, capable of producing a water droplet of known volume to a tolerance of +/- 10 %. If the droplet volume is determined by the pumped volume, are shall be taken that no air bubbles are present as they will cause significant volume error.

NOTE 1 The volume relates to the droplet produced at the end of a capillary, not necessarily to the volume applied to the test piece surface. After release of the droplet there will always be a small portion of water remaining on the capillary, the amount of which will relate to the water, capillary diameter, capillary type and material, and droplet application technique.

NOTE 2 The dispensing volume and reproducibility of dosing system can be checked using an impermeable reference surface.

NOTE 3 Some contact angle measuring apparatus enable the volume calculation directly from pendant droplet in which case air bubbles won’t affect the final droplet volume.

6.1.4 Droplet application system (key item 6), allowing a water droplet of specified volume to be suspended at the end of a capillary before being applied to a paper or board surface under controlled conditions. The diameter and material of the capillary shall be chosen for application of water in accordance to Clause 5 and the droplet application system. The droplet application system shall be adjusted so that the droplet has minimum kinetic energy when it impacts the test piece surface.

The exact method of droplet application will differ between instruments. The method shall allow the droplet to detach from the capillary so as not to inhibit contact angle measurement after a contact time of at most 20 ms to 40 ms.

The material of the capillary shall be chosen

— to hinder wicking of the water up the exterior of the capillary;

— to be inert, such that it does not affect the liquid surface tension, and

— to allow release of the droplet with a minimum of effort.

Care shall be taken when interpreting behaviour of water on very hydrophobic surfaces, since the droplet may bounce for several hundred milliseconds due to the way some droplet application systems impart kinetic energy, which causes an apparent oscillation in contact angle values. Where such behaviour is observed, contact angle values during this period shall not be reported. This behaviour does not tend to occur on hydrophilic surfaces when using water.

6.1.5 Image capturing system (key item 7), incorporating an optical magnification mechanism that allows the shape and dimensions of the droplet in contact with the paper or board to be measured accurately.

The video camera shall be capable of taking a minimum of 50 frames per second, and the exposure of each frame shall take no more than 1 ms.

The ratio between image width (mm) and the number of horizontal pixels in the displayed image shall not exceed 0,02 mm/pixel to provide sufficient detail for image analysis.

The depth of field shall be sufficient to encompass the whole of the droplet contour on the paper or board surface.

6.1.6 Timing system, which is triggered when the droplet is released from the capillary, triggered directly before the droplet contacts the test piece surface or which is based on a captured video starting before the drop reaches the substrate. This timing system shall initiate before, or at the latest within 2 ms after contact, and shall allow each digital image to be assigned a unique time stamp with an accuracy of ±2 ms.

NOTE The nature of the trigger mechanism differs between instruments. In general, it will be a camera trigger or a software trigger. For example, in some instruments the droplet is dislodged from the capillary by a mechanical pulse, which simultaneously initiates the timer mechanism. In others, the timer starts when the falling droplet approaches a point just above the test piece surface. Alternatively, a continuous video can be taken starting before the droplet contacts the paper or board surface, such that the contact time can be back-calculated taking the point at which contact between the liquid and solid takes place as 'zero'.

6.1.7 Suitable image analysis software, that can process each digital image taken by the camera, producing values for droplet height, H, droplet base diameter, D, and contact angle, Θ, and in some cases an estimate of the droplet volume.

NOTE Various algorithms can be used to analyse the droplet shape or contour and provide contact angle values. The contact angle value determined is influenced by the mathematical procedure chosen. Many manufacturers use their own proprietary algorithms, depending on what shape the individual droplets of water assume on the sample surface[4], which causes variability between instruments. For this reason, the circle equation [see Formula (1)] has been chosen as the only algorithm to be compliant with this document.

7 Sampling

If the test is being made on a lot of paper or board, the sample shall be selected in accordance with ISO 186. If the test is being made on another type of sample, report the source of the sample and, if possible, the sampling procedure used, and make sure that the test pieces taken are representative of the sample received.

8 Conditioning

Place the contact measuring apparatus and distilled water (5.1) in the conditioned atmosphere specified in ISO 187 for sufficient time that they come to equilibrium Condition the sample in accordance with ISO 187. Keep the sample, and the test pieces from it, in the conditioned atmosphere throughout the test.

9 Preparation of test pieces

Prepare, in the same atmospheric conditions used to condition the sample, sufficient test pieces to perform at least valid 5 measurements per side. Avoid contact of the test area with hands or fingers, and avoid folds, creases, cracks and other defects, including watermarks.

Test piece dimensions depend on the characteristics of the holder mechanism (6.1.1) and on the contact angle measuring apparatus (6.1). All test piece widths shall be a minimum of 10 mm but shall not be so wide that it is difficult to maintain flatness. The width shall be sufficient to prevent the droplet from

