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SIST EN 828:2014

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 828**

January 2013

ICS 83.180

Supersedes EN 828:1997

English Version

## Adhesives - Wettability - Determination by measurement of contact angle and surface free energy of solid surface

Adhésifs - Mouillabilité - Détermination par mesurage de l'angle de contact et de l'énergie superficielle libre de la surface solide

Klebstoffe - Benetzbarkeit - Bestimmung durch Messung des Kontaktwinkels und der freien Oberflächenenergie fester Oberflächen

This European Standard was approved by CEN on 24 November 2012.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

<b>Contents</b>		<b>Page</b>
Foreword.....		<b>3</b>
<b>1</b> <b>Scope.....</b>		<b>4</b>
<b>2</b> <b>Normative references.....</b>		<b>4</b>
<b>3</b> <b>Terms and definitions .....</b>		<b>4</b>
<b>4</b> <b>Principle.....</b>		<b>6</b>
<b>5</b> <b>Test equipment.....</b>		<b>6</b>
<b>6</b> <b>Measuring conditions .....</b>		<b>7</b>
<b>7</b> <b>Procedure .....</b>		<b>8</b>
<b>8</b> <b>Expression of results.....</b>		<b>9</b>
<b>9</b> <b>Test report .....</b>		<b>9</b>
<b>Bibliography.....</b>		<b>11</b>

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[SIST EN 828:2014](https://standards.iteh.ai/catalog/standards/sist/7562612f-0d07-4c73-8e1d-1f006c2338a3/sist-en-828-2014)

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## Foreword

This document (EN 828:2013) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 828:1997.

## Safety Statement

People applying this document, the user shall be familiar with normal laboratory practice. This standard does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish safety and health practices and to ensure their compliance with the provisions of any regulatory conditions.

## Environmental Statement

It is understood that some of the material permitted in this standard may have a negative environmental impact. As technological advantages lead to acceptable alternatives for these materials, they will be eliminated from this standard as far as possible.

At the end of the test, the user of the standard should take care to carry out appropriate disposal of waste, according to local regulations.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

**EN 828:2013 (E)****1 Scope**

This European Standard specifies a method for the determination of the surface free energy of a solid surface by measuring the contact angle of a liquid wetting the solid surface. It allows the prediction of the ability of a particular adhesive to wet a particular adherend. It can be used to characterise surfaces intended for pre-treatment, coating or bonding.

NOTE 1 In order to determine the surface free energy, the method of measuring the static contact angle is used in combination with a statistical interpretation.

NOTE 2 The measurement results are influenced by mechanical surface roughness and chemical homogeneity.

**2 Normative references**

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 923:2005+A1:2008, *Adhesives — Terms and definitions*

**3 Terms and definitions**

For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

**3.1  
triple point**

point where the solid, the liquid and the gas phases coincide with each other

Note 1 to entry: The triple point can be identified in the silhouette of a liquid drop situated on a test piece, at the intersection of the drop's contour line with the test piece surface.

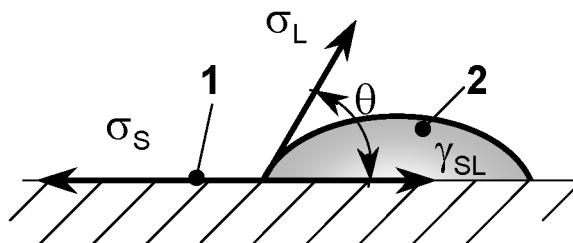
**3.2  
baseline**

in the case of plane test pieces, a straight line going through the two triple points

**3.3  
contact angle**

$\theta$

angle to the baseline, formed by a tangent to the drop contour going through one of the triple points (see Figure 1)

**Key**

1 solid body surface

2 liquid drop

 $\sigma_L$  surface tension (= surface energy) of the liquid in equilibrium with the gas phase $\sigma_S$  surface free energy of the solid body surface $\gamma_{SL}$  interfacial energy of the solid body surface in contact with the liquid $\theta$  contact angle**Figure 1 — Wettability****3.4****wettability**

a liquid coming into contact with a solid surface exhibits a typical drop shape. The characteristic of the drop is the angle formed by the tangent to the contour at the triple point (wetting point) (Figure 1). A contact angle = 0° indicates a surface that is completely wetted

**3.5****contour analysis**

image analysis procedure consisting in capturing the silhouette of a liquid drop on a surface by optical methods and calculating the contour profile of that silhouette

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**3.6****surface free energy of a solid** $\sigma_S$ 

energy measure (expressed in mN/m) which characterises the wettability of a solid material by a liquid that is based on the adsorption theory. The forces present in the state of equilibrium are described by Young's formula:

$$\sigma_S = \gamma_{SL} + \sigma_L \cdot \cos \theta \quad (1)$$

The Young-Dupré formula defines the work of adhesion obtained during wetting:

$$W_{ad} = \sigma_L + \sigma_L \cdot \cos \theta = \sigma_L \cdot (1 + \cos \theta) \quad (2)$$

The position of the thermodynamic equilibrium resulting in the formation of the static contact angle depends on both the pressure and the temperature conditions. When carrying out the measurement, standard conditions should be maintained.

Since the interfacial energy and the surface energy are based on interactive forces between atoms or molecules, it is necessary to take polarity into consideration when assessing the wettability.

Examples for polar interactions are as follows:

- dipole-dipole interactions;
- hydrogen bridge bonds;
- acid-base interactions.

## EN 828:2013 (E)

The non-polar (disperse) interactions are commonly described as London interactions.

In accordance with [1] and [2], the interfacial energy  $\gamma_{SL}$  between a solid body (S for "solid") and a liquid (L for "liquid") is the sum of the surface tensions of the two phases ( $\sigma_S + \sigma_L$ ), reduced by the disperse and the polar interactions at the phase border. These interactions are described as the doubled sum of the geometric mean values of the disperse ( $\sqrt{\sigma_S^D \cdot \sigma_L^D}$ ) and the polar ( $\sqrt{\sigma_S^P \cdot \sigma_L^P}$ ) tension components of the individual phases:

$$\gamma_{SL} = \sigma_S + \sigma_L - 2 \left( \sqrt{\sigma_S^D \cdot \sigma_L^D} + \sqrt{\sigma_S^P \cdot \sigma_L^P} \right) \quad (3)$$

Substitution  $\gamma_{SL}$  from the Young's formula (1) and rearrangement of formula (3) to the general form of a straight line

$$y = mx + b \quad (4)$$

leads to the following formula:

$$\underbrace{\frac{(1 + \cos\theta) \cdot \sigma_L}{2\sqrt{\sigma_L^D}}}_y = \underbrace{\sqrt{\frac{\sigma_S^P}{\sigma_S^D}}}_m \underbrace{\frac{\sqrt{\sigma_L^P}}{\sqrt{\sigma_L^D}}}_x + \underbrace{\sqrt{\frac{\sigma_S^D}{\sigma_S^P}}}_b \quad (5)$$

The square of the slope is the polar proportion of the solid body surface energy  $\sigma_S^P$ , the square of the ordinate intercept  $b$  is the disperse proportion  $\sigma_S^D$ . The sum of the two proportions is the total surface free energy of the solid body  $\sigma_S$ .

SIST EN 828:2014

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## 4 Principle

Ten drops each of at least three and up to eight known, different liquids are dosed onto a plane test piece surface. For each drop, the left and the right contact angles are measured. From the averaged contact angles of each liquid combined with its surface tension and its polar and disperse proportions, the surface free energy of the solid body is calculated, subdivided into the polar and the disperse proportions.

Preferably, the liquids used should exhibit different polar and disperse proportions of the interfacial tension. Recommended liquids and their characteristic interfacial tensions are listed in Table 1.

The drop volumes suitable for the measurement depend on the type of the liquid and should be adjusted such that the negative influence of gravity on the contact angle is kept to a minimum.

## 5 Test equipment

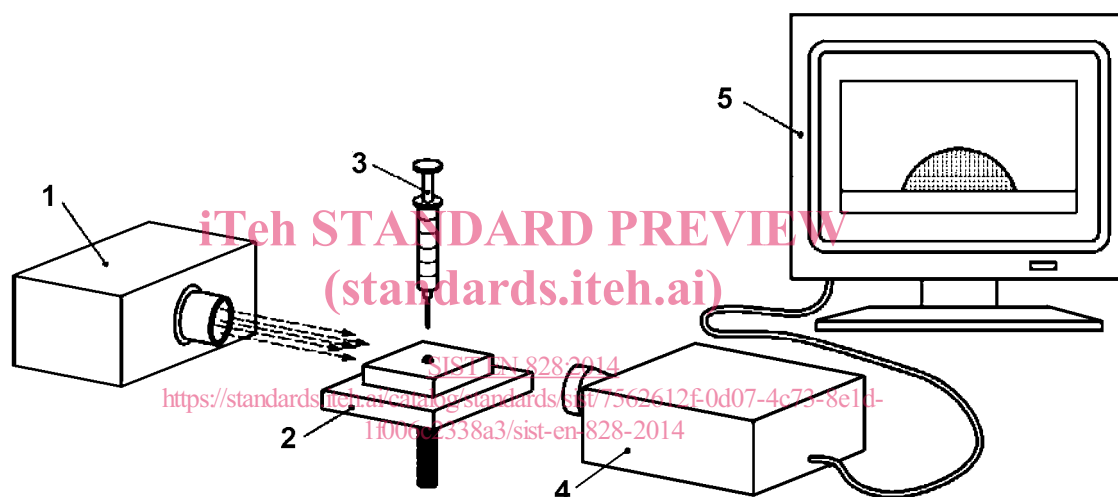
**5.1 Solid surface to be assessed**, substrate with pre-treated or untreated surface.

**5.2 Contact-angle measuring system**, any contact-angle measuring device, preferably systems comprising digital image acquisition and image analysis to comply with the current state of the art. Figure 2 gives an example of a schematic representation of a contact-angle measuring system.



Table 1 — Recommended test liquids

Designation of the test liquid	Surface tension mN/m	Disperse proportion mN/m	Polar proportion mN/m	Literature (Author)
Water	72,80	21,80	51,00	See [3]
Diiodomethane	50,80	50,80	0,00	See [3]
Ethylene glycol	47,70	30,90	16,80	See [3]
Glycerol	63,40	37,00	26,40	See [3]
Hexadecane	27,60	27,60	0,00	See [3]
$\alpha$ -Bromine naphthalene	44,60	44,60	0,00	See [3]
Benzyl alcohol	38,90	29,00	9,90	See [4]
Decalin	30,60	30,60	0,00	See [3]

**Key**

- 1 lighting
- 2 sample carrier
- 3 dosage
- 4 optical system
- 5 screen

Figure 2 — Contact-angle measuring system

**5.3 Reagents**, at least three and up to eight of the test liquids recommended in Table 1.

**WARNING** — These reagents are chemicals — carefully follow the safety advice on the labels and in the safety data sheets.

**5.4 Micropipette** (or suitable syringes for micro-dosing) which can be fastened above the sample carrier or the surface to be tested, respectively, by means of an appropriate holder.

## 6 Measuring conditions

Constant measuring conditions in accordance with the thermodynamic state function (temperature and pressure) shall be maintained.