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

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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

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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of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

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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

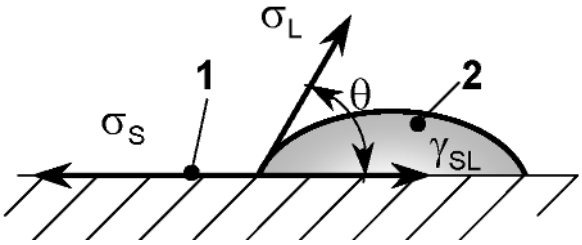
For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

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
θ
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

σ_L surface tension (= surface energy) of the liquid in equilibrium with the gas phase

σ_S surface free energy of the solid body surface

γ_{SL} interfacial energy of the solid body surface in contact with the liquid

θ 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 = 0º 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

3.6
surface free energy of a solid
σ_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.

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

In accordance with [1] and [2], the interfacial energy γ_{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 γ_{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:

$$\underbrace{\frac{(1+\cos\theta)\cdot\sigma_L}{2\sqrt{\sigma_L^D}}}_{y} = \underbrace{\sqrt{\sigma_S^P}}_{m}\underbrace{\frac{\sqrt{\sigma_L^P}}{\sqrt{\sigma_L^D}}}_{x} + \underbrace{\sqrt{\sigma_S^D}}_{b} \quad (5)$$

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

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.

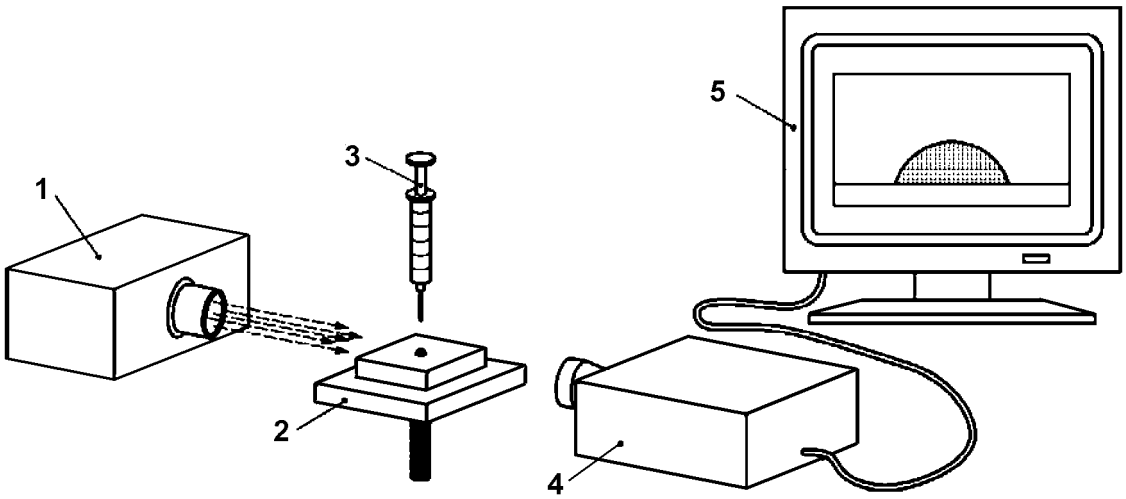
5 Test equipment

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]
α-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.

7 Procedure

7.1 The contact-angle measuring device shall be adjusted horizontally by means of the level.

7.2 The contact-angle measuring device, the lighting and the image acquisition and analysis system are switched on. The image display is adjusted to be sufficient with regard to brightness and contrast (the manufacturer's information should be taken into consideration).

The light source of the contact-angle measuring device shall be calibrated to obtain optimum contrast and grey scale value in the image of a drop and of a surface.

The dosing needle is moved to the upper edge of the image and the zoom of the contact-angle measuring device is set in a way that the contour width of the located drop covers two thirds of the width of the image. Afterwards the drop image shall be focussed.

7.3 The dosing system is filled with one of the selected liquids. It shall be ensured that any contaminations during filling are avoided.

7.4 A plane sample of the surface to be measured is placed on the sample carrier. The sample carrier shall be adjusted such that the sample surface is located in the lower half of the image and in a horizontal position.

7.5 The dosing needle is positioned approximately 1 mm above the sample surface. The drop shall be dosed such that its volume is between 2 µl and 6 µl, depending on the liquid selected. Recommended drop volumes are given in Table 2, depending on the liquid selected.

Table 2 — Recommended drop volumes

Liquid	Drop volume µl
Water	2–6
Diiodomethane	2–3
Ethylene glycol	2–6
Glycerol	2–6
Hexadecane	2–6
α-Bromine naphthalene	2–6
Benzyl alcohol	2–6
Decalin	2–6

7.6 One drop of the test liquid is dosed onto the surface. In doing so, the drop volumes given in Table 2 and the dosing rate of 100 µl/min shall not be exceeded.

7.7 The baseline shall be adjusted such that it intersects the triple points of the drop. In order to facilitate locating the triple points, a top-view angle of maximally 2° may be set. The maximum deviation of the baseline from the horizontal (slope) shall not exceed 0,1.

7.8 The measurement shall be started immediately (max. 15 s) after the completion of dosing. Over the contact period, no reaction and no mutual material conversion between the test liquid and the surface to be measured shall take place. As an example, it is recommended to carry out one measurement every second over a period of 20 s.

7.9 Depending on how the individual liquid drops behave on different solid body surfaces, different algorithms shall be applied for the accurate determination of the contact angle (e.g. equation of a circle for contact angles < 20°, a quadratic equation for contact angles between 20° and 120°, polynomial equation for contact angles > 120°).

7.10 Repeat sub-clauses 7.3 to 7.9 for a total ten drops of each liquid. In order to get sufficient information concerning the homogeneity of a sample, the measurements shall be carried out at different areas of the sample. Uncertain records which may have been caused by dust, contamination etc., are not included in the evaluation. The contact angle results from the mean value of the measurement values for each liquid.

7.11 The sub- clauses 7.3 to 7.9 shall be repeated for each test liquid selected. The liquids shall be selected so that a maximum variation of the polar and the disperse proportions is represented.

NOTE A higher number of test liquids increase the informative value of the results.

Used liquids shall not be filled back into the respective sample jar or storage bottle. They shall be discarded and properly disposed off after the end of the measurements.

8 Expression of results

For the evaluation of the surface free energy, the term $\frac{(1+\cos\theta)\cdot\sigma_L}{2\sqrt{\sigma_L^D}}$ is plotted against the term. $\frac{\sqrt{\sigma_L^P}}{\sqrt{\sigma_L^D}}$

where

θ are the measured and averaged contact angles for the individual liquids;

σ_L are the total surface tensions of the liquids;

σ_L^P and σ_L^D are the polar and the disperse proportions of the total surface tensions.

Subsequently, the slope m and the ordinate intercept b of the straight line are determined by linear regression. The square of the slope is the polar proportion of the solid body surface energy σ_S^P, the square of the ordinate intercept b is the disperse proportion σ_S^D. The sum of the two proportions is the total surface energy of the solid body σ_S.

9 Test report

The test report shall contain the following information:

a) a reference to this European Standard, i.e. EN 828;

b) complete description of the solid surface;

c) the test liquids used and their surface tensions as well as their polar and disperse proportions of the surface tension;

d) drop volumes and dosing rate used;

e) temperature conditions applied for measuring;

f) method (algorithm) of determining the contact angle of the drop;

g) number of drops per liquid;

h) average contact angle for the selected liquids and the polar proportion of the solid body surface energy σ_S^P, the disperse proportion σ_S^D and the total surface free energy of the solid body σ_S derived from that angle;

i) standard deviation of contact-angle measurement for each liquid;

j) statistical error of the surface free energy as well as of the polar and the disperse proportions of the surface free energy of the solid body, taking into consideration the statistical errors of the individual measurements;

k) all deviations from the described method and their potential influence on the results;

l) date when the test was performed.

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