SLOVENSKI STANDARD
oSIST prEN ISO 19403-2:2019

01-julij-2019

Barve in laki - Omočljivost - 2. del: Določevanje proste površinske energije površin trdnih teles z merjenjem stičnega kota (ISO 19403-2:2017)

Paints and varnishes - Wettability - Part 2: Determination of the surface free energy of solid surfaces by measuring the contact angle (ISO 19403-2:2017)

Beschichtungsstoffe - Benetzbarkeit - Teil 2: Bestimmung der freien Oberflächenenergie fester Oberflächen durch Messung des Kontaktwinkels (ISO 19403-2:2017)

Peintures et vernis - Mouillabilité - Partie 2: Détermination de l'énergie libre de surface des surfaces solides par la mesure de l'angle de contact (ISO 19403-2:2017)

Ta slovenski standard je istoveten z: **prEN ISO 19403-2**

ICS:

87.040	Barve in laki	Paints and varnishes

oSIST prEN ISO 19403-2:2019 **en,fr,de**

INTERNATIONAL STANDARD

ISO 19403-2

First edition
2017-06

Paints and varnishes — Wettability —

Part 2: Determination of the surface free energy of solid surfaces by measuring the contact angle

Peintures et vernis — Mouillabilité —

Partie 2: Détermination de l'énergie libre de surface des surfaces solides par la mesure de l'angle de contact

Reference number
ISO 19403-2:2017(E)

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Contents

Page

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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

A list of all parts in the ISO 19403 series can be found on the ISO website.

Paints and varnishes — Wettability —

Part 2: Determination of the surface free energy of solid surfaces by measuring the contact angle

1 Scope

This document specifies a test method to measure the contact angle for the determination of the surface free energy of a solid surface. The method can be applied for the characterization of substrates and coatings.

NOTE 1 For the determination of the surface free energy of polymers and coatings, either the method in accordance with Owens, Wendt, Rabel and Kaelble or the method in accordance with Wu is used preferably.

NOTE 2 The morphological and chemical homogeneity have an influence on the measuring results.

NOTE 3 The procedures indicated in this document are based on the state-of-the-art employing the drop projection method in penumbral shadow. Other methods are not excluded.

NOTE 4 Measuring the contact angle on powders is not part of this document. For further information, see the bibliography.

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 4618, *Paints and varnishes — Terms and definitions*

ISO 19403-1, *Paints and varnishes — Wettability — Part 1: Terminology and general principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and ISO 19403-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

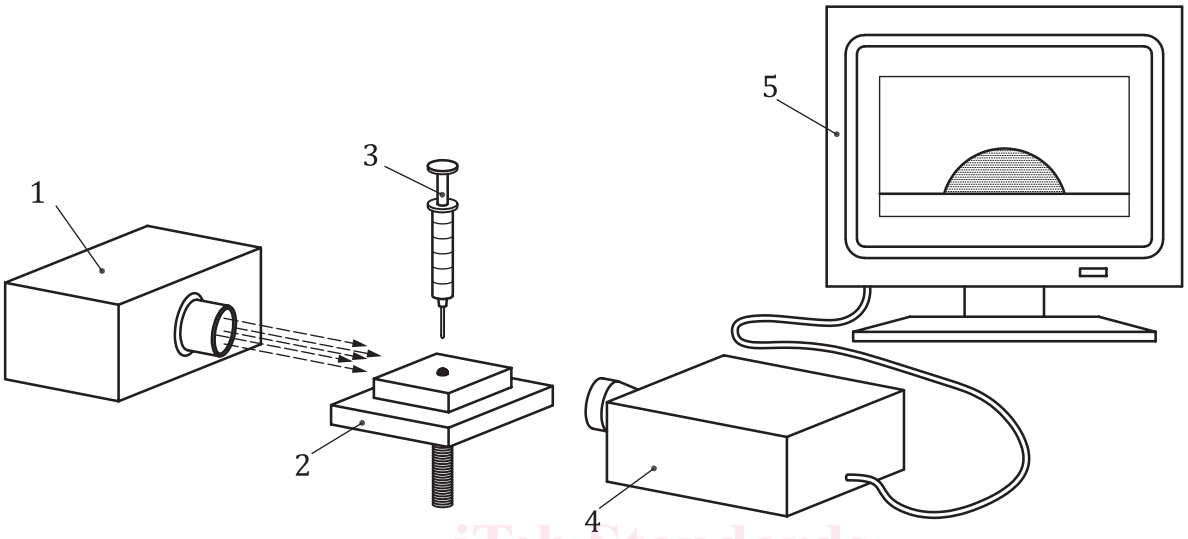
A minimum of three drops of at least two test liquids are dosed onto the flat surface of a test specimen. For every drop, the contact angle is measured. From the averaged contact angles of every liquid, their surface tensions, as well as their polar and dispersive fractions, the surface free energy of the solid is calculated by means of an appropriate model, divided into the polar and dispersive fractions.

5 Apparatus and materials

Ordinary laboratory apparatus, together with the following.

5.1 Contact angle measuring system.

Any state-of-the-art contact angle measuring device, preferably systems with digital image capture and analysis for measuring the contact angle. Figure 1 shows a schematic example of a contact angle measuring system.



Key

1 light source
2 specimen holder
3 graduaded micro syringe
4 optical system
5 screen

NOTE 1 The image capturing system is oriented in a way that the optimal image resolution ratio (ratio of width and height) can be used.

NOTE 2 The device used can differ from the schematic diagram in regard to light path and the arrangement of the components.

Figure 1 — Schematic diagram of a contact angle measuring system

5.2 Dosing unit.

The dosing unit makes it possible to precisely apply drops in the range of microlitres to the surface.

5.3 Test liquids.

At least two of the test liquids suggested in Table 1. The test liquids shall have at least “purity grade” for analysis. Water shall have a surface tension of at least 71,5 mN/m.

It is recommended to test the suitability of the liquids used in accordance with ISO 19403-3 or EN 14370 prior to measuring their surface tensions. For guidance, the values from the literature for the surface tension, σ_l, are indicated in Table 1. It is also possible to use an individually measured value of the surface tension as reference value. According to experience, the measured value should not deviate more than ±2 % from the value from the literature or the individually determined value.

The test liquids shall not physically or chemically affect the surface. The test liquids have a maximum of different polar and dispersive fractions of surface tension.

For at least one of the test liquids used, the polar fractions shall be larger than 0 mN/m (see Table 1).

In case only two test liquids are used, water and di-iodomethane are recommended. In case three liquids are used, ethylene glycol should be used additionally.

In case of high viscous test liquids like glycerol and ethylene glycol, a needle with higher inner diameter could be necessary to achieve the recommended dosing rate.

NOTE The values in Table 1 refer to 25 °C measuring temperature. For measuring under standard atmosphere (see 7.1.2), no significant deviations can be assumed.

Table 1 — Suggested test liquids

Test liquid	Surface tension σ_l mN/m	Dispersive fraction σ_l^d mN/m	Polar fraction σ_l^p mN/m	Source
Water	72,8	21,8	51,0	Reference [4]
Di-iodomethane[a]	50,8	50,8	0,0	Reference [4]
1,2-ethanediol (ethylene glycol)	47,7	30,9	16,8	Reference [4]
1,2,3-propanetriol (glycerol)	63,4	37,0	26,4	Reference [4]
Hexadecane	27,6	27,6	0,0	Reference [4]
1- bromo-naphthalene[b]	44,6	44,6	0,0	Reference [4]
Benzyl alcohol	38,9	29,0	9,9	Reference [5]
Decalin (isomer mixture)	30,6	30,6	0,0	Reference [4]
cis-Decalin	32,2	32,2	0,0	Reference [2]
trans-Decalin	29,9	29,9	0,0	Reference [2]

[a] Di-iodomethane is relative instable, yellowing after short time by splitting-off iodine.
Di-iodomethane swells and dissolves a lot of plastics and of organic coatings.
Di-iodomethane reacts with common metals (e.g. magnesium).

[b] 1-bromo-naphtalene reacts with common metals (e.g. magnesium).
1-bromo-naphtalene tends to swelling and dissolving of high-molecular compounds.

6 Sampling

Take a representative specimen of the substrate to be tested. The specimens shall not be contaminated before measuring.

Preferably, the specimen should have the minimum size of 10 cm × 10 cm.

See also Annex A.

7 Procedure

7.1 General for measuring on the horizontal drop

7.1.1 Setting up the contact angle measuring system

Choose the location of the contact angle measuring system so that it is not exposed to

— vibrations,

— intense air flows (e.g. caused by air conditioning), and

— intense exposure to light from outside (e.g. windows, bright lighting).

Align the contact angle measuring system horizontally.

7.1.2 Test conditions

Carry out the test at (23 ± 2) °C and a relative humidity of (50 ± 5) % (see ISO 3270) and make sure that all test media have this temperature.

7.1.3 Conditioning of the test panels

Condition the test panels at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) % for a minimum of 16 h prior to testing. Carry out the test immediately after conditioning.

7.2 Measurement

7.2.1 General

Place a preferably flat test specimen of the surface to be measured on the sample holder. Adjust the sample holder so that the surface of the test specimen is located in the lower half of the image and that it is horizontally aligned.

Fill the dosing system with the chosen liquid. Pay attention to fill without contamination or bubbles. Set up an image representation that is sufficient in regard to brightness and contrast (mind the specifications given by the manufacturer).

NOTE 1 If possible, set the light source of the contact angle measuring device so that the grey values within the drop close to the phase interface do not exceed the value 40 (referring to 256 grey value grades) and amount to a minimum of 170 on the outside of the drop.

NOTE 2 It can be reasonable to test the modes of operation of the optical components by means of two-dimensional images of drops. Such reference images are commercially available.

Move the needle to the upper margin of the image and bring into focus. Set up the zoom of the contact angle measuring device so that the width of the contour of the drop takes up two thirds of the width of the image.

7.2.2 Static method

Position the dosing needle approximately 3 mm to 6 mm above the surface of the test specimen. Dose the drop so that the volume of the drop is between 2 µl and 6 µl, depending on the chosen liquid (for di-iodomethane between 1 µl and 3 µl).

Apply one drop of the test liquid to the surface (see Figure 2).

NOTE 1 The contact between the drop and the solid surface can be achieved by putting down by means of the needle or by picking up by means of the specimen table.

