

SLOVENSKI STANDARD oSIST prEN ISO 19403-2:2023

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Barve in laki - Omočljivost - 2. del: Določevanje proste površinske energije površin trdnih teles z merjenjem stičnega kota (ISO/DIS 19403-2:2023)

Paints and varnishes - Wettability - Part 2: Determination of the surface free energy of solid surfaces by measuring the contact angle (ISO/DIS 19403-2:2023)

Beschichtungsstoffe - Benetzbarkeit - Teil 2: Bestimmung der freien Oberflächenenergie fester Oberflächen durch Messung des Kontaktwinkels (ISO/DIS 19403-2:2023)

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/DIS 19403-2:2023)

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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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 19403-2:2017), which has been technically revised.

The main changes are as follows: dards/sist/07d2e028-2460-4475-9490-c4e1f9de0830/osist-pren-iso-19403-2-2023

- the minimum size of the text samples has been changed to 4 cm × 4 cm;
- in <u>5.3</u>, the use of ethylene glycol as test liquid has been deleted;
- in <u>7.2.1</u>, the information on the camera tilt angle has been added;
- the text has been editorially revised and the normative references have been updated.

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

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.

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 s://standards.iteh.ai)

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 — Vocabulary

ISO 19403-1, Paints and varnishes — Wettability — Part 1: Vocabulary 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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Principle

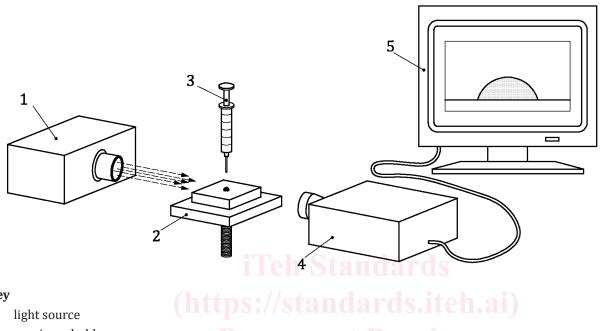
A minimum of three drops of at least two test liquids are dosed onto the flat surface of a test specimen. For every drop, the contact angle is measured. From the averaged contact angles of every liquid, their surface tensions, as well as their polar and dispersive fractions, the surface free energy of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

Apparatus and materials 5

Ordinary laboratory apparatus, together with the following.

Contact angle measuring system. 5.1

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

5

- 1
- 2 specimen holder
- 3 graduated micro syringe
- 4 optical system
- screen

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.

Test liquids. 5.3

At least two of the test liquids suggested in <u>Table 1</u>. The test liquids shall have at least "purity grade" for analysis. Water shall have a surface tension of at least 71,5 mN/m at standard climate, i.e. (23 ± 2) °C and (50 ± 5) % relative humidity.

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 <u>Table 1</u>. 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 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 <u>Table 1</u> refer to 25 °C measuring temperature. For measuring under standard atmosphere (see <u>7.1.2</u>), no significant deviations can be assumed.

1							
Test liquid	Surface tension	Dispersive fraction	Polar fraction	Source			
	$\sigma_{ m l}$	$\sigma_{ m l}^{ m d}$	$\sigma^{ m p}_{ m l}$				
	mN/m	mN/m	mN/m				
Water	72,8	21,8	51,0	Reference [<u>4</u>]			
Di-iodomethane ^a	50,8	50,8	0,0	Reference [<u>4</u>]			
1,2-ethanediol (ethylene glycol)	47,7	30,9	16,8	Reference [<u>4</u>]			
1,2,3-propanetriol (glycerol)	63,4 Teh	tan 37,0 rds	26,4	Reference [<u>4</u>]			
Hexadecane	27,6	27,6	0,0	Reference [<u>4</u>]			
1- bromo- naphthalene ^b	44,6	44,6	0,0	Reference [<u>4</u>]			
Benzyl alcohol	38,9	29,0	9,9	Reference [<u>5</u>]			
Decalin (isomer mixture)	30,6 oSIST prEM	30,6 (ISO 19403-2:2023	0,0	Reference [<u>4</u>]			
ndar cis-Decalin atal	g/standarc32,2st/07d2e02	8-2460- 32,2 -9490-c	4e1f9d 0,0 330/osi	st-pReference [2])3-2			
trans-Decalin	29.9	29.9	0.0	Reference [2]			

Table 1 — Suggested test liquids

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 $4 \text{ cm} \times 4 \text{ cm}$.

See also Annex A.

a Di-iodomethane is relatively 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.

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

If not otherwise agreed, 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.

NOTE Other conditioning parameters can be necessary, if the surface of the test sample changes its chemical state at $50\,\%$ relative humidity.

7.2 Measurement

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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. log/standards/sist/07d2e028-2460-4475-9490-c4e199de0830/osist-pren-iso-19403-2-2023

Tilt the camera from back to front to be able to see the contact point. The camera tilt angle should be between 0° to 4° . The tilting helps seeing the reflection of the drop and detecting the baseline, accurately.

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 twodimensional 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.