



**International
Standard**

ISO 19403-2

**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*

**Second edition
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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 document 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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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.

<https://standards.iteh.ai/catalog/standards/iso/d36ff26b-df42-4d3e-a69d-12b91ef090c7/iso-19403-2-2024>

The main changes are as follows:

- the minimum size of the text samples has been changed to 4 cm × 4 cm;
- definition [3.1](#) on “recently advanced contact angle” has been added;
- the use of ethylene glycol as test liquid has been deleted;
- in [7.2.1](#), the information on the camera tilt angle has been added;
- 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, it is preferred to use either the method according to Owens, Wendt, Rabel and Kaelble [3],[4],[5] or the method according to Wu.

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

Measuring the contact angle on powders is not part of this document.

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 — Vocabulary* ISO 19403-2:2024

ISO 19403-1:2022, *Paints and varnishes — Wettability — Part 1: Vocabulary and general principles* ISO 19403-2:2024

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618, ISO 19403-1 and the following 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/>

3.1

recently advanced contact angle

contact angle of a droplet at rest after the three-phase contact line has advanced over a previously dry surface

Note 1 to entry: The recently advanced contact angle is thermodynamically not defined.

4 Principle

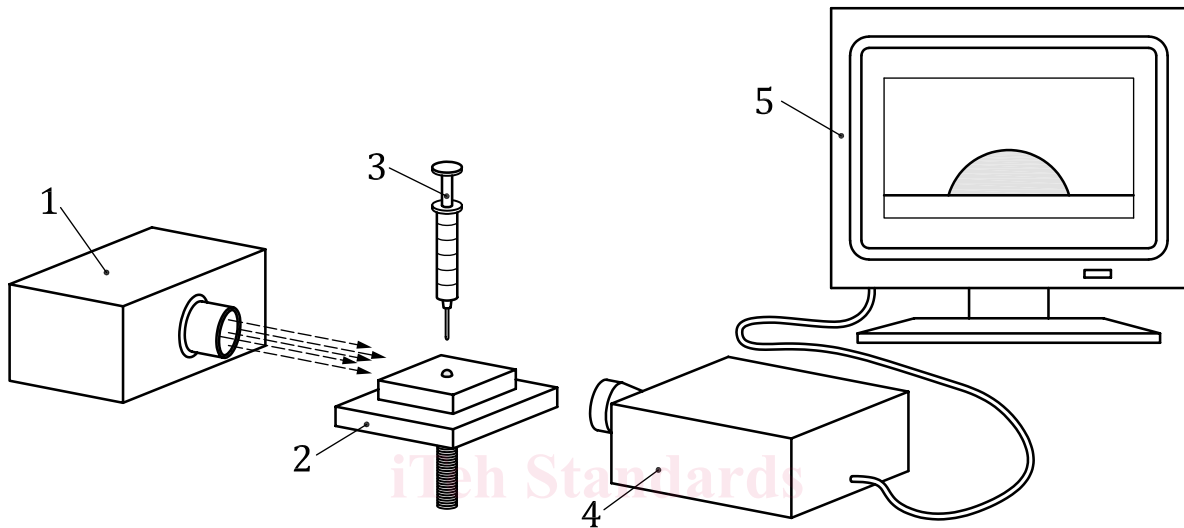
A minimum of three drops of at least two test liquids are dosed onto the flat surface of a test specimen. For every drop, the contact angle is measured. From the averaged contact angles of every liquid, their surface

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 shall be used.

5.1 Contact angle measuring system, i.e. any state-of-the-art contact angle measuring device, preferably 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 graduated micro syringe or pressure based liquid needle dosing system
- 4 optical system

5 computer 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, which makes it possible to precisely apply drops in the range of microlitres to the surface. It should facilitate a drop deposition guaranteeing that one of the contact angles (as described in ISO 19403-1:2022, 3.1.9) can be reproducibly assessed. The recently advanced contact angle after liquid needle drop deposition has the least human influence and is thus recommended.

5.3 Test liquids, including at least two of those 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 at standard climate, i.e. $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity.

It is recommended to measure the surface tension of the liquids to be used in accordance with ISO 19403-3. 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 a reference value. According to

experience, the measured value should not deviate more than $\pm 2\%$ from the value specified in the literature or the individually determined value.

The test liquids shall not physically or chemically affect the surface. The test liquids shall be chosen so that they have a difference as large as possible in the polar and dispersive parts of the 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.

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_1 mN/m	Dispersive fraction σ_1^d mN/m	Polar fraction σ_1^p mN/m	Source
Water	72,8	21,8	51,0	Reference [6]
Di-iodomethane ^a	50,8	50,8	0,0	Reference [6]
1,2,3-propanetriol (glycerol)	63,4	37,0	26,4	Reference [6]
Hexadecane	27,6	27,6	0,0	Reference [6]
1-bromo- naphthalene ^b	44,6	44,6	0,0	Reference [6]
Benzyl alcohol	38,9	29,0	9,9	Reference [6]
Decalin (isomer mixture)	30,6	30,6	0,0	Reference [6]
cis-Decalin	32,2	32,2	0,0	ISO 19403-3
trans-Decalin	29,9	29,9	0,0	ISO 19403-3

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

^b 1-bromo-naphthalene reacts with common metals (e.g. magnesium).
1-bromo-naphthalene tends to swell and dissolve 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 4 cm × 4 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

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

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 is horizontally aligned.

Tilt the camera from back to front to see the contact point. The camera tilt angle should be between 0° to 4°. The tilting allows the reflection of the drop to be more visible and the baseline to be more easily detected, 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 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 typically available from the instrument manufacturers.

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.

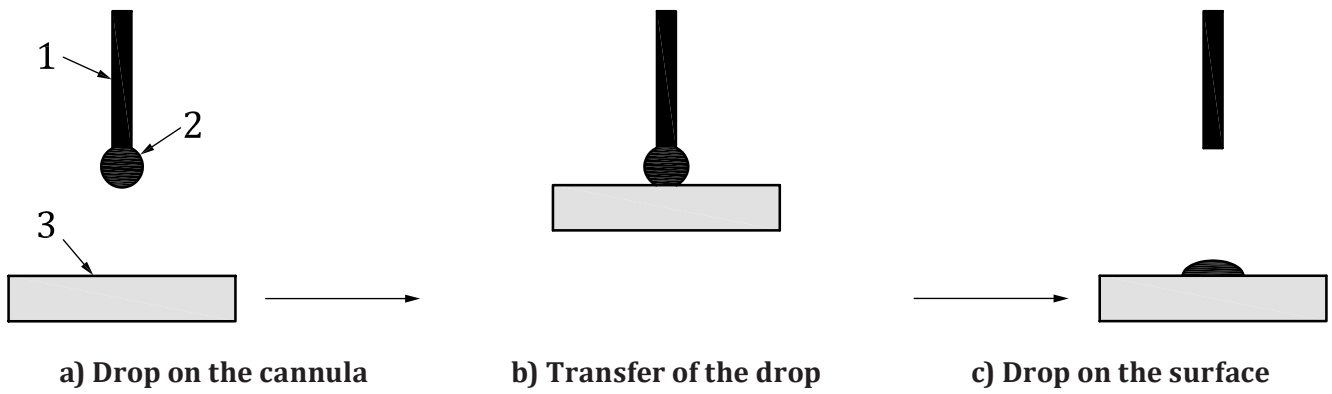
Prior to and during measuring, no mutual transformation of materials between the test liquid and the surface shall occur.

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 diiodomethane between 1 µl and 3 µl).

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

NOTE 1 Contact between the drop and the solid surface can be achieved either by means of the needle “putting down” the drop or by the specimen table rising to “pick up” the drop.



Key

- 1 cannula
- 2 test liquid
- 3 surface of test specimen

Figure 2 — Putting down or picking up the drop

Align the baseline so that it runs through the three-phase points of the drop.

Start measuring the contact angle within 5 s after finishing dosing.

The static method should only be used when the test liquids do not change the surface of the test sample.

NOTE 2 On low energy surfaces, it is possible that the drop does not detach completely from the needle.

7.2.3 Dynamic method (progressive contact angle)

Choose the distance between the dosing needle and the surface so that the influence on the expected contour of the drop is as slight as possible.

NOTE 1 As a first guidance for the distance between the needle and the surface of the test specimen, the one-and-a-half-times diameter of the needle can be used.

Especially for low contact angles, minimize the pull-up of liquid on the needle, if necessary, by using a poorly wettable material of the needle.

Choose the slowest dosing speed possible, so that the contact angle of the drop is as close as possible to the thermodynamic equilibrium contact angle.

NOTE 2 Typical dosing rates are in the range of 10 µl/min.

NOTE 3 It is common to start measuring only after a dosing of 3 µl minimal volume.

NOTE 4 Due to the limited image section, it is not ideal to measure contact angles below 10° by means of the dynamic method. For such cases, captive bubble method can be used.

Align the baseline so that it runs through the three-phase points of the drop.

Start measuring the contact angle immediately after dosing the minimal volume. Record the measuring values as function of time. The spreading process of the drop in a first step should be observed in order to then determine the time at which the contact angle is measured after the drop has settled.

NOTE 5 In comparison to the measuring method of the static contact angle, interfering transformations of material often occur less in regard to the dynamic contact angle.

7.2.4 Determination of the contact angle

Determine the contact angle by means of that numerical method, which describes best the contour of the drop. The contact angle is then determined according to its definition (see ISO 19403-1:2022, 3.1.9).

Depending on how the individual drops of liquid perform on different solid surfaces, different methods for the determination of the contact angle shall be used [e.g. circle equation for contact angles below 20°, conic section equation for contact angles between 20° and 110°, polynomial equation or Young-Laplace equation for contact angles above 110° (see notes in [Annex A](#))]. For the dynamic method, the recommendations given by the manufacturer should be noted; normally, the polynomial method is advantageous, because of the asymmetrical nature of the drop.

Measure on a minimum of three different measuring points on the test specimen in order to obtain sufficient information on the homogeneity of a test specimen. Previously wetted positions shall not be used. Arguable readings which can be caused by dust, contaminations, etc. shall not be included in the calculation of the mean value.

Repeat the measuring with at least one more liquid, which was selected in accordance with the criteria indicated in [5.3](#).

8 Evaluation

8.1 General

The value of the contact angle for every liquid is calculated as the arithmetic mean value of the measuring values. It is recommended to use the dynamic advanced contact angle or the recently advanced contact angle as well-defined contact angles. In any case, the contact angles used to calculate the surface free energy shall be reported in the surface free energy calculation.

For the determination of the surface free energy, the standard deviation for the static method should not be more than 3° and for the dynamic method not more than 5°.

In case the standard deviation is more than 5° for the dynamic method, the individual measuring values shall be checked. In order to improve the reliability, the mean value can be calculated for smaller periods.

For the calculation of the surface free energy, the Owens-Wendt-Rabel-Kaelble method is preferably used. For low-energy surfaces, the calculation in accordance with Wu and equation of state^[11] can be necessary in regard to the range of a surface free energy of (20 ± 2) mJ/m² or lower.

It is recommended for both methods to obtain the standard deviation of the surface free energy and their components in accordance with the Gauss error propagation (see References [\[9\]](#) and [\[10\]](#)).

8.2 Owens-Wendt-Rabel-Kaelble method (OWRK method)

For evaluation, the polar and dispersive fractions of the test liquids are used (see [Table 1](#)). For the calculation of the surface free energy, [Formula \(1\)](#):

$$\frac{(1 + \cos\theta) \cdot \sigma_1}{2\sqrt{\sigma_1^d}} \quad (1)$$

is plotted for every individual test liquid against [Formula \(2\)](#):

$$\sqrt{\frac{\sigma_1^p}{\sigma_1^d}} \quad (2)$$