
Paints and varnishes — Wettability —

Part 5:

**Determination of the polar and
dispersive fractions of the surface
tension of liquids from contact
angles measurements on a solid with
only a disperse contribution to its
surface energy**

Peintures et vernis — Mouillabilité —

Partie 5: Détermination des fractions polaires et disperses de la tension superficielle des liquides à partir de l'angle de contact avec un solide n'ayant qu'une contribution de dispersion à son énergie de surface



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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 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 5:

Determination of the polar and dispersive fractions of the surface tension of liquids from contact angles measurements on a solid with only a disperse contribution to its surface energy

1 Scope

This document specifies a test method to determine the polar and dispersive fractions of the surface tension of liquids by optical methods. The method can be applied for the characterization of liquid coating materials.

The applicability can be restricted for liquids with non-Newtonian rheology¹⁾.

This document assumes that the information of surface tension of the liquid to be tested and the surface free energy of the dispersive reference solids is known.

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 1409, *Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of surface tension by the ring method*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

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

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 19403-3, *Paints and varnishes — Wettability — Part 3: Determination of the surface tension of liquids using the pendant drop method*

EN 14370, *Surface active agents — Determination of surface tension*

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>

1) This term is defined in DIN 1342-1.

4 Principle

Step 1: The surface tension of the liquid to be tested is determined in accordance with ISO 19403-3, EN 14370 or ISO 1409.

Step 2: The surface free energy of a reference solid without polar fraction of the surface free energy is determined in accordance with ISO 19403-2.

Step 3: Measurement of the contact angle between the reference solid and the liquid to be tested is carried out in accordance with ISO 19403-2.

Step 4: The dispersive fraction of the surface tension of the liquid is calculated in accordance with Owens, Wendt, Rabel and Kaelble (OWRK) or in accordance with Wu.

Step 5: The polar fraction of the surface tension of the liquid is calculated from the dispersive fraction of the surface tension and the surface tension measured in step 1.

5 Reference solid

Sufficiently, chemically and topologically homogenous dispersive solid, e.g. made of paraffin or PTFE, shall be used.

NOTE For the application of this document, surfaces of solids with a polar fraction of $<0,5 \text{ mJ/m}^2$ are sufficiently dispersive.

Sufficiently homogenous PTFE reference surfaces preferably have a surface free energy of $(18,5 \pm 0,5) \text{ mJ/m}^2$ in accordance with the OWRK method, whereas locally measured surface free energies on the reference surface for the determination of the standard deviation (see 8.1) are measured in accordance with 7.3. For the PTFE surfaces, as well as the paraffin surfaces, precise reference objects with a mean roughness value $R_a^{2)}$ of less than $0,3 \mu\text{m}$ are to be preferred. The paraffin reference surfaces preferably have a surface free energy of $(25,5 \pm 0,5) \text{ mJ/m}^2$ under the same conditions as the PTFE reference surfaces.

6 Sampling

Take a representative sample of the liquid to be tested in accordance with ISO 15528.

7 Procedure

7.1 Test conditions

Carry out the test at $(23 \pm 2) ^\circ\text{C}$ and a relative humidity of $(50 \pm 5) \%$ (see ISO 3270) and make sure that all test media have this temperature.

7.2 Determination of the surface tension of the liquid to be tested

Measure the surface tension of the liquid to be tested in accordance with ISO 19403-3, EN 14370 or ISO 1409.

2) The roughness value is defined in ISO 4288 and ISO 25178-2.

7.3 Determination of the surface free energy of the reference solid

If the surface free energy of the reference solid is unknown, determine it in accordance with ISO 19403-2 using at least 10 drops of each of at least three test liquids indicated in ISO 19403-2:2017, Table 1. Calculate the surface free energy in accordance with ISO 19403-2:2017, 8.2 or 8.3.

NOTE Since a reference solid with predominantly dispersive interactions is used, it can be assumed that the dispersive fraction of the surface tension of the liquid can be determined from a single contact angle value.

7.4 Determination of the contact angle of the liquid to be tested on the reference solid

Clean, dry and condition the reference solid in accordance with ISO 19403-2. Measure the contact angle between the reference solid and the liquid to be tested in accordance with ISO 19403-2. Use at least 10 drops for this. Use the same method (static or dynamic) as in 7.3 for measurement of the contact angle.

8 Evaluation

8.1 General

The standard deviations should not exceed the following values:

- for the surface tension of the tested surface: ± 1 %;
- for the surface free energy of the reference solid: preferably $0,3 \text{ mJ/m}^2$, not exceeding $0,5 \text{ mJ/m}^2$;
- for the contact angles of the tested liquid on the reference solid: for the static method 1° and for the dynamic method 3° .

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

For reference surfaces with a surface free energy $>20 \text{ mJ/m}^2$, calculate the dispersive fraction of the surface tension of the liquid, σ_1^d , in accordance with [Formula \(1\)](#):

$$\sigma_1^d = \frac{(1 + \cos \theta)^2 \cdot \sigma_1^2}{4\sigma_s} \quad (1)$$

where

θ is the mean value of the measured contact angles between the tested liquid and the reference solid;

σ_1 is the surface tension of the tested liquid, measured in accordance with [7.2](#);

σ_s is the surface free energy of the reference solid, measured in accordance with [7.3](#).

8.3 Wu method

For reference surfaces with a surface free energy <20 mJ/m², calculate the dispersive fraction of the surface tension of the liquid, σ_1^d , in accordance with [Formula \(2\)](#):

$$\sigma_1^d = \frac{\sigma_1 \sigma_s^d (1 + \cos \theta)}{4\sigma_s^d - \sigma_1 (1 + \cos \theta)} \quad (2)$$

where

θ is the mean value of the measured contact angles between the tested liquid and the reference solid;

σ_1 is the surface tension of the tested liquid, measured in accordance with [7.2](#);

σ_s^d is the dispersive fraction of the surface energy of the reference solid.

8.4 Calculation of the polar fraction of the surface tension of the liquid

Calculate the polar fraction of the surface tension of the liquid, σ_1^p , in accordance with [Formula \(3\)](#):

$$\sigma_1^p = \sigma_1 - \sigma_1^d \quad (3)$$

where

σ_1 is the surface tension of the tested liquid, measured in accordance with [7.2](#);

σ_1^d is the dispersive fraction of the surface tension of the tested liquid.

9 Precision

At the time of publication, information on precision is not available.

10 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the tested product;
- b) a reference to this document, i.e. ISO 19403-5;
- c) the surface tension of the liquid to be tested including all information in accordance with the test report of the method used;
- d) the surface free energy of the reference solid including all information in accordance with the test report of the method used or reference and material and manufacturer, cleaning and preparation method of the reference solid, if applicable;
- e) for the determination of the polar and dispersive fractions of the surface tension of the liquid:
 - 1) the information on whether it was measured statically or dynamically,
 - 2) the used drop volumes or volume range and dosing speed,

- 3) the method with which the contact angle of the drop was obtained,
 - 4) the amount of drops,
 - 5) the amount of measuring points per drop,
 - 6) the result of the measurement of the contact angle (arithmetic mean value and standard deviation),
 - 7) the result of the calculation of the polar and dispersive fraction of the surface tension of the liquid,
 - 8) the information on whether it was calculated in accordance with Owens-Wendt-Rabel-Kaelble or Wu;
- f) the random error for the calculation of the surface free energy of the reference solid as well as the polar and dispersive fractions of the surface tension of the liquid;
 - g) all deviations from the specified method and their possible influences on the results;
 - h) any unusual observation during the test;
 - i) the type of device;
 - j) the name of the test person;
 - k) the date of the test.

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