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Paints and varnishes — Wettability —

Part 4: Determination of the polar and dispersive fractions of the surface tension of liquids from an interfacial tension

Peintures et vernis — Mouillabilité —
Partie 4: Détermination des fractions polaires et disperses de la tension de surface des liquides à partir de la tension interfaciable

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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 4: Determination of the polar and dispersive fractions of the surface tension of liquids from an interfacial tension

1 Scope

This document specifies a test method to determine the polar and dispersive fraction of the surface tension of liquids with optical methods. The method can be applied for the characterization of liquid coating materials, especially when drying effects occur during measurement. The applicability can be restricted for liquids with non-Newtonian rheology[1)].

This document assumes that the information of surface tension of the liquid to be tested, as well as at least one suitable reference liquid, 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 19403-3, *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

4 Principle

One drop of the respective liquid to be tested is reproduced within an optical cell, which is completely filled with a reference liquid, hanging from or ascending from a needle. The reproduced drop shall deviate significantly from the spherical shape due to its mass difference from the reference liquid.

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

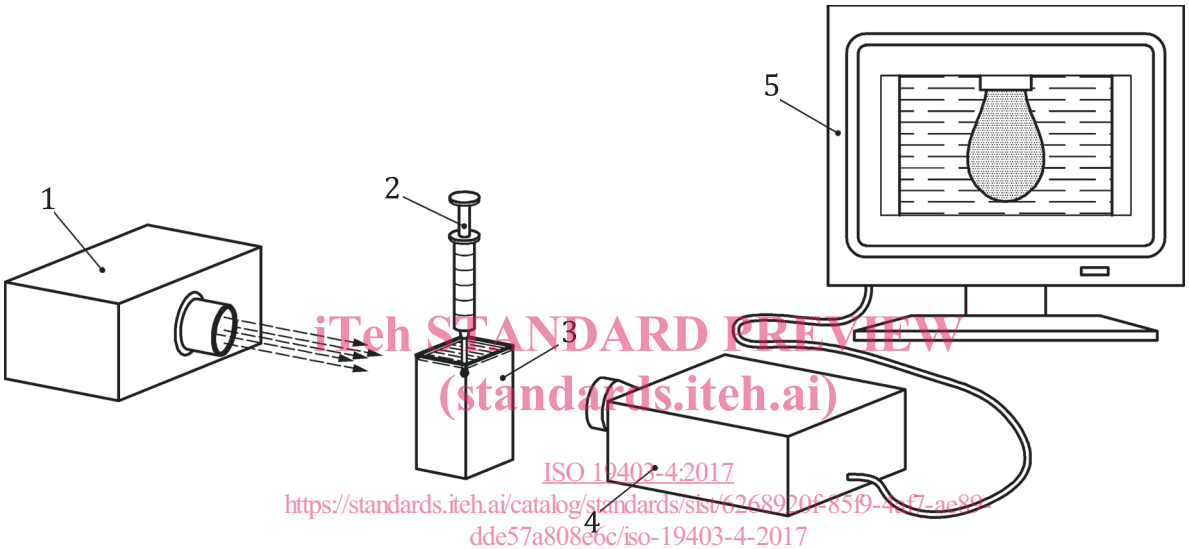
The interfacial tension is calculated from the shape of the reproduced pendant or ascending drop in accordance with the Young-Laplace equation. The polar and dispersive fraction of the surface tension of the liquid to be tested can be determined from the obtained interfacial tension and the known surface tensions of the liquid to be tested and the reference liquid.

5 Apparatus and materials

Ordinary laboratory apparatus, together with the following.

5.1 Drop contour analysis system, for measurement of the surface tension of pendant drops.

Any state-of-the-art drop contour analysis system with digital image capture and analysis. Figure 1 and Figure 2 show a schematic example of a drop contour analysis system.



Key

1 light source
2 dosing unit with graduated microsyringe
3 optical cell
4 image taking and analysing unit
5 screen

Figure 1 — Drop contour analysis system with pendant drop

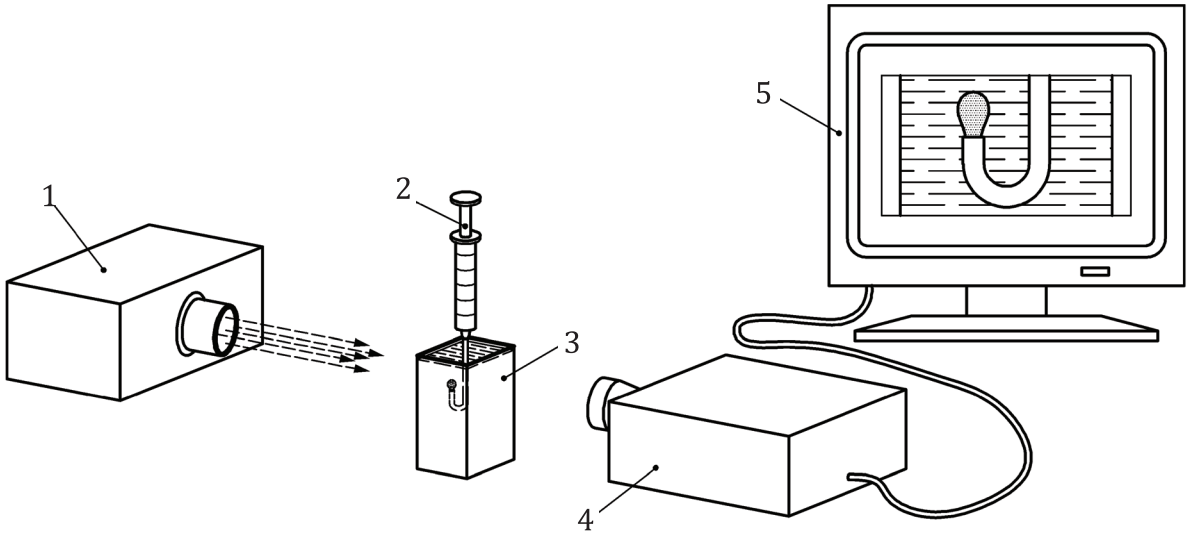


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Figure 2 — Drop contour analysis system with ascending drop

The image taking system should be oriented in a way that the optimal image resolution ratio (ratio of width and height) can be used.

NOTE The device used can differ from the schematic diagram in regard to light path and the set-up of the components.

5.2 Dosing unit.

The dosing unit makes it possible to dose a pendant liquid drop, which deviates significantly from the spherical shape due to its own mass, on a circular-cylindrical needle with constant wall thickness within the detection area of the camera.

The needle used can be straight or bent in a J-shape.

J-shaped needles, whose free end points upwards are necessary when the density of the liquid to be tested, is lower than the density of the reference liquid.

For the measurement of the surface tension on the pendant or ascending drop, usually a larger outside diameter of the needle is needed than for the measurement of the contact angle on the horizontal drop. The outside diameters of the needles used shall be in the range between 0,5 mm and 2,5 mm. The ideal outside diameter of the needle depends on the relationship between the interfacial tension, σ_{LR}, and the density difference, $\Delta\rho_{LR} = \rho_L - \rho_R$, of the liquid to be tested, ρ_L, and the reference liquid, ρ_R. The higher the quotient, $\sigma_{LR}/\Delta\rho_{LR}$, the larger should be the outside diameter of the needle.

5.3 Optical cell.

The optical cell used shall have plane and side walls running parallel to each other. Through these sides made of optically impeccable clear-transparent materials (e.g. made of glass or insoluble, optically useable plastic), the observation of the formed drop is carried out in a way so that a representation

without optical errors is possible. In addition, the inner and outer walls of the optical cell shall be free of contaminations (e.g. finger prints, adhering particles and surfactants).

5.4 Reference liquids.

The reference liquid to be used and the liquid to be tested shall not be miscible and shall be able to form a meniscus. Reference liquids shall be chemically homogenous, strictly dispersive and colourless with a melting point lower than 20 °C. Preferably, liquid hydrocarbons or perfluorohydrocarbons free from polar contaminations can be used. Among the hydrocarbons, especially *n*-decane, *n*-dodecane, *n*-tetradecane and *n*-hexadecane are suitable as reference liquids. As an alternative, also perfluoroalkanes, such as *n*-perfluorohexane and *n*-perfluorooctane, can be used as test liquids. The perfluoroalkanes can be used as reference liquids if the solubility of the liquid to be tested is too high compared to the hydrocarbons.

NOTE The viscosity of the mentioned reference liquids at 23 °C is in the range of that of water. This is of advantage for the setting of the equilibrium.

6 Sampling

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

7 Procedure

7.1 General

7.1.1 Setting up the drop contour analysis device

Choose the location of the drop contour analysis device, 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 drop contour analysis device horizontally.

Obtain the value of the local acceleration of gravity of the installation location and enter in the respective position of the manufacturer software.

7.1.2 Test conditions

Carry out the test at (23 ± 2) °C (see ISO 3270) and make sure that all test media have this temperature.

7.1.3 Cleaning and conditioning of the reference liquid

The measurable interfacial tension compared to water shall be at least 52 mN/m at 23 °C after the cleaning of the recommended *n*-alkanes (see 5.4)[5].

Especially, the residual content of ketones in the *n*-alkanes can be removed by the following cleaning procedure. The cleaning of the hydrocarbons used as reference liquids can be carried out in chromatography columns with a minimum length of 60 cm, which are filled with silica gel (e.g. particle size mesh 60). The silica gel used shall have polar surfaces in order to sufficiently absorb the polar contaminations that originate from the production process during passing through the hydrocarbons. Cleaning by distillation is also possible.

The cleaned *n*-alkanes shall be stored in light-proof glass bottles in the refrigerator at a maximum temperature of 4 °C.

Perfluorohydrocarbons need not be cleaned beforehand, since after the normal industrial production process (electrofluorination) of these liquids, no measurable content of polar contaminations is to be expected. An interfacial tension compared to water at 23 °C for the preferably used perfluoroalkanes (*n*-perfluorohexane and *n*-perfluorooctane) of at least 54 mN/m at 23 °C should be observed[6].

7.2 Determination of the interfacial tension of the liquid

7.2.1 Preparations

In case it is not given, obtain the density of the liquid to be tested as well as the density of the reference liquid and calculate the density difference.

Fill the optical cell with the reference liquid to about 80 %.

Determine the outside diameter of the needle to ±0,005 mm, e.g. by means of a microscope or a micrometer in accordance with ISO 2808.

It shall be taken into account that especially in the case of J-shaped dosing needles, the end of the needle runs out circular-cylindrical.

Fill the dosing system with the liquid to be tested. Pay attention to fill without contamination and bubbles.

Move the needle to the upper margin of the image in case of a pendant drop or to the lower margin of the image in case of an ascending drop and bring the edges of the needle into focus. Set up the zoom of the drop contour analysis device so that the diameter of the needle takes up at least one eighth of the width of the image; refocus, if necessary.

Set up an image representation that is sufficient in regard to brightness and contrast (observe the information given by the manufacturer). If possible, set the light source of the drop contour analysis 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 between 170 and 200 on the outside of the drop.

7.2.2 Procedure

Produce a preferably large pendant or ascending drop within the optical cell. The drop shall touch neither the boundaries of the optical cell nor the meniscus of the ambient liquid phase in the upper area of the optical cell.

In order to avoid interferences of the pendant or ascending drop, as well as image blurring due to convection currents, equilibrium in the optical cell shall be awaited. To achieve complete equilibrium for the interfacial tension, additional waiting time may be necessary depending on the viscosity and solubility of the liquid to be tested. The exact time until (thermodynamic) equilibrium depends on the size of the optical cell, the ambient thermostatic control and the properties of the liquids themselves.

In case single parts of the liquid to be tested dissolve in the reference liquid, the reference liquid shall be changed. In this case, perfluoroalkanes can be used as reference. A further possibility is the measurement on dispersive reference surfaces (see ISO 19403-5).

After final focusing/zooming, carry out a length calibration of the imaging device. Update the lighting, if necessary.

Do not change the zoom and focus after this determination of scale.

Take an image of the pendant or ascending drop.

For traceability reasons, the original image should be saved.

Repeat measurement on at least two more drops. Calculate the mean value and the standard deviation of the interfacial tension from all single measurement values.

