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

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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 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.

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 19403-1:2022, *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, 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.
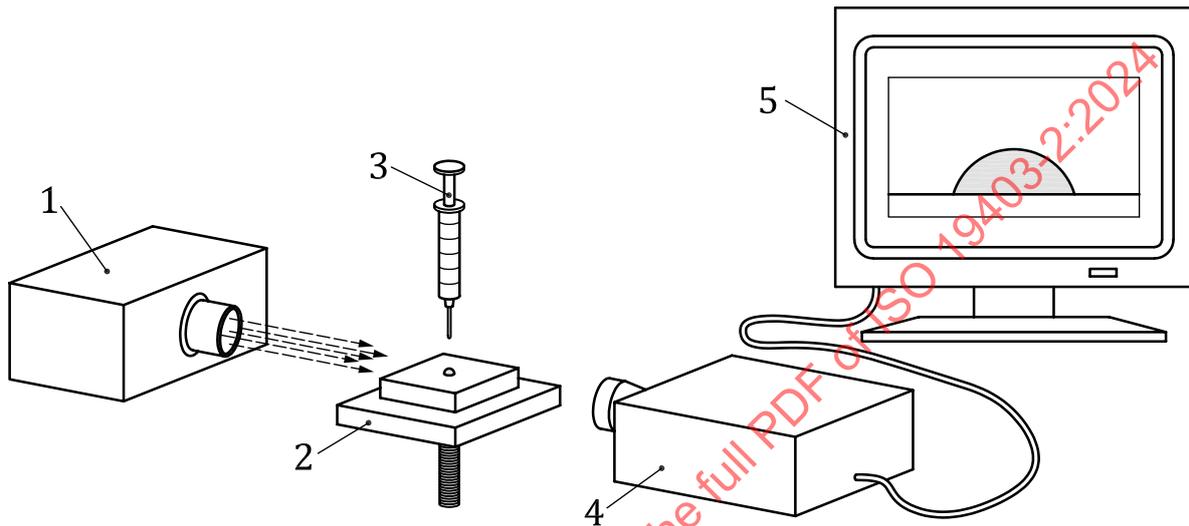
Key

1   light source
2   specimen holder
3   graduated micro syringe or pressure based liquid needle dosing system
4   optical system
5   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 ± 2) °C and (50 ± 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 ±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_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 [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-naphtalene reacts with common metals (e.g. magnesium).

  1-bromo-naphtalene 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 di-iodomethane 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.

a) Drop on the cannula   **b) Transfer of the drop**   **c) Drop on the surface**
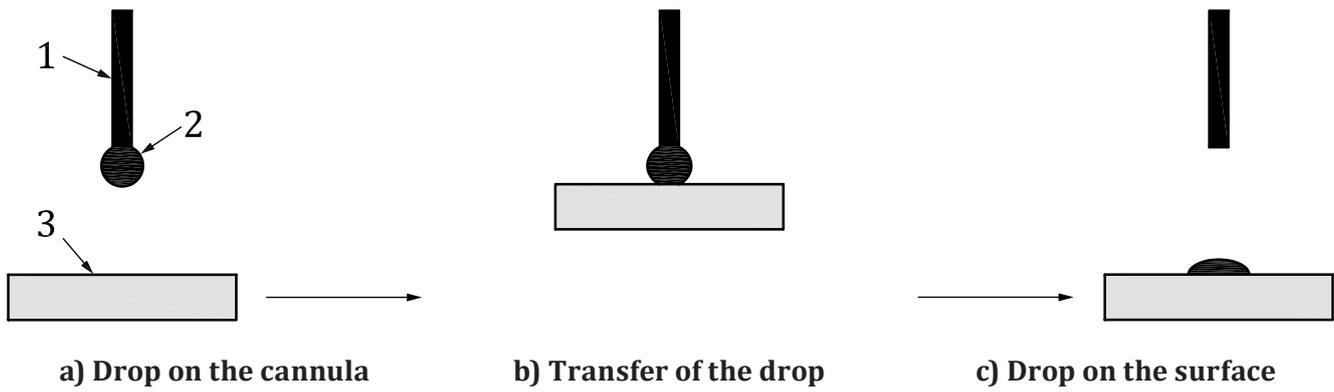
Key
1   cannula
2   test liquid
3   surface of test specimen

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/m^2 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):

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

is plotted for every individual test liquid against Formula (2):

$$\sqrt{\frac{\sigma_l^p}{\sigma_l^d}} \tag{2}$$

where

θ is the mean value of the measured contact angle for the respective liquid;

σ_l is the surface tension of the respective liquid;

σ_l^p and σ_l^d are the polar and dispersive fractions of the surface tension.

The values for $\sqrt{\dfrac{\sigma_l^p}{\sigma_l^d}}$ (= x) and $\dfrac{\sigma_l}{2\sqrt{\sigma_l^d}}$ [= $y/(1+\cos\theta)$] can be taken from Table 2.

Table 2 — Calculated values for insertion into the linear equation

Liquid	$\sqrt{\dfrac{\sigma_l^p}{\sigma_l^d}}$ = x	$\dfrac{\sigma_l}{2\sqrt{\sigma_l^d}}$ = $y/(1+\cos\theta)$ mN/m
Water	1,529 5	7,796 0
Di-iodomethane	0,000 0	3,563 7
1,2,3-propanetriol (glycerol)	0,844 7	5,211 4
Hexadecane	0,000 0	2,626 8
1-bromo-naphthalene	0,000 0	3,339 2
Benzyl alcohol	0,584 3	3,611 8
Decalin (isomer mixture)	0,000 0	2,765 9
cis-Decalin	0,000 0	2,837 3
trans-Decalin	0,000 0	2,734 0

The value from the middle column is inserted for x. The value from the right column is multiplied with the value of $(1 + \cos\theta)$ for the respective liquid and the result is inserted for y. Subsequently, a linear regression is done in order to obtain the gradient m and the ordinate section b of the straight line. From the square of the gradient, the polar fraction of the free surface energy of the solid σ_s^p is calculated and from the square of the ordinate section b, the disperse fraction σ_s^d.

NOTE   Alternatively, the statistics methods offered by the manufacturers of the devices can be used.

8.3 Wu method

The polar and dispersive fractions of the surface free energy are numerically obtained by means of the nonlinear regression of Formula (3):

$$\sigma_l(1+\cos\theta) = 4\left(\frac{\sigma_s^d \cdot \sigma_l^d}{\sigma_s^d + \sigma_l^d} + \frac{\sigma_s^p \cdot \sigma_l^p}{\sigma_s^p + \sigma_l^p}\right) \qquad (3)$$

NOTE 1   It is preferable to use an error square minimization in accordance with Gauss for the numerical calculation (see Reference [8]).

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

NOTE 2   When using more than two liquids, the quality of the regression can be indicated by means of a regression coefficient.

9 Precision

9.1 General

For the determination of the precision, an interlaboratory test with seven participants and three test specimens was conducted. The test specimens were conditioned in accordance with the specifications in 7.1.3. All participants used water, di-iodomethane and ethylene glycol as test liquids. The participants conducted the tests in accordance with this document. However, no specifications in regard to the measuring method (dynamic or static) and the evaluation method were given. The evaluation of the results gave quite a satisfactory repeatability. However, the reproducibility was inacceptable. During a second interlaboratory test with seven participants and one test specimen, the measuring method (static), as well as the evaluation method, were clearly described. For the determination of the contact angle, di-iodomethane, water and ethylene glycol were used as test liquids. The results of this interlaboratory test were used for the determination of the precision data, indicated in 9.2 and 9.3.

9.2 Repeatability limit, r

The repeatability limit, r, is the value below which the absolute difference between two single test results, each the mean of valid duplicates, can be expected to lie with a probability of 95 % when this method is used under repeatability conditions. In this case, the test results are obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method. In this document, the repeatability limit, r, is on average 2,4° for the determination of the contact angle.

9.3 Reproducibility limit, R

The reproducibility limit, R, is the value below which the absolute difference between two single test results, each the mean of valid duplicates, can be expected to lie with a probability of 95 % when this method is used under reproducibility conditions. In this case, the test results are obtained on identical material by operators in different laboratories using the standardized test method. In this document, the reproducibility limit, R, is on average 6,7° for the determination of the contact angle.

9.4 Calculation in accordance with the Owens-Wendt-Rabel-Kaelble method

For the calculation of surface energies in accordance with the Owens-Wendt-Rabel-Kaelble (OWRK) method, the resulting deviations have been obtained based on the contact angle measuring results (see Table 3).

Table 3 — Calculations in accordance with the OWRK method

	Polar fraction σ_s^p mN/m	Dispersive fraction σ_s^d mN/m	Surface energy σ_s mN/m
Mean value	4,9	36,5	41,4
Standard deviation	0,73	1,01	0,93
Minimal value	3,9	35,2	40,4
Maximal value	6,1	37,9	43,0
Value range	2,2	2,7	2,6

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 19403-2:2024;

c) the test liquids used, their surface tensions, as well as their polar and dispersive fractions of the surface tension;

d) the designation, if the contact angle was measured in dynamic mode (dynamic advancing contact angle or dynamic receding contact angle); the designation, if the recently advanced contact angle, static contact angle or recently receded contact angle was measured using either a solid needle or liquid needle dosing system;

e) for dynamic mode, specify the used dosing speed. For recently advanced contact angle, the static contact angle or recently receded contact angle, specify the used dosing system and final drop volume;

f) the method by which the contact angle of the drop was obtained;

g) the designation of the top-view angle and, if necessary, the correction used;

h) the amount of drops per liquid;

i) the period between putting down the drop and measuring (waiting time);

j) the amount of measuring points per liquid tested;

k) the result of the contact angle measurement of every liquid (mean value and standard deviation);

l) the determined polar fraction of the surface free energy of the solid, σ_s^p, the dispersive fraction, σ_s^d, and the total surface free energy of the solid, σ_s ;

m) the model used for the calculation of the surface energy;

n) the random error for the calculation of the surface free energy;

o) all deviations from the specified method and their possible influences on the results;

p) any unusual observation (deviation) during the test;

q) the type of device;

r) the name of the test person(s);

s) the date of the test.

Annex A
(informative)

Notes on measuring practice

A.1 Sampling and treatment of test specimen

A.1.1 Selection of test specimen

In regard to the surface property, the test specimen should be representative for the material to be tested.

A.1.2 Ambient conditions

For materials which shall be tested under general conditions, the notes on sample conditioning indicated in 7.2.1 are valid. For materials which shall be tested under process conditions, these conditions should be simulated in regard to pressure, temperature and humidity.

A.1.3 Sources of contamination and cleaning

During sampling, the contact of the test specimen with air containing smoke or aerosol with liquids or other solid surfaces should be avoided in order not to transfer surface-active substances. The test specimen shall only be touched in locations which are not intended for measuring. For transport, an air-tight container is recommended.

In case a test specimen is contaminated during process, it can be cleaned using a solvent which evaporates quickly and residue-free and does not chemically alter the material.

Surface-active cleaning agents shall not be used.

It is necessary that solvents used for cleaning evaporate without residue.

An ultrasonic bath is recommended for cleaning. After cleaning, the test specimen should dry in clean room air for one hour.

Drying with pressurized air increases the danger of contamination and is not recommended.

For comparative measurements, the same cleaning procedure should be conducted in each case even if no contamination occurred.

A.1.4 Electrostatic charging

Electrostatic charging of the test specimen alters the wettability performance and should be avoided. In case test specimens are inclined to electrostatic charging due to process or material, this can be dissipated by means of commercially available ionizers.

A.2 Keeping clean the test liquids

A.2.1 General

The test liquids shall be pure (see 5.3) and completely free of surface-active substances. Glass containers are preferred over plastic containers.

A.2.2 Water

Water purified by means of ion exchangers should not be used.

A.2.3 Di-iodomethane

Di-iodomethane decomposes under the influence of air and shall be kept protected from light in a brown glass bottle. Reddish or brown-yellow coloured di-iodomethane should be tested in accordance with 5.3 and, if necessary, no longer be used.

A.2.4 Glycerol

Glycerol is hygroscopic. The surface tension changes with increasing water content. Storage containers shall be kept protected from moisture and after taking the amount for measuring, shall be closed immediately.

A.3 Notes on the static method

When using automatic dosing units, dosing speeds of 100 µl/min and generally the drop volumes indicated in 7.2.2 should not be exceeded.

For higher-viscosity test liquids, e.g. glycerol, a waiting time until setting the equilibrium value shall be clearly set.

A.4 Notes on the dynamic method

A.4.1 Dynamic method

The dynamic method is frequently used in order to measure the contact angle on several, closely adjacent positions along the wandering three-phase contact point. The contact angle is plotted against time (see 7.2.3). In case the values change when starting measuring and run into a plateau, the mean value can be calculated from the values of the plateau.

For the determination of the surface free energy, the same method (static or dynamic) shall be used for all test liquids. For comparison measurements on different materials, the same method shall be used as well.

A.4.2 Preconditions

The following preconditions apply when using the dynamic method:

— the measuring device is equipped with a dosing unit, which enables a uniform dosing;

— it is possible to measure the contact angle on live-video screen or evaluation of a recorded video;

— software features a method, which enables measuring the contact angle on a contour shape altered by the needle;

— the needle (see 7.2.3) is appropriately positioned in order to minimize an influence on the contour of the drop in the area of the three-phase contact points;

— the maximum dosing speed is 10 µl/min (see 7.2.3);

— the contact-angle values are above 10° for all test liquids (see 7.2.3).

A.5 Image generation and evaluation

A.5.1 Brightness and contrast

The measuring device should be set up in a location without intense exposure to light from outside (windows, bright lighting).

Outshining the contour of the drop due to lighting, which is set too bright, shall be avoided.

A.5.2   Width of the drop

The width of the drop should take up approximately two thirds of the width of the image (see 7.2.1). For automatic measuring with several liquids, it shall be taken into account for the setting of the zoom that the drop widens in the case of larger volumes and lower contact angles.

A.5.3   Base line recognition

For measuring devices featuring automatic base line recognition, this should be used if possible. Only if this does not operate reliably, should the base line be determined manually.

NOTE   Problems are anticipated with the automatic base line recognition for images of poor contrast, for little or non-reflecting test specimens or for contact angles around 90°.

In order to help set the base line, the contrast of the image can be intensified.

For repeated measurements on a drop, the automatic base line recognition should be used for every measurement and not only once.

A.6   Methods of measuring the contact angle

Table A.1 comprises and supplements the criteria for the selection of the contact angle measuring method indicated in 7.2.4.

In Table A.1, the symbol ✓ indicates that the respective method can be used for the case indicated in the first column.

Table A.1 — Usable measuring methods

	Circle	Conic section	Polynomial	Young-Laplace
Measuring range				
0° to 20°	✓		✓	
20° to 110°		✓	✓a	✓
110° to 180°			✓	✓
Drop mass (volume × density)				
small	✓	✓	✓	✓
large		✓	✓	✓
very large			✓	✓
Dosing				
static (contour without cannula)	✓	✓	✓	✓
dynamic (contour with cannula)		✓	✓	
Contour shape				
symmetrical	✓	✓	✓	✓
slightly asymmetrical		✓	✓	
highly asymmetrical			✓	
a The polynominal method can be inaccurate if the contact angle is close to 90°.				

NOTE 1   Instead of using the formula of a circle, it is also possible to measure the height and width of the contour of the drop (height-width method), which is considered as a circular arc. When using software providing both methods, the equation of a circle is preferred.