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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

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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*.

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

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

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 — Terms and definitions*

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

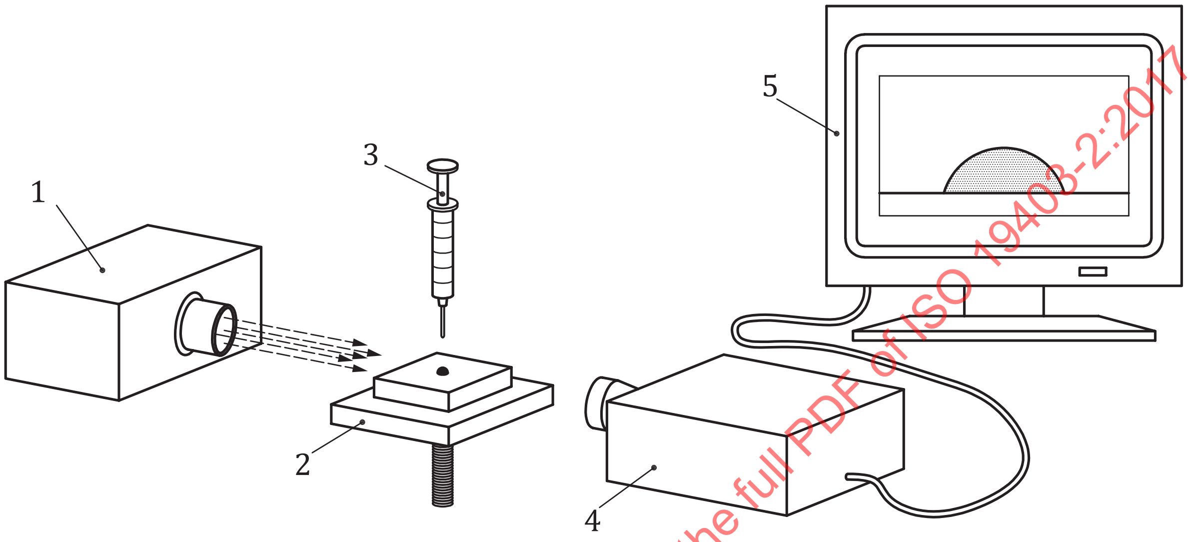
A minimum of three drops of at least two test liquids are dosed onto the flat surface of a test specimen. For every drop, the contact angle is measured. From the averaged contact angles of every liquid, their surface tensions, as well as their polar and dispersive fractions, the surface free energy of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

5.1 Contact angle measuring system.

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

1	light source
2	specimen holder
3	graduaded micro syringe
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.

The dosing unit makes it possible to precisely apply drops in the range of microlitres to the surface.

5.3 Test liquids.

At least two of the test liquids 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.

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 Table 1. 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 three liquids are used, ethylene glycol should be used additionally.

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 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** $\sigma_\mathrm{l}^\mathrm{d}$ mN/m	**Polar fraction** $\sigma_\mathrm{l}^\mathrm{p}$ mN/m	**Source**
Water	72,8	21,8	51,0	Reference [4]
Di-iodomethane[a]	50,8	50,8	0,0	Reference [4]
1,2-ethanediol (ethylene glycol)	47,7	30,9	16,8	Reference [4]
1,2,3-propanetriol (glycerol)	63,4	37,0	26,4	Reference [4]
Hexadecane	27,6	27,6	0,0	Reference [4]
1- bromo-naphthalene[b]	44,6	44,6	0,0	Reference [4]
Benzyl alcohol	38,9	29,0	9,9	Reference [5]
Decalin (isomer mixture)	30,6	30,6	0,0	Reference [4]
cis-Decalin	32,2	32,2	0,0	Reference [2]
trans-Decalin	29,9	29,9	0,0	Reference [2]

[a] Di-iodomethane is relative 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.

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 10 cm × 10 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

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.

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 that it is horizontally aligned.

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 two-dimensional images of drops. Such reference images are commercially available.

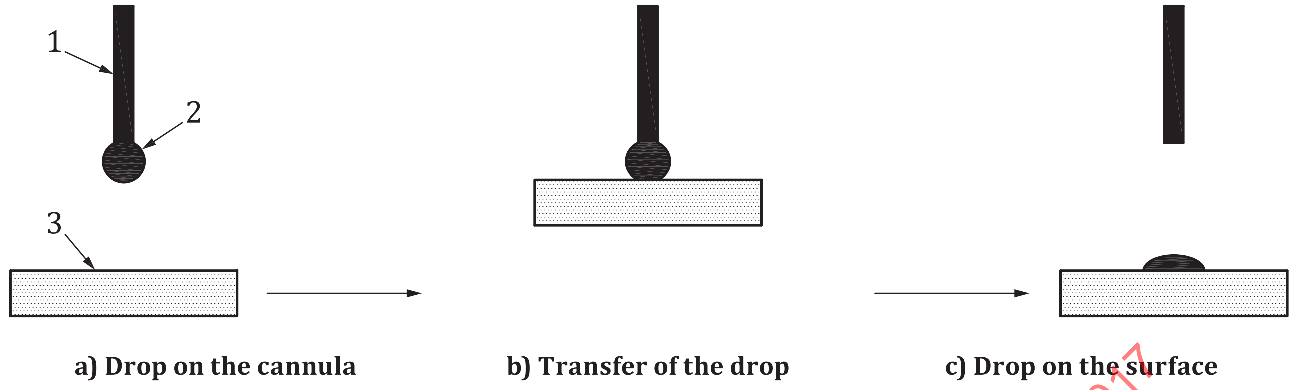
Move the needle to the upper margin of the image and bring into focus. Set up the zoom of the contact angle measuring device so that the width of the contour of the drop takes up two thirds of the width of the image.

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 The contact between the drop and the solid surface can be achieved by putting down by means of the needle or by picking up by means of the specimen table.



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.

NOTE 2 A top-view angle can be set to help find the three-phase points.

Start measuring the contact angle immediately after finishing dosing. Prior to and during measuring, no mutual transformation of materials between the test liquid and the surface should occur.

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

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.

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

NOTE 5 A slight top-view angle of the camera to the horizontal can be set to help find the three-phase points. The top-view angle causes an image error of the drop projection. This may have an influence on the measuring result of the contact angle and can be corrected.

Start measuring the contact angle immediately after dosing the minimal volume. Record the measuring values as function of time. Prior to and during measuring, no mutual transformation of materials between the test liquid and the surface shall occur.

NOTE 6 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 the numerical method, which describes the contour of the drop best.

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°, polynom 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 polynom method is of advantage.

Measure on a minimum of three different measuring points on the test specimen in order to obtain sufficient information in regard to the homogeneity of a test specimen. Previously wetted positions shall not be used. Arguable readings which may 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 arithmetic mean value of the measuring values.

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 used preferably. For low-energy surfaces, the calculation in accordance with Wu 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 [7] and [8]). It is recommended to obtain the error in measurement of σ_l^d in accordance with the Gauß error propagation (see References [7] and [8]).

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;

$\sigma_{\mathrm{l}}^{\mathrm{p}}$ and $\sigma_{\mathrm{l}}^{\mathrm{d}}$ are the polar and dispersive fractions of the surface tension.

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

Table 2 — Calculated values for insertion into the linear equation

Liquid	$\sqrt{\frac{\sigma_{\mathrm{l}}^{\mathrm{p}}}{\sigma_{\mathrm{l}}^{\mathrm{d}}}} = x$	$\frac{\sigma_{\mathrm{l}}}{2\sqrt{\sigma_{\mathrm{l}}^{\mathrm{d}}}} = y/(1+\cos\theta)$ mN/m
Water	1,529 5	7,796 0
Di-iodomethane	0,000 0	3,563 7
1,2-ethanediol (ethylene glycol)	0,737 4	4,290 5
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 $\sigma_{\mathrm{s}}^{\mathrm{p}}$ is calculated and from the square of the ordinate section b, the disperse fraction $\sigma_{\mathrm{s}}^{\mathrm{d}}$. From the sum of both fractions, the surface energy of the solid, σ_{S}, is calculated.

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):

$$\sigma_{\mathrm{l}}\left(1+\cos\theta\right) = 4\left(\frac{\sigma_{\mathrm{s}}^{\mathrm{d}}\cdot\sigma_{\mathrm{l}}^{\mathrm{d}}}{\sigma_{\mathrm{s}}^{\mathrm{d}}+\sigma_{\mathrm{l}}^{\mathrm{d}}}+\frac{\sigma_{\mathrm{s}}^{\mathrm{p}}\cdot\sigma_{\mathrm{l}}^{\mathrm{p}}}{\sigma_{\mathrm{s}}^{\mathrm{p}}+\sigma_{\mathrm{l}}^{\mathrm{p}}}\right) \tag{3}$$

NOTE 1 Preferably, an error square minimization in accordance with Gauß is used for the numerical calculation (see Reference [6]).

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 [7] and [8]).

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

For the calculation of surface energies in accordance with the 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;

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

d) the designation, if measured in dynamic or static mode;

e) the drop volumes or volume range and dosing speed;

f) the method by which means 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 are to be tested under general conditions, the notes on sample conditioning indicated in 7.2.1 are valid. For materials which are to 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 to be 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 Ethylene glycol and glycerol

Both liquids are 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 (see 7.2.2)

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 (see 7.2.3)

A.4.1 Dynamic method

The dynamic method is frequently used in order to measure the contact angle time-independently and 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 for using the dynamic method

- measuring device equipped with dosing unit, which enables a uniform dosing;
- possibility of measuring the contact angle on live-video screen or evaluation of a recorded video;
- software featuring a method, which enables measuring the contact angle on a contour shape altered by the needle;
- appropriate positioning of the needle (see 7.2.3) in order to minimize an influence on the contour of the drop in the area of the three-phase contact points;
- maximum dosing speed of 10 µl/min (see 7.2.3);
- contact-angle values 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.

NOTE Decisively for the assessment of the drop image is not the subjective evaluation of brightness and contrast but the concentration on the grey value grades indicated in 7.2.1.

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

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 (see 7.2.4)

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

NOTE 1 The symbol ✓ indicates that the respective method can be used for the case indicated in the left column.

Table A.1 — Usable measuring methods

	Circle	Conic section	Polynomial	Young-Laplace
Measuring range				
0° to 20°	✓			
20° to 110°		✓	✓	✓
110° to 180°			✓	✓
Drop mass (volume × density)				
small	✓	✓	✓	✓
large		✓	✓	✓
very large			✓	✓
Dosing				
static (contour without cannula)	✓	✓	✓	✓
dynamic (contour with cannula)		✓	✓	
Contour shape				