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Paints and varnishes — Wettability —

Part 7: Measurement of the dynamic contact angles and the roll-off angle on a tilt stage

Peintures et vernis — Mouillabilité —

Partie 7: Mesurage des angles de contact dynamiques et de l'angle de roulement sur un plan incliné

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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 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 19403-7:2017), which has been technically revised.

The main changes are as follows:

— the title of the document has been changed;

— the term 3.2 “advancing angle” has been replaced by “dynamic advancing contact angle” and the definition has been reworded;

— the term 3.3 “receding angle” has been replaced by “dynamic receding contact angle” and the definition has been reworded;

— the minimum size of the text samples has been changed to 4 cm × 4 cm;

— the use of ethylene glycol as test liquid has been deleted;

— 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.

Introduction

Dynamic contact angles describe the processes on the interface liquid or solid during volume increase (dynamic advancing contact angle) or volume decrease (dynamic receding contact angle) of a drop in horizontal position. As an alternative to the static method (see ISO 19403-2) a surface area is always wetted for the dynamic advancing contact angle, which was previously unwetted. For the dynamic receding contact angle, the contact angle is observed during dewetting. By determining the difference between the dynamic advancing contact angle and the dynamic receding contact angle, information on chemical homogeneity and roughness can be concluded. The dynamic receding contact angle is not suitable for the determination of the surface energy.

Paints and varnishes — Wettability —

Part 7: Measurement of the dynamic contact angles and the roll-off angle on a tilt stage

1 Scope

This document specifies a method for the dynamic measurement of the roll-off angle on a tilt stage of a liquid drop on a solid surface. This document also specifies how the dynamic advancing and receding contact angles of the drop rolling off can be determined. The roll-off angle determined through this method can be applied when evaluating easy-to-clean or anti-adherent surfaces.

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, *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
roll-off angle
α_s
tipping of the surface of the solid body, which arises from the rolling off of a liquid drop that is put down onto this surface

3.2
dynamic advancing contact angle
dynamic advancing angle
θ_a
contact angle measured at the three-phase line during advancing the liquid phase

Note 1 to entry: The values depend on the method.

[SOURCE: ISO 19403-6:2024, 3.2]

3.3
dynamic receding contact angle
dynamic receding angle
θ_r
contact angle measured at the three-phase line during receding the liquid phase

Note 1 to entry: The values depend on the method.

[SOURCE: ISO 19403-6:2024, 3.3]

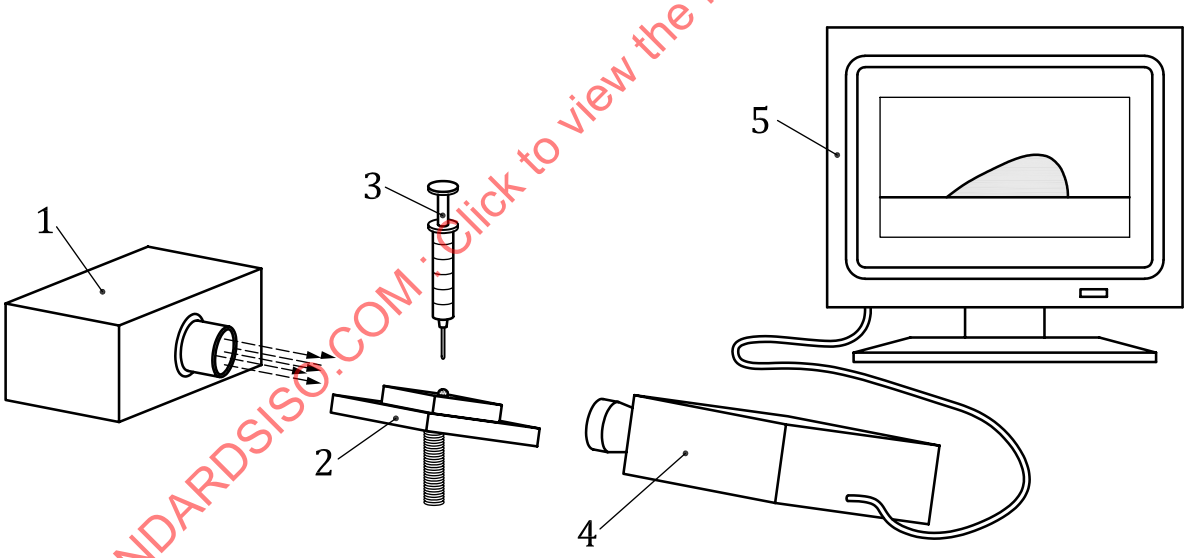
4 Principle

A drop is put down onto the surface to be tested. The surface is tipped with constant inclination speed until the drop rolls off. The progressing angle is determined from the temporal tracking of the wetting three-phase point and the dynamic receding contact angle from the temporal tracking of the dewetting three-phase point.

5 Apparatus and materials

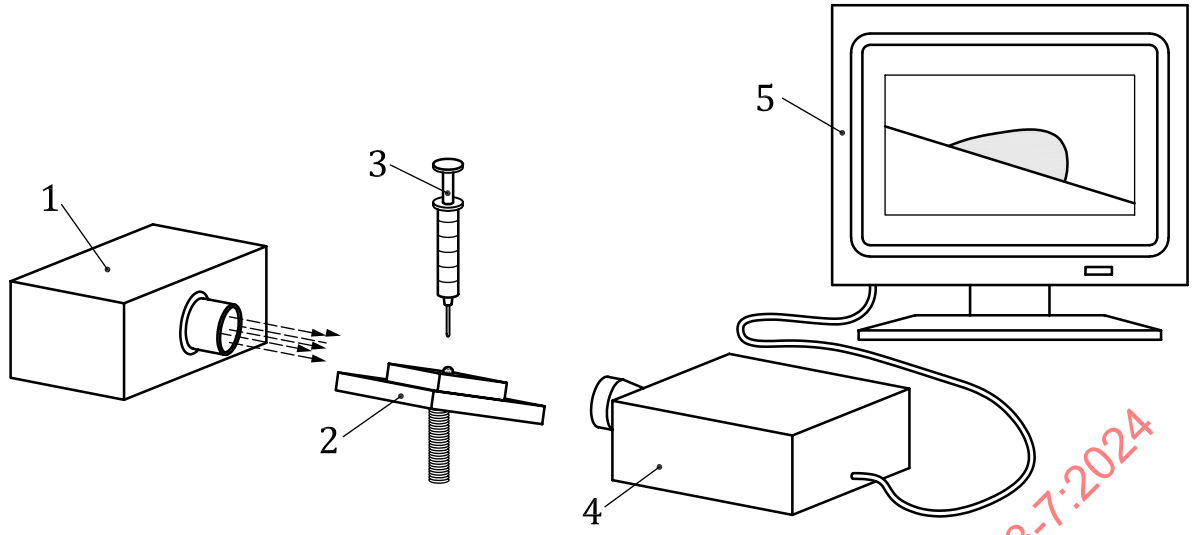
Ordinary laboratory apparatus, together with the following, shall be used.

5.1 Contact angle measuring system, i.e. any state-of-the-art contact angle measuring device fitted with a tilting device. It is preferred to use systems with digital image capture and analysis for measuring the contact angle. Figure 1 shows a system for which the camera as well as the sample table are inclined. Figure 2 shows a schematic example of a contact angle measuring system for which only the sample table is inclined.



Key
1 light source
2 specimen holder
3 graduated microsyringe
4 optical system
5 screen

Figure 1 — Schematic diagram of a contact angle measuring system for which the camera as well as the sample table are inclined



Key

1 light source
2 specimen holder
3 graduated microsyringe
4 optical system
5 screen

NOTE The device used can differ from the schematic diagram regarding light path and the arrangement of the components.

Figure 2 — Schematic diagram of a contact angle measuring system for which only the sample table is inclined

The image capturing system should be oriented in a way that the drop is within the left third of the image (when the table is inclined to the right).

5.2 Dosing unit, which makes it possible to precisely apply drops in the range of microlitres to the surface.

5.3 Test liquids, of 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.

If not otherwise agreed, use at least one of the test liquids suggested in Table 1.

It is recommended to measure the surface tension of the liquids to be used according to 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. They shall not have a yield point.

The test liquids shall not cross-link, show any skinning or evaporate during the measurement.

Liquids having a vapour pressure higher than water at 30 °C shall be measured in the saturated vapour phase.

NOTE The values given 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 [5]
Di-iodomethane	50,8	50,8	0,0	Reference [5]
1,2,3-propanetriol (glycerol)	63,4	37,0	26,4	Reference [5]
Hexadecane	27,6	27,6	0,0	Reference [5]
1-bromo-naphthalene	44,6	44,6	0,0	Reference [5]
Benzyl alcohol	38,9	29,0	9,9	Reference [6]
Decalin (isomer mixture)	30,6	30,6	0,0	Reference [5]
cis-Decalin	32,2	32,2	0,0	Reference [7]
trans-Decalin	29,9	29,9	0,0	Reference [7]

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 and be as flat as possible.

For advice on sampling and sample preparation, see Annex A.

7 Procedure

7.1 General for measuring the roll-off angle

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 and secure a preferably flat specimen with the surface to be measured on the specimen holder to avoid slipping during tilting. Adjust the specimen 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.

Adjust an image representation that is sufficient regarding 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.

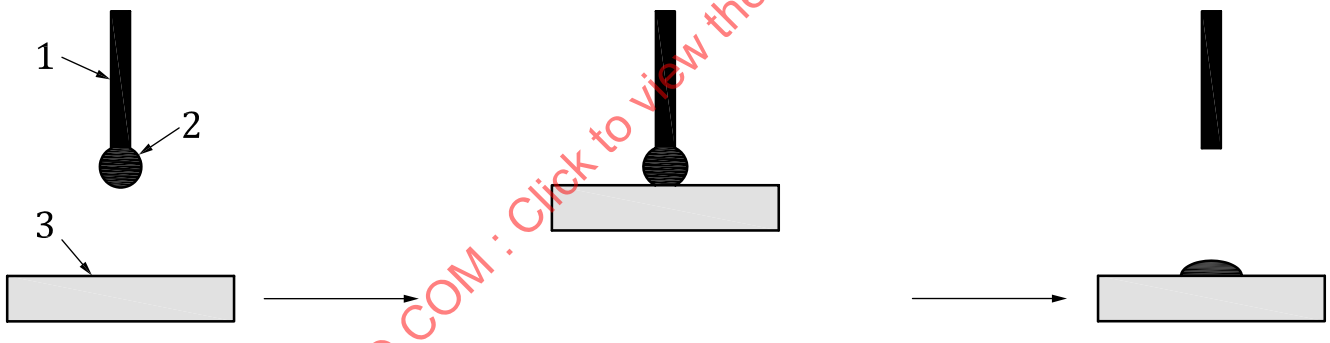
Move the needle to the upper margin of the image and bring into focus.

7.2.2 Application of the drop

Position the dosing needle approximately 3 mm to 6 mm above the surface of the test specimen. The volume of the drop depends on the used liquid, the test specimen, the inclination speed and shall be included in the test report.

Apply a drop of the test liquid on the surface (see Figure 3).

NOTE 1 The contact between the drop and the solid surface can be achieved by putting down using the needle or by picking up using the sample table.



a) Drop on the needle **b) Transfer of the drop** **c) Drop on the surface**

Key

1 needle
2 test liquid
3 surface of test specimen

Figure 3 — 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 adjusted to help find the three-phase points.

Begin measuring the contact angle and the inclination of the tilt table, or tilt table and camera, immediately after dispensing has ended.

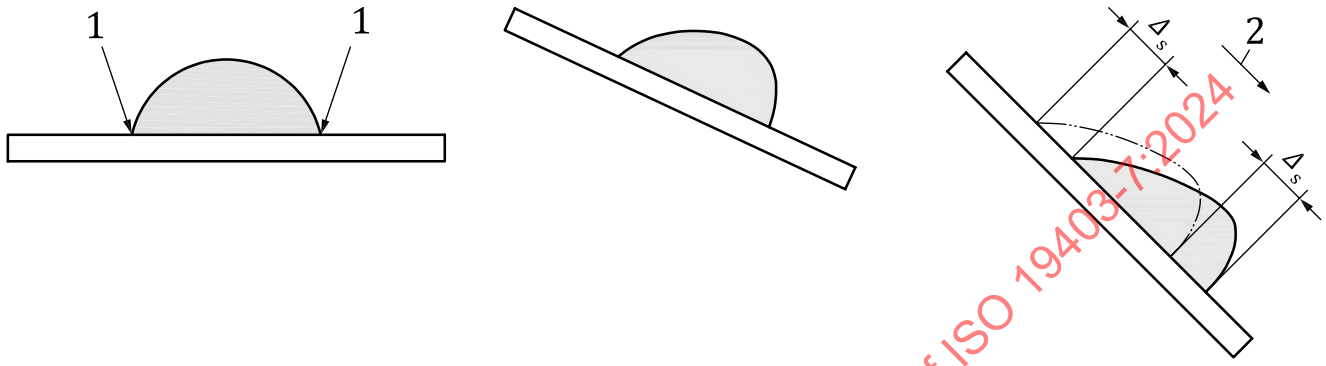
NOTE 3 On low energy surfaces, it is possible that the drop does not detach completely from the needle.

7.2.3 Determination of the roll-off angle

Tip the test specimen with a constant inclination speed without vibration. During tipping, observe the left and right three-phase point. The roll-off angle has been reached as soon as both of the three-phase points have moved over a specified distance, Δ_s, (see Figure 4). To a large extent, the roll-off angle depends on the drop volume and the inclination speed (see also Annex B).

At least Δ_s = 1 mm is required. The frame rate shall be adjusted to ensure a proper determination of Δ_s.

The drop volume should reflect the real-life application of the surface.



a) Test specimen horizontal **b) Test specimen inclined about 25°** **c) Test specimen inclined about 45°**

Key

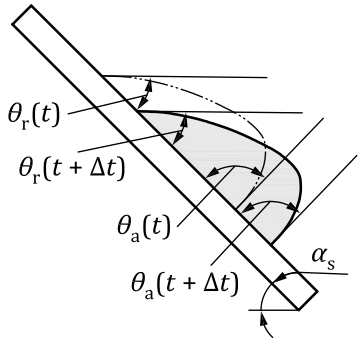
1 left and right three-phase point

2 movement

Δ_s specified distance

Figure 4 — Tipping of the test specimen

Upon reaching the roll-off angle, the dynamic advancing and receding contact angles can also be evaluated (see Figure 5).

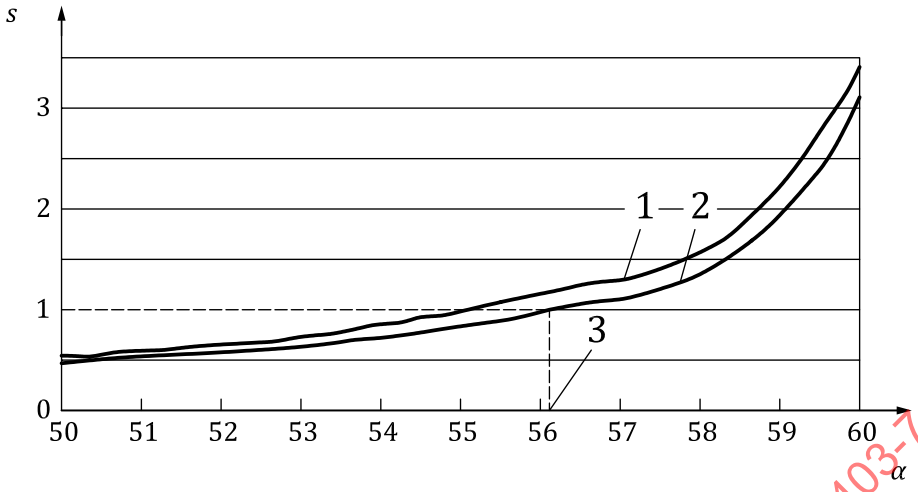


Key

$\theta_r(t)$	dynamic receding contact angle at time t
$\theta_r(t + \Delta t)$	dynamic receding contact angle at time $t + \Delta t$
$\theta_a(t)$	dynamic advancing contact angle at time t
$\theta_a(t + \Delta t)$	dynamic advancing contact angle at time $t + \Delta t$
α_s	roll-off angle

Figure 5 — Roll-off angle: inclination angle, for which the receding three-phase point has moved by about 1 mm

Generally, the dynamic receding contact angle is lower than the dynamic advancing contact angle and the right three-phase point moves before the left one does (see Figure 6).

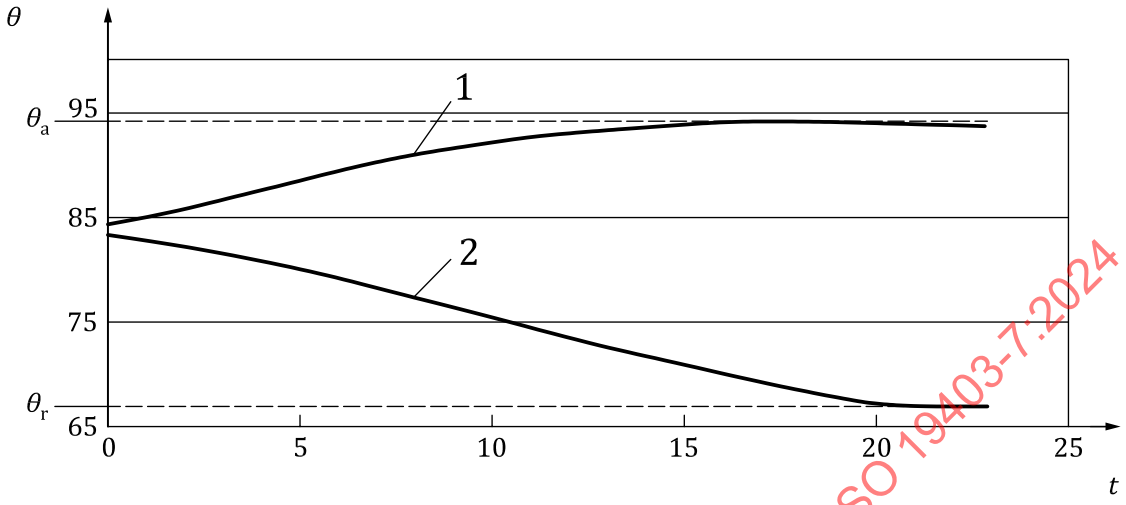


Key

s distance, in millimetres
α inclination angle, in degrees
1 three-phase point (right)
2 three-phase point (left)
3 roll-off angle

Figure 6 — Change of position of the three-phase points depending on the roll-off angle

Figure 7 shows the change of the dynamic advancing and receding contact angle over time after reaching the roll-off angle. After a phase of acceleration, the drop reaches constant speed and the dynamic advancing and receding contact angles reach a stable plateau.



Key

θ contact angle
θ_a dynamic advancing contact angle
θ_r dynamic receding contact angle
t time, in seconds
1 time curve of the dynamic advancing contact angle
2 time curve of the dynamic receding contact angle

Figure 7 — Time curve of the dynamic advancing and receding contact angles after reaching the roll-off angle

Measure on a minimum of three different measuring points on the 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.

8 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the test specimen (manufacturer, product identification, batch number, etc.);

b) a reference to this document, i.e. ISO 19403-7:2024;

c) the used test liquid(s);

d) the used drop volume used;

e) the dosing speed and waiting times, if necessary;

f) the method with which the contact angle of the drop was obtained, if deviating from the polynomial method;

g) an indication of the tilting speed;

h) the top-view angle and, if necessary, the used correction;

i) the contact angle vs. time diagram;

j) the amount of measuring points per test liquid;

k) the dynamic advancing and receding contact angle, if necessary, including mean value and standard deviation;

l) any deviations from the specified method and their possible influences on the results;

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

n) the type of device;

o) the name of the person who conducted the test;

p) the date of the test.

Annex A
(informative)

Notes on sampling and treatment of test specimens

A.1 Ambient conditions

For materials which shall be tested under general conditions, the notes on sample conditioning indicated in 7.1.3 apply. For materials, which shall be tested under process conditions, these conditions should be simulated regarding pressure, temperature and humidity.

A.2 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 to not 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.

Contaminated surfaces of test specimens can also be measured. These contaminations can influence the wettability behaviour and dewettability behaviour.

If it is necessary to clean a contaminated surface, this can be done by using a rapidly and residue-free evaporating solvent, which does not chemically alter the material.

Surface-active cleaning agents shall only be used if they can be removed residue-free.

It is possible that solvents used for cleaning do not evaporate without residue.

An ultrasonic bath is recommended for cleaning. After cleaning, the test specimen should dry in clean room air for 1 h.

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.3 Electrostatic charging

Electrostatic charging of the test specimen alters the wettability behaviour 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.