BS ISO 19403-6:2017

BSI Standards Publication

Paints and varnishes — Wettability

Part 6: Measurement of dynamic contact angle

bsi.

National foreword

This British Standard is the UK implementation of ISO 19403-6:2017.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2017

ISBN 978 0 580 87399 7

ICS 01.040.87; 87.040

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2017.

Amendments/corrigenda issued since publication

Date	Text affected

BS ISO 19403-6:2017

INTERNATIONAL STANDARD

ISO 19403-6

First edition
2017-06

Paints and varnishes — Wettability —

Part 6: Measurement of dynamic contact angle

Peintures et vernis — Mouillabilité —

Partie 6: Mesurage de l'angle de contact dynamique

Reference number
ISO 19403-6:2017(E)

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

Contents

Page

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

Introduction

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

INTERNATIONAL STANDARD **ISO 19403-6:2017(E)**

Paints and varnishes — Wettability —

Part 6: Measurement of dynamic contact angle

1 Scope

This document specifies a method to measure the dynamic contact angle with an optical method. The advancing and the receding angles are determined.

By means of this defined measurement, the wetting and dewetting properties can be characterized. It can also be concluded on the morphological and chemical homogeneity of interfaces.

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 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 free surface energy of solid surfaces by measuring the contact angle*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and ISO 19403-1, and the following 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

3.1
dynamic contact angle
contact angle, which is measured during advancing or receding of the three-phase point

Note 1 to entry: For the definition of “contact angle”, see ISO 19403-1:2017, 3.1.9.

Note 2 to entry: The advancing or receding of the three-phase point can be achieved by changing the volume of the liquid drop to be measured, by relative movement (immersing and pulling out) of a solid body to an interface, or by moving the drop over the interface (e.g. rolling off).

3.2
advancing angle
θ_a
contact angle, which is measured during advancing of the three-phase point

Note 1 to entry: Generally, the advancing angle is used for the determination of the interface energy, in which case, the measurement should be carried out close to the thermodynamic equilibrium. This is approximately reached if there is no influence of, for example, the dosing speed on the contact angle.

3.3
receding angle
θ_r
contact angle, which is measured during receding of the three-phase point

3.4
contact angle hysteresis
θ_{ar}
difference between *advancing angle* (3.2) and *receding angle* (3.3)

3.5
polynomial method
<contact angle> image-analysing evaluation method for the contact angle which can also be applied when the dosing needle is still inside the drop

4 Principle

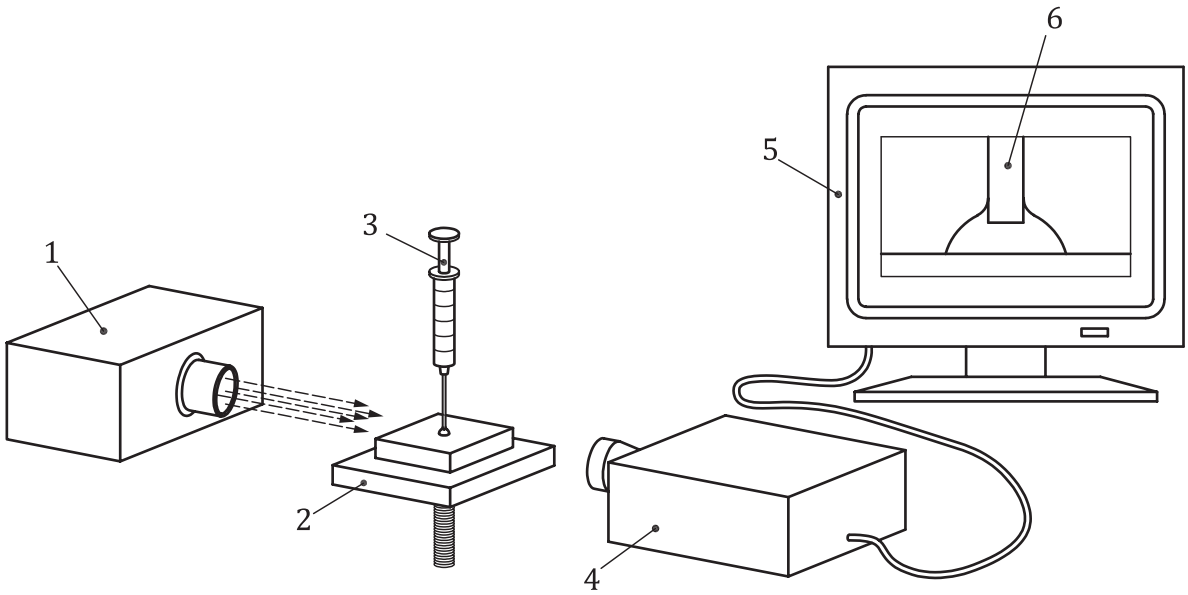
At least three drops of the respective test liquids are dosed onto the flat surface of a test specimen. The volume of the respective drop is continuously increased (advancing angle) or decreased (receding angle). The contact angle is preferably determined by means of the polynomial method, synchronously with the dosing. If the polar and dispersive fraction of the surface free energy is to be determined according to ISO 19403-2, the advancing angle shall be used.

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 system with microlitre syringe for continuous dosing
4 optical system
5 screen
6 needle positioned in the drop

Figure 1 — Schematic diagram of a contact angle measuring system

The image capturing 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 arrangement of the components.

5.2 Dosing unit.

Dosing unit, which makes it possible to continuously change the drop volume on the surface in the range of microlitres.

NOTE Typical dosing rates for test liquids for the determination of the surface energy are in the range of 10 µl/min.

5.3 Test liquids.

The test liquids shall not physically or chemically affect the surface. They shall not have a distinct yield point.

NOTE A notable yield value is shown when a lamella of the liquid teared with a needle does not level within a given time limit (e.g. 30 s).

The test liquids shall not crosslink during measuring, not form skins and not volatilize distinctly.

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

6 Sampling

Take a representative specimen of the solid substrate to be tested as described in ISO 15528. The specimens shall not be contaminated before measuring.

For advice on sampling and sampling preparation, 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.

7.1.4 Conditioning of the test liquids

Condition the liquids in a closed container at a temperature of (23 ± 2) °C 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 specimen holder. 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 in regard to brightness and contrast (mind the specifications given by the manufacturer).

If possible, adjust 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 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.

Bring the needle into focus. Adjust the zoom of the contact angle measuring device so that the maximum width of the contour of the drop is imaged entirely at maximum expansion.

7.2.2 Measuring method

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 needle of poorly wettable material.

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.

Typical dosing speeds for the determination of the surface energy are in the range of 10 µl/min. For test liquids with higher viscosity than most of the test liquids given in ISO 19403-2:2017, Table 1, the dosing speeds shall be reduced. This especially applies for glycerol.

NOTE 2 It is common to start measuring only after a dosing of 3 µl minimal volume.

NOTE 3 Due to the limited image section and the limited precision, it can be difficult to measure advancing 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 4 A top-view angle of the camera to the horizontal can be adjusted 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.

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. This especially applies for short measuring times.

7.2.3 Determination of the contact angle

Preferably, determine the advancing and receding angle by means of the polynomial method.

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. Discarded, possibly contaminated test liquid shall not be reused.

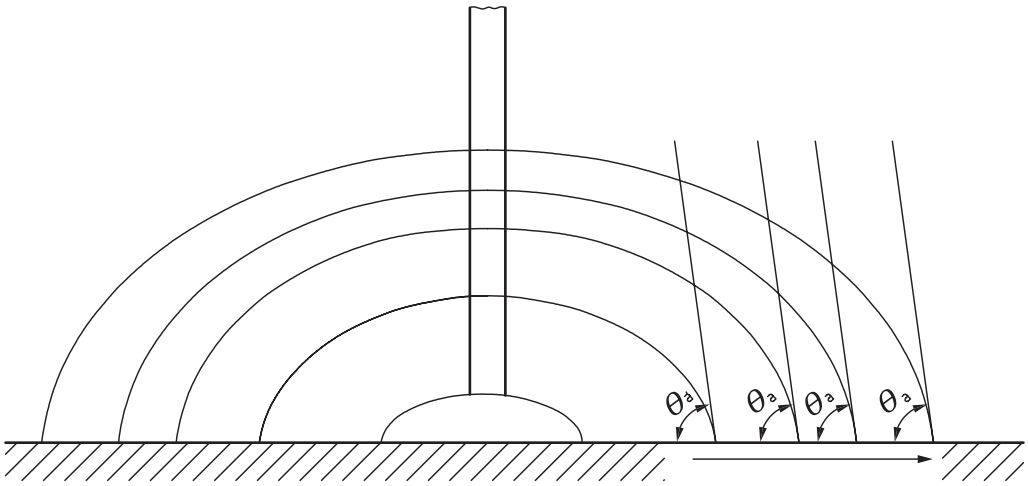
Repeat the measuring with at least one more liquid, which was selected in accordance with the criteria given in 5.3.

8 Evaluation

Present the measured contact angles as function of time. Determine the advancing and receding angle from these data (see Figure 2 and Figure 3). An advancing and receding angle can only be described if a respective plateau forms in the contact angle/time diagram (see Figure 4). This requires that the measured advancing or receding angle remains constant over a certain time period or at least varies around a mean value.

The wettability behaviour is characterized by the advancing angle. The dewettability behaviour is characterized by the receding angle.

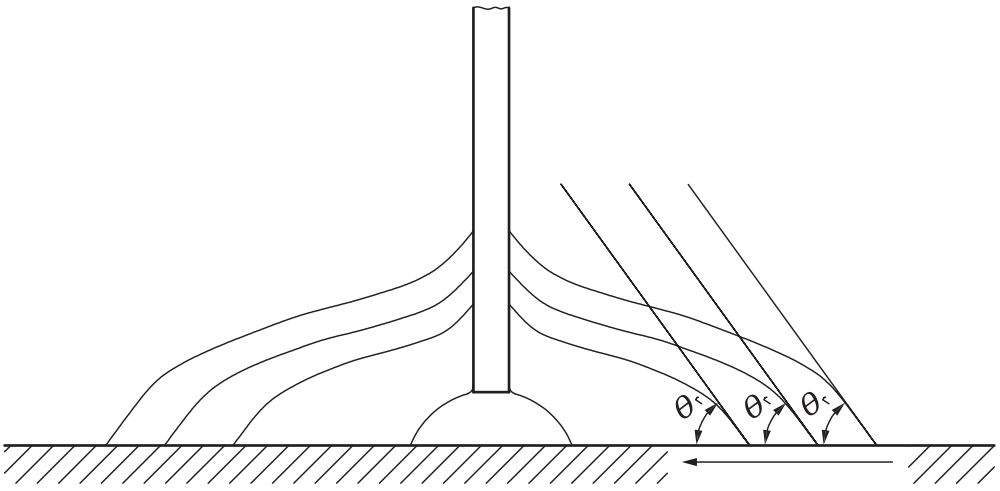
The method for determining the surface free energy is specified in ISO 19403-2.



Key

θ_a advancing angle

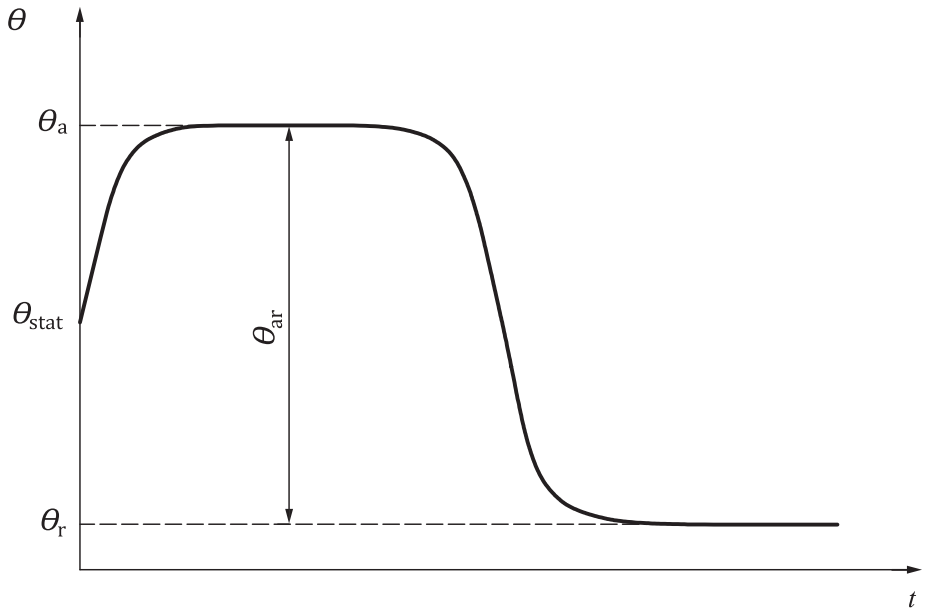
Figure 2 — Applying a drop through a needle for the dynamic measurement of the advancing angle



Key

θ_r receding angle

Figure 3 — Extracting a drop through a needle for the dynamic measurement of the receding angle

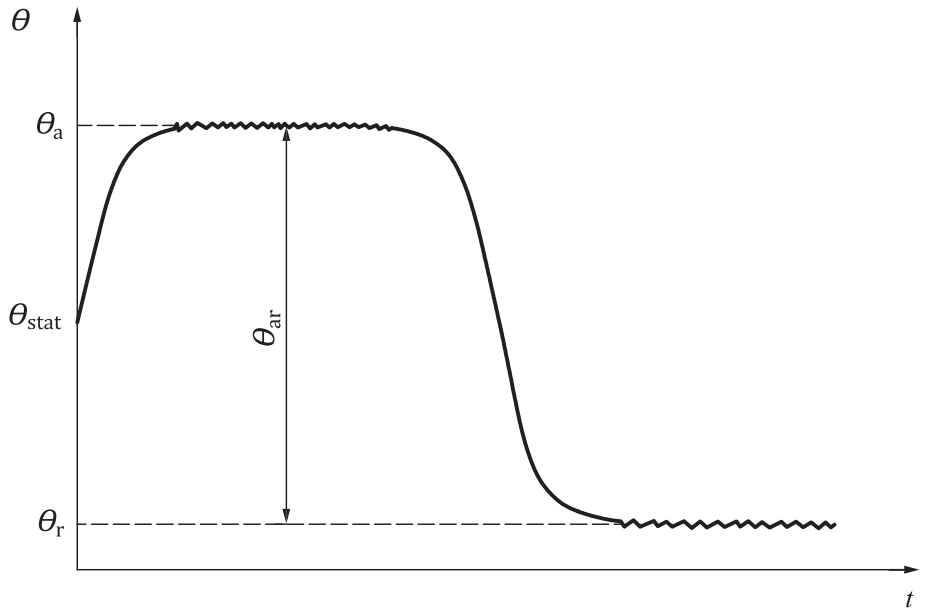


Key

θ	dynamic contact angle
θ_a	advancing angle
θ_{stat}	static contact angle
θ_r	receding angle
θ_{ar}	contact angle hysteresis
t	time

Figure 4 — Time curve of the contact angle during advancing and receding measuring

In the case of rough and chemically inhomogeneous surfaces, severe time variations of the advancing angle can occur. These occur when the diameter of the drop does not change even though the volume of the drop is continuously increased. This results in an increase of the contact angle until this contact angle is decreased instantly by the abrupt expansion of the drop. This phenomenon is called slipstick (see Figure 5).



Key

θ dynamic contact angle
θ_a advancing angle
θ_{stat} static contact angle
θ_r receding angle
θ_{ar} contact angle hysteresis
t time

Figure 5 — Slipstick behaviour of the time curve of the contact angle during advancing and receding measuring

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 test specimen (manufacturer, product identification, batch number, etc.);

b) a reference to this document, i.e. ISO 19403-6;

c) the used test liquids;

d) the used drop volumes at the beginning and upon completion of measurement;

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

h) the contact angle/time diagram;

i) the amount of measuring points per test liquid;

j) the advancing and receding angle, if necessary, including mean value and standard deviation;

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

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

m) the type of device;

n) the name of the test person;

o) the date of the test.

Annex A
(informative)

Notes on sampling and treatment of test specimens

A.1 Ambient conditions

For materials which are to be tested under general conditions, the notes on sample conditioning indicated in 7.1.3 apply. For materials which are to be tested under process conditions, these conditions should be simulated in regard to 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 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.

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

In case a contaminated surface is to be cleaned, this can be done by using rapidly and residue-free evaporating solvent, which does not chemically alter the material.

Solvents used for cleaning may possibly not evaporate without residue.

If surface-active cleaning agents are used, it shall be noted that possibly they cannot be removed residue-free.

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.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.

Bibliography

[1] ISO 3270, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing*

ICS 01.040.87
Price based on 11 pages

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.

Standards purchased in hard copy format:

- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services
Tel: +44 345 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 345 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

bsi.