BRITISH STANDARD

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Conservation of cultural property — Test methods — Determination of static contact angle

ICS 97.195

BSI
British Standards

National foreword

This British Standard is the UK implementation of EN 15802:2009.

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

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Erhaltung des kulturellen Erbes - Prüfverfahren - Messung des statischen Kontaktwinkels

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15802:2009) has been prepared by Technical Committee CEN/TC 346 “Conservation of cultural property”, the secretariat of which is held by UNI.

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Introduction

This test method can be applied if it does not change the value of the cultural property and follows relevant ethical codes of conservation practice.

1 Scope

This European Standard specifies a method for the measurement of the static contact angle of a water drop on porous inorganic materials used for and constituting cultural property. The method may be applied to porous inorganic materials either untreated or subjected to any treatment or ageing.

2 Normative references

The following referenced documents are indispensable for the application 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.

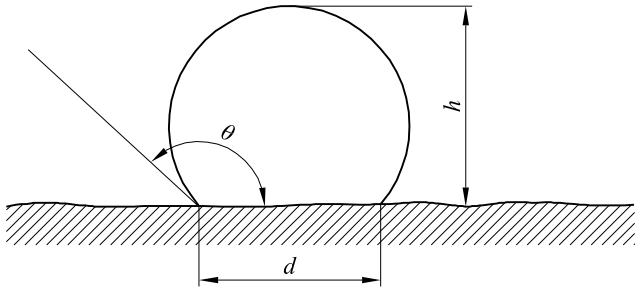
prEN 15898:2009, *Conservation of cultural property — Main general terms and definitions concerning conservation of cultural property*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN 15898:2009 and the following apply.

3.1
porous inorganic materials
materials including natural stones, e.g. sandstone, limestone, marble, as well as artificial materials, such as mortar, plaster, brick and others

3.2
static contact angle
angle θ, in degrees, formed by the surface of the specimen and the tangent to the water drop at the contact point, as shown in Figure 1



Key

d diameter of the contact surface, in mm

h height, in mm

θ static contact angle, in degrees

Figure 1 — Static contact angle at time *t*

4 Principle

Determination of the static contact angle between a water drop and the test surface of the specimen.

5 Symbols and abbreviations

For the purposes of this document, the following symbols and abbreviations apply:

d diameter of the contact surface, in mm

h height, in mm

θ static contact angle, in degrees

6 Test equipment

6.1 Test equipment for the measurement of static contact angle consists of:

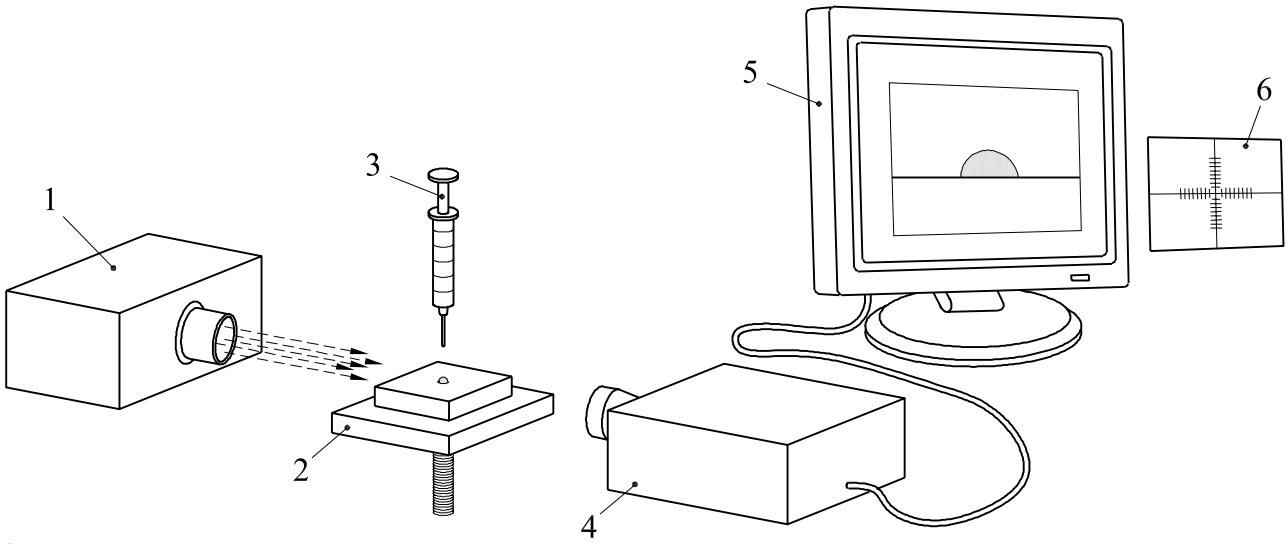
i) an illumination source (1) that will not affect the temperature of the drop/specimen system,

ii) a flat sample holder (2),

iii) a drop deposition system, usually consisting of a graduated micro-pipette (3), with a flat head needle, positioned above and perpendicular to the specimen surface and able to deliver reproducible drops,

iv) an optical system (4) that projects the image of the deposited water drop onto a screen (5) on which the height of the water drop and the diameter of the contact surface can be marked with a ruler (6). This part of the instrument can consist of a camera (4) able to record the image on a screen (5) where the parameters can be measured manually or automatically.

The measurement principle of most commercial instruments available on the market is shown in Figure 2.



Key

1 illumination source
2 sample holder
3 graduated micro-pipette
4 optical system
5 screen
6 ruler

Figure 2 — Schematic sketch of the measurement system

6.2 Sand paper with grain size of 82 μm (corresponding to grit number P180 according to the FEPA[1)] classification).

6.3 A soft brush.

6.4 Desiccator filled with desiccant such as indicating silica gel or other drying agents.

6.5 A ventilated oven which can maintain a temperature of (60 ± 2) °C.

6.6 An analytical balance with an accuracy of 0,1 mg for sample less than 200g and accuracy of 1 mg for samples more than 200 g.

6.7 A chronometer with an accuracy of 1 s.

6.8 Deionised or distilled water (with max. conductivity of 6 μS).

7 Preparation of test specimens

7.1 Number and dimensions of test specimens

The shape and dimensions of the specimens shall be conformed to the requirements of the chosen test equipment (usually 10 mm to 20 mm of thickness). The test surface shall be flat. Specimens that do not have parallel surfaces can only be tested using apparatus that adjusts for this.

The number and dimensions of specimens are dependent on the heterogeneity of the material. Each series shall consist of at least 3 specimens. All dimensions shall have a ± 0,5 mm tolerance.

7.2 Pre-conditioning of test specimens

The surface chosen for the determination of static contact angle shall be flat and wet or dry polished with sand paper (6.2). After polishing, the specimens shall be washed with water, gently brushed with a soft brush and immersed in deionised water for 30 min. In case of water-sensitive materials, for example gypsum containing materials, only dry polishing and compressed air shall be used. After smoothing and washing, the surface should not to be touched with hands.

The above procedure does not necessary apply to treated specimens or specimens taken from exposed surfaces.

Test specimens shall be dried to constant mass in a ventilated oven at a temperature of (60 ± 2) °C until a constant weight is reached, and stored in a desiccator until the test starts. If the material is temperature-sensitive, the pre-conditioning shall be conducted in a desiccator filled with desiccant or in a ventilated oven at a temperature of (40 ± 2) °C till constant mass is reached.

Constant mass is reached when the difference between two successive weighings at an interval of 24 h is not greater than 0,1% of the mass of the specimen.

8 Test procedure

Fill the micro-pipette with deionised/distilled water taking care that the needle is clean, outside and inside, to prevent air bubbles or drop deformation.

The specimen is mounted on the sample holder in such a way that the test surface is horizontal.

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1) FEPA – Federation of European Producers of Abrasives

Build up a drop with a volume in the range between 5 µl and 10 µl. Deposit the drop on the specimen surface by slowly lowering the micro-pipette and by taking care that the drop is detaching. The measurement of *d, h* and *θ* (see Figure 1) shall be carried out 10 s after the deposition of the drop. In case of macroscopically asymmetric drops, measurements shall be repeated.

If the drop loses at least 50 % of its volume in the first 10 s measurement is not possible and this shall be noted in the test report.

For each representative specimen for which the material and treatment are the same, the number of measurements shall be at least 15, including cases of fast absorption. Drops shall be deposited at a distance of at least 3 mm from each other to avoid overlapping.

9 Expression of results

9.1 Calculation of the static contact angle

Assuming a spherical drop, the contact angle is calculated according to the following equation:

$$\theta = 2arctg\frac{2h}{d}$$

where

d is the diameter of the contact surface, in mm;

h is the height, in mm;

θ is the static contact angle, in degrees.

If a different algorithm or method is used, it shall be reported in the test report.

10 Test report

The test report shall contain the following information:

a) reference to this European Standard;

b) name and address of the test laboratory in which the test was carried out;

c) date of testing (yy-mm-dd);

d) type, name, provenance, description of the porous inorganic material including chemical, petrographical, mineralogical and physical characteristics (if available) in accordance with existing standards;

e) number, shape, dimensions of the specimens and orientation of anisotropy present, if any;

f) description of pre-conditioning;

g) description of the test surface of the specimens, the date when the specimens were prepared, type and date of the treatment applied, if any;

h) type of instrument;

i) type of micro-pipette (material and volume of micro drop);

j) measuring methodology, if different from the one described in Clause 8;

k) number of measurements for each sample;

l) contact angle of single measurements;

m) mean value of the measurements obtained on one specimen and the mean of the set of specimens;

n) standard deviation;

o) number of events, such as drop absorption, for which a correct measurement of the drop was not possible;

p) all deviations from this European Standard and their justification;

q) any additional remarks.

Annex A
(informative)

Physical meaning of the static contact angle

The contact angle θ of a liquid on a surface is widely used to predict wetting and adhesion properties of the solid by calculating its solid-vapour surface tension. The contact angle θ of a liquid on a surface is frequently used to estimate the wetting properties of the solid by calculating its surface tension.

Its definition is based on the equilibrium of a drop on an ideal (i.e. flat, horizontal, smooth, homogeneous, isotropic and rigid) surface, according to the Young relationship:

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos\theta$$

where γ_{SV} is the solid-vapour interfacial tension, γ_{SL} is the solid-liquid interfacial tension and γ_{LV} is the liquid surface tension.

Looking at a cross-section of a drop on a solid, θ is the inclusive angle between the direction of γ_{SL} and the direction of γ_{LV}, tangent to the external surface of the drop, with the vertex at the three-phase liquid-solid-vapour intersection. Looking at a cross-section of a drop on a solid, θ is the inclusive angle between vector γ_{SL} and vector γ_{LV}, tangent to the external surface of the drop and having its vertex at the intersection of the three-phase system.

Under these hypotheses, a contact angle corresponds to the thermodynamic value that minimizes the surface free energy.

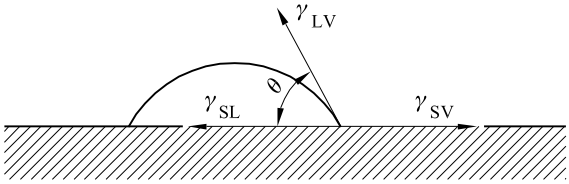

Figure A.1 — Cross-section of static contact angle

Instead, on a real surface it is generally found that a range of contact angles is possible experimentally causing wetting or contact angle hysteresis. The reason is that contact angle phenomena are very complicated. Contact angles on “non-ideal” surfaces are not only influenced by the interfacial tensions according to Young relationship, but also by many other phenomena, such as surface roughness, chemical heterogeneity, sorption layers, molecular orientation, swelling and partial solution of the constituents in the material. These effects have to be considered and the initial hypothesis of “ideal surface” is no longer valid. The contact angle value, where the system assumes its absolute minimum of surface free energy, is called equilibrium contact angle, while the other possible contact angles correspond to different metastable equilibrium states depending on the surface and on different initial conditions. The highest value in this range is commonly defined as advancing contact angle, while the lowest is defined as contact angle. The advancing angle refers to a wetting process, where liquid is changing, or has changed, a dry solid surface into a wet one. The word "advancing" comes from the requirement that the drop is just ready to spread further on the substrate surface. Thus, the advancing contact angle is the largest possible angle with the drop still at steady state and such an angle is more sensitive to the most hydrophobic micro domains of the surface. The receding contact angle on the contrary is the de-wetting angle and is the smallest steady state angle. It is more sensitive to the most hydrophilic micro domains of the surface.

The static contact angle, because of the mechanism of drop deposition, will be an advancing angle. The measurement of the angle of static contact is therefore only a partial depiction of the wettability because it is related to the characterization of the lowest surface energy areas of the surface. Accordingly, an elevated value of the static contact angle can be indicative of the presence of a protective agent, but it can not be used to evaluate the effectiveness and uniformity of the protection.

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London, W4 4AL, UK
Tel +44 (0)20 8996 9001
Fax +44 (0)20 8996 7001
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