



BSI Standards Publication

**Conservation of Cultural  
Heritage — Surface protection  
for porous inorganic materials  
— Laboratory test methods  
for the evaluation of the  
performance of water repellent  
products**

**National foreword**

This British Standard is the UK implementation of EN 16581:2014.

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

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English Version

## Conservation of Cultural Heritage - Surface protection for porous inorganic materials - Laboratory test methods for the evaluation of the performance of water repellent products

Conservation du patrimoine culturel - Protection de surface des matériaux inorganiques poreux - Méthodes d'essai en laboratoire pour l'évaluation des performances des produits hydrofuges

Erhaltung des kulturellen Erbes - Oberflächenschutz für poröse anorganische Materialien - Laborprüfverfahren für die Ermittlung der Wirksamkeit von wasserabweisenden Produkten

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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## **Foreword**

This document (EN 16581:2014) has been prepared by Technical Committee CEN/TC 346 "Conservation of Cultural Heritage", the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2015, and conflicting national standards shall be withdrawn at the latest by June 2015.

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## Introduction

As part of the conservation of built heritage, a variety of surface treatments can be carried out to delay the decay processes.

This document focuses on water repellent treatments of porous inorganic materials. The main goal of a water repellent is to reduce the penetration of liquid water and the substances dissolved in the water into porous material by changing its surface properties.

A water repellent product when applied to the surface of a material decreases its surface tension and prevents wetting of the surface. The water repellent treatment is applied to the surface and penetrates into the pores of the material, the depth of penetration being dependent on the capillary properties of the material, the properties of the hydrophobic, the type and duration of application as well as the moisture content of the substrate and the temperature.

Many deterioration mechanisms result from the presence of water and therefore the reduction of water absorption without significantly decreasing water vapour permeability may positively influence the preservation of porous inorganic materials.

Coatings including varnishes and paints are not considered within this European Standard.

A water repellent should fulfil the following requirements to:

- reduce the absorption of liquid water in the substrate,
- minimize change of water vapour permeability of the substrate,
- minimize change in colour and gloss of the substrate,
- produce no harmful by-products after the application,
- maintain its physical and chemical stability.

Water repellent products should be applied on the surface of heritage objects only after they have been tested on representative samples of porous inorganic materials in the laboratory. Field trials on small areas are strongly recommended prior to final application.

This European Standard for the evaluation of water repellent treatments is based on the measurement of appropriate parameters to assess the performance of the product using standardized test methods.

*In situ* application methods include brushing, spraying, immersion, capillary rise absorption and poultice. Due to the dimensions of samples and the requirements to perform reproducible treatment procedures for laboratory testing, the capillarity method is specified. Where a treatment cannot be applied according to the standard method (for example when an emulsion is used) the application method should be clearly described in the test report.

Technical and chemical data sheets of treatment under evaluation should be provided; the data sheets which detail at least the chemical formulas of the active substances and concentrations, the names and the ratio of solvents, if applicable.

In order to evaluate the durability and in service performance of a water repellent product applied on the substrate, ageing tests representing the environment in which the porous inorganic material is located can be carried out.

## 1 Scope

This European Standard specifies the methodology for laboratory evaluation of the performance of water repellent products on porous inorganic materials.

It is based on the measurement of several parameters which assess the performance of the product using standard test methods before and after ageing.

Acceptable performance within the laboratory does not constitute a blanket endorsement of application in every situation. The particular context of the heritage object, including such factors as material designation, condition, exposure, salt content and problems related to water ingress requires further investigation.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15801, *Conservation of cultural property - Test methods - Determination of water absorption by capillarity*

EN 15802, *Conservation of cultural property - Test methods - Determination of static contact angle*

EN 15803, *Conservation of cultural property - Test methods - Determination of water vapour permeability ( $\delta p$ )*

EN 15886, *Conservation of cultural property - Test methods - Colour measurement of surfaces*

EN 15898, *Conservation of cultural property - Main general terms and definitions*

EN 16085, *Conservation of Cultural property - Methodology for sampling from materials of cultural property - General rules*

EN 16302, *Conservation of cultural heritage - Test methods - Measurement of water absorption by pipe method*

EN 16322, *Conservation of Cultural Heritage - Test methods - Determination of drying properties*

EN ISO 2813, *Paints and varnishes - Determination of specular gloss of non-metallic paint films at 20°, 60° and 85° (ISO 2813)*

## 3 Terms and definitions

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

### 3.1

#### **water-repellency**

ability of the substrate to resist the ingress of liquid water

### 3.2

#### **water repellent product**

product increasing the water-repellency of the treated surface of porous inorganic materials

### 3.3

#### **water repellent treatment**

application of a water repellent product to the surface of a material by a specified methodology

- 3.4**  
**protection degree by capillarity**  
reduction of the amount of water absorption by capillarity before and after the treatment
- 3.5**  
**protection degree by pipe**  
reduction of the amount of water absorption by pipe before and after the treatment
- 3.6**  
**specular gloss**  
ratio of the luminous flux reflected from an object in the specular direction for a specified source and receptor angle to the luminous flux reflected from glass with a refractive index of 1,567 in the specular direction

## 4 Symbols and abbreviations

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

- $m_{0B}$  dry mass after characterization tests and before the treatment, in kg;  
 $m_{0A}$  dry mass after the treatment, in kg;  
 $t_i$  time elapsed from the beginning of the test, in s;  
 $Q_i$  water absorbed by capillarity per unit area, in  $\text{kg}/\text{m}^2$ ;  
AC capillary water absorption coefficient, in  $\text{kg}/(\text{m}^2 \cdot \text{s}^{1/2})$ ;  
 $Q_p$  water repellent product absorbed, as a mass %;  
 $PD_{Ci}$  Protection Degree by capillarity at time  $t_i$ , as a mass %;  
 $Q_{Bi}$  amount of absorbed water by capillarity of untreated specimen at time  $t_i$ , in  $\text{kg}/\text{m}^2$ ;  
 $Q_{Ai}$  amount of absorbed water of treated specimen at time  $t_i$ , in  $\text{kg}/\text{m}^2$ ;  
 $\bar{\delta}_p$  water vapour permeability, in  $\text{kg}/(\text{m} \cdot \text{s} \cdot \text{Pa})$ ;  
 $\bar{\delta}_{pB}$  water vapour permeability before treatment, in  $\text{kg}/(\text{m} \cdot \text{s} \cdot \text{Pa})$ ;  
 $\bar{\delta}_{pA}$  water vapour permeability after treatment, in  $\text{kg}/(\text{m} \cdot \text{s} \cdot \text{Pa})$ ;  
 $\bar{\delta}_{p\text{red}}$  reduction of water vapour permeability, as a %;  
 $L^*$  lightness coordinate. The scale for  $L^*$  ranges from 0 (black) to 100 (white);  
 $a^*$  red/green coordinate, with  $+a^*$  indicating redness and  $-a^*$  indicating greenness;  
 $b^*$  yellow/blue coordinate, with  $+b^*$  indicating yellowness and  $-b^*$  indicating blueness;  
 $L^*_B$  Lightness coordinate before treatment;  
 $L^*_A$  Lightness coordinate after treatment;  
 $a^*_B$  red/green coordinate before treatment;  
 $a^*_A$  red/green coordinate after treatment;  
 $b^*_B$  yellow/blue coordinate before treatment;  
 $b^*_A$  yellow/blue coordinate after treatment;  
 $\Delta E^*$  total colour difference;  
 $PD_{LP}$  Protection Degree by pipe method, as a %;  
 $(W_f)_B$  amount of absorbed water by pipe method at the end of the test before the treatment, in  $\text{ml}/\text{cm}^2$ ;  
 $(W_f)_A$  amount of absorbed water by pipe method at the end of the test after the treatment, in  $\text{ml}/\text{cm}^2$ ;



$D_{1B}$	drying <sub>2</sub> rate corresponding to the first drying phase before the treatment, in $\text{kg}/(\text{m}^2 \cdot \text{h})$ ;
$D_{1A}$	drying <sub>2</sub> rate corresponding to the first drying phase after the treatment, in $\text{kg}/(\text{m}^2 \cdot \text{h})$ ;
$D_{1\text{red}}$	reduction of the drying rate, as a %;
$\theta$	contact angle, in °.

## 5 Test methods for evaluation

The methods listed here below are used for the evaluation of a water repellent product in relation to the possible changes of the characteristics of porous inorganic materials.

- 1) Determination of water absorption by capillarity (EN 15801)
- 2) Determination of static contact angle (EN 15802)
- 3) Determination of water vapour permeability (EN 15803)
- 4) Colour measurement of surfaces (EN 15886)
- 5) Determination of water absorption by pipe method (EN 16302)
- 6) Determination of drying properties (EN 16322)
- 7) Gloss measurement (EN ISO 2813)

## 6 Evaluation of long term performances

Water repellents are expected to demonstrate suitable durability on exposure to natural weathering or laboratory tests (such as light irradiation, freeze and thaw, wetting and drying, thermal cycles, salt crystallization, pollutant simulation, biological growth, etc.) representing the environment in which the object is located.

In the event that a specific methodology for the assessment of environmental degradation is standardised in the field of conservation of cultural heritage, it shall be used. Where these do not exist, a suitable assessment method for Cultural Heritage should be used and described in the test report.

It is important to monitor the performances of water repellent products after ageing repeating the tests listed in Clause 5, taking into account that different comparisons of the results are possible (i.e. after ageing-before treatment or after ageing-before ageing, etc...).

The comparison of the results allow to evaluate the durability of products.

## 7 Test equipment for the treatment (apparatus and reagents)

### 7.1 Crystallization vessel

7.2 **Bedding layer** such as filter paper, foam or cotton.

7.3 **Chronometer** with an accuracy of 1 s.

7.4 **Ventilated oven** which can maintain a temperature of  $(40 \pm 2)$  °C.

7.5 **Analytical balance** with an accuracy of at least 0,01 g.

- 7.6 Linear measuring device (calliper)** with an accuracy of 0,1 mm.
- 7.7 Chamber** capable of maintaining a constant temperature of  $(23 \pm 2)$  °C.
- 7.8 Sand paper** with grain size of 82  $\mu\text{m}$  (corresponding to grit number P180 according to the FEPA <sup>1)</sup> classification).
- 7.9 Desiccator** filled with desiccant such as self-indicating silica gel or other drying agents.
- 7.10 Deionised water** (with max. conductivity of 6  $\mu\text{S/cm}$ ).
- 7.11 Soft bristle brush**
- 7.12 Ammonium chloride saturated solution**, or a climatic chamber capable of maintaining a relative humidity of  $(80 \pm 5)$  %.
- 7.13 Magnesium nitrate saturated solution**, or a climatic chamber capable of maintaining a relative humidity of  $(53 \pm 5)$ %.
- 7.14 Glass spheres or bars** (e.g. 3 mm to 4 mm diameter).

## **8 Test procedure**

### **8.1 Steps of evaluation of water repellent products**

The evaluation of water repellent products applied on porous inorganic materials should be carried out according to the following steps (Figure 1).

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<sup>1)</sup> Federation of European Producers of Abrasives

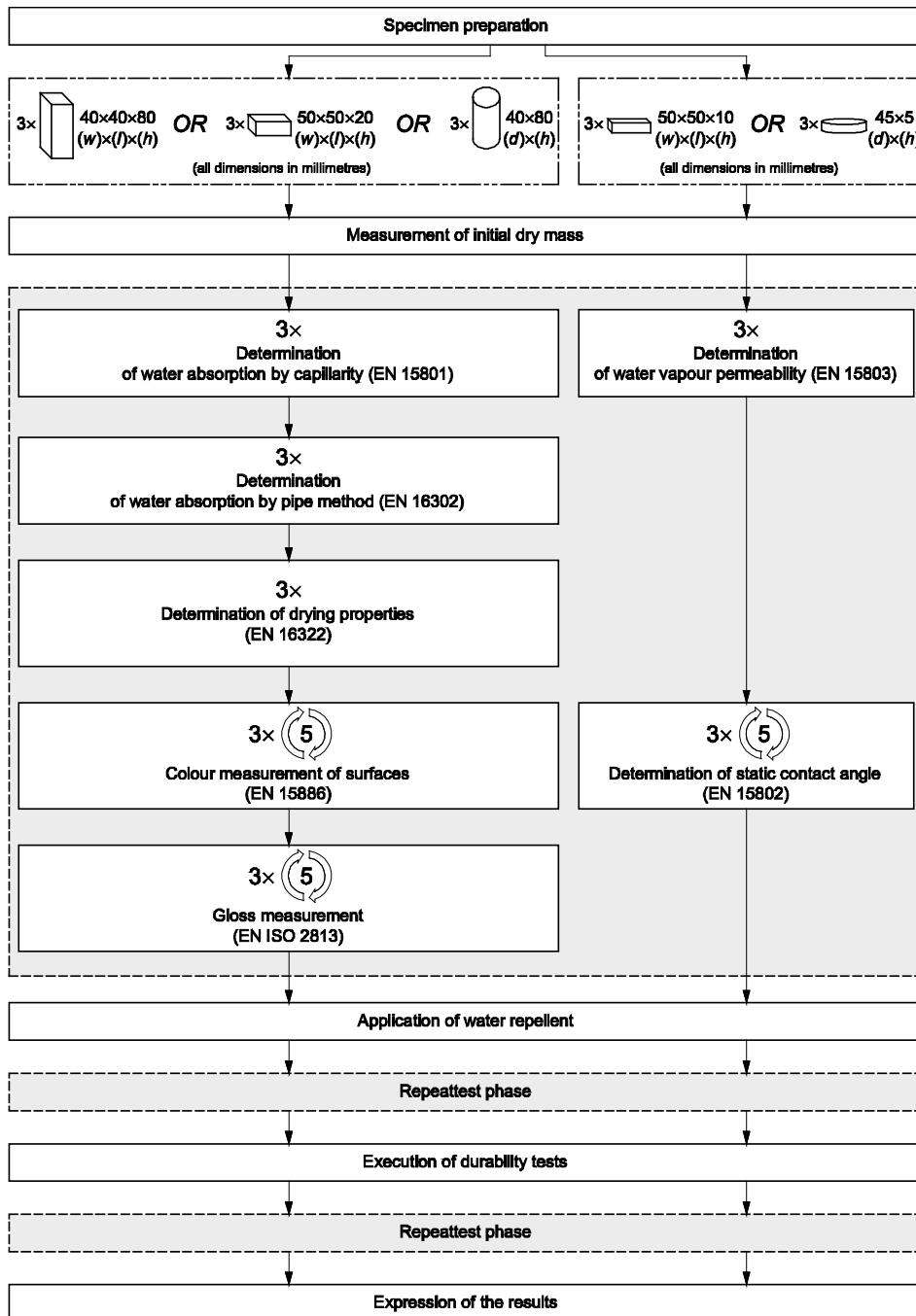










Figure 1 — Flow diagram for recommended test procedure

## 8.2 Specimen preparation and determination of dry mass

### 8.2.1 Number and dimensions of the test specimens

Specimens shall have a regular shape such as cubes, parallelepipeds or cylinders (see Table 1).

**Table 1 — Number and dimensions of the test specimens**

Test method	Characteristics of test specimens		
	Test specimens (numbers of measurements for each test specimen)	Shape	Dimensions mm
Determination of water absorption by capillarity	3	 OR 	50 × 50 × 20 OR 40 × 40 × 80 OR 40 × 80
Determination of static contact angle	3 (5 measurements on each test specimen)	flat surface	-
Determination of water vapour permeability	3		50 × 50 × 10
			45 × 5
Colour measurement of surfaces	3 (5 measurements on each test specimen)	flat surface	-
Determination of water absorption by pipe method NOTE Supplied dimensions relate to the use of vertical pipe.	3	 OR 	50 × 50 × 20 OR 40 × 40 × 80 OR 40 × 80
Determination of drying properties	3	 OR 	50 × 50 × 20 OR 40 × 40 × 80 OR 40 × 80
Gloss measurement	3 (5 measurements on each test specimen)	flat surface	-

In the case of non-homogeneous materials, such as mortars, containing coarse aggregates, the dimensions shall be at least three times (and preferably five times) the largest grain size.

The number of specimens is also dependent on the heterogeneity of the material. In case of anisotropy, the specimens shall always be tested according to the same orientation. All dimensions shall have a  $\pm 0,5$  mm tolerance.

The constraints of sampling material from the cultural heritage may limit sizes and numbers of samples. However, they should be as close to the requirements above as possible.

When evaluating inorganic porous materials from cultural heritage, the sampling shall follow EN 16085.

### 8.2.2 Surface preparation

The surface chosen for the application of a treatment and evaluation of its performance shall be flat and wet or dry polished with sand paper. After polishing, the specimens shall be washed with water, gently brushed with a soft brush and immersed in deionised water for  $30 \pm 5$  min. In case of water sensitive materials, for example gypsum containing materials, only dry polishing and compressed air shall be used.

### 8.2.3 Initial characterization before treatment

Measurements for the initial characterization are carried out before the treatment according to standards listed in Clause 5. Where the listed standards allow a choice of apparatus, the one selected shall be used for both before and after treatment.

### 8.2.4 Measurement of dry mass after characterization tests and before the treatment ( $m_{0B}$ )

After all classification tests have been carried out, the specimens shall be dried to constant mass in a ventilated oven at a temperature of  $(60 \pm 2)^\circ\text{C}$ . 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  $(40 \pm 2)^\circ\text{C}$  until constant mass is reached.

Constant mass ( $m_{0B}$ ) is reached when the difference between two successive weighing at an interval of 24 h is not greater than 0,1 % of the mass of the specimen.

In the case of materials with porosity less than 5 % constant mass is reached when the difference between two successive weighing at an interval of 24 h is not greater than 0,01 % of the mass of the specimen.

## 8.3 Methodology of treatment application

### 8.3.1 General

The application method detailed in following clauses is to be used unless specifically contraindicated by material incompatibility or by an alternative application method specified by the manufacturer. The application method shall be reported and any deviations from the standard have to be described and justified in the test report.

### 8.3.2 Conditioning of the specimens

Before treatment with water repellent products, specimens shall be pre-conditioned for 24 h at a temperature of  $(23 \pm 2)^\circ\text{C}$  and  $(50 \pm 5) \% \text{RH}$ , by using a climatic chamber or a desiccator maintained at  $(23 \pm 2)^\circ\text{C}$  containing a saturated solution of  $\text{Mg}(\text{NO}_3)_2$ .

### 8.3.3 Duration of water repellent application

The duration of treatment depends on the chemical composition and concentration of the product/agent, on the type of the solvent and evaporation rate, and on the porosity of material. Therefore the duration of the treatment shall be established through preliminary tests on specimens and should take into account the recommendations of the manufacturer if any (technical data sheet).

Where specific recommendations do not exist, preliminary tests to determine the duration of the treatment shall be carried out at the following intervals: 1 min, 5 min, 10 min, 30 min, 1 h, 2 h, and the protection degree by capillarity ( $\text{PD}_C$ ), see 10.2, shall be calculated for each individual interval of duration of the treatment.

### 8.3.4 Water repellent application by capillarity and measurement of dry constant mass after treatment ( $m_{oA}$ )

- a) A drying bedding layer (7.2) 1 cm of thickness and the diameter at least 1 cm larger than the maximum size of the test specimen is placed on the bottom of a crystallization vessel (7.1).

- b) The bedding layer has to be saturated to within a few millimetres of the upper surface with water repellent solution. The solution level should not exceed the upper surface of the bedding layer and should be maintained at constant height throughout the test by adding further solution when necessary.
- c) The crystallization vessel is placed in a desiccator containing a saturated solution of  $Mg(NO_3)_2$  and conditioned at  $(23 \pm 2) ^\circ C$  for 1 h or alternatively held for 1 h at  $(23 \pm 2) ^\circ C$  and  $(53 \pm 5) \% RH$ , by using a climatic chamber.
- d) Specimens are laid in contact with the bedding layer with the test face down and in contact with the water repellent solution for the time previously determined (see 8.3.3).
- e) At the end of the treatment specimens are turned (test face up) and placed on small glass balls or glass rods and maintained at  $(20 \pm 2) ^\circ C$  and  $(80 \pm 5) \% RH$ . This environmental condition can be obtained either in a climatic chamber at  $(80 \pm 5) \% RH$  or in a desiccator containing a saturated solution of  $NH_4Cl$ .
- f) Specimens shall be weighed during the stabilization as described in e) after 15 days and successively every seven days until constant mass is reached ( $m_{0A}$ ). Constant mass is reached when the difference between two successive weighing at an interval of 24 h is not greater than 0,01 % of the mass of the specimen ( $m_{0A}$ ). Evaluation of constant mass should be done in the same conditions as for the determination of weight before treatment otherwise the calculation of the amount of product will not be correct (see note).
- g) After the stabilization of treatment the specimens shall be dried in a ventilated oven at a temperature of  $(60 \pm 2) ^\circ C$ . If the material is temperature-sensitive, drying shall be conducted in a desiccator filled with desiccant or in a ventilated oven at a temperature of  $(40 \pm 2) ^\circ C$  until constant mass is reached.

Stabilization of treatment indicates full evaporation of solvents and/or curing reactions have taken place, although guidance from the manufacturer should be taken into account. Constant mass indicates that stabilization has been achieved. Characterization tests should be carried out only when constant mass is reached.

Care should be taken during the application and stabilization of samples to prevent cross contamination, particularly when using volatile molecules as active agents.

## 9 Determination of the amount of water repellent applied

Specimen is weighed before and after the treatment ensuring that complete evaporation of the solvent according to g) of procedure 8.2.3 has occurred.

The amount of water repellent product absorbed ( $Q_p$ ) by the specimen is determined by the following formula as the difference between dry weights before and after the treatment according to:

$$Q_p = \frac{(m_{0A} - m_{0B})}{m_{0B}} \cdot 100 \quad (1)$$

where

- $Q_p$  is the amount of applied product, expressed as a percentage (mass fraction);
- $m_{0B}$  is the constant mass before the treatment (see 8.2.4), in kg;
- $m_{0A}$  is the constant mass after the treatment (see 8.3.4), in kg.

## 10 Measurements and methods for treatment evaluation

### 10.1 Determination of water absorption by capillarity

The water absorption experiment provides information about the material's transport properties for liquid water.

EN 15801 is based on the process of water capillary rise to calculate the water absorption coefficient (AC) and to determine the amount of water absorbed ( $Q_i$ ) at different time. Capillarity measurements are carried out on untreated specimens and repeated after treatments and/or ageing of treated material on the same specimen and measuring the amount of absorbed water at the same time intervals.

Before treatment with water repellent products, specimens shall be pre-conditioned for 24 h at a temperature of  $(23 \pm 2)$  °C and  $(53 \pm 5)$  % RH, by using a climatic chamber or a desiccators maintained at  $(23 \pm 2)$  °C containing a saturated solution of  $Mg(NO_3)_2$ .

The shape of the curve and the inclination of its linear section provide an indication of both the performances of a protective treatment applied on specimen and of the material's characteristics.

Untreated samples initially show a high rate of water absorption followed by the rate of uptake rapidly decreasing towards an asymptotic value.

This value is the maximum amount of water absorbed by a material at atmospheric pressure and is related to the porosity accessible to water.

The different inclination of the curve's initial linear section after treatment shows the decrease of absorption rate, while the curve's second part gives information about the distribution of the product: on the surface or inside the material.

The evaluation of the efficacy of a water repellent is not only based on the AC, but also on the shape of the absorption curve. Very frequently different products have the same AC but show different curve shapes after long testing times.

A greater decrease of AC and  $Q_i$  for a treated specimen with respect to the untreated one indicates a greater capacity of the treatment to give a water repellent property to a specimen. This capacity is calculated by the calculation of Protection Degree (see 10.2).

### 10.2 Protection Degree by capillarity $PD_c$

The effect of the treatment on water absorption at specific times is defined as protection degree and is calculated according to the following formula:

$$PD_{Ci}(\%) = \frac{(Q_{Bi} - Q_{Ai})}{Q_{Bi}} \cdot 100 \quad (2)$$

where

$Q_{Bi}$  is the amount of absorbed water of untreated specimen at time  $t_i$ ;

$Q_{Ai}$  is the amount of absorbed water of treated specimen at time  $t_i$ ;

$PD_c$  shall be measured at elapsed times ( $t_i$ ) of 1, 4, 8, 24 h and at the end point of the test.

The  $PD_{ci}$  assessed over time gives an indication of the resistance to the ingress of liquid water exerted by the water repellent.

Values of PDC close to hundred percent mean that the water repellent applied is efficiently reducing the penetration of liquid water into the substrate.

### 10.3 Determination of water vapour permeability ( $\delta_p$ )

EN 15803 describes the method for measuring the amount of water vapour passing through the specimen over time in static conditions. A flux of water vapour through the specimen occurs when the partial pressure of water vapour differs between the two opposite surfaces of the specimen.

As air temperature strongly influences the difference in vapour partial pressure between specimen surfaces, it shall be strictly controlled. When equilibrium conditions are reached, the density of water vapour flow rate (in kg/m<sup>2</sup> for 24 h) is calculated by measuring the weight loss of the box every 24 h. The equilibrium conditions are generally reached after some days. The test specimen shall be mounted in the cup with the test i.e. treated face up so it is perpendicular to the direction of water vapour flow.

$\delta_p$  measurements are carried out on untreated specimens and repeated after treatments and/or ageing of treated material on the same specimens with the same system (in EN 15803 two different systems are described).

The thickness and the shape of test specimen shall be the same when comparing different water repellents.

### 10.4 Reduction of water vapour permeability ( $\delta_{p,red}$ )

The assessment of the reduction of water vapour permeability  $\delta_{p,red}$  is obtained by comparing the water vapour permeability before ( $\delta_{p,bt}$ ) and after the treatment ( $\delta_{p,at}$ ) at the equilibrium with the following formula:

$$\delta_{p,red}(\%) = \frac{(\delta_{pB}) - (\delta_{pA})}{(\delta_{pB})} \cdot 100 \quad (3)$$

where

$\delta_{p,red}$  reduction of water vapour permeability, as a %,

$\delta_{pB}$  water vapour permeability before treatment,

$\delta_{pA}$  water vapour permeability after treatment,

$\delta_{p,red}$  values close to zero indicate that the water repellent product has a negligible effect on water vapour flow in the specimen.

### 10.5 Determination of static contact angle

EN 15802 is used to assess the degree of water-repellency of a surface.

Determination of static contact angle is carried out on untreated specimens and repeated after treatments and/or ageing of treated material on the same specimens.

The contact angle  $\theta$  of a liquid on a surface is used to estimate the wetting properties of the material by calculating its solid-liquid-vapour surface tension.

An increase of contact angle value is indicative of an increase of water repellency of the surface.

Very frequently contact angle measurements cannot be performed on untreated surfaces due to the fast water absorption by the surface specimen. The same specimens once treated show a measurable contact angle and therefore this can be interpreted as an increase in the contact angle value.

Values lower than 90° show a scarce surface water repellency, while values larger than 90° are indicative of a good surface water repellency. The higher the value the greater the indication of more effective treatment.



## 10.6 Colour measurement of surfaces

The measurement of the surface colour of a specimen is performed according to EN 15886 on untreated specimens and repeated after treatments and/or ageing of treated material on the same specimens.

When the number of readings has been determined, the measuring points for the after-treatment colour measurement shall be localized by reference coordinates in order to ensure precise repetition of the measurement. A grid delimiting the measurement field may be useful for this purpose, depending on specimen dimensions.

The calculation of total colour differences  $\Delta E^*$  between two measurements ( $L^*_B a^*_B b^*_B$  and  $L^*_A a^*_A b^*_A$ ) is the geometrical distance between their positions in the CIELAB colour space. It is calculated according to the formula reported in EN 15886:

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

$\Delta E^*$  values close to zero mean that no colour change has occurred.  $\Delta E^*$  values less than 2 correspond to acceptable changes because they are hardly perceived by the naked eye.

In addition, it is necessary to report the difference for the single colourimetric coordinate calculated as follows:

$$\Delta L^* = L^*_A - L^*_B;$$

$$\Delta a^* = a^*_A - a^*_B;$$

$$\Delta b^* = b^*_A - b^*_B;$$

## 10.7 Determination of water absorption by pipe method

Measurements are carried out on untreated specimens and the measurements repeated after treatment and/or ageing of treated material on the same specimens using the same system (in EN 16302 different systems are described).

This test method is useful for measuring the rate at which water moves through a porous materials. The test is performed by measuring the volume of water absorbed through a defined surface under low pressure and within a specified time. This test can be performed *in situ* or in the laboratory and can be used to measure vertical and horizontal water transport. Penetration of driving rain into wall surfaces results in horizontal transport. Under actual conditions, the rate of rain penetration depends on prevailing wind conditions as well as on the composition and condition of the exposed surfaces.

## 10.8 Protection Degree of water absorption by pipe method $PD_{LP}$

The protection degree at low pressure is evaluated by using the following formula:

$$PD_{LP}(\%) = \frac{(W_f)_B - (W_f)_A}{(W_f)_B} \cdot 100 \quad (5)$$

where

$PD_{LP}$  is the protection degree at low pressure;

$(W_f)_B$  amount of absorbed water at the end of the test before the treatment,

$(W_f)_A$  amount of absorbed water by at the end of the test after the treatment,

It is possible to determine the protection degree in the laboratory as well as *in situ* before and after treatment.

Values of  $PD_{LP}$  close to 100 % mean that the water repellent applied is efficiently reducing the penetration of liquid water into the substrate.

## 10.9 Determination of drying properties

The drying experiment provides important information on the hygric and hydric properties of porous materials and it allows the quantification of the effect of liquid water and vapour transport phenomena through them (EN 16322).

The drying behaviour of porous materials depends on the:

- properties of the material
- boundary conditions (Temperature, Relative Humidity, air velocity).

The material properties influence the rate and volume of moisture transport within the material.

The combination of climatic and transfer conditions, i.e. the boundary conditions, define the rate at which moisture is transferred to the atmosphere.

Evaporation takes place when there is a moisture gradient between the external environment and the material.

The time-dependent weight loss can be divided into two phases:

- The first drying phase (in which the surface is wet) is characterized predominantly by transport of liquid water to the surface followed by evaporation. The drying process is limited by the boundary conditions.
- The second drying phase (in which the surface is dry) is characterized by a decrease in liquid water transport. The increase in water vapour diffusion is limited by material properties.

The time of transition between the first and the second drying phases is called knick-point.

The knick-point occurs when transport of liquid water to the surface is no longer possible and only the less efficient vapour diffusion mechanism remains available.

The assessment of the reduction of the drying rate  $D_{1\text{Bred}}$  is obtained by comparing the drying rate before ( $D_{1\text{B}}$ ) and after the treatment ( $D_{1\text{A}}$ ) with the following formula:

$$D_{1\text{red}}(\%) = \frac{D_{1\text{B}} - D_{1\text{A}}}{D_{1\text{B}}} \cdot 100 \quad (6)$$

where

$D_{1\text{B}}$  drying rate corresponding to the first drying phase before the treatment,

$D_{1\text{A}}$  drying rate corresponding to the first drying phase after the treatment,

$D_{1\text{red}}$  reduction of the drying rate, as a %,

Low  $D_{1\text{red}}$  values indicate that the water repellent product gives a low interference to the drying behaviour.

When comparing different products, for the same protection degree, a lower  $D_{1\text{red}}$  indicates less detrimental change.

## 11 Test report

### 11.1 General information

- a) reference to this European Standard;
- b) the name and address of the principal test laboratory;
- c) the name and address of the location at which treatments and tests were carried out, where different from that stated in b);
- d) type, name, provenance, description of the porous inorganic material including chemical, petrographic, mineralogical and physical characteristics (if available), in accordance with existing standards;
- e) number, shape and dimension of each set of samples used for the different performed tests; specify the treated surface if any orientation of anisotropy is present. In case of more than one treatment specify the number of specimens for treatment;
- f) identification of the water repellent/s detailing the chemical formulas of the active substances and concentrations, the names and the ratio of solvents, if applicable normally derived from technical and chemical data sheets;
- g) description of the water repellent application method used in the test, including application times and stabilization times;
- h) the dry mass after characterization tests and before the treatment;
- i) the dry mass after the treatment;
- j) the amount of water repellent remained after stabilization (i.e. evaporation of solvent and or curing);
- k) date of treatment and testing (yy-mm-dd).

### 11.2 Determination of water absorption by capillarity

- a) date of testing (yy-mm-dd);
- b) capillary water absorption coefficient (AC) before and after the treatment;
- c)  $Q_i$  values and the time intervals ( $t_i$ ) before and after the treatment or the graphs of  $Q_i$  values as a function of square root of time  $t_i^{(1/2)}$
- d)  $Q_{Bi}$ , the amount of absorbed water of untreated specimen at times  $t_i$ ;
- e)  $Q_{Ai}$ , the amount of absorbed water of treated specimen at times  $t_i$ ;
- f)  $PD_{Ci}$  Protection Degree by capillarity at time  $t_i$ ;

### 11.3 Determination of water vapour permeability

- a) date of testing yy-mm-dd;
- b) test environmental conditions (T, RH) and the cup system selected;
- c)  $\delta_{pB}$  the water vapour permeability before treatment;

- d)  $\bar{\delta}_{pA}$  the water vapour permeability after treatment;
- e)  $\bar{\delta}_{pred}$  the reduction of water vapour permeability;

#### 11.4 Colour measurement of surfaces

- a) date of testing (yy-mm-dd) technical specification of the geometry, standard observer illuminant, and whether the specular component is excluded, the colour data set ( $L^*_B$ ,  $a^*_B$ ,  $b^*_B$  and  $L^*_A$ ,  $a^*_A$ ,  $b^*_A$ ) before and after treatment;
- b) total colour differences  $\Delta E^*$ ;
- c) the difference for the single colourimetric coordinate  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ;

#### 11.5 Measurement of water absorption by pipe method

- a) date of testing (yy-mm-dd) the type of pipe used and the diameter of the measurement area;
- b) the duration of the test;
- c)  $W_{fB}$  the amount of absorbed water at the end of the test before the treatment;
- d)  $W_{fA}$  the amount of absorbed water at the end of the test after the treatment;
- e) the Protection Degree of water absorption by pipe method;

#### 11.6 Determination of drying properties

- a) date of testing (yy-mm-dd);
- b)  $D_{1B}$  the drying rate corresponding to the first drying phase before the treatment;
- c)  $D_{1A}$  the drying rate corresponding to the first drying phase after the treatment;
- d)  $D_{1red}$  the reduction of the drying rate;

#### 11.7 Determination of static contact angle

- a) dates of testing (yy-mm-dd);
- b) type of instrument and type of micro-pipette (material and volume of micro drop);
- c)  $\theta_B$  B static contact angle data set before treatment;
- d)  $\theta_A$  A static contact angle, after treatment;

#### 11.8 Gloss measurement

- a) date of testing (yy-mm-dd);
- b) the angle of incidence;
- c) the results of the test, as indicated in Clause 10 of the standard EN ISO 2813;
- d) any deviation from the test method specified.

### **11.9 Long term performances after ageing tests**

In case of evaluation of long term performances through ageing test the data obtained after each step of ageing test shall be reported. Regarding the methodology of ageing if it is not standardized a description of the methodology used shall be reported.

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