



SLOVENSKI STANDARD
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Fotokataliza - Kemično ukrepanje proti onesnaženju z uporabo adsorbiranih organskih snovi v pogojih trden/trden - 1. del: Barvila na poroznih površinah

Photocatalysis - Anti-soiling chemical activity using adsorbed organics under solid/solid conditions - Part 1: Dyes on porous surfaces

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Photocatalyse - Activité chimique anti-salissures à l'aide de matières organiques adsorbées dans des conditions solide/solide - Partie 1 : Colorants sur des surfaces poreuses <https://standards.iteh.ai/catalog/standards/sist/e09bc6fb-b3dd-46e7-bda0-b9277c725414/sist-en-16845-1-2017>

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25.220.20 Površinska obdelava Surface treatment

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Photocatalysis - Anti-soiling chemical activity using adsorbed organics under solid/solid conditions - Part 1: Dyes on porous surfaces

Photocatalyse - Activité chimique anti-salissures à l'aide de matières organiques adsorbées dans des conditions solide/solide - Partie 1 : Colorants sur des surfaces poreuses

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (prEN 16845-1:2015) has been prepared by Technical Committee CEN/TC 386 "PHOTOCATALYSIS", the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

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1 Scope

This European Standard specifies a test method for the evaluation of the photocatalytic self-cleaning performance of materials showing photocatalytic activity, usually based on semiconducting metal oxides such as titanium dioxide, by the measurement under solid/solid conditions of the decolouring ability under irradiation with ultraviolet light (UV-A) of a test sample on which a dye solution is sprayed and dried.

This European Standard is intended for use with opaque and rough surfaces of different kinds, such as construction materials in flat sheet, board or plate shape, that are the basic forms of materials for various applications.

This European Standard also applies to fabric, plastic or composites containing photocatalytic materials that are not soluble in acetone. This European standard does not apply to photocatalytic glass, granular materials (unless they are deposited in compact films or layers over flat solid surface) and flat non porous materials.

The method evaluates only the self-cleaning ability of the material under ultraviolet light irradiation. It cannot be applicable to evaluate other performance attributes of photocatalytic materials, i.e., decomposition of water contaminants in liquid or gas phases contacting the material, and antifogging and antibacterial actions.

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.

CEN/TS 16599:2014, *Photocatalysis — Irradiation conditions for testing photocatalytic properties of semiconducting materials and the measurement of these conditions*

EN ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in *Glossary of Photocatalytic Terms CEN/TC386 WI00386014* and the following apply.

- 3.1**
self-cleaning
ability of a material to maintain clean or to clean itself if soiled on its surface
- 3.2**
photocatalytic self-cleaning
self-cleaning ability of a material as a consequence of the irradiation of the material surface with UV-Vis-IR radiation
- 3.3**
spraying distance
distance from the outlet of the spraying gun (see experimental setup) and the surface of the test sample
- 3.4**
Covered Area (CA)
area of the sample where the colour intensity is $\geq \exp(-2) \approx 13,5\%$ of the maximum intensity
- 3.5**
Dirt Parameter (DP)
measurement of the dye amount spread or persistent over the sample surface

3.6

Dirt Parameter (DP) Calibration Function

mathematical function that describes the relation between the Dirt Parameter and the amount of dye spread over the sample surface

4 Symbols and abbreviations

a, b, c	polynomial constant parameters obtained by the fit using Formula.(7);
$A_{\text{net}}(\lambda)$	Net Absorbance of the dye covering of the sample surface at the wavelength λ ;
CA	Covered Area (cm^2);
C_{dye}	concentration of the dye in the spraying solution (g cm^{-3});
d_{ac}	density of acetone at the temperature of the measurement (g cm^{-3});
DP	dirt parameter (nm);
DR	deposition rate ($\text{g sec}^{-1} \text{cm}^2$);
f	volumetric spraying flow ($\text{cm}^3 \text{sec}^{-1}$);
F_i	mass flow (g sec^{-1});
k_{dye}	first order kinetic constant of the specified dye for the photocatalytic self-cleaning process (min^{-1});
n	number of steps used for deposition of the dye on the sample surface. Typically, $n=5$, but can be larger if the surface excessively wet;
$R(\lambda)$	Spectral Reflectance at the wavelength λ of the sample surface; $R(\lambda)$ has indexes i and j referring to steps of spraying and illumination, respectively;
$R_{\text{background}}(\lambda)$	reflectance of the pristine surface at the wavelength λ ;
SC	standard dye covering defined in Table 2 (g cm^{-2});
T	temperature in $^{\circ}\text{C}$;
t_i	irradiation time (min);
t^{spr}	spraying time (sec), calculated as $t^{\text{spr}} = t_{\text{std}}^{\text{spr}} / n$;
W	the full width at half maximum (FWHM) of the sprayed dye colour peak (cm);
β	average covering of dye at the surface (g cm^{-2}); indexes i and j refer to steps of spraying and illumination, respectively;
β_0	maximum average covering of dye at the surface (g cm^{-2}), obtained at $t_{\text{std}}^{\text{spr}}$;
λ	wavelength (nm);
$t_{\text{std}}^{\text{spr}}$	standard spraying time (sec);
$\tau_{1/2}^{\text{dye}}$	half-life of the dye for the photocatalytic self-cleaning process (min);

5 Principle

This norm concerns the comparison and the quality assurance of photocatalytic materials used as self-cleaning materials. The method described is intended to measure the photocatalytic self-cleaning performance of a photocatalytic material by evaluating its ability to clean its surface, previously covered by a known amount of coloured organic compound, as a consequence of the exposition to ultraviolet light. A controlled amount of a dye solution dissolved in a volatile solvent (acetone) is spread on the tested surface by using a spraying gun.

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The photocatalytic material turns out to be covered by the solid dye. The relation between the amount of the spread dye and the spectrophotometric reflectance is defined in the calibration step. The calibration function involves the measurement of the reflectance spectra of the sample surface as a function of the dyes covering. Dyes used in separate experiments are Metanil Yellow, Rhodamine B, and Methylene Blue. The test shall be carried out with the dye showing the maximum optical contrast with the material to be tested. Criteria for the choice of the best dye are here given (see 8.3.2). Optionally, the test can be carried out with the others dyes as the reactivity of each dye can depend on the specimen. The measurement with more than one dye is encouraged, but it is not compulsory. The self-cleaning activity measured by this test shall be referred to the dye used

The soiled surface is then irradiated in air by UV-A light under defined conditions and the decrease of the dye amount on the surface is monitored by measuring the reflectance spectra of the surface of the test sample in the visible range. By using the calibration function the change of the reflectance spectra can be related to the kinetic of disappearance of the dye from the surface. The photocatalytic self-cleaning performance is determined as the half-life (minutes) of the dyes applied to the surface.

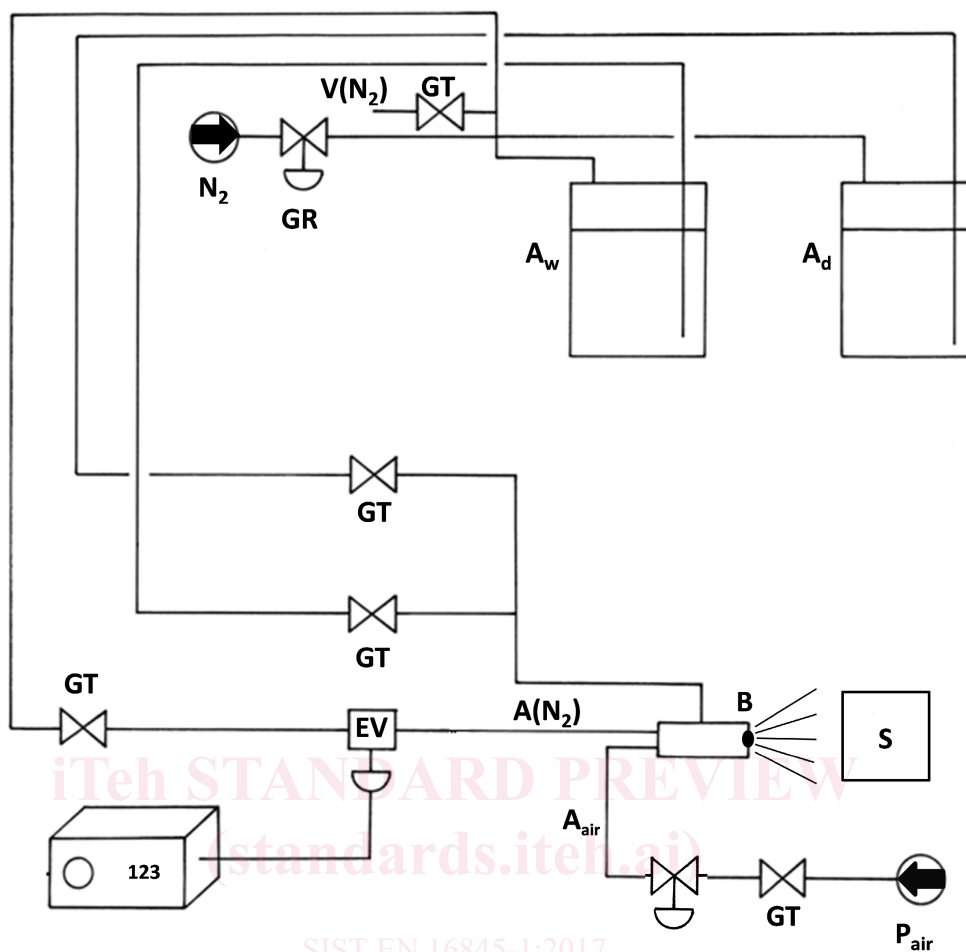
6 Instruments

6.1 Spraying system

The method described in this norm is based on the possibility to cover in a controlled way the sample surface with the dye. A spraying system shall be used to spread over the sample surface a solution of the dye (dye solution) in volatile solvent (acetone). The spraying system consists of a sample support and in a pneumatic system under pressure able to spray for different definite times the dye solution over the sample. The dye solution shall be spread by using a spraying gun that forms a circular spot. This involves a normal (Gaussian) distribution of the amount of dye centred in the spot of the dye on the surface. The amount of solution spread over the test sample is controlled by changing the spraying time (t^{spr}) with a timer that opens and closes, with a precision of $\pm 0,01$ sec, the dye solution flow. The relative distance and orientation between the gun and the sample shall be changed in a way to obtain a symmetric covering of the surface of the test sample and the desired surface covered area. Due to the normal distribution of the colour intensity at the surface test piece, the dye surface covered area is defined as the area of the sample where the colour intensity is $\geq \exp(-2) \approx 13,5\%$ of the maximum intensity (see 8.2). The optimal distance from the gun outlet to the surface of the test sample is referred as the spraying distance.

A sketch of the pneumatic spraying system and of the sample support is shown in Figure 1.

One pressurized ($3,0 \pm 0,1$ bar) bottle containing water and at least one pressurized ($3,0 \pm 0,1$ bar) bottle containing the dye solution (spraying liquids) are connected to a spraying gun by tubes made of materials resistant to the used solvent (for example PTFE (polytetrafluoroethylene)). Also the bottles and gun materials shall be chemically inert to water and acetone. The desired spraying liquid can be selected acting on the corresponding valves. The gun is connected to air and N_2 pressurized lines ($3,0 \pm 0,1$ bar) that supply the atomization and the actuator gases. The actuator line is controlled by an electrovalve connected to a digital timer. The amount of solution spread over the test sample is controlled by changing the spraying time with a digital timer that opens and closes the actuator line with a precision of $\pm 0,01$ sec.



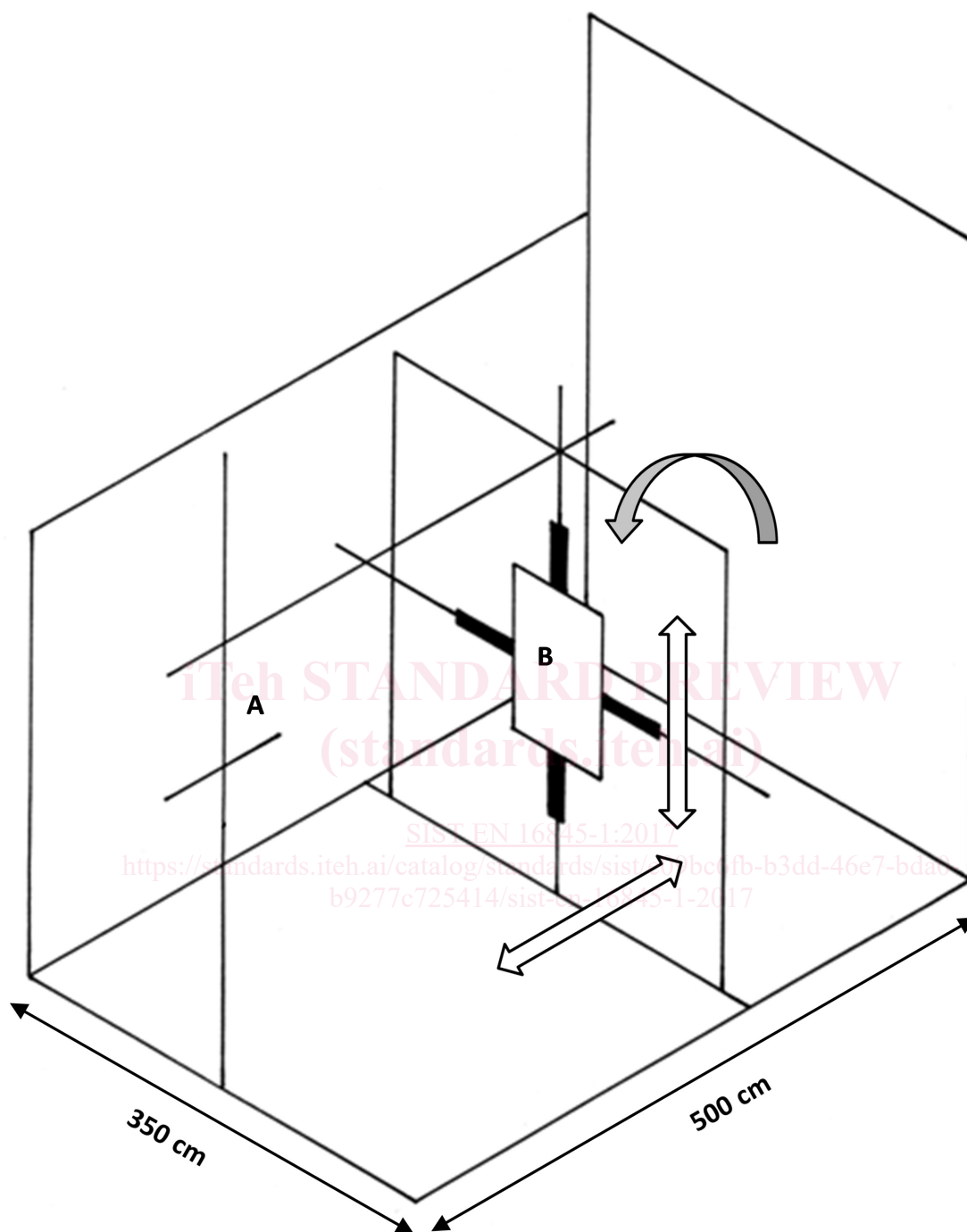
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Key

A (N ₂)	Actuators (N ₂)
A _{air}	Atomization AIR
A	Pressurized bottles
A _w	Water
A _d	Dye/Dyes solution
B	Spraying gun
123	Digital timer
EV	Electrovalve
GR	Gas regulator with manometer
GT	Gastap
P _{air}	Pressurized AIR

Figure 1 — Pneumatic spraying system

**Key**

A spraying gun

B sample

Arrows translation direction of the sample position

Figure 2 — Sample support

The spraying distance between the gun and the sample is typically fixed after setup of the instrumentation (see 8.2); the test samples are placed orthogonally to the spraying flow direction. The spraying gun shall

provide a circular sprayed spot. Using a 0,8 mm fluid nozzle orifice under the given pressure, the typical spray distance is 200 mm. Typically, the fluid flow through the gun shall be regulated to obtain a value close to $0,2 \text{ cm}^3 \text{ sec}^{-1}$. For the accurate measurement of the spraying flow see 8.2.2. The atomization air flow is typically $270 \pm 20 \text{ Ndm}^3 \text{ min}^{-1}$.

The system shall be operated under a ventilated chemical hood. Safety precautions are taken for use of acetone (CAS No: 67-64-1, safety glasses, good ventilation, removal of sources of ignition from the working area).

6.2 Analytical balance

An analytical balance with the precision of 10^{-4} g is used for all the gravimetric operations.

6.3 Diffuse Reflectance Spectrometer

The diffuse reflectance spectra of the sample surfaces shall be measured by using a diffuse reflectance spectrophotometer working in the Vis wavelength range (400-750 nm) excluding the specular component. Any commercial spectrophotometer with integrating sphere accessory can be used, including low cost instruments having wavelength band pass ≤ 3 nm. The reflectance spectra are measured by using a diffuse reflectance standard such as BaSO_4 as a reference of 100% reflectance material.

6.4 Light source

The light source should agree with CEN/TS 16599. It shall provide UV-A irradiation within a wavelength range of 305 nm to 380 nm for a specimen containing TiO_2 . Suitable sources include the so-called black light (BL) and black light blue (BLB) fluorescent lamps, with a maximum at 351 nm or 368 nm, and xenon arc lamps with optical filters that block radiation below 300 nm. In the case of xenon arc lamp, a cooling system shall be used.

The test sample shall be irradiated uniformly. The distance between the light source and the sample shall be adjusted so that the UV irradiance (300 nm to 400 nm) at the sample surface is $20 \pm 0,5 \text{ W m}^{-2}$. A UV radiometer in conformity with CEN/TS 16599 shall be put at the same distance as the surface of the test sample to be tested. The irradiance along the length of the test sample shall also be constant within $\pm 5\%$. The temperature of the sample during the test shall be 25 ± 5 °C.

6.5 Other experimental needs

- a) A bottle with a neck diameter larger than the spot dye spot size, as determined under 2) in 8.2.3.
- b) Ventilated chemical hood.
- c) Sonication bath
- d) Safety glasses.

7 Materials

7.1 Dyes used

Three different dyes can be used in the test (see Table 1). The dyes shall be dissolved in acetone (2-propanone), a volatile organic solvent that allows a perfect solubilisation of the dyes. The dyes have different optical contrast on the test sample depending on their colour. When a dye is chosen, a calibration function shall be performed (see 8.3). Depending on the effective test sample chromatic properties and the obtained calibration function, the proper dye is selected.