SLOVENSKI STANDARD
SIST-TS CEN/TS 16599:2014

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Fotokataliza - Obsevalni pogoji za preskušanje fotokatalitičnih lastnosti polprevodnih snovi in meritve teh pogojev

Photocatalysis - Irradiation conditions for testing photocatalytic properties of semiconducting materials and the measurement of these conditions

Photokatalyse - Bestrahlungsbedingungen zum Prüfen photokatalytischer Eigenschaften von halbleitenden Werkstoffen und die Messung dieser Bedingungen

Photocatalyse - Détermination des conditions d'irradiation pour tester les propriétés photocatalytiques de matériaux semi-conducteurs

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English Version

Photocatalysis - Irradiation conditions for testing photocatalytic properties of semiconducting materials and the measurement of these conditions

Photocatalyse - Détermination des conditions d'irradiation pour tester les propriétés photocatalytiques de matériaux semi-conducteurs

Photokatalyse - Bestrahlungsbedingungen zum Prüfen photokatalytischer Eigenschaften von halbleitenden Werkstoffen und die Messung dieser Bedingungen

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Contents

Page

Foreword

This document (CEN/TS 16599:2014) has been prepared by Technical Committee CEN/TC 386 “Photocatalysis”, the secretariat of which is held by AFNOR.

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Environmental statement

It is understood that some of the material permitted in this standard may have negative environmental impact. As technological advantages lead to better alternatives for these materials, they will be eliminated from this standard to the extent possible.

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Introduction

Photocatalysis is a very efficient advanced oxidation technique which enables the production of hydroxyl radicals (·OH) or perhydroxyl radicals (·OOH), capable of partly or completely mineralising/oxidising the majority of organic compounds. Its principle is based on the simultaneous actions of photons and of a catalytic layer which allows degradation of molecules. The most commonly used photocatalyst is titanium dioxide (TiO_2), the latter being thermodynamically stable, non-toxic and economical. It can be used in powder form or deposited on a substrate (glass fibre, fabrics, plates/sheets, etc.). The objective is to introduce performance standards for photo-induced effects (including photocatalysis). These standards will mainly concern test and analysis methods.

1 Scope

This Technical Specification prescribes the conditions for irradiating photocatalytic surfaces in order to perform photocatalytic efficiency tests. In addition, the measurement and documentation of these irradiation conditions with respect to the spectral distribution, irradiance and homogeneity are given.

2 Symbols and abbreviations

APD	avalanche photodiode
$A(\lambda)$	decadic absorbance
CA	chemical actinometry
E	irradiance
FWHM	full width at half maximum
h_d	height difference
h_{max}	maximum height difference
h_s	measurement plane
LED	light emitting diode
PC-A	photocatalytic amber
PC-B	photocatalytic blue
PC-C	photocatalytic cyan
PC-G	photocatalytic green
PC-R	photocatalytic red
PC-U	photocatalytic ultraviolet
PC-UC	photocatalytic ultraviolet C
PC-V	photocatalytic violet
$QP_{abs}(\lambda)$	total amount of absorbed photons
$q_p^{\circ}(\lambda)$	incident photon flux
λ	wavelength
$\varphi(\lambda)$	quantum yield

In Annex A, further examples concerning literature, terms and definitions, quantities and figures are listed for information.

3 Specification of spectral areas and irradiance values

As shown in Table 1, different spectral areas in combination with the specified irradiance should be used for irradiation during photocatalytical analysis. The test procedures themselves are described in their according standards, e.g. ISO 22197-1 [6] for the abatement of nitrogen monoxide.

Table 1 — Specification of spectral areas and irradiance values

Range	Abbreviation	Colour	Peak nm	$_{max}$FWHM nm	Cut-on-Limit E < 2 % nm	Cut-off-Limit E < 5 % nm	Irradiance [a] W/m^2
UV	*PC-UC*	Ultraviolet C	254 ± 5	not defined	not defined	not defined	not defined
	PC-U	Ultraviolet	365 ± 5	20	345	385	10,0 (± 10 %)
VIS	*PC-V*	Violet	405 ± 5	15	370	440	9,0 (± 7 %)
	PC-B	Blue	450 ± 5	20	400	495	8,1 (± 5 %)
	PC-C	Cyan	500 ± 5	27	440	560	7,3 (± 5 %)
	PC-G	Green	530 ± 5	30	465	595	6,8 (± 5 %)
	PC-A	Amber	590 ± 5	15	555	620	6,2 (± 5 %)
	PC-R	Red	630 ± 5	15	595	655	5,8 (± 5 %)

NOTE 1 For more information about the definition of UV- and VIS-range see reference [7].

NOTE 2 The above mentioned irradiance values are named as guidelines for the level of irradiance. As well as the unification to use the same photon flux is a suggestion in order to have a valid basis on the same concentration of photogenerated active species with respect to the typical heterogeneous catalytic reactions standing behind photocatalytic reactions. If special photocatalytic measurements need to use different parameters, it is important that these deviations be named and refer to this Technical Specification. The basis of 10 W/m^2 UVA-radiation is a compromise of outside day and night irradiance during the whole year in Central Europe and therefore also a compromise between Northern Europe, e.g. Scandinavia, which usually has less irradiance, and Southern Europe, e.g. Mediterranean Area, which usually has more irradiance. This is the same assumption as to have a compromise between indoor (most of the time lower) and outdoor (most of the time higher) irradiation conditions.

[a] A min. 75 % of the irradiance has to be within FWHM and a min. 93 % of the irradiance has to be within Cut-on- and Cut-off-Limit. The used irradiance values should represent the same flux of photons within the described part of the spectra.

Presently only *PC-U*, *PC-V*, *PC-B*, *PC-C* and *PC-G* are important for photocatalytic applications. *PC-A* and *PC-R* are only important for future innovations in photocatalytic materials, which use these defined wavelengths for photo-oxidation processes. Examples of available and suitable filters and LEDs which fulfil these conditions are shown in A.3 and A.4.

4 Lamp types and filters

4.1 Examples of different lamp types

4.1.1 Xenon lamps

In a pure xenon lamp, the light generation volume is cone-shaped, and the luminous intensity falls off exponentially moving from cathode to anode. Electrons passing through the plasma cloud strike the anode, causing it to heat. Pure xenon short-arc lamps have a "near daylight" spectrum, that is, the light output of the lamp is relatively flat over the entire colour spectrum. All xenon short-arc lamps generate significant amounts of ultraviolet radiation while in operation. Xenon has strong spectral lines in the UV bands, and these readily pass through the fused quartz lamp envelope. Unlike the borosilicate glass used in standard lamps, fused quartz does not attenuate UV radiation. The UV radiation released by a short-arc lamp can cause a secondary problem of ozone generation. Equipment that uses short-arc lamps as the light source shall contain UV radiation and prevent ozone build-up. Many lamps have a low-UV blocking coating on the envelope and are sold as "Ozone Free" lamps. Some lamps have envelopes made out of ultra-pure synthetic, which roughly doubles the cost, but which allows them to emit useful light into the so-called vacuum UV region. These lamps are normally operated in a pure nitrogen atmosphere.

NOTE Xe-Arc-bow lamps show the disadvantage when broader areas than 100*100 mm^2 have to be irradiated homogeneously.

4.1.2 Halogen lamps

A halogen lamp, also known as a tungsten halogen lamp, is an incandescent lamp with a tungsten filament contained within an inert gas and a small amount of a halogen such as iodine or bromine. The combination of the halogen gas and the tungsten filament produces a chemical reaction known as a halogen cycle which increases the lifetime of the filament and prevents darkening of the bulb by redepositing tungsten from the inside of the bulb back onto the filament. Because of this, a halogen lamp can be operated at a higher temperature than a standard gas-filled lamp of similar power and operating life. The higher operating temperature results in light of a higher colour temperature (blue shift). Because of their smaller size, halogen lamps can be used advantageously with optical systems that are more efficient in how they cast emitted light. Like all incandescent light bulbs, a halogen lamp produces a continuous spectrum of light, from near ultraviolet to deep into the infrared.

4.1.3 Fluorescence lamps

A fluorescent lamp or fluorescent tube is a gas-discharge lamp that uses electricity to excite mercury vapour. The excited mercury atoms produce short-wave ultraviolet radiation that then causes a phosphor to fluorescence, producing visible light. A fluorescent lamp converts electrical power into useful light more efficiently than an incandescent lamp. Lower energy cost typically offsets the higher initial cost of the lamp. The lamp fixture is more costly because it requires a ballast to regulate the current through the lamp. While larger fluorescent lamps have been mostly used in commercial or institutional buildings, the compact fluorescent lamp is now available in the same popular sizes as incandescent and is used as an energy-saving alternative in homes.

NOTE 1 The United States Environmental Protection Agency classifies fluorescent lamps as hazardous waste, and recommends that they be segregated from general waste for recycling or safe disposal.

Fluorescence lamps have very different spectral distributions, but typically a combination of three or five different phosphors lead to a three- or five-band fluorescent lamp. Each phosphor used gives characteristic emission spectra. Comparable peak wavelengths of the phosphors might also shift during the period of use due to ageing effects (red shift). Some examples for different emission spectra of fluorescence tubes are given in A.6.

NOTE 2 Due to the different emission bands of various fluorescence tubes, a comparison of photocatalytic activities will be very difficult.

4.1.4 Mercury vapour lamps

A mercury vapour lamp is a gas discharge lamp that uses mercury in an excited state to produce light. The arc discharge is generally confined to a small fused quartz arc tube mounted within a larger borosilicate glass bulb. The outer bulb may be clear or coated with a phosphor; in either case, the outer bulb provides thermal insulation, protection from ultraviolet radiation, and a convenient mounting for the fused quartz arc tube. Mercury vapour lamps (and their relatives) are often used because they are relatively efficient. Phosphor coated bulbs offer better colour rendition than either high- or low-pressure sodium vapour lamps. Mercury vapour lamps also offer a very long lifetime, as well as intense lighting for several special purpose applications.

NOTE Hg-low-pressure lamps are only usable for PC-UC.

4.1.5 Light emitting diodes (LED)

A light-emitting diode is a semiconductor light source. LEDs are used as indicator lamps in many devices and are increasingly used for other lighting. When a light-emitting diode is switched on, electrons are able to recombine with electron holes within the device, releasing energy in the form of photons. This effect is called electroluminescence and the colour of the light (corresponding to the energy of the photon) is determined by the energy gap of the semiconductor. A LED is often small in area (less than 1 mm^2), and integrated optical components may be used to shape its radiation pattern. LEDs present many advantages over incandescent light sources including lower energy consumption, longer lifetime, improved robustness, smaller size, faster switching, and greater durability and reliability. LEDs powerful enough for room lighting are relatively expensive and require more precise current and heat management than compact fluorescent lamp sources of comparable output.

NOTE Light emitting diodes are available in all spectral ranges defined in Clause 3.

4.1.6 Sunlight

Sunlight, in the broad sense, is the total frequency spectrum of electromagnetic radiation emitted from sun. On earth, sunlight is filtered through the earth's atmosphere, and solar radiation is obvious as daylight when the sun is above the horizon. When the direct solar radiation is not blocked by clouds, it is experienced as sunshine, a combination of bright light and radiant heat. When it is blocked by the clouds or reflects off other objects, it is experienced as diffused light. The World Meteorological Organization uses the term "sunshine duration" to mean the cumulative time during which an area receives direct irradiance from the sun of at least 120 W/m^2. Sunlight may be recorded using a sunshine recorder, pyranometer or pyrheliometer. Bright sunlight provides irradiance of approximately 700 W/m^2 to 900 W/m^2 at the earth's surface. Sunlight is a key factor in photosynthesis, a process vital for life on earth.

NOTE Sunlight is not applicable for testing due to fluctuations in intensity, weathering and changes of angle of incidence.

4.2 Controlling of the ageing behaviour of the used lamp

Lamps have to be checked every 500 h of operation regarding the spectral distribution and the output power (see also irradiance level in Table 1). If there is a shift, e.g. for fluorescence tubes, of more than 10 nm, lamps have to be replaced by new ones. If there is a loss in output power, lamps also have to be replaced by new ones or if it is possible the output power has to be adjusted by changing the distance between the lamp and the sample.

4.3 Filters

4.3.1 Cut-on/Cut-off-filters for irradiation of large areas

A long-pass-filter (cut-on-filter) is a coloured glass or a plastic foil filter that attenuates shorter wavelengths and transmits (passes) longer wavelengths over the active range of the target spectrum (ultraviolet, visible, or infrared). In contrast, a short-pass-filter (cut-off-filter) blocks longer wavelengths and transmits shorter wavelengths. Long-pass-filters and short-pass-filters, which can have a very sharp slope (referred to as edge filters), are described by the cut-on respectively cut-off wavelength at 50 % of peak transmission.

4.3.2 Band-pass-filters for irradiation of small areas

A band-pass-filter is mainly an interference filter which works to mask out frequencies that are too low or too high, giving easy passage only to frequencies within a certain range. The largest available commercial dimensions for band-pass-filters are limited to (50 × 50) mm^2. Dimensions for customers' specifications are very expensive in manufacturing. Therefore, a combination of a long-pass- and a short-pass-filter could simulate a band-pass-filter with respect to the possibility of irradiating larger areas.

4.3.3 Interference filters

Interference is a characteristic of the wave nature of electromagnetic radiation. Two or more coherent wave trains of the same wavelength and polarisation state that are superimposed enhance or compensate each other, depending on the phase relationship and amplitudes of the electric field strength. These filters utilize the interference effect to transmit or reflect certain spectral ranges of the electromagnetic radiation. Hereto numerous thin layers with differing refractive indices are applied to a substrate. The optical thicknesses of these layers are usually a quarter of a given design wavelength or multiples thereof. When electromagnetic radiation encounters such a multilayer system, the incident beam is split at every interface between two layers of differing refractive indices into a transmitted and a reflected beam. This process is repeated at every successive interface, resulting in the formation of numerous superimposing secondary beams that give rise to interference, either in a constructive or a destructive manner. A wide variety of spectral characteristics with high transmission or high reflection ratings can be produced by varying the nature, number, thicknesses and order of the layers. The coatings of the interference filters are manufactured by the process of vapour deposition under high vacuum. In the case of so-called "soft" coatings, additional measures are normally taken to protect the filters from damage e.g. by handling or from moisture. This is usually achieved by supplementary cementing with suitable glass. The upper temperature limit for these filters is essentially determined by the nature of the optical cement used. Within certain areas of the UV spectrum, it is not possible to use optical cements due to the inherent absorption involved. In such cases, the coated substrates are fitted into appropriate mounts and protected by suitable glasses. In the case of so-called "hard" coatings, the layers of which normally consist of very stable metal oxides, there is generally no need for additional protection. Depending on the substrate selected, interference filters with hard coatings can be operated at temperatures up to approximately 350 °C.

5 Diffusers

Commonly used diffuser technologies include prismatic glass integrating bars, ground glass, opal glass, holographic diffusers and diffractive diffusers. Prismatic glass integrating bars, though sometimes used in high end systems, are limited in capability, are expensive, and occupy a great deal of precious space. Ground and opal glass scatter light equally in all directions but offer limited light-control capabilities. In addition, efficiency is often very poor with these simple diffusers. A holographic diffuser is a step ahead of these diffusers and enables the production of simple light distribution patterns. Holographic diffusers, however, have limited control over the light distribution pattern. In general, only round or elliptical patterns can be produced and only with non-uniform intensity variation, typically of a Gaussian nature. In terms of general beam shaping capability, diffractive elements can shape an input beam arbitrarily. These are mostly limited to monochromatic applications with coherent light sources. Diffractive elements are also limited to narrow diffusion angles due to fabrication limitations, can be strongly sensitive to input beam variations, and present the well-known problem of zero order, a bright spot co-linear with the incident beam. In many applications, the zero order is unacceptable and the requirement of single wavelength operation is very restricting. Diffusers are found in many applications where a bright light source is used to create uniform irradiation over a broad area. Applications include outdoor lighting, rear projection televisions, and consumer electronic displays. Traditional ground or opal glass diffusers are inefficient and have limited capabilities when it comes to controlling the shape of the irradiated area. More modern holographic diffusers can typically be made so that round or elliptical areas are irradiated but the intensity of the radiation is usually only Gaussian within a small angular width. In addition, holographic diffusers are often limited to monochromatic applications using coherent light.

6 Measuring systems

6.1 General

The used physical measuring systems have to be calibrated in the same range of later use, where the irradiation sources are available. All incident radiation shall not be cosine function corrected with respect to the incident angle.