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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials under indoor lighting environment — Measurement of water contact angle

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National foreword

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials under indoor lighting environment — Measurement of water contact angle

Céramiques techniques — Méthode d'essai relative aux propriétés autonettoyantes des matériaux photocatalytiques semiconducteurs dans un environnement d'éclairage intérieur — Mesurage de l'angle de contact de l'eau

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

INTERNATIONAL STANDARD ISO 19810:2017(E)

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials under indoor lighting environment — Measurement of water contact angle

1 Scope

This document specifies a test method for the determination of the self-cleaning performance of sheet-form materials that contain an indoor-light-active photocatalyst or have indoor-light-active photocatalytic films on the surface, under indoor lighting environment.

This method is used to measure the change of water contact angle under indoor lighting environment, which is one of the indices reflecting the self-cleaning performance of semiconducting photocatalytic materials.

This document is not applicable to permeable materials on which water droplets cannot hold and rough materials which obscure water droplets. This document is not applicable to materials of which the changes in the water contact angle due to decomposition of adhered organic matter cannot be evaluated because even if the surface is clean the water contact angle is remarkably large or the water contact angle cannot be sufficiently increased by attaching organic matter to the surface.

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 10677, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ultraviolet light source for testing semiconducting photocatalytic materials*

ISO 14605, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Light source for testing semiconducting photocatalytic materials used under indoor lighting environment*

ISO 27448, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials — Measurement of water contact angle*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 27448 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
photocatalyst
material in which or on which the photocatalyst is added by coating, impregnation, mixing, etc.

Note 1 to entry: Materials include ceramic, metal, plastic, paper, cloth, etc. for general purposes.

3.2
photocatalytic materials
material in which or on which the photocatalyst is added by coating, impregnation, mixing, etc.

3.3
semiconducting photocatalyst
substance that displays photocatalytic action based on its electronic band structure

Note 1 to entry: This applies to metal oxides like titanium dioxide, and sulfides. Photocatalysts which are not semiconducting includes metal complexes.

3.4
self-cleaning effect
maintenance of surface cleanliness of a material by employing a photocatalyst loaded onto the surface

Note 1 to entry: Self-cleaning using photocatalysis is achieved through decomposition of surface contaminants by redox reactions, and/or hydrophilicity that allows stains or dirt to be easily removed by the flow of (rain) water over the surface.

Note 2 to entry: Examples include glass, tiling and other facings for buildings, and plastics and coatings for general purposes.

3.5
indoor lighting environment
indoor lighting environment with an artificial light source for general lighting service that does not include sunlight

Note 1 to entry: For the purposes of photocatalytic activity characterization, a clear definition of spectral range and intensity is normally required.

3.6
indoor-light-active photocatalyst
substance that carries out many functions based on oxidization and reduction reactions produced by an artificial light source for general lighting service, including decomposition and removal of air and water contaminants, deodorization, and antibacterial, antifungal, self-cleaning and antifogging actions

3.7
contact angle before pretreatment
θ_1
water contact angle before pretreatment by UV irradiation and coating with organic matter

3.8
contact angle after UV irradiation and before coating
θ_2
water contact angle after pretreatment by UV irradiation and before coating with organic matter

3.9
initial contact angle
θ_3
water contact angle after pretreatment by UV irradiation and coating with organic matter and immediately before starting visible light irradiation (water contact angle after 0 h of visible light irradiation)

3.10
contact angle after *n* h of visible light irradiation
$\theta_4(n)$
water contact angle after applying visible light irradiation for n h

Note 1 to entry: The unit of time may also be in days, minutes, and seconds in addition to hours.

3.11
initial contact angle halving time
$n_{1/2}$
time required for water contact angle to reach half the value of the initial contact angle θ_3 due to visible light irradiation

3.12
contact angle reduction time (10°)
$n_{10°}$
time required for water contact angle to reach 10° due to visible light irradiation

3.13
test piece set
multiple test pieces of the same material, treated under the same conditions, to investigate time-series changes in a water contact angle by sequential measurement under identical visible light irradiation conditions

4 Principle

This test method measures the time until a water contact angle increased by attaching organic matter to a test piece is reduced due to decomposition of the organic matter by the photocatalytic effect of visible light irradiation, thus provides an index of the self-cleaning performance of an indoor-light-active photocatalytic material. First, the test piece is irradiated with UV light to remove any organic matter adsorbed to its surface, and organic matter for test purposes (stearic acid) is then applied to the test piece by a previously established method. Next, the initial contact angle is measured, and the test piece is then irradiated with a given amount of visible light. The time-series changes in the contact angle due to visible light irradiation are measured, and the elapsed time from the start of visible light irradiation until the contact angle reaches half of the initial value and until the contact angle reaches 10° or lower are determined.

5 Test apparatus

5.1 Instruments and apparatus

5.1.1 Black light blue fluorescent lamp, as specified by ISO 10677.

NOTE In general, the lamp recommended for use is an ultraviolet fluorescent lamp which produces ultraviolet rays termed UVA and has a peak emission at 351 nm, employing blue glass which absorbs visible light.

5.1.2 Ultraviolet light irradiation apparatus, as specified by ISO 27448.

5.1.3 Ultraviolet light radiometer, as specified by ISO 10677.

5.1.4 Visible light source (fluorescent lamp and UV cut filter).

Indoor illumination environment condition (Condition A) shall be used with a cool white halophosphate fluorescent lamp and a UV sharp cut filter designated as Type A from among those specified by ISO 14605, with an attached cover which transmits light longer than wavelengths of 400 nm. Fluorescent lamps shall be warmed up for 15 min before use to stabilize output.

5.1.5 Visible light irradiating apparatus.

To ensure uniform irradiation of test piece sets by light produced by the lamp, allow for blocking of light from surroundings, and allow for adjustment of illuminance, the test piece or the position of the lamp shall be movable. If a lamp reflector is attached, it shall employ a material with little absorption of visible light and degradation under visible light conditions and the structure shall allow for measurement of

illuminance where the test piece is located. Illuminance at the test piece surface shall be adjustable over a threefold or greater range.

5.1.6 Illuminometer, as specified by ISO 14605.

5.1.7 Contact angle measurement apparatus, as specified by ISO 27448.

5.2 Reagents

5.2.1 Stearic acid, of assay (cGC) 60,0 % or higher.

5.2.2 *n*-Heptane, of assay (cGC) 99,0 % or higher.

5.2.3 Water, distilled water or water of equivalent purity.

5.3 Laboratory temperature and humidity

The laboratory should be preferably kept at a temperature (23 ± 5) °C, relative humidity (50^{+20}_{-10}) % or a temperature (20 ± 5) °C, relative humidity (65 ± 10) %. The laboratory temperature and humidity in use shall be documented in the reports of test results.

6 Test piece preparation

Preparation of test pieces shall be as follows.

a) Test pieces: Test pieces shall be prepared by cutting a square 50 ± 2 mm in size from the flat portion of a semiconducting photocatalytic material. During preparation of test pieces, due care shall be taken to avoid contamination by oils or other such organic matter and cross-contamination between semiconducting photocatalytic materials. Test pieces should be taken from the semiconducting photocatalytic material itself, but if the shape of a semiconducting photocatalytic material makes preparation of test pieces difficult, test pieces may be prepared on a separate flat sheet made from the same starting material and processed identically. A single test piece set comprises multiple test pieces prepared from the same material under the same conditions to investigate time-series changes in a water contact angle through sequential measurement of the water contact angle under identical visible light irradiation conditions.

b) Number of test pieces: Each test piece set shall include a sufficient number of test pieces needed to carry out testing. Since the water contact angle is measured at different locations on the test piece surface, a greater number of test pieces are needed if the visible light irradiation time becomes longer and the number of water contact angle measurements increases. The number of test pieces needed can be estimated by preliminary testing and other means in advance. The number of test piece sets needed also corresponds to the number of visible light irradiation levels to be used in testing on two or more levels under visible light irradiation conditions with at least a threefold difference in illuminance.

7 Test procedures

7.1 Measurement of water contact angle

When water droplets are brought into contact with a test piece, water droplets are transferred to the test piece, and liquid droplets are formed. The contact angle at such time shall be measured rapidly, preferably 3 s to 5 s after water dripping. The amount of water dripped shall follow the specification for the contact angle meter used, and measurement is performed with a suitable amount. The value of the contact angle shall be always taken as the arithmetic mean for measurement of contact angles at

three different locations. Measurement shall not be repeated at a location on a test piece surface where the water contact angle has been measured previously. Likewise, if multiple measurements are made on the same test piece, measurement is made at a location sufficiently separated from locations where measurement was made previously, with care taken to obviate effects from previous measurement. Special care shall be needed in cases where the water contact angle is small and water droplets spread on the surface of the test piece.

7.2 Test piece pretreatment

Test pieces shall be pretreated by the following procedures, in which the test piece is irradiated with ultraviolet light to remove any organic matter adsorbed to the surface, and the test piece is then coated with stearic acid. When handling the test piece, care shall be taken to prevent direct contact with the test piece surface, so as to prevent contamination by hydrophobic substances or other such materials. Polyethylene or similar gloves should be worn to protect the test piece from contamination by hydrophobic substances or other such materials.

a) **Measurement of contact angle before pretreatment**, $\boldsymbol{\theta_1}$. For each test piece set, the contact angle at three locations shall be measured before pretreatment of the test pieces. The arithmetic mean of the measured values of contact angles at the three locations measured in this fashion shall be taken as the "contact angle before pretreatment, θ_1" of each test piece set. If multiple, entirely identical test pieces are divided into individual test piece sets, the measured value for a single "contact angle before pretreatment, θ_1" may be used for all such test piece sets.

b) **Preparation of ultraviolet irradiation apparatus**. The light receiving section of a ultraviolet light radiometer shall be installed on the base surface of the ultraviolet irradiation apparatus, and the apparatus shall be adjusted such that irradiance at the test piece surface is (2,0 ± 0,1) mW/cm^2 during use. When measuring irradiance, the light source of the irradiation apparatus shall be warmed up 15 min in advance to stabilize the level of irradiance.

c) **Removal of organic matter by ultraviolet irradiation and measurement of contact angle after UV irradiation and before stearic acid coating**, $\boldsymbol{\theta_2}$. The ultraviolet irradiation apparatus, with irradiance adjusted, shall be used to irradiate each test piece set with UV light for 24 h. Thereafter, the contact angle shall be measured at three locations for each test piece set. The arithmetic mean of the contact angles in the three locations measured for each test piece set shall be then taken as the "contact angle after UV irradiation and before coating, θ_2" of each test piece set. If the contact angle after UV irradiation and before stearic acid coating, θ_2, is not 10° or lower, the test piece set shall be again subjected to UV irradiation for 24 h, and the contact angle after UV irradiation and before stearic acid coating, θ_2, shall be re-measured. This process shall be repeated until the contact angle after UV irradiation and before stearic acid coating, θ_2, is 10° or lower. If the contact angle after UV irradiation and before stearic acid coating, θ_2, does not attain 10° or lower despite repeated UV irradiation, the test is judged invalid. If multiple, exactly identical test pieces have been divided into separate test piece sets and entirely identical ultraviolet irradiation has been performed for each such set, the measured value of a single "contact angle after UV irradiation and before coating, θ_2" may be used for all such test piece sets.

d) **Coating with stearic acid and measurement of initial contact angle**, $\boldsymbol{\theta_3}$. Test pieces shall be coated with stearic acid by the following method. A heptane solution of stearic acid (0,3 wt%) shall be prepared, and each test piece shall be spin-coated with this solution by dripping 1 ml at 2 000 r/min × 20 s, whereafter the test pieces shall be then dried for 10 min at 70 °C. The contact angle at three locations shall be subsequently measured for each test piece set. The arithmetic mean of the contact angles measured at the three locations is determined for each test piece set, and if this value is not 20° or greater, spin-coating with a heptane solution of stearic acid (0,3 wt%) and drying are again performed under the same conditions, and the contact angles shall be re-measured. This process shall be repeated until the arithmetic mean of the contact angle value measured at the three locations reaches 20° or greater, the value at which 20° or greater is attained shall be taken as the "initial contact angle, θ_3" for the test piece set, and the next stage of visible light irradiation can be carried out. If the contact angle does not reach 20° or greater despite repeated spin-coating with stearic acid, the test is judged invalid.

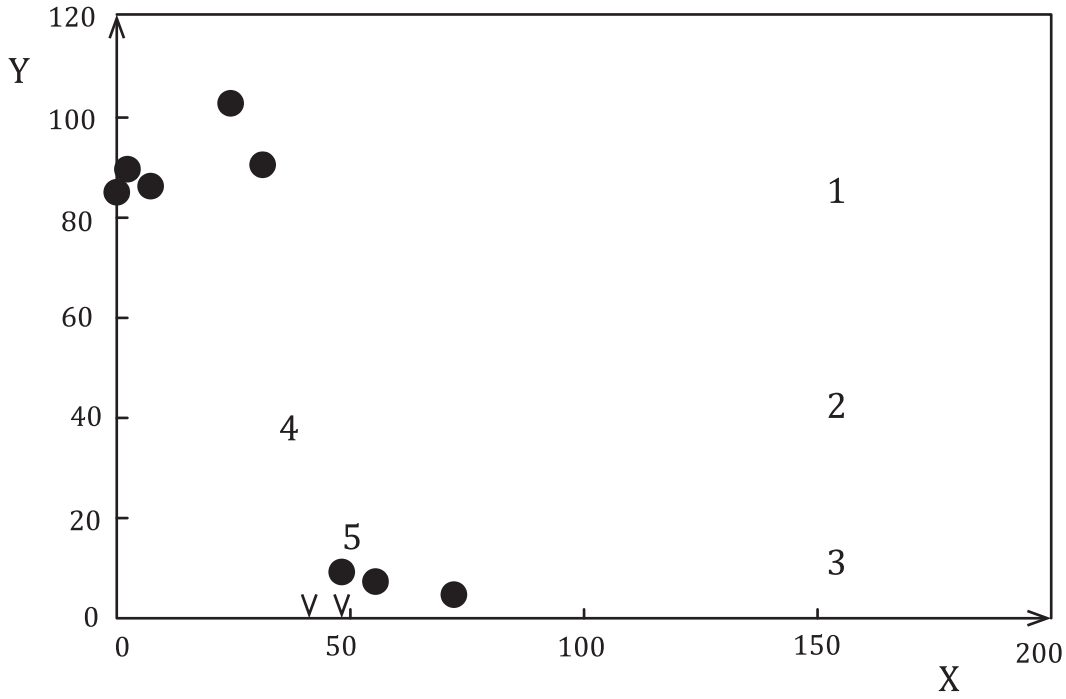
7.3 Visible light irradiation and measurement of contact angle after *n* h of visible light irradiation, $\theta_4(n)$

7.3.1 Measurement of illuminance and preparation of test piece placement location

The light receiving section of an illuminometer shall be installed on the base surface of the visible irradiation apparatus, and the apparatus shall be adjusted such that illuminance at the test piece surface is a predetermined value during use. Testing shall be performed on two or more levels under visible light irradiation conditions with at least a threefold difference in illuminance. When illuminance is measured, the light source of the irradiation apparatus shall be let for 15 min or more in advance to stabilize illuminance.

7.3.2 Measurement of contact angle after *n* h of visible light irradiation, $\theta_4(n)$

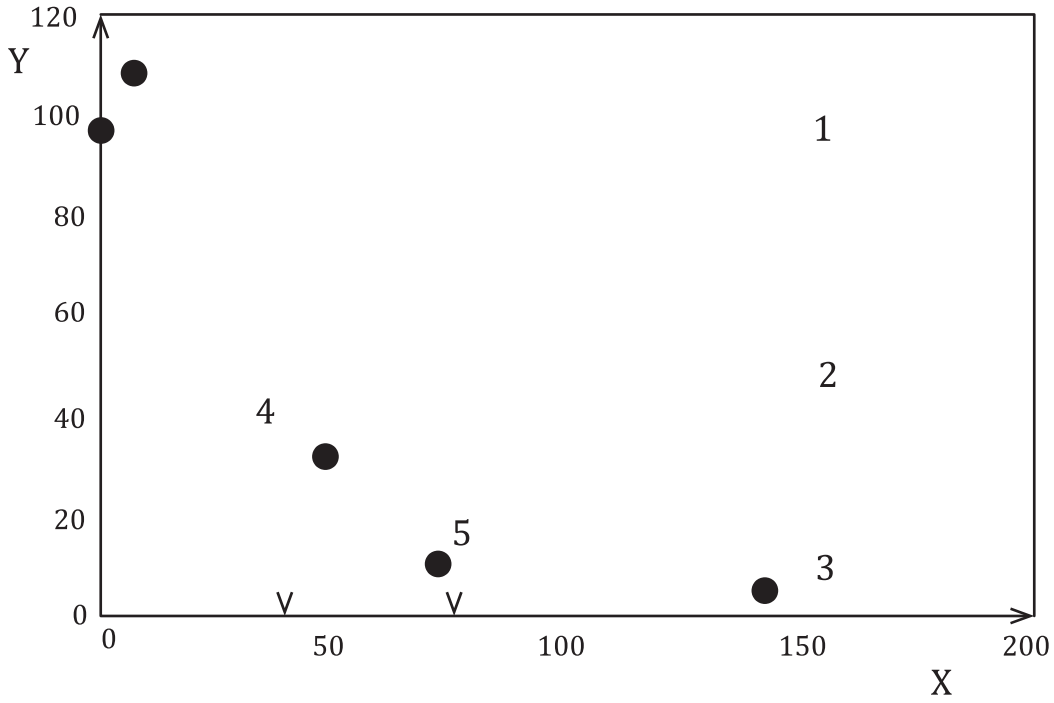
Once visible light irradiation of a test piece set is initiated, an appropriate interval of irradiation shall be allowed, and the contact angle at three locations shall be measured for each test piece set. The arithmetic mean of the contact angle measured at three locations after n h of visible light irradiation shall be taken as the "contact angle after n h of visible light irradiation, $\theta_4(n)$" for each test piece set. Visible light irradiation shall be continued and contact angle measurement is repeated until the contact angle is reduced enough to calculate the initial contact angle halving time, $n_{1/2}$, and the contact angle reduction time, $n_{10°}$. If the contact angle is not reduced to half the value of the initial contact angle and to 10° within the anticipated test duration, the tester shall record the elapsed irradiation time and contact angle when visible light irradiation is stopped, and testing ends. Figures 1 and 2 present measurement examples (2 level of visible light illuminance).



Key

X irradiation time, h
Y water contact angle, degrees
1 dotted line: θ_3 84,9°
2 dotted line: θ_3 /2 42,5°
3 dotted line: 10°
4 arrow: $n_{1/2}$ 41,0 h
5 arrow: $n_{10°}$ 47,9 h

Figure 1 — Measurement example for illuminance of 10 000 lx



Key

X irradiation time, h
Y water contact angle, degrees
1 dotted line: θ_3 96,7°
2 dotted line: θ_3 /2 48,4°
3 dotted line: 10°
4 arrow: $n_{1/2}$ 39,1 h
5 arrow: $n_{10°}$ 76,0 h

Figure 2 — Measurement example for illuminance of 3 000 lx

Though the number of measuring points in these measurement examples is small, testing shall include three or more measuring points between the point where the maximum contact angle is reached and the point where the contact angle becomes 10° or lower by performing preliminary testing or other such procedures in advance (including the point of maximum contact angle and the point at which 10° or lower is reached, five or more points shall be evaluated).

8 Calculation of test results

8.1 Guide to the rounding of numbers

The calculation values are usually rounded to one decimal place.

8.2 Calculation of contact angle

The arithmetic mean shall be determined for contact angles measured at three points for each test piece set, and the result is taken as the “contact angle for the test piece set” under the conditions.

8.3 Conditions for a valid test

When the following conditions for a valid test are fulfilled, the associated test is judged valid. The contact angle after UV irradiation and before stearic acid coating, θ_2, shall be 10° or lower [fulfils Formula (1)].

$$\theta_2 \leq 10° \tag{1}$$

where

θ_2 is the contact angle after UV irradiation and before stearic acid coating.

The initial contact angle, θ_3, shall be 20° or higher [fulfils Formula (2)].

$$\theta_3 \geq 20° \tag{2}$$

where

θ_3 is the initial contact angle (contact angle after 0 h of visible light irradiation).

8.4 Initial contact angle halving time, $n_{1/2}$

The initial contact angle halving time, $n_{1/2}$, shall be calculated by the following formulae.

For each test piece set, when the contact angle is reduced by visible light irradiation and the contact angle at measurement of the contact angle after $n_{-1/2}$ h of visible light irradiation ($\theta_4(n_{-1/2})$) first reaches a size that is not more than half of the initial contact angle, θ_3 [fulfils Formula (4)], if the previous contact angle measurement was made after $n_{+1/2}$ h of visible light irradiation and the contact angle at such time is taken as $\theta_4(n_{+1/2})$, the initial contact angle halving time $n_{1/2}$ is defined by Formula (3), using these values.

$$n_{1/2} = \frac{\left(n_{+1/2} - n_{-1/2}\right) \cdot \frac{\theta_3}{2} + n_{-1/2} \cdot \theta_4(n_{+1/2}) - n_{+1/2} \cdot \theta_4(n_{-1/2})}{\theta_4(n_{+1/2}) - \theta_4(n_{-1/2})} \tag{3}$$

$$\theta_4\left(n_{-1/2}\right) \leq \frac{\theta_3}{2} \tag{4}$$

where

$n_{-1/2}$ is the elapsed time of visible light irradiation (h) at contact angle measurement when the size of the contact angle, θ_4, first reaches half or less that of the initial contact angle, θ_3;

$n_{+1/2}$ is the elapsed time of visible light irradiation (h) at contact angle measurement immediately before contact angle measurement when the size of the contact angle, θ_4, first reaches half or less that of the initial contact angle, θ_3;

$\theta_4(n_{-1/2})$ is the contact angle (°) after $n_{-1/2}$ h of visible light irradiation;

$\theta_4(n_{+1/2})$ is the contact angle (°) after $n_{+1/2}$ h of visible light irradiation;

$n_{1/2}$ is the initial contact angle halving time (h);

θ_3 is the initial contact angle (°).

8.5 Contact angle reduction time, $n_{10°}$

Contact angle reduction time, $n_{10,°}$ shall be calculated by Formula (3), with the following substitutions.

When the contact angle first reaches 10° or lower at the time of contact angle measurement, visible light irradiation time at that time is taken as $n_{-1/2}$, and the contact angle is taken as $\theta_4(n_{-1/2})$. For the contact angle measurement immediately before the measurement above, visible light irradiation time

is taken as $n_{+1/2}$, and the contact angle is taken as $\theta_4(n_{+1/2})$. Assuming that the value of θ_3 at such time is 20°, $n_{1/2}$ calculated by Formula (3) shall be taken as $n_{10°}$.

9 Reporting of test results

Test results shall be reported for the following items:

a) a reference to this document, i.e. ISO 19810;

b) test date, temperature, relative humidity, etc.;

c) test piece type, size, material, and shape;

d) reagent manufacturer name, grade, etc.;

e) black light blue fluorescent lamp model, specification, number of lamps, peak emission wavelength;

f) visible light source (fluorescent lamp and UV cut filter) model, specification, number of lamps, peak emission wavelength;

g) ultraviolet light radiometer model, specification;

h) visible light illuminometer model, specification;

i) contact angle measurement apparatus model, specification;

j) spin coating equipment model, specification;

k) UV light irradiation time during pretreatment;

l) contact angle before pretreatment, θ_1, for each test piece set;

m) contact angle after ultraviolet light irradiation and before stearic acid coating, θ_2, for each test piece set;

n) spin coating conditions (e.g. number of repetitions);

o) initial contact angle ,θ_3, for each test piece set;

p) illuminance of irradiated visible light;

q) contact angle halving time, $n_{1/2}$, for each test piece set;

r) contact angle reduction time, $n_{10°}$, for each test piece set;

s) maximum contact angle after starting visible light irradiation (maximum value of θ_4) and duration of irradiation at such time [if monotone decreasing, $\theta_4(0)$];

t) if contact angle did not decrease to half of the initial contact angle value or to 10° during the test duration, documentation of same, and documentation of irradiation time and contact angle when visible light irradiation was stopped;

u) contact angle after n h of visible light irradiation, $\theta_4(n)$, for each test piece set;

v) noteworthy details concerning test and test pieces after testing;

w) besides the above, changes in the event of changes in test conditions for certain reasons.

10 Test measurement examples

Examples of test results are shown below.

Checking of conditions for a valid test

Figure 1/Table 1 measurement example: Test is validated based on following conditions.

Contact angle after UV irradiation and before stearic acid coating θ_2 = 5,0 → 5,0 ≤ 10, initial contact angle θ_3 = 84,9 → 84,9 ≥ 20

Figure 2/Table 2 measurement example: Test is validated based on following conditions.

Contact angle after UV irradiation and before coating θ_2 = 5,0 → 5,0 ≤ 10, initial contact angle θ_3 = 96,7 → 96,7 ≥ 20

Table 1 — Test results for measurement example in Figure 1 **(Illuminance of visible light irradiated 10 000 lx)**

θ_1	8,0°
θ_2	5,0°
θ_3	84,9°
Maximum of θ_4	102,7°
Irradiation time at maximum of θ_4	24 h
$n_{1/2}$	41,0 h
$n_{10°}$	47,9 h
$\theta_4(2)$	89,3°
$\theta_4(7)$	86,1°
$\theta_4(24)$	102,7°
$\theta_4(31)$	90,2°
$\theta_4(48)$	9,3°
$\theta_4(55)$	7,5°
$\theta_4(72)$	5,0°

Table 2 — Test results for measurement example in Figure 2 **(Illuminance of visible light irradiated 3 000 lx)**

θ_1	8,0°
θ_2	5,0°
θ_3	96,7°
Maximum of θ_4	108,1°
Irradiation time at maximum of θ_4	7 h
$n_{1/2°}$	39,1 h
$n_{10°}$	76,0 h
$\theta_4(7)$	108,1°
$\theta_4(48)$	31,8°
$\theta_4(72)$	10,3°
$\theta_4(142)$	5,0°

Bibliography

[1] ISO 4892-3, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

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