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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for complete decomposition performance of semiconducting photocatalytic materials under indoor lighting environment — Decomposition of acetaldehyde

Céramiques fines (céramiques avancées, céramiques techniques avancées) — Méthode d'essai pour déterminer la performance de décomposition complète des matériaux photocatalytiques semi-conducteurs dans un environnement d'éclairage intérieur — Décomposition de l'acétaldéhyde

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Foreword

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for complete decomposition performance of semiconducting photocatalytic materials under indoor lighting environment — Decomposition of acetaldehyde

1 Scope

This document specifies a test method for the determination of complete decomposition performance of indoor light-active photocatalytic materials under an indoor lighting environment using acetaldehyde. In this document, photocatalytic materials are usually made from semiconducting metal oxides, such as titanium dioxide, tungsten trioxide or other ceramic materials, and they are treated in powder form. This document does not apply to film, flat sheet, board and other plate-shape materials.

This method is not suitable for the determination of other performance attributes of photocatalytic materials, i.e. decomposition of water contaminants, self-cleaning, antifogging and antibacterial actions.

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 4224, *Ambient air — Determination of carbon monoxide — Non-dispersive infrared spectrometric method*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
photocatalyst
substance that performs one or more functions based on oxidation and reduction reactions under photoirradiation, including decomposition and removal of air and water contaminants, deodorization, and antibacterial, self-cleaning and antifogging actions

3.2
indoor lighting environment
environment with artificial light source for general lighting service

Note 1 to entry: Does not include sunlight.

3.3
indoor light-active photocatalytic materials

materials which perform one or more functions based on oxidation and reduction reactions under indoor lighting environment

3.4
zero-calibration gas

air which does not contain pollutants (i.e. common pollutants are below 0,01 µl/l) or carbon dioxide (i.e. carbon dioxide is below 1,0 µl/l.)

Note 1 to entry: The zero-calibration gas is supplied as synthetic air in a gas cylinder.

3.5
standard gas

diluted gases of known concentrations supplied in cylinders and certified by an accredited laboratory

3.6
test gas

mixture of air and pollutant(s) of known concentration prepared from a standard gas or a zero-calibration gas, to be used for the performance test of a photocatalytic material

3.7
dark conditions

test conditions of no light illumination by the light source for testing and room lighting

3.8
complete decomposition

complete mineralization through oxidative decomposition of organic compounds by a photocatalyst, producing carbon dioxide and water

4 Principle

This document has been established to develop, compare and evaluate the characteristics and capabilities of practical indoor light-active photocatalysts.

This document serves as an evaluative index for complete decomposition performance for indoor light-active photocatalytic materials under indoor light irradiation, selects acetaldehyde as the target gas for evaluation and establishes an objective and reproducible test method.

This test entails the use of a photoreactor with a sealable structure provided with a light-transmitting window and a gas inlet/collection port. A photocatalyst serving as a test sample is placed inside the photoreactor, which is then sealed. First, as a pretreatment, the test sample is photoirradiated until production of carbon dioxide ceases. After confirming that production of carbon dioxide has ceased, air in the photoreactor is displaced by a zero-calibration gas. Acetaldehyde gas is then injected into the reactor in a dark location until the concentration of acetaldehyde gas in the reactor reaches 100 ppm. The reactor is left in the dark for 1 h and then irradiated with indoor light. At regular intervals, the gas in the reactor is analysed, and the concentrations of acetaldehyde and carbon dioxide are measured. It is considered complete decomposition when the concentration of carbon dioxide reaches a twofold equivalent of the acetaldehyde injected, and the increase of carbon dioxide ceases simultaneously.

5 Apparatus

5.1 Test equipment

The test equipment is used to evaluate complete decomposition performance of acetaldehyde by a test sample of a photocatalytic powder when test gas is introduced to a photoreactor in which the test sample has been placed and sealed, then irradiated with indoor light. The test equipment consists of the photoreactor, light source, UV-cut filter, acetaldehyde concentration measuring unit and carbon dioxide concentration measuring unit shown below.

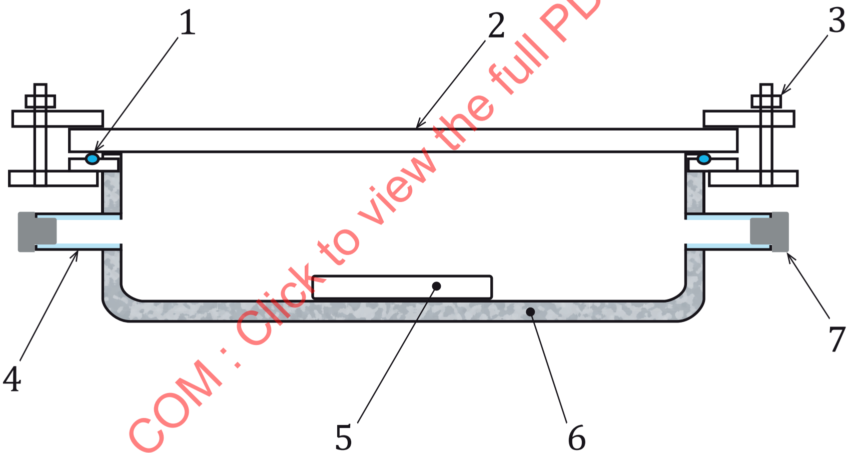
Since the test equipment uses air containing acetaldehyde, consideration shall be given to minimizing adsorptive and other such losses.

5.2 Photoreactor

The photoreactor has a capacity of 500 ml ± 25 ml and a sealable structure provided with a light-transmitting window, and is constructed from a material which adsorbs little acetaldehyde, emits no outgas and can withstand close UV radiation. Glass is a favourable material. The light-transmitting window shall be a quartz glass or borosilicate glass plate of low light absorption in the wavelength range at which photoirradiation is performed. Figure 1 shows an example of a photoreactor.

Gas inlets/collection ports are provided at two or three locations on the photoreactor to collect the gas in the reactor and for air displacement.

The airtightness of the photoreactor is important, and performing the following airtightness test is desirable before using it in this examination. The empty photoreactor is sealed and the air inside replaced with zero-calibration gas (carbon dioxide-free gas). It is then placed in the dark for 24 h. Ideally, the carbon dioxide concentration inside the photoreactor 24 h later should be zero, but if it is less than 5 ppm then the airtightness is good enough for this examination.



Key

1 elastic gasket
2 light-transmitting window
3 clamp holder
4 gas inlet
5 flat dish
6 photoreactor
7 rubber plug

Figure 1 — Example of a sealed photoreactor equipped with a glass window

5.3 Light source

Test samples shall be irradiated uniformly through a UV cut filter and the photoreactor window using a cool white halophosphate fluorescent lamp from among those specified by ISO 14605. The distance from the lamp to the photoreactor is also adjusted such that illuminance on the test sample surface is 10 000 lx. The irradiance shall be measured in illuminance (lx) by using an illuminance meter that has been calibrated by a calibration laboratory.

External light shall be prevented from entering the photoreactor, and the components used around the light source and photoreactor shall have a low or uniform reflectance of 360 nm or higher wavelengths.

5.4 UV cut condition

Irradiation wavelengths can be restricted using a Type A or B sharp cut-off filter specified by ISO 14605.

5.5 Analytical system of acetaldehyde

The concentration of acetaldehyde shall be determined by gas chromatography analysis. Apparatus equipped with a flame ionization detector (FID), or a micro-gas chromatograph equipped with a high-sensitivity thermal conductivity detector (TCD), shall be used. As long as the column is able to separate organic compounds, either a packed column or capillary column can be used. A gas-tight syringe or other such means allowing gas sampling with good reproducibility shall be used.

5.6 Analytical system for carbon dioxide

The concentration of carbon dioxide shall be determined using a non-dispersive infrared carbon dioxide analyser, a gas chromatograph equipped with a methanizer unit and an FID or a micro-gas chromatograph equipped with a high-sensitivity TCD. Calibration of the system shall be done in accordance with ISO 4224. In the case of gas chromatography, the test gas shall be sampled as described in 5.5.

6 Test sample

The test sample shall be photocatalytic powder in an amount of 0,3 g ± 0,01 g weighed into a flat dish (glass, inner diameter 60 mm) and spread uniformly over the entire dish. It is useful to add water (ion exchange water or purified water) to spread samples uniformly. If water is added, the sample is dispersed in the water homogeneously and then spreads uniformly over the entire dish by itself. It is effective to use an ultrasonic bath when the sample is dispersed in water. After that, the sample shall be dried by heating to an extent not causing physical or chemical changes (up to 120 °C), and shall be checked to see that it has achieved a constant weight.

7 Procedure

7.1 Pretreatment of test sample

a) Place the test sample in a flat dish into the photoreactor, then displace the air using the zero-calibration gas at 1,56 % ± 0,16 % volume of water vapour and a temperature of 25,0 °C ± 2,5 °C. Seal the vessel after air displacement.

b) Adjust the illuminance of the catalyst surface in advance to 10 000 lx ± 500 lx. Illuminance shall be adjusted with the window and UV cut filter in place, as used for acetaldehyde decomposition testing in 7.2. A black cloth or black paper should be kept spread under the photoreactor to prevent the reflection of light from the bottom surface.

c) Place a UV cut filter used in acetaldehyde decomposition testing (7.2) on the top surface of the sealed vessel containing the test sample, and place the vessel below the light source described in 5.3.

d) Start the photoirradiation after the illumination by the light source is stable. (For light sources requiring time for steady lighting, shutters are closed before the light source is lit, and shutters are then opened when the lighting is stable.)

e) Measure the concentration of carbon dioxide produced by photoirradiation with the analytical system for carbon dioxide described in 5.6. Continue the photoirradiation until the rate of carbon dioxide production reaches 2,0 ppm/h or lower.

   Ideally, the rate of carbon dioxide production should be zero, but this is difficult. Carbon dioxide which adsorbed to the sample inside is desorbed slowly, or decomposition of organic compounds included in the sample inside produces carbon dioxide. Therefore, a rate of carbon dioxide production of 2,0 ppm/h is permitted, because this is distinguishable from carbon dioxide production caused from decomposition of acetaldehyde from experience.

f) Stop the photoirradiation when the rate of carbon dioxide production reaches 2,0 ppm/h or lower.

7.2 Acetaldehyde decomposition testing

Acetaldehyde decomposition testing shall be performed according to the following procedures to investigate acetaldehyde and carbon dioxide concentrations under dark conditions and photoirradiation. Figure 2 shows an example of the measurement of acetaldehyde and carbon dioxide.

a) Displace air in the photoreactor containing the test sample using the zero-calibration gas at 1,56 % ± 0,16 % volume fraction of water vapour and a temperature of 25,0 °C ± 2,5 °C after the test sample is pretreated to remove organic matter, then seal the reactor. The water vapour concentration at this time corresponds to a relative humidity of 50 % ± 5 % at 25 °C. Measure the carbon dioxide concentration after displacement of air to check that the air has been fully displaced.

   NOTE 1 Humidity does not affect complete acetaldehyde decomposition by photocatalysts but is specified to ensure reproducibility of testing.

b) Darken the photoreactor with a blackout curtain or other means, and inject acetaldehyde gas into the reactor to create an acetaldehyde concentration of 100 ppm ± 5 ppm. Adjust the acetaldehyde concentration in the sealed, empty photoreactor using acetaldehyde gas of a known concentration by adjusting the amount of gas injected in advance such that a concentration of 100 ppm ± 5ppm is achieved. Collect the gas in the reactor immediately after the injection, and measure acetaldehyde and carbon dioxide concentrations. The acetaldehyde measurement apparatus and carbon dioxide measurement apparatus shall be warmed up in advance, and calibration curves shall be prepared.

c) Leave the reactor in the dark for 1 h or less. Sample the gas in the reactor and measure acetaldehyde and carbon dioxide concentrations after removing from the dark.

   NOTE 2 Although adsorption of acetaldehyde under dark conditions does not affect complete decomposition performance, retention is carried on in darkness for uniform dispersion of the acetaldehyde gas in the reactor.

d) Turn the light on in advance to stabilize illuminance. Adjust the illuminance at the surface position of the test sample to 10 000 lx ± 500 lx with the glass window and the UV cut filter used in the test on the top surface of the illuminometer sensor. Record the illuminance.

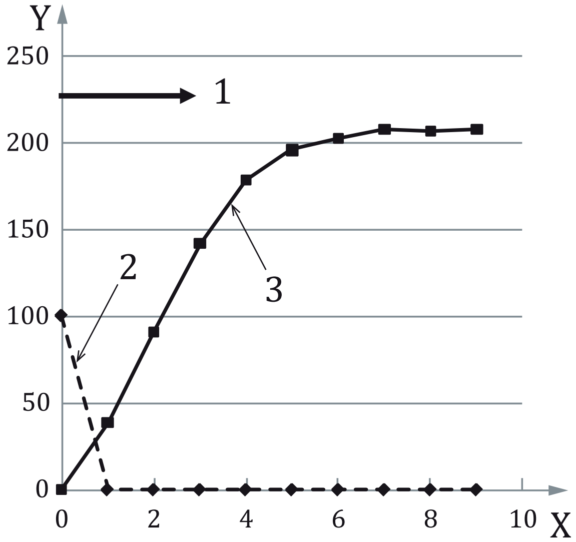
e) Place the UV cut filter on the top surface of the photoreactor and below the light source.

f) Turn the light on (for light sources requiring a long time for stable lighting, shutters are closed in advance when the light is lit, and shutters are then opened when lighting becomes stable). Irradiate the sample.

g) Sample the gas in the reactor at regular intervals under photoirradiation. Measure acetaldehyde concentration and carbon dioxide concentration. An example of the real measurement is shown in Annex A. After photoirradiation, complete the measurement based on the conditions in 8.3.

h) When the frequency of sampling is too high, the internal pressure of the reactor decreases, which can easily cause a leak. Therefore the sum total volume of sampling-gas should be less than 50 mL.

   The recommended volume of one sampling is less than 1 ml. With 1 ml of sampling gas, it is possible to measure both acetaldehyde concentration and carbon dioxide concentration exactly, if using a gas chromatograph. Up to 50 instances of sampling are then possible. Predicting the number of sampling instances and the interval of sampling until complete decomposition by a preliminary experiment is recommended.



Key

1 indoor-light irradiation
2 acetaldehyde concentration
3 carbon dioxide concentration
X irradiation time (h)
Y concentration of acetaldehyde or carbon dioxide (vol-ppm)

Figure 2 — Example of test results

8 Calculation

8.1 General

Test results shall be calculated as follows. The calculation values are usually rounded to one decimal place in accordance with ISO 80000-1.

8.2 Carbon dioxide concentration

The concentration of carbon dioxide produced by decomposition of acetaldehyde is the value obtained by subtracting carbon dioxide concentration measured before photoirradiation in darkness [7.2 c)] from carbon dioxide concentration measured after photoirradiation.

8.3 Measurement end point

After photoirradiation, measurement is complete when the following conditions are fulfilled.

a) When the rate of carbon dioxide production (α_1 and α_2) in three successive measurements is calculated according to Formulae (1) and (2) and fulfils the condition in Formula (3), measurement shall be complete (Figure 3, pattern A, C or E).

$$\alpha_1 = \frac{[\mathrm{CO_2}]t_2 - [\mathrm{CO_2}]t_1}{t_2 - t_1} \quad (1)$$

$$\alpha_2 = \frac{[\mathrm{CO_2}]t_3 - [\mathrm{CO_2}]t_2}{t_3 - t_2} \tag{2}$$

$$\alpha_1 \leqq 2{,}0 \text{ ppm/h and } \alpha_2 \leqq 2{,}0 \text{ ppm/h} \tag{3}$$

where

α_1 is the carbon dioxide production rate after t_1–t_2 h of photoirradiation (ppm/h);

α_2 is the carbon dioxide production rate after t_2–t_3 h of photoirradiation (ppm/h);

$[\mathrm{CO_2}]t_1$ is the carbon dioxide concentration after t_1 h of photoirradiation (ppm);

$[\mathrm{CO_2}]t_2$ is the carbon dioxide concentration after t_2 h of photoirradiation (ppm);

$[\mathrm{CO_2}]t_3$ is the carbon dioxide concentration after t_3 h of photoirradiation (ppm).

b) After photoirradiation, if the measured value of carbon dioxide concentration is between the lower limit (190 ppm) and upper limit of that during complete acetaldehyde decomposition (including factors such as leakage to the reactor), measurement shall be concluded after 45 h when the rate of carbon dioxide generation exceeds 2,0 ppm/h even after 45 h or more (Figure 3, pattern B).

c) After photoirradiation, if the concentration of carbon dioxide generated is 2,0 ppm/h or higher, and exceeds the upper limit of carbon dioxide concentration during complete acetaldehyde decomposition (including factors such as leakage to the reactor), measurement shall be concluded at such time (Figure 3, pattern F).

d) After photoirradiation, if the concentration of carbon dioxide generated for more than 45 h is less than the lower limit (190 ppm) of complete decomposition of acetaldehyde supplied, measurement is concluded in 45 h (Figure 3, Pattern D).

Ideally, the rate of carbon dioxide production should be zero, but this is difficult. Carbon dioxide which adsorbed to the sample inside is desorbed slowly, or decomposition of organic compounds included in the sample inside produces carbon dioxide. Therefore, a rate of carbon dioxide production of 2,0 ppm/h is permitted, because it is distinguishable from carbon dioxide production caused from decomposition of acetaldehyde from experience.

8.4 Determination of complete decomposition

a) **Patterns establishing complete decomposition:** After photoirradiation, complete decomposition is deemed established when the measured concentration of carbon dioxide fulfils Formula (4).

$$190 \text{ ppm} \leqq [\mathrm{CO_2}]_t \leqq (210 + 2 \times t) \text{ ppm} \tag{4}$$

where $[\mathrm{CO_2}]_t$ is the carbon dioxide concentration at time t after photoirradiation.

b) Patterns A and B in Figure 3 correspond to this case.

Pattern A fulfils Formulae (3) and (4).

Pattern B does not fulfil Formula (3) but fulfils Formula (4).

c) **Patterns not leading to complete decomposition:** After photoirradiation, if the measured concentration of carbon dioxide is below the lower limit (LL) of Formula (4), oxidative decomposition strength is assessed as insufficient. Patterns C and D in Figure 3 correspond to this case.

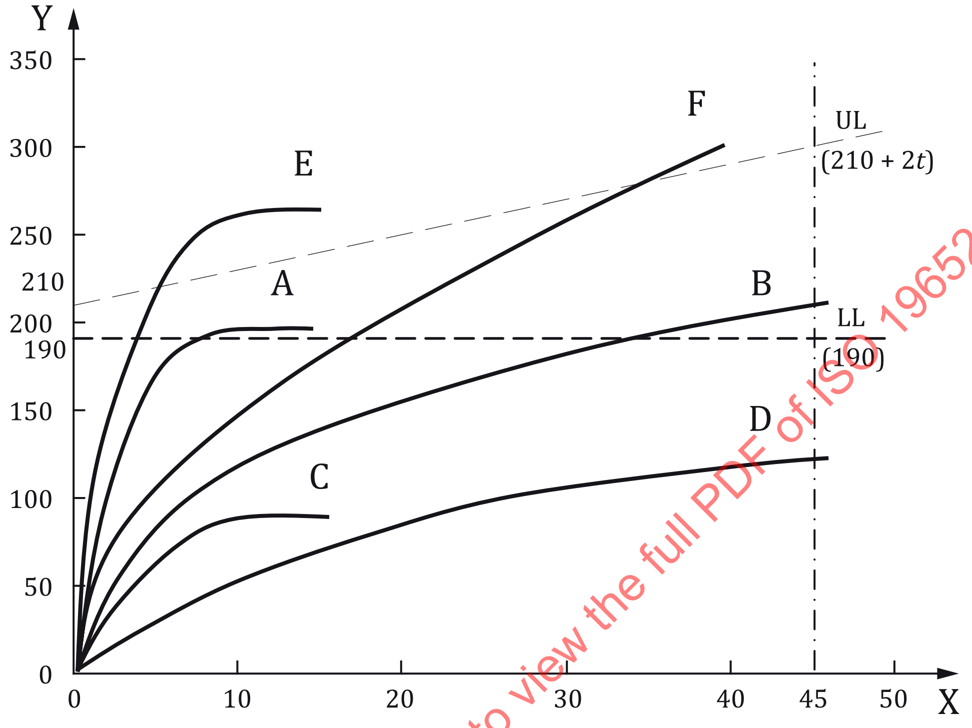
Pattern C fulfils Formula (3) but is below the lower limit (LL) of Formula (4).

Pattern D is below the lower limit (LL) of Formula (4) in a measurement time of 45 h.

d) **Patterns which invalidate the test:** After photoirradiation, if the measured concentration of carbon dioxide exceeds the upper limit (UL) of Formula (4), the test is not considered valid. Patterns E and F in Figure 3 correspond to this case.

Pattern E fulfils Formula (3) but exceeds the upper limit (UL) of Formula (4).

Pattern F exceeds the upper limit (UL) of Formula (4).



Key

X	irradiation time (h)
Y	concentration of carbon dioxide (vol-ppm)
A~F	patterns of curve-example
LL	lower limit for complete decomposition
UL	upper limit for complete decomposition

Figure 3 — Curve examples of carbon dioxide concentration

NOTE The stoichiometric amount of carbon dioxide produced by complete decomposition of acetaldehyde is a molar amount double that of acetaldehyde, according to Formula (5).

$$CH_3CHO + 5/2\ O_2 \rightarrow 2CO_2 + 2H_2O \qquad (5)$$

In the acetaldehyde decomposition testing in 7.2, the concentration of acetaldehyde supplied to the reactor is 100 ppm ± 5 ppm, and the carbon dioxide concentration resulting from complete decomposition is thus 190 ppm~210 ppm. Additionally, because the concentration of carbon dioxide produced by leakage to the reactor and decomposition of organic residue adhering to the test sample is 2,0 ppm/h or lower by pretreatment, the maximum concentration of carbon dioxide produced by the test sample after photoirradiation is thus (2 × measurement time) ppm. Consequently, the upper limit of carbon dioxide concentration measured by complete decomposition will be (210 + 2 × measurement time) ppm.