
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Test method for air-purification
performance of semiconducting
photocatalytic materials under indoor
lighting environment —**

**Part 4:
Removal of formaldehyde**

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of 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*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Photocatalyst is a substance that performs decomposition and removal of contaminants, self-cleaning, antifogging, deodorization and antibacterial actions under photoirradiation. Its application has expanded considerably in recent years. The application of photocatalysts for indoor spaces has increasingly been sought as a solution to indoor environmental problems. Since conventional photocatalysts are responsive only to ultraviolet light, studies have been made to develop an indoor-light-active photocatalyst that makes effective use of indoor light, which room lights mainly emit, and thus demonstrates high photocatalytic performance indoors. The development has recently led to the commercialization of various indoor-light-active photocatalytic products, and there has been demand for the establishment of test methods to evaluate the performance of this type of photocatalyst.

This document, with ISO 22197-1, ISO 22197-2 and ISO 22197-3 as the basis, is intended to provide a testing method to determine the performance of indoor-light-active photocatalytic materials with regards to the removal of formaldehyde, one of the most interesting aromatic volatile organic compound (VOC)-caused sick-house syndromes, enabling swift distribution of photocatalytic products and thus contributing to a safe and clean environment.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for air-purification performance of semiconducting photocatalytic materials under indoor lighting environment —

Part 4: Removal of formaldehyde

1 Scope

This document specifies a test method for the determination of the air-purification performance, with regards to the removal of formaldehyde, of materials that contain a photocatalyst or have photocatalytic films on the surface, usually made from semiconducting metal oxides such as titanium dioxide or other ceramic materials, by continuous exposure of a test piece to the model air pollutant under illumination from indoor light.

This document is intended for use with different kinds of materials, such as construction materials in flat sheet, board or plate shape, which are the basic forms of materials for various applications. This document also applies to materials in honeycomb form, and to plastic or paper materials containing ceramic microcrystals and composites. This document does not apply to powder or granular photocatalytic materials.

This test method is usually applicable to photocatalytic materials produced for air purification. 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 6145-7, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 7: Thermal mass-flow controllers*

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

ISO 16000-3, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 17168-1, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for air purification performance of semiconducting photocatalytic materials under indoor lighting environment — Part 1: Removal of nitric oxide*

ISO 80000-1, *Quantities and units — Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 17168-1 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/>

4 Symbols

f	flow rate of test gas converted into that at the standard state (0 °C, 101,3 kPa)
ϕ_F	formaldehyde volume fraction at the reactor exit ($\mu\text{l/l}$)
ϕ_{FD}	formaldehyde volume fraction at the reactor exit under dark conditions ($\mu\text{l/l}$)
ϕ_{F0}	supply volume fraction of formaldehyde ($\mu\text{l/l}$)
n_F	removal quantity, by test piece, of formaldehyde (μmol)
R_F	removal percentage, by test piece, of formaldehyde (%)

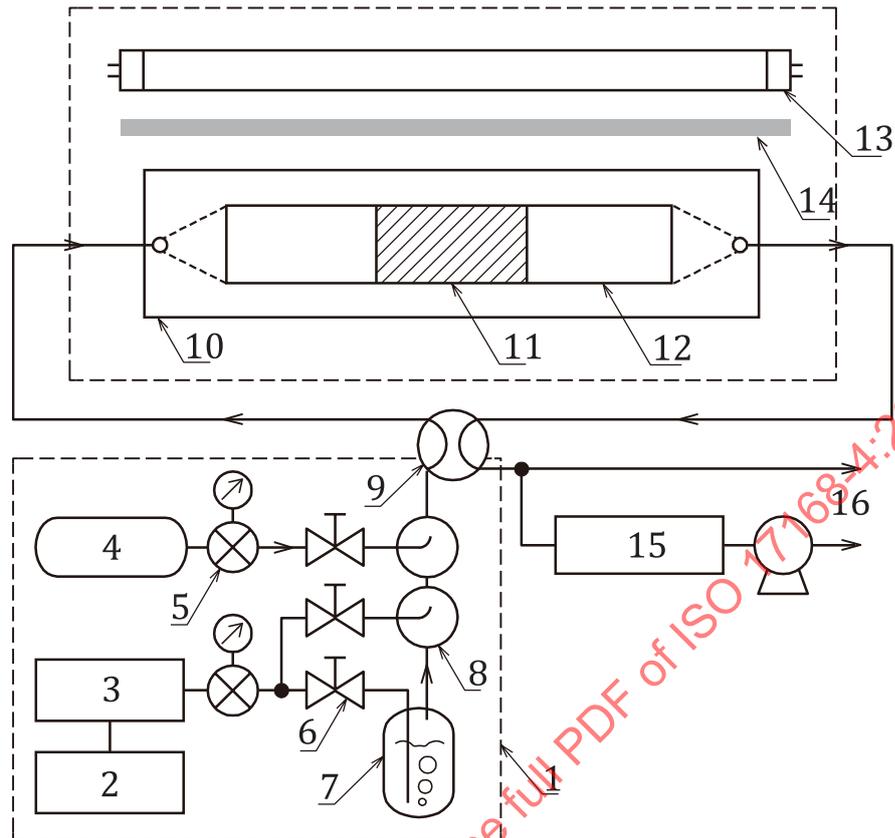
5 Principle

This document deals with the development, comparison, quality assurance, characterization, reliability and design data generation of photocatalytic materials^[1]. The method described is intended to obtain the air-purification performance of photocatalytic materials by exposing a test piece to model polluted air under illumination by indoor light. Formaldehyde (HCHO) is chosen because it is a typical indoor air pollutant that causes the so-called sick building syndrome^[2]. The test piece, placed in a flow-type photoreactor, is activated by indoor light illumination, and adsorbs and oxidizes gas-phase formaldehyde to form carbon dioxide (CO₂) and other oxidation products. The air purification performance is determined from the amount of formaldehyde removed by the test piece (μmol). The simple adsorption of HCHO by the test piece (not due to photocatalysis) is evaluated by tests in the dark. However, some test pieces are very absorbent, and a stable concentration of formaldehyde may not be attained in the designated test time. The photocatalytic activity may depend on physical and chemical properties of pollutants, mainly due to the adsorption process involved. For a better evaluation of air purification performance of photocatalytic materials, it is recommended that one or more suitable test methods are combined, as provided in other parts of the ISO 17168 series.

6 Apparatus

6.1 Test equipment

The test equipment enables a photocatalytic material to be examined for its pollutant-removal capability by supplying the test gas continuously, while providing photoirradiation to activate the photocatalyst. It is the same as that used in the test method for the removal of nitric oxide (see ISO 17168-1) and consists of a test gas supply, a photoreactor, a light source, a UV sharp cut-off filter and pollutant measurement equipment. Since low concentrations of pollutants are to be tested, the system shall be constructed with materials of low absorption and resistant to ultraviolet (UV) radiation, for example acrylic resin, stainless steel, glass and fluorocarbon polymers. An example of a testing system is shown in [Figure 1](#).



Key

- | | | | |
|---|---------------------------------|----|-------------------------|
| 1 | test gas supply | 9 | four-way valve |
| 2 | air compressor | 10 | photoreactor |
| 3 | air-purification system | 11 | test piece |
| 4 | standard gas (pollutant) | 12 | airtight optical window |
| 5 | pressure regulator with a gauge | 13 | light source |
| 6 | mass-flow controller | 14 | sharp cut-off filter |
| 7 | humidifier | 15 | analyser |
| 8 | gas mixer | 16 | vent |

Figure 1 — A schematic of the testing equipment

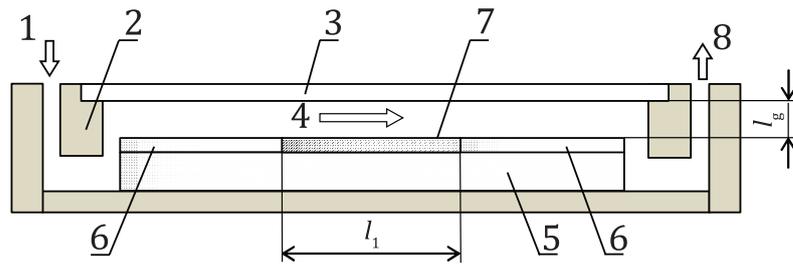
6.2 Test gas supply

The test gas supply provides air polluted with model contaminant at a predetermined concentration, temperature and humidity, and supplies it continuously to the photoreactor. It consists of flow regulators, a humidifier, gas mixers and so on. The flow rate of each gas should be within 5 % of the designated value, which is easily attained by using thermal mass-flow controllers with a knowledge of temperature and gas type at calibration in accordance with ISO 6145-7. The expression of gas flow rate in this document is that converted to the standard state (0 °C, 101,3 kPa). Typical capacities of flow controller for pollutant gas, dry air and wet air are 100, 1 000 and 1 000 ml/min, respectively. The standard formaldehyde gas in a cylinder, normally balanced with nitrogen, shall have a volume fraction of ca. 20 µl/l. If formaldehyde gas generated relevantly can be used besides the standard formaldehyde gas in a cylinder, it is necessary to generate a constant concentration of formaldehyde for the designated test time, which shall not contain impurities (e.g. methanol).

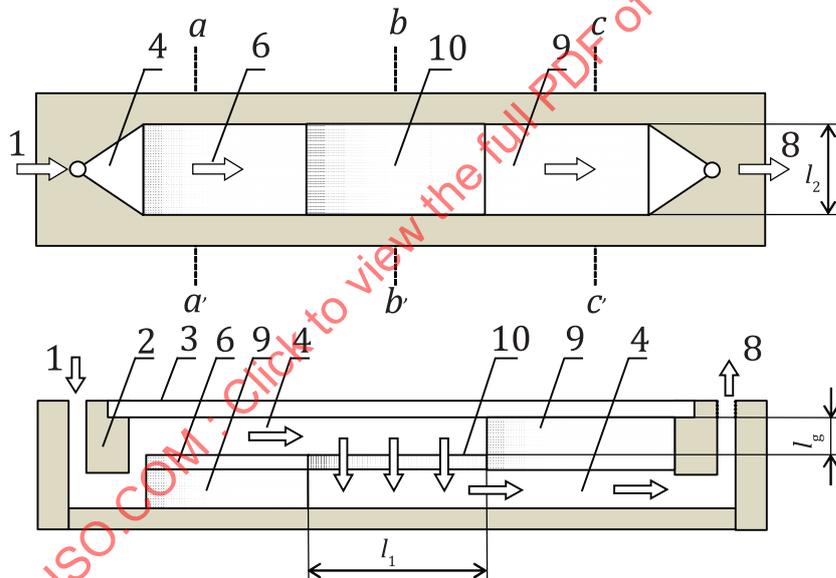
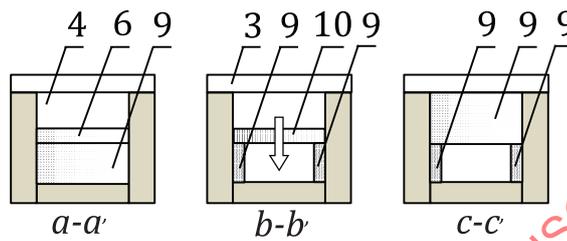
6.3 Photoreactor

The photoreactor holds a planar test piece within a 50-mm wide trough, with its surface parallel to an airtight optical window for photoirradiation. The reactor shall be fabricated from materials that adsorb little test gas and withstand irradiation of near-UV light. The test piece shall be separated from the window by a $5,0 \text{ mm} \pm 0,5 \text{ mm}$ thick air layer. The test gas shall pass only through the space between the test piece and the window. This gap shall be accurately set up according to the thickness of the test piece, for example by using height-adjusting plates with different thicknesses, as shown in [Figure 2 a\)](#). When a filter-type material is tested, an alternative type of test-piece holder shall be used, which holds the test piece while allowing the test gas to pass through the cells of the filter under illumination [[Figure 2 b\)](#)]. Quartz or borosilicate glass that absorbs minimal light at wavelengths longer than 300 nm should be used for the window.

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a) For flat test pieces



b) For filter-type test pieces

Test piece length l_1	Test piece width l_2	Air layer thickness l_g
99,0 ± 1,0 mm	49,0 ± 1,0 mm	5,0 ± 0,5 mm

Key

- | | |
|---------------------------|-----------------------------|
| 1 test gas inlet | 6 auxiliary plate |
| 2 baffle | 7 test piece (flat-type) |
| 3 airtight optical window | 8 test gas outlet |
| 4 flow channel | 9 test piece holder |
| 5 height-adjusting plate | 10 test piece (filter-type) |

Figure 2 — Cross-sectional views of photoreactor

6.4 Light source

A cool white halophosphate fluorescent lamp and a UV sharp cut-off filter as specified in ISO 14605 shall be used. The test piece shall be illuminated uniformly through the window by the light source. When testing honeycomb-form photocatalysts, the light source shall illuminate one face of the test piece. A light source that requires warming up shall be equipped with a shutter. The distance between the light source and the reactor shall be adjusted so that the illuminance at the test piece surface is $6\,000\text{ lx} \pm 300\text{ lx}$. The illuminance along the length of the test piece shall also be constant within $\pm 5\%$. The indoor illuminance shall be measured with an illuminance meter which conforms to ISO 14605. The reactor and light source shall be shielded from external light. The parts around the light source, such as luminaire and shielding device, shall have small reflectance, or flat spectral reflectance over the wavelength range of indoor light.

6.5 UV sharp cut-off filter

The UV sharp cut-off filter shall remove a small amount of UV light radiated from the light source. The transmittance of the UV sharp cut-off filter shall be less than 0,1 % (wavelength $< 365\text{ nm}$), less than 1 % (at 380 nm) and higher than 80 % ($400\text{ nm} < \text{wavelength} < 780\text{ nm}$), and the wavelength where the transmittance is half of the average transmittance between 400 nm and 780 nm is located at $390\text{ nm} \pm 5\text{ nm}$.

6.6 Analytical system

The test gas is sampled using a sampling cartridge, pump and flow controller, as specified in ISO 16000-3. The concentration of formaldehyde shall be determined by 2,4-dinitrophenylhydrazine-derivatised high-performance liquid chromatography (DNPH-HPLC). The reagents, equipment and procedure specified in ISO 16000-3 shall be used.

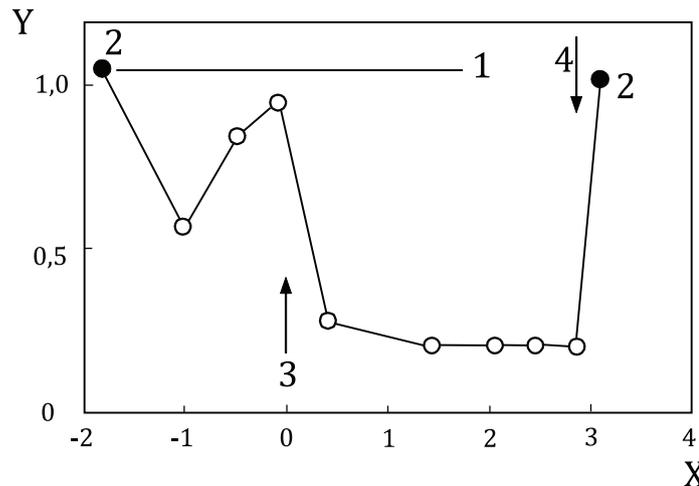
7 Test piece

The test piece shall be a flat material or a honeycomb filter $49,0\text{ mm} \pm 1,0\text{ mm}$ wide and $99,0\text{ mm} \pm 1,0\text{ mm}$ long. It may be cut to these dimensions from a larger bulk material or coated sheet, or may be specially prepared for the test by coating a pre-cut substrate. The thickness of the test piece shall ideally be less than 5 mm, in order to minimize the contribution from the side faces. If thicker test pieces are to be tested, the side faces shall be sealed with an inert material before testing. The honeycomb test piece shall not be thicker than 20 mm.

8 Procedure

8.1 General aspects

The test procedure consists of pretreatment of a test piece, adsorption process in the dark, and measurements of removal of formaldehyde under photoirradiation. An example of concentration change of formaldehyde during the test is shown in [Figure 3](#). Some test pieces may not give accurate removal of formaldehyde due to lower photocatalytic activity. In this case, loading of formaldehyde per test piece can be reduced following the procedure in [Clause 10](#).

**Key**

- 1 formaldehyde feed level
- 2 concentration at reactor inlet
- 3 lights on
- 4 lights off
- X time (h)
- Y formaldehyde concentration (µl/l)

Figure 3 — Examples of formaldehyde concentration measurement

8.2 Pretreatment of test piece

8.2.1 The test piece shall normally be pretreated as specified in 8.2.2 and 8.2.3 in this order. When it is anticipated that the test piece has hydrophobic contamination, 8.2.3 may be followed by 8.2.2. The procedure in 8.2.2 can be omitted if it causes damage to the test piece. If the test pieces are not to be tested immediately after this pretreatment, they shall be kept in an airtight container.

8.2.2 Immerse the test piece in deionized water for 2 h or more, remove it and air-dry at room temperature. The test piece may be dried by heating within a temperature range that does not cause physical and chemical changes to the test piece (maximum 120 °C). Dryness is confirmed when a constant mass is reached. The method of drying and any observations, such as the appearance of sediment in the wash water, shall be recorded.

8.2.3 Irradiate the test piece with an ultraviolet lamp for at least 12 h (up to 24 h) to decompose residual organic matter on the test piece. The UV irradiance at the sample surface shall be high enough to secure complete decomposition of organic matter (10 W/m² – 20 W/m²).

8.3 Preparation for the test

8.3.1 Adjust the test gas supply beforehand so that it can stably supply the test gas containing 1,0 ± 0,1 (µl/l) of formaldehyde and 1,56 % ± 0,08 % of volume fraction of water vapour at 25,0 °C ± 2,5 °C. This water-vapour volume fraction is equivalent to a relative humidity of 50 % at 25 °C. The relative humidity shall be measured by using a hygrometer (with accuracy of ± 3 % RH) that has been calibrated by a method traceable to a certified reference standard. Set the flow rate to be 1,0 l/min (0 °C, 101,3 kPa) at the entrance of the photoreactor. Measure and record the illuminance from the light source. Warm up and calibrate the pollutant analyser during this period.

8.3.2 Place the test piece in the centre of the photoreactor and attach the glass window after adjusting the air layer between the test piece and window to a thickness of $5,0 \text{ mm} \pm 0,5 \text{ mm}$. If necessary, height-adjusting plates are used for this purpose, and adjusting based on the top of the test piece. Check that the reactor is sealed by visual examination of the sealing material, such as an O-ring to tightly connect to the glass window.

8.3.3 Place the UV sharp cut-off filter at the space between the light source and the photoreactor.

8.4 Pretest

The concentration of formaldehyde cannot be obtained instantaneously by the DNPH-HPLC method. Therefore, the time of the adsorption of formaldehyde reaching saturation in dark conditions cannot be confirmed during the test. For this reason, the following pre-test shall be carried out. If the time for saturation can be confirmed during the test, there is no need for the pre-test.

After pretreatment of the test piece in 8.2 and preparation for the test in 8.3, introduce the test gas into the reactor. Measure the concentration of formaldehyde under dark conditions every 15 min. When the concentration of formaldehyde exceeds 90 % of the supply gas concentration for the first time, then that time and the concentration at that time are defined as the time of the dark conditions and concentration of the dark conditions, respectively. When the concentration of formaldehyde is still less than 90 % after 90 min, then this document shall not apply.

8.5 Removal test

8.5.1 Follow the pretreatment procedure in 8.2 and preparation in 8.3.

8.5.2 If the pre-test in 8.4 has been carried out already, introduce the test gas into the photoreactor under dark conditions for the previously confirmed time. If the pre-test has not been done yet, carry out the following procedure. Introduce the test gas into the photoreactor and measure the formaldehyde concentration until the concentration of acetaldehyde exceeds 90 % of the supply gas concentration. At this time the concentration is defined as the concentration of the dark conditions. When the concentration of acetaldehyde is still less than 90 % after 90 min, then this document shall not apply. If the requirement mentioned above is satisfied, it is possible to continue the dark conditions for the purpose of increasing the concentration of the dark conditions.

8.5.3 Turn on the light source. (In the time required to light the light source, prevent the UV rays from making contact with the test pieces. Set a partition while the light source is on. After the test piece stabilizes, remove the partition.) Record the concentration under irradiation for 3 h. When the photocatalytic decomposition begins, the concentration decreases as in Figure 3 and eventually becomes constant. The formaldehyde concentration shall be measured at more than one point in an hour. Measurement shall be made at more than three points as in the last 1 h (120 min to 180 min after the start of irradiation). The concentration of formaldehyde shall be obtained from the average value based on the concentrations measured in the last 1 h.

8.5.4 Stop the gas supply to the reactor and remove the test piece from the reactor. When the test piece that was used in the pre-test is going to be reused in the actual test, the pretreatment in 8.2 shall be repeated.

9 Calculation

If ϕ_A does not satisfy Formula (1), i.e. the difference between formaldehyde concentrations under dark conditions and under photoirradiation is less than 5 % of the formaldehyde concentration supplied, this test method shall not be applied. The test results shall be calculated as follows. The calculated values are usually rounded to one decimal place according to ISO 80000-1. The flow rate of test gas f is the observed flow rate normalized for $0 \text{ }^\circ\text{C}$, 101,3 kPa.