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

Part 4: Removal of formaldehyde

Céramiques techniques — Méthodes d'essai relatives à la performance des matériaux photocatalytiques semi-conducteurs pour la purification de l'air —

Partie 4: Élimination du formaldéhyde

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 22197-4:2013), which has been technically revised.

The main changes to the previous edition are as follows:

— references to ISO 4892-3 and ISO 6145-7 deleted from Clause 2;

— gas flow measurement changed from dry-gas basis to wet-gas basis in 6.2;

— tolerance on dimensions of test piece changed in Clause 7;

— procedures for removing water-soluble contaminants added to 8.2;

— criterion for acceptable adsorption of formaldehyde added to Clause 9.

A list of all parts in the ISO 22197 series can be found on the ISO website.

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.

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

Part 4: Removal of formaldehyde

1 Scope

This document specifies a test method for the determination of the air-purification performance 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 irradiation with long-wave ultraviolet (UV) light. This document is intended for use with different kinds of materials, such as construction materials in flat sheet, board or plate shape, that are the basic forms of materials for various applications. This document also applies to structured filter materials including honeycomb-form, woven and non-woven fabrics, and to plastic or paper materials if they contain 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. It concerns the removal of formaldehyde.

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 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*

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
photocatalytic material
material in which or on which the *photocatalyst* (3.1) is added by, for example, coating, impregnation or mixing

Note 1 to entry: Such photocatalytic materials are intended primarily for use as building and road construction materials to obtain the functions performed by photocatalysts.

3.3
zero-calibration gas
air that does not contain pollutants (i.e. in which common pollutants are below 0,01 µl/l)

Note 1 to entry: The zero-calibration gas is prepared from indoor air using a laboratory air purification system or supplied as a synthetic air in a gas cylinder.

3.4
formaldehyde gas
diluted gas of known formaldehyde concentration used for testing and calibration

3.5
test gas
mixture of air and pollutant(s) of known concentration, prepared from a standard gas or a *zero-calibration gas* (3.3), to be used for the performance test of a *photocatalytic material* (3.2)

3.6
dark condition
test condition with no light irradiation by the light source for testing and room lighting

4 Symbols

For the purposes of this document, the following symbols apply.

f flow rate of test gas converted into that at the standard state (0 °C and 101,3 kPa) (l/min)

ϕ_F volume fraction of formaldehyde at the reactor exit (µl/l)

ϕ_{F0} supply volume fraction of formaldehyde (µl/l)

ϕ_{FD} volume fraction of formaldehyde at the reactor exit under dark conditions (µl/l)

n_F removal quantity, by test piece, of formaldehyde (µmol)

R_F removal percentage, by test piece, of formaldehyde (%)

5 Principle

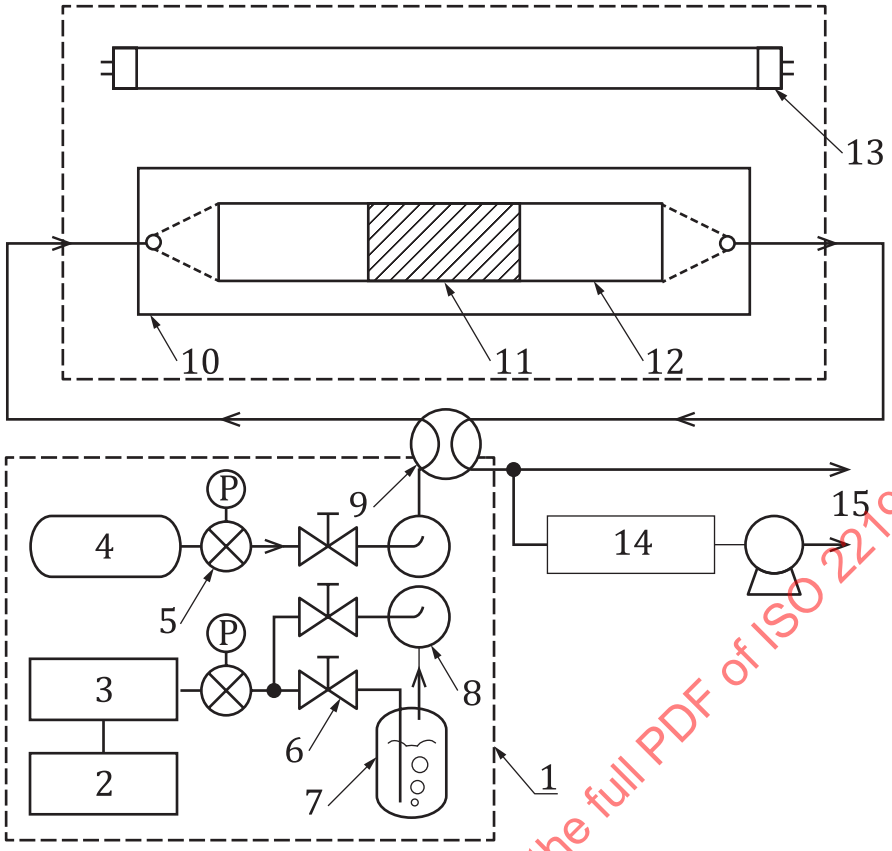
This document concerns the development, comparison, quality assurance, characterization, reliability and design data generation of photocatalytic materials. The method described is intended to obtain the air-purification performance of photocatalytic materials by exposing a test piece to model polluted air under irradiation by UV light. Formaldehyde (HCHO) is chosen because it is a typical indoor air pollutant that causes the so-called sick-building syndrome. The test piece, placed in a flow-type photoreactor, is activated by UV irradiation, and adsorbs and oxidizes gas-phase formaldehyde to form carbon dioxide (CO_2) and other oxidation products. The air purification performance is determined from the net 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 absorb formaldehyde very strongly, and it is not always possible to attain a stable concentration of formaldehyde in the designated time of test. The photocatalytic activity can 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 as provided in the other parts of the ISO 22197 series are combined.

6 Apparatus

6.1 Test equipment

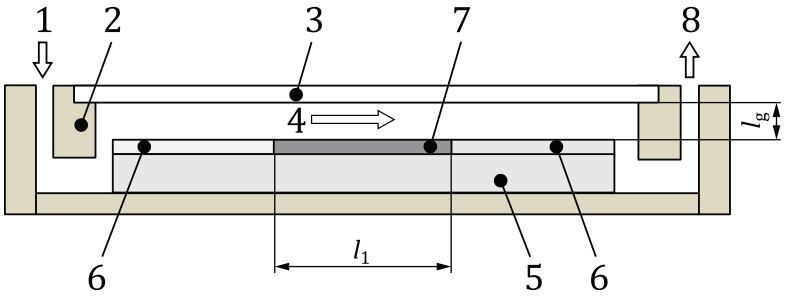
The test equipment enables a photocatalytic material to be examined for its pollutant-removal capability by supplying the test gas continuously, while providing photoirradiation to activate the photocatalyst. It is the same as that used in the test method for the removal of nitric oxide (see ISO 22197-1) and consists of a test gas supply, a photoreactor, a light source 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 UV radiation, e.g. acrylic resin, stainless steel, glass and fluorocarbon polymers. An example of a test system is shown in Figure 1.



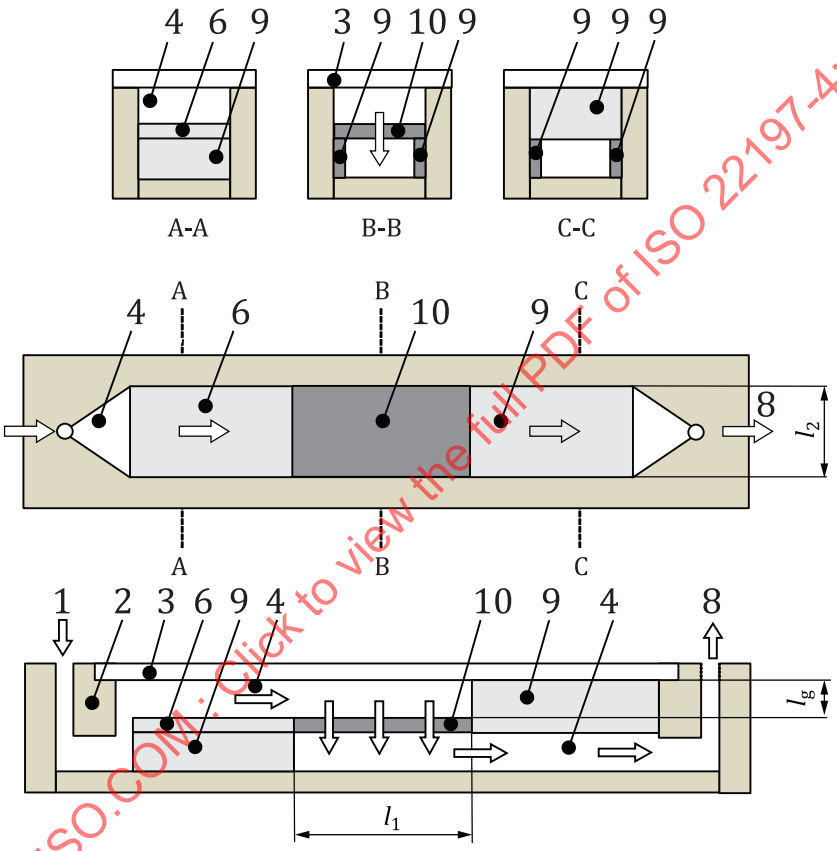
Key

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	air-tight optical window
5	pressure regulator	13	light source
6	mass-flow controller	14	analyser
7	humidifier	15	vent
8	gas mixer		

Figure 1 — Schematic diagram of the test equipment



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
2 buffle
3 air-tight optical window
4 flow channel
5 height-adjusting plate
6 auxiliary plate
7 test piece (flat-type)
8 test gas outlet
9 test piece holder
10 test piece (filter-type)

Figure 2 — Cross-sectional views of photoreactor

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 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 and 101,3 kPa). Typical capacities of flow controller for pollutant gas, dry air and wet air are 200 ml/min, 2 000 ml/min and 2 000 ml/min, respectively. The standard formaldehyde gas in a cylinder, normally balanced with nitrogen, shall have a volume fraction of about 20 µl/l.

6.3 Photoreactor

The photoreactor holds a planar test piece within a 50-mm-wide trough, with its surface parallel to an 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 an air layer 5,0 mm ± 0,5 mm thick. 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 irradiation [Figure 2 b)]. Quartz or borosilicate glass that absorbs minimal light at wavelengths longer than 300 nm shall be used for the window.

6.4 Light source

The light source shall provide UV irradiation within a wavelength range of 300 nm to 400 nm. Suitable sources include the so-called black light (BL) and black light blue (BLB) fluorescent lamps, with a maximum at 351 nm, as specified in ISO 10677. The test piece shall be irradiated uniformly through the window by the light source. If testing filter-type photocatalysts, the light source shall irradiate 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 UV irradiance (300 nm to 400 nm) at the sample surface is 10 W/m^2 ± 0,5 W/m^2. The irradiance along the length of the test piece shall also be constant within ±5 %. The UV irradiance shall be measured with a radiometer which conforms to ISO 10677. The reactor shall be shielded from external light if necessary.

6.5 Analytical system

The test gas shall be 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-dinitorphenylhydrazine-derivatised high-performance liquid chromatography (DNPH-HPLC). The reagents, equipment and procedure as specified in ISO 16000-3 shall be used. Other analytical methods that give equivalent or better performance can be used.

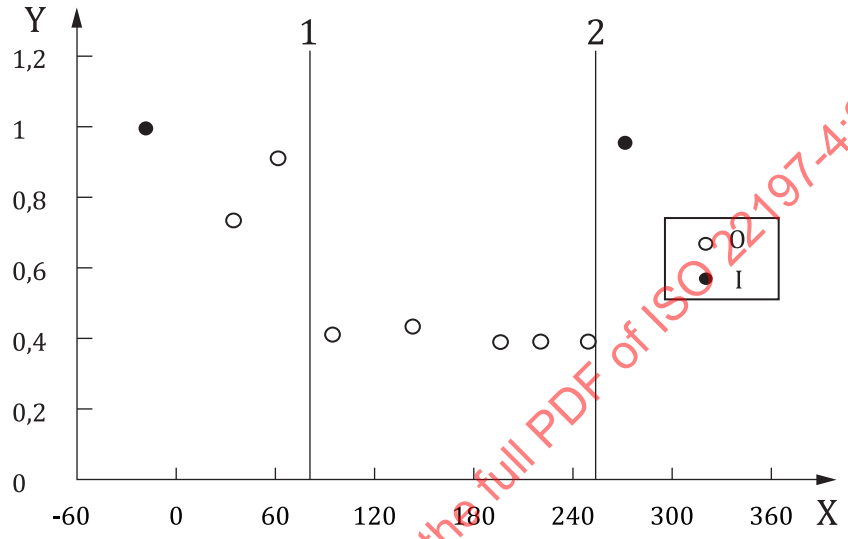
7 Test piece

The test piece shall be a flat material or a filter-type 49,0 mm ± 1,0 mm wide and 99,0 mm ± 1,0 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 filter-type test piece shall not be thicker than 20 mm.

8 Procedure

8.1 General aspects

The test procedure consists of pretreatment of the test piece, an adsorption process in the dark and measurements of removal of formaldehyde under photoirradiation. An example of the concentration change of formaldehyde during the test is shown in Figure 3. Some test pieces will possibly not give accurate removal of formaldehyde due to lower photocatalytic activity. In these cases, loading of formaldehyde per test piece may be reduced following the procedure in Clause 10.



Key

X time (min)
Y concentration of formaldehyde (µl/l)
1 irradiation start
2 irradiation stop
O outlet
I inlet

Figure 3 — Typical trace of formaldehyde concentration during the test operation

8.2 Pretreatment of test piece

8.2.1 The test piece shall normally be pretreated according to 8.2.2 and 8.2.3, in this order. When it is anticipated that the test piece will have hydrophobic contamination, 8.2.3 may be followed by 8.2.2. The procedure in 8.2.2 may 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 or 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 UV 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/m^2 to 20 W/m^2).

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 % ± 0,16 % 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. Adjust the flow regulator in order for the flow rate at the inlet of the reactor to be 3,00 l/min ± 0,15 l/min (0 °C and 101,3 kPa). Measure and record the irradiance from the light source at the surface of the test piece. For the light source that requires warming up, turn the power on well before the measurement of irradiance and irradiation for the formaldehyde removal test. Use the shutter appropriately to avoid unnecessary irradiation to the photoreactor.

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 be 5,0 mm ± 0,5 mm thick, using height-adjusting plates. If necessary, adjust the air layer thickness before and after the test piece to be within 1,0 mm difference based on the top of the test piece, using auxiliary plates. Check that the reactor is sealed by visual examination of the sealing material, such as an O-ring to tightly contact the glass window.

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 a dark condition cannot be confirmed during the test. For this reason, the following pretest shall be carried out. If the time for saturation can be confirmed during the test, there is no need for the pretest.

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 the dark condition every 15 min for 90 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 condition and concentration of the dark condition, respectively. When the concentration of formaldehyde is still less than 90 % of the concentration after 90 min, then this document shall not apply.

8.5 Removal test

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

8.5.2 If the pretest in 8.4 has been done, supply the test gas into the photoreactor until the time of dark conditions which was checked beforehand (if the time is less than 30 min, supply for 30 min). If the pretest has not been done, proceed as follows. Supply the test gas to the photoreactor and record the concentration of formaldehyde under the dark conditions. If the concentration of formaldehyde exceeds 90 % of the supply gas concentration, then that time and the concentration at that time are defined as the time of the dark condition and concentration of the dark condition, respectively. If it does not exceed 90 % even after 90 min, stop measurement and report that this test is not applicable to the test piece used.

8.5.3 Maintain the gas flow and commence irradiation of the test piece. For a light source that requires warming up, the same procedure shall be applied as described in 8.3.1. 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 one hour. Measurement shall be made at more than three points as in the final hour (120 min to 180 min after the start of irradiation). The concentration of formaldehyde shall be obtained by the average value based on the concentrations measured in the final hour.

8.5.4 Stop the gas supply to the reactor and remove the test piece from the reactor.