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

**Part 5:
Removal of methyl mercaptan**

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

Partie 5: Élimination du mercaptan méthylique



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Published in Switzerland

Contents

| | Page |
|--|-----------|
| Foreword | iv |
| 1 Scope | 1 |
| 2 Normative references | 1 |
| 3 Terms and definitions | 1 |
| 4 Symbols | 2 |
| 5 Principle | 2 |
| 6 Apparatus | 3 |
| 6.1 Test equipment | 3 |
| 6.2 Test gas supply | 6 |
| 6.3 Photoreactor | 6 |
| 6.4 Light source | 6 |
| 6.5 Analytical system | 6 |
| 7 Test piece | 7 |
| 8 Procedure | 7 |
| 8.1 General aspects | 7 |
| 8.2 Pretreatment of test piece | 8 |
| 8.3 Removal test | 8 |
| 9 Calculation | 9 |
| 10 Test method for test pieces with lower performance | 10 |
| 11 Test report | 10 |
| Bibliography | 11 |

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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 22197-5:2013), which has been technically revised.

The main changes to the previous edition are as follows:

- references to ISO 2718 (withdrawn), ISO 4677-1 (withdrawn), ISO 4892-3 and ISO 6145-7 deleted from [Clause 2](#);
- precaution relating to the handling of methyl mercaptan added to [6.1](#);
- 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](#);
- valid dark concentration level of methyl mercaptan changed in [8.3](#);
- criterion for acceptable adsorption of methyl mercaptan 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 5: Removal of methyl mercaptan

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 methyl mercaptan.

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/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 standard gas

diluted gas of known concentration supplied in cylinders and certified by an accredited laboratory

3.5 test gas

mixture of air and pollutant(s) of known concentration prepared from a *standard gas* (3.4) 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_M | volume fraction of methyl mercaptan at the reactor exit (µl/l) |
| ϕ_{M0} | supply volume fraction of methyl mercaptan (µl/l) |
| ϕ_{MD} | average volume fraction of methyl mercaptan under dark condition (µl/l) |
| ϕ_{MD1} | volume fraction of methyl mercaptan under dark condition before the test (µl/l) |
| ϕ_{MD2} | volume fraction of methyl mercaptan under dark condition after the test (µl/l) |
| n_M | removal quantity, by test piece, of methyl mercaptan (µmol) |
| R_M | removal percentage, by test piece, of methyl mercaptan (%) |

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. Methyl mercaptan (methanethiol, CH₃SH) is chosen as a typical malodorous substance. The test piece, placed in a flow-type photoreactor, is activated by UV irradiation, and adsorbs and oxidizes gas-phase methyl mercaptan. The air purification performance is determined from the amount of the net removal of methyl mercaptan (µmol). The simple adsorption and desorption of CH₃SH by the test piece (not due to photocatalysis) is evaluated by tests in the dark. It is noted that CH₃SH undergoes not only adsorption but also catalytic oxidation to give other malodorous

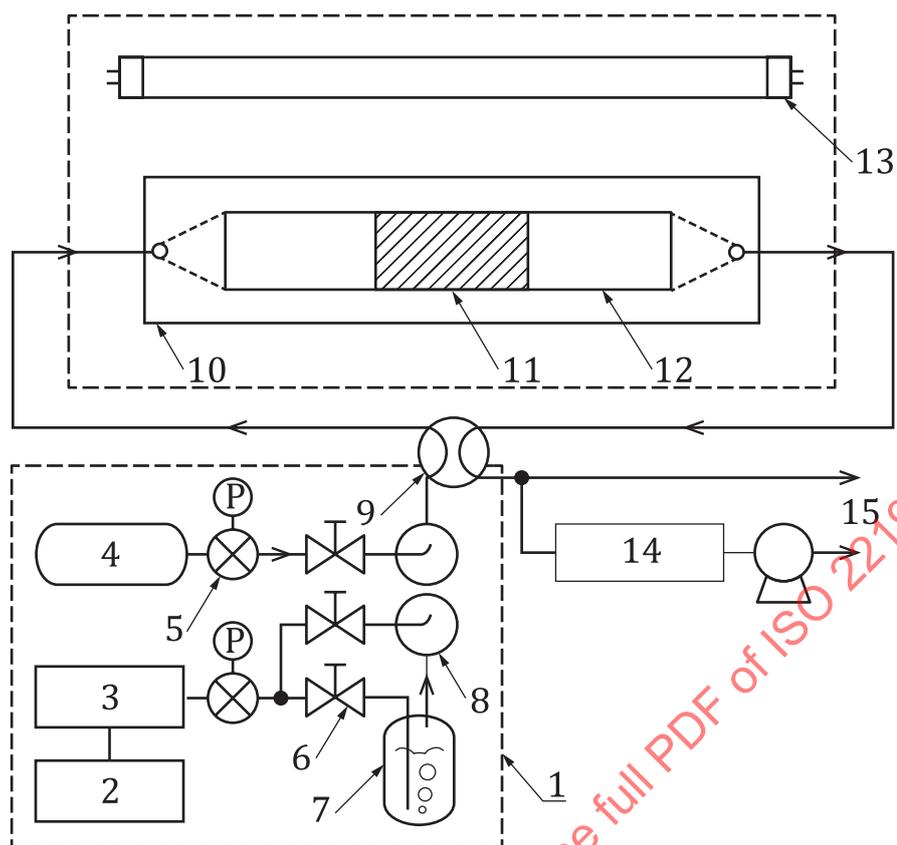
products such as dimethyl disulphide by some test pieces under the dark condition. Therefore, this document adopts a different dark test condition from that in the other parts of the ISO 22197 series. However, this document cannot be applied to a test piece which does not give a stable concentration of methyl mercaptan 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 complete evaluation of air purification performance of photocatalytic materials, it is recommended that one or more suitable test methods as provided in 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](#).

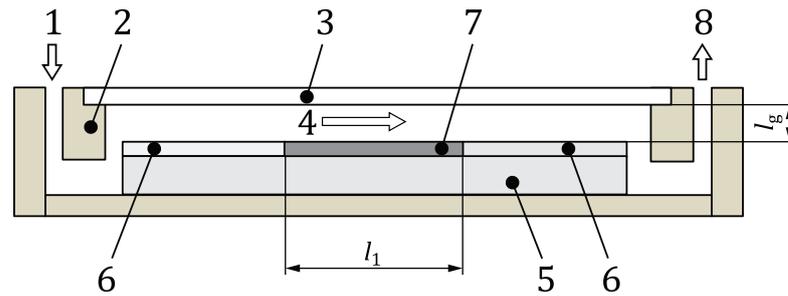
Tests should be carried out in a fume hood with a scrubber or similar ventilation system in order to protect the laboratory personnel from the strong, offensive odour of methyl mercaptan and its reaction products.



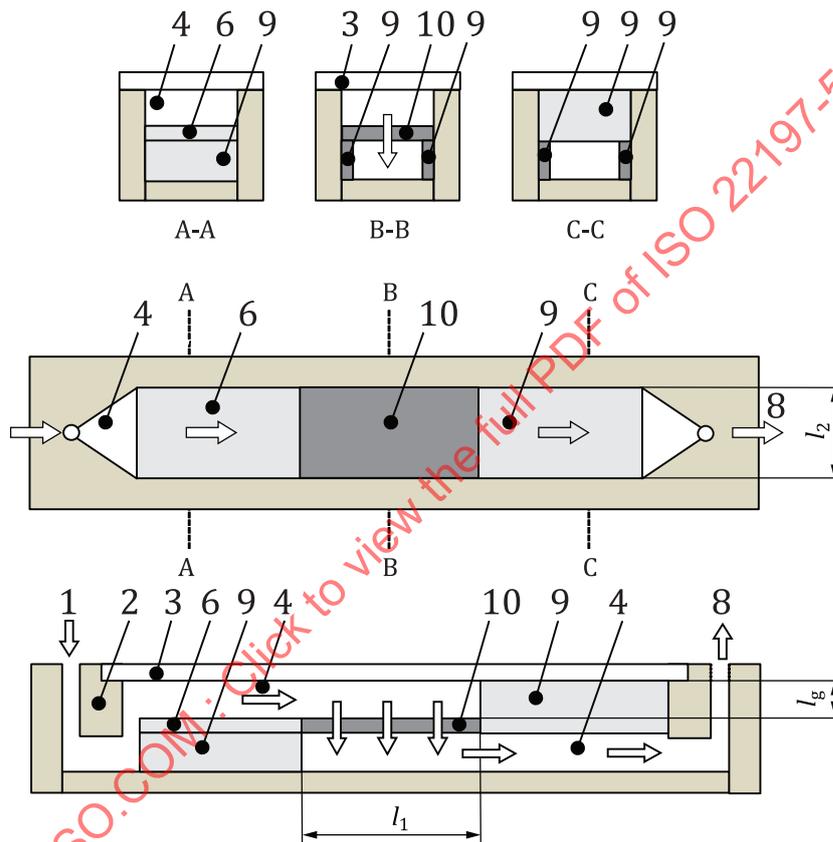
Key

- | | | | |
|---|--------------------------|----|--------------------------|
| 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 |
|-------------------------|------------------------|---------------------------|
| $99,0 \pm 1,0$ mm | $49,0 \pm 1,0$ mm | $5,0 \pm 0,5$ mm |

Key

- | | |
|----------------------------|-----------------------------|
| 1 test gas inlet | 6 auxiliary plate |
| 2 baffle | 7 test piece (flat-type) |
| 3 air-tight 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

6.2 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 50 ml/min, 1 000 ml/min and 1 000 ml/min, respectively. The standard methyl mercaptan gas in a cylinder, normally balanced with nitrogen, shall have a volume fraction of 100 µl/l to 1 000 µl/l.

6.3 Photoreactor

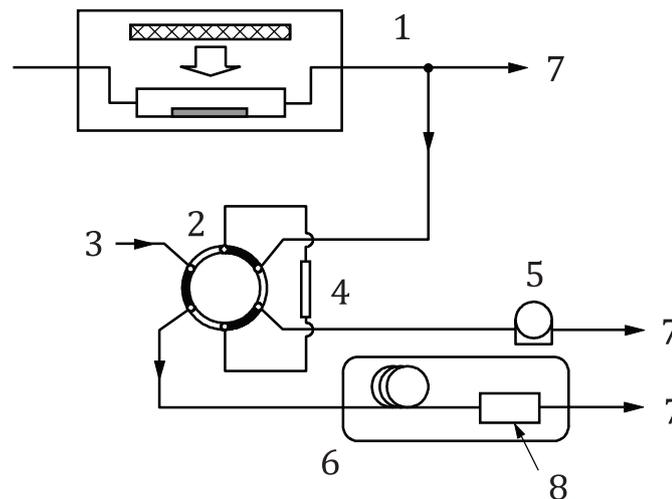
The photoreactor holds a planar test piece within a 50-mm-wide trough, with its surface parallel to an optical window for photoirradiation. The reactor shall be fabricated from materials that adsorb little test gas and withstand irradiation of near-UV light. The test piece shall be separated from the 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 $10 \text{ W/m}^2 \pm 0,5 \text{ W/m}^2$. 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 concentration of methyl mercaptan shall be determined by gas chromatography. Either packed column or capillary column may be used as long as it can separate methyl mercaptan from related organic compounds. The detection shall be made by either a flame ionization detector (FID) or a flame photometric detector (FPD). The test gas shall be sampled with a gas-tight syringe. However, use of a six-way valve is recommended for reproducible and automatic sampling. The flow diagram when a six-way valve is used is shown in [Figure 3](#). A small sampling pump continuously ventilates the metering tube with the test gas. The pump is stopped when the test gas is sampled by switching the six-way valve. The volume of the metering tube is typically 0,5 ml, but it shall be determined by the sensitivity of the analytical system.



Key

- 1 photoreactor
- 2 six-way valve
- 3 carrier gas
- 4 metering tube
- 5 sampling pump
- 6 gas chromatograph
- 7 vent
- 8 flame ionization detector

Figure 3 — Gas sampling system

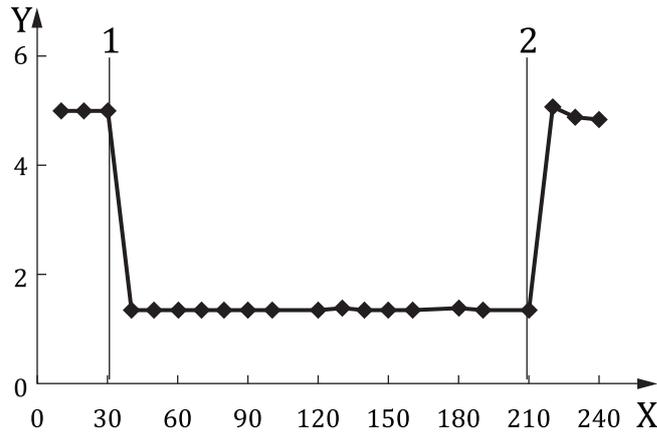
7 Test piece

The test piece shall be a flat material or a filter-type of $49,0 \text{ mm} \pm 1,0 \text{ mm}$ 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 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 methyl mercaptan under photoirradiation. An example of the concentration change of methyl mercaptan during the test is shown in [Figure 3](#). Some test pieces will possibly not give accurate removal of methyl mercaptan due to lower photocatalytic activity. In these cases, loading of methyl mercaptan per test piece may be reduced following the procedure in [Clause 10](#).



Key

- X time (min)
- Y concentration of methyl mercaptan (µl/l)
- 1 irradiation start
- 2 irradiation stop

Figure 4 — Typical trace of methyl mercaptan 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 a 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/m² to 20 W/m²).

8.3 Removal test

8.3.1 Adjust the test gas supply beforehand so that it can stably supply the test gas containing 5,0 µl/l ± 0,25 µl/l of methyl mercaptan 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 1,00 l/min ± 0,05 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 methyl mercaptan 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