INTERNATIONAL STANDARD

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

Part 1:
Removal of nitric oxide

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

Partie 1: Élimination de l'oxyde nitrique

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 22197-1 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

ISO 22197 consists of the following parts, under the general title *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for air-purification performance of semiconducting photocatalytic materials*:

— *Part 1: Removal of nitric oxide*

The following parts are under preparation:

— *Part 2: Removal of acetaldehyde*

— *Part 3: Removal of toluene*

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

Part 1: Removal of nitric oxide

1 Scope

This part of ISO 22197 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 illumination with ultraviolet light. This part of ISO 22197 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 part of ISO 22197 also applies to materials in honeycomb-form, and to plastic or paper materials if they contain ceramic microcrystals and composites. This part of ISO 22197 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 nitric oxide.

2 Normative references

The following referenced documents are indispensable for the application 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 31-0:1992, *Quantities and units — Part 0: General principles*

ISO 4677-1:1985, *Atmospheres for conditioning and testing — Determination of relative humidity — Part 1: Aspirated psychrometer method*

ISO 4892-1:—[1], *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance*

ISO 4892-3:2006, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 6145-7:2001, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 7: Thermal mass-flow controllers*

1) To be published. (Revision of ISO 4892-1:1999.)

ISO 7996:1985, *Ambient air — Determination of the mass concentration of nitrogen oxides — Chemiluminescence method*

ISO 10304-1:—[2)], *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

ISO 10523:1994, *Water quality — Determination of pH*

ISO/IEC 17025:2005, *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.

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

NOTE Such photocatalytic materials are intended primarily for use as building and road construction materials to obtain the above-mentioned functions.

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

NOTE The zero-calibration gas is prepared from indoor air using a laboratory air-purification system, or supplied as synthetic air in a gas cylinder.

3.4
standard gas
diluted gases of known concentrations 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 or a zero-calibration gas, to be used for the performance test of a photocatalytic material

3.6
purified water
water to be used for elution, etc., with a conductivity lower than 1 μS, prepared by the ion exchange method or distillation

2) To be published. (Revision of ISO 10304-1:1992.)

4 Symbols

f	air-flow rate converted into that at the standard state (0 °C, 101,3 kPa, and dry gas basis) (l/min)
ϕ_{NO}	nitric oxide volume fraction at the reactor exit (µl/l)
ϕ_{NO_i}	supply volume fraction of nitric oxide (µl/l)
ϕ_{NO_2}	nitrogen dioxide volume fraction at the reactor exit (µl/l)
ϕ_{NO_x}	the volume fraction of nitrogen oxides (ϕ_{NO} + ϕ_{NO_2}) at the reactor exit (µl/l)
ρ_{NO_2-}	nitrite ion concentration in the eluent from the test piece (mg/l)
ρ_{NO_3-}	nitrate ion concentration in the eluent from the test piece (mg/l)
t	time of adsorption, removal or desorption operation (min)
n_{ads}	the amount of NO_x adsorbed by the test piece (µmol)
n_{des}	the amount of NO_x desorbed from the test piece (µmol)
n_{NO}	the amount of NO removed by the test piece (µmol)
n_{NO_2}	the amount of NO_2 formed by the test piece (µmol)
n_{NO_x}	the amount of NO_x removed by the test piece (µmol)
n_w	the amount of nitrogen eluted from the test piece (µmol) [w_1, w_2 are the 1st and 2nd elutions, respectively]
V_w	the volume of collected washings (ml) [w_1, w_2 are the 1st and 2nd elutions, respectively]
η_w	the fractional recovery of nitrogen

5 Principle

This part of ISO 22197 concerns 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 ultraviolet (UV) light[2]. Nitric oxide (NO) is chosen as a typical air pollutant that gives nonvolatile products on the photocatalyst. The test piece, placed in a flow-type photoreactor, is activated by UV illumination, and adsorbs and oxidizes gas-phase NO to form nitric acid (or nitrate) on its surface[3]. A part of the NO is converted to nitrogen dioxide (NO_2) on the test piece. The air-purification performance is determined from the amount of the net removal of nitrogen oxides (NO_x) (= NO removed – NO_2 formed). The simple adsorption and desorption of NO by the test piece (not due to photocatalysis) is evaluated by tests in the dark. Although the photocatalytic activity is reduced by the accumulation of reaction products, it is usually restored by washing with water[4]. The elution test provided here gives information about the ease of regeneration and material balance of the pollutants.

6 Apparatus

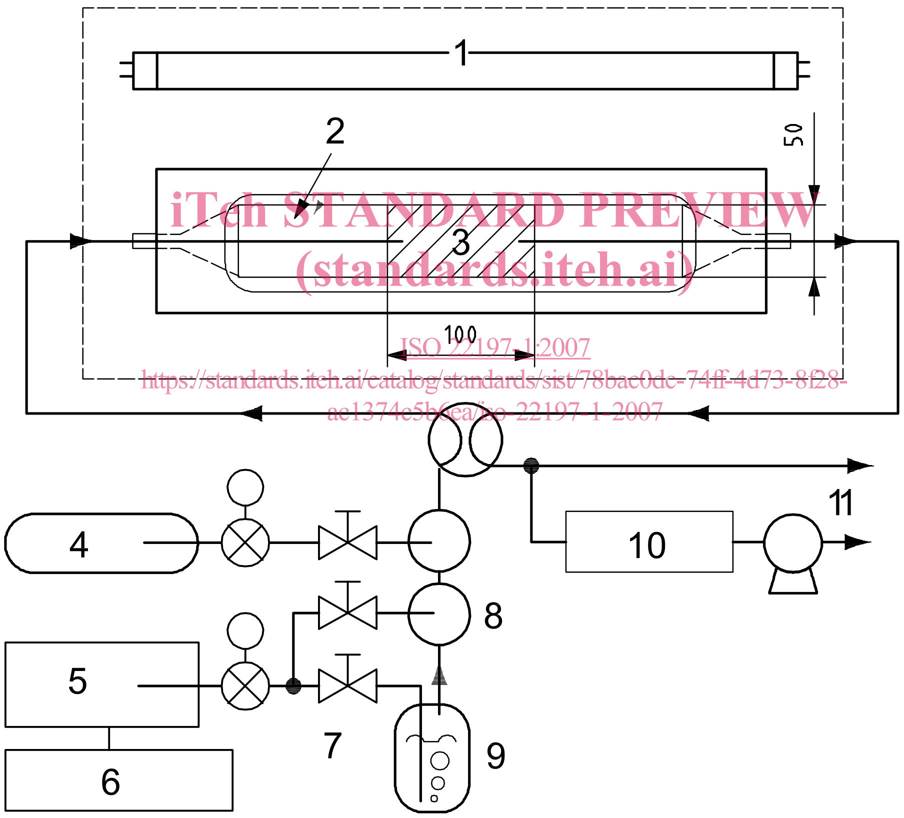
6.1 Test equipment

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 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 adsorption and resistant to ultraviolet (UV) radiation, for example, acrylic resin, stainless steel, glass and fluorocarbon polymers. An example of a test system is shown in Figure 1.

6.2 Test gas supply

The test gas supply provides air polluted with the 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, etc. 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 calibrated gas flow rate and temperature in accordance with ISO 6145-7. Typical capacities of the flow controller for pollutant gas, dry air and wet air are 0,1 l/min, 2,0 l/min and 2,0 l/min, respectively. The expression of gas flow rate in this part of ISO 22197 is that converted to the standard state (0 °C, 101,3 kPa, and dry gas basis). The standard NO gas, normally balanced with nitrogen in a cylinder, shall have a volume fraction of 30 to 100 μl/l, because the oxidation of NO to NO_2 upon mixing with purified air becomes prominent with a higher concentration of NO.

Dimensions in millimetres

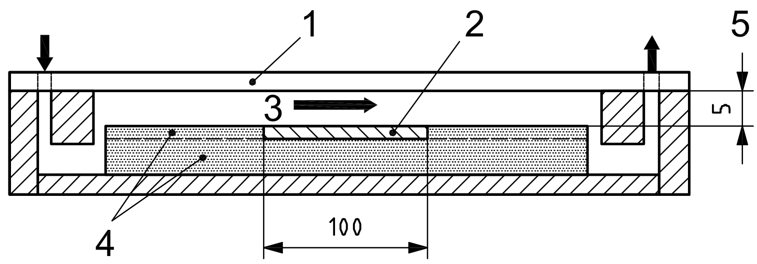


Key

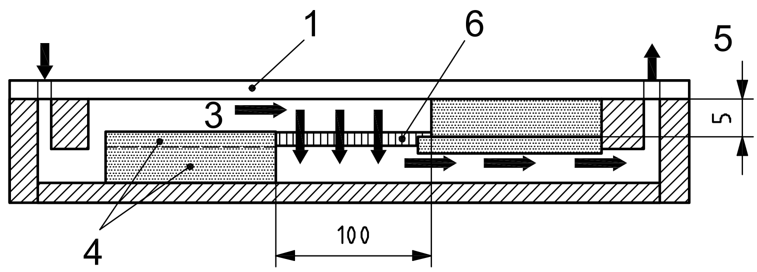
1 light source
2 optical window
3 test piece
4 standard gas (pollutant)
5 air-purification system
6 air compressor
7 mass-flow controllers
8 gas mixers
9 humidifier
10 analyser
11 vent

Figure 1 — A schematic of the test equipment

Dimensions in millimetres



a) For flat test pieces



b) For honeycomb filters

Key

1 optical window
2 test piece
3 test gas flow
4 height-adjusting plate
5 air layer thickness
6 test piece (honeycomb)

Figure 2 — Cross-sectional view of photoreactor

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 minimal test gas and withstand irradiation of near-UV light. The test piece shall be separated from the window by a 5,0 mm ± 0,5 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, for example by using height-adjusting plates with different thicknesses, as shown in Figure 2 a). When a filter-type photocatalyst 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 shall be used for the window.

6.4 Light source

The light source shall provide UV-A illumination 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 4892-3, and xenon lamps with optical filters that block radiation below 300 nm and above 400 nm. The test piece shall be irradiated uniformly through the window by the light source. In the case of 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 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 4892-1. The reactor shall be shielded from external light if necessary.