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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 3: Removal of toluene

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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 the following URL: www.iso.org/iso/foreword.html.

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

A list of all parts in the ISO 17168 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.

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 and ISO 22197-2 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 toluene, a representative aromatic volatile organic compound (VOC), enabling swift distribution of photocatalytic products and thus contributing to a safe and clean environment.

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

Part 3: Removal of toluene

1 Scope

This document specifies a test method for the determination of the air-purification performance, with regards to the removal of toluene, 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/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) (l/min)
ϕ_T	toluene volume fraction at the reactor exit (µl/l)
ϕ_{T0}	supply volume fraction of toluene (µl/l)
ϕ_{TD}	toluene volume fraction at the reactor exit under dark conditions (µl/l)
n_T	removal quantity, by test piece, of toluene (µmol)
R	removal percentage, by test piece, of toluene (%)

5 Principle

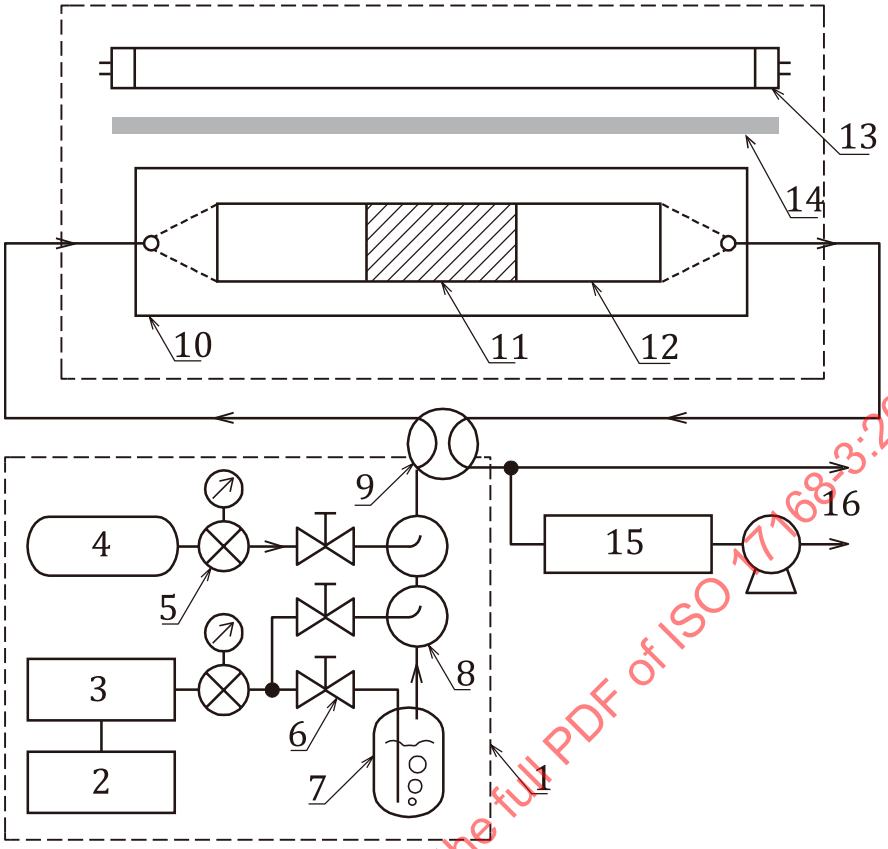
This document deals with the development, comparison, quality assurance, characterization, reliability and design data generation of photocatalytic materials[1]. The method described is intended to obtain the air-purification performance of photocatalytic materials by exposing a test piece to model polluted air under illumination by indoor light[2]. Toluene (C_7H_8) is chosen as a typical aromatic volatile organic compound (VOC) with offensive odour[3]. The test piece, placed in a flow-type photoreactor, is activated by indoor-light illumination, and adsorbs and oxidizes gas-phase toluene to form carbon dioxide (CO_2) and other oxidation products[4]–[6]. The air-purification performance is determined from the amount of toluene removed by the test piece (µmol). The simple adsorption by the test piece (not due to photocatalysis) is evaluated by the tests in the dark. However, some test pieces are very absorbent, and a stable concentration of toluene may not be attained in the designated test time. The photocatalytic activity may depend on the physical and chemical properties of pollutants, mainly due to the adsorption process involved. For a better evaluation of the 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.

The results of an interlaboratory test are given in Annex A.

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 adsorption. 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

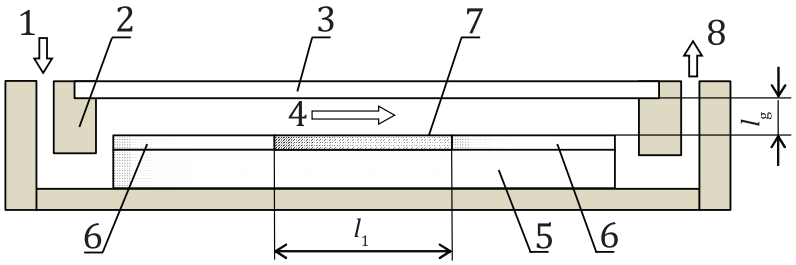
6.2 Test gas supply

The test gas supply provides air polluted with 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 and so on. The flow rate of each gas should be within 5,0 % 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, 101,3 kPa). Typical capacities of flow controller for pollutant gas, dry air and wet air are 10, 500 and 500 ml/min, respectively. The standard toluene gas before dilution, normally balanced with nitrogen in a cylinder, shall have a volume fraction of 10 µl/l to 50 µl/l.

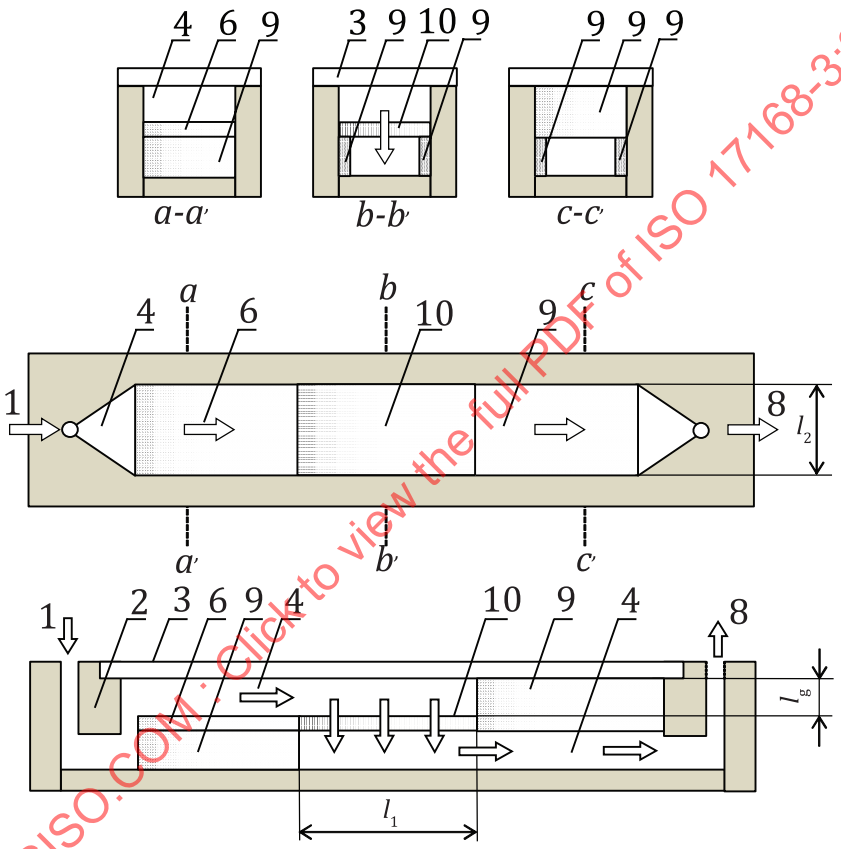
6.3 Photoreactor

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 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 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.



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 baffle
3 airtight 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

6.4 Light source

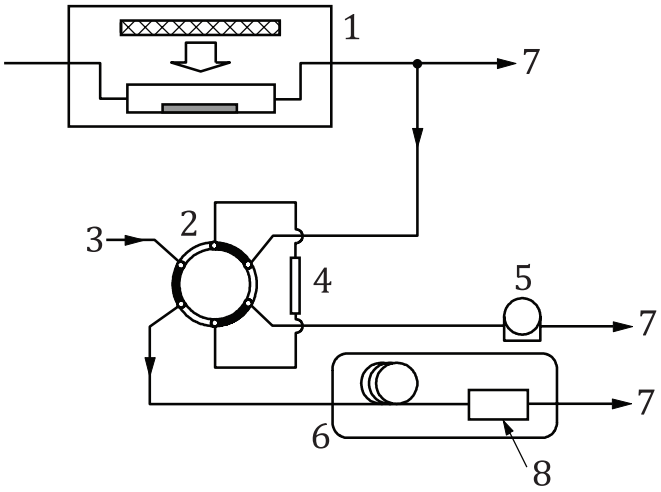
A cool white halophosphate fluorescent lamp and a UV sharp cut-off filter as specified in ISO 14605 shall be used. The test piece shall be illuminated uniformly through the window by the light source. When testing honeycomb-form photocatalysts, the light source shall illuminate one end of the test piece. 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 lx ± 300 lx. The illuminance along the length of the test piece shall also be constant within ± 5 %. The 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 nm), less than 1 % (at 380 nm) and higher than 80 % (400 nm < wavelength < 780 nm), and the wavelength where the transmittance is half of the average transmittance between 400 nm and 780 nm is located at 390 nm ± 5 nm.

6.6 Analytical system

The concentration of toluene shall be determined by gas chromatography. Either packed column or capillary column can be used as long as it can separate toluene from related organic compounds. The detection shall be made by either flame ionization detector (FID) or photoionisation detector (PID). The test gas is 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 FID

Figure 3 — Gas sampling system

7 Test piece

The test piece shall be a flat material or a honeycomb filter 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 5,0 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 quantity and percentage of toluene under photoirradiation. An example of concentration change of toluene during the test is shown in Figure 4. This document cannot be applied to certain test pieces that contain a large amount of adsorbent, due to unattained adsorption equilibrium. Some test pieces may not give accurate removal of toluene due to lower photocatalytic activity. In this case, loading of toluene per test piece can be reduced following the procedure in Clause 10.

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/m^2 – 20 W/m^2).

8.3 Toluene 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 toluene 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 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 so that the flow rate at the inlet of the reactor is 0,5 l/min (0 °C, 101,3 kPa). Measure and record the illuminance from the light source at the surface of the test piece.

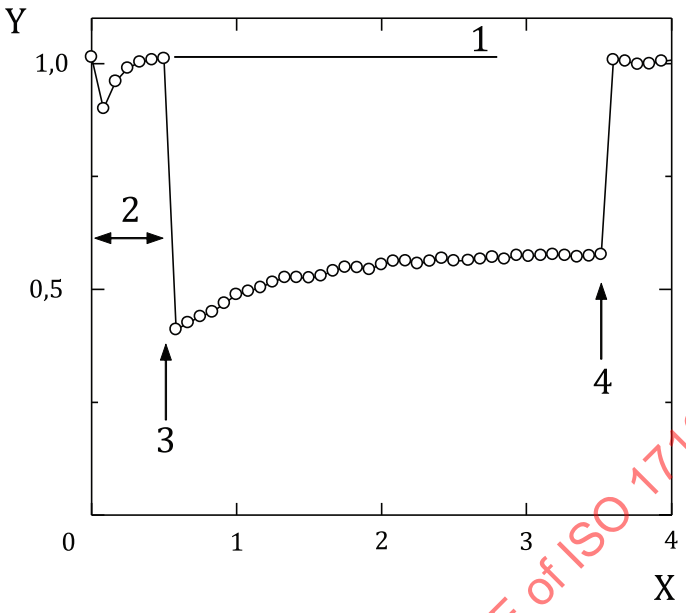
8.3.2 Place the test piece in the centre of the photoreactor and attach the glass window after adjusting the air layer between the test piece and window to a thickness of 5,0 mm ± 0,5 mm. If necessary, height-adjusting plates are used for this purpose, and adjusting based on the top of the test piece. 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.

8.3.4 Allow the test gas to flow into the photoreactor without photoirradiation. The flow rate shall be 0,5 l/min. Record the change in the concentrations of toluene under dark conditions for 30 min. Adsorption of toluene onto the test piece can be observed by this procedure. When the concentration at the outlet of the reactor returns to the supply gas concentration within 30 min, photoirradiation may be starting at the time. If the toluene concentration is less than 90 % of the concentration supplied, continue until it exceeds this. The toluene concentration under dark conditions is defined as the average toluene concentration of the last three measurement points. If the concentration does not exceed 90 % after 90 min, stop measuring and report that the test is not applicable to the test piece used.

8.3.5 Maintain the gas to flow and commence irradiation of the test piece, recording the concentration of toluene under photoirradiation for 3 h. The concentration decreases, as shown in Figure 4, if toluene is decomposed by photocatalyst and then stabilizes. The concentration of toluene at the outlet of the photoreactor shall be the average of three or more measurements in the last 1 h test period.

8.3.6 Stop the photoirradiation and confirm that the concentration of toluene has returned to the supply gas concentration. Stop the gas supply to the reactor and take the test piece out of the reactor.



Key

1 toluene feed level
2 dark conditions
3 lights on
4 lights off
X time (h)
Y toluene concentration (µl/l)

Figure 4 — Typical trace of toluene concentration during the test operation

9 Calculation

The test results shall be calculated as follows. If ϕ_T does not satisfy Formula (1), i.e. the difference between toluene concentrations under dark conditions and those under photoirradiation is less than 5 % of the toluene concentration supplied, this test method shall not be applied. The flow rate of test gas f is the observed flow rate normalized for 0 °C, 101,3 kPa.

The removal percentage of toluene (R) is calculated by Formula (2). The calculated values are usually rounded to one decimal place according to ISO 80000-1. When R is either below 5,0 % or more than 95,0 %, R is expressed as "below 5,0 %" or "more than 95,0 %", respectively. Then, the quantity of toluene removed per hour (n) is calculated by Formula (3). When n_T is below 5,0 % or more than 95,0 %, it is expressed as "below 5,0 %" or "more than 95,0 %", respectively.

$$\phi_{TD} - \phi_T \leq \phi_{T0} \times 0{,}05 \tag{1}$$

$$R = \frac{\phi_{T0} - \phi_T}{\phi_{T0}} \times 100 \tag{2}$$

$$\eta_T = R_T \times \frac{\phi_{T0} \times f \times 60}{100 \times 22{,}4} \tag{3}$$

where

R is the removal percentage, by test piece, of toluene (%);

ϕ_{T0} is the supply volume fraction of toluene (µl/l);

ϕ_T is the toluene volume fraction at reactor exit under indoor light irradiation(µl/l);

ϕ_{TD} is the toluene volume fraction at reactor exit under dark conditions (µl/l);

n_T is the removal quantity, by test piece, of toluene (µmol);

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

10 Test method for test pieces with lower performance

When the removal percentage is less than 5 % and a more certain result is demanded, the number of test pieces may be altered as shown in Table 1. However, the removal quantity of toluene which appears in the test report shall be half of the value calculated from Formula (2).

Table 1 — Alternative test conditions

Alterable test conditions	Value after change
Number of test pieces	Two pieces in series (surface of 50 mm × 200 mm)

11 Test report

The test report shall include the reporting provisions of ISO/IEC 17025 and the following information. Items g), h) and i) shall be reported for each test.

a) the name and address of the test establishment;

b) the date of the test, a unique identification of the report and of each page, customer name and address, signatory of the report;

c) a reference to this document, i.e. ISO 17168-3.

d) date of test, temperature, relative humidity;

e) description of the test piece (e.g. material, size, shape);

f) description of test equipment (e.g. specifications);

g) testing conditions (e.g. kind of pollutant gas, supply concentration, water-vapour concentration, flow rate, kind of light source, kind of UV sharp cut-off filter, illuminance, analyser and illuminance meter used, condition of pretreatment, modification under Clause 10);

h) the amount of toluene removed during the last 1 h, and removal percentage of toluene (optional); if the test or CO_2 measurement is not valid, the reasons for that;

i) any comments or observations, such as a change in the test piece noticed during the test.