INTERNATIONAL STANDARD

ISO 22197-3

Second edition
2019-10

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

Part 3: Removal of toluene

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

Partie 3: Élimination du toluène

Reference number
ISO 22197-3:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 22197-3:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— deletion of reference to ISO 2718 (withdrawn) from Clause 2 and 6.5;

— deletion of ISO 4677-1 (withdrawn) from Clause 2 and 8.3.1;

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

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

— addition of procedures for removing water-soluble contaminants (8.2);

— addition of criterion for acceptable adsorption of toluene (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 3: Removal of toluene

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 ultraviolet light (UV-A).

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 toluene.

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

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*

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

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 oxidization 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, 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 described in 3.1.

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 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 or a zero-calibration gas, to be used for the performance test of a photocatalytic material

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_T	volume fraction of toluene 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 condition (µl/l)
n_T	quantity of toluene removed by the test piece (µmol)
R	removal percentage, by test piece, of toluene (%)

5 Principle

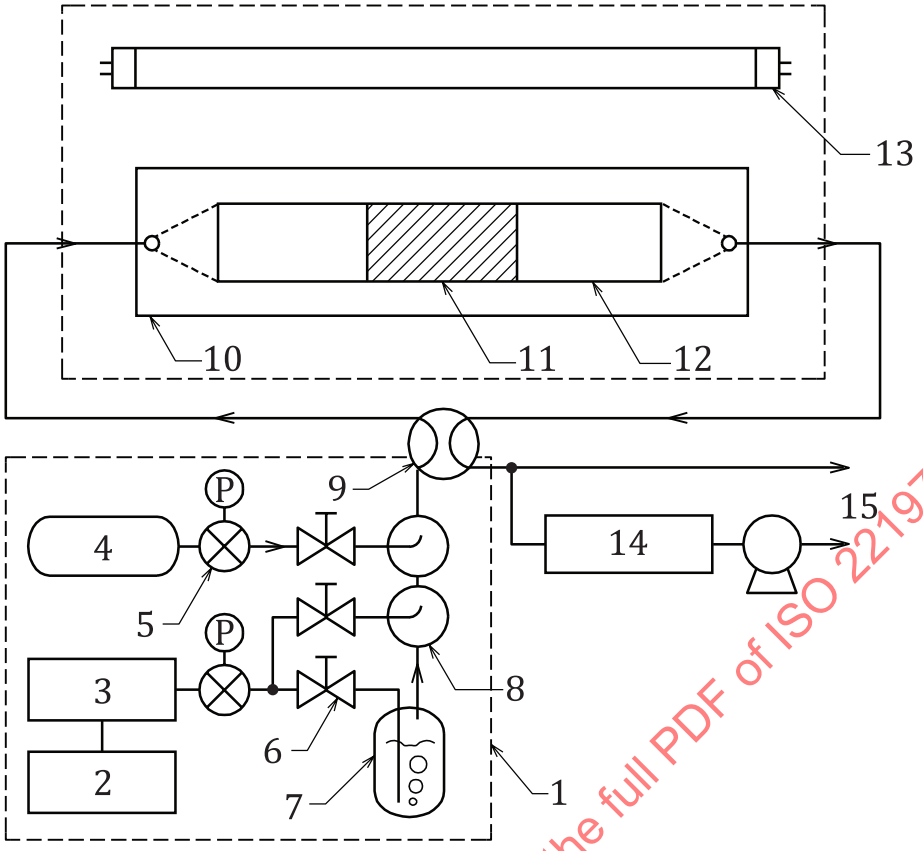
This document concerns the development, comparison, quality assurance, characterization, reliability, and design data generation of photocatalytic materials (see Reference [1]). The method described is intended to obtain the air-purification performance of photocatalytic materials by exposing a test piece to model polluted air under irradiation by ultraviolet (UV) light (Reference [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 UV irradiation, and adsorbs and oxidizes gas-phase toluene to form carbon dioxide (CO_2) and other oxidation products (References [4] to [6]). The air-purification performance is determined from the amount of toluene removed by the test piece, in

micromoles (μmol). The simple adsorption by the test piece (not due to photocatalysis) is evaluated by tests in the dark. However, some test pieces adsorb toluene very strongly, and a stable concentration of toluene may not be attained in the designated time of test. 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 described in other parts of the ISO 22197 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 (ISO 22197-1[2]) 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 adsorption and resistant to UV radiation (e.g. acrylic resin, borosilicate glass). An example of a testing system is shown in Figure 1.



Key

1	test gas supply	9	4-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 test equipment

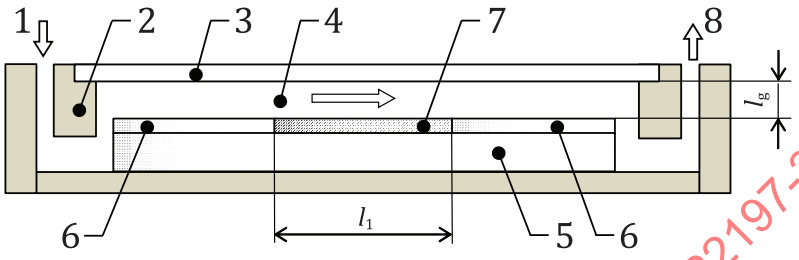
6.2 Test gas supply

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 forth. 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 the temperature and gas type at calibration in accordance with ISO 6145-7[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 10 ml/min, 500 ml/min 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.

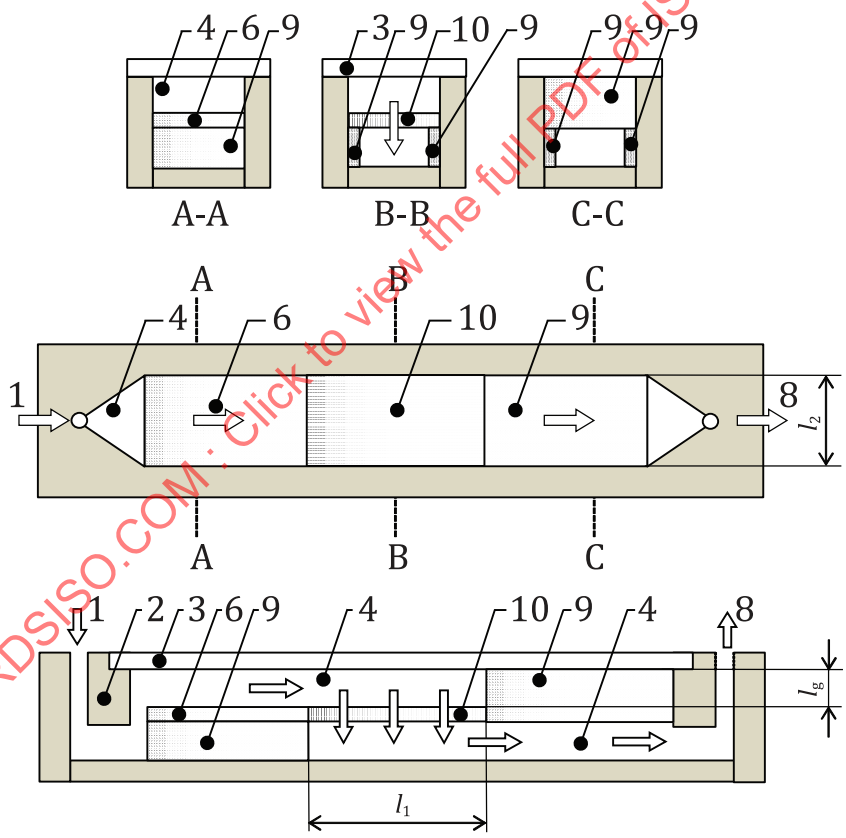
6.3 Photoreactor

The photoreactor holds a planar test piece within a 50 mm-wide trough, with its surface parallel to an 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 irradiation [Figure 2 b)]. Quartz or borosilicate glass that absorbs minimal light at wavelengths longer than 300 nm shall 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	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)

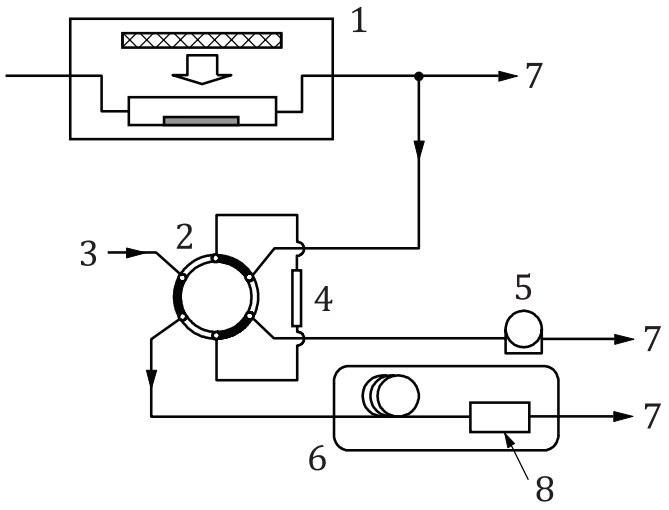
Figure 2 — Cross-sectional views of photoreactor

6.4 Light source

The light source shall provide UV-A 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 or 368 nm, as specified in ISO 4892-3, and xenon arc lamps with optical filters that block radiation below 300 nm. In the case of a xenon arc lamp, a cooling system shall be used in accordance with ISO 10677. The test piece shall be irradiated uniformly through the window by the light source. In the case of testing filter-type photocatalysts, the light source shall irradiate 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 UV irradiance (300 nm to 400 nm) at the sample surface is 10 W/m^2 ± 0,5 W/m^2. This distance shall be determined independently without using the photoreactor. A UV radiometer in conformity with ISO 10677 shall be put behind the optical window or its equivalent, at the same level as the test piece to be tested. The irradiance along the length of the test piece shall also be constant within ±5 %. The reactor shall be shielded from external light if necessary.

6.5 Analytical system

The concentration of toluene shall be determined by gas chromatography. Either a 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 a flame ionization detector (FID) or a photoionization detector (PID). The test gas is sampled with a gastight 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	5	sampling pump
2	six-way valve	6	gas chromatograph
3	carrier gas	7	vent
4	metering tube	8	FID

Figure 3 — Gas sampling system

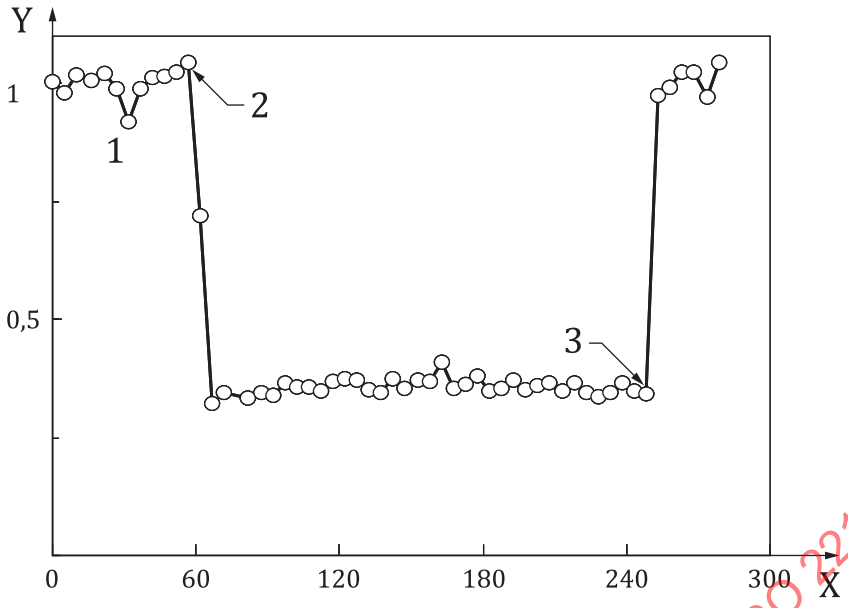
7 Test piece

The test piece shall be a flat material or a filter-type material 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 precut 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 quantity and percentage of toluene under photoirradiation. An example of the 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, the loading of toluene per test piece can be reduced following the procedure in Clause 10. Results of round-robin test are reported in Annex A.



Key

X time (min)
Y concentration of toluene (µl/l)
1 under dark
2 irradiation start
3 irradiation stop

Figure 4 — Typical trace of toluene 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 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 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 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 1,0 µl/l ± 0,05 µl/l of toluene 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 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 0,500 l/min ± 0,025 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 toluene 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. If necessary, height-adjusting plates are used for this purpose, adjusting the height before and after the test piece to be within 1,0 mm difference 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 contact the glass window.

8.3.3 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 started at the time. If the toluene concentration is less than 90 % of the concentration supplied, continue until it exceeds this. If the concentration does not exceed 90 % after 90 min, stop measurement and report that this test is not applicable to the test piece used.

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

8.3.5 Stop photoirradiation and confirm that the concentration of toluene returns to supply gas concentration. Stop the gas supply to the reactor and take the test piece out of the reactor.

9 Calculation

The test results shall be calculated as follows. If ϕ_T does not satisfy Formula (1), meaning that the difference between toluene concentrations under dark condition and those under photoirradiation is less than 5 % of the toluene concentration supplied as Formula (1), this test method shall not be applied. The calculated values shall be rounded to one decimal place in accordance with ISO 80000-1. The flow rate of the test gas f is 0,5 (l/min) at the standard state (0 °C and 101,3 kPa).

The removal percentage of toluene (R) is calculated using Formula (2). When R is either below 5 % or more than 95 %, it is expressed as “below 5 %” or “more than 95 %”, respectively. Then the quantity of toluene removed (n_T) is calculated using Formula (3). When n_T is either below 5 % or more than 95 %, it is expressed as “below 5 %” or “more than 95 %”, respectively.

$$\varphi_T \leq \varphi_{TD} - \varphi_{T0} \times 0,05 \quad (1)$$

$$R = \frac{\phi_{T0} - \phi_T}{\phi_{T0}} \times 100 \quad (2)$$

$$n_T = R \times \frac{\phi_{T0} \times f \times 60}{100 \times 22,4} \quad (3)$$

where

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

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

ϕ_{T0} is the supply volume fraction of toluene, in microlitres per litre (µl/l);

ϕ_T is the toluene volume fraction at the reactor exit, in microlitres per litre (µl/l);

n_T is the quantity of toluene removed by the test piece, in micromoles (µmol);

f is the flow rate of test gas converted into that at the standard state (0,5 l/min, 0 °C and 101,3 kPa).

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 to appear in the test report shall be half of the value calculated from Formula (3).

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 shall include the following information. Items g), h) and i) shall be reported for each test.

a) The name and address of the testing establishment.

b) The date of the test, a unique identification of the report and of each page, the customer's name and address, signatory of the report.

c) A reference to this document, i.e. determined in accordance with ISO 22197-3:2019.

d) Date of test, atmospheric temperature, relative humidity.

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

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

g) Testing conditions (e.g. kind of pollutant gas, supply concentration, water-vapour concentration, flow rate, detailed description of light source, irradiance, analyser and radiometer used, conditions of pretreatment, modification under Clause 10).

h) The amount of toluene removed during the final 1 h, and removal percentage of toluene (optional). If the test is not valid, the reasons for that (e.g. strong adsorption of toluene).

i) Any other matters of special importance, such as a change in the test piece noticed during the test.