BS ISO 22197-4:2021

BSI Standards Publication

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

Part 4: Removal of formaldehyde

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National foreword

This British Standard is the UK implementation of ISO 22197-4:2021. It supersedes BS ISO 22197-4:2013, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Published by BSI Standards Limited 2021

ISBN 978 0 539 15210 4

ICS 81.060.30

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2021.

Amendments/corrigenda issued since publication

Date	Text affected

BS ISO 22197-4:2021

INTERNATIONAL STANDARD

ISO 22197-4

Second edition
2021-07-05

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

Reference number
ISO 22197-4:2021(E)

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Foreword

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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).

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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.

INTERNATIONAL STANDARD ISO 22197-4:2021

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

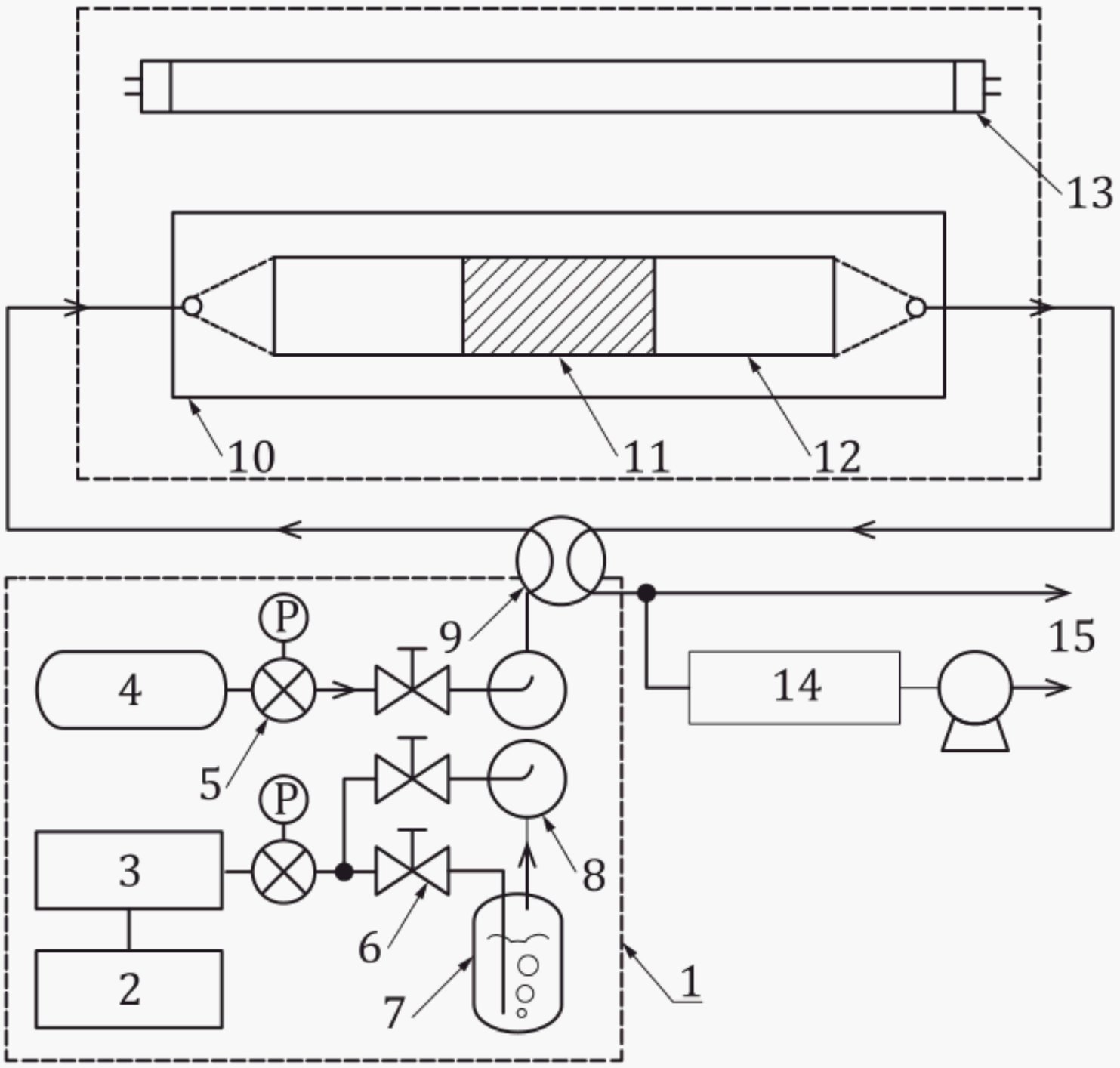
— IEC Electropedia: available at http://www.electropedia.org/

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

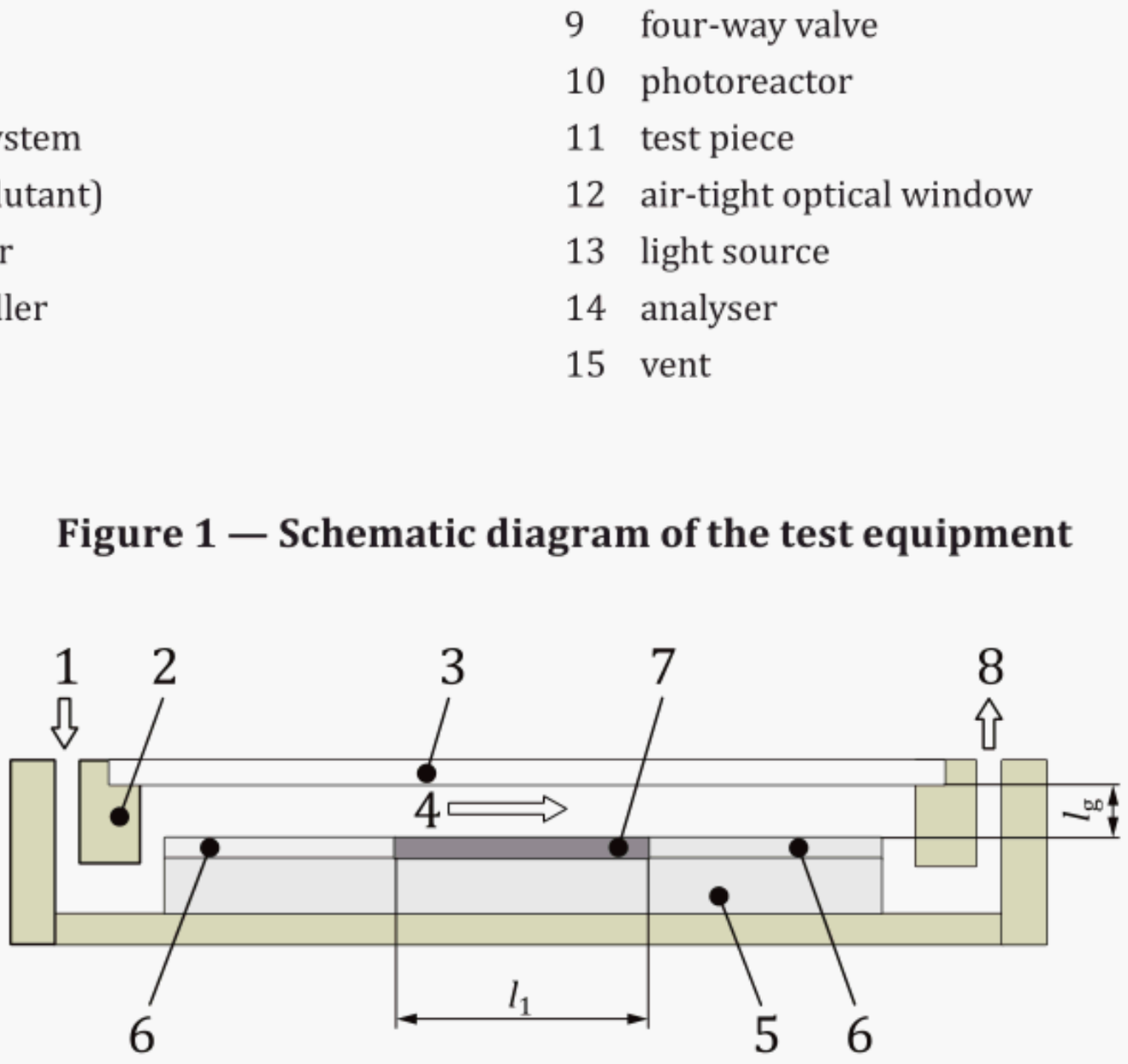
The test equipment enables a photocatalytic material to be examined for its pollutant-removal capability by supplying the test gas continuously, while providing photoirradiation to activate the photocatalyst. It is the same as that used in the test method for the removal of nitric oxide (see ISO 22197-1) 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.



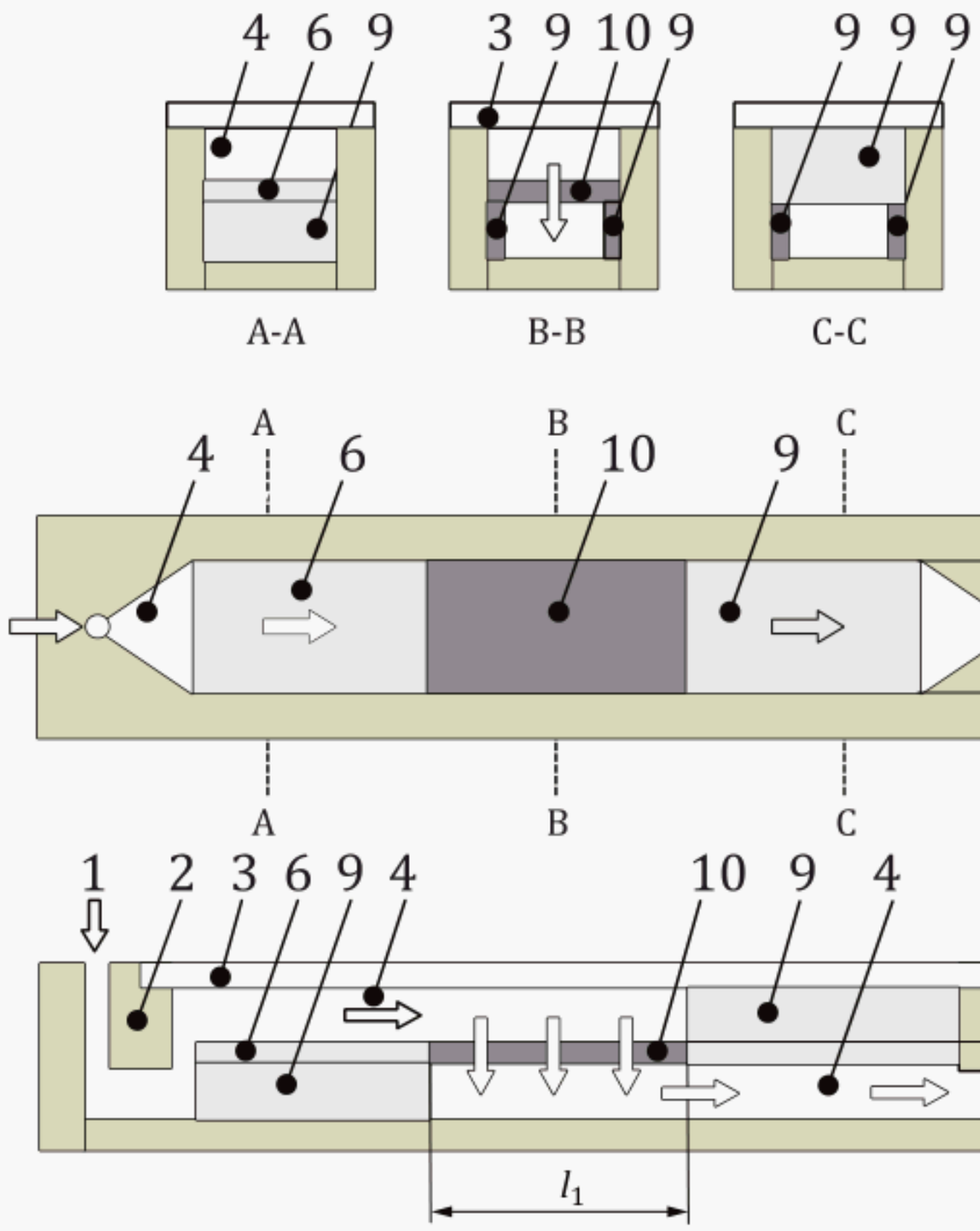
Key

1	test gas supply
2	air compressor
3	air-purification system
4	standard gas (pollutant)
5	pressure regulator
6	mass-flow controller
7	humidifier
8	gas mixer
9	four-way valve
10	photoreactor
11	test piece
12	air-tight optical window
13	light source
14	analyser
15	vent

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

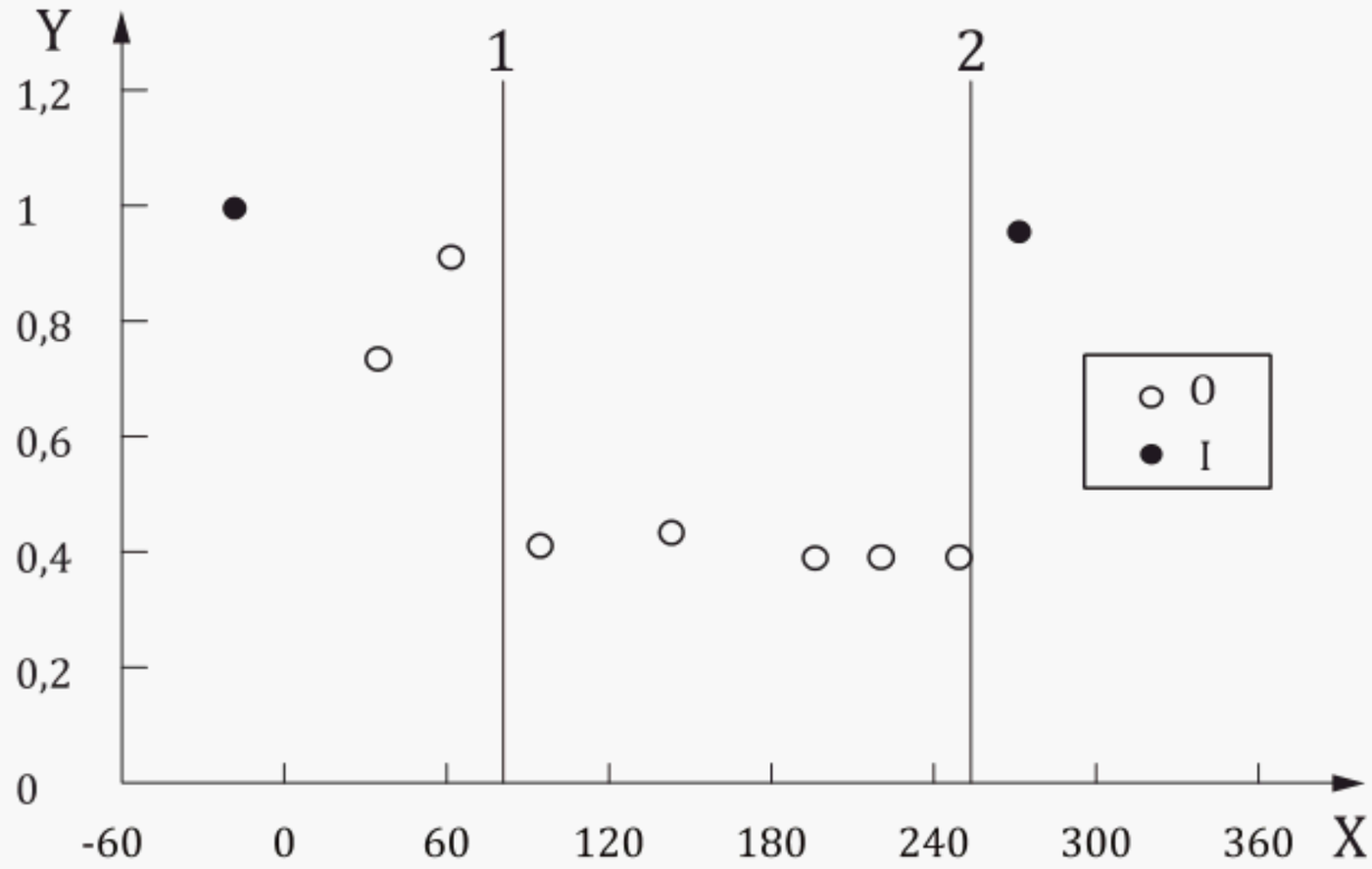
Key

1	test gas inlet	6	auxiliary plate
2	buffle	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 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.



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.

9 Calculation

The test results shall be calculated as follows. The calculated values shall be rounded to one decimal place. The flow rate of test gas f is 3,0 l/min at the standard state (0 °C and 101,3 kPa).

If ϕ_F does not satisfy Formula (1), meaning that the difference between formaldehyde concentrations under dark condition and under photoirradiation is less than 5 % of the formaldehyde concentration supplied, this test method shall not be applied. The removal percentage of formaldehyde (R_F) is calculated by Formula (2). When R_F is either below 5 % or more than 95 %, R_F is expressed as “below 5 %” or “more than 95 %”, respectively. Then, the quantity of formaldehyde removed (n_F) is calculated

by Formula (3). When R_F is either below 5 % or more than 95 %, n_F is expressed as "below (0,134 $\phi_{F0} f$) µmol" or "more than (2,545 $\phi_{F0} f$) µmol", respectively.

$$\phi_F \leq \phi_{FD} - \phi_{F0} \times 0{,}05 \quad (1)$$

$$R_F = \frac{\phi_{F0} - \phi_F}{\phi_{F0}} \times 100 \quad (2)$$

$$n_F = R_F \times \frac{\phi_{F0} \times f \times 60}{100 \times 22{,}4} \quad (3)$$

where

R_F is the removal percentage, by test piece, of formaldehyde (%);
n_F is the removal quantity, by test piece, of formaldehyde (µmol);
ϕ_{F0} is the supply volume fraction of formaldehyde (µl/l);
ϕ_F is the volume fraction of formaldehyde at the reactor exit (µl/l);
ϕ_{FD} is the formaldehyde volume fraction at the reactor exit under dark conditions (µl/l);
f is the flow rate of test gas converted into that at the standard state (0 °C and 101,3 kPa) (l/min).

10 Test method for test pieces with lower performance

In cases where the removal percentage is less than 5 % and a more certain result is demanded, the number of test pieces and the flow rate of test gas may be altered at the same time as shown in Table 1. However, the removal quantity of formaldehyde to appear in the test report shall be half of the values calculated from Formula (3), as well as using the flow rate of 1,5 l/min. When the test conditions are altered, it is necessary to confirm the time of adsorption (dark condition) at the altered test conditions.

Table 1 — Alternative test conditions

Alternative test conditions	Value after change
Test gas flow rate	1,5 l/min ± 0,075 l/min
Number of test pieces	Two pieces

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, the signatory of the report.

c) A reference to this document, i.e. determined in accordance with ISO 22197-4:2021.

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

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

f) Description of the test equipment (specifications).

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

h) The amount of formaldehyde removed during the final hour, removal percentage of formaldehyde (optional).

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

Bibliography

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

[2] ISO 22197-1, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for air-purification performance of semiconducting photocatalytic materials — Part 1: Removal of nitric oxide*

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