

BS ISO 22197-2:2011



BSI Standards Publication

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

Part 2: Removal of acetaldehyde

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of ISO 22197-2:2011.

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 secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© BSI 2011

ISBN 978 0 580 62882 5

ICS 81.060.30

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2011.

Amendments issued since publication

Date	Text affected
------	---------------

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

**Part 2:
Removal of acetaldehyde**

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

Partie 2: Élimination de l'acétaldéhyde



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.



COPYRIGHT PROTECTED DOCUMENT

© ISO 2011

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Symbols	3
5 Principle	3
6 Apparatus	3
6.1 Test equipment	3
6.2 Test gas supply	4
6.3 Photoreactor	5
6.4 Light source	6
6.5 Analytical system for acetaldehyde	6
6.6 Analytical system for CO ₂	7
7 Test piece	7
8 Procedure	7
8.1 General aspects	7
8.2 Pretreatment of test piece	8
8.3 Preparation for the test	8
8.4 Pretest	8
8.5 Test of acetaldehyde removal and CO ₂ conversion	8
8.6 Test of acetaldehyde removal (when the CO ₂ concentration cannot be measured)	9
9 Calculation	10
9.1 Calculation method	10
9.2 Removal percentage and removed quantity of acetaldehyde	10
9.3 Conversion to CO ₂	11
10 Test method for test pieces with lower performance	11
11 Test report	12
Annex A (informative) Results of round-robin test	13
Bibliography	14

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 22197-2 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*
- *Part 2: Removal of acetaldehyde*
- *Part 3: Removal of toluene*
- *Part 4: Removal of formaldehyde*
- *Part 5: Removal of methyl mercaptan*

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

Part 2: Removal of acetaldehyde

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, 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 (UV-A). 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 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 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 acetaldehyde.

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 80000-1:2009, *Quantities and units — Part 1: General*

ISO 2718:1974, *Standard layout for a method of chemical analysis by gas chromatography*

ISO 4224:2000, *Ambient air — Determination of carbon monoxide — Non-dispersive infrared spectrometric method*

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

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*

ISO 10677:—¹⁾, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ultraviolet light source for testing semiconducting photocatalytic materials*

ISO 16000-3:2001, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds — Active sampling method*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

ISO 22197-1:2007, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for air-purification performance of semiconducting photocatalytic materials — Part 1: Removal of nitric oxide*

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 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 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 and carbon dioxide is below 0,1 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.

NOTE The flow rate, concentration, etc., are expressed at the standard state (0 °C, 101,3 kPa) and dry-gas basis (exclusion of water vapour).

3.6 dark condition

test condition with no light illumination by the light source for testing and room lighting

NOTE Usually the test gas is supplied for comparison with the illuminated reaction.

1) To be published.

4 Symbols

For the purposes of this document, the following symbols apply.

f	the air-flow rate converted into that at the standard state (0 °C and 101,3 kPa, and dry-gas basis) (l/min)
ϕ_A	the volume fraction of acetaldehyde at the reactor exit ($\mu\text{l/l}$)
ϕ_{A0}	the volume fraction of acetaldehyde in the test gas ($\mu\text{l/l}$)
ϕ_{CO_2}	the carbon dioxide (CO_2) volume fraction generated by UV irradiation ($\mu\text{l/l}$)
$\phi_{\text{CO}_2,\text{L}}$	the CO_2 volume fraction at the reactor exit under UV irradiation ($\mu\text{l/l}$)
$\phi_{\text{CO}_2,\text{D}}$	the CO_2 volume fraction at the reactor exit under dark conditions ($\mu\text{l/l}$)
$\phi_{\text{CO}_2,\text{Dpost}}$	the CO_2 volume fraction in the dark after UV irradiation ($\mu\text{l/l}$)
$\phi_{\text{CO}_2,\text{Dpre}}$	the CO_2 volume fraction in the dark before UV irradiation ($\mu\text{l/l}$)
n_A	the quantity of acetaldehyde removed by the test piece (μmol)
n_{CO_2}	the quantity of CO_2 converted from acetaldehyde, in micromoles (μmol)
R_A	the removal percentage, by test piece, of acetaldehyde (%)
R_{CO_2}	the conversion from acetaldehyde to CO_2 (%)

5 Principle

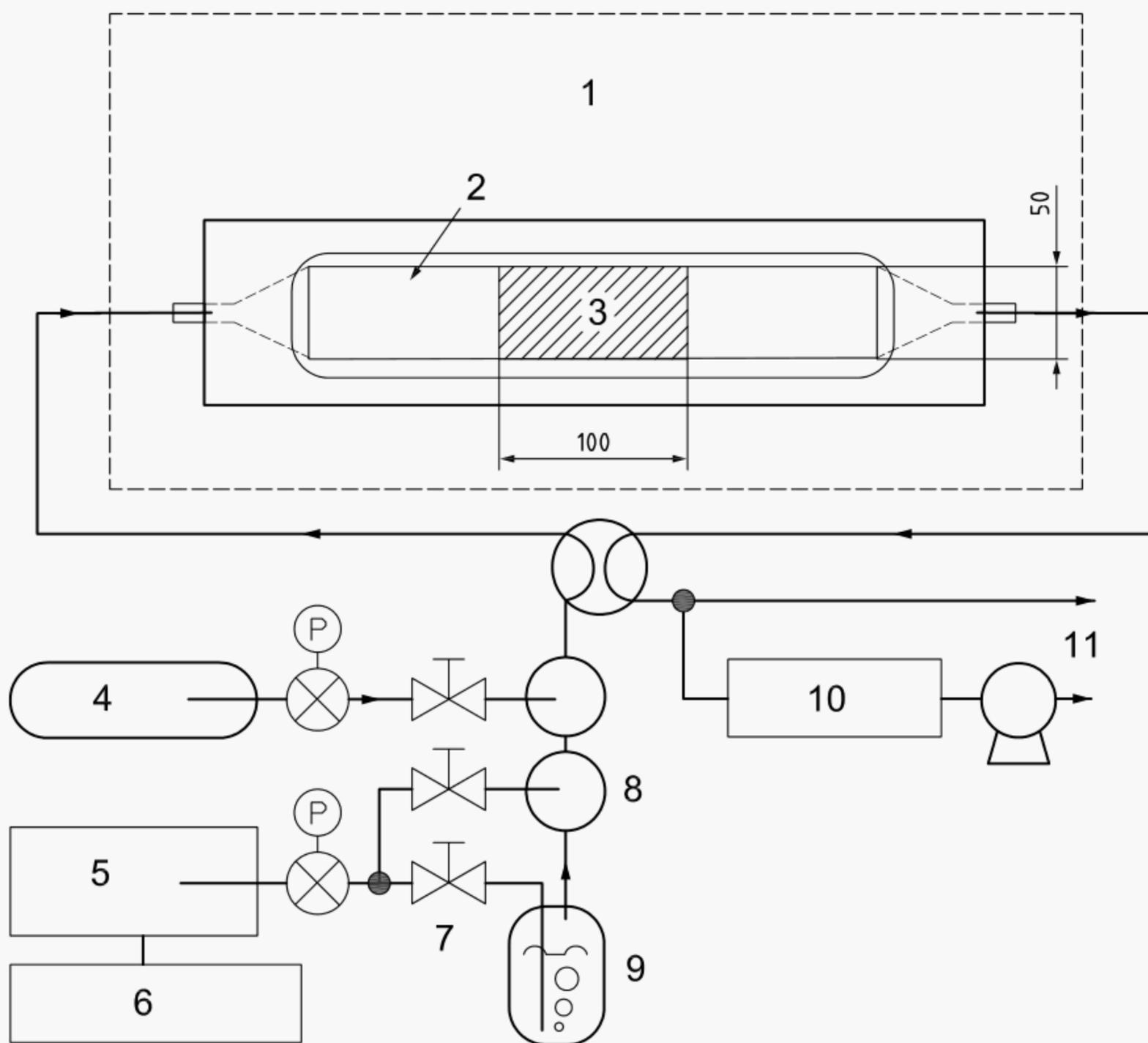
This part of ISO 22197 concerns the development, comparison, quality assurance, characterization, reliability, and design data generation of photocatalytic materials (Reference [3] in the Bibliography). The method described is intended to obtain the air-purification performance of photocatalytic materials by exposing a test piece to model polluted air under illumination by ultraviolet (UV) light (Reference [4] in the Bibliography). Acetaldehyde (CH_3CHO) is chosen as a typical volatile organic compound (VOC) with lower molecular mass and offensive odour. The test piece, put in a flow-type photoreactor, is activated by UV illumination, and adsorbs and oxidizes gas-phase acetaldehyde to form carbon dioxide (CO_2) and other oxidation products. The air-purification performance is determined from the amount of acetaldehyde, in percent, adsorbed by the test piece, in micromoles (μmol). The simple adsorption by the test piece (not due to photocatalysis) is evaluated by the tests in the dark. However, some test pieces adsorb acetaldehyde very strongly, and a stable concentration of acetaldehyde 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 to combine one or more suitable test methods as described in other parts of ISO 22197.

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) 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 ultraviolet (UV) radiation (e.g. acrylic resin, borosilicate glass). An example of a testing system is shown in Figure 1.

Dimensions in millimetres



Key

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

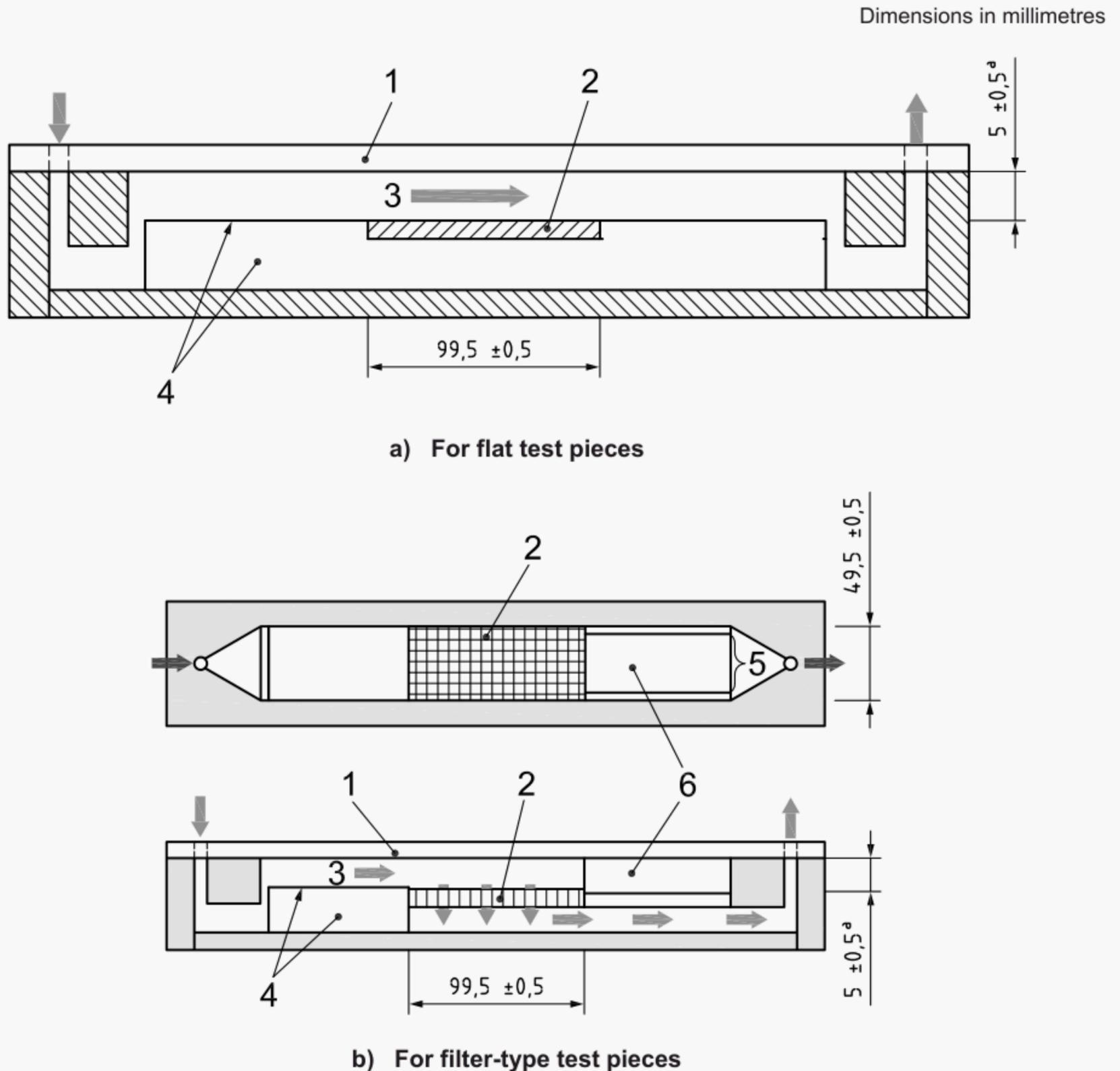
Figure 1 — Schematic diagram of test equipment

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, 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 knowledge of the temperature and gas type at calibration in accordance with ISO 6145-7. 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). Typical capacities of flow controller for pollutant gas, dry air and wet air are 100 ml/min, 1 000 ml/min and 1 000 ml/min, respectively. The standard acetaldehyde gas before dilution, normally balanced with nitrogen in a cylinder, shall have a volume fraction of 50 µl/l to 250 µl/l. Synthetic air (N₂ + O₂, such as that supplied in cylinders) shall be used for dilution when the CO₂ from acetaldehyde is also measured.

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 $5,0 \text{ mm} \pm 0,5 \text{ mm}$ 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.



Key

- | | |
|--------------|--------------------------|
| 1 window | 4 height-adjusting plate |
| 2 test piece | 5 flow channel |
| 3 test gas | 6 test-piece holder |

^a Air-layer thickness.

Figure 2 — Cross-sectional view of photoreactor

6.4 Light source

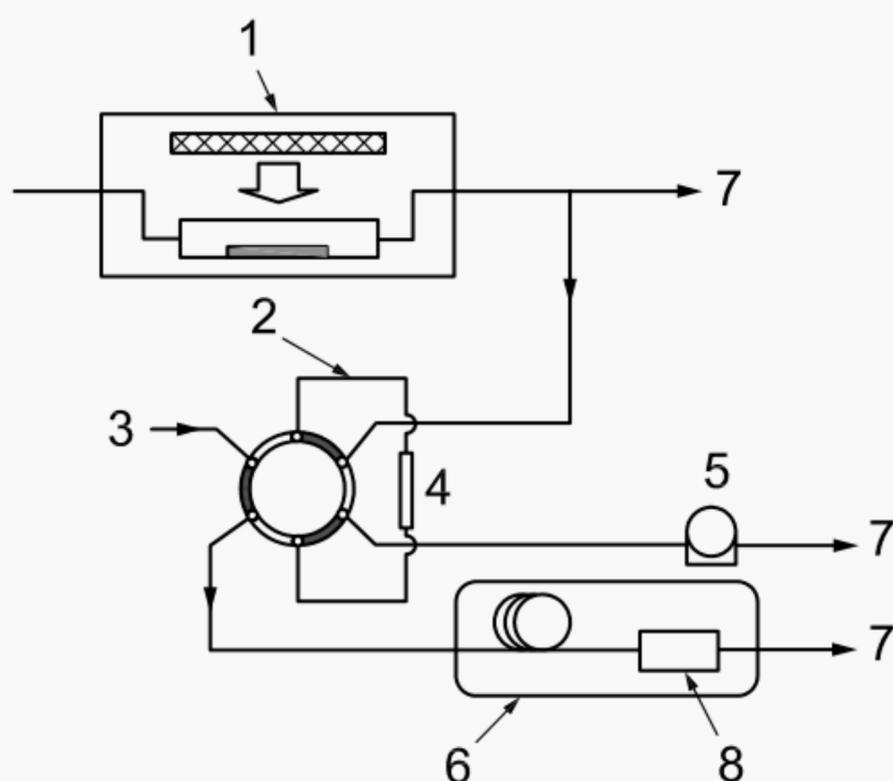
The light source shall provide UV-A illumination within a wavelength range of 300 nm to 400 nm. Suitable sources include the so-called black light (BL) and black light blue (BLB) fluorescent lamps, with a maximum at 351 nm or 368 nm, as specified in ISO 10677, and xenon arc lamps with optical filters 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 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 UV irradiance (300 nm to 400 nm) at the sample surface is $10 \text{ W/m}^2 \pm 0,5 \text{ W/m}^2$. 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 $\pm 5 \%$. The reactor shall be shielded from external light if necessary.

6.5 Analytical system for acetaldehyde

The concentration of acetaldehyde shall be determined by gas chromatography or 2,4-dinitrophenylhydrazine-derivatized high-performance liquid chromatography (DNPH/HPLC).

In the case of gas chromatography, either a packed column or capillary column, as described in ISO 2718, can be used, as long as it can separate lower organic compounds. The detection shall be made by either a flame ionization detector (FID) or 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.

In the case of the DNPH/HPLC method, the reagents, equipment and procedure specified in ISO 16000-3 shall be used.



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

6.6 Analytical system for CO₂

The concentration of CO₂ shall be determined using a non-dispersive infrared CO₂ analyser or a gas chromatograph with a methanizer furnace. Calibration of the system shall be done in accordance with ISO 4224 or ISO 2718. In the case of gas chromatography, the test gas shall be sampled as described in 6.5.

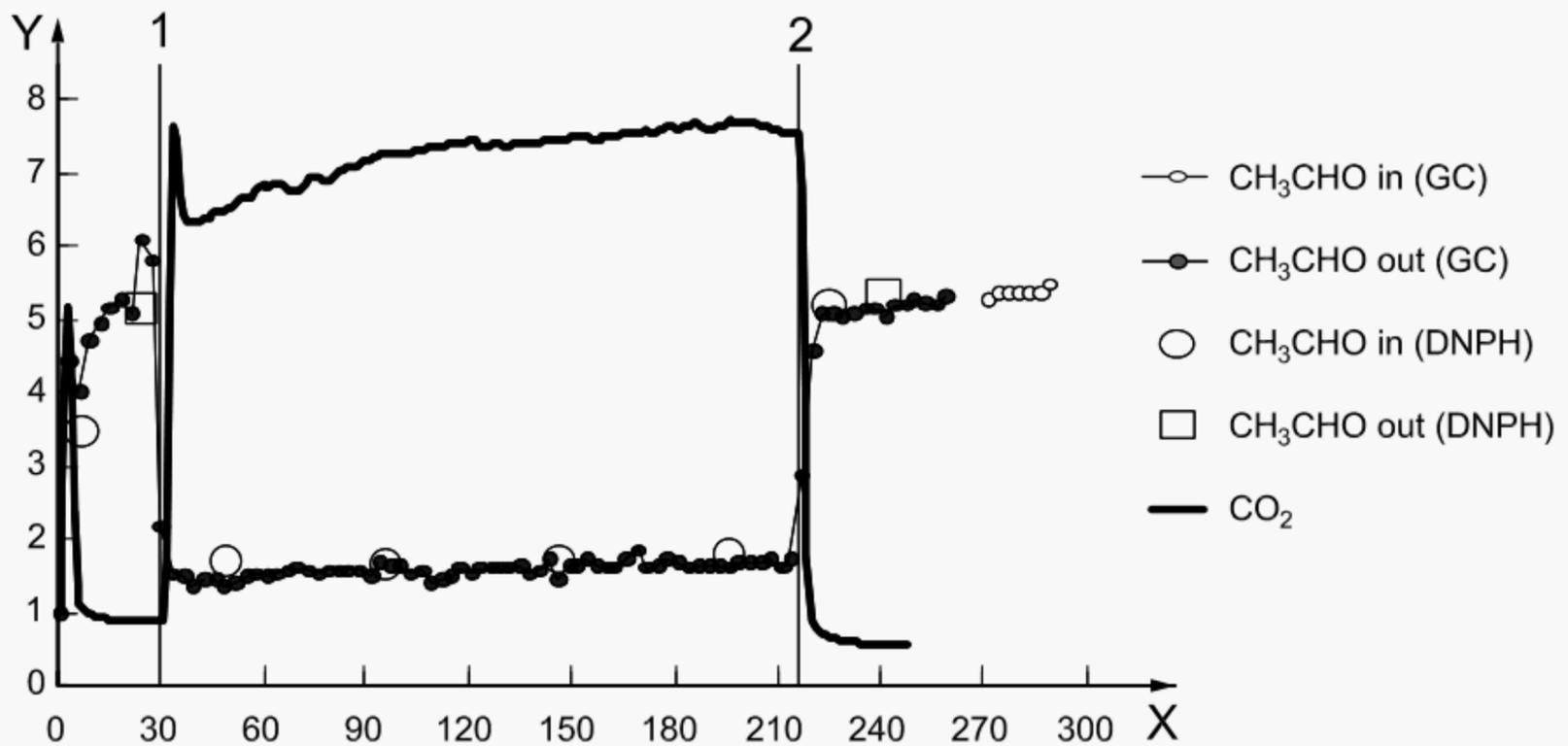
7 Test piece

The test piece shall be a flat material or a filter-type material 49,5 mm ± 0,5 mm wide and 99,5 mm ± 0,5 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 acetaldehyde and formation of CO₂ under photoirradiation. An example of the concentration change of acetaldehyde and CO₂ during the test is shown in Figure 4. The measurement of CO₂ may not always be feasible for some test pieces. Some test pieces may not give accurate removal of acetaldehyde due to lower photocatalytic activity. In this case, the loading of acetaldehyde per test piece can be reduced following the procedure in Clause 10.



Key

- X time (min)
 Y CH₃CHO, CO₂ (µl/l)
- 1 irradiation start
 2 irradiation stop

Figure 4 — Typical trace of acetaldehyde and CO₂ concentration during the test operation

8.2 Pretreatment of test piece

Irradiate the test piece with an ultraviolet lamp for at least 16 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 (15 W/m^2 or higher). If the test pieces are not to be tested immediately after this pretreatment, they shall be kept in an airtight container.

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 $5,0 \mu\text{l/l} \pm 0,25 \mu\text{l/l}$ of acetaldehyde and $1,56 \% \pm 0,16 \%$ of volume fraction of water vapour at $25,0 \text{ }^\circ\text{C} \pm 2,5 \text{ }^\circ\text{C}$. This water-vapour volume fraction is equivalent to a relative humidity of 50 % at $25 \text{ }^\circ\text{C}$. The measurement of humidity shall be made using the procedure in ISO 4677-1. Adjust the flow regulator in order for the flow rate at the inlet of the reactor to be $1,0 \text{ l/min}$ ($0 \text{ }^\circ\text{C}$, $101,3 \text{ kPa}$, and dry-gas basis). 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 acetaldehyde 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 $5,0 \text{ mm} \pm 0,5 \text{ mm}$ thick. If necessary, height-adjusting plates are used for this purpose, adjusting the height before and after the test piece to be within $1,0 \text{ mm}$ 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.

8.4 Pretest

If the concentration of acetaldehyde is determined by the DNPH/HPLC method, the concentration cannot be obtained instantaneously. Therefore, the time of the adsorption of acetaldehyde reaching saturation in a dark condition cannot be confirmed during the test. For this reason, the following pretest shall be carried out. If the concentration is determined by gas chromatography and 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 acetaldehyde under the dark condition every 15 min for 90 min. When the concentration at the outlet of the reactor becomes the same as the supply gas concentration within 30 min, then that time may be the time of the dark condition. When the concentration of acetaldehyde is less than 90 % of the concentration after 30 min, the time of the dark condition shall be the time when the concentration exceeds 90 % for the first time. When the concentration of acetaldehyde is still less than 90 % of the concentration after 90 min, then this part of ISO 22197 shall not apply.

8.5 Test of acetaldehyde removal and CO_2 conversion

8.5.1 In order to reduce the concentration of CO_2 in zero-calibration gas, carry out the test as follows using synthetic air in a gas cylinder with less than $0,1 \mu\text{l/l}$ of CO_2 .

8.5.2 Place the test piece according to 8.3. When the test piece used during pretesting is reused, the pretreatment described in 8.2 shall be done again.

8.5.3 Supply a large enough volume of air that does not contain CO_2 into the photoreactor to purge CO_2 from the system.

The flow rate shall not necessarily be $1,0 \text{ l/min}$. The higher the flow rate is, the faster the CO_2 in the system can be removed.

8.5.4 Supply zero-calibration gas with 1,56 % volume fraction of water vapour at $25,0\text{ °C} \pm 2,5\text{ °C}$ and measure the concentration of CO_2 . Make sure that the concentration of CO_2 is low and stable. Measure the concentration of CO_2 under illumination. Then turn the light off, and measure the concentration of CO_2 again. If the difference of CO_2 concentration is less than $1\text{ }\mu\text{l/l}$ between measurement with the light on and off, move forward to the next step. If a large amount of CO_2 is observed to generate when the light is turned on, it is conceivable that the test piece is not well pretreated and is contaminated with organic matter, or the binder in the test piece has been decomposed by UV irradiation. When the previously cited condition is not met even after pretreatment of enough UV irradiance, etc., then do not measure the CO_2 conversion (proceed to 8.6) and report that the test piece is not stable after pretreatment.

The change of CO_2 concentration in 30 min shall be no higher than $0,1\text{ }\mu\text{l/l}$.

8.5.5 Get ready for measurement using the procedure described in 8.3.1.

8.5.6 If the pretest has been done, supply the test gas into the photoreactor until the time of the dark condition (adsorption) which has been checked beforehand (if the time is less than 30 min, supply for 30 min) and measure the concentration of CO_2 . If the pretest has not been done, proceed as follows. At first, supply the test gas to the photoreactor, and record the change in the concentrations of acetaldehyde and CO_2 under the dark condition, to examine the adsorptivity of the test piece in 30 min. However, if the concentration of acetaldehyde is less than 90 % of the supply gas concentration after 30 min, continue until it exceeds this percentage. 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. The average of CO_2 concentrations measured for the last 30 min (more than 2 points) is to be the concentration of CO_2 ($\phi_{\text{CO}_2, \text{Dpre}}$) under the dark condition before UV irradiation.

8.5.7 Maintain the gas flow and commence irradiation of the test piece, and record the concentration of acetaldehyde and CO_2 under photoirradiation for 3 h continuously. As photocatalytic decomposition of acetaldehyde occurs, the concentration of acetaldehyde lowers and the concentration of CO_2 grows as shown in Figure 4, and they become stable in time. Continuous measurement of the concentrations is desired; however, if there is a limitation for the number of measurements, at least 1 point of measurement within 1 h has to be done. In addition, more than 3 points of measurement have to be done in the last 1 h (120 min to 180 min after irradiation). The concentration of acetaldehyde (ϕ_{A}) to be used for calculation of removal rate and the concentration of CO_2 ($\phi_{\text{CO}_2, \text{L}}$) is to be the mean value (more than 3 points) of the concentration measured during the last 1 h. If the relative standard deviation of the last 3 measurements of ϕ_{A} exceeds 20 %, report this instability with possible reasons, such as deactivation of the test piece.

8.5.8 Stop photoirradiation, bring back to the dark condition and measure the concentration of CO_2 . After the concentration of CO_2 has settled, the mean value (for more than 2 points) during this 30 min period is reported as the CO_2 concentration under dark conditions after UV irradiation ($\phi_{\text{CO}_2, \text{Dpost}}$).

The change of CO_2 concentration in 30 min shall be no higher than $0,1\text{ }\mu\text{l/l}$.

8.5.9 Stop the test gas supply to the reactor, and remove the test piece from the reactor.

8.6 Test of acetaldehyde removal (when the CO_2 concentration cannot be measured)

8.6.1 Pretreat the test piece and prepare for the test according to 8.2 and 8.3.

8.6.2 If the pretest (8.4) was done, allow the test gas to flow into the photoreactor until the time of the adsorption (dark condition) which had been determined beforehand. If the pretest was not done, do as follows. Supply the test gas to the photoreactor for 30 min under the dark condition, and measure the concentration of acetaldehyde. Start photoirradiation when the concentration of acetaldehyde becomes the same as the supply gas concentration. If the concentration of acetaldehyde is less than 90 % of the supply gas concentration after 30 min, continue until it exceeds this percentage. If the concentration does not exceed 90 % after 90 min, stop measurement and report that this test method is not applicable to this test piece.

8.6.3 Maintain the gas flow and commence irradiation of the test piece, and record the concentration of acetaldehyde under photoirradiation for 3 h. When photocatalytic decomposition of acetaldehyde occurs, the concentration lowers as shown in Figure 4 and becomes stable in time. The concentration of acetaldehyde is preferably measured continuously. However, if the number of times of measurement is limited due to the analytical method (i.e. DNPH/HPLC), more than 1 measurement shall be done at an interval of 1 h or less. In addition, more than 3 measurements shall be done for the last 1 h (120 min to 180 min after photoirradiation). The concentration of acetaldehyde to be used for calculation of the removal rate is the mean value of the concentration measured during the last 1 h. If the relative standard deviation of the last 3 measurements of ϕ_A exceeds 20 %, report this instability with possible reasons such as deactivation of the test piece.

8.6.4 Stop photoirradiation and confirm that the concentration of acetaldehyde returns to the supply gas concentration (5,0 $\mu\text{l/l}$).

8.6.5 Stop the gas supply to the reactor and take the test piece out of the reactor.

9 Calculation

9.1 Calculation method

The test results shall be calculated as follows. The calculated values are usually rounded to one decimal place in accordance with ISO 80000-1. The observed concentrations of acetaldehyde and CO_2 before water-vapour correction shall be used for calculation. The flow rate of test gas f is 1,0 l/min, normalized for 0 °C, 101,3 kPa and dry-gas basis, and is then multiplied by a factor of 1,016 for water-vapour correction.

9.2 Removal percentage and removed quantity of acetaldehyde

Removal percentage of acetaldehyde is calculated using Equation (1). When R is either below 5 % or more than 95 %, the removal percentage shall be expressed as “below 5 %” or “more than 95 %”, respectively, for uncertainty reasons. Then the removed quantity is calculated by Equation (2). Similarly to the case of R , when n_A is either below 5 % or more than 95 %, the removed quantity shall be expressed as “below 5 %” or “more than 95 %”, respectively.

$$R_A = \frac{\phi_{A0} - \phi_A}{\phi_{A0}} \times 100 \quad (1)$$

$$n_A = R_A \times \frac{\phi_{A0} \times f \times 1,016 \times 60}{100 \times 22,4} \quad (2)$$

where

R_A is the removal percentage, by test piece, of acetaldehyde;

ϕ_{A0} is the supply volume fraction of acetaldehyde, in microlitres per litre ($\mu\text{l/l}$);

ϕ_A is the volume fraction of acetaldehyde at the reactor exit, in microlitres per litre ($\mu\text{l/l}$);

n_A is the quantity of acetaldehyde removed, in micromoles (μmol), by test piece, during the last 1 h of irradiation;

f is the flow rate of test gas converted into that at the standard state, in litres per minute (l/min) at 0 °C, 101,3 kPa and dry-gas basis.

9.3 Conversion to CO₂

The volume of CO₂ formed by UV irradiation is calculated by Equation (3), and the conversion to CO₂ (R_{CO_2} , %) is calculated by Equation (3), and then the CO₂ conversion R_{CO_2} shall be calculated by Equation (4). When R_{CO_2} is either less than 5 % or more than 95 %, it shall be expressed as “below 5 %” or “more than 95 %”, respectively. The quantity of CO₂ converted (n_{CO_2}) is calculated by Equation (5). When R_{CO_2} is either less than 5 % or more than 95 %, n_{CO_2} is calculated by assigning 5 or 95 for R_{CO_2} , respectively.

$$\phi_{CO_2} = \phi_{CO_2,L} - \phi_{CO_2,D} \quad (3)$$

$$R_{CO_2} = \frac{\phi_{CO_2} \times 100}{2 \times \phi_{A0}} \quad (4)$$

$$n_{CO_2} = R_{CO_2} \times \frac{2 \times \phi_{A0} \times f \times 1,016 \times 60}{100 \times 22,4} \quad (5)$$

where

ϕ_{CO_2} is the CO₂ volume fraction generated by UV irradiation, in microlitres per litre (µl/l);

$\phi_{CO_2,L}$ is the CO₂ volume fraction at the reactor exit under UV irradiation, in microlitres per litre (µl/l);

$\phi_{CO_2,D}$ is the CO₂ volume fraction at the reactor exit under dark conditions, in microlitres per litre (µl/l);

R_{CO_2} is the conversion from acetaldehyde to CO₂, in percent;

n_{CO_2} is the quantity of CO₂ converted from acetaldehyde during the last 1 h, in micromoles (µmol).

The CO₂ volume fraction under the dark condition shall be the mean value before and after the UV irradiation period as calculated by Equation (6). It shall not change by 1,0 µl/l or more before and after UV irradiation.

$$\phi_{CO_2,D} = \frac{\phi_{CO_2,Dpre} + \phi_{CO_2,Dpost}}{2} \quad (6)$$

where

$\phi_{CO_2,Dpre}$ is the CO₂ volume fraction in the dark before UV irradiation, in microlitres per litre (µl/l);

$\phi_{CO_2,Dpost}$ is the CO₂ volume fraction in the dark after UV irradiation, in microlitres per litre (µl/l).

10 Test method for test pieces with lower performance

In the case 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 acetaldehyde and conversion to CO₂ to appear in the test report shall be half of the values calculated from Equations (2) and (5), as well as using the flow rate of 0,5 l/min. When the test conditions are altered, it is required to confirm the time of adsorption (dark condition) at the altered test conditions.

Table 1 — Alternative test conditions

Alterable test conditions	Value after change
Flow rate of test gas	0,5 l/min
Number of test pieces	2 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 part of ISO 22197, i.e. determined in accordance with ISO 22197-2:2011.
- d) Date of test, atmospheric temperature, relative humidity, etc.
- e) A description of the test piece (material, size, shape, etc.).
- f) A description of test equipment (specifications, etc.).
- g) Testing conditions (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, etc.).
- h) The amount of acetaldehyde removed and CO₂ formed during the last 1 h, removal percentage of acetaldehyde (optional) and conversion to CO₂ (optional). If the test or CO₂ measurement is not valid, the reasons for that (e.g. strong adsorption of acetaldehyde).
- i) Any other matters of special importance, such as a change in the test piece noticed during the test.

Annex A (informative)

Results of round-robin test

A round-robin test was undertaken between seven collaborating laboratories. Each laboratory (lab) prepared the test equipment, including the photoreactor, separately. The organizing laboratory visited each party during the test with a standard gas cylinder (about 5 µl/l acetaldehyde in nitrogen), calibrated gas flowmeter and UV-A radiometer to check and calibrate its equipment. Each laboratory made four tests using the sheet- and filter-type photocatalyst supplied. The original data are shown in Table A.1.

Table A.1 — Results of round-robin test

Unit: micromoles (µmol)

Test piece	Amount	Run	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G
Sheet-type	CH ₃ CHO removed	1	9,93	11,20	9,39	10,24	9,40	8,35	7,30
		2	10,23	11,31	9,07	10,14	8,92	9,10	7,19
		3	9,99	11,33	9,20	10,33	8,46	8,51	—
		4	9,99	11,06	9,13	10,28	8,67	8,75	—
	CO ₂ formed	1	13,79	13,82	—	15,61	—	—	13,86
		2	14,83	20,79	—	14,11	—	—	14,41
		3	14,88	17,50	—	15,69	—	—	13,47
		4	15,25	21,03	—	14,31	—	—	14,57
Filter-type	CH ₃ CHO removed	1	13,08	11,94	12,59	13,02	12,94	—	12,72
		2	13,02	11,75	12,66	12,89	12,52	—	12,24
		3	13,22	11,64	12,57	12,93	12,68	—	—
		4	13,18	11,65	12,62	12,98	12,76	—	—
	CO ₂ formed	1	22,78	12,86	—	18,41	—	—	18,88
		2	21,12	14,24	—	17,92	—	—	18,66
		3	20,54	19,29	—	14,31	—	—	—
		4	20,34	24,24	—	14,34	—	—	—
Analytical methods			GC/FID, NDIR	GC/FID, NDIR	DNPH/ HPLC	GC/FID, methanizer- GC/FID	GC/FID	DNPH/ HPLC	DNPH/ HPLC, NDIR

The overall mean, repeatability standard deviation and reproducibility standard deviation computed in accordance with ISO 5725-2 are summarized in Table A.2.

Table A.2 — Statistical data obtained in accordance with ISO 5725-2

Unit: micromoles (µmol)

Test piece	Overall mean		Repeatability standard deviation		Reproducibility standard deviation	
	CH ₃ CHO	CO ₂	CH ₃ CHO	CO ₂	CH ₃ CHO	CO ₂
Sheet-type	9,50	14,72	0,23 (2,4 %)	0,68 (4,6 %)	1,20 (13,0 %)	0,82 (5,6 %)
Filter-type	12,63	18,97	0,11 (0,9 %)	1,65 (8,7 %)	0,54 (4,3 %)	3,13 (16,0 %)

Relative standard deviations are shown in parentheses.

In further tests, the results obtained with different photocatalyst samples, such as sheet glass, paper, fabrics, paint, etc., varied from < 2,0 µmol to 29,3 µmol, which indicated that this method can differentiate the samples with different photocatalytic performance.

Bibliography

- [1] ISO 4892-1, *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance*
- [2] ISO 4892-3:2006, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*
- [3] FUJISHIMA, A., HASHIMOTO, K. and WATANABE, T. *TiO₂ Photocatalysis. Fundamentals and Applications*, BKC Inc., Tokyo (1999)
- [4] OHKO, Y., TRYK, D.A., HASHIMOTO K. and FUJISHIMA, A. Autoxidation of acetaldehyde initiated by TiO₂ photocatalysis under weak UV illumination, *J. Phys. Chem. B*, **102**, pp. 2699-2704 (1998)

ICS 81.060.30

Price based on 14 pages

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services

Tel: +44 845 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 845 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com