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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

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22197-2 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 (µl/l)
ϕ_{A0}	the volume fraction of acetaldehyde in the test gas (µl/l)
ϕ_{CO_2}	the carbon dioxide (CO_2) volume fraction generated by UV irradiation (µl/l)
$\phi_{CO_2,L}$	the CO_2 volume fraction at the reactor exit under UV irradiation (µl/l)
$\phi_{CO_2,D}$	the CO_2 volume fraction at the reactor exit under dark conditions (µl/l)
$\phi_{CO_2,Dpost}$	the CO_2 volume fraction in the dark after UV irradiation (µl/l)
$\phi_{CO_2,Dpre}$	the CO_2 volume fraction in the dark before UV irradiation (µl/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

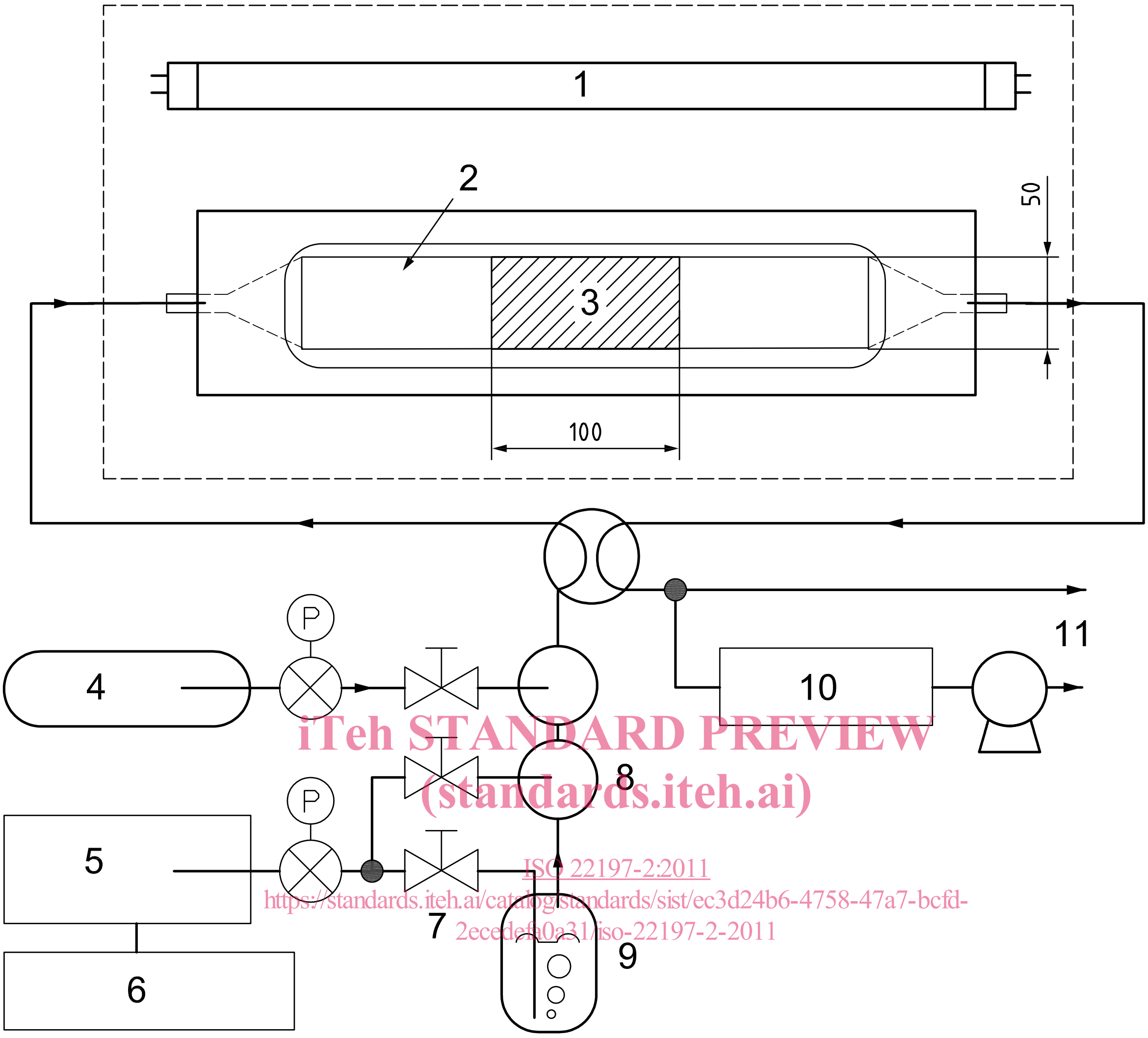
This part of ISO 22197 concerns the development, comparison, quality assurance, characterization, reliability, and design data generation of photocatalytic materials (Reference [3] in the Bibliography). The method described is intended to obtain the air-purification performance of photocatalytic materials 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
2 optical window
3 test piece
4 standard gas (pollutant)
5 air-purification system
6 compressor
7 mass-flow controller
8 gas mixers
9 humidifier
10 analyser
11 vent

Figure 1 — Schematic diagram of test equipment

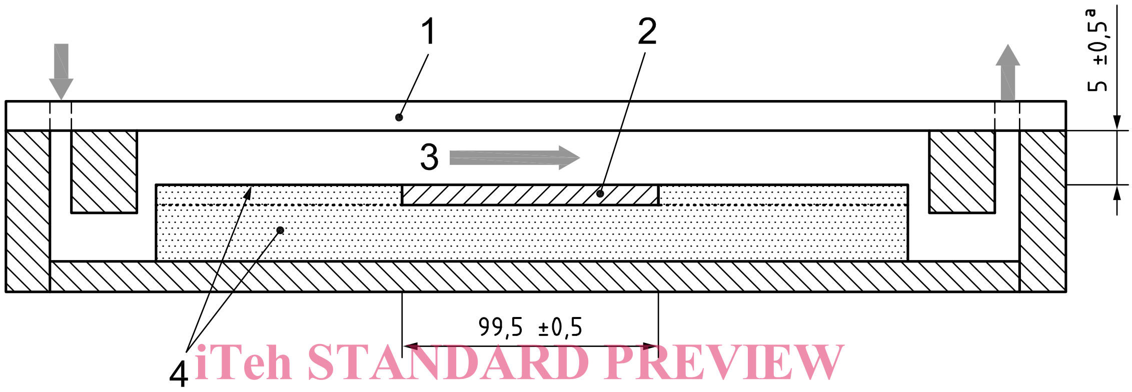
6.2 Test gas supply

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 ($N_2 + O_2$, such as that supplied in cylinders) shall be used for dilution when the CO_2 from acetaldehyde is also measured.

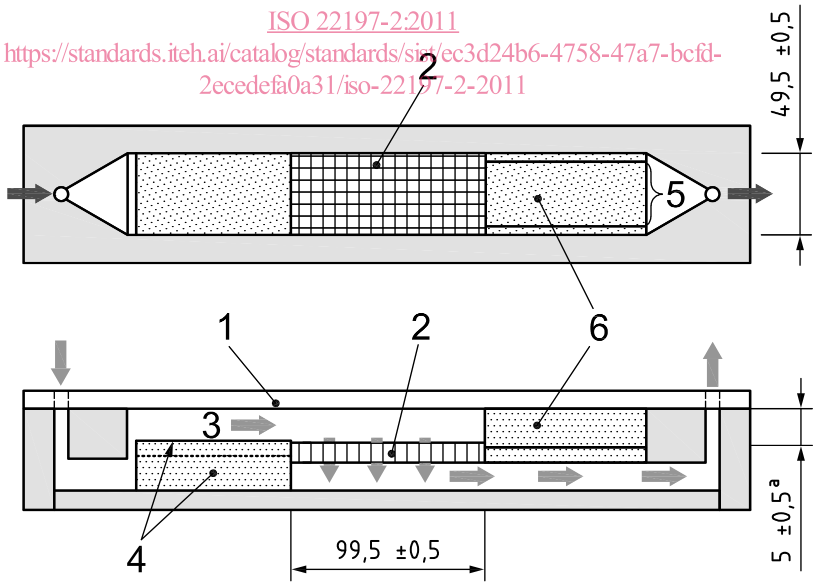
6.3 Photoreactor

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 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.

Dimensions in millimetres



a) For flat test pieces



b) For filter-type test pieces

Key

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

a Air-layer thickness.

Figure 2 — Cross-sectional view of photoreactor

6.4 Light source

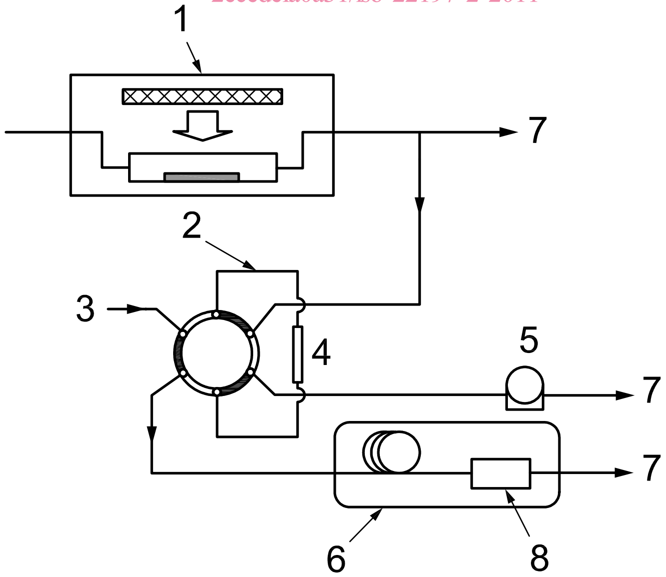
The light source shall provide UV-A illumination within a wavelength range of 300 nm to 400 nm. Suitable sources include the so-called black light (BL) and black light blue (BLB) fluorescent lamps, with a maximum at 351 nm or 368 nm, as specified in ISO 10677, and xenon arc lamps with optical filters 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 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 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
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

6.6 Analytical system for CO_2

The concentration of CO_2 shall be determined using a non-dispersive infrared CO_2 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.

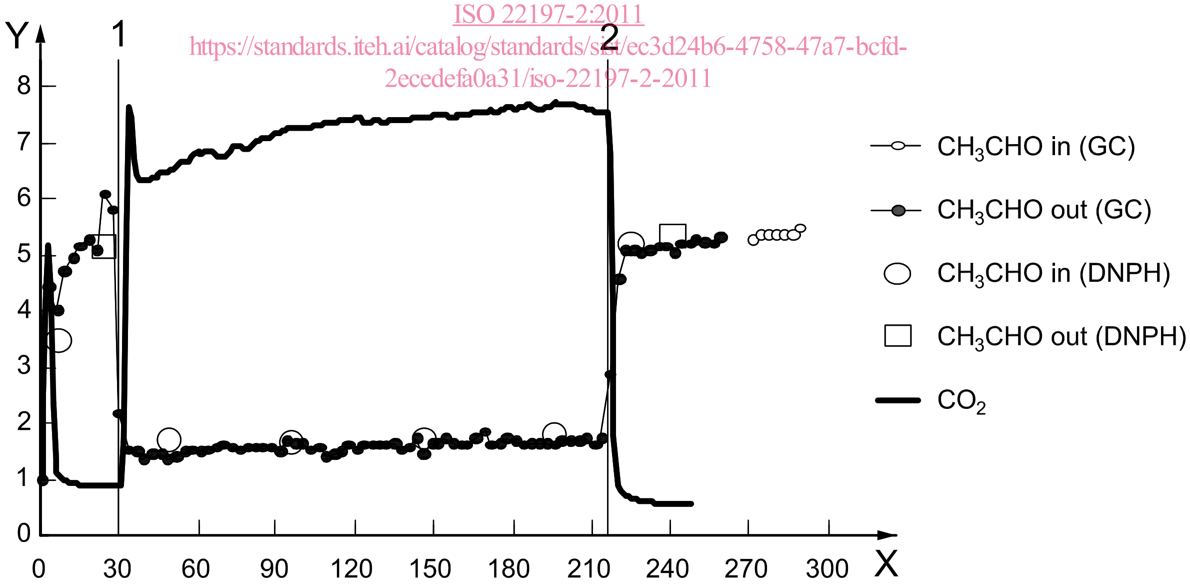
7 Test piece

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 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 of acetaldehyde and formation of CO_2 under photoirradiation. An example of the concentration change of acetaldehyde and CO_2 during the test is shown in Figure 4. The measurement of CO_2 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 CH_3CHO, CO_2 (µl/l)

1 irradiation start

2 irradiation stop

Figure 4 — Typical trace of acetaldehyde and CO_2 concentration during the test operation