
Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determination of phenol oxidative decomposition performance of semiconducting photocatalytic materials by quantitative analysis of total organic carbon (TOC)

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Published in Switzerland

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Since photocatalysts have a wide range of functions such as purification of air or water, antibacterial effect and self-cleaning, many application products that make use of said functions are developed. Photocatalyst-applied products for water purification are expected to exert oxidative decomposing performance against organic compounds composed of hydrocarbon, which is a water polluting substance. In line with this, there is a necessity for developing a testing method for measuring the oxidative decomposition against water polluting substances. This document stipulates a test method for measuring the performance of a semiconducting photocatalytic material irradiated with UV light in water for the oxidative decomposition of hydrocarbon such as phenol and the byproducts as intermediate produced by the photocatalysis, by the TOC method.

An examples of data evaluation is given in [Annex D](#) while the results of the interlaboratory tests are given in [Annex E](#).

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1 Scope

This document provides a testing method for testing phenol oxidative decomposition performance of semiconducting photocatalytic materials or made of a material adsorbed with photocatalyst to its surface for the purpose of purifying water polluting substances in water making use of photocatalytic performance as test piece. The test piece can be planar, spherical, flake or block shape. A test piece that can elute hydrocarbon or the like by immersion into water or phenol aqueous solution or by light irradiation, a semiconducting photocatalytic material that cannot maintain its shape or a powdery semiconducting photocatalytic material are excluded from the scope of application, since they cannot be evaluated.

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 8425, *Water quality—Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*

ISO 10523, *Water quality — Determination of pH*

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 19722, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for determination of photocatalytic activity on semiconducting photocatalytic materials by dissolved oxygen consumption*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

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*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 19722 and ISO 20507 and the following 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 test piece
planar, spherical, flake or block shaped semiconducting photocatalyst materials for water purification prescribed in ISO 20507 using semiconducting photocatalyst

3.2 illuminated condition
test condition under which a test is conducted with light source lit

3.3 dark condition
measuring condition under which no light source is lit and no light is incoming from outside

Note 1 to entry: The purpose of this condition is for comparing the result with that conducted under the illuminated condition.

4 Symbols

Symbol	Designation	Unit
C	concentration of hydrocarbon	mg/l
ΔTOC_D	phenol concentration adsorbed to the test piece in the dark	mg/l
C_{Di}	phenol concentration measured from the test solution as the initial concentration in the dark condition	mg/l
C_{Df}	phenol concentration measured from the test solution after 6 hours in the dark condition	mg/l
ΔTOC_L	phenol concentration adsorbed to the test piece and decomposed by photocatalysis	mg/l
C_{Li}	phenol concentration measured from the test solution as the initial concentration under the illuminated condition	mg/l
C_{Lf}	concentration of residual phenol and byproduct measured from the test solution after 6 hours under an illuminated condition	mg/l
ΔTOC	decomposed phenol concentration as hydrocarbon on the test piece by the photocatalysis	mg/l
V	volume of test solution	ml
λ	wavelength	nm
I	ultraviolet (UV) light irradiation intensity	mW/cm ²

5 Principle

Organic compound in waste water is oxidatively decomposed to CO₂ and water by the photocatalysis, so called as photocatalytic mineralization. Under the photocatalytic mineralization, many kinds of byproducts are usually produced and the production mechanism including the changes of a molecular structure and the concentration of byproducts are depending on the characteristics of photocatalyst, such as the surface structure, crystal phase and the photocatalytic activity. Accordingly, estimation of photocatalytic activity by the decrement of initial concentration of organic compounds means mainly changing the original structure of the organic compound. Preferable estimation method of the

photocatalytic activity for waste water treatment should oxidatively decompose organic compound to CO₂ and water as photocatalytic mineralization. On the basis of the conception, total organic carbon analysis as measurement of the photocatalytic mineralization is most suitable method because no the byproducts production as reaction mechanism should be taken into account.

6 Materials

6.1 Chemicals and implements

6.1.1 Reagent

Reagent is phenol and the assay is > 99 wt%.

6.1.2 Purified water

Water used for the preparation of all solutions shall be distilled or deionised water.

6.1.3 Purified air

Air prepared by removing dusts and volatile organic compounds from atmospheric air using a filter or the like, or synthetic air filled in a high-pressure container as provided in ISO 22197-1.

6.1.4 Purified water saturated with dissolved oxygen

Purified water prepared in accordance with the preparation procedure provided for in ISO 19722 by saturating water with dissolved oxygen at ± 1 °C of water temperature during the test.

7 Test device and instrument

7.1 Test vessel

Use a silica glass for the window glass plate for this test. The test vessel and O-ring used in this test shall be near-UV irradiation resistant and chemical resistant against phenol. For example, preferred material for an O-ring is fluorocarbon polymer, and preferred material for other parts is polytetrafluoroethylene.

The test vessel shall be inclined to avoid fogging the inner side of window glass by test solution because the fogged window glass induces extinction. Therefore, the test vessel shall be structured to maintain the test piece immersed under the test solution even when the test vessel is inclined as shown in [Annex A](#). Moreover, a weir shall be provided so as to avoid the test piece from falling off or swept away by the circulation of the test solution. The height of the weir shall have a clearance of 5 mm + 0 mm, -1 mm from the window plate.

The size of window glass shall be larger than the size of the place for folding the test piece

7.2 Reserve container

Provide a reserve container for maintaining the condition of the test solution to be supplied to the test vessel and for taking water for TOC measuring (see [Annex B](#)). Preferably, the volume capacity of the reserve container is 500 ml \pm 10 ml and it is made of borosilicate glass having chemical resistance against phenol. Since the test solution is stirring in the reserve container during the test, the reserve container shall have sufficient volume and structure that allows stirring. To maintain the temperature of the test solution within the predetermined range during the test, a water jacket shall be provided for fixing the reserve container that can circulate the thermostatic water. Provide a reserve container with a lid provided with an O-ring for keeping airtightness. The O-ring is preferably made of fluorocarbon polymer. This lid shall be provided with a through-hole for piping, a through-hole for air inflow, an air exhaust and a through-hole for fixing a thermometer. Diameter of the through-hole for air exhaust shall

be determined after conducting a test without using a test piece under a dark condition and under an environmental condition for conducting a test, and if the resulted variation of phenol concentration by TOC measuring is 1,5 % or less, such diameter is allowed. For example, around 3 mm + 0 mm, -1 mm of diameter of the through-hole for air exhaust is allowed.

For reference, specification of the reserve container, lid and water jacket are not limited to the above-mentioned examples, in other words, if they are of material and structure that satisfy the conditions specified here, they are allowed.

7.3 UV irradiation light source

Use the light source that is provided in ISO 10677. It is recommended to use UV fluorescent lamp having 351 nm of the maximum optical intensity that can irradiate UV ray.

7.4 UV radiometer

A radiometer with a detector whose sensitivity peak is at $\lambda = 351$ nm shall be used to measure the UV-light intensity. The radiometer shall be calibrated to closely match the characteristic of the UV light irradiation light source as specified in ISO 10677 or be corrected to ascertain sensitivity within the wavelength range to be adsorbed by the test piece.

7.5 UV light intensity

Adjust I to $2 \text{ mW/cm}^2 \pm 0,1 \text{ mW/cm}^2$ through the window glass when the light receiving portion of an UV illuminometer is positioned at the centre of the test piece.

7.6 pH meter

Measure pH of the test solution using the pH meter specified in ISO 10523.

7.7 TOC measuring device

Measure the total organic carbon in accordance with TOC analysis by combustion oxidizing and infrared system [Non-Purgeable Organic Carbon (NPOC) analysis] as provided in ISO 8425, using TOC automatic measuring instrument. For reference, pH resulted after oxidizing treatment as provided in ISO 8425 shall not necessarily be 2, rather, 3 or 4 is acceptable if non-organic carbon can be removed.

8 Arrangement of test method

8.1 Measuring device setup

The test device comprises a test vessel, a light source, a reserve container, a liquid feeding pump and a thermostatic bath/cryostat as shown in [Annex C](#). It is for evaluating the phenol oxidative decomposition performance using a semiconducting photocatalyst material by TOC analysis. Decrease of phenol contained in the test solution is measured by a test piece, irradiating light necessary for expression of semiconducting photocatalyst performance to the test piece and continuously supplying and circulating test solution. In this test, a lid for fixing the window glass plate for avoiding the test solution from leaking is provided, since the test vessel shall be inclined. The lid is also for limiting evaporation of the test solution. This is a semi-closed system where an air exhaust is provided which is necessary for air inflow. An example of the measuring device setup is shown in [Annex C](#).

8.2 Test solution feeding device

The test device as shown in [Annex C](#) has a liquid feeding pump and piping for controlling the flowing amount of the test solution circulated continuously at a predetermined speed from the reserve container

to the test vessel. Material for piping shall be chemical resistant against phenol and UV resistant. For example, a thermoplastic elastomer is recommended.

A liquid feeding pump shall be able to feed and circulate predetermined amount of the test solution at a constant speed for a long time and shall be chemical resistant against phenol. Furthermore, no material that may be eroded by phenol or that may be adsorbed with phenol shall be used in any part of the liquid feeding pump structure. For example, a peristaltic type pump is recommended, which compresses the liquid feeding tube formed of phenol resistant and UV resistant material by a roller to feed liquid.

8.3 Instruments for air flow

For air inflow of dissolved oxygen supplying air, hollow tubular instrument made of material that does not adsorb phenol nor elute impurities shall be used. For example, a disposable pipet made of borosilicate glass that is replaced for every test (volume of $3 \text{ ml} \pm 1 \text{ ml}$), or cylindrical gas injection pipet with diameter $10 \text{ mm} \pm 1 \text{ mm}$ and height $18 \text{ mm} \pm 1 \text{ mm}$ are good. Air inflow amount may be an inflow amount that can maintain the concentration of dissolved oxygen under the water temperature during the test. It is better if air inflow amount is controllable within the range from 400 ml/min to 600 ml/min.

9 Test material

9.1 Test piece

Sample size of the test piece shall be as follows:

Width — $49 \text{ mm} + 0 \text{ mm}, -1 \text{ mm}$

Length — $199 \text{ mm} + 0 \text{ mm}, -1 \text{ mm}$

The size of a test piece shall be able to be put at the place for folding the test piece as shown in [Annex A](#). The thickness of the test piece is not especially specified, it is acceptable if $5 \text{ mm} + 0 \text{ mm}, -1 \text{ mm}$ of clearance is secured between the uppermost surface of the test piece and the inner surface of the window glass plate. If a test piece of different thickness is used, the clearance should be adjusted using an accessory plate. Material for an accessory plate is preferably PTFE that has UV irradiation resistance and chemical resistance against phenol.

If the test piece is flake shape or the like, set it at the place for holding the test piece in the test vessel, and then flatten it and secure $5 \text{ mm} + 0 \text{ mm}, -1 \text{ mm}$ of clearance between the uppermost surface of the test piece and the inner surface of the window glass plate. If the test piece falls off by inclination of the test vessel or be swept away by the circulation of water, use an offset preventing structure at the place for holding the test piece.

9.2 Pre-treatment of the test piece

If any of the followings possibly occur, do proper pre-treatment. After any pre-treatment, the test piece shall be dried well for the test. At this time, adsorption of organic substances or other effect shall be avoided to the extent possible.

- a) If the resulted pH varies beyond the range from 5 to 8 during the test under a dark condition, wash the test piece with purified water. Repeat washing with purified water until pH stays between the range from 5 to 8, measuring pH after each washing.
- b) If elution of any organic substance from the test piece is suspected in a test under a dark condition, wash the test piece with purified water. In addition, irradiate light by an UV fluorescent lamp for more than 5 h in the atmosphere or in water as provided for in ISO 22197-1. Elution of organic substances from the test piece is allowed if C_{Df} value results in 95 % or more of C_{Di} value after conducting the procedure of the measurement [10.4 a\) to d\) and f\)](#).

Pre-treatment may be omitted for a test piece if it has been treated or synthesized with high temperature at 400 °C or more and no elution of organic substance into water is obvious that no residual inorganic salt after sufficient washing.

10 Procedure of the measurement

10.1 Test temperature

All tests shall be carried out at a room temperature of 23 °C ± 5 °C and water temperature shall be at 23 °C ± 2 °C. Water temperature should be controlled to a certain temperature by thermostatic bath/cryostat.

10.2 Other test condition

Secure a clearance of 5 mm + 0 mm, -1 mm between the light receiving portion and the window glass. Measure the UV illuminance after the UV fluorescence lamp lit for 10 min or more. The inclination angle of the test vessel shall be from 5° to 10°. Position it in a manner that the centre of the UV fluorescent lamp and the centre of the position for holding the test piece, and they shall be in parallel.

Air inflow amount shall be around 500 ml/min.

Inflow amount of the test solution into the test vessel shall be around 200 ml/min ± 2 ml/min.

10.3 Preparation for test

Use a vial made of borosilicate glass for taking the test solution. All glass instrument used under this document, including vials, shall be washed using neutral or alkaline detergent in order to prevent entrainment of impurities into the test solution and further shall be washed sufficiently with purified water.

10.4 Procedure of the measurement

Conduct two tests, one under a dark condition and the other under an illuminated condition. Under the dark condition, measure the adsorption of phenol to the test piece, and under the illuminated condition, measure the phenol oxidative decomposition of the test piece by photocatalytic action. The test procedures a) to c) shall be conducted first. Then proceed to d) and f) for dark condition, and e) and f) for illuminated condition.

If a TOC measuring instrument is directly connected to the reserve container, set the initial concentration to a value obtained by measuring TOC after the procedure c), and then measure TOC every 2 h under the procedures d) and e).

- a) Prepare 1,000 ml ± 5 ml of test solution with 5 mg/l ± 0, 5 mg/l of phenol concentration using dissolved oxygen saturated water. Use 500 ml ± 2 ml of the prepared test solution for the test under dark condition, and use the remaining test solution for the test under illuminated condition. Then, take 20 ml ± 1 ml of the test solution in a vial for measuring the initial concentration under dark condition. Further, take the same amount of water in the same manner for measuring under an illuminated condition. Measure pH of the prepared test solution. The test solution used in this test shall be tightly sealed and stored at the same temperature with that at the testing or stored in a cold and dark place.
- b) Set the test piece to the test vessel in a dark place.
- c) Put the stirrer bar and the test solution into the reserve container and close the lid to set it to the water jacket. Connect the piping between the reserve container and the test vessel and set the thermometer and other necessary instruments. Start stirring and air flow. Stirring and air flow shall be done continuously during the test. Circulate the temperature regulated water in the water jacket.

- d) Start measuring adsorption in a dark place simultaneously with the start of circulation of the test solution. Circulate the test solution for 6 h. Take 20 ml ± 1 ml of the test solution from the reserve container every 2 h for measuring C by TOC.
- e) Start light irradiation within 1 min from the start of circulation of test solution. Irradiate light for 6 h, and take 20 ml ± 1 ml of the test solution from the reserve container every 2 h for measuring C by TOC.
- f) Measure pH of the test solution in the reserve container.

11 Evaluation of results

Reports all values rounded to second decimal places according to ISO 80000-1.

11.1 Evaluation of dark condition

Calculate ΔTOC_D (mg/l) according to [Formula \(1\)](#), from the variation of TOC measured from the test solution for measuring the initial concentration and the test solution after 6 h of the test, both taken under dark condition.

$$\Delta TOC_D = C_{Di} - C_{Df} \quad (1)$$

If C_{Df} became 0 within the test duration, express the time when it became 0 and ΔTOC_D . If ΔTOC_D became a minus value and such value exceeds 5,0 % of C_{Di} , conduct the procedure b) of [9.2](#) and then conduct the test once again.

11.2 Evaluation of illuminated condition

Calculate ΔTOC_L (mg/l) according to [Formula \(2\)](#), from the variation of TOC measured from the test solution for measuring the initial concentration and the test solution after 6 h of the test, taken under illuminated condition.

$$\Delta TOC_L = C_{Li} - C_{Lf} \quad (2)$$

If C_{Lf} became 0 within the test duration, express the time when it became 0 and ΔTOC_D . If ΔTOC_D became a minus value under illuminated condition even though the ΔTOC_D value is proper under dark condition, it is highly possible that the test piece itself has been decomposed by the effect of photocatalyst during light irradiation. In such a case, the test ends up in failure because correct value cannot be obtained.

11.3 Phenol oxidative decomposition performance

Calculate ΔTOC as the phenol oxidative decomposition performance by the following [Formula \(3\)](#), using ΔTOC_L and ΔTOC_D .

$$\Delta TOC = \Delta TOC_L - \Delta TOC_D \quad (3)$$

11.4 Condition for establishing a successful test

A test is deemed established if the test satisfies the relationship of the following [Formula \(4\)](#).

$$\Delta TOC_L > \Delta TOC_D \quad (4)$$

If C_{Lf} and C_{Df} became 0 within the test duration, the test is deemed established if C_{Lf} became 0 earlier than C_{Df} .

12 Test report

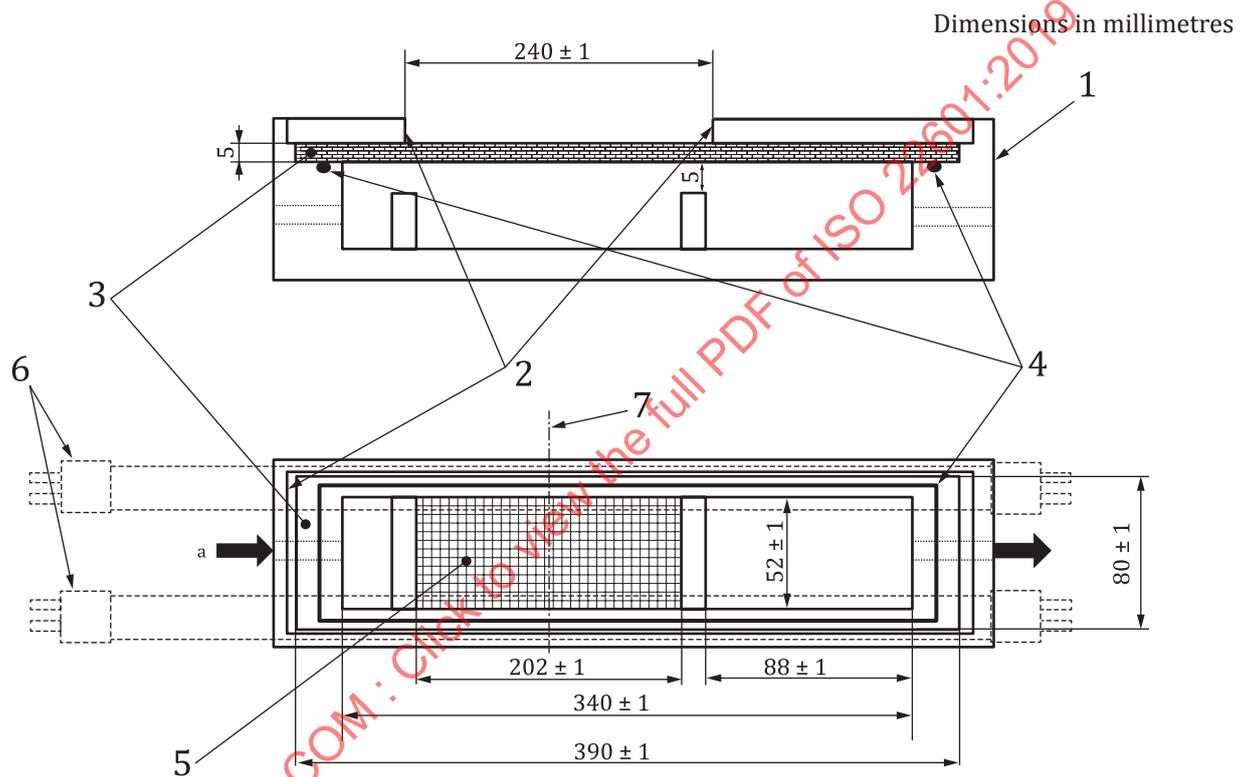
The test report shall be in accordance with the reporting provisions of ISO/IEC 17025, and shall include the following information:

- a) a reference to this document, i.e. ISO 22601:2019;
- b) date on which the test was conducted;
- c) name of measurer and testing laboratory;
- d) name of the manufacturer, degree of purity, class of the reagent;
- e) light source (name of the manufacturer, model No. and number of the lamps);
- f) name of the manufacturer and model No. of the irradiation illuminometer;
- g) model number and the name of manufacturer of TOC measuring instrument;
- h) name of the test piece and its LOT No.;
- i) shape of the test piece (planar, flake, spherical, etc.);
- j) size of the test piece (dimensions, number of pieces, diameter, mass, etc.);
- k) method for setting and attaching the test piece to the test vessel, set status, etc.);
- l) presence/absence of pre-treatment, and if pre-treatment was done, the cause that required the pre-treatment (pH or/and TOC);
- m) values of TOC or pH, both before and after the pre-treatment (in case where pre-treatment was done);
- n) inclination angle of the test vessel;
- o) room temperature;
- p) temperature of the test solution (both at the start of the test and at the end of the test);
- q) flowing amount of the test solution;
- r) pH of the test solution before and after the test;
- s) air inflow speed and instruments used in the test (name of the instrument, shape and name of the manufacturer);
- t) C_{Di} , C_{Df} , ΔTOC_D (mg/l), C_{Li} , C_{Lf} , ΔTOC_L (mg/l), ΔTOC (mg/l);
- u) remarks (condition, time, etc. if the phenol concentration became 0 within the test duration, both under the dark condition and the illuminated condition);
- v) details of any operation not specified in this document or in the International Standards to which reference is made, and any operations regarded as optional, as well as any incidents likely to have affected the results.

Annex A
(informative)

Example of suitable test vessel

A.1 Schematic diagram of test vessel



Key

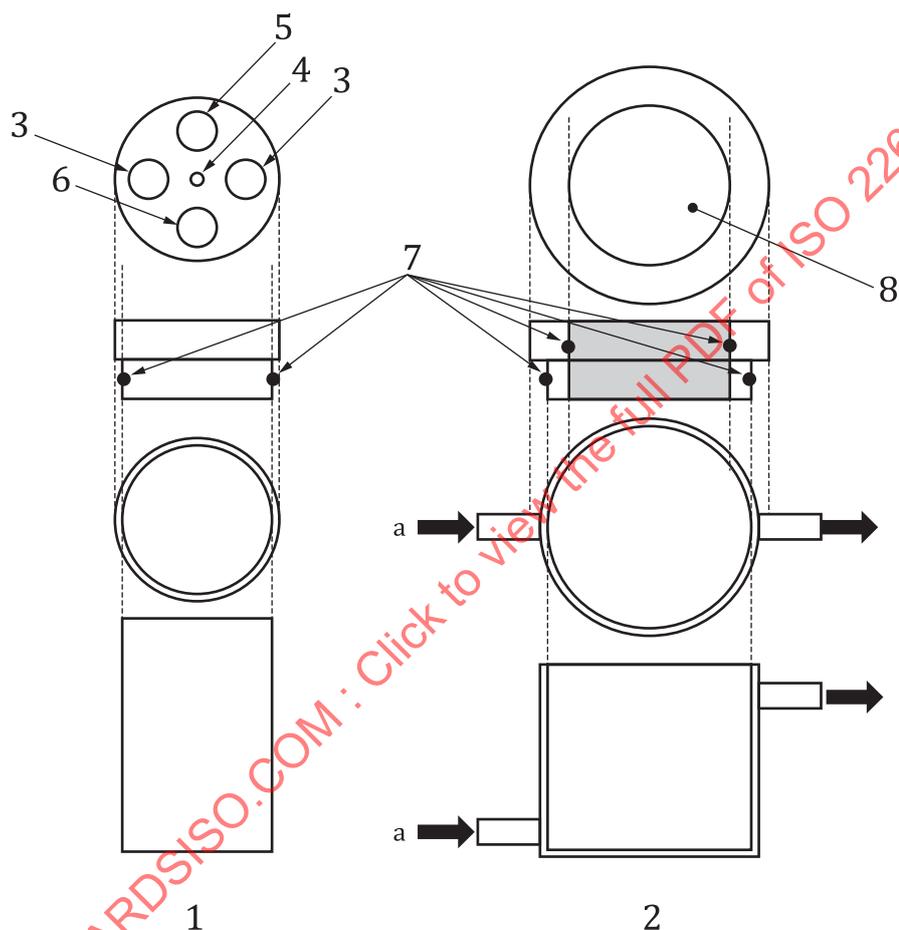
- | | |
|---|---|
| 1 test vessel | 5 test piece |
| 2 lid for fixing the window glass plate | 6 light source |
| 3 window glass plate | 7 centre of the light source and test piece |
| 4 O-ring | a Test solution flow. |

Figure A.1 — Schematic diagram of structure of a test vessel

Annex B (informative)

Example of suitable test vessel

B.1 Schematic diagram of reserve container and water jacket



Key

- | | |
|--|--|
| <p>1 reserve container/lid and main body</p> <p>2 water jacket/lid and main body</p> <p>3 through-hole for piping</p> <p>4 air exhaust hole</p> <p>5 thermometer/through-hole for test solution intake</p> | <p>6 through-hole for air inflow</p> <p>7 O-ring</p> <p>8 through-hole for fixing the reserve container</p> <p>a Temperature regulated water flow.</p> |
|--|--|

NOTE The reserve container and the water jacket are made of borosilicate glass.

Figure B.1 — Schematic diagram of a structure of reserve container and water jacket

Annex C
(informative)

Example of test position of test vessel and suitable measuring device component

C.1 Schematic diagram of framework for test device

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