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

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National foreword

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

Céramiques techniques — Méthode d'essai relative à la détermination de l'activité photocatalytique sur matériaux photocatalytiques semiconducteurs par la consommation d'oxygène dissous

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
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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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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Introduction

International Standards covering test methods for determination of photocatalytic activity have been published. A wide variety of photocatalytic functions, such as water and air purification, antibacterial effect, and self-cleaning, require different evaluation methods. However, much easier methods to evaluate a common semiconducting photocatalytic activity are strongly demanded, in particular in research and development activities for testing of performance of semiconducting photocatalyst and photocatalytic materials under development.

INTERNATIONAL STANDARD ISO 19722:2017(E)

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

1 Scope

This document specifies the test method for determination of concentration of dissolved oxygen consumed due to photocatalytic oxidation of phenol in aqueous phase by semiconducting photocatalytic substances. The method is applicable to powder test sample or film test piece of semiconducting photocatalystic material targeting water contaminants. This test method is not applicable for evaluating the materials conjugated with other base material, such as organic binder which can also be decomposed by the photocatalytic activity.

This document is applicable to the test method for the activity of powder test sample or film test piece of semiconducting photocatalystic material targeting water contaminants.

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 5814, *Water quality — Determination of dissolved oxygen — Electrochemical probe method*

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*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
semiconducting photocatalyst
substance that displays photocatalytic action based on its electronic band structure

Note 1 to entry: This applies to metal oxides like titanium dioxide and sulfides. Photocatalysts which are not semiconducting includes metal complexes.

3.2
photocatalytic materials
material in which or on which the photocatalyst is added by coating, impregnation, mixing, etc.

Note 1 to entry: Materials include ceramic, metal, plastic, cloth, etc. for general purpose.

3.3
DO
dissolved molecular oxygen in aqueous phase

3.4
DO analyser
measuring instrument for continuous measurement of *DO* (3.3) in aqueous using *DO electrode* (3.5)

3.5
DO electrode
electrode to measure *DO* (3.3) in aqueous phase

3.6
photocatalytic oxygen demand
POD
quantity of molecular oxygen in aqueous phase consumed in photocatalysis

3.7
blank POD(%)
percentage of concentration of *DO* (3.3) consumed under a test condition without phenol addition

4 Symbols

Designation	Symbol	Unit
room temperature	R.T.	°C
water temperature	W.T.	°C
concentration	c	mol/l
concentration of DO	c_{DO}	mg/l
photocatalytic oxygen demand	POD	mg/l
c_{DO} before UV light irradiation in the dark	c_{iDO}	mg/l
c_{DO} after UV light irradiation in the dark	c_{fDO}	mg/l
volume of test solution	V	ml
wavelength	λ	nm
UV light irradiation intensity	I	mW/cm^2

5 Principle

Photocatalysis in pure water generally produces molecular oxygen(O_2) from water molecule (H_2O) oxidation[3]. In the water polluted by some of the organic compounds, major photocatalysis oxidizes the organic compounds[4]. Photocatalyst needs O_2 to oxidize organic compounds to CO_2 and water in the environment[4]. Then the quantity of O_2 that photocatalysis needs is much larger than O_2 production from H_2O oxidation because the organic compound is photocatalytically oxidized much easier than water molecule. O_2 then has major three functions in semiconducting photocatalyst. The first function is to improve charge separation by accepting conduction band electron. The second one is to produce active oxygen species that have the ability to oxidize organic compounds. The final one is oxidation. O_2 combines with organic radicals (intermediates) produced by semiconducting photocatalytic oxidation; O_2 is an indispensable species in the semiconducting photocatalysis[4]. Therefore, the photocatalysis to oxidize organic compound means O_2 consumption. In the photocatalysis to oxidize and mineralize the organic compounds, partially oxygenated by-products are produced. Under the progress in the continuous oxidation of partially oxygenated by-products, the photocatalysis consume O_2 to mineralisation. On the basis of the photocatalytic mineralization and functions of O_2, the semiconducting photocatalytic activity can be evaluated by determining O_2 consumption. This test method is especially effective in the photocatalysis in aqueous phase. Target photocatalytic materials are either powder test samples or a film test pieces.

6 Materials

6.1 Reagent

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

6.2 Purified water

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

6.3 Purified air

Air in the atmosphere aerated through 1 000 ml purified water.

6.4 Purified water saturated with dissolved oxygen

Purified water at R.T. ± 1 °C saturated with DO.

6.5 Test solution

Suspension with the powder test sample in phenol solution or phenol solution for the film test piece.

7 Test apparatus

7.1 General

Apparatus shall be used to evaluate the semiconducting photocatalytic materials with a suitable test method. The powder test sample is suspended and the film test piece is immersed in the test solution. The following apparatus is required.

7.2 Ultraviolet (UV) — Irradiation light source

Use black light fluorescence lamps as black light lamp (BL) and black light blue lamps (BLB). The black light fluorescence lamps shall have a peak wavelength λ = 351 nm as specified in ISO 10677.

7.3 UV radiometer

A radiometer with a detector whose sensitivity peak is at λ = 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 powder test sample or the film test piece with suitable approaches.

7.4 UV light intensity

I is adjusted to be 1,5 mW/cm^2 at the centre of a test vessel for the powder test sample or at the centre of the film test piece surface for the film test piece (see Annex C).

7.5 DO analyser

To measure c_{DO}, an electrode of DO analyser has an oxygen permeable membrane and the performance of electrode and equipment is specified in ISO 5814. Sensitivity correction and operation of the electrode and the DO analyser shall be performed following their manuals of suppliers and manufactures. Notice that portable equipment does not have stability for voltage, if indicated values are unstable, it is necessary to use stabilised power supplies.

7.6 Magnetic stirrer and magnetic stirring bar

A rotational number for stirring is 1 400 r/min to 2 100 r/min. The size of a magnetic stirring bar is approximately o.d. 7 mm × L 20 mm which is adjusted to be the size of the test vessel. Digital laser tachometers (non-contact system) are suitable for measurement of a rotational number. The rotational number is measured in the air.

8 Arrangement of test method

8.1 Measuring device setup

The apparatus shall be used to evaluate the activity of photocatalytic materials by measuring a decrease in c_{DO} in the test solution with photo irradiation necessary for the photocatalytic reaction after suspending the powder test sample or immersing the film test piece in the test solution, consisting of the test vessel, the light source, the DO analyser, a thermostatic bath, and a pump. This test is in a closed system to measure the decrease in c_{DO} in the test solution. An example of the measuring device setup is shown in Annex A.

8.2 Test vessel and implement

Schematic diagrams are shown in Annex B. The test vessel and glass implement shall be made of borosilicate glass which can resist near-UV light irradiation of $\lambda > 300$ nm and absorbs less UV. The test vessel for the test solution shall be cylindrical and the volume shall be 200 ml.

The test solution temperature has to be kept within a certain definite range during the test. A water jacket in which water can circulate at a constant temperature shall be used and a test vessel holder (upper and lower) to fix the test vessel inside the water jacket shown in Annex C shall be prepared. A holder is held has O-rings to keep airtightness of the closed system and with a through-hole as well to overflow extra suspension/test solution because the amount of suspension/test solution equal to the DO electrode volume is overflowed when the DO electrode is inserted into the holder. A plug shall be used to close the through-hole following the step. The holder has a space to hold the film test piece. A spacer adjusted to a thickness of the film test piece shall be used to fill a gap in a holding part for the film test piece when the film test piece is held in the holder. In the case of the suspension with the powder test sample, the spacer to fill an orifice of the holding part for the film test piece shall be used. This test method is not limited to the specification of this measuring device setup shown in Annex A, but the test vessel shall be made of airtight, chemical resistant, and non-photoresponsive material and be able to keep the W.T. constant and be what an adequate amount of light for the photocatalytic activity is gained under stirring. The O-rings and other implements shall be near-UV resistant and chemical resistant, and it is preferable to use fluoro-carbon polymer for the O-rings and polytetrafluoroethylene for the others.

9 Test material

9.1 Powder test sample

The powder test sample is the photoctalytic material synthesized for treatment of environmental pollution and does not contain so many organic compounds as plastic cement. The quantity of powder test sample shall be 0,110 g ± 0,005 g.

9.2 Film test piece

Sample size of the film test piece shall be as follows:

— width: 29,5 mm ± 0,5 mm;

— length: 59,5 mm ± 0,5 mm;

— thickness: 5 mm ± 0,5 mm.

The film test piece is held vertically (lengthwise) using a holder and a minimum effective area of 49,5 mm × 29,5 mm of the film is subjected to the test.

NOTE 1 If the film test piece cannot maintain the shape in the solution, it is not applicable to an adequate evaluation in this test method.

NOTE 2 A film test piece of less than 5 mm thickness is also available by using adjustable spacer.

10 Procedure of the measurement

10.1 Test temperature

All tests shall be carried out at R.T. of 23 °C ± 5 °C and W.T. shall be at R.T. ± 1 °C. W.T. should be controlled to a certain temperature by thermostatic bath/cryostat.

10.2 Preparation of water

Purified water quality shall be distilled or deionised water. The DO in purified water of 1 000 ml is saturated by aeration of 1 500 ml/min for 60 min and the DO saturation is determined by the DO analyser. Then the c_{DO} obeys the concentration specified in ISO 5814. The air for the aeration should be passed through another 1 000 ml of distilled or deionized water to clean itself.

10.3 Powder test sample

10.3.1 Preparation of suspension from powder test sample

Powder test sample is added into 1 000 ml of the purified water and agitated by a sonicator (within 23 kHz to 43 kHz) for 15 min. After sonication, the suspension is irradiated by a UV lamp at a UV light irradiation intensity of 1 mW/cm^2 under aeration by purified air in vigorous stirring for 180 min. Then the aeration flow rate shall be above 1 500 ml/min. A cylindrical glass vessel (a size of o.d. 80 mm × H 300 mm, the volume is above 1 000 ml) is made of borosilicate glass. After UV light irradiation, the pH value of the suspension shall be within 5 to 7. If it is not such a range, the powder test sample shall be washed to remove materials which change the pH. The temperature of the suspension is adjusted to R.T. Then the saturated c_{DO} shall be the DO value ±0,3 mg/l at the test temperature, which is specified in ISO 5814.

10.3.2 Procedure of the measurement

A volume of 0,5 ml of phenol stock solution is added into 195 ml of the suspension and the concentration of phenol shall be finally 0,33 mmol/l. The magnetic stirring bar is then put into the suspension. Here, the saturated c_{DO} shall be the DO value ±0,3 mg/l at the test temperature, which is specified in ISO 5814. Set the holder into the test vessel with the suspension overflowed. To avoid forming air bubbles in the suspension, insert the DO electrode into the holder with the suspension overflowed and set the plug into the through-hole of the holder. The test vessel is then put into the water jacket. Start stirring and recording the c_{DO} every minute at least for 5 min in the dark. Start and carry on the UV light irradiation for 60 min. After stopping the UV light irradiation, keep recording the c_{DO} every minute for 5 min in the dark. If the c_{DO} becomes 0 before regulated examination time, the time and the POD should be included in the test report.

NOTE The flowing water in the water jacket is purified water and the W.T. is adjusted to R.T.

10.4 Film test piece

10.4.1 Preparation of film test piece

Set the film test piece into the holder and then set the holder in the purified water-filled test vessel. The film test piece is irradiated by a UV lamp at a UV light irradiation intensity of 1 mW/cm^2 under

aeration by purified air in vigorous stirring for 180 min. Then the aeration flow rate shall be above 1 500 ml/min. W.T. is adjusted to be R.T. and then the saturated c_{DO} shall be the DO value ±0,3 mg/l at the test temperature, which is specified in ISO 5814. In addition, the pH value of the solution shall be within 5 to 7. If it is not such a range, the film test piece shall be washed to remove materials which change the pH.

NOTE This preparation procedure can be skipped when it is apparent that the film test piece is clean.

10.4.2 Procedure of the measurement

Of Phenol solution, 1 mmol/l adjusted with the purified water saturated with dissolved oxygen shall be prepared for the test solution. Pour the test solution into the test vessel. Put the magnetic stirring bar in the test vessel. Set the film test piece into the holder. Set the holder into the test vessel with the test solution overflowed. To avoid forming air bubbles in the test solution, insert the DO electrode into the holder with the test solution overflowed and then put the plug into the through-hole of the holder. The test vessel is placed into the water jacket. Start stirring and recording the c_{DO} every minute at least for 5 min in the dark. Start and carry on the UV light irradiation for 180 min. After stopping the UV light irradiation, keep recording the c_{DO} every minute for 5 min in the dark. If the c_{DO} becomes 0 before regulated examination time, the time and the POD should be included in the test report.

NOTE The flowing water is purified water and the W.T. is adjusted to R.T.

10.5 Blank POD(%)

If reproducible result is not obtained, as the powder test sample or the film test piece is polluted by organic compounds and the pollution affects the test result, Blank POD(%) measurement is needed to check cleanliness of the test sample.

After the preparation procedure of powder test sample/film test piece without the UV light irradiation is performed, the POD shall be measured without the phenol addition. The POD is calculated by Formula (2) as explained in Clause 11. On the other hand, the POD shall be measured without the phenol addition following the normal preparation procedure of powder test sample/film test piece (with the UV light irradiation). Each Blank POD(%) shall be calculated using Formula (1). If the Blank POD(%) after preparation procedure with the UV light irradiation is not to be less than 15 %, the preparation procedure of powder test sample/film test piece should be repeated until the Blank POD(%) becomes less than 15 %. The calculated values are usually rounded to second decimal place according to ISO 80000-1:

$$\text{blank POD}(\%) = \text{POD} / c_{iDO} \times 100\ \% \quad (1)$$

11 Evaluation of results

11.1 General

Report all values rounded to second decimal places.

11.2 Evaluation of POD

For the quantitative data treatment, use only the dark parts before and after the UV light irradiation, i.e. the average c_{DO} in the dark before and after the UV light irradiation. The POD analysed by this test method is calculated using Formula (2). If POD becomes 0 mg/l within the measurement period of 60 min or 180 min, the termination time and the POD should be reported. The calculated values are usually rounded to the second decimal place according to ISO 80000-1:

$$\text{POD} = c_{iDO} - c_{fDO} \quad (2)$$

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 19722;

b) name of measurer and testing laboratory;

c) date of the test;

d) details concerning the measurement device (DO analyser, data logger, thermometer);

e) details concerning the light intensity, supplier, number of UV light sources used (BL or BLB);

f) details concerning the sample name, composition, purity, quality/reagent grade, supplier (powder, film, phenol, water);

g) temperature in the test room in the laboratory;

h) temperature of test solution;

i) POD (mg/l);

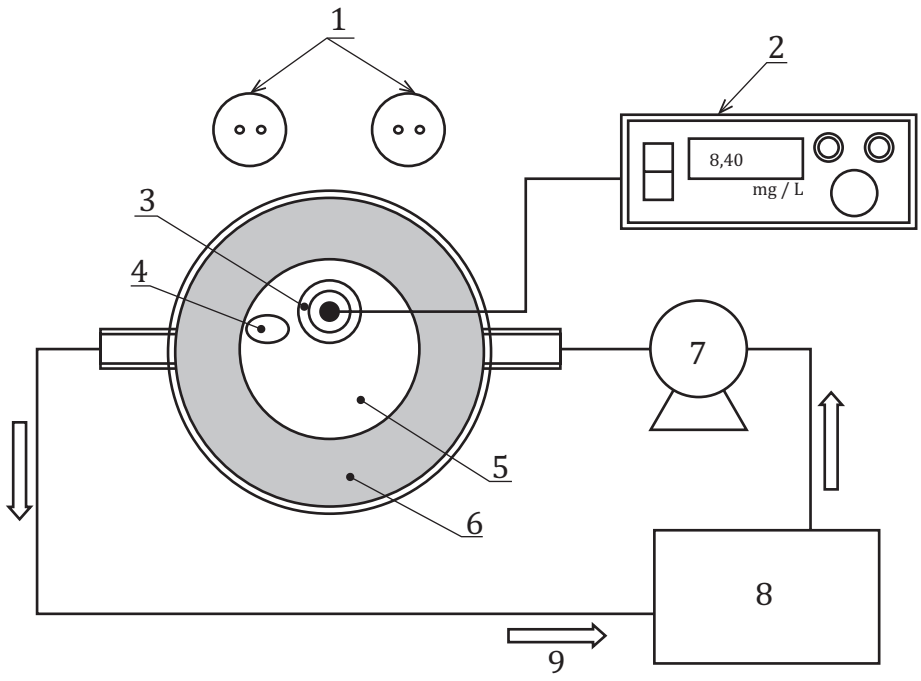
j) time and POD (mg/l) if the POD becomes 0 before regulated examination time;

k) Blank POD(%) and repeated number of the procedure;

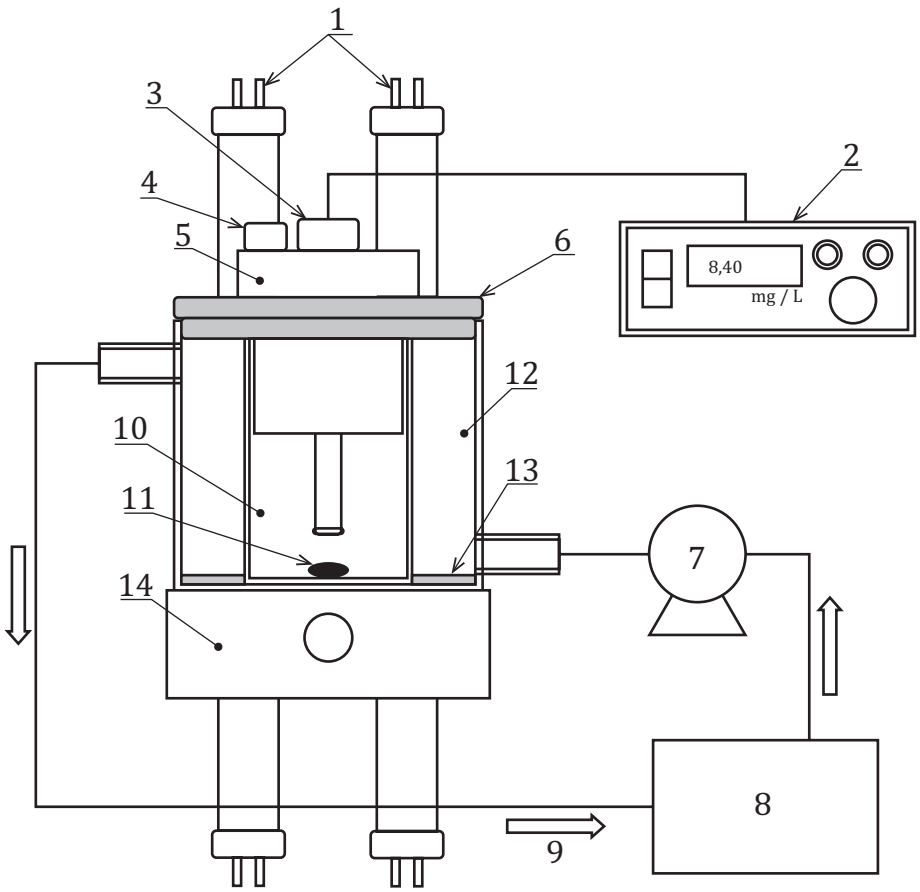
l) details of any operation not specified in this document or in the International Standards to which reference is made, and any operations regarded as optional, as well as any incidents likely to have affected the results.

Annex A
(informative)

Example of suitable measuring device component



a) Plane view



b) Side view

Key

1	UV light source	8	thermostatic bath/cryostat
2	DO analyser	9	water flow direction
3	DO electrode	10	test vessel
4	plug	11	stirring bar
5	holder	12	water jacket
6	test vessel holder (upper)	13	test vessel holder (lower)
7	pump	14	magnetic stirrer

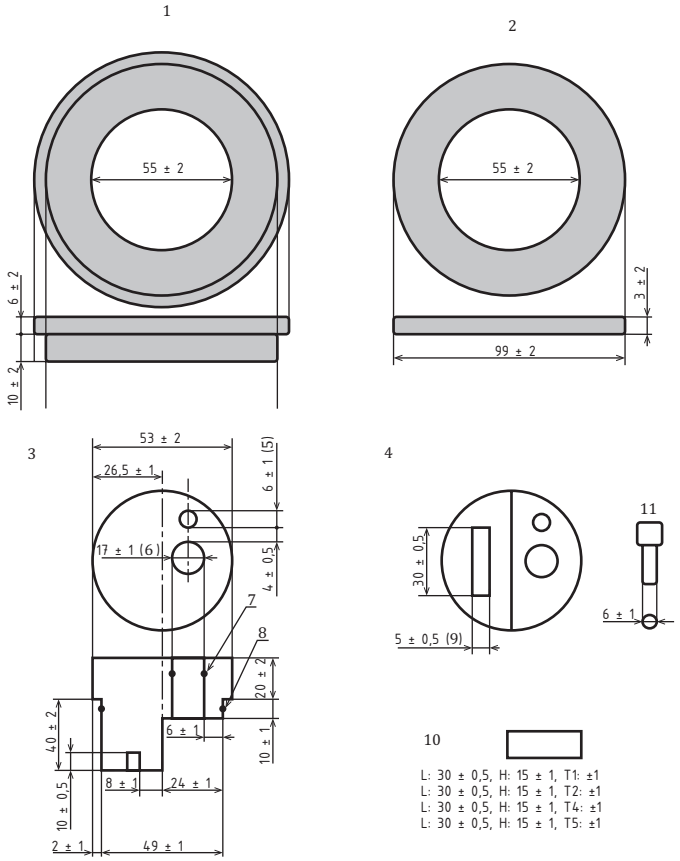
Figure A.1 — Schematic diagram of measuring device setup

Annex B
(informative)

Example of suitable test vessel and implement

B.1 Schematic diagram of test vessel and implement

Dimensions in millimetres



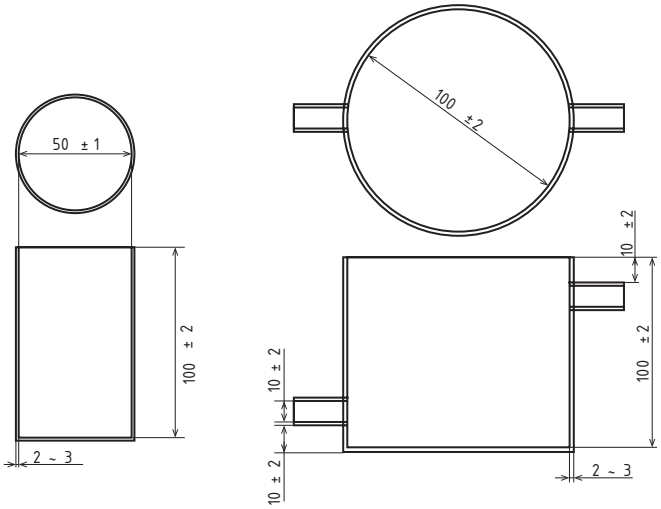
Key

1 test vessel holder (upper)
2 test vessel holder (lower)
3 holder upper view
4 holder lower view
5 through-hole for suspension or test solution
6 hole for DO electrode
7 O-ring (P16)
8 O-ring (P46)
9 holding part for film test piece
10 spacer
11 plug for 5

Figure B.1 — Schematic diagram of test vessel holder and holder for DO electrode and film test piece

B.2 Schematic diagram of test vessel and water jacket

Dimensions in millimetres



Key

1 test vessel

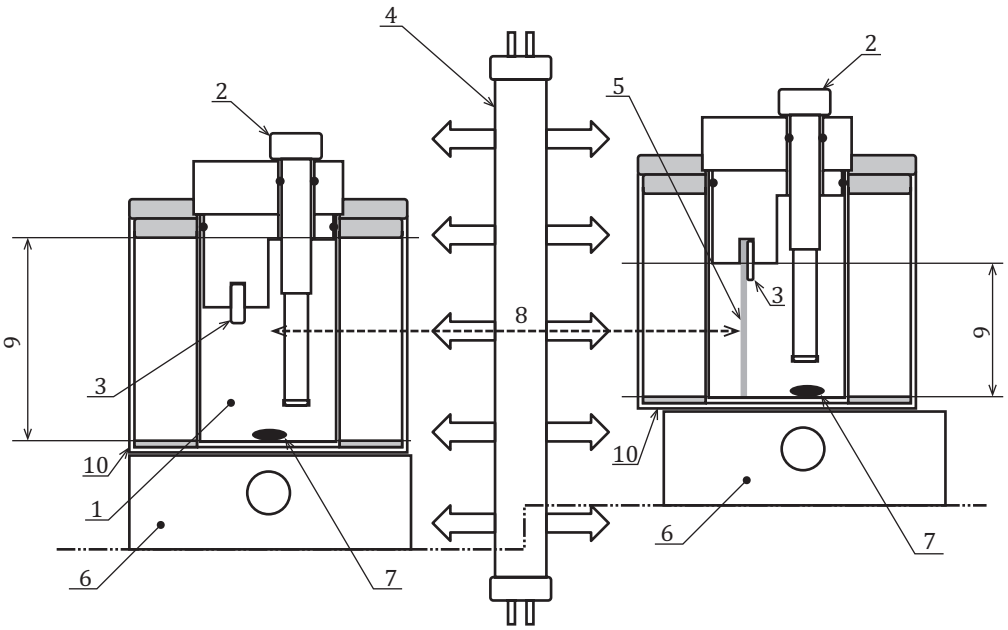
2 water jacket

Figure B.2 — Schematic diagram of test vessel and water jacket

NOTE The test vessel and the water jacket are made of borosilicate glass.

Annex C
(informative)

Example of test position of test vessel



Key

1	powder test sample (suspension)	6	magnetic stirrer
2	DO electrode	7	magnetic stirring bar
3	spacer	8	centre of light
4	UV light source	9	reaction area
5	film test piece	10	position of stirring bar and test vessel

Figure C.1 — Schematic diagram of test position of measuring device setup for powder suspension and film test piece

Film test piece

Light intensity is adjusted at the centre of the film test piece surface. The centre of film test piece surface shall then be placed in the middle of the light source. The stirring bar position is under the DO electrode.

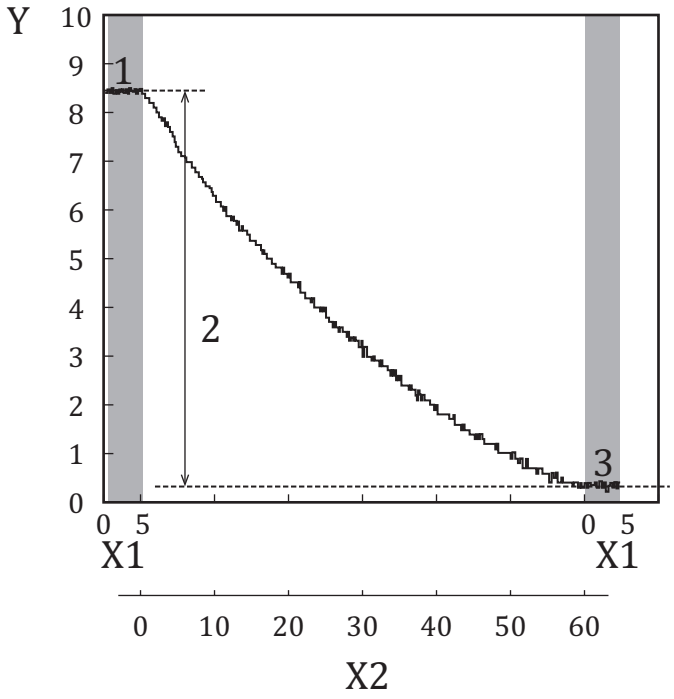
Powder test sample

Light intensity is adjusted at the centre of the test vessel. The centre of the test vessel shall then be placed in the middle of the light source. The stirring bar position is in the centre of the test vessel.

Annex D
(informative)

Example of a data evaluation

The measurements were performed in the test vessel designed as shown in Annexes A, B and C and the UV light irradiation wavelength was λ = 351 nm and I was 1,5 mW/cm^2. The DO analyser was UC12 supplied by CENTRAL KAGAKU®[1)]. The time interval of measurement is that 5 min in the dark after the start of measurement, the UV light irradiation starts at 5 min and is carried on for 60 min, the UV light irradiation stops at 65 min and 5 min more in the dark. The data was recorded by a data logger at 100 ms as a time interval. The POD was calculated using Formula (1). Powder test sample (PL) was used as an example. Figure D.1 shows an example of experimental results and the results for this example calculation are listed in Table D.1.



Key

1 c_{iDO}
2 POD
3 c_{fDO}
X1 dark condition time (min)
X2 UV light irradiation time (min)
Y c_{DO}

NOTE Decrement of DO with powder test sample PL by decomposition of c = 0,33 mmol/l phenol.

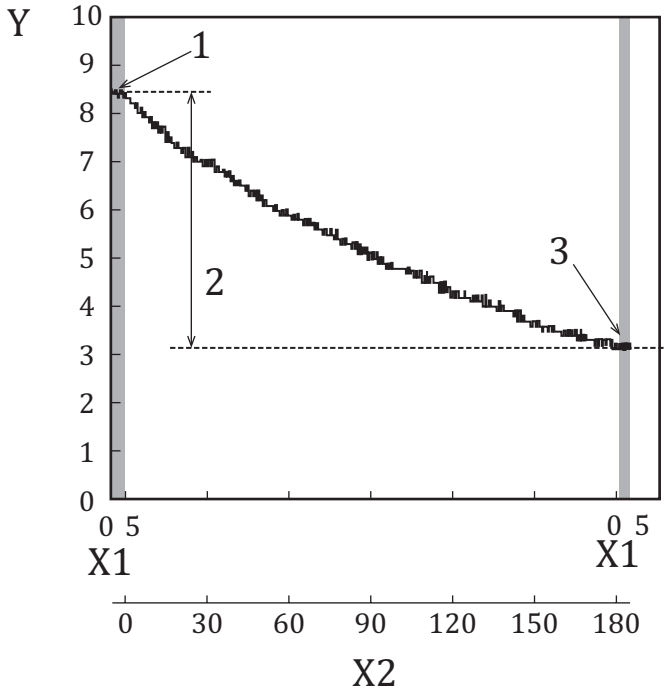
Figure D.1 — Result obtained from powder test sample

1) Example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Table D.1 — Evaluation of POD Reproducibility for given example

Powder test sample name (PL)	POD (mg/l)
Test 1	8,07
Test 2	8,08
Test 3	8,12
Mean value	8,09
Reproducibility standard deviation	0,01
Coefficient of variation of reproducibility (CV), %	0,15

Film test piece (FX) was also used as an example. Figure D.2 shows an example of experimental results and the results for this example calculation are listed in Table D.2.



Key

1 c_{iDO}
2 POD
3 c_{fDO}
X1 dark condition time (min)
X2 UV light irradiation time (min)
Y c_{DO}

NOTE Decrement of DO with film test piece FX by decomposition of c = 1 mmol/l phenol.

Figure D.2 — Result obtained from film test piece

Table D.2 — Evaluation of POD reproducibility for given example

Film test piece name (FX)	POD (mg/l)
Test 1	5,40
Test 2	5,22
Test 3	4,91
Mean value	5,18
Reproducibility standard deviation	0,12
Coefficient of variation of reproducibility (CV), %	2,26

Annex E
(informative)

Results of the interlaboratory test

Round robin tests were undertaken among four collaborating laboratories. Each laboratory conducted the tests three times for each sample, with three kinds of samples for powder test sample or film test piece. Mean value, reproducibility standard deviation and coefficient of variation of reproducibility, which are obtained from each laboratory, are summarized in Tables E.1 and E.2.

Table E.1 — Results of interlaboratory test (powder test sample)

Powder test sample	Number of individual values	POD (mg/l)		
		PL	PM	PN
Laboratory 1	3	8,09	6,03	2,50
Laboratory 2	3	7,35	4,90	2,20
Laboratory 3	3	6,98	6,07	3,72
Laboratory 4	3	7,60	6,20	3,73
Mean value		7,51	5,80	3,04
Reproducibility standard deviation		0,20	0,26	0,35
Coefficient of variation of reproducibility (CV), %		2,69	4,51	11,5

Table E.2 — Results of interlaboratory test (film test piece)

Film test sample	Number of individual values	POD (mg/l)		
		FX	FY	FZ
Laboratory 1	3	5,18	2,54	1,46
Laboratory 2	3	6,16	3,75	2,67
Laboratory 3	3	4,60	3,14	1,66
Laboratory 4	3	6,50	3,10	2,20
Mean value		5,61	3,13	2,00
Reproducibility standard deviation		0,38	0,21	0,24
Coefficient of variation of reproducibility (CV), %		6,76	6,84	11,9

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BSI Group Headquarters

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