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Fine bubble technology — Evaluation method for determining gas content in fine bubble dispersions in water —

Part 1: Oxygen content

Technologie des fines bulles — Méthode d'évaluation pour déterminer la teneur en gaz dans les dispersions de fines bulles dans l'eau —

Partie 1: Teneur en oxygène

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This document was prepared by Technical Committee ISO/TC 281, *Fine bubble technology*.

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Introduction

Fine bubble dispersion in water has been used in various industries in recent years. Particularly in the fishery and food-processing industries, fine bubble technology is widely accepted as means for controlling dissolved oxygen level. For example, air fine bubbles are used to prevent oxygen depletion in the water of aquafarm and nitrogen fine bubbles are applied to reduce oxidization of fresh fish fillet.

The determination of the oxygen content in water is necessary to monitor the quality of object to be controlled by fine bubble dispersion in water. In the measurement of the oxygen content in fine bubble dispersion in water, however, attention should be paid to the possibility that the presence of fine bubbles themselves influences the measurement results.

In the case of air microbubble, air inside the bubbles is being dissolved during their slow floatation resulting in the increase in the oxygen content when oxygen is not oversaturated. In contrast, ultrafine bubbles (UFBs) have little influence on the oxygen content because the total amount of oxygen in UFBs is negligibly small compared to the intrinsic dissolved oxygen content in raw water. Furthermore, there is a possibility that the precipitation of visible bubbles on the surface of oxygen sensor, which is originated from dissolved gas, influences its measurement result.

Therefore, to evaluate the oxygen content of fine bubble dispersion in water, the state of bubbles in a sample water during the measurement is figured out.

This document is intended to specify the evaluation method of the oxygen content in fine bubble dispersion in water by three measurement methods: optical sensor, electrochemical probe and iodometric methods, which are widely accepted in industries. The standardized evaluation method for the oxygen content enables easy and solid comparison among fine bubble dispersion in various states.

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Fine bubble technology — Evaluation method for determining gas content in fine bubble dispersions in water —

Part 1: Oxygen content

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is essential that tests conducted in accordance with this document be carried out by suitably trained staff.

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1 Scope

This document specifies evaluation methods for the oxygen content in fine bubble dispersion in water.

Three test methods which are adopted include the optical sensor, the electrochemical probe and the iodometric method. The first two methods have an advantage in availability of in situ and real-time measurement, and high accessibility to commercially available instruments. The last one, composed of a well-established chemical analysis procedure, is advantageous in the situation where the instruments to be used in the first two methods are unavailable.

The detection limits of the electrochemical and optical sensor methods are stated in the instruction manuals of the instruments, in most cases 0,1 mg/l or 0,2 mg/l. The upper limit depends on the specification of the instrument used. Most instruments allow measurement of a supersaturated sample.

Measurement range of the iodometric method is between 0,2 mg/l and 20 mg/l.

NOTE Chemical analysis methods other than the iodometric method can be applied[1] as an alternative.

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 5813:1983, *Water quality — Determination of dissolved oxygen — Iodometric method*

ISO 5814:2012, *Water quality — Determination of dissolved oxygen — Electrochemical probe method*

ISO 17289:2014, *Water quality — Determination of dissolved oxygen — Optical sensor method*

ISO 20480-1, *Fine bubble technology — General principles for usage and measurement of fine bubbles — Part 1: Terminology*

ISO/TR 23015, *Fine bubble technology — Measurement technique matrix for the characterization of fine bubbles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20480-1 apply.

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

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

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

3.1
UFB dispersion
UFBD
liquid which contains ultrafine bubbles

[SOURCE: ISO 21255:2018, 3.2[2]]

4 Interferences

4.1 Iodometric method

Readily oxidizable organic substances such as tannins, humic acid and lignins, interfere. Oxidizable sulfur compounds such as sulfides and thiourea also interfere.

To avoid such interferences, it is preferable to use the electrochemical probe or the optical sensor method described in ISO 5714 or ISO 17289 respectively.

In the presence of suspended matter capable of fixing or consuming iodine, or if in doubt about the presence of such matter, the modified procedure described in ISO 5813:1983, Annex, shall be used.

Preferably, however, determine the oxygen content with the electrochemical probe or the optical sensor method described in ISO 5714 or ISO 17289 respectively.

4.2 Electrochemical probe method

Gases and vapours such as chlorine, hydrogen sulfide, amines, ammonia, bromine and iodine which diffuse through the membrane can interfere.

Solvents, oils, sulfides, carbonates and biofilms can also interfere with the measured current by causing obstruction and deterioration of the membrane or corrosion of the electrodes.

If in doubt about such interferences, it is preferable to use the optical sensor method described in ISO 17289.

5 Implication of measurement result

In the measurement of MB dispersion in water, the oxygen content should be determined after the bubbles are completely dissolved in the water sample. When MBs remain in water, the measured data indicate those in the process of dissolving of MBs.

In the measurement of UFB dispersion in water, the implication of measurement results differs depending on whether UFBs are present or not during the measurement. When UFBs are present, the data indicate the dissolved oxygen in coexistence with UFBs. When they are not present, the data indicate the total oxygen content of dissolved oxygen and UFBs.

The presence of UFBs shall be verified with one of the characterization techniques described in ISO/TR 23015.

6 Requirement

6.1 General

During sampling, transportation and storage of the water sample to be measured, oxygen uptake and oxygen stripping shall be minimized.

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6.2 Correction for the salinity of sample water

If the optical sensor or electrochemical probe methods are used for saline water such as sea or estuarine waters, the salinity of sample water shall be input into the instrument. The salinity is estimated by the procedure described in ISO 17289:2014, Annex A for the optical sensor method or ISO 5814:2012, Annex A for the electrochemical probe method.

If the instrument does not have compensatory function for salinity, compensating procedure is described in ISO 5814:2012, Annex A.

7 Apparatus

7.1 Optical sensor method

The specification of the measuring instrument is described in ISO 17289:2014, Clause 6.

If extremely high oxygen level is expected, an optical sensor of sufficiently wide measuring range should be chosen.

If an adequate sensor is not available, the sample can be diluted before measurement according to the procedure described in ISO 20298-1.[3] Attention should be paid to a possibility that the oxygen content is varied during dilution.

7.2 Electrochemical probe method

The specification of the measuring instrument is described in ISO 5814:2012, Clause 6.

NOTE Usually, a temperature sensor and a barometer are part of the instrument used for the electrochemical probe and optical sensor methods.

7.3 Iodometric method

Ordinary laboratory equipment and narrow-mouthed glass flasks specified in ISO 5813:1983, Clause 5 are required.

8 Procedure

8.1 General

To evaluate the increase or decrease of the oxygen content by the generation of fine bubbles, the dissolved oxygen level of blank water shall be determined with the methods specified in 8.2 to 8.4, and the value shall be deducted from the oxygen content measurements of fine bubble dispersions.

The difference among the measurement results by the three methods, iodometric, electrochemical probe and the optical sensor methods, can be found in Annex A.

The influence of bubble attachment to sensor surface on the measurements of the oxygen content by the optical sensor and electrochemical probe methods can be found in Annex B.

8.2 Optical sensor method

8.2.1 General

The procedure is described in ISO 17289:2014, Clause 7. Normally, the measurement shall be carried out directly on-site in the water body to be analysed. If direct measuring is not possible, the measurement using a gastight connected flow-through device[4] is an alternative. Measuring immediately after discrete sampling is another alternative.

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Any discrete sampling procedure will result in a higher measurement uncertainty.

8.2.2 Sampling, measuring technique and precautions to be taken

Sampling procedure shall be in accordance with ISO 17289:2014, 7.1.1. Measuring techniques and precautions to be taken shall be in accordance with ISO 17289:2014, 7.2.

In the measurement of MB or UFB dispersion in water during the operation of bubble generator, the formation of visible bubbles on the surface of sensor shall be minimized.

NOTE To reduce the adverse effect of visible bubbles, the following can show practical effect:

— shaking off bubbles by tapping sensor body routinely;

— making sensor surface upward or inclined from the perpendicular.

In the discrete sampling of MB or UFBD using a sample vessel, confirm the disappearance of MBs by dissolution or flotation ahead of the sampling by the naked eye. The formation of visible bubbles on the surface of sensor shall also be minimized.

8.2.3 Calibration

Calibration shall be in accordance with ISO 17289:2014, 7.3.

8.2.4 Determination

Determination shall be in accordance with ISO 17289:2014, 7.4.

8.3 Electrochemical probe method

8.3.1 General

The procedure is described in ISO 5814:2012, Clause 7. The measurement shall be carried out directly on-site in the water body to be analysed. If direct measuring is not possible, the measurement using gastight connected flow-through device[4] is an alternative. Measuring immediately after discrete sampling is another alternative.

Any discrete sampling procedure will result in a higher measurement uncertainty.

8.3.2 Sampling, measuring technique and precautions to be taken

Sampling procedure shall be in accordance with ISO 5814:2012, 7.1.1. Measuring techniques and precautions to be taken shall be in accordance with ISO 5814:2012, 7.2.

Additional measuring techniques and precautions to be taken are the same as those for the optical sensor method described in 8.2.2.

8.3.3 Calibration

Calibration shall be in accordance with ISO 5814:2012, 7.3.

8.3.4 Determination

Determination shall be in accordance with ISO 5814:2012, 7.4.

8.4 Iodometric method

The procedure is described in ISO 5813:1983, Clause 6.

Confirm the disappearance of micro bubbles in the flask after sampling.

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The presence of oxidizing or reducing substances can be checked by the procedure described in ISO 5813:1983, 6.2.

In the presence of oxidizing substances, follow the procedure of ISO 5813:1983, 9.1.

In the presence of reducing substances, follow the procedure of ISO 5813:1983, 9.2.

In the absence of oxidizing or reducing substances, follow the procedure of ISO 5813:1983, 6.3 to 6.6.

9 Calculation and expression of results

9.1 Optical sensor and electrochemical probe methods

Most instruments are equipped with an automatic calculation. If required, calculate the percentage saturation of dissolved oxygen in water according to the procedure described in ISO 17289:2014, Clause 8 or ISO 5814:2012, Clause 8.

9.2 Iodometric method

The dissolved oxygen content is expressed in milligrams of oxygen per litre.

In the absence of oxidizing or reducing substances, use the calculating formula described in ISO 5813:1983, Clause 7.

In the presence of oxidizing or reducing substances, calculating formula is given in ISO 5813:1983, 9.1 or 9.2, respectively.

10 Test report

The test report shall include the following information:

a) the test method used, together with a reference to this document, i.e. ISO 7383-1:2024, and information about the measuring instruments used:

— name of instrument, type of test method and its manufacture's name;

b) the characterization method to verify the presence of UFBs:

— name of instrument, its manufacture's name, version of software, etc.;

c) the nature of the water used:

— property of the water sample such as pH, electroconductivity, etc.;

d) the measuring conditions:

— the temperature of the water when the measurement was carried out;

— the atmospheric pressure when the measurement was carried out;

— the elapsed time between the shutdown of bubble generator or the end sampling and the beginning of measurement test;

e) the result in accordance with Clause 9;

f) all circumstances that can have influenced the result.

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Annex A
(informative)

Influence of UFB on the measurement of the oxygen content

A.1 General

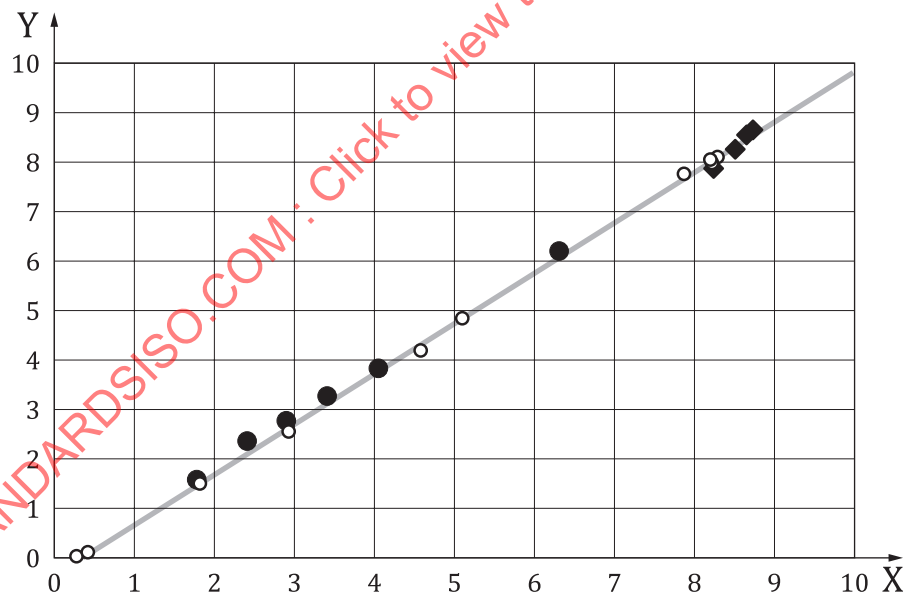
This annex provides the information about the influence of UFBs on the measurements of the oxygen content by three different methods: iodometric, electrochemical probe and the optical sensor methods. The difference among the measurement results by the three methods is also determined.

A.2 Electrochemical probe and optical sensor methods

The oxygen content adjusted water was prepared as a control. The oxygen content was reduced by dissolution of nitrogen gas by sparging. Sodium sulfite was dissolved to have the oxygen content close to zero. The oxygen saturated water was prepared by sparging of filtered air for about 1 h.

The number concentration of air containing UFBs and that of N_2 gas containing ones measured by the particle tracking analysis method are $1{,}26 \times 10^8$ 1/ml and $1{,}73 \times 10^9$ 1/ml respectively. The oxygen content of UFBD was adjusted by the time of exposure to the atmosphere.

The 1-point calibration in water saturated air was carried out for both, the electrochemical probe and optical sensor methods.



Key

X	oxygen content by optical sensor method, in mg/l
Y	oxygen content by electrochemical probe method, in mg/l
◆	air UFBD

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● N_2 UFBD

○ oxygen content adjusted water

——— linear regression of oxygen content adjusted water

NOTE Outputs of electrochemical probe and optical sensor can exhibit the non-linearity when the oxygen content is significantly more than a level of saturation.

Figure A.1 — Comparison of the oxygen content measurement results by the optical sensor and electrochemical probe methods

The comparison of the oxygen content measurements by the optical sensor and electrochemical probe methods is shown in Figure A.1. The oxygen content of UFBD was plotted along the regression line for that of the control, implying that UFBs have little influence on the measurement of the oxygen content and that measurement results by the two methods do not differ substantially.

A.3 Iodometric method

The oxygen content measurement by the iodometric method was carried out at a third-party chemical analysis lab for safety reasons. The samples in air-tight glass containers were transported with phase-change heat storage material to keep a given temperature ranging from 21,3 °C to 24,7 °C.

Oxygen saturated water was prepared as a control. UFBD was exposed to the atmosphere for 24 h after generation to reduce its oxygen content to a level of saturation.

The number concentration of UFBD measured on the same day as the oxygen content measurement by the particle tracking analysis method after the exposure to atmosphere was $1{,}26 \times 10^8$ 1/ml.

Table A.1 shows the comparison of the oxygen content between water and UFBD measured by the iodometric method.

Table A.1 — Oxygen content of water and UFB dispersion by iodometric method

	Iodometric method mg/l	
	Batch 1	Batch 2
O_2 saturated pure water	7,7	7,8
O_2 saturated UFB dispersion	7,8	7,8

The oxygen content of UFBD has a good agreement with that of oxygen saturated water, implying that UFBs have little influence on the measurement of the oxygen content by the iodometric method.

A.4 Comparison between iodometric and electrochemical probe methods

The same samples as Clause A.3 were measured by the electrochemical probe method in the same chemical analysis lab on the same day as the measurement by the iodometric method.

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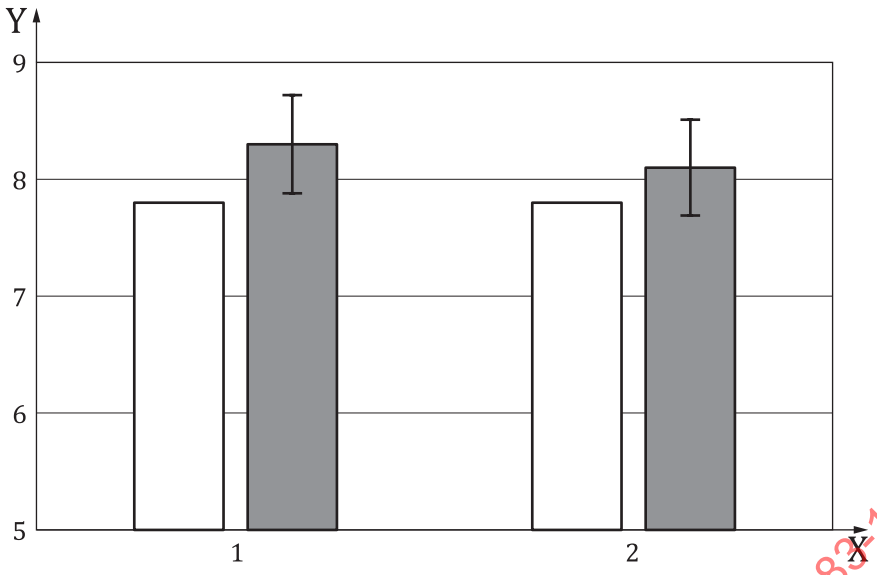

Key

X	sample specification
Y	oxygen content (mg/l)
1	oxygen saturated water
2	oxygen saturated UFB dispersion
□	iodometric method
■	electro chemical probe method

NOTE The error bar denotes standard deviation.

Figure A.2 — Oxygen content measurement of the same sample in Clause A.3 by electrochemical probe

Figure A.2 shows the comparison of the measurement results by the electrochemical probe method with those by the iodometric method. Oxygen contents by the iodometric method were slightly lower than those by the electrochemical probe method and close to the lower end of error bars corresponding to nominal repeat accuracy thereof.

Measurement results are not substantially different between the iodometric and electrochemical methods. Given the results in Figure A.1, it can be argued that measurement results by the three different methods, iodometric, electrochemical probe and the optical sensor methods, do not differ substantially even in the presence of UFBs.

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Annex B
(informative)

Influence of bubble attachment to sensor surface

B.1 General

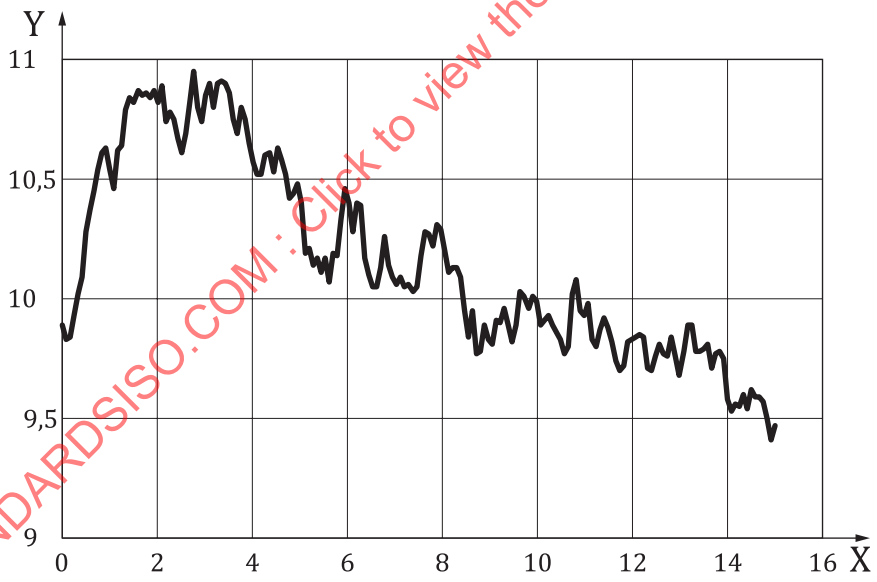
This annex provides the information about the influence of bubble attachment to sensor surface on the measurements of the oxygen content by the optical sensor and electrochemical probe methods.

B.2 Optical sensor method

The measurement by the optical sensor method is based on the principle that luminescent quenching by oxygen molecule depends on the partial pressure of oxygen regardless of dissolved in liquid or gas. Therefore, the influence of bubble attachment to sensor surface is predicted in principle.

The temporal change in the oxygen content during the operation of UFB generator was monitored by an optical sensor, whose upper limit of measurement range was 50 mg/l, for air and N_2 gas containing UFBs.

The bubbles are generated by hydrodynamic cavitation followed by pulverization by shear force in vortex flow and micro- and milli-bubbles coexist with UFBs during the operation of generator, resulting in the attachment of visible bubbles to the sensor surface.



a) Air UFB dispersion

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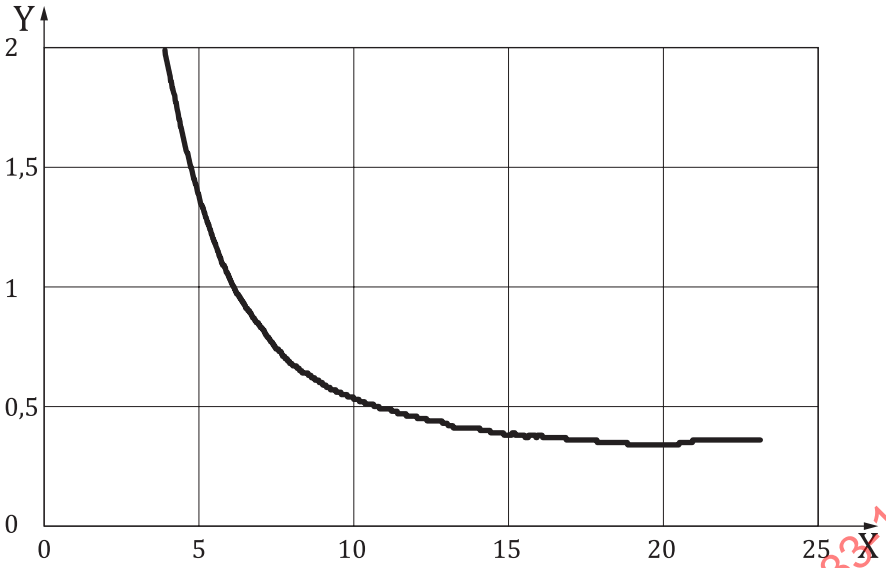

b) N_2 UFB dispersion

Key

X elapsed time (min)

Y oxygen content (mg/l)

Figure B.1 — Temporal change in oxygen content during the operation of UFB generator measured by the optical sensor method

The profile of the oxygen content of air UFBD includes high-frequency component as shown in Figure B.1 a). Meanwhile, Figure B.1 b) exhibits uniform decrease in the oxygen content during the generation of N_2 gas UFBs.

These results demonstrate the influence of bubbles containing oxygen gas on the temporal measurement of the oxygen content by the optical sensor method.

B.3 Electrochemical probe method

The temporal change in the oxygen content during the operation of UFB generator was also monitored by electrochemical probe, whose upper limit of measurement range was 20 mg/l, during the generation of air and N_2 gas UFBs.

Trend of temporal change in the oxygen content exhibited in Figure B.2 is similar to that in measurement results by the optical sensor method.

These results demonstrate that measurement by electrochemical probe method is influenced by the bubbles containing oxygen gas.

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