IEC 60746-4

Edition 2.0 2018-12

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Expression of performance of electrochemical analyzers – Part 4: Dissolved oxygen in water measured by membrane-covered amperometric sensors

Expression des qualités de fonctionnement des analyseurs électrochimiques – Partie 4: Oxygène dissous dans l'eau mesuré par des capteurs ampérométriques recouverts d'une membrane

IEC 60746-4:2018-12(en-fr)

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

COMMISSION
ELECTROTECHNIQUE
INTERNATIONALE

ICS 19.080; 71.040.40

ISBN 978-2-8322-6274-0

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FOREWORD

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International Standard IEC 60746-4 has been prepared by subcommittee 65B: Measurement and control devices, of IEC technical committee 65: Industrial-process measurement, control and automation.

This second edition cancels and replaces the first edition published in 1992. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) terms and definitions have been revised to meet the requirements of ISO/IEC Directives Part 2:2016.

b) ISO 5814:2012 is cited as reference for solubility tables of dissolved oxygen in water with variable salt content at different pressure and temperature.

The text of this International Standard is based on the following documents:

FDIS	Report on voting
65B/1128/FDIS	65B/1138/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 60746 series, published under the general title *Expression of performance of electrochemical analyzers*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

EXPRESSION OF PERFORMANCE OF ELECTROCHEMICAL ANALYZERS –

Part 4: Dissolved oxygen in water measured by membrane-covered amperometric sensors

1 Scope

This part of IEC 60746 is intended:

- to specify terminology, definitions and requirements for statements by manufacturers for analyzers, sensor units and electronic units used for the determination of dissolved oxygen partial pressure or concentration;
- to establish performance tests for such analyzers, sensor units and electronic units;
- to provide basic documents to support the applications of quality assurance standards [1][1].

This document applies to analyzers using membrane covered amperometric sensors. It applies to analyzers suitable for use in water containing liquids, ultrapure waters, fresh or potable water, sea water or other aqueous solutions, industrial or municipal waste water from water bodies (e.g. lakes, rivers, estuaries), as well as for industrial process streams and process liquids. Whilst in principle amperometric oxygen-analyzers are applicable in gaseous phases, the expression of performance in the gas phase is outside the scope of this document.

This document is applicable to analyzers specified for permanent installation in any location (indoors or outdoors) using membrane-covered amperometric sensors.

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.

IEC 60746-1:2003, *Expression of performance of electrochemical analyzers – Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60746-1 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

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[1] Numbers in square brackets refer to the Bibliography.

3.1 Oxygen sensor properties

3.1.1

membrane covered amperometric sensor

sensor with a membrane covered metal cathode, a metal or metal/metal salt anode, optional auxilliary or reference electrodes and temperature sensors

Note 1 to entry: The electrochemical reduction of molecular oxygen occurs at the membrane-covered cathode. The metal or metal/metal salt anode provides a well-defined, electrochemically reversible oxidation reaction within the special filling electrolyte solution of the sensor. A gas-permeable membrane is employed to separate the electrolyte filling solution from the test medium to avoid contamination and to achieve a stable diffusion potential. In the case of applications in the gaseous phase the unit, volume fraction (v/v) are used.

Note 2 to entry: The cell can function as an electrochemical two-electrode cell, or as a potentiostatic three-electrode cell, or either as a galvanic cell. Within potentiostatic three-electrode cells, a high-impedance, current-free reference electrode is additionally implied to optimize the polarization voltage.

Note 3 to entry: Membrane-covered amperometric sensors are provided with temperature sensors located at the active sites of the electrode cells being in thermal contact with the media to be measured.

3.1.2

sensor sensitivity

change in electrode current of the amperometric cell caused by the application of a partial pressure of oxygen

3.1.3

zero current of sensor

current delivered by the sensor when the dissolved oxygen concentration or partial pressure is zero

Note 1 to entry: The zero current is expressed in µA or in nA.

3.1.4

polarization

voltage to be applied to an amperometric cell during a time period to increase the stability of the sensor

3.1.5

stabilization time

time period necessary for obtaining a stable measurement after reconnecting the sensor unit to the electronic unit for polarization purposes (see 3.1.4)

Note 1 to entry: Regeneration of the sensor with a new membrane or an electrolyte change or installation of a replacement sensor will require a subsequent stabilization time.

3.1.6

oxygen consumption

electrochemical reduction of molecular oxygen at the cathode of amperometric oxygen sensors

3.2 Electronics

3.2.1

electronic unit

electronic device of an electrochemical two-electrode cell comprises a stabilized DC voltage source and an electronic circuitry with a low impedance, direct current amplifier and optional microprocessor circuitries

Note 1 to entry: If the electronic unit of the analyzer comprises a microprocessor device, the detected amperometric signal will be digitised and evaluated to analyse the oxygen content.

Note 2 to entry: To quantify the amperometric signal of the oxygen reduction, software-based algorithmic routines or data sets comprising solubility tables of oxygen in water (with consideration of temperature, pressure and salinity) [2] and a vapour pressure table of water are used.

3.2.2
auxiliary electronics
electronic devices provided for potentiostatic circuitries and for temperature and pressure transducers

Note 1 to entry: In electronic devices for three-electrode cells, additional potentiostatic circuitries are implemented.

3.3 Measurement units and solubility of oxygen

3.3.1
partial pressure
units for the expression of the oxygen partial pressure (pO_2):

millibar (mbar) or kilopascal (kPa)

Note 1 to entry: In the case of special applications, the units mm Hg (Torr) and inch Hg may be used with known conversion ratio [2].

Note 2 to entry: In the case of applications in the gaseous phase the unit, volume fraction (v/v) are used.

Volume fraction is the quotient of the volume of a specified component and the sum of the volumes of all components of a gas mixture before mixing, all volumes referring to the pressure and the temperature of the gas mixture. Volume fraction will be expressed in %. The volume fraction and volume concentration take the same value if, at the same state conditions, the sum of the component volumes before mixing and the volume of the mixture are equal. However, because the mixing of two or more non-ideal gases at the same state conditions is usually accompanied by a slight contraction (or, less frequently, a slight expansion), this is not generally the case.

[SOURCE: EN 50104:2010, 3.1.5]

3.3.2
concentration
units for expression of the dissolved oxygen concentration (cO_2):

parts per million (ppm) = milligram · kg^{-1}

parts per billion (ppb) = $\mu g \cdot kg^{-1}$

Note 1 to entry: The mass of the test medium means in this case the mass of the sample water, including salts or other substances dissolved or suspended in it.

Note 2 to entry: Although ppm (parts per million) units are used in this document, it is sometimes convenient to use the units of milligrams per litre ($mg \cdot dm^{-3}$) or of micrograms per litre ($\mu g \cdot dm^{-3}$).

3.3.3
oxygen saturation index
unit for the expression of the dissolved oxygen in percent (%) to express the relative saturation of the actual oxygen concentration (parts per million), as fraction of the theoretical oxygen concentration (parts per million) of the air-saturated solution at the actual conditions of pressure, temperature and salinity

3.3.4
oxygen solubility
maximal possible dissolved oxygen concentration of the water sample in contact and equilibrium with air (air-saturated solution) at the actual conditions of pressure, temperature and salinity

Note 1 to entry: The oxygen solubility in water is dependent on other dissolved organic and inorganic compounds, dissolved electrolytes, salt-content in seawater (salinity) [2].

3.4 Test media

3.4.1
test medium
stable mixture of gases (nitrogen, nitrogen + oxygen), saturated with water vapour or humidifed reference air showing a known concentration or a known partial pressure of oxygen used for performance tests

Note 1 to entry: The concentration (see 3.3.2) or partial pressure or volume fraction (see 3.3.1) of dissolved oxygen and its uncertainty range shall be known.

3.4.2
calibration solution
stable mixture of humidified (saturated with water vapour) gases (nitrogen, oxygen) or humidifed reference air exhibiting a known stable concentration or partial pressure or volume fraction of oxygen (with tracable uncertainties) used for calibration of the analyzer

3.4.3
zero oxygen solution
solution with dissolved gas (pure nitrogen) or dissolved molecular or ionogenic compounds (substitutes) used to eliminate the content of free oxygen or a gaseous phase (pure nitrogen) saturated with water vapourSymbols

4 Influence quantities for membrane covered amperometric sensors

4.1 Temperature

In addition to the temperature dependent solubility-effect of oxygen [2], the temperature has an impact on membrane-covered amperometric sensors owing to the temperature-dependent variation of the diffusion of oxygen molecules at the liquid interfaces of membrane-covered sensors.

4.2 Pressure

Pressure affects the dissolved oxygen saturation value, or respectively the maximal oxygen concentration or the oxygen partial pressure [2]. The air-pressure of the ambient media (calibration with water-vapour saturated air) or respectively the sample pressure has to be determined applying an appropriate pressure sensor or has to be manually entered at the electronic unit (see 3.2.1).

4.3 Dissolved substances

Dissolved inorganic substances (salts, acids, alkalies) as well as organic substances have an impact on the oxygen solubility in water.

In seawater, the salinity (salt content by weight) shall be considered in addition to the measured temperature by employing combined salinity-temperature tables in order to determine the correct oxygen solubility [2].

4.4 Flow

Membrane covered amperometric sensors show in principle a small, but intrinsic, oxygen-consumption, which requires an adjusted flow-rate given at rated operation conditions.

5 Procedure for specification

5.1 Zero and span drift

Statements shall be made on zero and span drift in accordance with IEC 60746-1:2003, 6.2.5

5.2 Additional specifications for the sensor unit

5.2.1 Electrode and sensor materials

The manufacturer shall state the composition of the electrodes of the sensor and the construction materials in contact with the sample.

5.2.2 Dimensions of the sensor

Dimensions of the sensor shall be stated.

5.2.3 Permitted temperature and pressure range

Limiting conditions and rated ranges of use for sample conditions shall be stated including flow rate (if appropriate), pressure and temperature.

5.2.4 Temperature measurement and temperature compensation

The temperature measurement and the temperature compensation shall be specified.

5.2.5 Pressure compensation

The sensor pressure compensation technique shall be stated if relevant.

5.2.6 Zero current

The zero current of the sensor expressed in µA or in nA shall be stated.

5.2.7 Sensor sensitivity

The change in electrode current in µA or nA caused by the application of oxygen shall be stated at rated operation conditions, at rated reference conditions of 25 °C and at reference pressure of 1 013 hPa.

5.2.8 Stabilization time

The period of time (minutes, hours) shall be stated that is necessary for obtaining a stable measurement after reconnecting the sensor to the electronic unit for polarization purposes.

5.2.9 Oxygen consumption

The oxygen consumption expressed in ng or µg of oxygen per hour shall be stated at rated operation conditions, at rated reference conditions of 25 °C and at reference pressure of 1 013 hPa.

5.2.10 Flow rate

Sample flow-rate requirements, if relevant.

5.2.11 Method and extent of sensor regeneration

The recommended time interval for sensor regeneration and the procedure for sensor regeneration shall be stated.

6 Recommended standard values and ranges of influence quantities affecting the performance of electronic units

See Annex A of IEC 60746-1:2003.

7 Verification of values

7.1 General

7.1.1 General aspects of verification of values

See IEC 60746-1:2003, 6.1 and 6.2.

7.1.2 Testing procedure for linearity of the electronic unit

Modify the input current in accordance with IEC 60746-1:2003, 6.2.2.

7.1.3 Rated reference conditions for testing

During all tests, sample pressure and applied flow-rate shall remain constant within ± 1 % at the stated mean working value. If this is not possible (e.g. in the case of change of the barometric pressure), appropriate corrections shall be made. Excepted for tests at various temperatures, all other testing done in Clause 7 shall be conducted with the sample temperature within ± 0,3 °C of the mean working temperature. Unless otherwise stated, the mean temperature is 25 °C.

7.2 Simulator for testing electronic units

Testing of electronic units for linearity, zero and span adjustment, temperature compensation and effect of influence quantities such as supply voltage and frequency, room temperature, etc., shall be accomplished by using a suitable current source to substitute the sensor. Caution should be used to make sure that auxiliary electronics, temperature compensation devices or substitutes are still part of the electronics, so that the unit is fully operable and that, with electronic units employing a polarization voltage source for the sensor, no interaction occurs between the voltage source and the sensor simulating a current source.

7.3 Calibration solutions

See Annexes B and C.

7.4 Testing procedures for complete analyzer (sensor unit connected to electronic unit)

7.4.1 Intrinsic uncertainty

A signal representing approximately the mid-scale value of the rated input range, resulting from the exposure of the sensor unit to an appropriate level dissolved oxygen, is used to test for intrinsic uncertainty as described in 6.2.1 of IEC 60746-1:2003.

7.4.2 Linearity uncertainty

7.4.2.1 Realisation of procedure

Testing procedures are given in IEC 60746-1:2003, 6.2.2.

7.4.2.2 Sensor unit

Using an appropriate electronic unit, perform the test described in 7.4.2.3.

7.4.2.3 Analyzer

The sensor unit is exposed to calibration solutions of known dissolved oxygen content representing nearly zero, nearly full scale and at least three intermediate calibration points whose values are approximately uniformly distributed. It is important to provide adequate stirring. The final output of the analyzer is recorded. These steps are repeated once, and the linear least squares curve fit is calculated using both readings for each calibration point. The linearity error is determined graphically or numerically and expressed in terms of percent of rated range and corresponds to the maximum deviation for the least squares fit line. For reasons of efficiency, this test can be combined with the test for repeatability (see 7.4.3.3).

7.4.3 Repeatability

7.4.3.1 Realisation of procedure

Testing procedures are given in IEC 60746-1:2003, 6.2.5.

7.4.3.2 Sensor unit

Using an appropriate electronic unit, perform the test described in 7.4.3.3.

7.4.3.3 Analyzer

The sensor unit is exposed to test solutions representing as nearly as possible the minimum, the maximum and the median rated values. The steps are repeated N times (where $N > 6$) in each test solution in turn, at intervals of at least ten times the instruments' 90 % (T_{90}) response time. The recorded readings are converted to concentration units. The standard deviation is calculated for each set of recorded values for each solution and reported as repeatability.

7.4.4 Interference uncertainty (whole analyzer)

See IEC 60746-1:2003, 6.2.8.1.

Since the interrelationships which exist between influence quantities and results obtained are complex, the method used for testing interfering errors is left for agreement between the manufacturer and the user.

7.4.5 Zero drift and span drift

7.4.5.1 Sensor unit

Using an appropriate electronic unit, perform the test described in 7.4.5.2.

7.4.5.2 Analyzer

The sensor is exposed for 20 times T_{90} to a near-zero calibration solution. The data collection device reading is adjusted to approximately 5 % of full scale to serve as a "live zero". The sensor is then exposed for 20 times T_{90} to a calibration solution or a test solution that gives a reading between 75 % and 95 % of full scale. The difference between the two readings is noted. The test is repeated without further adjustment after the specified time interval, which is used for stability determinations (see IEC 60746-1:2003, 6.2.5), has elapsed. The zero and span drift uncertainties are reported in terms of percent of the rated range.

7.4.6 Output fluctuation of the analyzer

The sensor unit is exposed to test solutions representing as nearly as possible the minimum, the maximum and the median rated values. The steps are repeated N times (where $N > 6$) in each test solution in turn, at intervals of at least ten times the instruments 90 % time. The data collection device readings are converted to concentration units. The standard deviation is calculated for each set of recorded values for each solution and reported as repeatability.

7.4.7 Delay times T_{10} and 90 % rise or fall times T_{90}

7.4.7.1 Sensor unit

Using an appropriate electronic unit, perform the test as described in 7.4.7.2 or 7.4.7.3. depending on the sensor type.

7.4.7.2 Analyzers incorporating flow-cell sensor

Two calibration solutions or stable test solutions shall be provided with means of supplying either to the sensor inlet, at rated flowrate, selected by an appropriate two-way valve at the inlet port. The two test solutions shall differ in dissolved oxygen concentration by at least 50 % of the rated range. With a data-collection device connected to its output terminal, the analyzer is flushed with near-zero test solution until a constant reading is obtained. Then the upscale test solution is introduced, and a flag is set at the recording of the data collection device. Flow is continued until a constant reading is obtained. The values for delay time and 90 % rise time are determined from the data record. Successively, the first near-zero test solution is introduced once more, until the constant reading is obtained, and delay time as well as 90 % fall time are determined from the data record.

7.4.7.3 Analyzers incorporating immersion-type sensor

The test is similar, but the two test solutions are held in open containers of at least 10 litres capacity and provided with appropriate stirring. The sensor is transferred from one container to the other and a flag is set at the recording of the data collection device at the moment of immersion. Care should be taken to prevent exchange of oxygen from ambient air to the test solutions.

7.4.8 Temperature compensation

7.4.8.1 Temperature compensation test

- To compensate the temperature dependency of the temperature dependent diffusion rate of the sensor membrane; and
- to perform the correct expression of the oxygen analyzer using the stated measuring units (see 3.3) for the dissolved oxygen measurement;
- based on the known interrelations of: temperature, oxygen partial pressure, water vapor pressure, solubility in water, salinity effects, ambient atmospheric and sample pressure [2].

The complete test is performed twice with the temperature of the test solutions and at least two different test temperatures, depending on the application of the analyzer.

7.4.8.2 Temperature compensation test – measuring unit, concentration

Within the maximum temperature – and concentration – range, the compensation of variations of the sample temperature will be tested.

7.4.8.3 Temperature compensation test – measuring unit, partial pressure

Within the maximum temperature – and partial pressure – range, the compensation of variations of the sample temperature will be tested.