SVENSK STANDARD
SS-EN ISO 5814:2012

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Vattenundersökningar – Bestämning av halten löst syre – Elektrokemisk metod (ISO 5814:2012)

Water quality – Determination of dissolved oxygen – Electrochemical probe method (ISO 5814:2012)

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Europastandarden EN ISO 5814:2012 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 5814:2012.

Denna standard ersätter SS-EN 25814, utgåva 1.

The European Standard EN ISO 5814:2012 has the status of a Swedish Standard. This document contains the official version of EN ISO 5814:2012.

This standard supersedes the Swedish Standard SS-EN 25814, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemiska vattenundersökningar, SIS/TK 424.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 5814

October 2012

ICS 13.060.50

Supersedes EN 25814:1992

English Version

Water quality - Determination of dissolved oxygen - Electrochemical probe method (ISO 5814:2012)

Qualité de l'eau - Dosage de l'oxygène dissous - Méthode électrochimique à la sonde (ISO 5814:2012)

Wasserbeschaffenheit - Bestimmung des gelösten Sauerstoffs - Elektrochemisches Verfahren (ISO 5814:2012)

This European Standard was approved by CEN on 4 August 2012.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Foreword

This document (EN ISO 5814:2012) has been prepared by Technical Committee ISO/TC 147 “Water quality” in collaboration with Technical Committee CEN/TC 230 “Water analysis” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2013, and conflicting national standards shall be withdrawn at the latest by April 2013.

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

This document supersedes EN 25814:1992.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 5814:2012 has been approved by CEN as a EN ISO 5814:2012 without any modification.

Water quality — Determination of dissolved oxygen — Electrochemical probe method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies an electrochemical method for the determination of dissolved oxygen in water by means of an electrochemical cell which is isolated from the sample by a gas permeable membrane.

Measurement can be made either as a concentration of oxygen in milligrams per litre, percentage saturation (% dissolved oxygen) or both. The method measures oxygen in water corresponding to 1 % to 100 % saturation. However, most instruments permit measurement of values higher than 100 %, i.e. supersaturation.

NOTE Supersaturation is possible when the partial pressure of oxygen is higher than in air. Especially when strong algal growth is present, supersaturation of up to 200 % and above can occur.

The method measures oxygen in water with a saturation higher than 100 %, when special arrangements to prevent the outgassing of oxygen during the handling and measurement of the sample are made.

The method is suitable for measurements made in the field and for continuous monitoring of dissolved oxygen, as well as measurements made in the laboratory. It is the preferred method for highly coloured and turbid waters, and also for analysis of waters not suitable for the Winkler titration method because of iron- and iodine-fixing substances, which can interfere in the iodometric method specified in ISO 5813[1].

The method is suitable for drinking waters, natural waters, waste waters, and saline waters. If used for saline waters, such as sea or estuarine waters, a correction for salinity is essential.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

Immersion of a probe, consisting of a cell enclosed by a selective membrane and containing the electrolyte and at least two metallic electrodes, in the water to be analysed.

NOTE The membrane is effectively impermeable to water and ionic dissolved matter, but is permeable to oxygen and a certain number of other gases.

One of the electrodes is made of a noble metal like gold or platinum. Oxygen is reduced at its surface by an electrochemical process. In order to make this process possible, a suitable electrochemical potential is established at this electrode. For polarographic probes, this is achieved by applying an external voltage related to a second electrode. Galvanic probes are able to build up the potential by themselves.

The current resulting from the reduction of oxygen is directly proportional to the rate of transport of oxygen through the membrane and the layer of electrolyte, and hence to the partial pressure of the oxygen in the sample at a given temperature.

Temperature has two different influences. The first relates to the variation of gas permeability of the membrane with temperature. So the primary signal of the probe has to be compensated with a built-in temperature sensor. Meters manufactured recently are able to do this automatically. The second is the temperature effect on the electrode reactions.

To calculate the percentage of saturation of samples in contact with an atmosphere, it is necessary to include the effective pressure. This can be performed manually or by implementing a pressure sensor for automatic compensation. Salinity can also be an influence.

4 Interferences

Gases and vapors such as chlorine, hydrogen sulfide, amines, ammonia, bromine, and iodine which diffuse through the membrane can interfere, if present, by affecting the measured current.

Other substances present in the sample can interfere with the measured current by causing obstruction, deterioration of the membrane or corrosion of the electrodes. These include solvents, oils, sulfides, carbonates, and biofilms.

5 Reagents

During analysis, use only reagents of recognized analytical grade.

5.1 **Water**, grade 2, as specified in ISO 3696, optionally from commercial sources.

5.2 **Sodium sulfite**, anhydrous, Na_2SO_3 or heptahydrate, $Na_2SO_3 \cdot 7H_2O$.

5.3 **Cobalt(II) salt**, for example cobalt(II) chloride hexahydrate, $CoCl_2 \cdot 6H_2O$.

5.4 **Nitrogen gas,** N_2, purity 99,995 % volume fraction or better.

6 Apparatus

6.1 **Measuring instrument**, comprising the components specified in 6.1.1 and 6.1.2.

6.1.1 **Measuring probe**, either of the galvanic type (e.g. lead/silver) or the polarographic type (e.g. silver/gold) with, if required, a temperature-sensitive compensating device.

6.1.2 **Meter**, graduated to show the concentrations of dissolved oxygen directly, and/or percentage saturation with oxygen.

6.2 **Thermometer**, graduated in at least 0,5 °C divisions.

NOTE Commonly a temperature sensor is integrated into the instrument (6.1).

6.3 **Barometer**, graduated to 1 hPa.

NOTE Usually the barometer is integrated into the instrument (6.1).

7 Sampling and analysis procedure

7.1 Sampling

7.1.1 General

Samples should always be handled so that transfer of oxygen between water sample and air is inhibited.

As a matter of principle, the oxygen concentration shall be measured directly on site in the water body to be analysed.

If direct measurement in the water body is not possible, the measurement can also be taken in a gas-tight connected flow-through device or immediately after sampling as a discrete sample.

Any discrete sampling procedure results in a higher measurement uncertainty.

While filling the sample vessel during sampling, oxygen uptake or oxygen stripping shall be minimized. Sample transfer shall occur without any turbulence, i.e. by maintaining a laminar flow.

7.1.2 Dip-sampling (e.g. surface waters)

Take the sample by carefully and slowly dipping the sample vessel.

7.1.3 Sampling from taps

Connect an inert sampling tube, in a gas-tight fashion, to the tap and insert the sampling tube all the way down to the bottom of the sampling vessel. Ensure that the volume of water allowed to overflow is at least three times the capacity of the vessel.

7.1.4 Sampling with pumps

Only water-displacing submersible pumps should be used. Pumps that function according to the principle of air displacement are *not* suitable. Fill the sample vessel from the bottom, using a sampling tube, and allow the water to overflow. During sample transfer, the volume flow rate shall be controlled in order to guarantee a mainly laminar flow. Ensure that the volume of water allowed to overflow is at least three times the capacity of the vessel.

7.2 Measuring technique and precautions to be taken

The measuring system shall be in a proper state as specified in the manufacturer's instructions. For example:

— ensure the membrane is not damaged;

— allow an adequate polarization time;

— calibrate the system when necessary.

When a measurement is performed, ensure that the sample flows past the membrane with sufficient velocity according to the manufacturer's instructions. This can be achieved by natural streaming, movement of the sensor or stirring, e.g. with a magnetic stirrer. This is necessary to prevent loss of signal because of consumption of oxygen by the sensor.

Take care that there is no exchange of oxygen from a gas reservoir to the sample or vice versa. Therefore, avoid formation of any air bubbles in the samples that are measured in a vessel. When measuring on-site, do not generate any air bubbles, which may affect the signal.

For storing and maintenance of the probe, consult the manufacturer's instructions.