SLOVENSKI STANDARD
SIST EN 25813:1996

01-oktober-1996

Kakovost vode - Ugotavljanja raztopljenega kisika - Jodometrijska metoda (ISO 5813:1983)

Water quality - Determination of dissolved oxygen - Iodometric method (ISO 5813:1983)

Wasserbeschaffenheit - Bestimmung des gelösten Sauerstoffs - Iodometrisches Verfahren (ISO 5813:1983)

Qualité de l'eau - Dosage de l'oxygène dissous - Méthode iodométrique (ISO 5813:1983)

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ICS:

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EUROPÄISCHE NORM

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English version

Water quality - Determination of dissolved oxygen - Iodometric method (ISO 5813:1983)

Qualité de l' eau - Dosage de l'oxygène dissous - Méthode iodométrique (ISO 5813:1983)

Wasserbeschaffenheit - Bestimmung des gelösten Sauerstoffs - Iodometrisches Verfahren (ISO 5813:1983)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist 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 Central Secretariat has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

This European Standard is the endorsement of ISO 5813. Endorsement of ISO 5813 was recommended by CEN/Technical Committee 230 "Water analysis" under whose competence this European Standard will henceforth fall.

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of an identical text or by endorsement, at the latest by April 1993, and conflicting national standards shall be withdrawn at the latest by April 1993.

The Standard was approved and in accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard : Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Endorsement notice

The text of the International Standard ISO 5813:1983 was approved by CEN as a European Standard without any modification.

International Standard ISO 5813

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Determination of dissolved oxygen — Iodometric method

Qualité de l'eau — Dosage de l'oxygène dissous — Méthode iodométrique

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5813 was developed by Technical Committee ISO/TC 147, *Water quality*, and was circulated to the member bodies in July 1982.

It has been approved by the member bodies of the following countries:

Australia
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China
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Sweden
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Thailand
United Kingdom
USA
USSR

No member body expressed disapproval of the document.

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Water quality — Determination of dissolved oxygen — Iodometric method

1 Scope and field of application

This International Standard specifies an iodometric method for the determination of dissolved oxygen in water by the so-called "Winkler procedure" modified in order to make allowance for certain interferences.

The iodometric method is the reference method for the determination of dissolved oxygen in water. It is applicable to all types of water having dissolved oxygen concentrations greater than 0,2 mg/l, up to double saturation of oxygen (approximately 20 mg/l), which are free from interfering substances. Readily oxidizable organic substances such as tannins, humic acid and lignins, interfere. Oxidizable sulphur compounds such as sulphides and thiourea also interfere, as do actively respiring systems which readily consume oxygen. In the presence of such substances, it is preferable to use the electrochemical probe method specified in ISO 5814.

Nitrites up to a concentration of 15 mg/l do not interfere with the determination because they are destroyed by the addition of sodium azide.

If oxidizing or reducing substances are present, it is necessary to make modifications to the method; these are described in clause 9.

If suspended matter, capable of fixing or consuming iodine, is present, the method may be used with the modification described in the annex, but it is preferable to use the electrochemical probe method.

2 Reference

ISO 5814, *Water quality — Determination of dissolved oxygen — Electrochemical probe method.*[1]

3 Principle

Reaction of dissolved oxygen in the sample with freshly precipitated manganese(II) hydroxide [formed by the addition of sodium or potassium hydroxide to manganese(II) sulphate]. Acidification, and oxidation of iodide by the higher valency manganese compound so formed, liberating an equivalent quantity of iodine. Determination of the quantity of iodine liberated by titration with sodium thiosulphate.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Sulphuric acid, solution.[2]

Cautiously add 500 ml of concentrated sulphuric acid (ϱ = 1,84 g/ml) to 500 ml water, stirring continuously.

4.2 Sulphuric acid, solution, $c(1/2\ H_2SO_4)$ = 2 mol/l.

4.3 Alkaline iodide-azide reagent.

WARNING — Sodium azide is an extremely strong poison. If nitrites are known to be absent, this reagent may be omitted.

Dissolve 35 g of sodium hydroxide (NaOH) [or 50 g of potassium hydroxide (KOH)] and 30 g of potassium iodide (KI) [or 27 g of sodium iodide (NaI)] in approximately 50 ml of water.

Dissolve separately 1 g of sodium azide (NaN_3) in a few millilitres of water.

Mix the two solutions and dilute to 100 ml.

Store the solution in a stoppered, brown glass flask.

After dilution and acidification, this reagent should not show any colour in the presence of the indicator solution (4.7).

4.4 Manganese(II) sulphate anhydrous, 340 g/l solution (or **manganese sulphate monohydrate,** 380 g/l solution).

Alternatively, use manganese(II) chloride tetrahydrate, 450 g/l solution.

Filter any solution which is not clear.

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[1] At present at the stage of draft.

[2] If the presence of trivalent iron is suspected, use phosphoric acid (H_3PO_4), ϱ = 1,70 g/ml.

4.5 Potassium iodate, $c(1/6\ KIO_3)$ = 10 mmol/l, standard solution.

Dry a few grams of potassium iodate (KIO_3) at 180 °C. Weigh 3,567 ± 0,003 g and dissolve in water. Dilute to 1 000 ml.

Withdraw 100 ml and dilute with water to 1 000 ml in a volumetric flask.

4.6 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3) \approx$ 10 mmol/l.

4.6.1 Preparation

Dissolve 2,5 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) in freshly boiled and cooled water. Add 0,4 g of sodium hydroxide (NaOH) and dilute to 1 000 ml.

Store the solution in a dark glass bottle.

4.6.2 Standardization

Dissolve, in a conical flask, approximately 0,5 g of potassium or sodium iodide (KI or NaI) in 100 to 150 ml water. Add 5 ml of 2 mol/l sulphuric acid solution (4.2).

Mix and add 20,00 ml of the standard potassium iodate solution (4.5). Dilute to about 200 ml and immediately titrate the liberated iodine with the sodium thiosulphate solution, adding the indicator solution (4.7) towards the end of the titration, when a pale straw colour is reached, and then titrating until complete decoloration.

The concentration c, expressed in millimoles per litre, is given by the equation

$$c = \frac{6 \times 20 \times 1{,}66}{V}$$

where V is the volume, in millilitres, of sodium thiosulphate solution used for the titration.

Standardize the solution daily.

4.7 Starch, freshly prepared 10 g/l solution.

NOTE — Other suitable indicators may be used.

4.8 Phenolphthalein, 1 g/l ethanolic solution.

4.9 Iodine, approximately 0,005 mol/l solution.

Dissolve 4 to 5 g of potassium or sodium iodide in a little water and add approximately 130 mg of iodine. After dissolution of the iodine, dilute to 100 ml.

4.10 Potassium iodide or **sodium iodide.**

5 Apparatus

Ordinary laboratory equipment, and

5.1 Narrow-mouthed glass flasks, of capacity between 130 and 350 ml, calibrated to the nearest 1 ml, with stoppers (Winkler flasks, or any other suitable flasks, preferably with straight shoulders). Each flask and its stopper shall bear the same identification number. The volume of each flask may be determined by weighing.

6 Procedure

6.1 In the presence of suspended matter capable of fixing or consuming iodine, or if in doubt about the presence of such matter, proceed as described in the annex or, preferably, determine dissolved oxygen by the electrochemical probe method specified in ISO 5814.

6.2 Check for the presence of oxidizing or reducing substances

If oxidizing or reducing agents can be expected to interfere with the results, collect 50 ml of the water to be analysed and neutralize it in the presence of 2 drops of the phenolphthalein solution (4.8). Add 0,5 ml of the sulphuric acid solution (4.2), a few crystals (of mass approximately 0,5 g) of the potassium or sodium iodide (4.10) and a few drops of the indicator solution (4.7).

If the solution turns blue, oxidizing substances are present.

If the solution stays colourless, add 0,2 ml of the iodine solution (4.9) and shake. Leave for 30 s. If no blue colour appears, reducing substances are present.[1)]

In the presence of oxidizing substances, proceed as specified in 9.1.

In the presence of reducing substances, proceed as specified in 9.2.

In the absence of oxidizing or reducing substances, proceed as specified in 6.3, 6.4 and 6.5.

6.3 Collection of samples

Unless it is essential to proceed otherwise, collect the sample in the flask (5.1) in which the determination is to be made.

The test sample consists of all the contents of the filled flask.

NOTE — In the presence of oxidizing or reducing substances, it is necessary to take a second test sample (see 9.1.2.1 and 9.2.3.1).

[1)] Further addition of iodine solution permits evaluation of the volume of sodium hypochlorite solution to be added in 9.2.3.

6.3.1 Sampling surface waters

Fill the flask (5.1) to overflowing, taking care to avoid any change in the concentration of dissolved oxygen.

For shallow waters, it is preferable to use the electrochemical probe method.

After elimination of any air bubbles that may be adhering to the glass, immediately fix the dissolved oxygen (see 6.4).

6.3.2 Sampling water from distribution pipelines

Connect a tube of inert material to the inlet and extend the tube outlet to the bottom of the flask (5.1).

Fill the flask by flushing it with a volume of water which is about ten times the volume of the flask. After elimination of any air bubbles that may be adhering to the glass, immediately fix the dissolved oxygen (see 6.4).

6.3.3 Sampling water at varying depths

Use a special sampler, containing the flask (5.1), equipped with a rubber inlet tube extending to the bottom of the flask.

The flask is filled by air displacement. Avoid turbulence. Certain types of samplers permit filling of several flasks at the same time.

6.4 Fixing of oxygen

After the sample has been taken, preferably on site, immediately add to the flask, containing the sample, 1 ml of the manganese(II) sulphate solution (4.4) and 2 ml of the alkaline reagent (4.3). Add reagents below the surface using narrow tipped pipettes. Replace the stopper carefully to avoid the inclusion of air bubbles.

If some other system is used, take the precautions necessary to ensure that the oxygen content of the sample is not modified.

Turn the flask upside down several times to mix the contents thoroughly. Allow the precipitate which is formed to settle for at least 5 min and then mix by inversion again to ensure that the mixture is homogeneous.

The flask may then be transported to the laboratory.

If protected from light, the sample may be stored for up to 24 h.

6.5 Liberation of iodine

Ensure that the precipitate which has been formed has settled so that it is in the lower third of the flask.

Add slowly 1,5 ml of the sulphuric acid solution (4.1) [or the corresponding volume of the phosphoric acid solution (see the footnote to 4.1)], stopper the flask, then shake until all the precipitate has been dissolved and the iodine is evenly distributed.

NOTE — If the titration is to be carried out directly in the flask, a corresponding portion of the clear, supernatant liquid should be cautiously siphoned off without whirling up the sediment.

6.6 Titration

Transfer the contents of the flask or of an aliquot portion (volume V_1) to a conical flask.

Titrate with the sodium thiosulphate solution (4.6), using either the starch solution (4.7), added near the end of the titration, or another suitable indicator.

7 Expression of results

The dissolved oxygen content, expressed in milligrams of oxygen per litre, is given by the formula

$$\frac{M_r V_2 c f_1}{4\ V_1}$$

where

M_r is the relative molecular mass of oxygen ($M_r = 32$);

V_1 is the volume, in millilitres, of the test sample or the aliquot portion ($V_1 = V_0$ if all the contents of the flask were titrated);

V_2 is the volume, in millilitres, of sodium thiosulphate solution (4.6) used to titrate the contents of the flask or the aliquot portion;

c is the actual concentration, expressed in millimoles per litre, of the sodium thiosulphate solution (4.6);

$$f_1 = \frac{V_0}{V_0 - V'}$$

where

V_0 is the volume, in millilitres, of the flask (5.1),

V' is the sum of the volumes of the manganese(II) sulphate solution (4.4) (1 ml) and the alkaline reagent (4.3) (2 ml).

Report the result to one decimal place.

8 Reproducibility

Replicate determinations, with 10 degrees of freedom, of dissolved oxygen in air-saturated water (range 8,5 to 9 mg/l), carried out in four separate laboratories, gave within batch standard deviations between 0,03 and 0,05 mg of dissolved oxygen per litre.