International Standard



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Water quality — Determination of dissolved oxygen — lodometric method

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

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Foreword

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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.

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

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

Australia Austria Belgium Canada Chile China Czechoslovakia Denmark Egypt, Arab Rep. of Finland France Germany, F.R. Hungary India Iran Iraq Italy Japan Korea, Rep. of Mexico Netherlands Norway Poland Romania South Africa: Rep. of Spain Sweden Switzerland United Kingdom USA USSR

No member body expressed disapproval of the document.

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Water quality — Determination of dissolved oxygen lodometric 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 tarting, 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 ($\rho = 1.84$ g/ml) to 500 ml water, stirring continuously.

- **4.2** Sulphuric acid, solution, $c(1/2 H_2SO_4) = 2 \text{ mol/l}$.
- 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 (NaN₃) 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.

¹⁾ At present at the stage of draft.

²⁾ If the presence of trivalent iron is suspected, use phosphoric acid (H_3PO_4), $\rho = 1,70$ g/ml.