

Methods of testing

Water used in industry —

Part 101: Dissolved oxygen

IMPORTANT NOTE. It is essential that this Part be read in conjunction with the information in Part 100 of this standard, “Foreword, scope and general requirements”, which is published separately.

UDC 628.1:663.63.01:543.371

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW

BSI
Documents

AVA

Document Sharing Hub

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Environment and Pollution Standards Committee, was published under the authority of the Board of BSI and comes into effect on 29 February 1984

© BSI 03-2000

The Committees responsible for this British Standard are shown in Part 100.
The following BSI references relate to the work on this standard:
Committee reference EPC/37/1
Draft for comment 77/50234 DC

ISBN 0 580 13505 5

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.
This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

	Page
0 Introduction	1
1 Scope	1
2 Iodometric titration (manganese hydroxide precipitation) method	1
3 Indigo-carmin Nessleriser method	2
4 Amperometric (manganese (II) hydroxide precipitation) method	5
Appendix A Colours of dissolved oxygen disk	12
Figure 1 — Modified Nessler cylinder	4
Figure 2 — Sampling vessels	7
Figure 3 — Platinum/platinum electrode system	9
Figure 4 — Titration assembly	10
Table 1 — Colours of dissolved oxygen disk	12
Publications referred to	Inside back cover

0 Introduction

BS 2690-101, BS 2690-102 and BS 2690-103¹⁾ together supersede BS 2690-2:1965. This Part supersedes the clauses concerned with determination of dissolved oxygen.

1 Scope

The three methods described are for the determination of dissolved oxygen in water for industrial use.

NOTE The titles of the publications referred to in this standard are given on the inside back cover.

2 Iodometric titration (manganese hydroxide precipitation) method

2.1 Range. From 0.02 mL/L or 0.03 mg/L up to supersaturation in up to a 250 mL test portion.

2.2 Principle. The dissolved oxygen in the sample is fixed by the addition of hypochlorite, alkaline iodide solution and manganese (II) hydroxide until the sample can be analyzed in the laboratory. The iodine released from the alkaline iodide solution by the dissolved oxygen is then titrated with standard volumetric sodium thiosulphate solution, using starch as indicator.

NOTE The method is applicable in the presence of nitrites, iron salts, dissolved or suspended organic matter, thiosulphate, sulphites, sulphides or residual chlorine.

2.3 Interferences

2.3.1 General. A small number of substances have been found to interfere with the determination. These are given in 2.3.2 to 2.3.5, together with modifications to the procedure to overcome them.

2.3.2 Residual chlorine and chloramines. If the water contains residual chlorine and chloramines, the addition of the sodium hypochlorite solution (2.4.1) may be omitted. In any case, no more hypochlorite than is necessary should be added and, with an unknown water, it is desirable first to test the blank and then to adjust the strength of the hypochlorite solution so that a small positive reading is obtained.

2.3.3 Dextrose, peptones and phenols. Dextrose, peptones and phenols are capable of direct oxidation by dissolved oxygen in alkaline solution and, if their presence is suspected, the manganese (II) sulphate solution (2.4.3) should be added to the test portion before the alkaline iodide solution (2.4.2) so as to remove dissolved oxygen before it can react. The addition of the alkaline iodide solution (2.4.2) to the blank should then be followed immediately by the addition of the acid/azide solution (2.4.4).

2.3.4 Manganese. Traces of manganese present in the blank will be oxidized by the excess sodium hypochlorite solution (2.4.1) rather than by dissolved oxygen. If larger amounts are present, the acid/azide solution (2.4.4) should be added to the blank before the alkaline iodide solution (2.4.2) in order to avoid the formation of a precipitate of manganese hydroxide.

2.3.5 Sea water. 5 mL of 500 mL/L sulphuric acid solution and 2.5 mL of the alkaline iodide solution (2.4.2) should be used to precipitate the magnesium hardness.

2.4 Reagents

2.4.1 Sodium hypochlorite solution. Dilute for use as required to approximately 4 g/L of available chlorine.

2.4.2 Alkaline iodide solution. Dissolve 700 g of potassium hydroxide in approximately 500 mL of water, stirring well, and cool. Dissolve 150 g of potassium iodide in approximately 150 mL of water. Mix together and dilute to 1 000 mL.

2.4.3 Manganese (II) sulphate solution. Dissolve 480 g of manganese (II) sulphate tetrahydrate, ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$), in water and dilute to 1 000 mL.

2.4.4 Acid/azide solution. Dissolve 4 g of sodium azide in approximately 55 mL of water and dilute to 1 L with orthophosphoric acid, 90 % (*m/m*).

CAUTION. Prepare this solution in a fume cupboard because toxic fumes of hydrazoic acid may be evolved.

2.4.5 Potassium iodate, 0.892 g/L standard volumetric solution²⁾. Weigh out exactly 0.892 g of potassium iodate, dried at 100 °C to 110 °C. Transfer to approximately 100 mL of water in a 250 mL beaker and, when dissolved, transfer the solution to a 1 000 mL one-mark volumetric flask. Wash the beaker several times with water, adding the washings to the flask, and dilute to the mark with water.

¹⁾ Part 103 is in course of preparation.

²⁾ Previously 0.025N

2.4.6 Sodium thiosulphate, 6.20 g/L solution.

2.4.6.1 Preparation. Weigh 6.20 g of sodium thiosulphate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). Transfer to 200 mL of water to which has been added approximately 0.2 g of sodium hydroxide, either as solid or as an equivalent volume of solution (e.g. 50 mL of 4 g/L solution) in a 400 mL beaker. When dissolved, transfer the solution to a 1 000 mL one-mark volumetric flask. Wash the beaker several times with water, adding the washings to the flask, and dilute to the mark with water. Mix well and standardize frequently (see 2.4.6.2).

2.4.6.2 Standardization. Pour 250 mL of water into a porcelain basin containing approximately 3 g of potassium iodide. Add 2 mL of 500 mL/L sulphuric acid solution. Introduce 10.0 mL of the potassium iodate solution (2.4.5) from a pipette or burette, and stir well. Titrate the solution with the sodium thiosulphate solution (2.4.6), stirring all the time, until the brown colour fades to a pale straw. Add approximately 1 mL of the starch solution (2.4.7) and continue the titration, with stirring, until the solution becomes colourless. If the volume of sodium thiosulphate solution required is V mL, the factor (F) for the sodium thiosulphate solution is $10/V$.

2.4.7 Starch solution. Mix 1 g of soluble starch and 0.2 g of sodium hydroxide (or 5 mL of a 40 g/L solution) with 15 mL of water. When dissolved, add 180 mL of water.

NOTE A proprietary indicator for iodine may be used.

2.5 Apparatus

2.5.1 Two 250 mL sample bottles with sloping shoulders, matched for equal size to within 1 % and fitted with ground glass stoppers which can be inserted without entrapping air bubbles.

2.6 Procedure. Take two 250 mL representative samples of the water to be tested, designating one the test portion and the other the blank. If the water is believed to be very low in dissolved oxygen, it is preferable to take the samples by the submerged bottle technique described in BS 1328, in which the bottle is placed in a large container into which the water to be tested overflows, sealing the bottle from direct access to the air; the reagents should be added beneath about 25 mm of sealing liquid. If using an immersion flushing sample collector (see Figure 4 of BS 1328:1969) to obtain a sample at some depth in a body of water, the blank may be filled with the water in the body of the collector.

Add 1 mL of the sodium hypochlorite solution (2.4.1) to the test portion, replace the stopper and mix well. Repeat with the blank. Add 1 mL of the alkaline iodide solution (2.4.2) to the test portion, replace the stopper and mix well. Repeat with the blank. Add 1 mL of the manganese (II) sulphate solution (2.4.3) to the test portion, replace the stopper and mix well. At this stage, if necessary, the bottles may be left for some hours and taken back to a base laboratory for completion of the determination. Add 4 mL of the acid/azide solution (2.4.4) to both bottles and mix well. Finally, add 1 mL of the manganese (II) sulphate solution (2.4.3) to the blank. If either solution is warm, cool to approximately 20 °C by immersion in cold water.

Titrate the test portion and blank, or portions thereof, with the standardized sodium thiosulphate solution (2.4.6) using the starch solution (2.4.7) as indicator.

2.7 Calculation. Dissolved oxygen, mg/L of the test sample = $200 \times (V_1 - V_2)/V_3$.

where

- V_1 is the volume of the sodium thiosulphate solution used for the titration of the test portion (in mL);
- V_2 is the volume of the sodium thiosulphate solution used for the titration of the blank (in mL);
- V_3 is the volume of the test portion (in mL).

3 Indigo-carmin Nessleriser method

3.1 Range. From 0.005 mL/L or 0.007 mg/L up to 0.12 mL/L or 0.17 mg/L.

3.2 Principle. The colours produced by indigo-carmin which has been reduced with glucose are compared with a series of standard colours. These range through orange, pink and mauve to blue, with increasing oxygen content.