International Standard ISO 8467

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Water quality — Determination of permanganate index

Qualité de l'eau — Détermination de l'indice de permanganate

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Water quality — Determination of permanganate index

0 Introduction

The permanganate index is a conventional measure of the contamination by organic and oxidizable inorganic matter in a water sample. It is primarily intended for judging the quality of potable water and raw waters, such as well and surface waters. More heavily contaminated waters may be analysed provided an appropriate pre-dilution step is adopted. The permanganate index can be determined for waters containing less than 500 mg/l of chloride ion. Reducing compounds such as iron(II) salts, nitrites or hydrogen sulfide may contribute to the permanganate index but are not classified as impurities.

The permanganate index cannot be considered as a measure of the theoretical oxygen demand or the total content of organic matter. Many organic compounds are only partially oxidized in this test as oxidation is generally incomplete. Volatile matter that evaporates before the addition of permanganate will not be included.

The method is not recommended for determining organic load in waste waters; for this purpose the chemical oxygen demand should be determined [see ISO 6060, *Water quality — Determination of the chemical oxygen demand*].

The method is simple and convenient for surveying the quality of large numbers of water samples.

1 Scope and field of application

This International Standard specifies a method for the determination of the permanganate index of water. It is primarily intended for water for human consumption and domestic use. It is applicable to waters having a chloride ion concentration of less than 500 mg/l. Samples having a permanganate index over 10 mg/l should be diluted before analysis. The lower limit of the optimum range of the test is 2,5 mg/l.

2 Definition

permanganate index (of water): The mass concentration of oxygen equivalent to the amount of permanganate ion consumed when a water sample is treated with that oxidant under defined conditions.

3 Principle

Heating of a sample in a boiling water-bath with a known amount of potassium permanganate and sulfuric acid for a fixed period of time (10 min), reduction of part of the permanganate by oxidizable material in the sample and determination of the consumed permanganate by addition of an excess of oxalate solution followed by titration with permanganate.

NOTE — The suggested maximum permanganate index of 10 mg/l is equivalent to a consumption of approximately 60 % of the added permanganate by the non-diluted sample.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. Do not use deionized water from an organic ion exchanger.

NOTE — Non-reducing water can be prepared as follows. Add 10 ml of sulfuric acid (4.2) and a small excess of the potassium permanganate stock solution (4.5) to 1 litre of distilled water. Distil in an all-glass apparatus and discard the first 100 ml of distillate. Store in a glass bottle with a glass stopper.

4.1 Sulfuric acid, $c(H_2SO_4) = 7,5$ mol/l.

Add slowly and with continuous stirring 420 ml of 18 mol/l sulfuric acid ($\varrho = 1,84$ g/ml) to 500 ml of water. Allow to cool and dilute to 1 litre.

4.2 Sulfuric acid, $c(H_2SO_4) = 2$ mol/l.

Add slowly and with continuous stirring 110 ml of 18 mol/l sulfuric acid ($\varrho = 1,84$ g/ml) to about 500 ml of water. Add slowly potassium permanganate solution (4.6) until a faint pink colour persists. Allow to cool, dilute with water to 1 litre and mix.

4.3 Sodium oxalate, stock solution, $c(Na_2C_2O_4) = 0,05$ mol/l.

Dry sodium oxalate at 120 °C for 2 h. Dissolve 6,700 g of the dried solid in water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

This solution is stable for 6 months if stored in a dark place.

4.4 Sodium oxalate, standard volumetric solution, $c(Na_2C_2O_4) = 5$ mmol/l.

Introduce, by means of a pipette, 100 ± 0,25 ml of the sodium oxalate stock solution (4.3) into a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix.

This standard volumetric solution is stable for 2 weeks.

NOTE — Commercially available ready-made solutions may be used.

4.5 Potassium permanganate, stock solution, $c(KMnO_4) \approx 20$ mmol/l.

Dissolve about 3,2 g of potassium permanganate in water and make up to 1 000 ml. Heat the solution to 90 to 95 °C for 2 h, cool and store for not less than 2 days. Decant the clear solution and store in a dark bottle.

4.6 Potassium permanganate, volumetric solution, $c(KMnO_4) \approx 2$ mmol/l.

Introduce, by means of a pipette, 100 ml of the stock solution (4.5) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

This volumetric solution is relatively stable for several months if stored in the dark. The procedure described in 7.5 automatically allows for its exact concentration to be included in the calculation in 8.1.

5 Apparatus

Usual laboratory apparatus, and

5.1 Water-bath, with a rack for test tubes, of sufficient capacity and power to ensure that the solutions in all test tubes quickly reach and are maintained at a temperature between 96 and 98 °C during both the initial heating and the reaction stages (clause 11).

5.2 Test tubes, of length 150 to 200 mm, and diameter 25 to 35 mm. Keep the test tubes exclusively for the determination of permanganate index.

Clean new test tubes by heating with acidified permanganate solution. This shall be checked by performing blank determinations until the values are adequately low and constant.

A blank value V_0 should typically not exceed 0,3 ml.

5.3 Burette, of capacity 10 ml, preferably of the piston type, graduated in divisions of 0,02 ml, and conforming to the requirements of ISO 385/1.

6 Sampling and samples

Immediately after receipt in the laboratory, add 5 ml of sulfuric acid (4.1) per litre of sample if this has not been done at the sampling in the field, regardless of whether the sample is to be stored before analysis.

Analyse the samples as soon as possible and not later than 2 days after sampling. Keep them in the dark at 0 to 5 °C if the storage time exceeds 6 h.

Shake the storage bottles and make sure that their contents are well homogenized when withdrawing a test portion for analysis.

7 Procedure

7.1 Check that all flasks and test tubes (5.2) used in the procedure are perfectly clean (see 5.2).

7.2 Dilute samples having a high permanganate index so that the results for the diluted samples fall within the range 2,5 to 10 mg/l.

7.3 Transfer, by means of a pipette, 25,0 ± 0,25 ml of the test sample (or the diluted test sample) to a test tube. Add 5 ± 0,5 ml of sulfuric acid (4.2) and mix by swirling gently.

Place the test tube in the boiling water-bath (5.1) for 10 ± 2 min.

Add 5 ± 0,05 ml of potassium permanganate volumetric solution (4.6) and commence timing.

After 10 min ± 15 s add 5 ± 0,05 ml of the sodium oxalate standard volumetric solution (4.4) and wait until the solution is colourless. Titrate, whilst hot, with potassium permanganate volumetric solution (4.6) to a faint pink colour which persists for about 30 s. Note the volume of permanganate solution consumed, as V_1.

7.4 Carry out a blank test in parallel with the determination, by the same procedure, but replacing the test portion with 25 ml of water. Note the volume of permanganate solution consumed, as V_0.

Retain the titrated solution for standardization of the potassium permanganate volumetric solution (4.6) as described in 7.5.

7.5 To the titrated solution retained from the blank test (7.4), add 5,00 ± 0,05 ml of the sodium oxalate standard volumetric solution (4.4). Reheat the solution, if necessary, to about 80 °C and titrate with potassium permanganate volumetric solution (4.6) until the appearance of a pink colour which persists for about 30 s. Note the volume of permanganate solution consumed, as V_2.

NOTE — It is good practice to leave the titrated solutions in the test tubes until they are required for the next determination of permanganate index.

8 Expression of results

8.1 Calculation

The permanganate index, expressed in milligrams of oxygen per litre, is given by the expression