International Standard 6333

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Water quality — Determination of manganese — Formaldoxime spectrometric method

Qualité de l'eau — Dosage du manganèse — Méthode spectrométrique à la formaldoxime

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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 manganese — Formaldoxime spectrometric method

0 Introduction

In water containing oxygen, most of the manganese will be present as undissolved forms, often associated with micro-organisms and as complexes with, for example, humic acid. If the water is free of oxygen or strongly acidic, all manganese will be present in dissolved forms.

1 Scope and field of application

This International Standard specifies a formaldoxime spectrometric method for the determination of total manganese (including dissolved, suspended and organically bound manganese) in surface and drinking water.

The method is applicable to the determination of manganese concentrations between 0,01 mg/l and 5 mg/l. Manganese concentrations above 5 mg/l may be determined after suitable dilution of the sample.

For known possible interferences, see clause 8.

NOTE — This method is not applicable to highly contaminated waters such as industrial waste water.

2 Principle

Addition of a formaldoxime solution to a test portion and spectrometric measurement of the orange-red complex at a wavelength of about 450 nm.

If suspended or organically bound manganese is present, pretreatment is required to convert manganese to forms capable of reacting with formaldoxime.

The manganese formaldoxime complex is stable between pH values of 9,5 and 10,5, and the intensity of the colour produced is proportional to the amount of manganese present. The relationship between concentration and absorbance is linear up to a concentration of 5 mg/l. Maximum absorbance occurs at about 450 nm (specific molar absorbance coefficient of 11×10^3 l/mol·cm).

3 Reagents

WARNING — The reagents described in 3.4, 3.5.1 and 3.5.3 should be regarded as special hazards. Hazardous operations should be carried out in a fume cupboard. Care must be taken to avoid ingestion or inhalation of vapours and to protect the hands, eyes and face. Gloves and goggles must be worn and any suspected skin contamination washed off immediately. Inhalation of the vapours of formaldehyde and formaldoxime will result in severe irritation and oedema of the upper respiratory tract.

During the analysis, use only reagents of recognized analytical grade, and only deionized water or water distilled from an all-glass apparatus with a manganese content that is as low as possible.

3.1 Oxidizing reagent.

Either potassium peroxodisulfate ($K_2S_2O_8$) or sodium peroxodisulfate ($Na_2S_2O_8$).

3.2 Sodium sulfite (Na_2SO_3), anhydrous.

3.3 EDTA, tetrasodium salt, solution, c(EDTA) = 0,24 mol/l.

Dissolve 90 g of disodium ethylenedinitrilotetraacetic acid (Na_2EDTA) dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$) and 19 g of sodium hydroxide (NaOH) in water and dilute to 1 000 ml.

Alternatively, dissolve 109 g of tetrasodium ethylenedinitrilotetraacetic acid (Na_4EDTA) tetrahydrate ($C_{10}H_{12}N_2Na_4O_8 \cdot 4H_2O$) or 100 g of tetrasodium ethylenedinitrilotetraacetic acid dihydrate ($C_{10}H_{12}N_2Na_4O_8 \cdot 2H_2O$) in water and dilute to 1 000 ml.

3.4 Formaldoxime solution.

Dissolve 10 g of hydroxylammonium chloride (NH_3OHCl) in about 50 ml of water. Add 5 ml of 35 % (m/m) methanal (HCHO) (formaldehyde) solution (ϱ = 1,08 g/ml) and dilute with water to 100 ml.

Keep the bottle in a dark and cool place. The solution has a shelf-life of at least 1 month.

3.5 Hydroxylammonium chloride/ammonia solution.

3.5.1 Hydroxylammonium chloride solution, $c(NH_3OHCl)$ = 6 mol/l.

Dissolve 42 g of hydroxylammonium chloride in water and dilute to 100 ml.