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Water quality — Determination of nitrate —

Part 3 :

Spectrometric method using sulfosalicylic acid

Qualité de l'eau — Dosage des nitrates —

Partie 3 : Méthode spectrométrique avec l'acide sulfosalicylique

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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 preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 7890-3 was prepared by Technical Committee ISO/TC 147, *Water quality*.

ISO 7890 consists of the following parts, under the general title *Water quality — Determination of nitrate* :

- *Part 1 : 2,6-Dimethylphenol spectrometric method*
- *Part 2 : 4-Fluorophenol spectrometric method after distillation*
- *Part 3 : Spectrometric method using sulfosalicylic acid*

Annex A forms an integral part of this International Standard.

Water quality — Determination of nitrate —

Part 3 :

Spectrometric method using sulfosalicylic acid

1 Scope

1.1 Substance determined

This part of ISO 7890 specifies a method for the determination of nitrate ion in water.

1.2 Type of sample

The method is suitable for application to raw and potable water samples.

1.3 Range

Up to a nitrate nitrogen concentration, ρ_N of 0,2 mg/l using the maximum test portion volume of 25 ml. The range can be extended upwards by taking smaller test portions.

1.4 Limit of detection¹⁾

Using cells of optical path length 40 mm and a 25 ml test portion volume the limit of detection lies within the range $\rho_N = 0,003$ to 0,013 mg/l.

1.5 Sensitivity¹⁾

A nitrate nitrogen concentration of $\rho_N = 0,2$ mg/l gives an absorbance of about 0,68 unit, using a 25 ml test portion and cells of optical path length 40 mm.

1.6 Interferences

A range of substances often encountered in water samples has been tested for possible interference with this method. Full details are given in annex A. The main potential interferents are chloride, orthophosphate, magnesium and manganese(II), as shown in annex A.

Other tests have shown that this method will tolerate a sample colour of up to 150 mg/l Pt providing the test portion absorption correction procedure is followed. (See 6.5.)

2 Principle

Spectrometric measurement of the yellow compound formed by reaction of sulfosalicylic acid (formed by addition to the

sample of sodium salicylate and sulfuric acid) with nitrate and subsequent treatment with alkali.

Disodium dihydrogen ethylenedinitrilotetraacetate (EDTANa_2) is added with the alkali to prevent precipitation of calcium and magnesium salts. Sodium azide is added to overcome interference from nitrite.

3 Reagents

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

3.1 Sulfuric acid, $c(\text{H}_2\text{SO}_4) \approx 18$ mol/l, $\rho = 1,84$ g/ml.

WARNING — When using this reagent, eye protection and protective clothing are essential.

3.2 Glacial acetic acid, $c(\text{CH}_3\text{COOH}) \approx 17$ mol/l, $\rho = 1,05$ g/ml.

WARNING — When using this reagent, eye protection and protective clothing are essential.

3.3 Alkali solution, $\rho_{\text{NaOH}} = 200$ g/l, $\rho[\text{CH}_2\text{-N}(\text{CH}_2\text{COOH})\text{CH}_2\text{-COONa}]_2 \cdot 2\text{H}_2\text{O} = 50$ g/l.

Cautiously dissolve $200 \text{ g} \pm 2 \text{ g}$ of sodium hydroxide pellets in about 800 ml of water. Add $50 \text{ g} \pm 0,5 \text{ g}$ of disodium dihydrogen ethylenedinitrilotetraacetate dihydrate (EDTANa_2) $\{[\text{CH}_2\text{-N}(\text{CH}_2\text{COOH})\text{CH}_2\text{-COONa}]_2 \cdot 2\text{H}_2\text{O}\}$ and dissolve. Cool to room temperature and make up to 1 litre with water in a measuring cylinder. Store in a polyethylene bottle. This reagent is stable indefinitely.

WARNING — When using this reagent, eye protection and protective clothing are essential.

3.4 Sodium azide solution, $\rho_{\text{NaN}_3} = 0,5$ g/l.

Carefully dissolve $0,05 \text{ g} \pm 0,005 \text{ g}$ of sodium azide in about 90 ml of water and dilute to 100 ml with water in a measuring cylinder. Store in a glass bottle. This reagent is stable indefinitely.

1) Information derived from a United Kingdom interlaboratory test involving four participants. Limit of detection was taken as 4,65 times the within-batch standard deviation of the blank.