International Standard 7890/2

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Water quality — Determination of nitrate — Part 2: 4-Fluorophenol spectrometric method after distillation

Qualité de l'eau — Dosage des nitrates — Partie 2: Méthode spectrométrique au fluoro-4 phénol après distillation

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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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Water quality — Determination of nitrate — Part 2: 4-Fluorophenol spectrometric method after distillation

1 Scope and field of application

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

This method is applicable to the analysis of all types of water, but is particularly intended for polluted waters and saline waters.

1.3 Range

Up to a nitrate nitrogen content of ϱ_N = 45 mg/l using a 5 ml test portion, and a cell of path length 10 mm. This range may be modified by using different test portion volumes and cell path lengths. (See clause 8.)

1.4 Limit of detection

A nitrate nitrogen content of ϱ_N = 0,22 mg/l.

1.5 Sensitivity

A nitrate nitrogen content of ϱ_N = 45 mg/l gives an absorbance of about 1,4 units in a cell of path length 10 mm.

1.6 Interferences

Potential interferences from nitrite and chloride are removed by the addition of amidosulfonic acid and tin(IV) sulfate respectively. Certain substances, for example nitrophenols, may distil over and colour the distillate. No other interferences are known.

2 Principle

Reaction of nitrate with 4-fluorophenol in acid solution to produce 2-nitro-4-fluorophenol. Recovery of this compound from the reaction mixture by steam distillation into sodium hydroxide solution. Spectrometric measurement of the absorbance of this distillate at 430 nm and reading of the nitrate concentration in the test portion from a calibration graph. Alternatively, extraction of the compound into toluene and then back-extraction into sodium sulfite solution prior to measurement of the absorbance.

3 Reagents

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

3.1 Toluene ($CH_3C_6H_5$).

WARNING — Toluene is highly flammable and harmful by inhalation. Avoid breathing vapour, or contact with skin and eyes. Keep away from sources of ignition.

3.2 Amidosulfonic acid mixture.

Using a mortar and pestle, grind together 46 ± 0,5 g of sodium sulfate (Na_2SO_4), 1,5 ± 0,1 g of sodium chloride (NaCl) and 2,5 ± 0,1 g of amidosulfonic acid (NH_2SO_3H). Mix thoroughly.

Store in a damp-proof container.

3.3 Sulfuric acid solution, ϱ = 1,74 g/ml.

WARNING — When using this acid solution, eye protection and protective clothing are essential. The solution should never be pipetted by mouth.

Place 250 ± 5 ml of water in a 2 litre glass beaker. Gradually and cautiously add 750 ± 10 ml of sulfuric acid (ϱ = 1,84 g/ml) to the water, with continual stirring and cooling. Cool to room temperature.

Store in a glass-stoppered bottle.

3.4 Tin(IV) sulfate, stock solution.

Place 70 ± 2 ml of water in a 500 ml glass beaker. Gradually and cautiously add 300 ± 5 ml of sulfuric acid (ϱ = 1,84 g/ml) to the water, with continual stirring and cooling. After cooling to room temperature, add 60 ± 0,5 g of tin(II) sulfate ($SnSO_4$) to the mixture, and dissolve. Then add, in small portions and with continual stirring, 30 ± 1 ml of 300 g/l hydrogen peroxide solution. After all the hydrogen peroxide has been added, heat the mixture to the boiling point in order to destroy excess hydrogen peroxide. Cool the solution to room temperature.

Store in a glass-stoppered bottle.

Although complete dissolution may not be achieved, the presence of a small amount of sediment in this reagent can be tolerated. Filtration is not necessary.