SLOVENSKI STANDARD
SIST ISO 29441:2011

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Kakovost vode - Določevanje celotnega dušika po UV razklopu - Metoda pretočne analize (CFA in FIA) in spektrometrijske detekcije

Water quality - Determination of total nitrogen after UV digestion - Method using flow analysis (CFA and FIA) and spectrometric detection

Qualité de l'eau - Dosage de l'azote total après digestion UV - Méthode par analyse en flux (CFA et FIA) et détection spectrométrique

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Water quality — Determination of total nitrogen after UV digestion — Method using flow analysis (CFA and FIA) and spectrometric detection

Qualité de l'eau — Dosage de l'azote total après digestion UV — Méthode par analyse en flux (CFA et FIA) et détection spectrométrique

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 29441 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

Methods using flow analysis enable wet chemistry procedures to be automated and are particularly suitable for the processing of many analytes in water in large series of samples at a high analysis frequency (up to 100 samples per hour).

A differentiation is made between flow injection analysis (FIA, References [1][2]) and continuous flow analysis (CFA, Reference [3]). Both methods share the feature of an automatic dosage of the sample into a flow system (manifold) where the analytes in the sample react with the reagent solutions on their way through the manifold. The sample preparation can be integrated into the manifold. The reaction product is measured in a flow detector (e.g. a flow photometer).

Water quality — Determination of total nitrogen after UV digestion — Method using flow analysis (CFA and FIA) and spectrometric detection

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

Waste containing cadmium in liquid or solid form shall be disposed of appropriately.

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of total nitrogen after inline UV digestion, in various types of waters, such as ground, drinking, surface, and waste waters, in mass concentrations ranging from 2 mg/l to 20 mg/l for total nitrogen, all in the undiluted sample.

Other mass concentration ranges are possible, provided the upper limit of the concentration range is exactly 10 times the lower limit (e.g. 0,2 mg/l to 2,0 mg/l). The range of application can be changed by varying the operating conditions.

NOTE Sea water can be analysed with changes in respect to sensitivity and adaptation of the carrier solution and calibration solutions to the salinity of the samples.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

ISO 13395, *Water quality — Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection*

3 Interferences

Samples with extreme pH values and samples with a high buffering capacity are prone to interferences. It is advisable to analyse several dilutions of the sample and to check the results for consistency.

High concentrations of organic substances can cause problems as the oxidation capacity may not be sufficient. For samples containing more than 100 mg/l of total organic carbon (TOC), reduced yields in the determination of nitrogen may arise. If the presence of more than 100 mg/l of TOC is suspected, the sample should be run with several dilutions in order to check for consistency, or standard addition techniques should be applied.

If the sample comprises larger particles (diameter, $d > 50$ µm), a homogenisation device (6.3.3) is necessary.

In sea water samples, high concentrations of calcium and magnesium may occur. In an alkaline medium, magnesium hydroxide or other hydroxides or calcium carbonate may be formed which interfere with the UV digestion.

4 Principle

The sample is pretreated with a buffered peroxodisulfate solution and thermal UV radiation. Nitrate is formed and determined either by flow injection analysis (FIA) or by continuous flow analysis (CFA). With FIA, the sample is fed into a continuously flowing buffer solution (carrier stream) by means of an injection valve, or, when applying CFA, it is continuously mixed with this buffer solution. Nitrate in the sample is reduced with metallic cadmium to nitrite (Reference [4]). Subsequently a phosphoric acid reagent solution also flowing continuously is admixed. Nitrite resulting from the reduction of nitrate diazotises sulfanilamide in acidic solution to the diazonium salt which is then coupled with *N*-(1-naphthyl)ethylenediamine to form a red soluble dye (see ISO 13395 and Reference [5]).

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade. Check the blank value of the reagents regularly (see 8.4).

5.1 General reagents

5.1.1 Water, complying with ISO 3696:1987, grade 1.

5.1.2 Phosphoric acid, $\rho(H_3PO_4) = 1{,}71$ g/ml.

5.1.3 Potassium peroxodisulfate, $K_2S_2O_8$.

5.1.4 Sodium hydroxide, NaOH.

5.1.5 Titanium tetrachloride, $TiCl_4$, liquid, commercially available.

5.1.6 Sulfanilamide, 4-aminobenzenesulfonamide, $C_6H_8N_2O_2S$.

5.1.7 *N*-(1-Naphthyl)ethylenediamine dihydrochloride, [*N*-(1–naphthyl)-1,2-diaminoethane dihydrochloride, $C_{12}H_{14}N_2 \cdot 2HCl$].

5.1.8 Sodium nitrite, $NaNO_2$, dried to constant mass at 150 °C.

5.1.9 Potassium nitrate, KNO_3, dried to constant mass at 150 °C.

5.1.10 Imidazole, $C_3H_4N_2$.

5.1.11 Hydrochloric acids.

5.1.11.1 Hydrochloric acid I, concentrated, w(HCl)= 37 %.

5.1.11.2 Hydrochloric acid II, c(HCl) = 4 mol/l.

5.1.11.3 Hydrochloric acid III, c(HCl) ≈ 0,1 mol/l.

5.1.12 Dichloromethane, CH_2Cl_2.

5.1.13 Copper(II) sulfate solution I, $\rho(CuSO_4 \cdot 5H_2O)$ = 2,5 g/l.

5.1.14 Copper(II) sulfate solution II, $\rho(CuSO_4 \cdot 5H_2O)$ = 20 g/l.

5.1.15 Boric acid, H_3BO_3.

5.1.16 Sulfuric acid I, $\rho(H_2SO_4)$ = 1,84 g/ml.

5.1.17 Sulfuric acid II, $c(H_2SO_4)$ = 1 mol/l.

5.1.18 Urea, $CO(NH_2)_2$.

5.1.19 Cadmium (Cd) granulate, grain size e.g. 0,3 mm to 1,5 mm (for FIA preferably 0,3 mm to 0,8 mm). A minimum reduction capacity of 90 % shall be reached (see 6.1.4 and 6.2.4).

5.1.20 Imidazole stock solution, $c(C_3H_4N_2)$ = 1 mol/l (Figure A.1, R1).

Dissolve 68,1 g of imidazole (5.1.10) in approximately 800 ml of water (5.1.1) in a 1 l beaker.

While stirring with a magnetic stirrer, add hydrochloric acid I (5.1.11.1) and adjust the pH to 7,5 using a pH electrode (6.3.2).

Transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water (5.1.1).

The solution is stable for 4 weeks if kept in a brown glass bottle at room temperature.

5.1.21 Urea stock solution, ρ(N) = 1 000 mg/l.

In a 500 ml one-mark volumetric flask, dissolve 1,071 7 g of urea (5.1.18) in water (5.1.1). Add 0,5 ml of dichloromethane (5.1.12) for preservation. Make up to the mark with water.

The solution is stable for 1 year if kept at (4 ± 2) °C.

5.1.22 Urea working solution, ρ(N) = 20 mg/l.

Dilute 5 ml of urea stock solution (5.1.21) in a 250 ml one-mark volumetric flask with water (5.1.1). Acidify the solution with sulfuric acid II (5.1.17) to pH ⩽ 2.

The solution is stable for 1 month if kept at (4 ± 2) °C.

5.1.23 Buffered copper(II) sulfate solution.

Mix 20 ml of the copper(II) sulfate solution II (5.1.14) and 20 ml of the imidazole stock solution (5.1.20) in a 50 ml beaker.

Prepare the solution freshly before use.