SLOVENSKI STANDARD
SIST EN 12260:2003

01-december-2003

Kakovost vode ï Doloļ evanje duġika ï Doloļ evanje vezanega duġika (TNb) po oksidaciji do duġikovih oksidov

Water quality - Determination of nitrogen - Determination of bound nitrogen (TNb), following oxidation to nitrogen oxides

Wasserbeschaffenheit - Bestimmung von Stickstoff - Bestimmung von gebundenem Stickstoff (TNb) nach Oxidation zu Stickstoffoxiden

Qualité de l'eau - Dosage de l'azote - Dosage de l'azote lié (TNb) apres oxydation en oxydes d'azote

Ta slovenski standard je istoveten z: **EN 12260:2003**

ICS:

13.060.50	Preiskava vode na kemiļ ne snovi	Examination of water for chemical substances

SIST EN 12260:2003 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12260

September 2003

ICS 13.060.50

Supersedes ENV 12260:1996

English version

Water quality - Determination of nitrogen - Determination of bound nitrogen (TN_b), following oxidation to nitrogen oxides

Qualité de l'eau - Dosage de l'azote - Dosage de l'azote lié (TN_b) après oxydation en oxydes d'azote

Wasserbeschaffenheit - Bestimmung von Stickstoff - Bestimmung von gebundenem Stickstoff (TN_b) nach Oxidation zu Stickstoffoxiden

This European Standard was approved by CEN on 1 August 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12260:2003 E

Contents

Foreword

This document (EN 12260:2003) has been prepared by Technical Committee CEN/TC 230 “Water analysis”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2004, and conflicting national standards shall be withdrawn at the latest by March 2004.

This document supersedes ENV 12260: 1996

Annexes A B, and C are informative annexes.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

WARNING — Persons using this 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.

It is absolutely essential that tests carried out according to this European Standard are carried out by suitably qualified staff.

This European Standard specifies a method for the determination of total bound nitrogen after oxidation of inorganic and organic nitrogen compounds by combustion to nitrogen oxides. Quantification is carried out by chemiluminescence.

The procedure referred to in the normative part is the reference method. Whilst staying within the scope of this standard, it is permissible to use such alternatives as given in annex A only, provided that their performance is equal to or better than that given in clause 13 and tables in annex B and C, when calculated using procedures given in ISO 5725-2, and when the comparison of performance data between this European Standard and any alternative technique is carried out using the procedures described in ISO 2854.

All references to nitrogen concentrations are expressed in units of mg/l N, i.e. milligrams of nitrogen per litre solution.

1 Scope

This European Standard specifies a method for the determination of nitrogen in water in the form of free ammonia, ammonium, nitrite, nitrate and organic compounds capable of conversion to nitrogen oxides under the oxidative conditions described. Determination is carried out instrumentally. Dissolved nitrogen gas is not determined by this method.

This method is applicable to the analysis of surface water, waste water and treated sewage effluent.

The concentration range of the method will depend on the injection volume used which is instrument specific. Total nitrogen can be determined in the range from 1 mg/l up to 200 mg/l. Higher concentrations can, if necessary, be determined by dilution of the sample. The limit of detection will depend on the instrument in use. Using a suitable injection volume, the limit of detection is typically round about 0,5 mg/l.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:1994)*.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic methods for the determination of repeatability and reproducibility of a standard measurement method.*

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 function.

3 Principle

Oxidation of the sample containing nitrogen by catalytic combustion in an oxygen atmosphere at > 700 °C, to nitrogen oxides. Quantification of nitrogen concentration is carried out by chemiluminescence detection (after reaction with ozone).

4 Interferences

Depending on the instrument in use, interferences may arise from memory effects. These may occur either from samples or standard solutions with high amounts of bound nitrogen.

NOTE Potential problems can arise with samples containing significant total organic carbon (TOC) concentrations. The analysis of samples containing large amounts of TOC will lead to the reporting of lower results for nitrogen. Suspected problems can be identified by determining nitrogen before and after suitable dilution, or by using standard addition techniques.

Not all organic nitrogen compounds are quantitatively converted to nitrogen oxides by the oxidation procedure used (see recovery rates in table B.1).

If a homogenized sample containing suspended material produces results (obtained from replicate measurements) which deviate by more than 10 %, the sample needs to be filtered through a 0,45 µm filter, free of nitrogen containing compounds. In this case, only the dissolved portion of the bound nitrogen will be determined and reported.

5 Reagents

5.1 General

Use reagents of recognized analytical grade.

5.2 Water

During analysis use Grade 3 purity water as specified in EN ISO 3696. The contents of bound nitrogen in water being used for the preparation of calibration solutions shall be negligibly low compared with the lowest concentration to be determined.

5.3 Hydrochloric acid, $\rho(HCl) = 1{,}12$ g/ml.

5.4 Nitrogen stock solution for calibration, $\rho(N) = 1{,}000$ g/l.

5.4.1 Dissolve (4,717 ± 0,001) g of ammonium sulfate, $(NH_4)_2SO_4$, previously dried at (105 ± 2) °C to constant weight, in a 1 000 ml volumetric flask, and make up to volume with water.

5.4.2 Dissolve (7,219 ± 0,001) g of potassium nitrate, KNO_3, previously dried at (105 ± 2) °C to constant weight, in a 1 000 ml volumetric flask, and make up to volume with water.

5.4.3 Mix equal volumes of the above solutions (see 5.4.1 and 5.4.2) to produce a mixed standard solution.

Stored in a refrigerator at (4 ± 2) °C, the standard solution can be used for about one month.

5.5 Nicotinic acid, $C_6H_5NO_2$, > 99,5 %.

5.6 Nitrogen stock solutions for system check, $\rho(N) = 1{,}000$ g/l.

5.6.1 Prepare separately stock solutions corresponding to 5.4.1 and 5.4.2.

5.6.2 Dissolve (8,793 ± 0,001) g of nicotinic acid (5.5), previously dried at (105 ± 2) °C to constant mass, transfer to a 1 000 ml volumetric flask and make up to volume with water (5.2).

Store in a refrigerator at (4 ± 2) °C, the standard solution can be used for about one month.

5.7 Oxygen, O_2, with a volume fraction of > 99,7 %.

6 Apparatus

6.1 Apparatus suitable for the determination of total bound nitrogen by oxidation

This apparatus will be instrument dependant and comprises for example reaction vessel, automatic sample injection device, sample homogenizing equipment, suitable detectors or sensors and computing system. A manual injection system may also be used. For requirements for a suitable system see clause 10.

6.2 Homogenizer

6.3 Filtration apparatus, with membrane filters (porewidth 0,45 µm).

6.4 Syringes

7 Sampling and sample preparation

When sampling, ensure that a representative sample is obtained (this is particularly important for samples containing any undissolved matter), and that the sample is not contaminated. In addition, see EN ISO 5667-3.

Withdraw samples in suitable containers consisting of glass or suitable plastics, appropriately sealed.

Analyze samples as soon as possible.

NOTE Delay may result in the reporting of low values (especially in the case of biologically active samples).

Samples may be stabilized by the addition of hydrochloric acid (5.3) to achieve a pH value of less than 2, and stored in a refrigerator at (4 ± 2) °C. Stabilised samples may be stored for a period of up to 8 days. Alternatively store smaller sample volumes (e.g. 50 ml to 100 ml) at -15 °C to -20 °C up to 14 days.

If necessary, homogenize the sample in order to obtain a representative aliquot for the determination (see clause 4). If this is impracticable, filter through a 0,45 µm filter (6.3).

8 Procedure

Follow the instrument manufacturer's instructions.

Prior to the determination of bound nitrogen, ensure that instrument operational tests and the verification of the responses to blank and standard solutions are carried out within the intervals specified by the manufacturer.

Adjust the instrument according to the manufacturer's instructions for each series of measurements.

If an instrument is fitted with an automated injection system, ensure that the sample is unlikely to cause any interference with the injection procedure by, for example, homogenizing the sample (see also clause 4).

Inject identical volumes of blank solutions and samples into the instrument according to the manufacturer's instructions and measure the responses obtained for aliquots of each solution at least three times.

Reject the first and use the mean response from at least two injections taking into account any memory effects likely to occur (see clause 4). Use the calibration graph (see clause 9) to determine the concentration of total bound nitrogen in the sample.

9 Establishment of the calibration curve

Use the mixed standard solution (5.4.3) to prepare calibration solutions in accordance with the range of concentrations of total bound nitrogen expected in the samples to be determined.

Use the calibration solutions on the day of preparation only. Calibrate within one decade of concentration. For the concentration range of 10 mg/l to 100 mg/l, proceed, for example, as follows:

Into 7 separate volumetric 100 ml flasks, pipette 0, (blank) 1,0 ml; 2,0 ml; 3,0 ml; 6,0 ml; 8,0 ml or 10,0 ml of the mixed standard solution (5.4.3) and make up to volume with water; these quantities correspond to mass