SLOVENSKI STANDARD
SIST EN ISO 11732:1999

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Kakovost vode - Določevanje amonijevega dušika s pretočno analizo (CFA in FIA) in spekrofotometrijsko detekcijo (ISO 11732:1997)

Water quality - Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732:1997)

Wasserbeschaffenheit - Bestimmung von Ammoniumstickstoff mit der Fließanalysis (CFA und FIA) und spektrometrischer Detektion (ISO 11732:1997)

Qualité de l'eau - Détermination de l'azote ammoniacal par analyse de l'écoulement (CFA et FIA) et détection spectrométrique (ISO 11732:1997)

Ta slovenski standard je istoveten z: **EN ISO 11732:1997**

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Water quality - Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection (ISO 11732:1997)

Qualité de l'eau - Détermination de l'azote ammoniacal par analyse de l'écoulement (CFA et FIA) et détection spectrométrique (ISO 11732:1997)

Wasserbeschaffenheit - Bestimmung von Ammoniumstickstoff mit der Fließanalysis (CFA und FIA) und spektrometrischer Detektion (ISO 11732:1997)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
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Foreword

The text of the International Standard ISO 11732:1997 has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with 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of an identical text or by endorsement, at the latest by January 1998, and conflicting national standards shall be withdrawn at the latest by January 1998.

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, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 11732:1997 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 11732

First edition
1997-07-01

Water quality — Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection

Qualité de l'eau — Détermination de l'azote ammoniacal par analyse en flux (CFA et FIA) et détection spectrométrique

Reference number
ISO 11732:1997(E)

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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11732 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A to D of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Introduction

Methods using flow analysis are automatized wet chemical procedures and are therefore particularly suitable for the processing of large sample series at a high analysis frequency (up to 100 samples per hour).

One differentiates between flow injection analysis (FIA) [1], [2] and continuous flow analysis (CFA) [3]. Both methods include the automatic dosage of the sample into a flow system (manifold) in which the analytes in the sample react with the reagent solutions on their way through the manifold. The sample preparation may be integrated in the manifold. The reaction product is analysed spectrometrically in a flow detector.

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Water quality — Determination of ammonium nitrogen by flow analysis (CFA and FIA) and spectrometric detection

1 Determination of ammonium nitrogen by flow injection analysis (FIA) and spectrometric detection

1.1 Scope

1.1.1 Field of application

This International Standard specifies a method suitable for the determination of ammonium nitrogen in various types of waters (such as ground, drinking, surface and waste waters) in mass concentrations ranging from 0,1 to 10 mg/l (in the undiluted sample). In particular cases, the range of application may be adapted by varying the operating conditions.

1.1.2 Interferences

Volatile amines will diffuse through the membrane and lead to a pH shift. If the concentrations of the volatile amines (e.g. methylamine or ethylamine) are equal to those of the ammonium, erroneously high results may be expected [12]. In significant cases, prior to analysis an (online) distillation of the sample, adjusted to a pH of 5,8 may be necessary.

Interferences may occur in exceptional cases when the sample does not reach a pH at least 12 after the addition of the alkaline reagent, since then ammonium will not be converted quantitatively into ammonia. In particular, this may occur with strongly acidic or buffered samples. In such cases the pH of the sample should be adjusted to 3 to 5 by the addition of sodium hydroxide solution (1.4.1 or 1.4.2).

High concentrations of metal ions which may precipitate as hydroxides will give poorly reproducible results. The addition of a suitable complexing agent, such as (ethylenedinitrilo)tetraacetic acid, disodium salt, to the alkaline reaction solution (1.4.17) in a sufficiently large concentration will prevent interference by Cu, Zn, Fe, Ca, Mg and Al; up to individual metal concentrations of 0,2 mg/l, a concentration of 30 g/l of ethylenedinitrilotetraacetic acid, disodium salt, in solution R_1 (see 1.4.17) is adequate.

For samples containing particulate matter, see 1.6 (last paragraph).

Samples with a total salt concentration of > 10 g/l should be diluted prior to measurement.

1.2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

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

1.3 Principle

The test sample containing ammonium is injected into a continuous carrier stream by means of an injection valve, and is mixed with a continuous flow of an alkaline solution. The ammonia formed is separated in a diffusion cell from the solution over a hydrophobic semipermeable membrane and taken up by a streaming recipient flow containing a pH indicator. Due to the resulting pH shift, the indicator solution will change colour; the colour change is monitored continuously in a flow spectrophotometer. Additional information concerning this analytical technique is given in [4], [5], [6], [7] and [8].

1.4 Reagents

Apart from the reagents listed in 1.4.4 to 1.4.6, use only reagents of analytical grade quality for the determination of nitrogen or, if not available, those of recognized analytical grade quality and water of grade 1 (in accordance with ISO 3696), freshly prepared. The ammonium content of the blank shall be checked regularly (see 1.7.3).

1.4.1 Sodium hydroxide solution I, c(NaOH) = 5 mol/l.

1.4.2 Sodium hydroxide solution II, c(NaOH) = 0,01 mol/l.

1.4.3 Ethylenedinitrilotetraacetic acid (EDTA), disodium salt, monohydrate, $Na_2C_{10}H_{14}N_2O_8 \cdot H_2O$.

1.4.4 Bromcresol purple, $C_{21}H_{16}Br_2O_5S$.

1.4.5 Bromthymol blue, $C_{27}H_{28}Br_2O_5S$.

1.4.6 Cresol red, $C_{21}H_{18}O_5S$.

1.4.7 Ammonium chloride, NH_4Cl, dried at 105 °C to constant weight.

1.4.8 Potassium chloride, KCl.

1.4.9 Boric acid, H_3BO_3.

1.4.10 Ethanol, C_2H_5OH, 95 % mass fraction.

1.4.11 Hydrochloric acid I, c(HCl) = 0,01 mol/l.

1.4.12 Hydrochloric acid II, c(HCl) = 0,1 mol/l.

1.4.13 Hydrochloric acid III, c(HCl) = 1,0 mol/l.

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

1.4.15 Mixed indicator.

In a mortar prepare a dry mixture consisting of 10 g of Bromcresol purple (1.4.4), 5 g of Bromthymol blue (1.4.5), 2,5 g of Cresol red (1.4.6) and 45 g of potassium chloride (1.4.8).

The given quantities can be reduced (e.g. by one-tenth), maintaining the ratio.

1.4.16 Carrier solution, C (see figure 1).

Use grade 1 water (ISO 3696), degassed by reduced pressure.

1.4.17 Alkaline reaction solution, R_1 (see figure 1).

Dissolve in a graduated flask, nominal capacity 1 000 ml, 30 g of EDTA disodium salt (1.4.3) in approximately 800 ml of water, and add 12,4 g of boric acid (1.4.9).

Add dropwise to the suspension 100 ml of sodium hydroxide solution I (1.4.1), and make up to volume with water.

Degas the solution by filtering it through a membrane filter assembly (see 1.5.2).

The pH of the solution will be approximately 13. When stored in a polyethylene bottle at room temperature, it will be stable for 1 month.

1.4.18 Indicator solution

In a graduated flask, nominal capacity 200 ml, dissolve 1 g of the mixed indicator (1.4.15) in a mixture of 10 ml of sodium hydroxide solution II (1.4.2) and 10 ml of ethanol (1.4.10).

Add approximately 150 ml of water.

The solution should have a bright orange-red colour. If it has a blue colour, add hydrochloric acid III dropwise (1.4.13) until the colour changes.

Make up to volume with water.

Filter off any undissolved particles.

This solution can be stored without deterioration at room temperature for 3 months in a brown glass bottle.

1.4.19 Ammonia recipient solution, R_2 (see figure 1)

Dilute 10 ml of the indicator solution (1.4.18) with approximately 480 ml of water.

Add dropwise sodium hydroxide solution II (1.4.2) until an absorbance value of 0,45 to 0,6 (pathlength 10 mm, wavelength 590 nm) is obtained. Make up to a volume of 500 ml with water.

Degas and purify the solution using the membrane filter assembly (see 1.5.2), pour it into the reagent reservoir and let it stand for at least 2 h.

Immediately before starting the measurement (1.7), check the absorbance again and adjust, if need be, to the absorbance range specified above by adding sodium hydroxide solution II (1.4.2) or hydrochloric acid I, II or III (1.4.11 to 1.4.13).

This solution can be stored without deterioration at room temperature for 2 weeks in a glass bottle.

1.4.20 Ammonium stock solution, $\rho_B(N) = 1\,000$ mg/l.

In a graduated flask, nominal capacity 1 000 ml, dissolve, 3,819 g of ammonium chloride (1.4.7) in approximately 900 ml water, acidify with sulfuric acid (1.4.14) to pH 2, and make up to volume with water.

This solution can be stored without deterioration in a refrigerator for at least 3 months.

1.4.21 Ammonium standard solution I, $\rho_B(N) = 100$ mg/l.

Pipette 10 ml of the ammonium stock solution (1.4.20) into a graduated flask, nominal capacity 100 ml, add approximately 80 ml water, acidify with sulfuric acid (1.4.14) to pH 2, and make up to volume with water.

This solution can be stored without deterioration in a refrigerator for at least 1 week.

1.4.22 Ammonium standard solution II, $\rho(N) = 10$ mg/l.

Pipette 1 ml of the ammonium stock solution (1.4.20) or 10 ml of the ammonium standard solution I (1.4.21) into a graduated flask, nominal capacity 100 ml, add approximately 80 ml water, acidify with sulfuric acid (1.4.14) to pH 2, and make up to volume with water.