

**Vee kvaliteet. Ammoniaagilämmastiku sisalduse
määramine pidevvoolumanalüüsidel (CFA ja FIA) ja
spektromeetrilisel detekteerimisel**

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN ISO 11732:1999 sisaldab Euroopa standardi EN ISO 11732:1997 ingliskeelset teksti.	This Estonian standard EVS-EN ISO 11732:1999 consists of the English text of the European standard EN ISO 11732:1997.
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EUROPEAN STANDARD

EN ISO 11732

NORME EUROPÉENNE

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English version

**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 Fließanalyse (CFA
und FIA) und spektrometrischer Detektor
(ISO 11732:1997)

This European Standard was approved by CEN on 1997-05-28. 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 Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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of an identical text or by endorsement, at the latest by January 1998, and conflicting national standards shall be withdrawn at the latest by January 1998.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.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

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.

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 R₁ (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, $c(\text{NaOH}) = 5 \text{ mol/l}$.

1.4.2 Sodium hydroxide solution II, $c(\text{NaOH}) = 0,01 \text{ mol/l}$.

1.4.3 Ethylenedinitrilotetraacetic acid (EDTA), disodium salt, monohydrate, $\text{Na}_2\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8 \cdot \text{H}_2\text{O}$.

1.4.4 Bromcresol purple, $\text{C}_{21}\text{H}_{16}\text{Br}_2\text{O}_5\text{S}$.

1.4.5 Bromthymol blue, $\text{C}_{27}\text{H}_{28}\text{Br}_2\text{O}_5\text{S}$.

1.4.6 Cresol red, $\text{C}_{21}\text{H}_{18}\text{O}_5\text{S}$.

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, $\text{C}_2\text{H}_5\text{OH}$, 95 % mass fraction.

1.4.11 Hydrochloric acid I, $c(\text{HCl}) = 0,01 \text{ mol/l}$.

1.4.12 Hydrochloric acid II, $c(\text{HCl}) = 0,1 \text{ mol/l}$.

1.4.13 Hydrochloric acid III, $c(\text{HCl}) = 1,0 \text{ mol/l}$.

1.4.14 Sulfuric acid, $\rho(\text{H}_2\text{SO}_4) = 1,84 \text{ g/ml}$.

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.