SLOVENSKI STANDARD
SIST ISO 10048:1996

01-junij-1996

Kakovost vode - Določanje dušika - Katalitski razklop po redukciji z Devardovo zlitino

Water quality -- Determination of nitrogen -- Catalytic digestion after reduction with Devarda's alloy

Qualité de l'eau -- Dosage de l'azote -- Minéralisation catalytique après réduction avec l'alliage de Devarda

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INTERNATIONAL STANDARD

ISO 10048

First edition
1991-11-01

Water quality — Determination of nitrogen — Catalytic digestion after reduction with Devarda's alloy

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Reference number
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SIST ISO 10048:1996

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 10048 was prepared by Technical Committee ISO/TC 147, *Water quality*, Sub-Committee SC 2, *Physical, chemical, biochemical methods*.

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Water quality — Determination of nitrogen — Catalytic digestion after reduction with Devarda's alloy

1 Scope

1.1 Substance determined

This International Standard specifies a method for the determination of nitrogen present in water samples in the form of ammonium, nitrite, nitrate, and organic nitrogen compounds capable of conversion to ammonium in the conditions of the method.

1.2 Type of sample

This method is applicable to the analysis of raw and polluted waters.

1.3 Range

A nitrogen content, ρ_N, of up to 200 mg/l may be determined. This range may be extended by using a smaller test portion.

1.4 Limit of detection[1]

Using a 50 ml test portion, the limit of detection is $\rho_N = 3$ mg/l when using the titrimetric method. Using the spectrometric method (see 7.2, note 2), the limit of detection is $\rho_N = 1$ mg/l.

1.5 Sensitivity

Using a 100 ml test portion, 1,0 ml of 0,02 mol/l hydrochloric acid is equivalent to $\rho_N = 2{,}8$ mg/l.

1.6 Interferences

Falsely low results may be obtained if the mineralization procedure is over-prolonged. To minimize this effect, the procedure in 7.2 shall be carefully observed.

Certain organic nitrogen compounds are known to be difficult to mineralize. Table 2 gives information on recovery of nitrogen from a variety of compounds.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7150-1:1984, *Water quality — Determination of ammonium — Part 1: Manual spectrometric method.*

3 Principle

Reduction of oxidized nitrogen compounds to ammonium ions using Devarda's alloy. After evaporation almost to dryness, conversion of all nitrogen compounds to ammonium sulfate in the presence of concentrated sulfuric acid containing a high concentration of potassium sulfate, to raise the boiling point of the mixture, and in the presence of copper which acts as a catalyst.

Liberation of ammonia from the mixture by the addition of alkali and distillation into boric acid/indicator solution. Determination of ammonium in the distillate either by titration with standard acid or by spectrometry at a wavelength of 655 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and ammonia-free water.

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[1] Information from Finland.

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4.1 Hydrochloric acid, $\rho = 1,18$ g/ml.

4.2 Sulfuric acid, $\rho = 1,84$ g/ml.

WARNING — This reagent causes severe burns.

The highest purity sulfuric acid shall be used. Pay particular attention to the manufacturer's specification with respect to its nitrogen content and ensure that it is as low as possible.

4.3 Sodium hydroxide, approximately 300 g/l solution.

WARNING — This reagent causes severe burns.

Dissolve 320 g ± 20 g of sodium hydroxide in about 800 ml of water. Cool to room temperature and dilute to 1 litre with water in a measuring cylinder.

Store in a polyethylene bottle.

4.4 Devarda's alloy [about 45 % (*m*/*m*) aluminium, 50 % (*m*/*m*) copper, and 5 % (*m*/*m*) zinc] powdered form.

Select, from commercially-available products, materials as low as possible in nitrogen content.

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4.5 Potassium sulfate, (K_2SO_4).

4.6 Boric acid/indicator solution.

4.6.1 Dissolve 0,10 g ± 0,01 g of bromocresol green and 0,020 g ± 0,005 g of methyl red in about 80 ml of ethanol and dilute to 100 ml with ethanol in a measuring cylinder.

4.6.2 Dissolve 20 g ± 1 g of boric acid (H_3BO_3) in warm water. Cool to room temperature. Add 10 ml ± 0,5 ml of indicator solution (see 4.6.1) and dilute to 1 litre with water in a measuring cylinder.

4.7 Hydrochloric acid standard volumetric solution, $c(\mathrm{HCl}) = 0,02$ mol/l.

This solution shall be prepared by dilution of hydrochloric acid (4.1) followed by standardization by normal analytical procedures. Alternatively, use a commercial solution of guaranteed concentration.

4.8 Glycine solution, $\rho_N = 1\,000$ mg/l.

Dissolve 5,362 g ± 0,002 g of glycine (H_2NCH_2COOH) in about 800 ml of water and dilute to 1 litre with water in a calibrated flask.

4.9 Glycine solution, $\rho_N = 10$ mg/l.

Pipette 10 ml of glycine solution (4.8) into a 1 000 ml one-mark volumetric flask and make up to the mark with water. Prepare this solution freshly for each batch of analyses.

4.10 Potassium standard stock solution, $\rho_N = 1\,000$ mg/l.

Dissolve 7,215 g ± 0,001 g of potassium nitrate ($KHNO_3$) (previously dried at 105 °C for at least 2 h) in about 750 ml of water. Quantitatively transfer to a 1 000 ml one-mark volumetric flask and make up to the mark with water.

Store the solution in a glass bottle for not more than 2 months.

4.11 Potassium nitrate standard solution, $\rho_N = 10$ mg/l.

Pipette 5 ml of the standard stock solution (4.10) into a 500 ml one-mark volumetric flask and make up to the mark with water.

Store the solution in a glass bottle for not more than 1 month.

4.12 Anti-bumping granules.

5 Apparatus

Ordinary laboratory apparatus and the following

5.1 Heating mantles or heating block, capable of maintaining a temperature of at least 400°C.

5.2 Kjeldahl flasks, of capacity 100 ml to 250 ml, compatible with the heating mantles (5.1), or **digestion tubes**, of capacity 100 ml to 250 ml, fitting snugly into the heating block (5.1).

5.3 Distillation apparatus, incorporating an anti-splash distillation head and a vertical condenser whose outlet may be submerged in the absorbent solution.

If the digestion vessels (5.2) are not suitable for direct attachment to the distillation apparatus, separate distillation flasks are necessary.

Alternatively, use an apparatus for steam distillation. Such apparatus is available commercially, but it shall be checked to ensure that ammonia is neither lost during distillation nor formed in the steam generated for distillation.

6 Sampling and samples

Collect laboratory samples in polyethylene or glass bottles. Analyse them as quickly as possible, or store them at between 2 °C and 5 °C until analysed.

NOTE 1 Acidification to pH 2 with sulfuric acid (4.2) may also be used as an aid to preservation, provided that

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possible contamination of the acidified sample by absorption of any atmospheric ammonia is avoided.

7 Procedure

7.1 Blank test

Proceed as described in 7.2, but use about 50 ml of water instead of a test portion. Record the volume, V_2, of hydrochloric acid (4.7) added.

7.2 Determination

WARNING — The mineralization procedure may evolve toxic sulfur dioxide gas. Hydrogen sulfide and/or hydrogen cyanide may also be liberated from polluted samples. The mineralization should therefore be carried out under an effective fume extraction system.

Pipette 50 ml of the laboratory sample (see note 2) into Kjeldahl flask (5.2). Add 4,0 ml ± 0,1 ml of sulfuric acid (4.2), 0,20 g ± 0,01 g of Devarda's alloy (4.4) and 2,00 g ± 0,05 g of potassium sulfate (4.5).

After at least 60 min, add a few anti-bumping granules (4.12) and boil the contents of the flask under an effective fume extraction system. The volume of the contents will decrease as water is boiled away.

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When white fumes begin to appear, insert a small filter funnel into the neck of the Kjeldahl flask in order to reduce evaporation. Do not allow the sample to dry. The temperature of the liquid shall not exceed 370 °C at this stage.

After white fume evolution has ended, periodically observe the mineralizate and, after it has become either colourless or light green in colour, continue heating for a further 60 min ± 5 min.

After mineralization, allow the flask to cool to room temperature. Meanwhile, measure 20 ml ± 2 ml of boric acid/indicator solution (4.6.2) into the receiving flask of the distillation apparatus (see note 3). Ensure that the delivery tip of the condenser is below the surface of the indicator solution.

Carefully add 10 ml ± 1 ml of water to the mineralization flask. Then add, from a measuring cylinder, 25 ml of sodium hydroxide solution (4.3) and immediately attach the flask to the distillation apparatus (see note 3).

Heat the distillation flask so that distillate collects at a rate of about 5 ml/min. Stop the distillation when about 30 ml have been collected. Titrate the distillate to a purple end-point, using the indicator already present in the receiving flask (4.6.2), with 0,02 mol/l hydrochloric acid (4.7) and record the volume added.

NOTES

2 The laboratory sample should have a nitrogen concentration of not more than 200 mg/l. Samples with higher concentrations than this shall be diluted with water prior to taking the 50 ml test portion.

3 Ammonium ions may be determined in the distillate by the spectrometric method described in ISO 7150-1. However, it is essential that the distillate should be collected in 1 % (V/V) hydrochloric acid instead of the boric acid/indicator solution. Clause 10 of ISO 7150-1:1984 gives details of this modification and of the modified calculation of result.

4 Where the flask is not compatible with the distillation apparatus, the contents should be transferred quantitatively to a suitable distillation flask, using a total volume of 10 ml ± 2 ml of water and rinsing three times.

8 Expression of results

8.1 Method of calculation

Calculate the total nitrogen concentration, ρ_N, expressed in milligrams per litre, using the formula

$$\rho_N = \frac{V_1 - V_2}{V_0} \times c(\mathrm{HCl}) \times 14{,}01 \times 1\,000$$

where

V_0	is the volume, in millilitres, of the test portion (which is usually 50 ml, see 7.2);
V_1	is the volume, in millilitres, of the standard volumetric hydrochloric acid used for titration (see 7.2);
V_2	is the volume, in millilitres, of the standard volumetric hydrochloric acid used for the titration of the blank test (see 7.1);
$c(\mathrm{HCl})$	is the exact concentration, expressed in moles per litre, of the hydrochloric acid used for titration;
14,01	is the relative atomic mass of nitrogen.

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Express the result either as the mass concentration of nitrogen ρ_N, in milligrams per litre, or as the amount-of-substance concentration of nitrogen, c_N, in micromoles per litre.

To convert ρ_N to c_N, use the formula

$$c_N = 71{,}4\ \rho_N$$

8.2 Repeatability and reproducibility

Repeatability and reproducibility standard deviations have been determined as shown in table 1.

Table 1 — Repeatability and reproducibility standard deviations

Sample[1]	Nitrogen concentrations ρ_N mg/l	Standard deviations	
		Repeatability r mg/l	Reproducibility R mg/l
4-Nitrophenol	20[2]	0,12 — 0,48	0,36
4-Nitroaniline	20[2]	0,12 — 1,98	0,78
Pyridine	20[2]	0,30 — 5,1	2,24
Purine	20[2]	0,06 — 1,06	0,29
Sodium glutamate	20[2]	0,20 — 1,58	0,74
A	5,303[3]	0,127	0,657
A	5,673[3]	0,125	0,743
B	6,994[4]	0,091	0,573
B	7,984[4]	0,199	0,583
B	21,84[4]	0,37	0,93
B	14,84[4]	0,45	0,92
B	23,35[5]	0,93	3,18
C	9,845[5]	0,57	1,93
D	2065[5]	3,3	7,6

1) A Pulp mill effluent, B Municipal effluent, C Surface water (river), D Industrial effluent.

2) Data from trials carried out by 8 laboratories in Finland and Germany.

3) Data from trials carried out by 14 laboratories in Finland.

4) Data from trials carried out by 17 laboratories in Finland.

5) Data from trials carried out by 12 laboratories in Germany.

Table 2 — Recovery of organic nitrogen

Compound	Nitrogen concentration ρ_N mg/l	Percentage recovery %
4-Nitrophenol	20	58,0 — 103,5[1]
4-Nitroaniline	20	90,8 — 133,8[1]
Pyridine	20	24,3 — 101,5[1]
Purine	20	82,2 — 108,0[1]
Benzonitrile	20	1,5 — 8,3[1]
Sodium glutamate	20	79,2 — 97,4[1]
4-Nitrophenol + nitrate	30[2]	85,2 — 97,4[3]
4-Nitroaniline + nitrate	30[2]	88,4 — 94,4[3]
Pyridine + nitrate	30[2]	77,6 — 87,9[3]
Purine + nitrate	30[2]	82,2 — 94,3[3]

1) Range of recoveries from between three and eight laboratories.

2) 10 mg/l from nitrate, 20 mg/l from the organic compound.

3) Results from two laboratories.

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