

SLOVENSKI STANDARD SIST ISO 5664:1996

01-junij-1996

Kakovost vode - Določanje amonija - Destilacijska in titracijska metoda

Water quality -- Determination of ammonium -- Distillation and titration method

Qualité de l'eau -- Dosage de l'ammonium -- Méthode par distillation et titrimétrie

Ta slovenski standard je istoveten z: ISO 5664:1984

| ICS: | <u>SIST ISC</u> https://standards.iteh.ai/catalog/stan c9d6b4c0be7b/ | <u>) 5664:1996</u> dards/sist/ac87c37c-0dee-4c2f-951a- 'sist-iso-5664-1996 | |
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| 13.060.50 | Preiskava vode na kemične snovi | Examination of water for chemical substances | |
| SIST ISO 56 | 64:1996 | en | |



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Determination of ammonium — Distillation and titration method

Qualité de l'eau – Dosage de l'ammonium – Méthode par distillation et titrimétrie

First edition – 1984-05-15Teh STANDARD PREVIEW (standards.iteh.ai)

<u>SIST ISO 5664:1996</u> https://standards.iteh.ai/catalog/standards/sist/ac87c37c-0dee-4c2f-951ac9d6b4c0be7b/sist-iso-5664-1996

UDC 543.342 : 543.24

Ref. No. ISO 5664-1984 (E)

Descriptors : water, tests, determination of content, ammonium ion, distillation method, titration.

SIST ISO 5664:1996

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5664 was developed by Technical Committee ISO/TC 147, Water quality, and was circulated to the member bodies in December 1982. eh.ai)

It has been approved by the member bodies of the following countries:

| Australia | https://standards.iteh.ai/catalog France c9d6b4c0l | z/standards/sist/ac87c37c-0dee-4c2f-951a- New Zealand Dedy/sta-so-3664-1996 |
|---------------------|---|---|
| Austria | Hungary | Poland |
| Belgium | India | Romania |
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| Canada | Iraq | Spain |
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The member body of the following country expressed disapproval of the document on technical grounds:

Germany, F.R.

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Water quality — Determination of ammonium — Distillation and titration method

1 Scope

This International Standard specifies a distillation and titration method for the determination of ammonium in raw, potable and waste water.

2 Field of application

2.1 Range

An ammonium nitrogen content of up to 10 mg in the test portion may be determined. Using a 10 ml test portion, this cor-**Q 4.1.2 Distillation method** responds to a sample concentration of up to $\rho_{\rm N} = 1\,000$ mg/l.

2.2 Limit of detection

4.1 Water, ammonium-free, prepared by one of the following methods.

4.1.1 Ion exchange method

Pass distilled water through a column of strongly acidic cationexchange resin (in the hydrogen form) and collect the eluate in a glass bottle provided with a well-fitting glass stopper. Add about 10 g of the same resin to each litre of eluate for storage purposes.

(standards.itAdd0, 10 ± 0,01 ml of sulfuric acid solution ($\rho = 1,84$ g/ml) to 1 000 ± 10 ml of distilled water and redistil in an all glass apparatus. Discard the first 50 ml of distillate, and then collect the

A practically determined (4 degrees of freedom) limit of detection, using a 250 ml test portion, is $\rho_N = 0,2$ mg/f. $\rho_N = 0,2$ mg/f.

2.3 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.

2.4 Interferences

The main possible interference arises from urea which, under the conditions specified, will distil as ammonia thus causing high results. Interference will also arise from volatile amines which will distil and react with the acid during the titration, thus causing high results. Chloramines present in chlorinated water samples will be determined in this manner.

3 Principle

Adjustment of the pH of a test portion to within the range 6,0 to 7,4. Addition of magnesium oxide to produce mildly alkaline conditions, distillation of the liberated ammonia and collection in a receiving flask containing boric acid solution. Titration of the ammonium in the distillate with standard volumetric acid solution using boric acid/indicator solution.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water prepared as described in 4.1.

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

4.3 Hydrochloric acid, standard volumetric solution, c(HCI) = 0,10 mol/l.

Prepare this solution by diluting the hydrochloric acid (4.2). Standardize it by normal analytical procedures. Alternatively, commercial solutions of guaranteed concentration may be used.

4.4 Hydrochloric acid, standard volumetric solution, c(HCI) = 0.02 mol/l.

Prepare this solution by diluting the hydrochloric acid (4.2). Standardize it by normal analytical procedures. Alternatively, commercial solutions of guaranteed concentration, or a diluted solution of the standard volumetric hydrochloric acid (4.3), may be used.

4.5 Boric acid/indicator, solution.

4.5.1 Dissolve 0.5 ± 0.1 g of water-soluble methyl red in about 800 ml of water and dilute to 1 litre.

4.5.2 Dissolve 1,5 \pm 0,1 g of methylene blue in about 800 ml of water and dilute to 1 litre.

4.5.3 Dissolve 20 \pm 1 g of boric acid (H₃BO₃) in warm water. Cool to room temperature. Add 10 \pm 0,5 ml of the methyl red indicator solution (4.5.1) and 2,0 \pm 0,1 ml of the methylene blue solution (4.5.2) and dilute to 1 litre.

4.6 Bromothymol blue, 0,5 g/l indicator solution.

Dissolve 0.5 ± 0.02 g of bromothymol blue in water and dilute to 1 litre.

4.7 Hydrochloric acid, 1 % (V/V) solution.

Dilute 10 \pm 1 ml of the hydrochloric acid (4.2) to 1 litre with water.

4.8 Sodium hydroxide solution, 1 mol/l.

Dissolve 40 \pm 2 g of sodium hydroxide in about 500 ml of water. Cool to room temperature and dilute to 1 litre.

4.9 Light magnesium oxide, carbonate-free.

Heat the magnesium oxide at 500 °C to remove carbonate.

Anti-bumping granules. 4.10

4.11 Anti-foaming agent, for example paraffin wax chips.

SIST ISO NOTE1996 chlorine is present in the test portion, a few small crystals of https://standards.iteh.ai/catalog/stand.sodiumthiosulfate.should.be_added in order to remove it.

5 Apparatus

Ordinary laboratory apparatus, and

Distillation apparatus, incorporating a distillation flask of capacity 800 to 1 000 ml attached to an anti-splash head and a vertical condenser arranged so that the outlet can be immersed in absorbent solution.

NOTE ON PRELIMINARY CLEANING OF THE DISTILLATION APPARATUS

Carry out this procedure whenever the apparatus has been out of use for more than a few days.

Place about 350 ml of the ammonium-free water (4.1) in the distillation flask. Add a few anti-bumping granules (4.10), assemble the apparatus, and distil until at least 100 ml of the water has been collected. Discard the distillate and the residue in the distillation flask.

Sampling and samples 6

Laboratory samples shall be collected in polyethylene or glass bottles. They should be analysed as quickly as possible, or else stored at between 2 and 5 °C until analysed. Acidification with sulfuric acid to < pH 2 may also be used as an aid to preservation, provided that possible contamination of the acidified sample by absorption of any atmospheric ammonia is avoided.

7 Procedure

7.1 Selection of volume of test portion

If the approximate ammonium content of the sample is known, select the volume of the test portion according to table 1.

Table 1

| Ammonium concentration, $\varrho_{ m N}$ | Volume of the test portion* |
|--|-----------------------------|
| mg/l | ml |
| up to 10 | 250 |
| 10 to 20 | 100 |
| 20 to 50 | 50 |
| 50 to 100 | 25 |

When using the standard volumetric hydrochloric acid (4.3) for the titration.

7.2 Determination

7.2.1 Transfer 50 \pm 5 ml of the boric acid/indicator solution (4.5) into the receiving flask of the distillation apparatus. Ensure iTeh STANDA that the delivery tip of the condenser is below the surface of the (standar boric acid solution. Measure the selected volume of test portion (see 7.1) into the distillation flask.

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Add a few drops of the bromothymol blue indicator solution (4.6) and, if necessary, adjust the pH to within the range 6,0 (indicator yellow) to 7,4 (indicator blue) using the sodium hydroxide solution (4.8) or the hydrochloric acid (4.7), as appropriate. Then make up the total volume in distillation flask to about 350 ml with the ammonium-free water (4.1).

Add to the distillation flask 0,25 \pm 0,05 g of the light magnesium oxide (4.9) and a few anti-bumping granules (4.10). [The addition of anti-foaming agent (4.11) may be necessary with some waste water samples.] Immediately attach the distillation flask to the apparatus.

7.2.2 Heat the distillation flask so that distillate collects at a rate of about 10 ml/min. Stop the distillation when about 200 ml have been collected.

7.2.3 Titrate the distillate to the purple end-point using the standard volumetric hydrochloric acid (4.4) and record the volume used.

NOTES

1 The standard volumetric hydrochloric acid (4.3) may be used for titration of distillates from samples having high ammonium contents.

2 Ammonia can be titrated as it distils into the receiving flask. If protracted collection of ammonia is thus revealed, it may indicate the presence of interfering substances which are undergoing slow hydrolysis to yield ammonia.

7.3 Blank test

Carry out a blank test by proceeding as described in 7.2, but replacing the test portion with about 250 ml of the ammoniumfree water (4.1).

Expression of results 8

8.1 Method of calculation

The ammonium nitrogen concentration, $\varrho_{\rm N},$ expressed in milligrams per litre, is given by the equation

$$\varrho_{\rm N} = \frac{V_1 - V_2}{V_0} \times c \times 14,01 \times 1000$$

where

(see 7.3);

 V_0 is the volume, in millilitres, of the test portion (see 7.1);

 V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid used for titration, I 🔊 I Al

c is the exact concentration, in moles per litre, of the hydrochloric acid used for the titration;

14,01 is the atomic mass, in grams per mole, of nitrogen.

The result may be expressed as the mass concentration of nitrogen, ϱ_N , ammonia, ϱ_{NH_3} , or ammonium ion, $\varrho_{NH_4}^+$, in milligrams per litre, or as the amount of substance concentration of ammonium ion, $c(NH_A^+)$, in micromoles per litre. The appropriate conversion factors are given in table 2.

| Table | 2 |
|-------|---|
|-------|---|

| | ₽ _N | ₽ _{NH3} | _{ен4} + | c(NH ₄ ⁺) |
|--|----------------|------------------|------------------|----------------------------------|
| | mg/l | mg/l | mg/l | µmol/l |
| $\varrho_{\rm N} = 1 {\rm mg/l}$ | 1 | 1,216 | 1,288 | 71,4 |
| $\varrho_{\rm NH_3} = 1 \rm mg/l$ | 0,823 | 1 | 1,059 | 58,7 |
| $\varrho_{\rm NH4}^{+} = 1 \rm mg/l$ | 0,777 | 0,944 | 1 | 55,4 |
| $c(\mathrm{NH}_4^+) = 1 \mu \mathrm{mol/l}$ | 0,014 | 0,017 | 0,018 | 1 |

Example:

An ammonium ion concentration $\varrho_{\rm NH4}^{+}$, of 1 mg/l corresponds to a nitrogen concentration of 0,777 mg/l.

8.2 Reproducibility V_2 is the volume, in millilitres, of the standard volumetric S ren.ai

hydrochloric acid used for the titration in the blank test Reproducibility standard deviations have been determined as indicated in table 3. SIST ISO 5664:

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| Sample | Ammonium content, _{ຂຸN} | Volume of test portion | Standard deviation | Degrees of freedom | |
|-------------------|-------------------------------------|---------------------------|--------------------|--------------------|--|
| | mg/l | ml | mg/l | _ | |
| Standard solution | 4,0 | 250 | 0,23 | 10 | |
| Standard solution | 40 | 250 | 0,56 | 11 | |
| Settled sewage | 35 | 100 | 0,70 | 16 | |
| Sewage effluent | 1,8 | 25 | 0,16 | 11 | |

Information from the United Kingdom.

Test report 9

The test report shall include the following information:

- a) reference to this International Standard;
- all details required for the complete identification of the sample; b)
- details concerning the storage and preservation of the laboratory sample before analysis; c)
- a statement of the repeatability achieved by the laboratory when using this method; d)
- e) the result and the method of expression used;

f) details of any deviation from the procedure specified in this International Standard or any other circumstance that may have influenced the result.