
International Standard



5664

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ 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 — 1984-05-15

UDC 543.342 : 543.24

Ref. No. ISO 5664-1984 (E)

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

Foreword

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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.

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

Australia	France	New Zealand
Austria	Hungary	Poland
Belgium	India	Romania
Brazil	Iran	South Africa, Rep. of
Canada	Iraq	Spain
China	Italy	Sweden
Czechoslovakia	Japan	Switzerland
Denmark	Korea, Dem. P. Rep. of	Thailand
Egypt, Arab Rep. of	Mexico	United Kingdom
Finland	Netherlands	USSR

The member body of the following country expressed disapproval of the document on technical grounds:

Germany, F.R.

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 corresponds to a sample concentration of up to $\rho_N = 1\,000\text{ mg/l}$.

2.2 Limit of detection

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

2.3 Sensitivity

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

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.

4.1 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 cation-exchange 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.

4.1.2 Distillation method

Add $0,10 \pm 0,01\text{ ml}$ of sulfuric acid solution ($\rho = 1,84\text{ g/ml}$) to $1\,000 \pm 10\text{ ml}$ of distilled water and redistil in an all glass apparatus. Discard the first 50 ml of distillate, and then collect the distillate in a glass bottle provided with a well-fitting glass stopper. Add about 10 g of strongly acidic, cation exchange resin (in the hydrogen form), to each litre of collected distillate.

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

4.3 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 0,10\text{ mol/l}$.

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, $c(\text{HCl}) = 0,02\text{ mol/l}$.

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 $0,5 \pm 0,1\text{ g}$ of water-soluble methyl red in about 800 ml of water and dilute to 1 litre.

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