International Standard 5663

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Water quality — Determination of Kjeldahl nitrogen — Method after mineralization with selenium

Qualité de l'eau — Dosage de l'azote Kjeldahl — Méthode après minéralisation au sélénium

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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 5663 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	Germany, F.R.	Norway
Austria	Hungary	Poland
Belgium	India	Romania
Brazil	Iran	South Africa, Rep. of
Canada	Iraq	Spain
China	Italy	Sweden
Czechoslovakia	Korea, Dem. P. Rep. of	Switzerland
Denmark	Mexico	Thailand
Egypt, Arab Rep. of	Netherlands	USSR
France	New Zealand	

The member bodies of the following countries expressed disapproval of the document on technical grounds:

Japan
United Kingdom

Printed in Switzerland

Water quality — Determination of Kjeldahl nitrogen — Method after mineralization with selenium

1 Scope and field of application

1.1 Substance determined

This International Standard specifies a method for the determination of nitrogen by a Kjeldahl-type method. Only trivalent negative nitrogen is determined. Organic nitrogen in the form of azide, azine, azo, hydrazone, nitrite, nitro, nitroso, oxime or semicarbazone is not determined quantitatively. Nitrogen may be incompletely recovered from heterocyclic nitrogen compounds.

1.2 Type of sample

This method is applicable to the analysis of raw, potable and waste waters.

1.3 Range

A Kjeldahl nitrogen content, ϱ_N, 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 ϱ_N = 1 000 mg/l.

1.4 Limit of detection

A practically determined (4 degrees of freedom) limit of detection, using a 100 ml test portion, is ϱ_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 ϱ_N = 2,8 mg/l.

2 Reference

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

3 Definition

For the purpose of this International Standard, the following definition applies:

Kjeldahl nitrogen: The content of organic nitrogen and ammoniacal nitrogen in a sample determined after mineralization.

It does not include nitrate and nitrite nitrogen, and does not necessarily include all organically bound nitrogen.

4 Principle

Mineralization of the sample to form ammonium sulfate, from which ammonia is liberated and distilled for subsequent determination by titration.

Conversion of the nitrogen compounds responding to the test to ammonium sulfate by mineralization of the sample with sulfuric acid, containing a high concentration of potassium sulfate in order to raise the boiling point of the mixture, in the presence of selenium which acts as a catalyst.[1)]

Liberation of ammonia from the ammonium sulfate by the addition of alkali and distillation into boric acid/indicator solution.

Determination of ammonium ion in the distillate by titration with standard acid.

Alternatively, direct determination of ammonium ion in the mineralizate by spectrometry at 655 nm. (See clause 11.)

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water prepared as described in 5.1.

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

5.1.1 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 collected eluate for storage purposes.

1) Selenium has been selected as the catalyst in preference to mercury because of concern in many countries about the toxicity of mercury. However, the toxicity of selenium must not be overlooked. See 11.2 for a suggested procedure for removal of selenium from mineralization residues.