International Standard 7150/1

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Water quality — Determination of ammonium — Part 1: Manual spectrometric method

Qualité de l'eau — Dosage de l'ammonium — Partie 1: Méthode spectrométrique manuelle

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Foreword

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Water quality — Determination of ammonium — Part 1 : Manual spectrometric method

1 Scope and field of application

1.1 Substance determined

This part of ISO 7150 specifies a manual spectrometric method for the determination of ammonium in water.

NOTE — An automated spectrometric method for the determination of ammonium will form the subject of ISO 7150/2.

1.2 Type of sample

The method is applicable to the analysis of potable water, and most raw and waste waters. Application to excessively coloured or saline waters shall be preceded by distillation (see clause 10).

For interferences, see clause 9.

1.3 Range

An ammonium nitrogen concentration, ϱ_N of up to 1 mg/l, using the maximum test portion of 40 ml, can be determined. Much higher concentrations can be determined by taking smaller test portions.

1.4 Limit of detection[1]

When using cells of optical path length 40 mm and a 40 ml test portion, the limit of detection lies within the range ϱ_N = 0,003 to 0,008 mg/l.

1.5 Sensitivity[1]

Using a 40 ml test portion and a cell of optical path length 40 mm, ϱ_N = 0,200 mg/l gives an absorbance of about 0,69 units.

Using a 40 ml test portion and a cell of optical path length 10 mm, ϱ_N = 0,750 mg/l gives an absorbance of about 0,65 units.

2 Reference

ISO 5664, *Water quality — Determination of ammonium — Distillation and titration method.*

3 Principle

Spectrometric measurement at about 655 nm of the blue compound formed by reaction of ammonium with salicylate and hypochlorite ions in the presence of sodium nitrosopentacyanoferrate(III) (sodium nitroprusside).

Hypochlorite ions are generated *in situ* by the alkaline hydrolysis of *N*, *N'*-dichloro-1,3,5-triazine-2,4,6 (1H, 3H, 5H)-trione, sodium salt (sodium dichloroisocyanurate). Reaction of the chloramine with sodium salicylate takes place at pH 12,6 in the presence of sodium nitroprusside. Any chloramines present in the sample are quantitatively determined as a consequence. Sodium citrate is incorporated in the reagent to mask interference from cations, notably calcium and magnesium.

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 collected eluate for storage purposes.

4.1.2 Distillation method

Add 0,10 ± 0,01 ml of sulfuric acid (ϱ = 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 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.

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[1] Data from a United Kingdom interlaboratory exercise involving five participants.

4.2 Colour reagent.

Dissolve 130 ± 1 g of sodium salicylate ($C_7H_6O_3Na$) and 130 ± 1 g of trisodium citrate dihydrate ($C_6H_5O_7Na_3 \cdot 2H_2O$) in water (4.1) in a 1 000 ml one-mark volumetric flask. Add sufficient water to give a total liquid volume of about 950 ml and then add 0,970 ± 0,005 g of sodium nitrosopentacyanoferrate(III) dihydrate {sodium nitroprusside, $[Fe(CN)_5NO]Na_2 \cdot 2H_2O$}. Dissolve the solid and then dilute to the mark with water.

Stored in an amber glass bottle, this reagent is stable for at least 2 weeks.

4.3 Sodium dichloroisocyanurate, solution.

Dissolve 32,0 ± 0,1 g of sodium hydroxide in 500 ± 50 ml of water (4.1). Cool the solution to room temperature and add 2,00 ± 0,02 g sodium dichloroisocyanurate dihydrate ($C_3N_3O_3Cl_2Na \cdot 2H_2O$) to the solution. Dissolve the solid and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water.

Stored in an amber glass bottle, this reagent is stable for at least 2 weeks.

4.4 Ammonium nitrogen, standard solution, $\varrho_N = 1\,000$ mg/l.

Dissolve 3,819 ± 0,004 g of ammonium chloride (dried at 105 °C for at least 2 h) in about 800 ml of water (4.1) in a 1 000 ml one-mark volumetric flask. Dilute to the mark with water.

1 ml of this standard solution contains 1 mg of ammonium nitrogen.

Stored in a stoppered glass bottle, this solution is stable for at least 1 month.

4.5 Ammonium nitrogen, standard solution, $\varrho_N = 100$ mg/l.

Pipette 100 ml of ammonium nitrogen standard solution (4.4) into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water.

1 ml of this standard solution contains 0,1 mg of ammonium nitrogen.

Stored in a stoppered glass bottle, this solution is stable for 1 week.

4.6 Ammonium nitrogen, standard solution, $\varrho_N = 1$ mg/l.

Pipette 1 ml of ammonium nitrogen standard solution (4.5) into a 100 ml one-mark volumetric flask. Dilute to the mark with water.

1 ml of this standard solution contains 1 µg of ammonium nitrogen.

Prepare this solution immediately before use.

4.7 Cleaning solution.

Dissolve 100 ± 2 g of potassium hydroxide in 100 ± 2 ml of water. Cool the solution and add 900 ± 50 ml of 95 % (*V*/*V*) ethanol.

Store the solution in a polyethylene bottle.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrometer, capable of operating at a wavelength of 655 nm with cells of optical path length between 10 and 50 mm.

5.2 Water-bath or **incubator,** capable of being maintained at 25 ± 1 °C.

NOTE ON CLEANING OF GLASSWARE

All glassware shall be carefully cleaned using the cleaning solution (4.7) followed by thorough rinsing with water (4.1).

6 Sampling and samples

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 Test portion

The maximum test portion volume is 40 ml, which can be used for the determination of ammonium nitrogen concentrations up to $\varrho_N = 1$ mg/l.

Smaller test portions may be used as appropriate in order to accommodate much higher ammonium nitrogen concentrations. Laboratory samples containing suspended matter should be allowed to settle, or filtered by gravity through a pre-rinsed glass fibre paper before taking the test portion. Alternatively, prior distillation of the sample may be used (see clause 10).

7.2 Preparation of test solution

Pipette the test portion (7.1) into a 50 ml one-mark volumetric flask, and, if necessary, dilute to 40 ± 1 ml with water (4.1).

7.3 Determination

7.3.1 Formation of the absorbing compound

Add 4,00 ± 0,05 ml of colour reagent (4.2) and mix well. Then add 4,00 ± 0,05 ml of sodium dichloroisocyanurate solution (4.3) and mix well.

NOTE — After this addition the pH of the solution should be 12,6 ± 0,1. Extremes of acidity or alkalinity in the sample may cause deviation.

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