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**Water quality — Determination of ammonium nitrogen in water — Small-scale sealed tube method**

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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 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

Ammonium nitrogen is one of the possible forms of nitrogen present in water. It can come directly from industrial or public waste, from fertilizers or can be generated by the microbial breakdown of urea and proteins under anaerobic conditions. Ammonium nitrogen can also be found naturally in water in aquifers with low flow velocities, which are confined and contain organic matter.

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# Water quality — Determination of ammonium nitrogen in water — Small-scale sealed tube method

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

## 1 Scope

This document specifies a method for the determination of ammonium nitrogen ( $\text{NH}_4\text{-N}$ ) in drinking water, groundwater, surface water, wastewater, bathing water and mineral water using the small-scale sealed tube method. The result can be expressed as  $\text{NH}_4$  or  $\text{NH}_4\text{-N}$  or  $\text{NH}_3$  or  $\text{NH}_3\text{-N}$ .

NOTE 1 In the habitual language use of sewage treatment and on the displays of automated sealed-tube test photometers or spectrophotometers,  $\text{NH}_4$  without indication of the positive charge has become the common notation for the parameter ammonium. This notation is adopted in this document even though not being quite correct chemical nomenclature.

This method is applicable to ( $\text{NH}_4\text{-N}$ ) concentration ranges from 0,01 mg/l to 1 800 mg/l of  $\text{NH}_4\text{-N}$ . The measuring ranges of concentration can vary depending on the type of small-scale sealed tube method of different manufacturers. Concentrations even slightly higher than the upper limit indicated in the manufacturers manual relating to the small-scale sealed tube method used, cannot be reported as accurate results. It is up to the user to choose the small-scale sealed tube test with the appropriate application range or to adapt samples with concentrations exceeding the measuring range of a test by preliminary dilution.

NOTE 2 The results of a small-scale sealed tube are most precise in the middle of the application range of the test.

All manufacturers' methods are based on the Berthelot reaction and its modifications to develop indophenol blue colour. Reagents mixtures can differ slightly based on manufacturers small-scale sealed tube method, see Clause 9. This method is applicable to non-preserved samples by using small-scale sealed tubes for the determination of drinking water, groundwater, surface water, wastewater and to preserved samples. The method is applicable to samples with suspended materials if these materials are removable by filtration.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste water*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods ~~and estimation of performance characteristics~~ — Part 1: ~~Statistical evaluation of the~~ Linear calibration function*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain ~~terminological~~terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

#### 3.1 small-scale sealed tube

glass tube commercially available on the market prefilled by different manufacturers with reagent(s) to develop a colour to be read by a photometer or spectrophotometer.

### 4 Principle

This method is based on the principle of Berthelot reaction and its modification to develop indophenol blue colour. In strongly alkaline solution, ammonia ions react with dichloroisocyanurate and salicylate ions or chlorophenol ions, forming blue indophenol. The presence of sodium nitroprusside as catalyst promotes the development of the blue colour. Small-scale sealed tube is to be read on a photometer or spectrophotometer.

The method serves to obtain the concentration of ammonium nitrogen present in the sample.

### 5 Interferences

Typical interferences are due to both cations and anions. Examples for maximum tolerable concentration values are listed in Table 1. These values refer to both small-scale sealed tube methods applicable at high concentrations and to those applicable at low concentrations of NH<sub>4</sub>-N.

Every manufacturer of small-scale sealed tubes shall provide information about interference levels above which the ion interferes. The concentration of interfering substances can depend on the ratio of sampled volume and predosed reagents, in the small-scale sealed tube.

**Table 1 — Example of interfering ions**

Ion	Maximum tolerable concentration mg/l
Cl <sup>-</sup>	1 000
SO <sub>4</sub> <sup>2-</sup>	1 000
K <sup>+</sup>	500

NO <sub>3</sub> <sup>-</sup>	250
Na <sup>+</sup>	500
CO <sub>3</sub> <sup>2-</sup>	50
Ca <sup>2+</sup>	500
Cu <sup>2+</sup>	50
Cr <sup>3+</sup>	50
Co <sup>2+</sup>	50
Zn <sup>2+</sup>	50
Cr(VI)	50

Primary amines can react under analysis conditions and lead to overestimated NH<sub>4</sub>-N values. All reducing substances interfere and lead to underestimated results. The presence of urea is tolerated up to concentrations which are 10 000 times greater than those of the ammonia present in the sample to be analysed.

Turbidity, colour and high salinity levels can interfere both in the instrumental measurement and in the speed of colour development.

As specified in ISO 5667-3, preliminary filtration is necessary using filters with 0,45 µm pore width. The filtration has to be performed on-site. Filtration shall be carried out under pressure and not under vacuum, to avoid ammonia leakage. Before filtering, the filter shall be washed with water with the same pH as the sample. Generally, it can be done with the sample itself by discarding the first millilitres of sample, already filtered.

When measuring a reagent blank value, follow the normal procedure but use pure water (7.1) instead of sample.

When measuring a blank value caused by a coloured sample, add the sample to a small-scale sealed tube but do not add further reagents. Then follow the normal procedure.

In some cases, interferences can be eliminated using appropriate dilutions such that interfering ions fall below the maximum tolerable concentration and NH<sub>4</sub>-N remains within the measurement range.

It is the laboratory's responsibility to identify interferences by, for example, spiking with reference material containing known amounts of ammonium nitrogen to the sample containing the suspected ion or to a solution of known concentration of the suspected ion.

In case of higher concentration of interfering substances, highly coloured wastewater or turbid suspension which cannot be easily filtered or diluted, as described above, proceed with a distillation as described in ISO 5664. An alternative method for distillation is described in Annex A.

## 6 Sampling and sample preparation

For methods of sampling and storing samples before analysis—see, [proceed according to](#) ISO 5667-1, ISO 5667-3 and ISO 5667-10.

Persons performing the analysis shall be aware of the risks associated with the method.

It shall be noted that, in the case of acidification of unknown samples, toxic gases such as HCN and H<sub>2</sub>S [may can](#) form. Work in a fume cupboard.

Non-preserved samples shall be analysed as soon as possible, [following in accordance with](#) ISO 5667-3. Longer storage periods require stabilization by acidification with sulfuric acid to pH  $\leq 2$ ; if the sample