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Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique)

*Qualité de l'eau — Dosage de l'arsenic — Méthode par spectrométrie
d'absorption atomique (technique hydrure)*



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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11969 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical, biochemical methods*.

Annexes A and B of this International Standard are for information only.

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Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique)

WARNING — Arsenic and arsenic compounds are toxic and are recognized as human carcinogens. Avoid any exposure by inhalation. Personal protection must be used in all cases where exposure to arsenic or arsenic compounds is possible.

1 Scope

This International Standard specifies a method for the determination of arsenic including organically bound arsenic in drinking waters, ground waters and surface waters, in a concentration range from 1 µg/l to 10 µg/l.

Higher concentrations can be determined by using a suitable dilution of the water sample.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*.

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques*.

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*.

3 Principle

The method is based on the atomic absorption measurement of arsenic generated by the thermal decomposition of arsenic(III) hydride.

Under the conditions of this method, only As(III) is quantitatively converted to the hydride. To avoid errors in determination, other oxidation states need to be converted to As(III) prior to the determination.

As(III) is reduced to gaseous arsenic(III) hydride (AsH₃) by reaction with sodium tetrahydroborate in a hydrochloric acid medium.

The absorbance is determined at a wavelength of 193,7 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade.

The arsenic content of the water and the reagents shall be negligible, compared with the lowest concentration to be determined.

4.1 Sulfuric acid (H₂SO₄), $\rho = 1,84$ g/ml.

4.2 Hydrochloric acid (HCl), $\rho = 1,15$ g/ml.

4.3 Hydrogen peroxide (H₂O₂), $w = 30$ % (m/m).

4.4 Sodium hydroxide (NaOH).

4.5 Sodium tetrahydroborate solution.

Dissolve 1 g of sodium hydroxide (4.4) in about 20 ml of water. Add 3 g of sodium tetrahydroborate (NaBH_4). Dilute to 100 ml with water.

Prepare the solution on the day of use.

NOTE 1 For flow-through systems, it is recommended to follow the instructions of the manufacturer. A solution containing 0,5 % of sodium tetrahydroborate and 0,5 % of sodium hydroxide is suitable. This solution is stable for at least one week.

4.6 Potassium iodide-ascorbic acid solution.

Dissolve 3 g of potassium iodide (KI) and 5 g of L(+)-ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in 100 ml of water.

Prepare the solution on the day of use.

NOTE 2 It is unnecessary to use ascorbic acid if a 20 % solution of potassium iodide is used.

4.7 Arsenic stock solution, corresponding to 1 000 mg of As per litre.

Place 1,320 g of arsenic(III) oxide (As_2O_3), in a volumetric flask of nominal capacity 1 000 ml. Add 2 g of sodium hydroxide (4.4) and dissolve in a small quantity of water. Dilute to volume with water.

This solution is stable for at least 1 year.

Arsenic stock solutions are commercially available. If the stock solution contains As(V), the standard solutions shall be treated in the same way as the sample for the reduction step (8.3.2).

4.8 Arsenic standard solution 1, corresponding to 10 mg of As per litre.

Pipette 10 ml of arsenic stock solution (4.7) into a volumetric flask of nominal capacity 1 000 ml. Add 20 ml of hydrochloric acid (4.2) and dilute to volume with water.

The solution is stable for about 1 month.

If a stock solution of arsenic(V) is used, arsenic(V) shall be reduced to arsenic(III) according to 8.3.2, before dilution to 1 000 ml.

4.9 Arsenic standard solution 2, corresponding to 0,1 mg of As per litre.

Pipette 10 ml of arsenic standard solution 1 (4.8) into a volumetric flask of nominal capacity 1 000 ml. Add 20 ml of hydrochloric acid (4.2) and dilute to volume with water.

Prepare the solution on the day of use.

5 Apparatus

Usual laboratory apparatus and

5.1 Atomic absorption spectrometer, fitted with a hydride system, and a suitable radiation source for the determination of arsenic, for example electrodeless discharge lamp or a hollow cathode lamp with a background correction facility, if necessary.

5.2 Gas supply, with argon or nitrogen.

5.3 Glassware, to be cleaned immediately before use with warm, dilute nitric acid [10 % (V/V)] and rinsed with water.

6 Sampling

Take samples according to ISO 5667-1 and ISO 5667-2.

Collect samples in polyethylene or borosilicate glass containers which have been previously cleaned with nitric acid [e.g. 10 % (V/V)] and then rinsed with water.

On site, add 20 ml of hydrochloric acid (4.2) to each 1 000 ml of the water sample.

If the pH of the sample is still greater than 2, add more hydrochloric acid until the pH is 2 or less.

For sample conservation, see ISO 5667-3.

7 Interferences

Most organic materials interfere with the arsenic determination. They shall be removed prior to the analysis by the digestion procedure described in 8.3.1. Samples forming foams when tetrahydroborate is added shall be pretreated (e.g. with an anti-foaming agent or by complete digestion). When an anti-foaming agent is added, it is also necessary to add it to the blank and calibration solutions.

Annex A gives details of the effect of potential interfering substances on the determination of arsenic. These results were obtained at the Laboratory of the Government Chemist (UK). Of the substances tested, only concentrations of copper greater than 2,0 mg/l, antimony greater than 0,2 mg/l, selenium greater than 0,05 mg/l and nitrate greater than 100 mg/l interfere at arsenic concentration levels of 1,0 µg/l.