International Standard 6595

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Water quality — Determination of total arsenic — Silver diethyldithiocarbamate spectrophotometric method

Qualité de l'eau — Dosage de l'arsenic total — Méthode spectrophotométrique au diéthyldithiocarbamate d'argent

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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.

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International Standard ISO 6595 was developed by Technical Committee ISO/TC 147, *Water quality*, and was circulated to the member bodies in November 1980.

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

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Austria	Germany, F.R.	South Africa, Rep. of
Belgium	Hungary	Spain
Brazil	India	Sweden
Canada	Italy	United Kingdom
Czechoslovakia	Korea, Rep. of	USA
Denmark	Mexico	USSR
Finland	Netherlands	

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

Japan
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Water quality — Determination of total arsenic — Silver diethyldithiocarbamate spectrophotometric method

The procedure specified in this International Standard is intended to be carried out by qualified chemists or by other suitably trained and/or supervised personnel. Attention is especially drawn to the toxic nature of arsenic and its solutions and of other reagents used in this method of analysis and to the need to take particular care in the handling and disposal of solutions. Pyridine and chloroform should be handled in a well-ventilated fume cupboard. Ephedrine is a scheduled drug and should be handled in accordance with appropriate regulations.

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1 Scope and field of application

This International Standard specifies a silver diethyldithiocarbamate spectrophotometric method for the determination of arsenic in water and waste water.

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It is applicable for the determination of arsenic concentrations in the range from 0,001 to 0,1 mg/l. In the case of arsenic compounds which are difficult to decompose, a method of digestion is described in the annex, clause A.1. By appropriate dilution of the test portion with arsenic-free water, higher concentrations of arsenic may also be determined.

Antimony interferes with the determination (see the annex, clause A.2). Chromium, cobalt, molybdenum, nickel, mercury, silver and platinum, in concentrations up to 5 mg/l, do not interfere with the determination.

2 Definition

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

total arsenic : The total amount of the element arsenic, in elementary form or bound in inorganic or organic compounds.

NOTE — Depending on the redox potential and the pH of the water, arsenic may be present in the trivalent state [for example as arsenite ions (AsO_3^{3-})], in the pentavalent state [for example as arsenate ions (AsO_4^{3-})], or as organically bound arsenic.

3 Principle

3.1 Oxidation of organic compounds or sulphides by heating with potassium permanganate and potassium peroxodisulphate.

3.2 Reduction of pentavalent arsenic to the trivalent state.

3.3 Reduction of the trivalent arsenic by nascent hydrogen in an acidic medium to arsenic trihydride (arsine).

3.4 Absorption of the arsine in a solution of silver diethyldithiocarbamate in either chloroform or pyridine, and spectrophotometric measurement of the red-violet complex thus formed, at a wavelength of 510 or 525 nm, respectively, according to the solvent.

4 Reagents

Unless otherwise specified, all reagents shall be of recognized analytical grade and the water used should be distilled or deionized water. The arsenic content of the reagents and the water should be negligibly small.

4.1 Sulphuric acid, ϱ = 1,84 g/ml.

4.2 Sulphuric acid solution, $c(1/2\ H_2SO_4)$ = 2 mol/l.

4.3 Sodium hydroxide solution, $c(NaOH)$ = 2 mol/l.

Store in a polyethylene bottle.

4.4 Potassium permanganate, 50 g/l solution.

Dissolve 50 g of potassium permanganate in water and dilute to 1 000 ml.

Take care to ensure complete dissolution of the reagent.

Store in a dark glass bottle.

4.5 Potassium peroxodisulphate, 40 g/l solution.

Dissolve 40 g of potassium peroxodisulphate in water and dilute to 1 000 ml.

4.6 Hydroxylamine hydrochloride, 100 g/l solution.

Dissolve 10 g of hydroxylamine hydrochloride in water and dilute to 100 ml.

The solution is stable for at least 1 month.

4.7 Potassium iodide, 150 g/l solution.

Dissolve 15 g of potassium iodide in water and dilute to 100 ml.

Store in a dark glass bottle.

The solution is stable for at least 1 month.

4.8 Tin chloride solution.

Dissolve 55 g of tin(II) chloride dihydrate in 25 ml of concentrated hydrochloric acid (ϱ = 1,19 g/ml) and dilute to 100 ml with water.

The solution is stable if stored in a refrigerator.

4.9 Absorption solution A.

Dissolve 0,500 g of silver diethyldithiocarbamate and 0,330 g of 1-ephedrine in chloroform and dilute with chloroform to 200 ml.

This solution is stable for at least 1 month if stored in a tightly-stoppered, dark glass bottle.

4.10 Absorption solution B.

Dissolve 1,000 g of silver diethyldithiocarbamate in pyridine and dilute with pyridine to 200 ml.

Store in a dark glass bottle.

4.11 Zinc, coarse powder, of particle size 0,5 to 1 mm.

4.12 Copper(II) sulphate solution.

Dissolve 15 g of copper(II) sulphate pentahydrate in water and dilute to 100 ml.

4.13 Arsenic, standard solution corresponding to 350 mg of As per litre.

Dissolve exactly 0,462 0 g of arsenic(III) oxide (As_2O_3), previously dried over silica gel to constant mass, in 12 ml of the sodium hydroxide solution (4.3). Neutralize with the sulphuric acid solution (4.2) and dilute to 1 000 ml with water.

1 ml of this standard solution contains 0,35 mg of arsenic.

4.14 Arsenic, standard solution corresponding to 3,5 mg of As per litre.

Dilute 10 ml of the standard arsenic solution (4.13) with water to 1 000 ml.

1 ml of this standard solution contains 3,5 µg of arsenic.

The solution is stable only for a few days.

Prepare the solution just before use.

4.15 Arsenic, standard solution corresponding to 0,35 mg of As per litre.

Dilute 1 ml of the standard arsenic solution (4.13) with water to 1 000 ml.

1 ml of this standard solution contains 0,35 µg of arsenic.

Prepare the solution just before use.

5 Apparatus

Usual laboratory equipment and

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5.1 Spectrophotometer, equipped with cells of optical path length 10 to 50 mm [for optical path lengths of more than 10 mm, use micro-cells of small total capacity (maximum 5 ml)].

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5.2 Reaction apparatus (as shown in the figure or its equivalent), comprising

— a conical flask, of capacity 500 ml, with a ground glass joint complying with the requirements of ISO 383;

— an absorption tube, with a ground glass joint complying with the requirements of ISO 383.

5.3 Volumetric flask, of capacity 1 000 ml.

5.4 Pipettes, of capacities 1 - 2 - 5 - 10 and 20 ml.

5.5 Measuring cylinders, of capacities 25, 100 and 500 ml.

6 Procedure

6.1 Test portion

Transfer 350 ml of the test sample to a measuring cylinder. If the arsenic content is expected to exceed 0,1 mg/l, take an appropriately smaller test portion and dilute with water to 350 ml.

6.2 Blank test

Carry out a blank test, using the same reagents in the same quantities as used in the determination and following the same procedures, including any pretreatment, but replacing the test portion by 350 ml of arsenic-free water.

6.3 Choice of absorption solution

The choice of absorption solution [A (4.9) or B (4.10)] is left to the discretion of the analyst. Pyridine has an unpleasant odour. It is, however, less volatile than chloroform, and the volume of absorption solution B is less likely to require adjustment during the analysis. The molar absorbance coefficient when using absorption solution B is about 30 % greater than that when using absorption solution A. The same absorption solution shall, therefore, be used in the determination, blank test and for preparation of the calibration graphs.

6.4 Preparation of calibration graphs

6.4.1 Preparation of standard matching solutions

6.4.1.1 Into each of two series of conical flasks (see 5.2), pipette the volumes of the standard arsenic solutions (4.14 and 4.15) shown in the following table, and make up the volume in each flask to 350 ml with water.

Volume of standard arsenic solution (4.14)	Corresponding arsenic content
ml	µg/l
0*	0
1,0	10
2,0	20
5,0	50
10,0	100
Volume of standard arsenic solution (4.15)	
ml	
0*	0
1,0	1
2,0	2
5,0	5
10,0	10
20,0	20

* Blank test of the reagents for calibration.

6.4.1.2 Add to each flask 20 ml of the sulphuric acid (4.1).

6.4.1.3 Add 10 ml of the potassium iodide solution (4.7) and 1 ml of the tin(II) chloride solution (4.8).

6.4.1.4 Transfer 5 ml of absorption solution A (4.9) or of absorption solution B (4.10), as appropriate (see 6.3), to the absorption tube.

Add 1 ml of the copper(II) sulphate solution (4.12) and 15 g of the zinc (4.11) to each flask. Immediately connect the absorption tube to the flask. To ensure that the reaction apparatus is airtight, a small amount of arsenic-free grease may be applied to the ground glass joint.

Allow to stand for 2 h to complete the evolution of arsine. Make up the volume of absorption solution to 5 ml to replace loss by evaporation by adding chloroform (in the case of absorption solution A) or pyridine (in the case of absorption solution B), as appropriate.

Shake the flasks gently from time to time so as to avoid the formation of precipitates in the entry zone of the absorption solution.

If protected from light, the coloured complex is stable for about 2 h; after complete evolution of arsine, carry out the spectrophotometric measurements within this time.

6.4.2 Spectrophotometric measurements

For each of the standard matching solutions (6.4.1.1) in turn, fill a cell with solution from the absorption tube and fill a reference cell with the appropriate absorption solution solvent (chloroform or pyridine, respectively).

Measure the absorbance of the test solution by means of the spectrophotometer (5.1), set at a wavelength of 510 nm when using absorption solution A (4.9) or set at 540 nm when using absorption solution B (4.10).

6.4.3 Plotting the graphs

Correct the measured absorbances of the solutions from the absorption tubes (see 6.4.2) corresponding to each of the standard matching solutions (6.4.1.1) by deducting the absorbance for the reagent blank.

For each series of standard matching solutions corresponding to the two standard arsenic solutions (4.14 and 4.15), plot a graph of the corrected absorbances as ordinates against the corresponding arsenic contents, in micrograms per litre, as abscissae.

Both plots should be linear.

Prepare new calibration graphs frequently and at least each time new reagent is used.

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6.5 Determination

6.5.1 Pretreatment

Transfer the test portion to a conical flask (see 5.2) and add 20 ml of the sulphuric acid (4.1), 5 ml of the potassium permanganate solution (4.4) and 50 ml of the potassium peroxodisulphate solution (4.5). Heat for 2 h at 90 °C (for example on a hot-plate or a water bath). Allow to cool to room temperature and add 20 ml of the hydroxylamine hydrochloride solution (4.6).

NOTE — The amount of oxidizing agent is sufficient for chemical oxygen demands up to 100 mg/l.

6.5.2 Development of colour

Proceed as described in 6.4.1.3 and 6.4.1.4.

6.5.3 Spectrophotometric measurement

Proceed as described in 6.4.2.

7 Expression of results

From the calibration graphs, determine the arsenic concentrations corresponding to the absorbances of the test solution and of the blank test solution. Take any dilution of the test portion (see 6.1) into consideration.

The arsenic content, expressed in milligrams per litre, is given by the formula

$$\frac{(A_2 - A_1) \times f}{l}$$

where

A_1 is the absorbance of the blank test solution;

A_2 is the absorbance of the test solution;

f is a calibration factor, in millimetre milligrams per litre;

l is the optical path length, in millimetres, of the cell.

Report the arsenic content, in milligrams per litre, rounding values below 0,1 mg/l to the nearest 0,001 mg/l and values above 0,1 mg/l to the nearest 0,01 mg/l. (For example, arsenic content 0,42 mg/l.) Alternatively, report the arsenic content in millimoles per litre (for arsenic, 1 mmol = 74,9 mg).

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8 Test report

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The test report shall include the following information :

a) a reference to this International Standard;

b) identification of the sample;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operating details not specified in this International Standard or regarded as optional.

Dimensions in millimetres

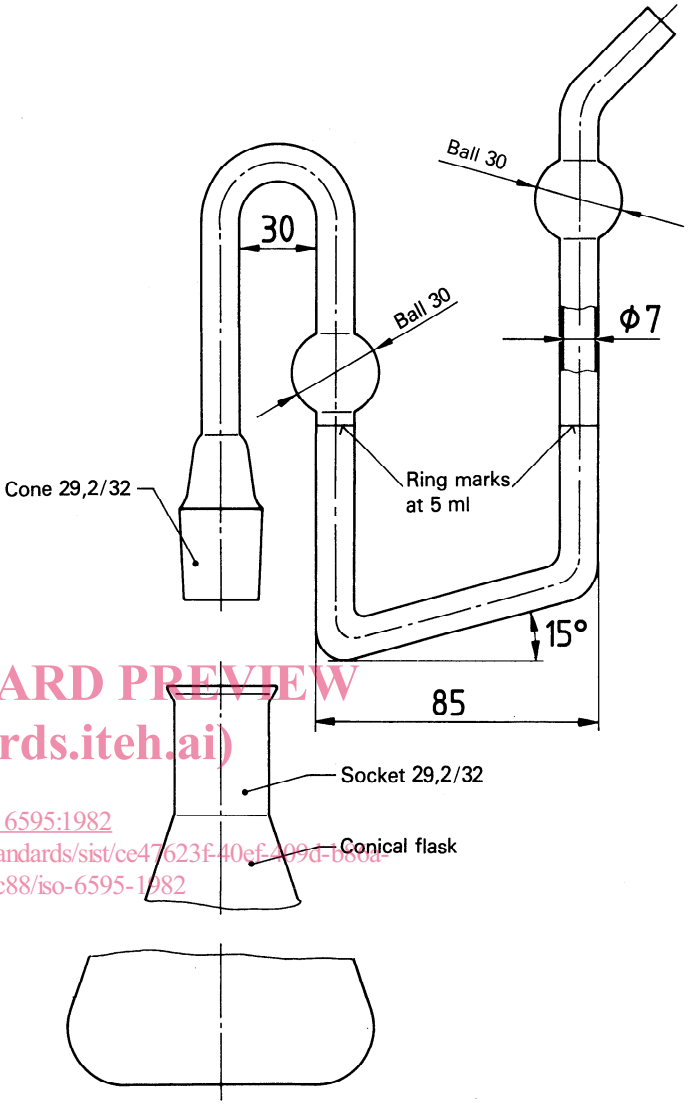

Figure — Example of reaction apparatus

Annex

Special case and interferences

A.1 Arsenic compounds which are difficult to decompose

For the determination of total arsenic in water containing silicon fluoride (for example effluents from glass etchings), or in water containing organic arsenic compounds which are difficult to decompose, it is necessary to use a method of digestion by addition of sulphuric acid and hydrogen peroxide. In such cases, heat the sample with sulphuric acid with multiple additions of hydrogen peroxide. Continue the procedure until fumes of sulphur trioxide appear, then dilute the sample with water and continue as specified in 6.4.2.

A.2 Antimony

Antimony salts are reduced under the test conditions to produce stibine (SbH_3), which reacts with the absorption solution to produce a red complex. If cells of optical path length 10 mm are used, an antimony concentration of 0,5 mg/l would contribute 0,015 % to the absorbance. Interference from antimony is thus only likely to be important when analysing certain waste waters or receiving streams. If it is present in large concentrations, this method should not be used.

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