



SLOVENSKI STANDARD
SIST EN 26595:1996

01-junij-1996

Kakovost vode - Določanje skupnega arzena - Spektrofotometrijska metoda s srebrovim dietilditiokarbamatom (ISO 6595:1982)

Water quality - Determination of total arsenic - Silver diethyldithiocarbamate spectrophotometric method (ISO 6595:1982)

Wasserbeschaffenheit - Bestimmung von Arsen - Photometrisches Verfahren mit Silberdiethyldithiocarbamat (ISO 6595:1982)

Qualité de l'eau - Dosage de l'arsenic total - Méthode spectrophotométrique au diéthylthiocarbamate d'argent (ISO 6595:1982)

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Ta slovenski standard je istoveten z: EN 26595:1992

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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EUROPEAN STANDARD

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NORME EUROPÉENNE

EUROPÄISCHE NORM

October 1992

UDC 628.1/.3:620.1:543.42:546.19

Descriptors: Water, quality, chemical analysis, determination of content, arsenic, spectrophotometric analysis, silver diethyldithiocarbamate

English version

**Water quality - Determination of total arsenic -
Silver diethyldithiocarbamate spectrophotometric
method (ISO 6595:1982)**

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Qualité de l'eau - Dosage de l'arsenic total -
Méthode spectrophotométrique au
diéthylthiocarbamate d'argent (ISO 6595:1982)

Wasserbeschaffenheit - Bestimmung von Arsen -
Photometrisches Verfahren mit
Silberdiethyldithiocarbamat (ISO 6595:1982)

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This European Standard was approved by CEN on 1992-10-05. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

This European Standard is the endorsement of ISO 6595. Endorsement of ISO 6595 was recommended by CEN/Technical Committee 230 "Water analysis" under whose competence this European Standard will henceforth fall.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 1993, and conflicting national standards shall be withdrawn at the latest by April 1993.

The Standard was approved and in accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard : Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom

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Endorsement notice

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The text of the International Standard ISO 6595:1982 was approved by CEN as a European Standard without any modification.

International Standard



6595

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

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

First edition — 1982-09-15

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UDC 614.777 : 543.42 : 546.19

Ref. No. ISO 6595-1982 (E)

Descriptors : water, quality, tests, determination of content, arsenic, spectrophotometric analysis.

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.

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

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

Australia	France	Poland
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
Switzerland

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.

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.

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, $\rho = 1,84$ g/ml.

4.2 Sulphuric acid solution, $c(1/2 \text{H}_2\text{SO}_4) = 2$ mol/l.

4.3 Sodium hydroxide solution, $c(\text{NaOH}) = 2$ 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.

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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 ($\rho = 1,19$ 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 μ g 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 μ g of arsenic.

Prepare the solution just before use.

5 Apparatus

Usual laboratory equipment and

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)].

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)	Corresponding arsenic content
ml	µg/l
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.

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.