SLOVENSKI STANDARD
SIST EN ISO 5961:1996

01-junij-1996

Kakovost vode - Določanje kadmija z atomsko absorpcijsko spektrometrijo (ISO 5961:1994)

Water quality - Determination of cadmium by atomic absorption spectrometry (ISO 5961:1994)

Bestimmung von Cadmium durch Atomabsorptionsspektrometrie (ISO 5961:1994)

Qualité de l'eau - dosage du cadmium par spectrométrie d'absorption atomique (ISO 5961:1994)

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English version

Water quality - Determination of cadmium by atomic absorption spectrometry (ISO 5961:1994)

Qualité de l'eau - Dosage du cadmium par spectrométrie d'absorption atomique (ISO 5961:1994)

Bestimmung von Cadmium durch Atomabsorptionsspektrometrie (ISO 5961:1994)

This European Standard was approved by CEN on 1994-11-14. 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.

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CEN

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

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

Foreword

This European Standard has been taken over by the Technical Committee CEN/TC 230 "Water analysis" from the work of ISO/TC 147 "Water quality" of the International Organization for Standardization (ISO).

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of an identical text or by endorsement, at the latest by September 1995, and conflicting national standards shall be withdrawn at the latest by September 1995.

According to 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.

Endorsement notice

The text of the International Standard ISO 5961:1994 has been approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 5961

Second edition
1994-01-15

Water quality — Determination of cadmium by atomic absorption spectrometry

Qualité de l'eau — Dosage du cadmium par spectrométrie d'absorption atomique

Reference number
ISO 5961:1994(E)

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.

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 5961 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical, biochemical methods*.

This second edition cancels and replaces the first edition (ISO 5961:1985), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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Water quality — Determination of cadmium by atomic absorption spectrometry

Section 1: General

1.1 Scope

This International Standard specifies two methods for the determination of cadmium: flame atomic absorption spectrometry (AAS) (Section 2) and electrothermal atomization (AAS) (Section 3).

1.1.1 Determination of cadmium using AAS in an air-acetylene flame

The method is applicable to the analysis of water and waste water when the concentration of cadmium is between 0,05 mg/l and 1 mg/l. Higher concentrations can be determined after dilution of the sample. The range of application of the method can be extended to lower concentrations by carefully evaporating the water sample, previously acidified with nitric acid. Cadmium can be determined in sludges and sediments after an appropriate digestion procedure avoiding the formation of a precipitate.

1.1.2 Determination of cadmium by electrothermal atomization AAS

The method is suitable for the determination of cadmium in water when, with a dosing volume of 10 µl, the concentration range is 0,3 µg/l to 3 µg/l. The range of application of the method can be extended to higher concentrations by diluting the water sample or by the use of smaller dosing volumes. Cadmium can be determined in sludges and sediments after an appropriate digestion procedure.

1.2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-3:—[1], *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

1) To be published. (Revision of ISO 5667-3:1985)

Section 2: Determination of cadmium using atomic absorption spectrometry in an air-acetylene flame

2.1 Interferences

The following ions will not interfere with the method as long as the mass concentrations specified below are not exceeded:

Sulfate	10 000 mg/l
Chloride	10 000 mg/l
Phosphate	10 000 mg/l
Sodium	10 000 mg/l
Potassium	10 000 mg/l
Magnesium	10 000 mg/l
Calcium	3 000 mg/l
Iron	3 000 mg/l
Copper	10 000 mg/l
Nickel	3 000 mg/l
Cobalt	10 000 mg/l
Lead	10 000 mg/l
Silicon	1 000 mg/l
Titanium	3 000 mg/l

The total salt content of the measuring solution shall be less than 15 g/l and the electrical conductivity shall be lower than 20 000 mS/m. Samples of unpredictable matrix effects shall be examined appropriately. This influence shall be compensated for either by diluting the sample or by applying the method of standard additions (see 3.6.2.2).

2.2 Principle

Aspiration of the acidified sample into the air-acetylene flame of an atomic absorption spectrometer. Measurement of the cadmium concentration at a wavelength of 228,8 nm.

2.3 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. The cadmium content of water used for blank determinations and for the preparation of standard solutions shall be negligibly low compared with the lowest mass concentration to be determined in the sample.

2.3.1 Nitric acid, $\rho = 1{,}40$ g/ml.

2.3.2 Hydrogen peroxide, $w(H_2O_2) = 30\ \%\ (m/m)$.

2.3.3 Cadmium stock solution I, $\rho(Cd) = 1\ 000$ mg/l.

Dissolve 1,000 g ± 0,002 g of cadmium in 10 ml of nitric acid (2.3.1) and 10 ml of water (see 2.3) in a 1 000 ml one-mark volumetric flask. Dilute to volume with water.

Store the solution in polyethylene or borosilicate glass containers. The solution is stable for 1 year.

Alternatively, use any commercially available stock solution containing 1,000 g/l ± 0,002 g/l of cadmium.

2.3.4 Cadmium standard solution I, $\rho(Cd) = 10$ mg/l.

Pipette 10 ml of the cadmium stock solution (2.3.3) into a 1 000 ml one-mark volumetric flask, add 10 ml of nitric acid (2.3.1) and dilute to volume with water.

Store the solution in polyethylene or borosilicate glass containers. The solution is stable for at least one month if stored at room temperature.

NOTE 1 The use of a microlitre pipette permits a standard solution of 100 ml to be prepared.

2.3.5 Cadmium calibration solutions

Prepare a minimum of five calibration solutions in accordance with the expected cadmium concentrations.

As an example, proceed as follows for the range from 0,05 mg/l to 1,0 mg/l:

Pipette 0,5 ml; 2,0 ml; 4,0 ml; 6,0 ml; 8,0 ml and 10,0 ml respectively of the cadmium standard solution (2.3.4) into 100 ml one-mark volumetric flasks.

Add 1 ml of nitric acid (2.3.1) to each of these solutions. Dilute to volume with water and mix.

The calibration solutions contain 0,05 mg/l; 0,2 mg/l; 0,4 mg/l; 0,6 mg/l; 0,8 mg/l and 1,0 mg/l of cadmium respectively.

2.3.6 Blank test solution

Pipette 1 ml of nitric acid (2.3.1) into a 100 ml one-mark volumetric flask, and dilute to volume with water (2.3.4).

If the sample requires a pretreatment by digestion, the blank shall be given the same pretreatment (see 2.5.2).

2.3.7 Solution for zero-setting the instrument

Use water (2.3.4) as a zero-setting solution. The blank solution (2.3.6) may also serve for zero-setting provided its cadmium concentration is negligibly low.

2.4 Apparatus

Immediately before use, clean the glassware with warm, dilute nitric acid, approximately 2 mol/l (e.g. by soaking for 24 h), followed by a thorough rinsing with water (2.3). Verify that each lot of pipette tips and single-use plastics vessels is free from potential cadmium contamination by carrying out blank measurements (see 2.6.1).

Usual laboratory apparatus and

2.4.1 Atomic absorption spectrometer, equipped with background correction and a radiation source for the determination of cadmium, operated according to the manufacturer's instructions.

2.4.2 Gas supply for air and acetylene. It is essential that the manufacturer's safety instructions be observed. The residual gas pressure for acetylene cylinders shall be at least 5×10^5 Pa.

2.4.3 Air-acetylene burner.

2.4.4 One-mark volumetric flasks of capacity 10 ml, 100 ml and 1 000 ml.

2.4.5 One-mark pipettes, of nominal capacity 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml, 8 ml, 10 ml, 20 ml, 30 ml and 40 ml.

2.4.6 Microlitre pipettes or **diluters**.

2.4.7 Beakers, of capacity 250 ml.

2.4.8 Heating device, for example a hotplate.

2.4.9 Membrane filtration device with filters, of pore size 0,45 µm, washed thoroughly with dilute nitric acid and rinsed with water.

2.5 Sampling and sample pretreatment

See ISO 5667-3.

2.5.1 Sampling

Collect the samples in polyethylene or borosilicate glass containers which have previously been cleaned with nitric acid and water.

2.5.2 Pretreatment and preparation of the sample solutions

2.5.2.1 Pretreatment for the determination of the content of dissolved cadmium

Filter the water sample as soon as possible after sampling (2.5.1) through a membrane filter of pore size 0,45 µm.

To stabilize the filtrate, add, for example, 10 ml of nitric acid (2.3.1) per litre of water sample to achieve a pH of less than 2; add more acid, if necessary, to ensure a pH of less than 2.

2.5.2.2 Pretreatment for the determination of cadmium after mineralization

Acidify the water sample as soon as possible after sampling by adding 1 ml of nitric acid (2.3.1) per litre of sample; add more acid, if necessary, to ensure a pH of less than 2.

Completely mix the sample, for example by thorough shaking.

Place 100 ml of the homogenized sample in a 250 ml beaker. Add 1 ml of nitric acid (2.3.1) and 1 ml of hydrogen peroxide (2.3.2).

Heat the beaker on a hotplate until about 0,5 ml remains.

It is essential that the sample is not reduced to dryness.