



SLOVENSKI STANDARD
SIST EN 1483:2007

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Nadomešča:
SIST EN 1483:1998

Kakovost vode - Določevanje živega srebra - Metoda z atomsko absorpcijsko spektrometrijo

Water quality - Determination of mercury - Method using atomic absorption spectrometry

Wasserbeschaffenheit - Bestimmung von Quecksilber - Verfahren mittels Atomabsorptionsspektrometrie

Qualité de l'eau - Dosage du mercure - Méthode par spectrométrie d'absorption atomique

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ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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EUROPEAN STANDARD
NORME EUROPÉENNE
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Water quality - Determination of mercury - Method using atomic absorption spectrometry

Qualité de l'eau - Détermination du mercure - Méthode par spectrométrie d'absorption atomique

Wasserbeschaffenheit - Bestimmung von Quecksilber - Verfahren mittels Atomabsorptionsspektrometrie

This European Standard was approved by CEN on 28 February 2007.

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 CEN Management Centre or to any CEN member.

This European Standard exists 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 CEN Management Centre has the same status as the official versions.

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Foreword

This document (EN 1483:2007) has been prepared by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

This document supersedes EN 1483:1997.

NOTE This revision of EN 1483:1997, without stating details, takes into account new state of the art methods. This revision also describes continuous flow methods whilst the batch tests have been deleted as they are no longer used.

In Annex A.1, a bromate bromide conservation/digestion step is included, thus allowing to avoid potassium permanganate resp. potassium chromate.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Introduction

It should be investigated as to whether, and to what extent, particular problems will require additional marginal specification conditions.

In natural water sources, mercury compounds generally occur only in very small concentrations of less than 0,1 µg/l, although higher concentrations may be found, for example, in waste water.

Both inorganic and organic compounds of mercury may be present as mercury can accumulate in sediment and sludge.

To fully decompose all of the mercury compounds, a digestion procedure is necessary. Digestion can be omitted only if it is certain that the mercury concentration can be measured without this pre-treatment.

For measurements in the low concentration range, highest purity reagents, clean reaction vessels, mercury-free air in the laboratory and a very stable measurement system are essential.

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

Mercury and mercury compounds are very toxic. Extreme caution should be exercised when handling samples and solutions which contain or may contain mercury.

Potassium dichromate is toxic. Caution should be exercised when handling the solid material or its solutions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

1 Scope

This European Standard specifies two methods for the determination of mercury. For the method described in Clause 4, tin(II) chloride is used as the reducing agent. For the method given in Clause 5, sodium borohydride serves as the reducing agent. The choice of method depends on the equipment available and the matrix (see Clause 3). Both methods are suitable for the determination of mercury in water, for example in drinking, ground, surface and waste waters, in a concentration range from 0,1 µg/l to 10 µg/l. Higher concentrations can be determined if the water sample is diluted. Lower concentrations in the range of 0,001 µg/l to 5 µg/l can be determined if special mercury analysers with an optimised instrument are used or if atomic fluorescence spectrometry is applied (see EN 13506 or ISO 17852).

2 Normative references

The following referenced documents are indispensable for the application 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.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques (ISO 5667-1:2006)*

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of water samples (ISO 5667-3:2003)*

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 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

3 General interferences

With mercury there is a risk that exchange reactions, that is adsorption and desorption, will occur on the walls of containers, reaction cell, gas- liquid separator etc.

Mercury vapour can diffuse through various plastics, thus this phenomenon needs to be considered in the choice of tubing material. Glass or special plastics tubing, e.g. FEP tubes (FEP = Fluorinated Ehtylene-Propylene) and pump tubes based on polypropylene compounds may be used. Silicone tubing, for example, is not suitable.

Volatile organic substances can absorb in the UV range and be mistaken for mercury. If present, these are, for the most part removed prior to reduction. Potassium permanganate is added until the solution is permanently coloured purple and an inert gas is bubbled through the solution for 10 min. Often, such interferences by non-specific absorption can also be eliminated by a background compensation system of atomic absorption equipment.

It is necessary to bring all solutions to the same temperature (< 25 °C) before reducing and stripping the mercury vapour. Water condensation on the cuvette windows can be prevented by heating the cuvette to temperatures slightly above 100 °C.

The interferences due to the presence of other elements in the matrix depend on the choice of reducing agent. Element concentrations in excess of those listed in Table 1 can cause negative bias.

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When using tin(II)chloride as reducing agent, the analysis is less prone to interferences from heavy metals compared with the application of sodium borohydride. Using flow systems, interference effects due to heavy metals can be less than indicated in Table 1.

Table 1 — Acceptable concentrations of some matrix elements in a measurement solution in milligrams per litre (mg/l)

Reducing agent	NaBH ₄	NaBH ₄	SnCl ₂
	directly	directly	directly
Medium	0,5 mol/l HCl	5 mol/l HCl + 0,2 g/l Fe(III)	0,5 mol/l HCl
Element			
Cu(II)	10	10	500
Ni(II)	1	500	500
Ag(I)	0,1	10	1
I ⁻	100	10	0,1
As(V)	0,5	0,5	0,5
Bi(III)	0,05	0,5	0,5
Sb(III)	0,5	0,5	0,5
Se(IV)	0,005	0,05	0,05

Tin(II) chloride causes such extensive contamination of the apparatus with tin that considerable interferences occur if sodium borohydride is used afterwards. Therefore, use separate systems for reductions with tin(II) chloride and with sodium borohydride.

4 Determination of mercury after tin(II) chloride reduction without enrichment

4.1 Principle

Mono- or di-valent mercury is reduced to the elemental form by tin(II) chloride in an acid medium. Elemental mercury is then stripped from the solution with the aid of a stream of inert gas or mercury-free air and, in the form of an atomic gas, transported into a cuvette. Absorbances are measured at a wavelength of 253,7 nm in the radiation beam of an atomic absorption spectrometer. Concentrations are calculated using a calibration curve.

4.2 Interferences

(See also Clause 3).

Iodide in concentrations > 0,1 mg/l causes interferences in the determination due to the formation of mercury complexes. In this case, use another method, such as reduction with sodium borohydride (see Clause 5).

Because of the redox potential of the tin(II) chloride solution, various inorganic mercury compounds such as mercury sulfide and organic mercury compounds cannot be fully reduced without digestion (4.6).

4.3 Reagents

As a minimum, use "analytical grade" reagents or those with particularly low mercury content. The mercury content of the water and reagents shall be negligible compared to the lowest analyte concentration.

4.3.1 Water, Grade 1, as specified in EN ISO 3696.

4.3.2 Nitric acid, $\rho(\text{HNO}_3) = 1,40 \text{ g/ml}$.

4.3.3 Sulfuric acid, $\rho(\text{H}_2\text{SO}_4) = 1,84 \text{ g/ml}$.

4.3.4 Hydrochloric acid, $\rho(\text{HCl}) = 1,16 \text{ g/ml}$.

4.3.5 Potassium permanganate solution

Dissolve 50 g of potassium permanganate, KMnO_4 , in 1 000 ml of water (4.3.1).

4.3.6 Stabilizer solution

Dissolve 5 g of potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, in 500 ml of nitric acid (4.3.2) and dilute to 1 000 ml with water (4.3.1).

4.3.7 Potassium peroxodisulfate solution

Dissolve 40 g of potassium peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$, in 1 000 ml of water (4.3.1).

4.3.8 Hydroxylammonium chloride solution

Dissolve 10 g of hydroxylammonium chloride, H_4CINO , in 100 ml of water (4.3.1).

4.3.9 Tin(II) chloride solution, $\rho(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) = 50 \text{ g/l}$

Dissolve 5 g of tin(II) chloride dihydrate, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, in 30 ml of hydrochloric acid (4.3.4) and dilute to 100 ml with water (4.3.1). In flow systems, use a solution of lower concentration (e.g. $\rho(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) = 20 \text{ g/l}$).

The concentration of the tin(II) chloride depends on flow conditions of the system. Follow the manufacturer recommendations.

If a high result for the blank (4.6) is obtained, pass nitrogen through the solution for 30 min to remove traces of mercury.

4.3.10 Mercury stock solution I, $\rho(\text{Hg}) = 1\,000 \text{ mg/l}$.

Use a commercially available quantitative stock solution with a mercury concentration of e.g. $(1\,000 \pm 2) \text{ mg/l}$. This solution is considered to be stable for at least one year, but in reference to guaranteed stability, see the recommendations of the manufacturer.

4.3.11 Mercury stock solution II, $\rho(\text{Hg}) = 10 \text{ mg/l}$.

Add 1 ml of stabilizer solution (4.3.6) to 1 ml of mercury stock solution I (4.3.10) and dilute to 100 ml with water (4.3.1).

The solution is stable for 1 week.

4.3.12 Mercury standard solution (1), $\rho(\text{Hg}) = 100 \mu\text{g/l}$.

Add 1 ml of stabilizer solution (4.3.6) to 1 ml of mercury stock solution II (4.3.11) and dilute to 100 ml with water (4.3.1).

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Prepare this solution freshly on the day of use.

4.3.13 Mercury standard solution (2), $\rho(\text{Hg}) = 50 \mu\text{g/l}$.

Add 1 ml of stabilizer solution (4.3.6) to 0,5 ml of mercury stock solution II (4.3.11) and dilute to 100 ml with water (4.3.1).

Prepare this solution freshly on the day of use.

4.3.14 Mercury calibration solutions

Prepare the calibration solutions appropriate for the volume and expected mercury concentrations of the measurement sample solutions.

For a concentration range from 0,5 $\mu\text{g/l}$ to 5 $\mu\text{g/l}$, for example, proceed as follows:

Pipette into a series of six 100 ml volumetric flasks 1 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml, of mercury standard solution (4.3.13).

Add 1 ml of stabilizer solution (4.3.6) to each 100 ml volumetric flask.

Fill to the mark with water (4.3.1) and mix thoroughly.

These calibration solutions contain 0,5 $\mu\text{g/l}$, 1 $\mu\text{g/l}$, 2 $\mu\text{g/l}$, 3 $\mu\text{g/l}$, 4 $\mu\text{g/l}$ and 5 $\mu\text{g/l}$ mercury. Prepare freshly before each series of measurements. If calibration measurements are to be run in duplicate, prepare another set of solutions.

Using dilutors (offline or integrated in the AAS system) for preparing the calibration solutions is also allowed. In this case the user shall prove that the performance criteria of the dilution system meet the specific requirements.

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4.3.15 Reagent blank solution

Prepare a volume of blank solution corresponding to that of the measurement solution by adding 10 ml of stabilizer solution (4.3.6) per 1 000 ml of water. Use the same digestion procedure as for the sample (4.6). Include the reagent blank in each batch of analyses.

4.3.16 Rinsing solution for glassware

Add to 500 ml of water (4.3.1) 150 ml of nitric acid (4.3.2) and dilute with water to 1 000 ml.

4.4 Apparatus

Before use, rinse all glassware thoroughly with diluted nitric acid (4.3.16) and then several times with water (4.3.1).

4.4.1 Atomic absorption spectrometer, equipped with a cold vapour generation system and a quartz or glass tube atomizer. An AAS system with background correction is recommended.

Automated stand-alone mercury flow systems (flow injection systems or continuous flow systems) are also suitable. They allow a concentration range lower by about one order of magnitude. Manually operated or semi-automatic batch systems are adequate as well.

4.4.2 Radiation source, for the determination of mercury, e.g. a hollow cathode or electrodeless discharge lamp. Stand alone mercury systems are usually equipped with a built-in mercury radiation source. The high resolution continuum source; AAS uses a continuum radiation source (e.g. a Xenon short arc lamp) for the determination of all elements inclusively mercury.

4.4.3 Mercury accessory, consisting of:

4.4.3.1 **Peristaltic pump**, with tubing to pump sample, reduction solution and carrier solution separately.

4.4.3.2 **Valves**, for the selection of the carrier gas flow.

4.4.3.3 **Reaction coil or reaction cell**, where the reagent streams are being mixed and mercury(I/II) is reduced to the Hg(0) state.

4.4.3.4 **Gas/liquid separator**, for separating the gas from the liquid.

4.4.3.5 **Absorption cell**, consisting of a borosilicate glass or quartz cuvette (depending on the AAS system)

4.4.4 **Volumetric flasks**, 100 ml, 200 ml, and 1 000 ml.

4.4.5 **Pipettes**, 0,5 ml, 1 ml, 5 ml, and 10 ml.

NOTE Instead of pipettes, it is advantageous to use a dispensing apparatus or a dilutor, as it can significantly reduce the risk of introducing trace contaminants.

4.5 Sampling and sample pre-treatment

Carry out sampling as specified in EN ISO 5667-1 and EN ISO 5667-3.

For sampling, use vessels made of borosilicate glass, quartz, polysulfone (PSU) or fluoridized ethylene-propylene-polymerisate (FEP).

Make sure that the sampling vessel contains no mercury and does not cause a loss of mercury by adsorption.

In order to limit any loss by, for example, adsorption on the vessel walls, add 10 ml of stabilizer solution (4.3.6) and make up to 1 000 ml with the sample.

Verify that the sample has a pH of 1 and shows a yellow-orange colour indicating an excess of dichromate.

If necessary, add additional stabilizer solution, and include the appropriate volume correction factor in the calculations.

4.6 Digestion method using potassium permanganate/potassium peroxodisulfate

Carry out the wet chemical digestion procedure as given below. Alternatively, use one of the digestion methods indicated in Annex A, Annex B or Annex C but verify that the efficiency of that method compared with the digestion method described in the normative part of this standard is equivalent.

Transfer 100 ml of the stabilized water sample (4.5) or an appropriate volume (maximum 1 000 ml) of sample to a flask made of one of the materials listed in 4.5.

Carefully add 15 ml of potassium permanganate solution (4.3.5), 1 ml of nitric acid (4.3.2) and 1 ml of sulfuric acid (4.3.3).

Shake the mixture well after each addition.

Allow the solution to stand for 15 min, then add 10 ml of potassium peroxodisulfate solution (4.3.7).

Place the loosely stoppered flask on a suitable heating device (e.g. a heating block or a water bath) and digest at $(95 \pm 3) ^\circ\text{C}$ for 2 h.