

Nadomešča:**SIST-TS CEN/TS 16175-2:2013**

Blato, obdelani biološki odpadki in tla - Določevanje živega srebra - 2. del: Metoda atomske fluorescenčne spektrometrije s tehniko hladnih par (CV-AFS)

Sludge, treated biowaste and soil - Determination of mercury - Part 2: Cold-vapour atomic fluorescence spectrometry (CV-AFS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Quecksilber - Teil 2: Kaldampf-Atomfluoreszenzspektrometrie (CV-AFS)

Boues, bio-déchets traités et sols - Détermination du mercure - Partie 2: Spectrométrie de fluorescence atomique en vapeur froide (SFA-VP)

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English Version

Sludge, treated biowaste and soil - Determination of mercury - Part 2: Cold-vapour atomic fluorescence spectrometry (CV-AFS)

Boues, biodéchets traités et sols - Détermination du mercure - Partie 2: Spectrométrie de fluorescence atomique de vapeur froide (SFA-VP)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Quecksilber - Teil 2: Kaltdampf-Atomfluoreszenzspektrometrie (CV-AFS)

This European Standard was approved by CEN on 19 March 2016.

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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-CENELEC Management Centre has the same status as the official versions.

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European foreword

This document (EN 16175-2:2016) has been prepared by Technical Committee CEN/TC 444 "Test methods for environmental characterization of solid matrices", the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16175-2:2013.

The preparation of the previous edition of this analytical method by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

This document contains the following technical changes in comparison with the previous edition:

- repeatability and reproducibility data have been added from a European interlaboratory comparison organized by the German Federal Institute for Materials Research and Testing BAM in 2013 (see Annex A).

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EN 16175, *Sludge, treated biowaste and soil* — *Determination of mercury* comprises the following parts:

- *Part 1: Cold-vapour atomic absorption spectrometry (CV-AAS);*
- *Part 2: Cold-vapour atomic fluorescence spectrometry (CV-AFS).*

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see Annex A for the results of validation).

Table 1 — Matrices for which this European Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost
Soil	Soil

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard does not purport to address all of the 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.

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

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1 Scope

This European Standard specifies a method for the determination of mercury in *aqua regia* or nitric acid digests of sludge, treated biowaste and soil, obtained according to EN 16173 or EN 16174 using cold-vapour atomic fluorescence spectrometry (CV-AFS). The lower working range limit is 0,003 mg/kg (dry matter basis).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16173, *Sludge, treated biowaste and soil — Digestion of nitric acid soluble fractions of elements*

EN 16174, *Sludge, treated biowaste and soil — Digestion of aqua regia soluble fractions of elements*

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

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3 Principle

Mono- and divalent mercury is reduced to the elemental form by tin(II) chloride solution or sodium borohydride in acid medium. Elemental mercury is stripped off from the solution in a closed system, by means of a stream of argon or nitrogen. The mercury vapour is injected into the cell of an atomic fluorescence spectrometer where the mercury atoms are excited by radiation of a specific wavelength, usually about 254 nm. The intensity of the fluorescence radiation is a function of mercury concentration. The concentrations are calculated using a calibration curve.

NOTE The matrix of the solution analysed is dominated by the acids used in the digestion step. Tin(II) chloride as a reduction substance is recommended in this European Standard, because sodium borohydride reduces many elements commonly found in soil, sludge and waste extract solutions, to the elemental state, which may cause matrix problems under particular circumstances. However, it is still possible to use sodium borohydride as reduction agent. The concentration range 0,1 µg/l to 10 µg/l in the test solution, corresponding to 0,003 mg/kg to 0,3 mg/kg of mercury, when a 3,0 g sample has been digested, can be determined directly. Higher concentrations can be determined if the test solution is diluted. Sensitivity can be increased by the amalgamation technique.

4 Interferences

The presence of water vapour or aerosol in the fluorescence cell may cause suppression due to quenching. Water vapour should be removed from the carrier gas stream using a hygroscopic membrane before entering the detector. The noble metals, such as gold and silver, amalgamate with mercury and, therefore, may cause suppression. Also anions, for instance sulfide, iodide and bromide, which complex strongly with mercury, can cause suppression.

Fewer interferences arise from heavy metals when tin(II) chloride is used rather than sodium borohydride. When flow systems are used, interference effects due to heavy metals may be less than indicated in Table 2.

Table 2 — Tolerable concentrations of some matrix elements

Element	Acceptable concentration in the test solution mg/l
Cu(II)	500
Ni(II)	500
Ag(I)	1

5 Reagents

For the determination of mercury at trace and ultra-trace level, the reagents shall be of adequate purity. The concentration of mercury or interfering substances in the reagents and the water should be negligible compared to the lowest concentration of mercury to be determined.

5.1 Water quality 2 according to EN ISO 3696 for all sample preparations and dilutions.

5.2 Hydrochloric acid, HCl, $\rho(\text{HCl}) = 1,18 \text{ g/ml}$, $c(\text{HCl}) = 12 \text{ mol/l}$, $w(\text{HCl}) = 370 \text{ g/kg}$.

The same batch of hydrochloric acid shall be used throughout the procedure.

5.3 Nitric acid, HNO_3 , $\rho(\text{HNO}_3) = 1,4 \text{ g/ml}$, $c(\text{HNO}_3) = 15 \text{ mol/l}$, $w(\text{HNO}_3) = 650 \text{ g/kg}$.

The same batch of nitric acid shall be used throughout the procedure.

5.4 Nitric acid, diluted solution.

Add 10 ml nitric acid (5.3) to 500 ml of water in a 1 000 ml volumetric flask, mix and fill to the mark with water.

5.5 Nitric acid, $c(\text{HNO}_3) = 2 \text{ mol/l}$, rinsing solution for glassware.

Add 150 ml of nitric acid (5.3) to about 500 ml of water, and dilute with water to 1 000 ml.

5.6 Aqua regia, diluted solution.

Add 21 ml hydrochloric acid (5.2) and 7 ml nitric acid (5.3) to 500 ml of water in a 1 000 ml volumetric flask, mix and fill to the mark with water.

5.7 Tin(II) chloride solution, $\rho(\text{SnCl}_2 \cdot 2 \text{H}_2\text{O}) = 100 \text{ g/l}$.

Dissolve 10 g of $\text{SnCl}_2 \cdot 2 \text{H}_2\text{O}$ in 30 ml of hydrochloric acid (5.2), transfer to a 100 ml volumetric flask and fill to the mark with water. The blank concentration of mercury can be reduced by bubbling a stream of nitrogen through the solution for 30 min, if necessary. Prepare this solution on the day of use.

A solution of lower concentration, e. g. 0,5 g in 100 ml, may be used with flow systems. Prepare this latter solution freshly on the day of use from the more concentrated solution by diluting with water.

5.8 Sodium borohydride solution, $\rho(\text{NaBH}_4) = 30 \text{ g/l}$.

1 g sodium hydroxide, NaOH, is weighed into a 100 ml volumetric flask and dissolved in water. 3 g sodium borohydride, NaBH_4 , are weighed into a 100 ml volumetric flask, dissolved and diluted to the mark with the sodium hydroxide solution.

5.9 Mercury standard stock solution, 1 000 mg/l

Use a commercially available quantitative stock solution with a mercury concentration of $(1\ 000 \pm 2)$ 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.

5.10 Mercury, standard solution I, 100 mg/l.

Pipette 10 ml of the mercury standard stock solution (5.9) into a 100 ml volumetric flask, add 10 ml nitric acid (5.3), mix and fill to the mark with water.

This solution is stable for one month.

5.11 Mercury, standard solution II, 1 mg/l.

Pipette 1 ml of the mercury standard solution I (5.10) into a 100 ml volumetric flask, add 10 ml nitric acid (5.3), mix and fill to the mark with water.

This solution is stable for 7 days.

5.12 Mercury, standard solution III, 100 µg/l

Pipette 10 ml of the mercury standard solution II (5.11) into a 100 ml volumetric flask, add 10 ml nitric acid (5.3), mix and fill to the mark with water.

This solution shall be freshly prepared on the day of use.

5.13 Carrier gas, argon or nitrogen, with a purity of $\geq 99,99$ %.

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6 Apparatus

6.1 Usual laboratory apparatus

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All glassware shall be carefully cleaned for low trace element determinations, e. g. by immersion in nitric acid rinsing solution (5.5) for a minimum of 6 h, followed by rinsing with water before use. The nitric acid shall be replaced each week.

6.2 Atomic fluorescence spectrometer (AFS)

Equipped with a specific Hg lamp, a fixed 254 nm filter, a photomultiplier tube for the detection of fluorescence radiation and a suitable software for processing the output signal. Operate at a current recommended by the lamp or the instrument manufacturer. The gas supply (argon or nitrogen) should be equipped with a two stage regulator. The use of a gas purifier consisting of activated carbon is recommended. The use of nitrogen instead of argon will cause a reduced sensitivity of the measurement.

6.3 Automated sample introduction system

Automated mercury flow systems (flow injection systems or continuous flow systems) are very common for atomic fluorescence spectrometry. They allow a measurement of mercury in a concentration range which is about one order of magnitude lower. Manually operated systems or semi-automated batch systems are adequate as well.

6.4 Cold-vapour generator, batch system or an automated flow injection analysis system (FIA)

The system should be adaptable to the atomic fluorescence spectrometer (6.2). A flow-controlled argon or nitrogen stream (5.13) is used as an inert carrier to transport mercury vapour into the cell. Time-controlled addition of tin(II) chloride solution (5.7) in combination with automatic start of the read signal of the spectrometer is required. For the atomic fluorescence spectrometer (6.2), condensation in the cell is avoided by the inclusion of a drying tube in the detection system.