

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16175-2:2016 sisaldab Euroopa standardi EN 16175-2:2016 ingliskeelset teksti.	This Estonian standard EVS-EN 16175-2:2016 consists of the English text of the European standard EN 16175-2:2016.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 19.10.2016.	Date of Availability of the European standard is 19.10.2016.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 13.030.01, 13.080.10

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:

Aru 10, 10317 Tallinn, Eesti; koduleht www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:

Aru 10, 10317 Tallinn, Estonia; homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

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.

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

CEN members are the national standards bodies of 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 United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

European foreword	3
Introduction	4
1 Scope	5
2 Normative references	5
3 Principle	5
4 Interferences	5
5 Reagents	6
6 Apparatus	7
7 Procedure	8
7.1 Test solution	8
7.2 Test blank solution	8
7.3 Preparation of the calibration solutions	8
7.4 Calibration	8
7.5 Measurement of test sample	8
8 Calculation and expression of results	9
8.1 Calculation	9
8.2 Expression of results	9
9 Performance data	9
10 Test report	9
Annex A (informative) Repeatability and reproducibility data	11
Bibliography	12

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).

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.

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)*

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.