

**Sludge, treated biowaste and soil - Determination of adsorbable organically bound halogens (AOX)**

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## EESTI STANDARDI EESSÕNA

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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 22.08.2012.	Date of Availability of the European standard is 22.08.2012.
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ICS 13.030.01, 13.080.10

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ICS 13.030.01; 13.080.10

English Version

**Sludge, treated biowaste and soil - Determination of adsorbable  
organically bound halogens (AOX)**

Boues, biodéchets traités et sols - Détermination des  
composés organiques halogénés adsorbables (AOX)

Schlamm, behandelter Bioabfall und Boden - Bestimmung  
von adsorbierbaren organisch gebundenen Halogenen  
(AOX)

This European Standard was approved by CEN on 24 May 2012.

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## Foreword

This document (EN 16166:2012) has been prepared by Technical Committee CEN/TC 400 "Project Committee - Horizontal standards in the fields of sludge, biowaste and soil", 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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 has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document 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.

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 document is the result of a desk study "Horizontal European standard for determination of AOX in sewage sludge and comparable matrices" in the project "Horizontal" and aims at evaluating the latest developments in assessing AOX in sludge, soil, treated biowaste and neighbouring fields. After an evaluation study, in which the ruggedness of the method was studied, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties involved in the evaluation.

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

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

<b>Matrix</b>	<b>Materials used for validation</b>
Sludge	Municipal sludge
Compost	Fresh compost
	Compost
Soil	Sludge amended soil
	Agricultural 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 an empirical method for the direct determination of organically bound chlorine, bromine and iodine (but not fluorine) adsorbed and occluded to the sample matrix. Non-volatile organically bound halogens adsorbable on activated carbon present in the aqueous phase of the sample prior to drying or adsorbed to sample surface are included in the determination.

This European Standard is intended for analysis of sludge, treated biowaste or soil in concentrations ranging from 5 mg/kg dry matter to approximately 6 g/kg dry matter. The exact concentration range covered depends on the instrument used for determination.

## 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 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

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

EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15)*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **adsorbable organically bound halogens**

#### **AOX**

equivalent amount of chlorine, bromine, and iodine contained in organic compounds, expressed as chloride when determined according to this European Standard

## 4 Principle

Activated carbon is added to a dried, homogenised solid sample. Inorganic halides are eluted and at the same time water soluble organic compounds are adsorbed onto the activated carbon by shaking with acidified nitrate solution.

The loaded carbon/sample mixture is combusted in an oxygen stream.

The hydrogen halides produced are absorbed followed by determination of the halide ions by an argentometric titration, such as microcoulometry. The result is expressed as the mass concentration of chloride.

## 5 Interferences

Sparingly soluble or occluded inorganic halides are included in the determination and may, if present, give a significant positive bias. Adequate washing is essential to remove inorganic interference.

NOTE Halogenated substances that volatilise at 105 °C are lost.

Organic bromine and iodine compounds may, during combustion, lead to the formation of elemental bromine or iodine respectively or to the formation of halogen oxides. The determination of these AOX fractions may be incomplete, thus leading to negative bias.