SLOVENSKI STANDARD
SIST EN 16166:2013

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Blato, obdelani biološki odpadki in tla - Določevanje organsko vezanih halogenov, sposobnih adsorpcije (AOX)

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

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

Boue, biodéchet traité et sol - Détermination des composés organiques halogénés adsorbables (AOX)

Ta slovenski standard je istoveten z: **EN 16166:2012**

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

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EUROPEAN STANDARD

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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)

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.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

Matrix	Materials used for validation
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.

6 Reagents

Use only reagents of recognised analytical grade and water grade 1 in accordance with EN ISO 3696.

The AOX contribution from water, reagents and gases should be significantly lower than the lowest AOX content to be determined. The overall AOX content of water, chemicals, and gases shall be checked by measuring the total blank (see 9.6).

6.1 Activated carbon, grain size approximately 10 µm to 50 µm.

For the storage of activated carbon, see Annex B.

The blank value of the washed activated carbon shall be less than 15 µg of chloride equivalent per gram of activated carbon.

6.2 Nitric acid, HNO_3, ρ = 1,4 g/ml, 65 % (mass fraction) solution.

6.3 Hydrochloric acid, c(HCl) = 0,100 mol/l.

The molarity shall be precisely known, since the acid is used for checking the micro-titration (see 9.4.3).

6.4 Sulfuric acid, H_2SO_4, ρ = 1,84 g/ml.

6.5 Gases for combustion, e.g. oxygen (O_2), or a mixture of oxygen and an inert gas.

6.6 Sodium nitrate, $NaNO_3$, for the preparation of stock solution.

6.7 Nitrate stock solution, acidified, $c(NaNO_3)$ = 0,2 mol/l.

Dissolve 17 g of sodium nitrate (6.6) in water in a 1 000 ml volumetric flask, add 15 ml of nitric acid (6.2), and make up to volume with water.

6.8 Nitrate washing solution, $c(NaNO_3)$ = 0,01 mol/l.

Pipette 50 ml of the nitrate stock solution (6.7) in a 1 000 ml volumetric flask, and make up to volume with water.

6.9 Methanol, CH_3OH.

6.10 4-Chlorophenol stock solution, C_6H_5ClO, equivalent to AOX = 2,0 g/l.

Dissolve 0,725 g of 4-chlorophenol in methanol (6.9) in a 100 ml calibrated flask and make up to volume with methanol (6.9).

6.11 4-Chlorophenol working solutions, equivalent to AOX = 0,1 g/l and 0,5 g/l AOX, respectively.

Pipette 5 ml and 25 ml of 4-chlorophenol stock solution (6.10) into two separate 100 ml calibrated flasks, and make up to volume with methanol (6.9).

The stock solution (6.10) may be stored for at least one month and the working solutions (6.11) for one week in a refrigerator in glass bottles.

7 Apparatus

7.1 Apparatus for combustion and detection

7.1.1 Combustion apparatus, a furnace capable of being heated to at least 950 °C, equipped with a quartz tube approximately 30 cm long with an internal diameter of between 2 cm and 4 cm (see Annex C).

NOTE It is essential that the combustion temperature is sufficient. Temperatures below 950 °C are likely to result in poor recovery and increased variability.

7.1.2 Quartz sample boat, to fit in the quartz tube.

7.1.3 Argentometric measuring device for determining halide concentrations, e.g. a microcoulometer, capable of determining at least 0,03 µmol/l chloride with a repeatability variation coefficient of less than 10 %, or an equivalent device to determine chloride ions.

7.1.4 Absorber, filled with sulfuric acid (6.4), to dry the gas stream and designed so that the acid does not backflush into the furnace.

7.1.5 Syringe, to pipette volumes of 1 µl to 10 µl of hydrochloric acid (6.3) or 4-chlorophenol solutions (6.10 and 6.11).

7.2 Equipment for adsorption

7.2.1 Filtration apparatus, e.g. with a funnel capacity of 0,15 l and a filter diameter of 25 mm.

7.2.2 Low-halide polycarbonate membrane filter, to fit the filtration apparatus (7.2.1), with a pore size of 0,45 µm, or any equivalent filtration material, such as a dedicated quartz filter for AOX determination.

7.2.3 Conical flask (Erlenmeyer flask) of 25 ml capacity with ground glass stopper or 12 ml to 20 ml screw cap vial with polytetrafluoroethene (PTFE) lined cap.

7.2.4 Mechanical shaker.

7.3 Equipment for sample preparation

7.3.1 Porcelain evaporating dish.

7.3.2 Oven with forced ventilation or natural ventilation through adjustable vents adjustable to (105 ± 5) °C.

7.3.3 Desiccator, provided with a suitable desiccant.

7.3.4 Analytical mill or porcelain mortar.

7.3.5 Precision balance.

8 Sample storage and pretreatment

8.1 Sample storage

For the sampling and storage of sludge samples refer to EN ISO 5667-15.

Samples shall be stored in suitable containers with an appropriate closure material such as PTFE. Samples to be frozen may be stored in aluminium containers pre-cleaned by heating to 450 °C for minimum 4 h or by rinsing with a non-chlorinated solvent.