



SLOVENSKI STANDARD
oSIST prEN 16166:2020
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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)

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

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EUROPEAN STANDARD
NORME EUROPÉENNE
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Will supersede EN 16166:2012

English Version

Sludge, treated biowaste, soil and sediments - Determination of adsorbed 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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 444.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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Contents	Page
European foreword	3
Introduction	4
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions.....	5
4 Principle	5
5 Interferences	5
6 Reagents.....	6
7 Apparatus	7
7.1 Apparatus for combustion and detection	7
7.2 Equipment for adsorption	7
7.3 Equipment for sample preparation.....	7
8 Sample storage and pretreatment.....	7
8.1 Sample storage.....	7
8.2 Sample pretreatment	8
9 Procedure	8
9.1 General.....	8
9.2 Adsorption and inorganic halide removal.....	8
9.3 Combustion	9
9.4 Preliminary tests and daily checks.....	9
9.5 Blank determination	10
10 Calculation.....	10
10.1 Mass concentration of adsorbed organically bound halogens [$\rho_{Cl}(AOX)$] in dried sample.....	10
10.2 Expression of results	11
11 Precision	11
12 Test report.....	11
Annex A (informative) Repeatability and reproducibility data.....	12
A.1 Materials used in the interlaboratory comparison study	12
A.2 Interlaboratory comparison results.....	13
Annex B (informative) Storage of activated carbon	14
Bibliography	15

European foreword

This document (prEN 16166:2020) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

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

This document will supersede EN 16166:2012.

In comparison with the previous edition, the following technical modifications have been made:

- Calculation harmonized with EN ISO 9562;
- Initial check and daily check procedure harmonized with EN ISO 9562 and extended to include full measuring process.

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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 Europe-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 subjected to discussions with all parties involved in the evaluation.

This document 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 document is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Compost	Fresh compost
	Compost
Soil	Sludge amended soil
	Agricultural soil
Sediments	Surface water sediments

WARNING — Persons using this document should be familiar with usual laboratory practice. This document 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 document be carried out by suitably trained staff.

1 Scope

This document specifies an empirical method for the direct determination of organically bound halogens adsorbable on activated carbon present in the aqueous phase of the sample prior to drying or adsorbed to sample surface.

This document is intended for analysis of sludge, treated biowaste, soil or sediments in concentrations ranging from 5 mg/kg dry matter. The upper limit and exact concentration range covered depend on the instrumentation used for determination.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp/ui>

3.1

adsorbed organically bound halogens, AOX

amount of chlorine, bromine, and iodine contained as organic compounds in the sample matrix, expressed as chloride when determined according to this document

Note 1 to entry: This includes halogenated organic compounds adsorbed at the dry sample matrix as well as water soluble halogenated compounds that can be adsorbed on to active carbon.

4 Principle

Activated carbon is added to a dried, homogenized 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 the determination of the halide ions, for example, by microcoulometric titration. The result is expressed as the mass concentration of chloride.

5 Interferences

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

Iodide contained in the sample can be adsorbed to the active carbon and not be washed off, thus leading to positive bias.

prEN 16166:2020 (E)

Organic bromine and iodine compounds can, 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 can be incomplete, thus leading to negative bias.

Samples with significant amounts of volatile, purgeable organic halogen compounds can lead to negative bias. In this case, the sample is to be suspended in water and purged with oxygen at 60 °C for 30 min. The purged gases are analysed by the analytical instrument. The resulting portion of purgeable organic halogens is added to the result of the regular determination.

6 Reagents

Use only reagents of recognized analytical grade. Water should be of high purity, for example, in accordance with [3] grade 1.

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 9.5).

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 activated carbon shall be less than 15 µg of chloride equivalent per gram of activated carbon.

6.2 Nitric acid, HNO₃, ρ = 1,4 g/ml, 65 % (m/m) solution.**6.3 Hydrochloric acid**, c(HCl) = 0,010 mol/l.**6.4 Sulfuric acid**, H₂SO₄, ρ = 1,84 g/ml.**6.5 Gases for combustion**, e.g. oxygen (O₂), or a mixture of oxygen and an inert gas.**6.6 Sodium nitrate**, NaNO₃, for the preparation of stock solution.**6.7 Nitrate stock solution**, acidified, c(NaNO₃) = 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(NaNO₃) = 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 4-chlorophenol, stock solution, ρ_{Cl}(AOX) = 200 mg/L.

Dissolve 72,5 mg of 4-chlorophenol (C₆H₅ClO) in water in a 100 ml volumetric flask and make up to volume with water.

6.10 2-chlorobenzoic acid, stock solution ρ_{Cl}(AOX) = 250 mg/L.

Dissolve 110,4 mg 2-chlorobenzoic acid in a 100 ml volumetric flask in water and make up to volume with water.

The dissolution of 2-chlorobenzoic acid is very slow. It is recommended to prepare this solution the day before using it. This stock solution may be stored for one month at 2 °C to 8 °C in a glass bottle.

7 Apparatus

7.1 Apparatus for combustion and detection

7.1.1 Combustion apparatus, consisting of furnace, heated to at least 950 °C, equipped with a combustion tube made of quartz glass or ceramic. Both vertically and horizontally arranged combustion tubes are suitable.

7.1.2 Combustion vessel, e.g. ceramic or quartz container, suitable to be inserted into the combustion tube.

7.1.3 Measuring device for determining halide concentrations, e.g. a microcoulometer, capable of determining 1 µg absolute amount of 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), 4-chlorophenol or 4-chlorobenzene solutions (6.9 and 6.10).

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 or ceramic 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 polytetrafluoroethylene (PTFE) lined cap.

7.2.4 Mechanical shaker device for the flasks, specified in 7.2.3, e.g. with a carrier plate.

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

7.3.6 pH measuring device

8 Sample storage and pretreatment

8.1 Sample storage

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

prEN 16166:2020 (E)

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 at least 4 h or by rinsing with a non-chlorinated solvent.

Samples shall be kept cold at a temperature < 8 °C and in the dark. The sample pretreatment for sludge samples should take place within 24 h of sampling. Alternatively, samples may be frozen (–18 °C) directly after sampling and kept frozen for a maximum of one month before sample pretreatment.

8.2 Sample pretreatment

Pretreat the samples according to EN 16179, if not otherwise specified.

Cool the dried test sample in a desiccator, comminute and homogenize in an analytical mill or porcelain mortar (7.3.4) to a particle size of no more than 0,1 mm.

Store the ground material in a desiccator or a tightly closed glass container.

The homogenized wet sample portion may alternatively be freeze-dried, which in some cases makes homogenizing of the dried sample easier.

9 Procedure**9.1 General**

The test sample taken for analysis shall have an AOX value within the working range of the instrument.

NOTE A typical working range is between 1 µg to 30 µg Chlorine (absolute titrated amount).

9.2 Adsorption and inorganic halide removal

Ensure that the dried, ground test sample is homogenized by stirring or shaking before taking the test portion. Prepare the test portion for combustion as follows:

- Transfer a homogenized test portion of 10 mg to 100 mg depending on the expected AOX content to a conical flask or screw cap vial (7.2.3).
- Add approx. 20 mg activated carbon (6.1) and 10 ml nitrate stock solution (6.7) to the sample. The amount of activated carbon shall be the same for all test samples, blank determinations, etc.
- Shake for 1 h using a mechanical shaker (7.2.4).
- Filter the suspension through a filtration apparatus (7.2.1) using nitrate washing solution (6.8). Wash the filter cake with small amounts of nitrate washing solution. Use a total volume of 25 ml to ensure complete transfer.
- Transfer the moist filter and the filter cake to the combustion apparatus according to manufacturer's instructions and proceed according to 9.3.

The laboratory should ensure that the nitrate washing procedure is sufficient to remove all inorganic halides typically found in the types of samples routinely analysed. If the concentration of AOX is found to decrease with additional washing stages, then the number of nitrate washes should be increased accordingly.

Laboratories should evaluate the methods' repeatability for the test sample mass routinely used. Test sample masses below 25 mg often result in increased repeatability standard deviation.

In case of difficult filtration properties, centrifuging the suspension before filtration is recommended.