



BSI Standards Publication

**Soil, treated biowaste and sludge — Determination
of adsorbed organically bound halogens (AOX)**

EUROPEAN STANDARD

EN 16166

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2021

ICS 13.030.01; 13.080.10

Supersedes EN 16166:2012

English Version

Soil, treated biowaste and sludge - Determination of adsorbed organically bound halogens (AOX)

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

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

This European Standard was approved by CEN on 22 November 2021.

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European foreword

This document (EN 16166:2021) has been prepared by Technical Committee CEN/TC 444 “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of an identical text or by endorsement, at the latest by June 2022, and conflicting national standards shall be withdrawn at the latest by June 2022.

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

This document supersedes 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.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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Introduction

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).

NOTE This method can also be applied to other environmental solid matrices, provided the user has verified the applicability.

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

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 operationally defined method for the direct determination of organically bound halogens (chlorine, bromine and iodine) adsorbed and occluded to the sample matrix. AOX being a methodologically defined parameter, it is essential that the procedure is applied without any modification.

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

NOTE This method can also be applied to other environmental solid matrices, provided the user has verified the applicability.

2 Normative references

There are no normative references in this document.

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 <https://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 activated 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 fraction 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.

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.

For samples with significant amounts of volatile, purgeable organic halogen compounds negative bias can occur. 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.

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 information for the storage of activated carbon, see Annex B.

The blank value of the activated carbon, expressed as chloride, shall be less than 15 µg per gram of activated carbon.

6.2 Nitric acid, HNO₃, ρ = 1,4 g/ml, 65 % 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.

If stored in a brown glass bottle, the solution is stable for three months.

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.

If stored in a brown glass bottle, the solution is stable for three months.

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.

If stored at (5 ± 3) °C the solution is stable for one month.

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, a microcoulometer, capable of determining 1 µg absolute amount of chloride with a repeatability variation coefficient of less than 10 %.

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-chlorobenzoic acid 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 Sampling and storage

For the sampling and storage of sludge samples, refer to existing standard, e.g. [2] and [3].

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.

Samples shall be kept at a temperature of (5 ± 3) °C and in the dark. The sample pretreatment for sludge samples should take place within 24 h of sampling. Alternatively, samples can be frozen (-18 ± 3) °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 [8], 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 homogenised 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 portion 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, without increasing the volume of washing solution.

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.

9.3 Combustion

The temperature in the combustion apparatus (7.1.1) shall be at least 950 °C; select other operating parameters in accordance with the manufacturer's instructions.

Introduce the sample into the heated zone of the combustion tube and combust according to manufacturer's instructions.

9.4 Chlorine determination, preliminary tests and daily checks

9.4.1 General

The capability of the measuring device to determine halogen concentrations (7.1.3) is to be checked over the whole working range according to manufacturer's instructions. It is recommended to check recovery of the hydrochloric acid solution (6.3) at least once before initial check of the overall procedure.

In case the same staff is carrying out analysis according to EN ISO 9562, using the same apparatus and applying the shaking method, the results of the initial check (see 9.5.2.2 of EN ISO 9562:2004) of the shaking method may be used instead of the initial check described in 9.4.2.1 of this document. The daily check according to 9.4.2.2 is to be carried out in all cases.

9.4.2 Checks on the overall procedure

9.4.2.1 Initial check

An initial check is necessary when the method is first introduced into the laboratory and if:

- on the daily check (9.4.2.2) a deviation of > 10 % is obtained repeatedly;
- different or new staff are employed to carry out the determinations.

The initial check is carried out as follows:

- Analyse at least one blank sample (9.5);
- Analyse at least 3 standard samples covering the working range prepared in the following way:
 - Suspend approx. 20 mg of activated carbon (6.1) in 10 ml of nitrate stock solution (6.7);
 - With a syringe (7.1.5), inject a suitable volume of 4-chlorophenol stock solution (6.9) or 2-chlorobenzoic acid stock solution (6.10) to give the desired amount of AOX. Table 2 gives an example for suitable volumes for injection;

Table 2 — Example injection volumes for initial check

Injected volume of 4-chlorophenol stock solution	Injected volume of 4-chlorobenzoic acid stock solution	Absolute amount of AOX
10 µl	8 µl	2 µg
50 µl	40 µl	10 µg
125 µl	100 µl	25 µg

— Proceed according to 9.2 and 9.3, starting with shaking.

— Test the correlation of the measured values by comparison with the nominal AOX values (recovery function).

The result is acceptable if the correlation coefficient is $\geq 0,999$ and the slope of the recovery function is within the range of 0,95 to 1,05.

9.4.2.2 Daily check

For every batch of sample, check the entire procedure by analysing at least one standard sample as described in 9.4.2.1 in the middle of the working range. The AOX values thus obtained and the theoretical value shall not deviate more than 10 % (recovery 90 % to 110 %).

9.5 Blank determination

Suspend approx. 20 mg of activated carbon (6.1) in 10 ml of nitrate stock solution (6.7) and proceed in the same way as described in 9.2.

The amount of activated carbon used for the blank determination should be equal to the amount used for the analysis of test samples.

Carry out at least two blank determinations in each series and use the average blank value for subsequent calculations.

The absolute mass of halogens determined in the blank, expressed as chloride, $m_{\text{Cl}}(\text{blank})$ shall not exceed 3 µg.

NOTE In practice, values $< 1 \mu\text{g}$ are easily obtainable.

10 Calculation

10.1 Mass concentration of adsorbed organically bound halogens [$\rho_{\text{Cl}}(\text{AOX})$] in dried sample

The mass fraction is calculated according to Formula 1:

$$w_{\text{Cl}}(\text{AOX}) = w_{\text{Cl}}(\text{sample}) - w_{\text{Cl}}(\text{blank}) = \frac{m_{\text{Cl}}(\text{sample}) - m_{\text{Cl}}(\text{blank})}{m(\text{sample})} \quad (1)$$

where

$w_{\text{Cl}}(\text{AOX})$	is the calculated mass fraction of adsorbed organically bound halogens, expressed as chloride, in mg/kg dm;
$w_{\text{Cl}}(\text{sample})$	is the uncorrected $w_{\text{Cl}}(\text{AOX})$ value for the test sample;
$w_{\text{Cl}}(\text{blank})$	is $w_{\text{Cl}}(\text{AOX})$ value of the blank determination;
$m_{\text{Cl}}(\text{sample})$	is the uncorrected absolute mass of halogens determined in the sample, expressed as chloride, in μg ;
$m_{\text{Cl}}(\text{blank})$	is the absolute mass of halogens determined in the blank, expressed as chloride in μg ;
$m(\text{sample})$	is the mass of the test sample, in g.

In case of microcoulometric titration, calculate the absolute mass of halogens (m_{Cl}) in sample or blank according to formula:

$$m_{\text{Cl}} = \frac{Q \times M}{F} \quad (2)$$

where

Q	is the measured quantity of charge, expressed in Coulomb (C);
M	is the molar mass of chloride ($M = 35,45 \times 10^6 \mu\text{g/mol}$);
F	is the Faraday constant ($F = 96\,484,56 \text{ C/mol}$).

NOTE This calculation is performed automatically by modern analytical equipment, and the mass of the halogens represents the raw data of the system.

10.2 Expression of results

The result shall be expressed in milligrams per kilogram (mg/kg) chloride on dry matter basis and reported to two significant figures.

11 Precision

The performance characteristics of the method have been evaluated, see Annex A for additional information.

12 Test report

The test report shall contain at least the following information:

- a) a reference to this document (EN 16166);
- b) complete identification of the sample;
- c) expression of results, according to 10.2;
- d) any details not specified in this document or which are optional, as well as any factor which may have affected the results.

Annex A (informative)

Repeatability and reproducibility data

A.1 Materials used in the interlaboratory comparison study

The interlaboratory comparison of adsorbed organically bound halogens (AOX) in sludge, treated biowaste and soil was carried out with 13 to 15 European laboratories on six materials. Detailed information can be found in the final report on the interlaboratory comparison study mentioned in [6].

Table A.1 lists the types of materials tested.

Table A.1 — Materials tested in the interlaboratory comparison of the determination of adsorbed organically bound halogens (AOX) in sludge, treated biowaste and soil

Grain size	Sample	Material
Sludge (< 0,5 mm)	Sludge 1	Mix of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
	Sludge 2	Mix of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
Fine grained (< 2,0 mm)	Compost 1	Fresh compost from Vienna, Austria
	Compost 2	Compost from Germany
	Soil 4	Sludge amended soil from Hohenheim, Germany
	Soil 5	Agricultural soil from Reading, United Kingdom

A.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table A.2).

Table A.2 — Results of the interlaboratory comparison studies of determination of adsorbed organically bound halogens (AOX) in sludge, treated biowaste and soil

Matrix	l	n	n_o	\bar{x} mg/kg	s_R mg/kg	$C_{V,R}$ %	s_r mg/kg	$C_{V,r}$ %
Sludge 1	17	69	1	241	27,8	13,0	19,6	9,2
Sludge 2	15	45	4	188	15,2	8,1	8,7	4,6
Compost 1	6	27	0	42,6	11,5	27,0	3,3	7,8
Compost 2	14	54	1	46,4	11,8	25,3	5,0	10,7
Soil 4	14	62	0	26,7	9,1	34,2	2,6	9,9
Soil 5	13	62	0	21,5	6,8	31,7	3,0	13,8

Explanation of symbols

l number of participating laboratories

N number of analytical results after outlier rejection

n_o number of outliers

\bar{x} total mean of results (without outliers)

s_R reproducibility standard deviation

$C_{V,R}$ coefficient of variation of reproducibility

s_r repeatability standard deviation

$C_{V,r}$ coefficient of variation of repeatability

Annex B (informative)

Storage of activated carbon

Activated carbon of an adequate adsorption capacity and of low content of inorganic halides, suitable for the AOX determination, is commercially available. After opening of the original container, the activated carbon can become contaminated with adsorbable compounds (including organic halogens), coming from the air. The activated carbon can become inactive within five days.

In order to keep the carbon blank low, take small quantities sufficient for one day work, e.g. 1,5 g to 2 g, and transfer to suitable sealed glass containers.

Use the contents of the container on the day of its opening. Discard the unused remainder at the end of the day.

Keep the remaining stock sealed. If the carbon blank is too high, use another batch after checking its blank.

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