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

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National foreword

This British Standard is the UK implementation of EN 16166:2012.

The UK participation in its preparation was entrusted to Technical Committee H/-/4, Environmental testing programmes.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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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COMITÉ EUROPÉEN DE NORMALISATION
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Contents

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.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

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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 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, $c(\text{HCl})$ = 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.

Samples shall be kept cold at a temperature < 8 °C and in the dark. The sample pretreatment 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 homogenised wet sample portion may alternatively be freeze-dried, which in some cases makes the homogenizing of the dried sample easier.

9 Procedure

9.1 General

The test sample taken for analysis shall have an AOX value within the optimal working range of the instrument, which is generally between 1 µg to 30 µg (absolute amount).

9.2 Adsorption and inorganic halide removal

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

Transfer a homogenised test portion of 5 mg to 100 mg depending on the expected AOX content to a conical flask or screw cap vial (7.2.3).

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.

Add 20 mg to 30 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.

Place the moist filter and the filter cake into the quartz sample boat (7.1.2) 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.

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.

Connect the gas supply to the quartz tube (7.1.1) and the quartz tube to the absorber (7.1.4). Do not allow back-siphoning of the sulfuric acid (6.4) into the quartz tube; this can happen if a drop in temperature or pressure occurs.

Adjust the flow of gas (6.5) to approximately 150 ml/min or follow manufacturer's instructions.

Introduce the quartz sample boat (7.1.2) into the heated zone of the tube, following the manufacturer's instructions.

NOTE A number of experimental variables, e.g. the amount of activated carbon applied, the test portion size, the dimensions of the furnace, residence time, combustion temperature, and the adjustment of the gas flow depend on the type of apparatus and inappropriate selection may adversely affect the result.

It was demonstrated that carbon to sample ratios lower than 1 (1:1) may lead to incomplete combustion of the sample/carbon mixture (seen as tailing during the argentometric detection). If this is the case, the amount of activated carbon should be raised when test portions larger than 20 mg to 30 mg are analysed. The amount of activated carbon for the corresponding blank determinations should be raised as well.

9.4 Initial calibration

9.4.1 General

Calibrate the analytical instrument (7.1) using one of the two methods described below. The calibration of the entire system is the preferred method (see 9.4.2). The method described in 9.4.3 is useful for checking the microcoulometric cell.

9.4.2 Calibration of the entire system

Calibrate the system by analysing at least three different levels of AOX covering the whole of the relevant working range (e.g. 0,5 µg, 5,0 µg and 10,0 µg Cl^- or 1,0 µg, 10,0 µg and 20,0 µg Cl^-) in the following way:

Transfer 20 mg to 30 mg activated carbon (6.1) to the filtration apparatus (7.2.1) and moisten it with a small amount of nitrate washing solution (6.8).

Place the moist filter and the activated carbon into the quartz sample boat (7.1.2).

Inject 5 µl or 10 µl of the 4-chlorophenol stock solution (6.10) or a 4-chlorophenol working solution (6.11) giving the desired amount of AOX (see Table 2) and proceed with combustion according to 9.3.

Table 2 — Suggested calibration solutions

Volume added µl	**Concentration of solution** µg/µl	**Amount of AOX** µg
5	0,1	0,5
10	0,1	1,0
10	0,5	5,0
5	2,0	10,0
10	2,0	20,0

Establish a linear relationship between the amount of AOX analysed and the instrument response according to Formula (1):

$$N_i = i + s \times m_{\mathrm{Cl}} \tag{1}$$

where

N_i is the instrument response, in Coulomb (C) or instrument-dependent units;

i is the y-axis intercept, in Coulomb (C) or instrument-dependent units;

s is the slope, in Coulomb (C) or instrument-dependent units per microgram (µg) Cl;

m_{Cl} is the amount of AOX, in micrograms (µg) Cl.

Use the slope (s) for subsequent calculation of mass concentrations of AOX (see 10.1.1).

9.4.3 Check of argentometric measuring device

In the case of microcoulometric determination, check the instrument within the relevant working range, using at least one test solution as follows:

Inject directly with a syringe (7.1.5) a known volume within the range of 5 µl to 8 µl of the hydrochloric acid solution (6.3) into the titration cell.

Measure the quantity of charge transferred in this test.

Coulometry theory assumes a 100 % current yield. But for practical purposes, it is important to determine the actual current yield. Obtain the correction factor by using Formula (2):

$$Q = a \times Q_{\mathrm{t}} \tag{2}$$

where

Q is the measured quantity of charge for the hydrochloric acid sample, in Coulomb (C);

Q_{t} is the theoretical quantity of charge for the hydrochloric acid sample, in Coulomb (C);

a is the correction factor.

Obtain the theoretical quantity of charge, Q_{t}, using Formula (3):

$$Q_{\mathrm{t}} = V \times c_{\mathrm{Cl}} \times F \tag{3}$$

where

V is the volume of the hydrochloric acid solution, in litres (l);

c_{Cl} is the chloride concentration of the hydrochloric acid solution, in mol per litre (mol/l);

F is the Faraday constant, F = 96 487 C/mol.

The measuring device is suitable for the analysis, if the correction factor, a, is in the range of 0,97 to 1,03.

9.5 Recalibration

For every batch of samples, analyse at least two calibration standards with the concentrations of (20 ± 10) % and (80 ± 10) % of the established linear range and calculate the straight line from these measurements. If the straight line falls within 95 % of the initial calibration relationship according to Formula (1), the initial calibration line is assumed to be valid. If not, a new calibration line shall be established according to 9.4.

9.6 Blank determination

Suspend 20 mg to 30 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.

10 Calculation

10.1 Method of calculation

10.1.1 Entire system calibration (according to 9.4.2)

Calculate the mass concentration of adsorbable organically bound halogens, expressed as chloride, using Formula (4):

$$Q_{\mathrm{Cl}}(\mathrm{AOX}) = \frac{N - N_0}{m_{\mathrm{T}} \times s} \tag{4}$$

where

$Q_{\mathrm{Cl}}(\mathrm{AOX})$	is the mass concentration of adsorbable organically bound halogens, expressed as chloride concentration on dry matter basis, in milligrams per kilogram (mg/kg);
N_0	is the average value for the blank, according to 9.6, in Coulomb (C) or instrument-dependent units;
N	is the measured value for the test sample, in Coulomb (C) or instrument-dependent units;
s	is the slope of the calibration curve as defined in Formula (1);
m_{T}	is the mass of the dried test sample, in grams (g).

10.1.2 Direct argentometric measurement device calibration (according to 9.4.3)

Calculate the mass concentration of adsorbable organically bound halogens, expressed as chloride, using Formula (5):

$$Q_{\mathrm{Cl}}(\mathrm{AOX}) = \frac{N - N_0}{m_{\mathrm{T}} \times s} \times \frac{M \times a}{F} \times 10^6 \tag{5}$$

where

$Q_{\mathrm{Cl}}(\mathrm{AOX})$	is the mass concentration of adsorbable organically bound halogens, expressed as chloride concentration on dry matter basis, in milligrams per kilogram (mg/kg);
N_0	is the average value for the blank, according to 9.6, in Coulomb (C);
N	is the measured value for the test sample, in Coulomb (C);
M	is the molar mass of chloride, M = 35,45 g/mol;
m_{T}	is the mass of the dried test sample, in grams (g);
a	is the factor relating the theoretical to the actual quantity of charge (see Formula (2));
F	is the Faraday constant, F = 96 487 C/mol.

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.

12 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 16166);

b) complete identification of the sample;

c) expression of results, according to 10.2;

d) any details not specified in this European Standard 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 adsorbable 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 adsorbable 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 adsorbable organically bound halogens (AOX) in sludge, treated biowaste and soil

Matrix	l	n	n_o	$\bar{\bar{x}}$ 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
$\bar{\bar{x}}$	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 halogenide, 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.

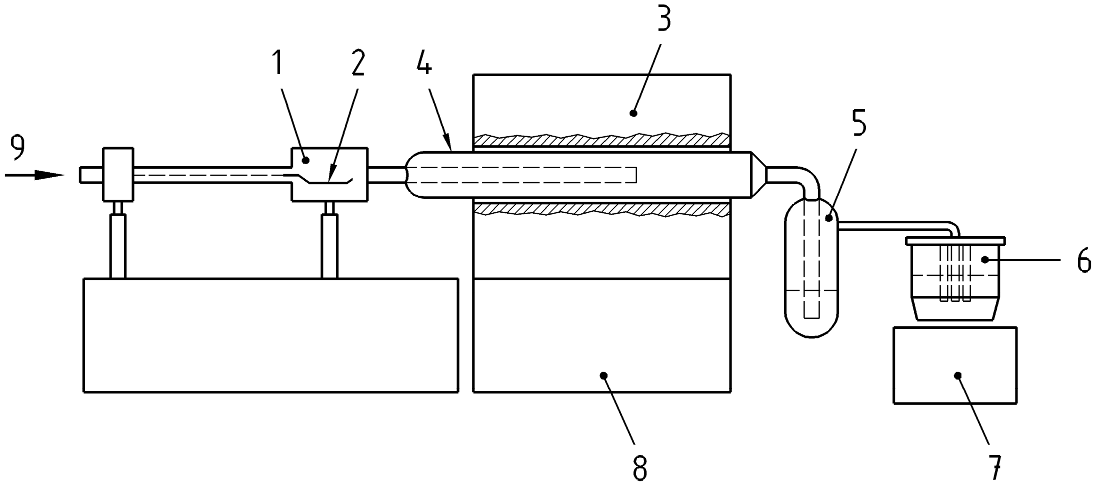
In order to keep the carbon blank low, take small quantities sufficient for one day work, e.g. 1,5 g to 2 g, and transfer to suitable sealed glass containers.

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.

Annex C
(informative)

Schematic diagram of an AOX apparatus



Key

1 sample inlet for AOX
2 AOX sample boat
3 furnace
4 quartz tube
5 absorber filled with sulfuric acid
6 titration cell
7 stirrer
8 control device for temperature, gas flow
9 combustion gas inlet

Figure C.1 — Schematic diagram of an AOX apparatus [EN ISO 9562]

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[5] DIN 38414-18, *German standard methods for the examination of water, waste water and sludge; sludge and sediments (group S); determination of adsorbed organically bound halogens (AOX) (S 18*)

[6] Horizontal Project Interlaboratory Comparison, www.horizontal.ecn.nl

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