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Solid recovered fuels — Methods for the determination of sulphur (S), chlorine (Cl), fluorine (F) and bromine (Br) content

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National foreword

This British Standard is the UK implementation of EN 15408:2011. It supersedes DD CEN/TS 15408:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

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

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Solid recovered fuels - Methods for the determination of sulphur (S), chlorine (Cl), fluorine (F) and bromine (Br) content

Combustibles solides de récupération - Méthodes pour la détermination de la teneur en soufre (S), en chlore (Cl), en fluor (F), et en brome (Br)

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Schwefel (S), Chlor (Cl), Fluor (F) und Brom (Br)

This European Standard was approved by CEN on 22 January 2011.

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Foreword

This document (EN 15408:2011) has been prepared by Technical Committee CEN/TC 343 “Solid recovered fuels”, the secretariat of which is held by SFS.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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Introduction

Determination of total sulphur, chlorine, fluorine and bromine content of solid recovered fuels (SRF) is necessary for environmental and technical reasons both in the production and combustion stage.

During the combustion process they are usually converted to sulphates and halides. These reaction products contribute significantly to corrosion and environmentally harmful emissions.

This method consists of an oxygen combustion procedure followed by trapping of sulphur, chloride, fluoride and bromide in an absorbing solution and subsequent determination by different techniques.

Alternatively, direct automatic techniques can be used for S and Cl determination. Other methods could also be used provided that it is demonstrated that they give the same results.

1 Scope

This European Standard specifies the determination of S, Cl, F and Br in solid recovered fuels of various origin and composition after combustion in oxygen atmosphere. This method is applicable for concentrations over 0,025 g/kg, depending on the element and on the determination technique. In the case of fluorine this method is applicable for concentration over 0,015 g/kg.

Insoluble halides and sulphate present in the original sample or produced during the combustion step are not completely determined by these methods.

This European Standard provides recommendations concerning standardised methods for determination of halides and sulphate in the solution obtained after combustion.

2 Normative references

The following referenced documents are indispensable for the application 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 15357:2011, *Solid recovered fuels — Terminology, definitions and descriptions*

EN 15413[1)], *Solid recovered fuels — Methods for the preparation of the test sample from the laboratory sample*

EN 15414-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 10304-1:2009, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007)*

EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2:2003)*

ISO 9297, *Water quality — Determination of chloride — Silver nitrate titration with chromate indicator (Mohr's method)*

ISO 10359-1, *Water quality — Determination of fluoride — Part 1: Electrochemical probe method for potable and lightly polluted water*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15357:2011 and the following apply.

3.1
halogen content
sum of halogens contained as organic and inorganic compounds in the solid recovered fuels, which can be converted to halides (fluoride, chloride, bromide, iodide) by combustion and then absorbed or dissolved in aqueous solution

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[1)] To be published.

NOTE The above definition is valid for this empirical European Standard only and do not comply with scientific definitions of halogen content.

3.2
oxygen combustion
combustion of material in oxygen atmosphere

4 Safety remarks

The safety in handling of potentially hazardous materials is dealt with relevant national and European regulations, which every laboratory should refer to.

In addition the following information is given:

— only experienced personnel can use the oxygen combustion apparatus, following the operating instructions described in the manufacturer manual;

— precautions shall be taken by the operator for reactive gas (oxygen) at high temperature and high pressure.

5 Principle

The determination of S, Cl, F and Br is carried out in two steps or by using automatic equipment:

— the sample is oxidized by combustion in a bomb containing oxygen under pressure. Halogenated and sulphur compounds are converted respectively to fluoride, chloride, bromide and sulphate which are absorbed and/or dissolved in an absorption solution (water or KOH 0,2 mol/l solution);

— analysis of Cl, F and S and Br by ion chromatography or other suitable technique, are reported in the reference documents listed in Clause 2: for Cl, F, S and Br in EN ISO 10304-1, for F only in ISO 10359-1. Br is preferably determined by “Inductively Coupled Plasma Mass Spectrometry” (ICP-MS) according to EN ISO 17294-2 since several oxidation states of Br occur after oxygen combustion.

6 Apparatus

Apart ordinary laboratory apparatus, the following devices are required:

6.1 Oxygen combustor

Equipped with a combustion bomb made of stainless steel or any other material that will not be affected by the combustion process or products (the materials used may adsorb or react with acid gases formed during combustion or it may be not possible to clean the bomb completely between combustions). The bomb is equipped with oxygen inlet and safety valve and electrical contacts for spark generation. Many commercially available systems can be used. The combustion bomb may be the same as used for the determination of the calorific value.

Carefully check the characteristics of the combustion bomb, in order to be sure that it is suitable for the processing of materials with significant chlorine content (chlorine resistant combustion bombs are commercially available).

The combustion apparatus is equipped with automatic ignition system and oxygen gas supply.

6.2 Balances

— Analytical balance resolution ± 0,1 mg;

— balance accuracy ± 0,1 g.

6.3 Ion chromatograph

An ion chromatograph with suitable anion separator column, pre-column, background suppressor and conductivity cell.

6.4 Apparatus for titrimetry

Any suitable apparatus can be used, with colorimetric or potentiometric final point determination.

6.5 Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Any suitable apparatus with normal resolution and pneumatic sample introduction system.

6.6 Automatic analyzer

Commercial instruments for S and Cl determination.

7 Reagents

All reagents shall be at least of analytical grade and suitable for their specific purposes. Particularly, they shall be free of sulphur and halogens.

7.1 Water of grade 1 as specified by EN ISO 3696.

7.2 Oxygen

Should be free of combustion material, minimum 99,99 % purity.

7.3 Nitrogen

Should be chromatographic grade for the ion chromatograph.

7.4 Eluent for ion chromatography

Carbonate/hydrogen carbonate mixed solution is suitable as eluent for ion chromatographic separation. Other eluents can be used, following the working instruction with the particular column used.

7.5 Absorbing solution

Water is appropriate for most application. If the content of chlorine is > 1 % or if bromine shall be determined, alkaline KOH 0,2 mol/l solution is more efficient for trapping the gases. As a preliminary check, XRF analysis can be used to check for the presence of Br or high chlorine content.

7.6 Stock standard solutions

1 000 mg/l chlorine, fluorine, bromine and sulphate commercially available standard solutions are used to prepared working and calibration solution by properly dilution.

7.7 Certified reference material (CRMs)

The trapping yield can be checked using a material with characteristic similar to SRF, e.g. a solid waste certified reference material.

7.8 Control mixtures

To create an appropriate control mixture, choose the control substances in combination so that all elements that shall be determined in the samples are represented. The amount of halogen and sulphur contents shall be in the same range of the element contents of the samples and approximately in the middle of the working range of the determination techniques. The mixture of the control substances needs to be homogenized using a pebble mill.

EXAMPLE An example of a mixture of control substances for the determination of fluorine, chlorine, bromine and sulphur is:

0,50 g 4-fluoro-benzoic acid; 2,0 g 4-chloro-benzoic acid; 0,25 g 4-bromo-benzoic acid; 0,25 g 4-iodo-benzoicacid; 2,0 g sulphanilic acid and 55,0 g cellulose are mixed. The mixture is homogenized, e.g. in a pebble mill. This mixture contains 1,13 g/kg fluorine; 7,547 g/kg chlorine; 1,656 g/kg bromine; 2,132 g/kg iodine and 6,17 g/kg sulphur.

8 Interferences and sources of error

The container in which the sample is delivered and stored can be a source of errors. Its material shall be chosen according to the elements to be determined. Grinding or milling samples includes a risk of contamination of the sample by the environment.

NOTE In fuels with high contents of sulphur bound to iron, the method can show different result.

9 Procedure

9.1 Sample conservation and pre-treatment

The laboratory samples shall be stored according to guidelines defined in Annex A.

9.2 Sample preparation

The test portion shall be prepared from the laboratory sample according to EN 15413.

In addition, for the purposes of this method, the target size should be 1 mm or below.

The amount of test portion is usually 1 g. For some instruments it may be necessary to weigh a lower amount: in this case a nominal size less than 1 mm is required in order to ensure the homogeneity of the test portion, according to EN 15413.

Whereas the determination is carried out on dry basis, the moisture content shall be determined according to EN 15414-3.

NOTE In some cases, when the sample is not homogeneous as regards chlorine-containing particles, finer particle size (e.g. 0,5 mm) would make better repeatability of analysis (see Annex C).

9.3 Bomb combustion

Set up the instrument following the manufacturer instructions.

Weigh about 1 g of test material. The test portion, weighed to 0,1 mg, can be:

— pressed to produce an unbreakable pellet;

— placed and weighed directly in a capsule (with 20 % water or without water);

— placed and weighed directly in a small PE bag (with 20 % water or without water);

— mixed with a combustion enhancer, like benzoic acid or any other not containing significant amounts of elements of interest.

The amount of sample used has to be reduced accordingly when a combustion aid (benzoic acid, PE-bag, capsule) is used because the combustion aid introduces an additional significant heating value. It is in many systems important not to exceed the max temperature rise in the bomb for safety reasons and for getting best results; the temperature rise is to be kept in a small band adjusting the amount of sample and combustion aid. This is expecially important when heating value is subsequently determined.

Add 10 ml of 0,2 mol/l KOH solution; fill the bomb with oxygen and set up the system following the operator instructions. If the chlorine content is < 1 % or bromine is not to be determined, water can be used instead of KOH solution.

Start the combustion. After combustion, equilibrate at room temperature for at least 10 min. After opening the bomb, check the completeness of combustion, by analysing residues. If unburned fractions are present the sample shall be discharged. Quantitatively recover the absorbing solution in a 100 ml tared bottle and fill up to the final volume with water.

Determine chloride, fluoride, sulphate and bromide by using the proper method, EN ISO 10304-1, ISO 9297, ISO 10359-1 or EN ISO 17294-2.

NOTE Robustness study (see Annex C), showed that the method is not applicable in its current form to some samples (e.g. containing tyre pieces) because the prepared pellet tend to break during combustion.

9.4 Calibration

Prepare a series of calibration solutions by dilution of the stock concentrated solution. These solutions are stable for at least 1 month if stored refrigerated.

9.5 Analysis of calibrations samples

Measure the calibration samples according to the instrument user’s manual.

Calculate the regression curve.

9.6 Analysis of samples

Measure the samples according to the instrument user’s manual.

The samples shall be analysed with the same analytical conditions as for the calibration samples.

10 Calculation and evaluation

10.1 General

Results are referred to the dry material. Calculation shall take into consideration any eventual intermediate dilution of the solution.

10.2 Total chlorine or total fluorine or total bromine

The total chlorine or fluorine or bromine content expressed in % dry weight is given by:

$$w = \frac{(c - c_0) \times V}{m} \times 100$$

where

c is the concentration of chloride, fluoride or bromide respectively in the solution [mg/l];

c_0 is the concentration of chloride, fluoride or bromide respectively in the blank solution [mg/l];

V is the volume of the solution [l];

m is the mass of the test portion used [mg].

10.3 Total sulphur

The total sulphur content expressed in % dry weight is given by:

$$w_s = \frac{(c - c_0) \times V}{m} \times 0{,}3338 \times 100$$

where

c is the concentration of sulphate in the solution [mg/l];

c_0 is the concentration of sulphate in the blank solution [mg/l];

V is the volume of the solution [l];

m is the mass of the test portion used [mg];

0,333 8 is the stoichiometric ratio of the relative masses of sulphur and sulphate.

11 Quality control

To detect possible contaminations from vessels and/or reagents, blank tests shall be carried out by the same sample preparation procedure, using the same quantities of reagents.

In order to check the reliability of the whole procedure, a control test should be carried out for each series of determinations, using a control mixture (7.8). A triplicate analysis of one point in the middle of the working range is sufficient. For each element, the mean recovery has to be between 90 % and 110 % with a coefficient of variation less than 10 %.

If available, the use of standard reference materials is recommended. It is recommended too, that quality control charts are used for control mixtures, as a minimum.

12 Performance characteristics

Data on performance characteristics of the present method are given in Annex B which presents the results of the QUOVADIS validation project (QUality management Organisation, VAlidation of standards, Developments and Inquiries for Solid-recovered fuels).

13 Test report

The test report shall contain at least the following information:

a) name, address and location of any laboratory involved in the analysis;

b) description and identification of the laboratory sample;

c) date of receipt of laboratory sample and date(s) of performance of test;

d) a reference to this European Standard, i.e. EN 15408;

e) reference to the analytical standard used for the determination for each element;

f) the analytical results, referring to Clause 10;

g) any details not specified in this European Standard or which are optional, and any other factors which may have affected the results;

h) unique identification of report (such as serial number) and of each page and total number of pages of the report.

The laboratory should keep a trace of any analytical steps and intermediate results (chromatograms, raw data and calculation details) that should be kept available in case of specific requirements (e.g. accreditation).

Annex A
(normative)

Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF

The following requirements apply when preparing the laboratory sample for the chemical characterisation of SRF samples according to this draft European Standard.

NOTE Equivalent requirements apply in all chemical test method specifications for SRF, i.e. EN 15407, this European Standard, EN 15410, EN 15411, CEN/TS 15412 and EN 15413.

A maximum amount of laboratory sample of 10 kg and maximum particle size of 1 cm is established on the basis of number and type of parameters to be determined, sample representativeness and practical reasons for handling samples. In the Table A.1 the requirements are summarised both for single or grouped chemical parameters.

Table A.1 — Requirements for the laboratory sample for the analysis of SRF

Parameter (single or group)	Minimum laboratory sample amount (g) [a]	Short term storage conditions before delivery to the lab	Long term storage condition before delivery to the lab	Container material
C, H, N	100	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Cl, S, Br, F	100	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Metallic Al	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements	400	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Trace elements excluding Hg	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Hg	100	In the same condition of SRF production	refrigeration 4 °C	glass or PFA bottle
C, H, N, Cl, S, Br, F	150	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Major elements + trace elements excluding Hg	500	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements + trace elements + Hg	600	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + plastic bottle or bag

Table A.1 — (*concluded*)

Major elements + trace elements + Hg + metallic Al	700	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + plastic bottle or bag
Complete analysis	800	In the same condition of SRF production	refrigeration 4 °C	glass bottle (100 g) + non-PVC plastic bottle or bag
[a] The maximum particle size (mm) is related to the laboratory sample amount (g) in order to guarantee sample homogeneity. It is established following the rules reported in EN 15413.				

Annex B
(informative)

Data on performance characteristics

The method described in this European Standard has been validated within the QUOVADIS project. In this validation project several SRF were selected to provide a reasonable coverage of SRF. They were tested according to the present standardised method. Results and robustness, repeatability and reproducibility data are reported in the document : "EUR 23552 EN - 2008 - Joint Research Centre – Institute for Environment and Sustainability - QUOVADIS Project - Organization of Validation Exercises - JRC Scientific and Technical Reports - ISBN 978-92-79-10396-4 - Luxembourg: Office for Official Publications of the European Communities".

Interlaboratory trials were carried out by laboratories in Austria, Belgium, France, Germany, Italy, the Netherlands and the United Kingdom. The variety of instruments and other analytical conditions used conformed to the quality parameters specified in the method.

The performance data according to ISO 5725-2 are presented in Tables B.1 to B.4.

The data derive from laboratories participating in the above-mentioned interlaboratory trials.

Table B.1 — Performance data for sulphur

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{\bar{x}}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	160	14	4,7	na	1,42	na	0,181	12,7	0,103	7,30
B	SRF produced from demolition wood	144	13	7,7	na	0,03	na	0,013	43,3	0,007	23,3
C	SRF produced from sewage sludge	156	14	7,1	na	1,06	na	0,144	13,6	0,059	5,60
D	SRF produced from municipal waste	154	14	8,3	na	0,25	na	0,029	11,6	0,029	11,6
E	SRF produced from municipal waste (paper and plastic reach)	154	14	8,3	na	0,13	na	0,026	20,0	0,013	10,0

Definition of symbols

l	is the number of outlier-free individual analytical values per level;
n	is the number of laboratories after outlier elimination;
o	is the percentage of outlying values from the replicate determination;
x_{ref}	is the accepted reference value on dry matter base;
$\bar{\bar{x}}$	is the overall mean on dry matter base;
η	is the recovery rate;
s_R	is the reproducibility standard deviation on dry matter base;
CV_R	is the coefficient of the variation of the reproducibility;
S_r	is the repeatability standard deviation on dry matter base;
CV_r	is the coefficient of the variation of the repeatability;
na	not applicable.

Table B.2 — Performance data for chlorine

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{x}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	138	13	11,5	na	0,050	na	0,024	48,0	0,016	32,0
B	SRF produced from demolition wood	156	14	7,1	na	0,294	na	0,056	19,0	0,044	15,0
C	SRF produced from sewage sludge	156	14	7,1	na	0,311	na	0,087	28,0	0,027	8,70
D	SRF produced from municipal waste	156	14	7,1	na	0,571	na	0,153	26,8	0,102	17,9
E	SRF produced from municipal waste (paper and plastic reach)	156	14	7,1	na	0,932	na	0,181	19,4	0,099	10,6
Definition of symbols: see Table B.1.											

Table B.3 — Performance data for bromine

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{x}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	114	10	5,0	na	0,030	na	0,0138	46,0	0,0067	22,2
B	SRF produced from demolition wood	49	5	18,3	na	0,0027	na	0,0012	4,40	0,0006	2,20
C	SRF produced from sewage sludge	60	6	16,7	na	0,0029	na	0,0008	27,6	0,0003	10,3
D	SRF produced from municipal waste	65	7	22,6	na	0,0065	na	0,0024	36,9	0,0024	36,9
E	SRF produced from municipal waste (paper and plastic reach)	76	7	9,5	na	0,0110	na	0,0042	38,2	0,0042	38,2
Definition of symbols: see Table B.1.											

Table B.4 — Performance data for fluorine

Sample	Matrix	l	n	o %	x_{ref} %	$\bar{\bar{x}}$ %	η %	s_R %	CV_R %	S_r %	CV_r %
A	SRF produced from shredded tyres	92	8	4,2	na	0,0063	na	0,0098	156	0,0059	93,7
B	SRF produced from demolition wood	86	9	20,4	na	0,0027	na	0,0005	18,5	0,0005	18,5
C	SRF produced from sewage sludge	144	12	0,0	na	0,0311	na	0,0106	34,1	0,0051	16,4
D	SRF produced from municipal waste	118	11	10,6	na	0,0098	na	0,0026	26,5	0,0019	19,4
E	SRF produced from municipal waste (paper and plastic reach)	117	11	11,4	na	0,0100	na	0,0053	53,0	0,0048	48,0
Definition of symbols: see Table B.1.											

Annex C
(informative)

Major results of ruggedness testing

The design of the ruggedness testing was carried out by applying the analytical method to be validated with some controlled variations of analytical parameters (composition and different grain size: 0,5 mm, 1 mm and 1,5 mm) in repeatability conditions, in order to evaluate separately the influence of each varying parameter on the final results.

Robustness study shows that the method is not applicable in its current form to some SRF samples (e.g. containing rubber) because the prepared pellet tends to break during combustion. When working with SFR containing these materials, it is important to check whether the combustion is quantitative. It could be also advisable to collect separately the combustion gases outside the bomb in a vessel containing the sorption solution.

Repeatability is worse at larger grain size for Br and Cl, while it is similar for S. Recovery appears to be influenced by grain size only for sulphur. In case there is a need of increasing repeatability it is recommended to work on sample with grain size of 0,5 mm or less.

Bibliography

[1] EN 15442, *Solid recovered fuels — Methods for sampling*

[2] EN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

[3] ASTM D4239, *Standard Test Method for Sulphur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion*

[4] EN 15407, *Solid recovered fuels — Methods for the determination of carbon (C), hydrogen (H) and nitrogen (N) content*

[5] EN 15410[2)], *Solid recovered fuels — Method for the determination of the content of major elements (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)*

[6] EN 15411[2)], *Solid recovered fuels — Methods for the determination of the content of trace elements (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Ti, V and Zn)*

[7] CEN/TS 15412, *Solid recovered fuels — Methods for the determination of metallic aluminium*

[8] ISO 5725-5, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

[2)] To be published.

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