BS EN 15289:2011

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Solid biofuels — Determination of total content of sulfur and chlorine

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National foreword

This British Standard is the UK implementation of EN 15289:2011. It supersedes DD CEN/TS 15289:2006 which is withdrawn.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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ISBN 978 0 580 71232 6

ICS 75.160.10

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2011.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15289

February 2011

ICS 75.160.10

Supersedes CEN/TS 15289:2006

English Version

Solid biofuels - Determination of total content of sulfur and chlorine

Biocombustibles solides - Détermination de la teneur totale en soufre et en chlore

Feste Biobrennstoffe - Bestimmung des Gesamtgehaltes an Schwefel und Chlor

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COMITÉ EUROPÉEN DE NORMALISATION
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Ref. No. EN 15289:2011: E

Contents

Foreword

This document (EN 15289:2011) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

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of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.

This document supersedes CEN/TS 15289:2006.

In the pre-normative project BIONORM I&II a robustness test has been performed to find out if all critical parameters in the standard were addressed. Based on the results of that test it has been concluded that all critical parameters were covered. Only minor technical changes were necessary which have been implemented in the revised text. The revision also includes a change of deliverable from Technical Specification to European Standard and updated normative references.

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Introduction

Sulfur and chlorine are present in solid biofuels in varying concentrations. During the combustion process they are usually converted to sulfur-oxides and chlorides. The presence of these elements and their reaction products may contribute significantly to corrosion and to environmentally harmful emissions.

Chlorine may be present in different organic and inorganic compounds and should exceed or equal the water soluble amount that can be determined by EN 15105 [2].

Oxygen combustion in a closed oxygen bomb is the preferred method to digest biomass samples for a determination of the total content of sulfur and chlorine. An advantage of the method is that the digestion may be carried out in connection with determination of the calorific value according to EN 14918. Decomposition in closed vessels is an appropriate alternative method. Other analytical techniques (e.g. high temperature combustion in a tube furnace and Eschka method) may also be used. The determination of the resultant chlorine and sulfur compounds can be done by different techniques, e.g. ion chromatography, ICP, titrimetry.

Automatic equipment and alternative methods may be used when these methods are validated with biomass reference samples of an adequate type and also meet the requirements of Clause 10.

A list with typical sulfur and chlorine contents of biofuels can be found in EN 14961-1.

1 Scope

This European Standard specifies methods for the determination of the total sulfur and total chlorine content in solid biofuels. The standard specifies two methods for digestion of the fuel and different analytical techniques for the quantification of the elements in the digest solutions. The use of automatic equipment is also included in this European Standard provided that a validation is carried out as specified.

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.

CEN Guide 13:2008, *Validation of environmental test methods*

EN 14588:2010, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

FprEN 14780, *Solid biofuels — Sample preparation*

EN 14918, *Solid biofuels — Determination of calorific value*

EN 14961-1, *Solid biofuels — Fuel specifications and classes — Part 1: General requirements*

EN 15290:2011, *Solid biofuels — Determination of major elements — Al, Ca, Fe, Mg, P, K, Si, Na and Ti*

EN 15296, *Solid biofuels — Conversion of analytical results from one basis to another*

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

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

ASTM D516 - 07, *Standard Test Method for Sulphate Ion in Water*

DIN 38405-1:1985, *German standard methods for the examination of water, waste water and sludge; anions (group D); determination of chloride ions (D 1)*

DIN 51727, *Testing of solid fuels — Determination of chlorine content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14588:2010 and the following apply.

3.1
Reference Material
RM
material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

3.2
Certified Reference Material
CRM
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

3.3
NIST Standard Reference Material®
SRM
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

4 Principle

4.1 General

The determination of total sulfur and total chlorine content is performed in two steps (4.2 and 4.3) or by using automatic equipment (4.4).

4.2 Decomposition of the biofuel and transfer of acidic gaseous components into solution

- Combustion in an oxygen bomb and absorption of the acidic gas components in an absorption solution (method A);
- Digestion in closed vessels as described in EN 15290:2011, Part A (method B).

4.3 Determination of sulphate and chloride in the receiving solution

- Ion chromatography applying the principles of EN ISO 10304-1;
- ICP, applying the principles of EN ISO 11885 (determination as sulfur and chlorine);
- Other validated analytical methods.

NOTE A large number of applicable methods for the quantification of sulphate and chloride exist but detection limits and precision vary significantly.

4.4 Automatic equipment

Automatic equipment may be used when the method is validated with biomass reference samples of an adequate biomass type. If automatic equipment is used, sulfur and chlorine compounds may be detected as gaseous components (e.g. by infrared methods). Examples for automatic analysers are e.g. elemental analysers, AOX-analysers.

X-ray fluorescence may be used to determine sulfur and chlorine. Usually, the test sample prepared in accordance with FprEN 14780 is pressed with or without elevated temperature into a wafer and the concentration of relevant elements are measured directly in the solid biofuel sample.

If automatic equipment or X-ray fluorescence are used, the method shall be validated for the respective main origin based biomass group (see EN 14961-1) according to CEN Guide 13:2008, Clause 3 validation of alternative methods with one of the two approaches:

- full validation as applies to reference methods;

- relative validation in which a comparison is made to the reference method e.g. by participation in interlaboratory comparison tests.

NOTE Equipment validated only with e.g. straw reference materials is not automatically suitable for the determination of sulfur and chlorine in e.g. wood samples because of the usually significant lower concentrations of the elements in wood and/or the unknown influences of the different matrix.

5 Reagents

The reagents listed below relate to the digestion method specified in 8.1.1 (method A). Reagents for the digestion method B and the different detection methods according to 8.2 are specified in the corresponding standards.

5.1 General

All reagents shall be at least of analytical grade and suitable for their specific purpose. Particularly, they shall contain negligible amounts of chlorine and sulfur, i.e. amounts that do not contribute significantly to the determination.

5.2 Water

De-ionised water will normally fulfil the requirements of 5.1.

5.3 Oxygen

Pure with an assay of at least 99.5 % (V/V).

5.4 Combustion aid/enhancer

Various substances may be used e.g. benzoic acid, paraffin oil, acetobutyrate capsules, polyethylene bags.

5.5 Use of Certified Reference Materials (CRM or SRM)

Use certified reference materials, issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1570 spinach leaves, NBS 1571 orchard leaves, NBS 1573 tomato leaves and NBS 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials, may solve these problems. In that case CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

6 Apparatus

6.1 General

- Analytical balance, with a resolution of at least 0,1 mg;

- general laboratory equipment such as volumetric flasks and measuring cylinders.

6.2 Method A

6.2.1 Pellet press, capable of applying a force of 0,1 Nm equipped with a die to press a pellet with a diameter of about 13 mm.

6.2.2 Combustion bomb, suitable for the determination of sulfur and chlorine. The combustion bomb may be the same as used for the determination of the calorific value (see EN 14918).

The bomb shall not leak during the test and shall permit a quantitative recovery of the liquid. Its inner surface may be made of stainless steel or any other material that will not be affected by the combustion process or products.

Note that not all calorimeter bombs can be used because the principle of construction, the materials used for construction, or the surfaces in the bombs, may adsorb or react with the acidic gases formed during combustion or it may not be possible to clean the bomb completely.

6.3 Method B

Sample digestion vessels and an associated heating device, see EN 15290.

7 Preparation of the test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm prepared in accordance with FprEN 14780.

NOTE For some instrumental methods and/or solid biofuels it may be necessary to prepare a test sample with a lower nominal top size than 1 mm (e.g. 0,25 mm) in order to keep the stated precision and repeatability limits.

As the results are to be calculated on dry basis, the moisture content of the test sample shall be determined concurrently by the method described in EN 14774-3, using another portion of the test sample.

8 Procedure

8.1 Decomposition

8.1.1 Method A: Combustion in a closed bomb

Solid biofuel samples are usually combusted in a pellet form due to the low density and their combustion behaviour.

- Take a sample of approximately 1 g (unless the combustion bomb is designed for other sample amounts).
- Press the sample with a suitable force to produce a compact unbreakable pellet that is weighed to 0,1 mg. If the calorific value is determined simultaneously, the sample amount may eventually be adjusted according to the specification in EN 14918.
- Transfer the sample into a quartz glass or metal crucible.

The combustion may be carried out using a combustion aid:

a) Liquid combustion aid: after the mass of the sample pellet is determined, the auxiliary liquid material shall be added dropwise on the pellet placed in the crucible (allowing the liquid to be adsorbed); the added amount has to be determined exactly by weighing;

b) Combustion bag or capsule: the sample may be filled in powdered form into a combustion bag or capsule with precisely known weight. The sample mass is calculated by weighing the sample in the combustion bag or capsule and subtracting the mass of the bag or capsule.

c) Solid combustion aid: after the mass of the sample is determined, add an appropriate amount of the aid (e.g. benzoic acid) and determine the added amount of the aid exactly by weighing. Mix the sample and the aid carefully and prepare a pellet of the whole mix as described above. Ensure that the weight of the pellet equals the weight of the sample and the weight of the aid.

— Add 1 ml water into the bomb as receiving solution (also see EN 14918 if the calorific value is determined simultaneously). Alternatively, the water may be omitted or more water (up to 5 ml) may be used. If the content of chlorine or sulfur exceeds 2 % (m/m) alkaline solutions may be used to neutralize the acidic compounds produced. When ion chromatography is used for determination, the absorption solution may be the mobile phase e.g. a carbonate/bicarbonate solution. In all cases, the calibration of the method and the blank tests have to be done with the same amount and the same kind of receiving solution.

— Place the crucible in position and arrange the firing wire. Assemble the bomb and tighten the cover securely. Before ignition, the bomb is filled with 30 bar oxygen.

NOTE 1 If the chlorine content of the sample is very low, the cotton thread usually used for ignition may contribute significantly to the measured chlorine content. This can be avoided by using highly pure combustible crucibles without cotton threats.

NOTE 2 The bomb may be flushed with oxygen to reduce the nitrous oxide formation during the combustion process especially when the concentration of sulfur and chlorine is determined by ion chromatography (some peaks in the chromatogram may not be separated satisfactorily).

— After combustion, release the bomb pressure slowly before opening the bomb.

— Transfer the absorption solution to a volumetric flask (50 ml or 100 ml).

— Thoroughly rinse the bomb, the cover and the crucible including the combustion residues in the crucible with water and collect all the rinse water carefully. Transfer it into the volumetric flask and fill the flask to volume. While rinsing, take care that the ash that was formed after the combustion is collected in the flask as well.

NOTE 3 If the sample contains high concentrations of sulfur and/or chlorine (> 2 %), the combustion gas should be let through a gas washing bottle with a disk to assure that all acidic gas components are dissolved. The solution from this gas washing bottle can be combined with the bomb washings or may be analysed separately from the solution of the combustion bomb.

NOTE 4 If the sample contains high amounts of ash then chlorine and sulfur may be trapped in the combustion residues. In that case the combustion residue can be analysed for the chlorine and sulfur content. Alternatively, a low sample intake in combination with a combustion aid can be used.

NOTE 5 Special care is necessary when samples with low contents of chlorine and/or sulfur (e.g. virgin wood) are analysed after samples with high contents of these elements (e.g. herbaceous samples or waste samples). The most efficient way of cleaning the bomb is multiple combustions of pure benzoic acid; see 8.1.3, Blank test.

For some subsequent analytical methods, a chemical treatment of the solution is necessary before filling up to volume. Some analytical methods require a filtered solution.

The calorific value may be determined simultaneously. In this case, the requirements of EN 14918 shall be observed. The content of other halogens (fluoride, bromide, iodide) may be determined by a similar method, see EN 14582 [1].

8.1.2 Method B: Digestion in a closed vessel

The method for the digestion in closed vessels is described in EN 15290. For the determination of chlorine an amount of 0,8 ml H_2O_2 per 100 mg of sample shall be used for the digestion. This larger amount compared to EN 15290 is used to avoid losses of chlorine.

8.1.3 Blank test

Carry out a blank test, using the same procedures and methods as described in 8.1.1 (method A) or 8.1.2 (method B), respectively, using benzoic acid for method A. This assesses both, the contents of the elements in the reagents and any contamination from equipment and the laboratory atmosphere. This contribution shall not be quantitatively significant.

The measured blank value has to be subtracted from the sample value.

NOTE At high element level the blank should be less than 10 % of the sample content. For low element level (a content below 500 mg/kg in the sample), it is adequate that the contents of the elements in the blank solution are 30 % or less of the contents of the elements in the sample solution.

8.2 Detection methods

8.2.1 Ion chromatography

Ion chromatography is the recommended method for the detection of sulphate and chloride. The determination should be according to the principles of EN ISO 10304-1.

NOTE The solution obtained from the digestion may be filtered using a syringe equipped with a 0,45 µm pore size filter tip to avoid damage of the ion chromatograph.

8.2.2 Other detection methods

The following methods are standardized at international levels and can be used:

Table 1 — Other methods for the detection of sulfate and chloride

Method	Cl	S	References (examples)
ICP	X	X	EN ISO 11885
Photometric (colorimetric)	X		DIN 51727
Turbidimetric		X	ASTM D516 - 07
Coulometric	X		DIN 38405-1:1985 (method D1-3)
Potentiometric titration	X		DIN 38405-1:1985 (method D1-2)

8.3 Calibration of the apparatus

When the analytical system is evaluated for the first time, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of Standard Reference Material (SRM) and or CRM, control samples and create control charts. The calibration and quality control scheme shall be organised and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (Annex A) demonstrates what is achievable with commercial instruments that are used by experienced laboratories.

8.4 Analyses of the digests

Analyse test portions of the digests in accordance with the manufacturer's instructions.

9 Expression of results

9.1 General

The results shall be reported as the mean of duplicate determinations. The results shall be calculated on dry basis according to 9.2 and 9.3. The results may be calculated to other bases e.g. to as received basis according to EN 15296.

9.2 Total chlorine

The total content of chlorine in the sample on dry basis, $w_{\mathrm{Cl,d}}$, expressed in % m/m, is given by the equation:

$$w_{Cl,d} = \frac{(c - c_0) \times V}{m} \times 100 \times \frac{100}{(100 - M_{ad})} \tag{1}$$

where

- c is the concentration of chloride in the solution in mg/l;
- c_0 is the concentration of chloride in the solution of the blank experiment in mg/l;
- V is the volume of the solution in l;
- m is the mass of the test portion used in mg;
- M_{ad} is the moisture content in the analysis test sample in % m/m.

9.3 Total sulfur

The total content of sulfur in the sample on dry basis, $w_{\mathrm{S,d}}$, expressed in % m/m, is given by the equation:

$$w_{S,d} = \frac{(c - c_0) \times V}{m} \times 0{,}3338 \times 100 \times \frac{100}{(100 - M_{ad})} \tag{2}$$

where

- c is the concentration of sulphate in the solution in mg/l;
- c_0 is the concentration of sulphate in the solution of the blank experiment in mg/l;
- V is the volume of the solution in l;
- m is the mass of the test portion used in mg;
- 0,3338 is the stoichiometric ratio of the relative molar masses of sulfur and sulphate;
- M_{ad} is the moisture content in the analysis test sample in % m/m.

10 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European intercomparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. These two samples represent the extremity of the method. The wood chip sample represents samples with low contents of sulphur and chlorine and the olive residue samples with high amounts of sulfur and chlorine.

11 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of sample tested;

c) reference to this European Standard (EN 15289);

d) methods used for digestion and for determination;

e) results of the test including the basis in which they are expressed, as indicated in Clause 9;

f) any unusual features noted during the determination;

g) any operation not included in this European Standard, or regarded as optional.

Annex A
(informative)

Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, the Netherlands, Spain, Sweden and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips and exhausted olive residues produced in the EU-project BioNorm in 2008 in accordance with CEN/TS 14780. The sample “wood chips” was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample “exhausted olive residues” was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data is reported on dry basis.

The performance data according to ISO 5725-2 [5] are presented in Tables A.1 and A.2.

NOTE 1 See Table A.1 for definition of the symbols used in Tables A.1 and A.2.

NOTE 2 A guideline can be found in EN 15296 on how to use these validation parameters.

Table A.1 —Performance data for sulphur (S)

Sample	n	l	o	x	s_R	CV_R	s_r	CV_r
			%	% m/m	% m/m	%	% m/m	%
wood chips	20	90	5,2	0,009	0,003	34	0,001	12
exhausted olive residues	23	111	1,8	0,12	0,021	17	0,007	5,4

Definition symbols	
n	is the number of laboratories after outlier elimination
l	is the number of outlier free individual analytical values
o	is the percentage of outlying values from replicate determination
x	is the overall mean
s_R	is the reproducibility standard deviation
CV_R	is the coefficient of the variation of the reproducibility
s_r	is the repeatability standard deviation
CV_r	is the coefficient of the variation of the repeatability

Table A.2 — Performance data for chlorine (Cl)

Sample	n	l	o	x	s_R	CV_R	s_r	CV_r
			%	% m/m	% m/m	%	% m/m	%
wood chips	17	75	7,4	0,006	0,003	52	0,001	13
exhausted olive residues	16	75	0	0,20	0,02	8,0	0,01	2,8

Bibliography

[1] EN 14582, *Characterization of waste — Halogen and sulphur content — Oxygen combustion in closed systems and determination methods*

[2] EN 15105, *Solid biofuels — Determination of the water soluble chloride, sodium and potassium content*

[3] ISO 351, *Solid mineral fuels — Determination of total sulfur — High temperature combustion method*

[4] ISO 352, *Solid mineral fuels — Determination of chlorine — High temperature combustion method*

[5] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[6] ISO/TS 21748, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

[7] DIN 51724-1, *Testing of solid fuels — Determination of sulfur content — Part 1: Total sulfur*

[8] ASTM D3177, *Standard Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke*

[9] ASTM D4239-02a, *Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods*

[10] NIST definitions: http://ts.nist.gov/MeasurementServices/ReferenceMaterials/DEFINITIONS.cfm

[11] NIST Technical note 1297:1994, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, *http://www.nist.gov/physlab/pubs/tn1297/index.cfm*

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