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Trdna biogoriva - Določevanje celotnega žvepla in klora

Solid biofuels - Determination of total content of sulfur and chlorine

Feste Biobrennstoffe - Bestimmung des Gesamtgehaltes an Schwefel und Chlor

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

Ta slovenski standard je istoveten z: EN 15289:2011

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of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.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