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Trdna biogoriva - Določevanje vodotopnega klorida, natrija in kalija

Solid biofuels - Determination of the water soluble chloride, sodium and potassium content

Feste Biobrennstoffe - Bestimmung des wasserlöslichen Gehaltes an Chlorid, Natrium und Kalium

Biocombustibles solides - Méthodes de détermination de la teneur en chlorure, sodium et potassium solubles dans l'eau

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English Version

Solid biofuels - Determination of the water soluble chloride, sodium and potassium content

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Foreword

This document (EN 15105:2011) 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of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.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 15105:2005.

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

The elements chlorine, sodium and potassium are present in solid biofuels. They can contribute significantly to utilisation problems such as corrosion, fouling and slagging in furnaces. Also they affect the gaseous emissions from the thermal processes.

The chlorine content in solid biofuels is mainly present as water soluble inorganic salts such as sodium and potassium chlorides or other ion-exchangeable forms. Determination of the water soluble chloride content is thus an alternative and simple method to achieve information of the level of chlorine in solid biofuels. The content of water soluble chloride shall however not be mistaken for the total content of chlorine in the fuels.

In solid biofuels sodium and potassium can be present as both minerals and salts. The salts of these elements are extractable with water and are readily volatile during thermal conversion. By determination of the water soluble content of sodium and potassium an estimate of the aggressive content of the elements in relation to potential slagging and fouling problems can be achieved. For some biofuels, such as straw, experience has shown that the water soluble content of sodium and potassium correspond to the total content of the elements. The content of water soluble sodium and potassium shall not be mistaken for the total content of the elements.

1 Scope

This European Standard specifies a method for the determination of the water soluble chloride, sodium and potassium content in solid biofuels by extraction with water in a closed container and their following quantification by different analytical techniques.

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 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 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)*

ISO 9964-1, *Water quality — Determination of sodium and potassium — Part 1: Determination of sodium by atomic absorption spectrometry*

ISO 9964-2, *Water quality — Determination of sodium and potassium — Part 2: Determination of potassium by atomic absorption spectrometry*

ISO 9964-3, *Water quality — Determination of sodium and potassium — Part 3: Determination of sodium and potassium by flame emission spectrometry*

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
water soluble content of chloride, sodium and potassium
amount of the elements chloride, sodium and potassium which can be extracted with water using the extraction procedure specified in this European Standard

4 Principle

The fuel sample is heated with water in a closed container at 120 °C for 1 hour. The concentrations of chloride, sodium and potassium in the obtained water extract are determined by one of the following techniques:

— chloride: Ion-Chromatography (IC) or potentiometric titration with silver nitrate;

NOTE Be aware that when potentiometric titration with silver nitrate is used, any contents of water soluble bromide and iodide will be included in the determination.

— sodium and potassium: Flame Emission Spectroscopy (FES) or Flame Atomic Absorption Spectroscopy (FAAS) or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

5 Reagents

Water, containing negligible amounts of chloride, sodium and potassium i.e. amounts that do not contribute significant to the determinations. Deionised water will normally fulfil this requirement.

6 Apparatus

6.1 Heating oven or autoclave, capable of being maintained at a temperature of (120 ± 5) °C.

6.2 Vessel, made of fluoropolymer with a volume of about 100 ml and provided with a tight screw cap.

The vessel and the cap shall be capable of withstanding at least 125 °C at 232 kPa. If only the water soluble content of chloride is to be determined, an equivalent Pyrex glass vessel can be used.

6.3 Balance, with a resolution of at least 1 mg.

6.4 General laboratory equipment as volumetric flasks and measuring cylinders.

If sodium and potassium are to be determined, the use of glass equipments shall be avoided.

6.5 Membrane filtering apparatus, with membrane filters of mean pore size 0,45 µm.

7 Preparation of the test sample

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

If the results are to be calculated other than on an “as determined” 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 Extraction

a) Weigh, in an empty clean vessel (see 6.2), 1,0 g of the analysis sample to the nearest 1 mg.

b) Add 50,0 ml water, swirl the content and close the vessel tight.

c) Leave the closed vessel in a heating oven or an autoclave at 120 °C for 60 min.

d) Take the closed vessel out of the oven or the autoclave and let it cool to room temperature.

WARNING — Do not attempt to open the vessel before it is cool.

e) Transfer the content of the vessel to a 100 ml volumetric flask. Wash the inside of the vessel with small portions of water; add the washings to the volumetric flask and fill it to the 100 ml volume with water.

f) Filter a portion of the solution (see e)) through a membrane filter of pore size 0,45 µm, discarding the first portion of the filtrate. Alternatively the filtering can be carried out using a syringe equipped with a 0,45 µm pore size filter tip.

NOTE If only the water soluble content of chloride is to be determined, filtering may be omitted or a coarse folded filter paper may be used instead of the membrane filter.

8.2 Detection methods

8.2.1 General

Complete the determination by measuring the concentration of the elements in the prepared solution; for chloride by using one of the methods stated in 8.2.2 and for sodium and potassium by using one of the methods stated in 8.2.3.

8.2.2 Methods for the determination of chloride concentration

For the determination of the chloride concentration one of the following methods shall be used:

— Ion-chromatographic determination according to the principles of EN ISO 10304-1;

— Potentiometric titration with silver nitrate according to Std. Meth. 4500-Cl- D [7] or equivalent national standards e.g. [3], [4] or [6].

Other methods may be used, provided that it can be proved that the obtained results are comparable to results obtained by determinations using one of the above stated methods, within the performance characteristics of these methods.

8.2.3 Methods for the determination of sodium and potassium concentration

For the determination of the concentration of sodium and potassium one of the following methods shall be used:

— ICP-OES according to the principles of EN ISO 11885;

— FAAS according to the principles of ISO 9964-1 and ISO 9964-2;

— FES according to the principles of ISO 9964-3.

For the instrumental technique used, an initial control for eventual interferences shall be performed using a standard addition method and/or a dilution method.

Other methods may be used, provided that it can be proved that the obtained results are comparable to results obtained by determinations using one of the above stated methods, within the performance characteristics of these methods.

8.3 Blank test

Carry out a blank test, using the same procedure and methods as described in 8.1 and 8.2 but omitting the test portion. This assesses both the contents of the elements in the reagents and any contamination from equipments and in the laboratory atmosphere. This 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.