SLOVENSKI STANDARD
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Trdna biogoriva ï Metode za doloļ evanje vodotopnega klorida, natrija in kalija

Solid biofuels - Methods for determination of the water soluble content of chloride, sodium and potassium

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

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

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Foreword

This Technical Specification (CEN/TS 15105:2005) has been prepared by Technical Committee CEN/TC 335 “Solid Biofuels”, the secretariat of which is held by SIS.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

The elements chlorine, sodium and potassium are to a larger or a smaller extent 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 as e.g. 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 latter forms of the elements are extractable with water and are readily volatile. 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, as e.g. straw, it is an experience 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 Technical Specification describes a method for the determination of the water soluble content of chloride, sodium and potassium in solid biofuels by extraction with water in a closed container and their following quantification by different analytical techniques.

The method is applicable for all solid biofuels with water soluble contents more than 50 mg/kg for chloride and more than 10 mg/kg for sodium and potassium.

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/TS 14588, *Solid biofuels – Terminology, Definitions and Descriptions.*

CEN/TS 14774-3, *Solid biofuels – Methods for the determination of moisture content – Oven dry method - Part 3: Moisture in general analysis sample.*

prCEN/TS 14780, *Solid biofuels – Methods for sample preparation.*

prCEN/TS 15296, *Solid biofuels – Calculation of analyses to different bases.*

EN ISO 10304-1, *Water quality – Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions – Part 1: Method for water with low contamination (ISO 10304-1:1992).*

EN ISO 11885, *Water quality – Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy (ISO 11885:1996).*

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 Technical Specification, the terms and definitions given in CEN/TS 14588 and the following apply.

3.1
water soluble content of chloride, sodium and potassium
amount of the element which can be extracted with water using the extraction procedure specified in this Technical specification

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 Emissions Spectroscopy (ICP-OES).

5 Reagents

5.1 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 controlled at a temperature within the range 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 to withstand at least 125 °C (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 prCEN/TS 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 CEN/TS 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 minutes.

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 Completion

Complete the determination by measuring the concentration of the elements in the prepared solution; for chloride by using one of the methods stated in 8.2.1 and for sodium and potassium by using one of the methods stated in 8.2.2.

8.2.1 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 EN ISO 10304-1;

— Potentiometric titration with silver nitrate according to Std. Meth. 4500-Cl^- D [1] or equivalent national standards e.g. [2], [3] or [4].

Other methods may be used, provided that it can be proved that equivalent results are obtained, within the overall precision stated in Clause 10, compared to determinations carried out using one of the above stated methods.

8.2.2 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 EN ISO 11885;

— FAAS according to ISO 9964-1 and ISO 9964-2;

— FES according to 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 equivalent results are obtained, within the overall precision stated in Clause 10, compared to determinations carried out using one of the above stated 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.