SLOVENSKI STANDARD
SIST-TS CEN/TS 15411:2007

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Trdno alternativno gorivo - Metode za določ evanje elementov v sledovih (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V in Zn)

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, Tl, V and Zn)

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Spurenelementen (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V and Zn)

Combustibles solides de récupération - Méthodes pour la détermination de la teneur en éléments traces (As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mo, Mn, Ni, Pb, Sb, Se, Tl, V et Zn)

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English Version

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, Tl, V and Zn)

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The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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Foreword

This document (CEN/TS 15411:2006) has been prepared by Technical Committee CEN/TC 343 “Solid Recovered Fuels”, the secretariat of which is held by SFS.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN 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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

Accurate determination of trace element content in solid recovered fuels is important for environmental and technical reasons both in the production and combustion stage. After digestion of the solid recovered fuels using different methods, a number of analytical techniques can be used for the quantification of the trace element content. They include Inductively Coupled Plasma with optical or mass detection, graphite furnace Atomic Absorption Spectrometry and, when available, dedicated specific method (e.g. for mercury).

1 Scope

This Technical Specification specifies three methods of digestion for solid recovered fuels:

a) microwave assisted digestion with hydrofluoric, nitric and hydrochloric acid mixture;

b) hot water bath digestion with hydrofluoric, nitric and hydrochloric acid mixture, after ashing of the SRFs sample;

c) oven digestion with nitric, perchloric and hydrofluoric acid mixture.

Instrumental determination of As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Sb, Se, Tl, V, Zn is performed by Inductively Coupled Plasma with optical or mass detection or graphite furnace Atomic Absorption Spectrometry. Hg can be analysed only after the microwave assisted procedure or, alternatively, by a direct analysis method.

The effectiveness of the digestion can be verified by qualitative X-ray fluorescence (XRF) analysis on the remaining residue. If necessary an alternative digestion method (among those proposed) is used.

Method a) is recommended for general use, but the amount of the test portion can be very low in case of high concentration of organic matter.

Method b) is recommended for SRFs with high organic matter concentration that can be difficult to digest with the other methods. This method is not suitable for mercury.

Method c) is recommended for SRFs samples for which the other methods leave a significant insoluble residue.

2 Normative references

The following referenced documents are indispensable for the application of this Technical Specification. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13656, *Characterization of waste — Microwave assisted digestion with hydrofluoric (HF), nitric (HNO_3) and hydrochloric (HCl) acid mixture for subsequent determination of elements*

CEN/TS 15357:2006, *Solid recovered fuels — Terminology, definitions and descriptions*

prCEN/TS 15403, *Solid recovered fuels — Methods for the determination of the ash content*

CEN/TS 15413, *Solid recovered fuels — Methods for the preparation of the test sample from the laboratory sample*

prCEN/TS 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)*

3 Terms and definitions

For the purposes of this Technical Specification, the terms and definitions given in CEN/TS 15357:2006 and the following apply.

3.1
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacted with a reagent mixture

3.2
microwave unit
whole microwave digestion system (oven and associated equipment)

4 Safety remarks

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

In addition the following information is given:

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

— most of reagents used within this Technical Specification are strongly corrosive and toxic. Safety precautions are absolutely necessary due to strong corrosive reagents, high temperature and high pressure;

— all procedures have to be performed in a hood or in closed force-ventilated equipment. By the use of strong oxidising reagents the formation of explosive organic intermediates is possible especially when dealing with samples with a high organic content. Do not open pressurised vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

5 Principle

The test portion is digested using one of the proposed methods with a suitable acid mixture. The digested sample is then analysed by the most appropriate spectrometric technique, such as atomic absorption or emission spectroscopy.

6 Apparatus

6.1 Microwave unit

Intended for laboratory use and preferably equipped with temperature control.

6.2 Resistance heating oven

A resistance heated oven or heating block that can be used at a temperature of at least 220 °C and an accuracy of ± 10 °C.

6.3 Digestion vessels

The vessels used in the microwave unit shall be equipped with a pressure relieve valve or another technical equipment which avoids the bursting of the vessels at suddenly occurring excess pressure. The material of the vessels has to be inert to the acids used for digestion. The digestion vessel shall withstand the pressure of at least 8 bar. If the amount of organic carbon exceeds 100 mg it has to be ensured that the digestion vessel is capable of withstanding higher pressures.

6.4 Inductively coupled plasma

Normal commercial instrumentation with optical or mass spectrometric detector (ICP-OES, ICP-MS).

6.5 Atomic absorption spectrophotometer

Normal commercial instrumentation, equipped with graphite furnace or hydride generation systems or cold vapour (GF-AAS, HG-AAS, CV-AAS).

6.6 X-ray fluorescence spectrometer

Energy or wavelength dispersion system suitable for qualitative analysis of the elements listed in this Technical Specification (with the exception of beryllium).

6.7 Balances

Analytical balance resolution ± 0,1 mg.

6.8 General equipment

General laboratory equipment, including volumetric graduated flasks and pipettes of adequate size.

Filter equipment of adequate chemical resistance and purity or centrifuge.

The use of glass ware shall be excluded when free hydrofluoric acid is present.

The glassware used in the digestion procedure should be carefully pre-cleaned with, for example, 10 % nitric acid solution.

7 Reagents

All reagents shall be at least of analytical grade and suitable for their specific purposes.

Other specific reagents are listed and described in the reference methods for digestion or instrumental determination listed in Clause 2.

NOTE Acids used in the preparation of standards and for sample processing are of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Nitric acid at less than 2 % (v/v) is recommended for ICP-MS to minimize damage to the interface and to minimize isobaric molecular-ion interferences with the analytes. Many more molecular-ion interferences are observed when hydrochloric and sulfuric acids are used.

7.1 Water of grade 1 as specified by EN ISO 3696:1995.

7.2 Nitric acid (HNO_3), 65 % (w/w), ρ 1,40 g/ml.

7.3 Hydrofluoric acid (HF), 40 % (w/w), ρ 1,14 g/ml.

7.4 Perchloric acid ($HClO_4$), 70 % (w/w), ρ 1,62 g/ml.

7.5 Hydrochloric acid (HCl), 65 % (w/w), ρ 1,40 g/ml.