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Solid recovered fuels - Method for the determination of the content of major elements (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)

Combustibles solides de récupération - Méthodes pour la détermination de la teneur en éléments majeurs (Al, Ca, Fe, K, Mg, Na, P, Si et Ti)

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Hauptelementen (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)

This Technical Specification (CEN/TS) was approved by CEN on 25 March 2006 for provisional application.

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Contents

Page

Foreword 3
Introduction 4
1 Scope 5
2 Normative references 5
3 Terms and definitions 6
4 Safety remarks 6
5 Principle 7
6 Apparatus 7
7 Reagents 8
8 Procedure 8
9 Digestion procedure 9
10 Analysis of the digestion solutions 9
11 Expression of results 10
12 Quality control 11
13 Performance characteristics 11
14 Test report 11
Annex A (normative) Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF 12
Bibliography 14

Foreword

This document (CEN/TS 15410:2006) 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. The determination of major elements such as Al, Ca, Fe, Mg, P, K, Si, Na and Ti can be helpful to predict the melting behaviour and slagging of the ash. 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, Flame Atomic Spectroscopy, Graphite Furnace Atomic Absorption Spectrometry and X-ray fluorescence spectrometry. X-ray fluorescence allows the simultaneous determination of these elements after ashing of SRF. Direct analysis of the SRF material is not possible by XRF due to the sample inhomogeneity and because suitable certified reference materials for calibration are not available.

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 of 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 Si, Al, K, Na, Ca, Mg, Fe, P, and Ti is performed by Inductively Coupled Plasma Spectrometry with optical detection or other suitable spectroscopic techniques such as Flame Atomic Spectroscopy.

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) shall be used.

XRF can be used for the analysis of Si, Al, K, Na, Ca, Mg, Fe, P, Ti, after ashing (550 °C) of the sample: other elements can be analysed by XRF providing that the concentration levels are above the instrumental detection limits of the XRF instrumentation and after proper preliminary testing.

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.

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

All the listed methods are suitable for the determination of Si, provided that closed containers are used for sample dissolution. XRF is highly recommended for Si, P and Ti analysis.

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*

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*

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

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

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

EN ISO 12020, *Water quality — Determination of aluminium — Atomic absorption spectrometric methods (ISO 12020:1997)*

EN ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace (ISO 15586:2003)*

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 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's manual;

— most of the 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;

— the X-ray fluorescence spectrometers on the market are generally approved fully protected apparatus. This means that the user is not subjected to any radiation when operating the apparatus. All the apparatus are subject to specific official approval and acceptance conditions;

— the person responsible for managing or supervising the operation of X-ray equipment shall provide evidence of his knowledge of radiation protection according to national regulations.