

SLOVENSKI STANDARD SIST-TS CEN/TS 15410:2007

01-marec-2007

HfXbc'UhYfbUhjj bc'[cf]j c'!'A YhcXU'nU'Xc'c Yj Ub'Y'['Uj b]\ 'Y Ya Ybhcj 'f5'ž7 Už: Yž?žA[žBUžDžG|žHk

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

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

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

https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-

Ta slovenski standard je istoveten z: CEN/TS 15410:2006

ICS:

75.160.10 Trda goriva Solid fuels

SIST-TS CEN/TS 15410:2007 en

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST-TS CEN/TS 15410:2007 https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-b75e92e1335c/sist-ts-cen-ts-15410-2007

TECHNICAL SPECIFICATION

CEN/TS 15410

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

September 2006

ICS 75.160.10

English Version

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.

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.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

CEN members are the national standards bodies of 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.

SIST-TS CEN/TS 15410:2007 https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-b75e92e1335c/sist-ts-cen-ts-15410-2007



EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

CEN/TS 15410:2006 (E)

| COI | ntents | Page |
|--------------|---|------|
| Fore | word | 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 iTeh STANDARD PREVIEW | 10 |
| 12 | Quality control(standards.iteh.ai) | 11 |
| 13 | Performance characteristics | 11 |
| 14 | Test report SIST-TS CEN/TS 15410:2007 https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7- | |
| Anne | https://standards.iteh.ai/catalog/standards/sist/11acbbct-70te-4c84-83c7- ex A (normative) Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF | |
| Bibli | ography | 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.

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST-TS CEN/TS 15410:2007 https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-b75e92e1335c/sist-ts-cen-ts-15410-2007

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 AI, 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.

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST-TS CEN/TS 15410:2007 https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-b75e92e1335c/sist-ts-cen-ts-15410-2007

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.

SIST-TS CEN/TS 15410:2007

All the listed methods are suitable for the determination of Sipprovided that closed containers are used for sample dissolution. XRF is highly recommended for Sipp 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 (HNO₃) and hydrochloric (HCI) 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)

CEN/TS 15410:2006 (E)

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 STANDARD PREVIEW

3.2

microwave unit

(standards.iteh.ai)

whole microwave digestion system (oven and associated equipment)

SIST-TS CEN/TS 15410:2007

https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-

b75e92e1335c/sist-ts-cen-ts-15410-2007

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.

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 inductively coupled plasma atomic emission spectroscopy.

For XRF analysis the sample is ashed at 550 °C and the ash is homogenised in a ball mill to obtain a uniform size dimension of the particles. The ash is then pressed in the form of pellet or fused with tetraborate. Both techniques are suitable for the analysis by XRF. Coal ash and other ashes of various origins can be used for instrument calibration.

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 $^{\circ}$ C and an accuracy of \pm 10 $^{\circ}$ 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 SIST-TS CEN/TS 15410:2007

https://standards.iteh.ai/catalog/standards/sist/11acbbcf-70fe-4c84-83c7-

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

6.5 X-ray fluorescence spectrometer

Energy or wavelength dispersion system suitable for quantitative/qualitative analysis of the elements listed in this Technical Specification.

6.6 Atomic Absorption Spectrometer

Normal commercial instrumentation with air-acetylene burner or with graphite tube atomizer and background correction system and with hollow cathode lamps.

6.7 Press

6.8 Balances

Analytical balance with a resolution of \pm 0,1 mg.

6.9 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 glassware shall be excluded when free hydrofluoric acid is present.