# SLOVENSKI STANDARD
# oSIST prEN 15410:2010

**01-marec-2010**

---

**Trdna alternativna goriva - Metode za določevanje glavnih elementov (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)**

Solid recovered fuels - Methods 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 Hauptbestandteilen (Al, Ca, Fe, K, Mg, Na, P, Si, Ti)

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



**Ta slovenski standard je istoveten z: prEN 15410**



---

**ICS:**

75.160.10 Trda goriva Solid fuels

**oSIST prEN 15410:2010 en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

# DRAFT
# prEN 15410

January 2010

ICS 75.160.10

Will supersede CEN/TS 15410:2006

English Version

# Solid recovered fuels - Methods 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 élémentes majeurs (Al, Ca, Fe, K, Mg, Na, P, Si et Ti)

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

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 343.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, 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.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

**Warning** : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.



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

**Management Centre: Avenue Marnix 17, B-1000 Brussels**



Ref. No. prEN 15410:2010: E

# Contents

Page

## Foreword

This document (prEN 15410:2010) has been prepared by Technical Committee CEN/TC 343 “Solid Recovered Fuels”, the secretariat of which is held by SFS.

This document is currently submitted to the CEN Enquiry.

This document will supersede CEN/TS 15410:2006.

## 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 European Standard 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 document. 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*

prEN 15357, *Solid recovered fuels — Terminology, definitions and descriptions*

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

prEN 15414-3, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

prEN 15403, *Solid recovered fuels — 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 12020:2000)*



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 document, the terms and definitions given in prEN 15357 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 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 European Standard 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 °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 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 European Standard.

### 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 ± 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 glass ware shall be excluded when free hydrofluoric acid is present.



The glassware used in the digestion procedure should be accurately pre-cleaned with 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 should be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Nitric acid at less than 2 % (v/v) is required 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.