

## SLOVENSKI STANDARD SIST-TS CEN/TS 15408:2007

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Solid recovered fuels - Methods for the determination of sulphur (S), chlorine (CI), fluorine (F) and bromine (Br) content

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Schwefel (S), Chlor (Cl), Fluor (F) und Brom (Br) ANDARD PREVIEW

Combustibles solides de récuperation - Méthodes pour la détermination de la teneur en soufre (S), en chlore (CI), en fluor (F), et en brome (Br)

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Ta slovenski standard je istoveten z: CEN/TS 15408:2006

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75.160.10 Trda goriva

Solid fuels

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en

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# TECHNICAL SPECIFICATION SPÉCIFICATION TECHNIQUE TECHNISCHE SPEZIFIKATION

### **CEN/TS 15408**

September 2006

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**English Version** 

# Solid recovered fuels - Methods for the determination of sulphur (S), chlorine (Cl), fluorine (F) and bromine (Br) content

Combustibles solides de récupération - Méthodes pour la détermination de la teneur en soufre (S), en chlore (Cl), en fluor (F), et en brome (Br)

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an Schwefel (S), Chlor (CI), Fluor (F) und Brom (Br)

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.

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

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### Contents

Foreword	
Introduction4	
1	Scope
2	Normative references5
3	Terms and definitions5
4	Safety remarks
5	Principle
6	Apparatus6
7	Reagents7
8	Interferences and sources of error8
9	Procedure
10	Calculation and evaluation9
11	Quality control iTeh STANDARD PREVIEW
12	Performance characteristics
13	Test report10
Annex A (normative) Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF	
	of SRF
56115e086ce0/sist-ts-cen-ts-15408-2007 Bibliography	

### Foreword

This document (CEN/TS 15408:2006) 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.

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### Introduction

Determination of total sulphur, chlorine, fluorine and bromine content of solid recovered fuels is necessary for environmental and technical reasons both in the production and combustion stage.

During the combustion process they are usually converted to sulphur oxides and halides. These reaction products contribute significantly to corrosion and environmentally harmful emissions.

This method consists of an oxygen combustion procedure followed by trapping of sulphur, chloride, fluoride and bromide in an absorbing solution and subsequent determination by different techniques.

Alternatively, direct automatic techniques can be used for S and Cl determination. Other methods could also be used provided that it is demonstrated that they give the same results.

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### 1 Scope

This Technical Specification describes the determination of S, Cl, F and Br in solid recovered fuels of various origin and composition after combustion in oxygen atmosphere. S and Cl can be alternatively determined by direct automatic analysis (see Bibliography for examples of available methods). Other methods could also be used provided that it is demonstrated that they give the same results.

This method is applicable for concentrations over 0,025 g/kg, depending on the element and on the determination technique.

Insoluble halides and sulphate present in the original sample or produced during the combustion step are not completely determined by these methods.

This Technical Specification provides recommendations concerning standardised methods for determination of halides and sulphate in the solution obtained after combustion.

### 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.

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 - S Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample log/standards/sist/cada57be-ce87-446f-b450-56115e086ce0/sist-ts-cen-ts-15408-2007

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

EN ISO 10304-1:1995, 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 10304-2:1996, Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water (ISO 10304-2:1995)

EN ISO 17294-2, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2:2003)

ISO 9297, Water quality — Determination of chloride — Silver nitrate titration with chromate indicator (Mohr's method)

ISO 10359-1, Water quality — Determination of fluoride — Part 1: Electrochemical probe method for potable and lightly polluted water

### 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

### halogen content

sum of halogens contained as organic and inorganic compounds in the solid recovered fuels which can be converted to halides (fluoride, chloride, bromide, iodide) by combustion and then absorbed or dissolved in aqueous solution

NOTE Be aware that the above definition is valid for this empirical Technical Specification only and do not comply with scientific definitions of halogen content.

### 3.2

### oxygen combustion

combustion of material in oxygen atmosphere

### 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 oxygen combustion apparatus, following the operating instructions described in the manufacturer manual;
- precautions shall be taken by the operator for reactive gas (oxygen) at high temperature and high pressure.

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### 5 Principle

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The determination of S, CI, Fland/Briscarried out in two steps of by using automatic equipment: 56115e086ce0/sist-ts-cen-ts-15408-2007

- the sample is oxidized by combustion in a bomb containing oxygen under pressure. Halogenated and sulphur compounds are converted respectively to fluoride, chloride, bromide and sulfate which are absorbed and/or dissolved in an absorption solution (water or KOH 0,2 M solution);
- analysis of Cl, F and S and Br by ion chromatography or other suitable technique, reported in the reference documents listed in Clause 2. Br is preferably determined by ICP-MS since several oxidation states of Br occur after oxygen combustion.

### 6 Apparatus

Ordinary laboratory apparatus, and:

### 6.1 Oxygen combustor

Equipped with a combustion bomb made of stainless steel or any other material that will not be affected by the combustion process or products (the materials used may adsorb or react with acid gases formed during combustion or it may be not possible to clean the bomb completely between combustions). The bomb is equipped with oxygen inlet and safety valve and electrical contacts for spark generation. Many commercially available systems can be used. The combustion bomb may be the same as used for the determination of the calorific value.

Carefully check the characteristics of the combustion bomb, in order to be sure that it is suitable for the processing of materials with significant chlorine content (chlorine resistant combustion bomb are commercially available).

The combustion apparatus is equipped with automatic ignition system and oxygen gas supply.

### 6.2 Balances

- Analytical balance resolution ± 0,1 mg.
- Balance resolution  $\pm$  0,1 g.

### 6.3 Ion chromatograph

An ion chromatograph with suitable anion separator column, pre-column, background suppressor and conductivity cell.

### 6.4 Apparatus for titrimetry

Any suitable apparatus can be used, with colorimetric or potentiometric final point determination.

#### 6.5 Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Any suitable apparatus with normal resolution and pneumatic sample introduction system.

### 6.6 Automatic analyzer

Commercial instruments for S and Cl determination.

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### 7 Reagents

All reagents shall be at least of analytical grade and suitable for their specific purposes. Particularly, they shall be free of sulphur and halogens. <u>SIST-TS CEN/TS 15408:2007</u>

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7.1 Water of grade 1 as specified by ENcISO 3696:19955408-2007

### 7.2 Oxygen

Free of combustion material, minimum 99,99 purity.

### 7.3 Nitrogen

Chromatographic grade for the ion chromatograph.

#### 7.4 Eluent for ion chromatography

Carbonate/hydrogen carbonate mixed solution is suitable as eluent for ion chromatographic separation. Other eluents can be used, following the working instruction with the particular column used.

#### 7.5 Absorbing solution

Water is appropriate for most application. If the content of chlorine is >1 % or if bromine shall be determined, alkaline KOH 0,2 M solution is more efficient for trapping the gases. As a preliminary check, XRF analysis can be used to check for the presence of Br or high chlorine content.

#### 7.6 Stock standard solutions

1 000 mg/l chlorine, fluorine, bromine and sulphate commercially available standard solutions are used to prepared working and calibration solution by properly dilution.