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Trdno alternativno gorivo - Metode za določ evanje aluminija v kovinski obliki

Solid recovered fuels - Methods for the determination of metallic aluminium

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an metallischem Aluminium

Combustibles solides de récupération - Méthodes pour la détermination de l'aluminium métal

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Combustibles solides de récupération - Méthodes pour la détermination de l'aluminium métal

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an metallischem Aluminium

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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 15412:2006) 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

The metallic aluminium in solid recovered fuels is very problematic in combustion processes. Aluminium can form deposit on heat transfer surfaces and superheaters. For these reasons a method for the determination of total metallic aluminium is necessary. Other methods with low melting point such as tin, lead and zinc may cause similar problems but their content in solid recovered fuels is usually very low and then their effect is not significant.

1 Scope

This Technical Specification specifies two different methods for the determination of metallic aluminium in solid recovered fuels:

— method a: dissolution of metallic aluminium and analysis by Inductively Coupled Plasma Optic Emission Spectrometry (ICP-OES) or by Flame Atomic Absorption Spectrometry (FAAS);

— method b: Differential Thermal Analysis (DTA) on the solid SRF.

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 — 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 the 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)*

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
metallic aluminium
aluminium that could be extract from SRF by using a 0,75 mol/l NaOH solution, after leaching with 0,14 mol/l HNO_3 solution. This includes the metallic aluminium and some chemical forms of aluminium non-soluble in nitric acid but easily soluble in alkaline media

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:

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

— the reaction of metallic aluminium (and other metals such as zinc, lead and tin) with NaOH solution generates gaseous hydrogen that can form explosive mixtures in the air;

— 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

5.1 Method A

The test portion of 1 mm maximum particle size is leached with 0,14 mol/l nitric acid solution and shaken. After that the mixture is filtered. The elemental aluminium is digested by heating the sample with alkali. After that the mixture is filtered and then the aluminium content is determined by ICP-OES or FAAS.

5.2 Method B

The test portion of 1 mm maximum particle size is ashed and then introduced with the proper program in the DTA analyser and the DTA curve is recorded.

6 Apparatus

6.1 Method A

6.1.1 Balances

Analytical balance resolution ± 0,1 mg.

6.1.2 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 NaOH is used.

6.1.3 Shaking table

6.1.4 Hotplate

Resistance heated, with temperature regulation up to 120 °C.

6.1.5 Inductively coupled plasma (ICP-OES)

Normal commercial instrumentation.

6.1.6 Flame atomic absorption spectrometer (FAAS)

Normal commercial instrumentation.

6.2 Method B

6.2.1 Differential thermal analyser (DTA)

Commercial differential thermal analyser or differential thermal analyser/thermogravimetric analyser (DTA/TGA)

6.2.2 Platinum pans

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.

7.1 Method A

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

7.1.2 Sodium hydroxide (NaOH), 0,75 mol/l.

7.1.3 Nitric acid (HNO_3), 0,14 mol/l.

7.1.4 Concentrated nitric acid ((HNO_3), 14 mol/l.

7.1.5 Aluminium standard solution, 1 000 mg/l.

Commercial available standard solution for spectroscopy.

7.1.6 Calibration solutions

The calibration solutions are made by mixing 10 ml of 0,75 mol/l NaOH (7.1.2) with 3 ml concentrated HNO_3 (7.1.3) in 100 ml graduated flask. A suitable amount of aluminium standard is then added and the solution is diluted to 100 ml with water.

7.2 Method B

7.2.1 Aluminium powder, purity 96,7 % or higher.

7.2.2 Sand

Purified, high quartz content sand.

7.2.3 Aluminium calibration mixture

Appropriate solid mixtures prepared by mixing sand (7.2.2) and metallic aluminium (7.2.1), according to the expected content of metallic aluminium in SRF samples.

8 Preparation of the test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm or less, which shall be prepared in accordance with CEN/TS 15413 and for the method B prepared in accordance with prCEN/TS 15403.

For the method A the sample can be either dry or air-dried and for the method B the sample can be either dry or air-dried fuel ash, prepared according to prCEN/TS 15403.