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Trdna alternativna goriva - Metode za določevanje 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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English Version

Solid recovered fuels - Methods for the determination of metallic aluminium

Combustibles solides de récupération - Méthode de détermination de l'aluminium total

Feste Sekundärbrennstoffe - Verfahren zur Bestimmung des Gehaltes an metallischem Aluminium

This Technical Specification (CEN/TS) was approved by CEN on 27 March 2010 for provisional application.

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Foreword

This document (CEN/TS 15412:2010) has been prepared by Technical Committee CEN/TC 343 “Solid Recovered Fuels”, the secretariat of which is held by SFS.

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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 metals 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 document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

prEN 15357:2009, *Solid recovered fuels — Terminology, definitions and descriptions*

prEN 15403, *Solid recovered fuels —Determination of the ash content*

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*

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

EN ISO 11885:2009, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

EN ISO 12020:2000, *Water quality — Determination of aluminium — Atomic absorption spectrometric methods (ISO 12020:1997)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN 15357:2009 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

NOTE 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 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

6.1.3 Hotplate

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

6.1.4 Inductively coupled plasma (ICP-OES)

Normal commercial instrumentation.

6.1.5 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

7.1 General

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.

7.2 Method A

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

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

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

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

7.2.5 Aluminium standard solution, 1 000 mg/l.

Commercial available standard solution for spectroscopy.

7.2.6 Calibration solutions

The calibration solutions are made by mixing 10 ml of 0,75 mol/l NaOH (7.2.2) with 3 ml concentrated HNO_3 (7.2.3) in 100 ml graduated flask. A suitable amount of aluminium standard is then added and the solution is diluted to 100 ml with water.

7.3 Method B

7.3.1 **Aluminium powder**, purity 96,7 % or higher.

7.3.2 Sand

Purified, high quartz content sand.

7.3.3 Aluminium calibration mixture

Appropriate solid mixtures prepared by mixing sand (7.3.2) and metallic aluminium (7.3.1), 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 prEN 15413 and for the method B prepared in accordance with prEN 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 prEN 15403.

NOTE 1 In some cases Calcite (calcium carbonate) in the sample can effect on results interpretation in method B. In such cases ashing is recommended at higher temperatures, e.g. 850 °C.

NOTE 2 Special care should be taken to avoid segregation of aluminium particles when preparing test portion (method A).

9 Procedure

9.1 Sample conservation and pre-treatment

The laboratory samples shall be stored according to guidelines defined in Annex A.

Furthermore any possible source of contamination during the laboratory sample preparation (e.g. grinding with metallic apparatus, mainly aluminium or aluminium alloy) shall be avoided or reduced as much as possible.

The laboratory sample should be stored and delivered in sealed high-density plastic containers.

9.2 Sample preparation

The test portion shall be prepared from the laboratory sample according to prEN 15413.

In addition, for the purposes of this method, the target size should be 1 mm or below.

For method B the test portion is obtained by ashing an appropriate amount of the original sample.

Depending on the used method, the amount of test portion ranges between 50 mg (method B) to 2 g (method A).

Whereas the determination is carried out on dry basis, the moisture content shall be determined according to prEN 15414-3.