DD CEN/TS 15412:2010

BSI Standards Publication

Solid recovered fuels — Methods for the determination of metallic aluminium

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It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for its conversion to an international standard. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards.*

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The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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.

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 15412:2010) 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.

9.3 Method A

9.3.1 General

Accurately weight about 2 g of the test sample in 250 ml Erlenmeyer flask. Carry out two blank tests, using the same procedure but omitting the test portion.

NOTE 1 A glass fibre filter, 10 cm, GF 6 (retention of particles larger than 1 µm) with Büchner funnel with suction may be used when necessary.

NOTE 2 Some turbidity may remain after filtration and should not interfere with the determination.

9.3.2 Leaching of lightly soluble aluminium compounds

Add 100 ml of 0,14 mol/l nitric acid (7.2.3) put the Erlenmeyer flask on the shaking table. Shake the sample for one hour. Filter the sample and rinse 3 times the Erlenmeyer flask and the filter with 20 ml aliquots of water (7.2.1).

9.3.3 Dissolution of metallic aluminium

Transfer the filter with the sample into a narrow PTFE container. Add 100 ml of 0,75 mol/l sodium hydroxide (7.2.2), heat to boiling and let the solution gently boil for 1,5 h. Remove the sample from the heating plate and add 100 ml water and let the solution cool down. The solution is filtered and the filtrate is transferred into plastic 500 ml graduated flask. Rinse the PTFE vessel and the filter with 3 aliquots of 20 ml each of water (7.2.1). Add 15 ml concentrated nitric acid (7.2.4) into the graduated flask and dilute with water to 500 ml.

9.4 Method B

The sample is ashed according to the general principle of prEN 15403. Weight about 50 mg of ash directly in the platinum pan. The thermogram is recorded.

9.5 Determination of metallic aluminium

9.5.1 Method A

The content of aluminium in the final solution from method A shall be determined by FAAS or ICP-OES according to the general principles of EN ISO 12020 or EN ISO 11885.

9.5.2 Method B

The metallic aluminium content shall determined from the DTA curve by integrating the melting peak of aluminium at 660 °C. The DTA peak is downward. The reaction is endothermic. The calibration shall be made by similar aluminium standards made in sand matrix.

10 Calculation and evaluation

10.1 General

Results are referred to the dry material. Calculation shall take into consideration any eventual intermediate dilution of the solution.

10.2 Method A

The content of aluminium on dry basis, w, is calculated according to following equation. The concentration is expressed as Al % w/w in the weighted sample.

$$w = \frac{(c - c_0) \times V}{m} \times 100$$

where

c is the concentration of aluminiumin the final solution (mg/l);

c_0 is the concentration of aluminium in the solution of the blank test (mg/l);

m is a mass of the sample of the test portion used (mg);

V is the volume of the final solution (l).

10.3 Method B

The aluminium content of the sample is expressed as percentage on a dry mass basis. The results are calculated from the calibration curve.

11 Quality control

To detect possible contaminations from vessels and/or reagents, blank tests shall be carried out by the same sample preparation procedure, using the same quantities of reagents.

If available, the use of standard reference materials is recommended. The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM) if existing. Calculate the analytical result for the CRM/RM, and compare it with the reference or certified value.

Recovery rate of the test method could be checked using spiked samples, e.g. virgin woody material mixed with known amount of metallic aluminium (of different film thicknesses).

12 Performance characteristics

Data of performance of the methods await for future validation.

13 Test report

The test report shall contain at least the following information:

a) name, address and location of any laboratory involved in the analysis;

b) description and identification of the laboratory sample;

c) date of receipt of laboratory sample and date(s) of performance of test;

d) a reference to this Technical Specification, i.e. CEN/TS 15412;

e) reference to the analytical standard used for the determination for each element;

f) analytical results, referring to Clause 10;

g) any details not specified in this Technical Specification or which are optional, and any other factors which may have affected the results;

h) unique identification of report (such as serial number) and of each page and total number of pages of the report.

The laboratory should keep a trace of any analytical steps and intermediate results (thermograms, raw data and calculation details) that should be kept available in case of specific requirements.

Annex A
(normative)

Guidelines - Characteristics of the laboratory sample for chemical analysis of SRF

The following requirements apply when preparing the laboratory sample for the chemical characterisation of SRF samples according to this Technical Specification.

NOTE Equivalent requirements apply in all chemical test method specifications for SRF, i.e. prEN 15407, prEN 15410, prEN 15411, prEN 15412 and prEN 15413.

A maximum amount of laboratory sample of 10 kg and maximum particle size of 1 cm is established on the basis of number and type of parameters to be determined, sample representativity and practical reasons for handling samples. In Table A.1 the requirements are summarised both for single or grouped chemical parameters.

Table A.1 – Requirements for the laboratory sample for the analysis of SRF

Parameter (single or group)	Minimum laboratory sample amount (g) [a]	Short term storage conditions before delivery to the lab	Long term storage condition before delivery to the lab	Container material
C, H, N	100	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Cl, S, Br, F	100	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Metallic Al	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements	400	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Trace elements excluding Hg	200	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Hg	100	In the same condition of SRF production	refrigeration 4 °C	Glass or PFA bottle
C, H, N, Cl, S, Br, F	150	In the same condition of SRF production	refrigeration 4 °C	non-PVC plastic bottle or bag
Major elements + Trace elements excluding Hg	500	In the same condition of SRF production	refrigeration 4 °C	plastic bottle or bag
Major elements + Trace elements + Hg	600	In the same condition of SRF production	refrigeration 4 °C	Glass bottle (100 g) + plastic bottle or bag

Table A.1 – (*concluded*)

Major elements + Trace elements + Hg + metallic Al	700	In the same condition of SRF production	refrigeration 4 °C	Glass bottle (100 g) + plastic bottle or bag
Complete analysis	800	In the same condition of SRF production	refrigeration 4 °C	Glass bottle (100 g) + non-PVC plastic bottle or bag

[a] The maximum particle size (mm) is related to the laboratory sample amount (g) in order to guarantee sample homogeneity. It is established following the rules reported in prEN 15413.

Bibliography

[1] prEN 15442, *Solid recovered fuels — Methods for sampling*

[2] prEN 15443, *Solid recovered fuels — Methods for the preparation of the laboratory sample*

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