BRITISH STANDARD

BS EN 14775:2009

Solid biofuels — Determination of ash content

ICS 75.160.10

BSI
British Standards

National foreword

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Biocombustibles solides - Méthode de détermination de la teneur en cendres

Feste Biobrennstoffe - Bestimmung des Aschegehaltes

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Foreword

This document (EN 14775:2009) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2010, and conflicting national standards shall be withdrawn at the latest by May 2010.

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1 Scope

This European Standard specifies a method for the determination of ash content of all solid biofuels (CEN/TS 14588).

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.

CEN/TS 14588:2003, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

CEN/TS 14778 (all parts), *Solid biofuels – Sampling*

CEN/TS 14780, *Solid biofuels – Methods for sample preparation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 14588:2003 and the following apply.

3.1
ash content
on dry basis, mass of inorganic residue remaining after ignition of a fuel under specified conditions expressed as a percentage of the mass of the dry matter in the fuel

4 Principle

The ash content is determined by calculation from the mass of the residue remaining after the sample is heated in air under rigidly controlled conditions of time, sample weight and equipment specifications to a controlled temperature of (550 ± 10) °C.

Automatic equipments may be used when the method is validated with biomass reference samples of an adequate biomass type. This equipment shall fulfill all the requirements given in Clause 7 regarding sample size, heating procedure, atmosphere, temperatures and weighing accuracy.

NOTE Difference in the ash content determined at a higher temperature, 815 °C, according to ISO 1171, compared to 550 °C is explained by the decomposition of carbonates forming CO_2, by losses of volatile inorganic compounds and further oxidation of inorganic compounds (to higher oxidation stage). In the ash content found in practise, for instance at a combustion plant, some of the released inorganic compounds are likely to be recovered in the fly ash while CO_2 and other gaseous compounds are traversed to air and will not form a part of the total amount of ash produced.

5 Apparatus

5.1 Dish

A dish of inert material, such as porcelain, silica or platinum and of such size that the sample loading does not exceed 0,1 g/cm^2 bottom area.

5.2 Furnace

Furnace capable of giving a zone of uniform temperature at the levels required by the procedure and reaching these levels in the specified times. The ventilation rate through the furnace should be such that no lack of oxygen for combustion arises during the heating procedure.

NOTE A ventilation rate of between five and ten air changes per minute should be suitable.

5.3 Balance

A balance having sufficient accuracy to enable the dish containing the sample to be weighed to the nearest 0,1 mg.

5.4 Desiccator

With desiccant.

WARNING Ashes from solid biofuels are very hygroscopic and there is a risk that moisture bound in the desiccant can be absorbed in the sample. Therefore the desiccant shall be controlled frequently and dried if necessary.

6 Preparation of test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm or less, prepared in accordance with CEN/TS 14780. The determination of ash content shall be done either:

a) directly on the prepared general analysis test sample, including a concurrently determination of the moisture content of the general analysis test sample according to EN 14774-3; or

b) from a test portion of the general analysis sample which has been dried using the same drying procedure as in the determination of the moisture content of the general analysis sample and kept absolutely dry before the weighing for the ash content determinations (test portion is kept in a closed container in a desiccator).

NOTE For some solid biofuels it may be necessary to prepare a test sample with a lower nominal top size than 1 mm (e.g. 0,25 mm) in order to keep the stated precision.

7 Procedure

7.1 General

A minimum of two determinations shall be carried out on the test sample.

7.2 Heat the empty dish in the furnace to (550 ± 10) °C for at least 60 min. Remove the dish from the furnace. Allow the dish to cool on a heat resistant plate for 5 min to 10 min and then transfer to a desiccator without desiccant and allow to cool to ambient temperature. When the dish is cool, weigh to the nearest 0,1 mg and record the mass.

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NOTE 1 Several dishes can be handled at the same time.

NOTE 2 For determination of the ash content at 815 °C, see bibliography.

7.3 The general analysis sample shall be mixed carefully before weighing. Place minimum 1 g of sample on the bottom of the dish and spread in an even layer over the bottom surface. Weigh the dish plus the sample to the nearest 0,1 mg and record the mass. If the test sample previously has been oven-dried, both the dish and the sample should be dried at 105 °C and then weighed as a precautionary measure for absorption of moisture.

NOTE If the ash content is expected to be very low, use a larger sample size (and a larger dish) to improve the accuracy.

7.4 Place the dish in a cold furnace. Heat the sample in the furnace according to the following heating routine:

a) Raise the furnace temperature evenly to 250 °C over a period of 30 min to 50 min (i.e. a heating rate of 4,5 °C/min to 7,5 °C/min). Maintain at this temperature level for 60 min to allow the volatiles to leave the sample before ignition;

b) Continue to raise the furnace temperature evenly to (550 ± 10) °C over a period of 30 min (i.e. a heating rate of 10 °C/min). Maintain at this temperature level for at least 120 min.

7.5 Remove the dish with its content from the furnace. Allow the dish and its content to cool on a heat resistant plate for 5 min to 10 min and then transfer to a desiccator without desiccant and allow to cool to ambient temperature. Weigh the ash and the dish to the nearest 0,1 mg as soon as ambient temperature is reached and record the mass. Calculate the ash content of the sample as detailed in Clause 8.

7.6 If there is any doubt of incomplete incineration (for instance presence of soot at visual inspection) then:

a) the sample is reloaded into the hot furnace (at 550 °C) for further 30 min periods until the change in mass is lower than 0,5 mg; or

b) droplets of distilled water or ammonium nitrate are added to the sample before it is reloaded into the cold (at room temperature) furnace, and reheated to (550 ± 10) °C and kept at this temperature for further 30 min periods until the change in mass is lower than 0,5 mg.

8 Calculations

The ash content on dry basis, A_d, of the sample expressed as a percentage by mass on a dry basis shall be calculated using the following formula:

$$A_d = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100 \times \frac{100}{100 - M_{ad}} \quad (1)$$

where

m_1 is the mass, in g, of the empty dish;

m_2 is the mass, in g, of the dish plus the test sample;

m_3 is the mass, in g, of the dish plus ash;

M_{ad} is the % moisture content of the test sample used for determination.

The result shall be reported as the mean of duplicate determinations to the nearest 0,1 %.

9 Precision

9.1 General

Precision must be given in a standard (comments and input on precision from your experience please).

9.2 Repeatability

The result of duplicate determinations, carried out over a short period, but not simultaneously, in the same laboratory by the same operator with the same apparatus on two representative portions taken from the same general analysis sample, should not differ more than the above value.

9.3 Reproducibility

The means of result of duplicate determinations carried out in two different laboratories, on representative portions taken from the same general analysis sample, should not differ more than the values given in Table 1.

Table 1 – Repeatability and reproducibility of the method

Ash content %	Maximum acceptable differences between results	
	Same laboratory (Repeatability)	Different laboratories (Reproducibility)
Less than 10 %	0,2 % absolute	0,3 % absolute
Equal to or greater than 10 %	2,0 % of the mean result	3,0 % of the mean result

10 Test report

The test report shall include at least the following information:

— identification of the laboratory and the testing date;

— identification of the product or sample tested (see CEN/TS 14778);

— a reference to this European Standard;

— any deviation from this European Standard;

— test result on dry basis;

— conditions and observations, i.e. unusual features during the test procedure, which may affect the result.

Bibliography

[1] ISO 1171, *Solid Mineral Fuels – Determination of ash content*

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