TUHASUSE MÄÄRAMINE

1. Töö eesmärk

Määrata kahe etteantut kütuse tuhasus toetudes standardile EN 14775.

2. Tööks vajalikud vahendid

- Muhvelahi
- Kaks kütust: pelletid (niiskus 12%) ja põlevkivi (niiskus 0.5%)
- Tiiglid
- Tangid
- Eksikaator

3. Teoreetilised alused

EN 14775

4. Töö käik

a) Kaaluda tühi tiigel
b) Kaaluda tühja tiiglisse 0.3±0.1 g kütust
c) Korrata protseduuri järgmiste tiiglitega
d) Kontrollida, et ahi oleks saavutanud nõutud temperatuuri
e) Asetada tangide abil tiiglid ahju
f) 10 minuti pärast eemaldada tangide abil tiiglid ahjust, hinnata silmaga kas on põlemata kütust
g) Asetada tiiglid tangide abil eksikaatorisse jahtuma (10 minutit)
h) Kaaluda tiiglid
i) Tühjendada tiiglid ja kaaluda uuesti tühjad tiiglid

5. Katseandmete töötlemine

Arvutada standardi abil kütuste tuhasus ja hinnata, kas mõõtmine õnnestus (määramatus jääb nõutud piiridesse).

6. Kirjandus

EN 14775:2004 Solid biofuels – Method for the aetermination of ash content

EESTI STANDARD CEN/TS 14775:2004

Solid biofuels - Method for the determination of ash content

Solid biofuels - Method for the determination of ash content

EESTI STANDARDIKESKUS

EESTI STANDARDI EESSÕNA NATIONAL FOREWORD

Käesolev Eesti standard CEN/TS 14775:2004 sisaldab Euroopa standardi CEN/TS 14775:2004 ingliskeelset teksti. Käesolev dokument on jõustatud 11.11.2004 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes. Standard on kättesaadav Eesti standardiorganisatsioonist.	This Estonian standard CEN/TS 14775:2004 consists of the English text of the European standard CEN/TS 14775:2004. This document is endorsed on 11.11.2004 with the notification being published in the official publication of the Estonian national standardisation organisation. The standard is available from Estonian standardisation organisation.

Käsitlusala: This document specifies a method for the determination of ash content of all solid biofuels (CEN/TS 14588).	**Scope:** This document specifies a method for the determination of ash content of all solid biofuels (CEN/TS 14588).

ICS 75.160.10

Võtmesõnad:

Hinnagrupp E

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 14775

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English version

Solid biofuels - Method for the determination of ash content

Feste Biobrennstoffe - Verfahren zur Bestimmung des Aschegehaltes

This Technical Specification (CEN/TS) was approved by CEN on 5 February 2004 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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Foreword

This document (CEN/TS 14775:2004) has been prepared by Technical Committee CEN/TC 335 “Solid Biofuels”, the secretariat of which is held by SIS.

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, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document 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 description*

CEN/TS 14774-3, *Solid Biofuels – Method for the determination of moisture content – Oven dry method – Part 3: Moisture in general analysis sample*

prCEN/TS 14779, *Solid Biofuels – Methods for preparing sampling plans and sampling certificates*

prCEN/TS 14780, *Solid Biofuels – Methods for sample reduction*

3 Terms and definitions

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

3.1
Ash content, 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.

NOTE Difference in the ash content determined at a higher temperature, 815 °C, according to ISO 1171-1997, compared to 550 °C can be explained by the loss of volatile inorganic compounds, further oxidation (higher oxidation state) of inorganic compounds and the decomposition of carbonates forming CO_2. 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 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 minutes 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

Without desiccant.

NOTE The use of desiccator without desiccant are specified in ISO 1171:1997 Solid Mineral Fuels – Determination of ash content for coal ashes and emphasised here since ashes from solid biofuels are often more hygroscopic than coal ashes.

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 prCEN/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 CEN/TS 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

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

7.1 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 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.

NOTE Several dishes can be handled at the same time.

7.2 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 previous has been oven-dried, both the dish and the sample should be dried at 105 ^{o}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.

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

- Raise the furnace temperature evenly to 250 °C over a period of 50 minutes (i.e. a rise of 5 °C/min). Maintain at this temperature level for 60 min to allow the volatiles to leave the sample before ignition.

- Continue to raise the furnace temperature evenly to (550 ±10) °C over either a period of 60 minutes, or a rise of 5 °C/min, and keep this temperature level for at least 120 min.

7.4 Remove the dish with its content from the furnace. Allow the dish and its content to cool on a **heat resistant** plate for 5 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.

7.5 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 0,2 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 0,2 mg.

8 Calculations

The ash content on dry basis, A_{db}, 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 grams, of empty dish;

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

m_3 is the mass, in grams, 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

Because of the varying nature of the solid biofuels covered by this document it is not possible at this time to give a precision statement (repeatability or reproducibility) for this test method.

See Annex A

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 prCEN/TS 14779);

- a reference to this document;

- any deviation from the standard;

- test result on dry basis;

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

Annex A

(Informative)

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.

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 above value.

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

NOTE The values given in Table 1 are based on precision data for coal and coke given in; ISO 1171:1997 Solid Mineral Fuels – Determination of ash content.

Bibliography

[1] ISO 1171:1997, *Solid Mineral Fuels – Determination of ash content*