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Second edition
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Solid biofuels — Determination of ash content

Biocombustibles solides — Détermination de la teneur en cendres

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 238, *Solid biofuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 335, *Solid biofuels,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 18122:2015), which has been technically revised.

The main changes are as follows:

— more detailed descriptions of the ashing furnace and ashing procedure;

— repeatability and reproducibility performance data updated;

— several references updated;

— minor editorial corrections.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Ash content is an important parameter for fuel deliveries since ash is a by-product of combustion and ends up as bottom ash or fly-ash and needs to be removed. Depending on the jurisdiction, ash may be deposited or used for production of other products or as fertilizer. Knowing how much ash comes with a fuel can have economic consequences. Since the chemical composition of ash contributes to slagging and corrosion in the combustion equipment, it is therefore important to know the amount of ash contained in a fuel. Other testing standards are used for determining the chemical composition of ash.

Solid biofuels — Determination of ash content

1 Scope

This document specifies a method for the determination of ash content of all solid biofuels.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 14780, *Solid biofuels — Sample preparation*

ISO 16559, *Solid biofuels — Vocabulary*

ISO 18134-3, *Solid Biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

ISO 18135, *Solid Biofuels — Sampling*

ISO 21945, *Solid biofuels — Simplified sampling method for small scale applications*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16559 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The ash content is determined by calculating 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 final temperature. The final temperature of 550 ± 10 °C is considered standard; however, other final temperatures may be used provided all other furnace conditions (e.g. air flows, temperature ramp rates, hold times) remain the same and the alternative final temperature is clearly referenced on the test report. Alternative final temperatures referenced in other test methods include 710 °C and 815 °C. The repeatability and reproducibility limits provided in Clause 9 are not applicable to alternative final temperatures.

Automatic equipment (such as gravimetric analysers) may be used when the method is validated with biomass reference samples of an adequate biomass type. The automatic equipment shall fulfil all the requirements given in Clause 7 regarding sample size, heating procedure, atmosphere, temperature and weighing accuracy.

NOTE The difference in the ash content if determined at a higher temperature, 815 °C, according to Reference [1], rather than 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 states).

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

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

NOTE If the test portion loading exceeds 0,1 g/cm^2 of bottom area there is a risk of incomplete incineration (in the lower sample layer) or absorption of CO_2 in the ash layer at the top (as $CaCO_3$) of calcium-rich samples (as e.g. pure wood).

5.2 Furnace, capable of providing a zone of uniform heat at the temperatures required and reaching these temperatures within the specified times. The air exchange in the furnace shall be sufficient to remove the flue gasses SO_2 and CO_2 formed during decomposition of the biofuel before these gases react with the ash components during the heating procedure. Furnace parameters are listed in 7.3.

NOTE For preparation of coal ashes according to ISO 1171, five to 10 air exchanges per minute are necessary to eliminate reaction of SO_2 and CO_2 with the ash. For biomass there is currently no scientific proof for the influence of air exchange in the ashing furnace, although an influence is expected. Biomass usually has a lower ash content and ash is lighter in weight than coal ash. This can cause the ash to be blown from the ashing crucible, which can limit air exchange possibilities. A sensitivity analysis of variation of these parameters on the result of ashing can be valuable for a certain set-up.

5.3 Balance, capable of reading to the nearest 0,1 mg.

5.4 Desiccator with appropriate **desiccant**, required to prevent absorption of moisture from the atmosphere by the test sample.

WARNING — Ash from solid biofuel is 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. In addition, lids shall be used to cover dishes while in the desiccator to prevent the absorption of moisture.

6 Sample preparation

6.1 General

A laboratory sample for the determination of ash content shall be obtained in accordance with ISO 18135 or ISO 21945. From the laboratory sample a general analysis sample shall be prepared in accordance with ISO 14780 and have a nominal particle top size of 1 mm or less. The general analysis sample shall include material sufficient for determination of ash content and moisture content.

6.2 Sample conditioning

The determination of ash content shall be done either:

a) directly on a test portion of the general analysis sample, including a concurrent determination of the moisture content of a similar test portion in accordance with ISO 18134-3; 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 test portion and kept absolutely dry before the weighing for the ash content determinations (the test portion shall be kept in a closed container in a desiccator with desiccant).

NOTE For some solid biofuels it might be necessary to prepare a general analysis sample to a nominal top size of less than 1 mm (e.g. 0,25 mm) in order to keep the stated precision.

7 Procedure

7.1 General

The standard final temperature of ashing is (550 ± 10) °C. The description of the procedure is based on this final temperature. If other final temperatures are used all other furnace conditions (e.g. air flows, temperature ramp rates, hold times) shall remain the same.

NOTE Ashing at higher temperatures removes carbonates from the ash and can possibly remove low melting salts resulting in a lower ash content.

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

7.2 Conditioning of dish

Heat the empty dish in the furnace to (550 ± 10) °C for at least 60 min to remove any biogenic material and moisture. 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 with 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.

7.3 Conditioning of the general analysis sample

The general analysis sample shall be mixed carefully before weighing the test portion. Place a minimum of 1 g of test portion at the bottom of the dish and spread in an even layer over the bottom surface. Weigh the dish plus the test portion to the nearest 0,1 mg and record the mass. If the test portion has previously been oven-dried, both the dish and the test portion shall 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 test portion (and a larger dish) to improve the accuracy.

7.4 Ashing of test portion

Place the dish in a cold furnace and heat the test portion in accordance with the following temperature programme:

— 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 the temperature at this level for 60 min to allow the volatiles to leave the test portion before ignition.

— 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 the temperature at this level for at least 120 min.

7.5 Weighing

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 with desiccant and allow to cool to ambient temperature. Weigh the dish with ash to the nearest 0,1 mg as soon as ambient temperature is reached and record the mass. Calculate the ash content of the test portion as detailed in Clause 8.

7.6 Completion of ashing

If there is any doubt of complete incineration (e.g. presence of soot at visual inspection), reload the dish with ash into the hot furnace (at 550 °C) for additional 30 min periods until the change in mass is lower than 0,05 % of the initial sample mass.

NOTE If the incineration is incomplete, it can be improved by adding a few droplets of distilled water to the sample before it is reloaded into a cold (at room temperature) furnace, reheated to (550 ± 10) °C and kept at this temperature for further 30 min periods until the change in mass is lower than 0,05 % of the initial sample mass.

8 Calculation

The ash content on dry basis, A_d, of the sample expressed as a mass fraction in % on a dry basis shall be calculated using Formula (1):

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

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 portion;

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

M_{ad} is the percentage moisture content of the test portion as determined.

The result shall be calculated to two decimal places and the mean value shall be rounded to the nearest 0,1 % for reporting.

9 Performance characteristics

The information provided in Annex A shows the results obtained by multiple international laboratory comparison studies carried out on wood pellets, almond kernels, coconut shells, wheat straw and olive residues. These five samples represent a variety of solid biofuel material types and are an indication of the repeatability and reproducibility that can be achieved with this method.

10 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) a reference to this document, i.e. ISO 18122:2022;

d) final ashing temperature used if other than (550 ± 10) °C;

e) results of the test on dry basis as indicated in Clause 8;

f) any unusual features noted during the determination which can affect the result;

g) any deviation from this document, or operations regarded as optional;