INTERNATIONAL STANDARD

ISO 18122

First edition
2015-10-15

Solid biofuels — Determination of ash content

Biocombustibles solides — Méthode de détermination de la teneur en cendres

Reference number
ISO 18122:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. 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. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 238, *Solid biofuels*.

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 and knowing how much ash comes with a fuel may have economic consequences. In addition, the chemical composition of ash contributes to slagging and corrosion in the combustion equipment and 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 International Standard specifies a method for the determination of ash content of all solid biofuels.

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.

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

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

EN 14778[1)], *Solid Biofuels — Sampling*

EN 14780[2)], *Solid Biofuels — Sample preparation*

3 Terms and definitions

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

3.1
nominal top size
aperture of the sieve where at least 95 % by mass of the material passes

[SOURCE: ISO 16559]

3.2
laboratory sample
combined sample or a sub-sample of a combined sample for use in a laboratory

[SOURCE: ISO 16559]

3.3
test sample
laboratory sample after an appropriate preparation made by the laboratory

[SOURCE: ISO 16559]

3.4
test portion
sub-sample either of a laboratory sample or a test sample

[SOURCE: ISO 16559]

1) To be replaced by ISO 18135.

2) To be replaced by ISO 14780.

3.5
general analysis sample
sub-sample of a laboratory sample having a nominal top size of 1 mm or less and used for a number of chemical and physical analyses

[SOURCE: ISO 16559]

4 Principle

The ash content is determined by calculation 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 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 Difference in the ash content if determined at a higher temperature, 815 °C according to Reference [1] as 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 states).

5 Apparatus

5.1 Dish

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

NOTE If the test sample 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

The furnace shall be capable of providing a zone of uniform heat at the temperatures required and reaching these temperatures within the specified times. The ventilation rate through the furnace shall be such that no lack of oxygen for combustion arises during the heating procedure.

NOTE A ventilation rate of between five and 10 air changes per hour is sufficient.

5.3 Balance

The balance shall be capable of reading to the nearest 0,1 mg.

5.4 Desiccator and desiccant

A desiccator with appropriate desiccant is 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

A laboratory sample for the determination of ash content shall be obtained in accordance with EN 14778. From the laboratory sample a general analysis sample is prepared in accordance with EN 14780 and has a nominal particle top size of 1 mm or less.

6.1 Sample size

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 may 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 Conditioning of dish

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 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 1 Several dishes can be handled at the same time.

NOTE 2 For determination of the ash content at 815 °C, see Reference [1].

7.2 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 previously has 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 size test portion (and a larger dish) to improve the accuracy.

7.3 Ashing of test portion

Place the dish in a cold furnace and heat the test portion in accordance with the following temperature program.

— Raise the furnace temperature evenly to 250 °C over a period of 30 min to 50 min (i.e. 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. heating rate of 10 °C/min). Maintain temperature at this level for at least 120 min.

7.4 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.

7.5 Completion of ashing

If there is any doubt of complete incineration (for instance 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 0,5 mg.

For improved incineration, droplets of distilled water or ammonium nitrate solution shall be 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.

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

8 Calculation

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 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 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 % moisture content of the test portion used for determination.

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

9.1 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 test portions taken from the same general analysis sample, shall not differ more than the values stated in Table 1.

9.2 Reproducibility

The mean value of results of duplicate determinations carried out in two different laboratories, on representative test portions taken from the same sample, shall not differ more than the values stated in Table 1, see Reference [2].

Table 1 — Repeatability and reproducibility of the method

Ash content %	Maximum acceptable differences between results	
	Repeatability	Reproducibility
<1 %	0,1 % absolute	0,2 % absolute
>1 %	10 % relative	20 % relative

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 International Standard, i.e. ISO 18122;

d) results of the test on dry basis (alternatively for all standards: results of the test including the basis in which they are expressed, as indicated in Clause 8);

e) any unusual features noted during the determination; which may affect the result;

f) any deviation from this International Standard, or operations regarded as optional.