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Solid recovered fuels — Determination of ash content

Combustibles solides de récupération — Détermination de la teneur en cendres

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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 (see www.iso.org/directives).

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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 300, *Solid recovered fuels*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 343, *Solid recovered fuels*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

This document covers the determination of ash content of solid recovered fuels. It is primarily geared toward laboratories, producers, suppliers and purchasers of solid recovered fuels but is also useful for the authorities and inspection organizations.

The method A specified in this document is based on EN 15403^[5].

For information about environmental aspect, see [Annex B](#).

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Solid recovered fuels — Determination of ash content

1 Scope

This document specifies methods for the determination of ash content of all solid recovered fuels.

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 21637, *Solid recovered fuels — Vocabulary*

ISO 21645¹⁾, *Solid recovered fuels — Methods for sampling*

ISO 21646²⁾, *Solid recovered fuels — Sample preparation*

ISO 21660-3³⁾, *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

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3 Terms and definitions (standards.iteh.ai)

For the purposes of this document, the terms and definitions given in ISO 21637 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

ash

ash content on dry basis

total ash

A

mass of inorganic residue remaining after combustion of a fuel under specified conditions, typically expressed as a percentage of the mass of dry matter in fuel

Note 1 to entry: Depending on the combustion efficiency the ash may contain combustibles.

Note 2 to entry: If a complete combustion is realized, ash contains only inorganic, non-combustible components.

[SOURCE: ISO 16559:2014, 4.13, modified — “Note 1 to entry” was removed and the following ones renumbered, and symbol “A” was italicized.]

3.2

total ash content

mass of inorganic residue remaining after ignition of a fuel under specified conditions, expressed as mass fraction in percent of the dry matter in the fuel, which also includes removed ash contributors

- 1) Under preparation. (Stage at the time of publication: ISO/FDIS 21645:2020.)
- 2) Under preparation. (Stage at the time of publication: ISO/DIS 21646:2020.)
- 3) Under preparation. (Stage at the time of publication: ISO/FDIS 21660-3:2020.)

3.3

removed ash contributors

rac

coarse inert material (i.e. metals, glass, stones, tiles etc.) removed from the pre-dried sample before preparation, in order to avoid damage to the preparation equipment

Note 1 to entry: Removed ash contributors are included in the total ash content calculations.

[SOURCE: ISO 21637:2020, 3.62, modified: Note 1 to entry was added]

3.4

total organic matter

combustible part of solid recovered fuels, which consists of the sum of volatile matter and fixed carbon

Note 1 to entry: It is calculated as: 100 - moisture content - ash content.

Note 2 to entry: It is the mass fraction of the matter lost by ignition, also known as "Loss of Ignition" (LOI).

3.5

volatile matter

relative part of the analysed sample, after moisture removal, that is lost when material is heated up under specific conditions of temperature, time and in a reduced atmosphere (anoxic conditions)

3.6

fixed carbon

relative part of carbon contained in a material that can only be degraded in oxic conditions and high temperature

Note 1 to entry: It is calculated as: 100 - moisture content - volatile matter content - ash content.

4 Principle

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The sample is heated in air atmosphere up to a temperature of (550 ± 10) °C for Method A or (815 ± 10) °C for Method B under rigidly controlled conditions of time, sample mass and equipment specifications. The ash content is determined by calculation from the mass of the residue remaining after heating.

NOTE Difference in the ash content if determined at 815 °C compared to 550 °C is explained by decomposition of carbonates forming CO₂, losses of volatile inorganic compounds (see also 3.5) and further oxidation of inorganic compounds^[1]. In common standard practise, 550 °C is used for the determination of ash content in SRFs with a high content of biomass. 550 °C can also be used major and minor elemental determination (see also EN 15410^[6]).

Automatic equipment (such as thermogravimetric analysers) may be used as long as the equipment is validated by parallel measurements to the reference method. The automatic equipment shall fulfil all the requirements regarding sample size, heating procedure, temperature, atmosphere and weighing accuracy. Deviations from this paragraph shall be reported and justified.

5 Apparatus

5.1 Dish, consisting of inert material such as porcelain, silica or platinum, with a depth from 10 mm to 20 mm and such a size that the sample loading does not exceed 0,1 g/cm² bottom area.

5.2 Furnace, capable of maintaining a zone of uniform temperature at the levels required in [Clause 7](#) and to reach these levels in the specified heating rates. The ventilation rate through the furnace should be such that no lack of oxygen arises during the heating procedure.

NOTE A ventilation rate from 5 air changes/min to 10 air changes/min are suitable.

5.3 Balance, capable of weighing the dish containing the sample to the nearest 0,1 mg.

5.4 Desiccator, without desiccant.

NOTE The use of a desiccator without desiccant is specified in ISO 1171 and emphasised here since ashes from solid recovered fuels are often more hygroscopic than coal ashes.

5.5 Sieve, with an aperture size of ≤ 1 mm (according ISO 3310-1 or ISO 3310-2).

5.6 Container, sealed airtight.

6 Sampling and sample preparation

6.1 General

The general analysis sample shall be taken and prepared in accordance with ISO 21645 and ISO 21646. It shall be ground to pass through a sieve with an aperture size of ≤ 1 mm. The general analysis sample shall be received in the container (5.6). The general analysis sample shall either be oven-dried or its moisture content determined in accordance with ISO 21660-3. The general analysis sample shall be mixed carefully before weighing (see also Clause 7).

6.2 Pre-drying

Pre-drying shall be performed according to ISO 21646.

Pre-drying of wet samples is carried out to minimize moisture loss in the subsequent sample-division processes, to facilitate the sample preparation processes, and to minimize biological activity. If it is necessary to dry a sample by heating, it shall be dried in an oven at a temperature not exceeding the temperature according ISO 21646.

6.3 Removed ash contributors (rac)

Coarse inert material (i.e. metals, glass, stones) removed from the pre-dried sample before preparation, in order to avoid damage to the preparation equipment.

Removed ash contributors (rac) from pre dried samples shall be considered as dry.

A_{rac} is weighed separately, calculated as mass percent and added to the determined ash content according to calculation in 8.2.

7 Procedure

7.1 General

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

7.2 Method A – ash content at 550 °C

For the determination of the biomass fraction, use the temperature of 550 °C for all cases.

Heat the empty dish (5.1) in the furnace (5.2) to (550 ± 10) °C for at least 60 min. Allow the dish to cool down in a desiccator. After the dish is cooled, weigh it to the nearest 0,1 mg and record the mass.

Place about 1 g of the general analysis 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 general analysis sample is oven-dried, both the dish and the sample shall be dried at (105 ± 10) °C as a precautionary measure against water absorption and then weighed.

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

- a) raise the furnace temperature evenly to (250 ± 10) °C over a period of 50 min (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;
- b) continue to raise the furnace temperature evenly to (550 ± 10) °C over a period of 60 min (i.e. a rise of 5 °C/min) and keep this temperature level for at least 120 min.

Remove the dish with its content from the furnace. Allow the dish and its content to cool on a thick metal 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](#). If there is any doubt of complete incineration (for instance presence of soot at visual inspection), then add droplets of water or ammonium nitrate to the sample before it is reloaded into the cold furnace and reheated to (550 ± 10) °C for a period of further 30 min until the change in mass is lower than 0,2 mg.

Automatic equipment may be used if the method is validated with biomass reference samples of an adequate biomass type. This equipment shall fulfil all the requirements given in this clause regarding sample size, heating procedure, atmosphere, temperatures and weighing accuracy. Deviations from this paragraph shall be reported and justified.

7.3 Method B – ash content at 815 °C

Heat the empty dish ([5.1](#)) in the furnace ([5.2](#)) to (815 ± 10) °C for at least 60 min. Allow the dish to cool down in a desiccator. After the dish is cooled, weigh it to the nearest 0,1 mg and record the mass.

Place about 1 g of the general analysis 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 general analysis sample is oven-dried, both the dish and the sample shall be dried at (105 ± 10) °C as a precautionary measure against water absorption and then weighed.

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

- a) raise the furnace temperature evenly to (250 ± 10) °C over a period of 50 min (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;
- b) continue to raise the furnace temperature evenly to (815 ± 10) °C over a period of 60 min (i.e. a rise of 15 °C/min) and keep this temperature level for at least 120 min.

Remove the dish with its content from the furnace. Allow the dish and its content to cool on a thick metal 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](#). If there is any doubt of complete incineration (for instance presence of soot at visual inspection), then add droplets of water or ammonium nitrate to the sample before it is reloaded into the cold furnace and reheated to (815 ± 10) °C for a period of further 30 min until the change in mass is lower than 0,2 mg.

Automatic equipment may be used if the method is validated with biomass reference samples of an adequate biomass type. This equipment shall fulfil all the requirements given in this clause regarding sample size, heating procedure, atmosphere, temperatures and weighing accuracy. Deviations from this paragraph shall be reported and justified.

8 Calculation

8.1 General analysis sample

The ash content on wet basis, A_{ad} , of the general analysis sample “as analysed”, expressed as mass fraction in percent, shall be given by [Formula \(1\)](#):

$$A_{ad} = \frac{m_3 - m_1}{m_2 - m_1} \cdot 100 \quad (1)$$

The ash content on dry basis, A_{db} , of the general analysis sample, expressed as mass fraction in percent, shall be calculated by [Formula \(2\)](#):

$$A_{db} = \frac{m_3 - m_1}{m_2 - m_1} \cdot 100 \cdot \frac{100}{100 - M_{ad}} = A_{ad} \cdot \frac{100}{100 - M_{ad}} \quad (2)$$

where

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

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

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

M_{ad} is the mass fraction of moisture of the general analysis sample on wet basis, in percent.

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

8.2 Calculation of total ash content including removed ash contributors, on an as received and dry basis

The total ash content on an as received basis, $A_{total,ar}$, of the sample, expressed as mass fraction in percent, shall be calculated by [Formula \(3\)](#):

$$A_{total,ar} = A_{ad} \cdot \left(1 - \frac{M_p}{100} - \frac{A_{rac,ar}}{100} \right) + A_{rac,ar} \quad (3)$$

where $A_{rac,ar}$ is according to [Formula \(4\)](#):

$$A_{rac,ar} = \frac{m_{rac}}{m_{ar}} \cdot 100 \quad (4)$$

The total ash content on a dry basis, $A_{total,db}$, of the sample, expressed as mass fraction in percent, shall be calculated by [Formula \(5\)](#):

$$A_{total,db} = A_{total,ar} \cdot \frac{100}{100 - M_T} \quad (5)$$

where the mass fraction of moisture content on an as received basis, M_{ar} , in percent, is according to [Formula \(6\)](#):

$$M_T = M_p + M_{ad} \cdot \left(1 - \frac{M_p}{100} - \frac{A_{rac,ar}}{100} \right) \quad (6)$$

where