INTERNATIONAL STANDARD

ISO 21404

First edition
2020-01

Solid biofuels — Determination of ash melting behaviour

Biocombustibles solides — Méthode de détermination de la fusibilité des cendres

Reference number
ISO 21404:2020(E)

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

Published in Switzerland

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 (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).

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 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 238, *Solid biofuels*.

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

The test method described in this document provides information about fusion and melting behaviour of the composite inorganic constituents of the solid biofuel ash at high temperatures.

Ash melting is a complex process where also sintering, shrinkage and expansion or swelling can occur.

The test method is empirical. The ash used for the test is a homogeneous material, prepared from the fuel by ashing at 550 °C (alternatively, ashing temperatures of 710 °C or 815 °C may be used). The determination is performed at a controlled rate of heating in a controlled atmosphere. In contrast, under full-scale conditions, the complex processes of combustion and fusion involve heterogeneous mixtures of particles, variable heating rates and gas compositions.

The determined characteristic temperatures in the test can be used for comparison of the tendency of the ashes from different types and qualities of solid biofuels to form fused deposits or to cause bed agglomeration on heating.

The method is based on the methods described in DIN 51730:1998[1], ISO 540:2008[2] and CEN/TS 15370-1[3]. The terms ash fusibility and ash softening are synonyms to ash melting.

Solid biofuels — Determination of ash melting behaviour

1 Scope

This document specifies a method for the determination of the characteristic temperatures for the ash melting behaviour of 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*

3 Terms and definitions

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

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

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

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

3.1
shrinkage starting temperature
SST
temperature is defined as when the area of the test piece falls below 95 % of the original test piece area at 550 °C (or other ashing temperature used) due to shrinking of the test piece

Note 1 to entry: Shrinkage may be due to liberation of carbon dioxide and volatile alkali compounds. It may also be due to sintering and may be a first sign of partial melting.

3.2
deformation temperature
DT
temperature at which the first signs of melting occur

Note 1 to entry: It is common that DT will occur shortly before HT/FT. If the test results show a small temperature difference between SST and DT and a large temperature gap between DT and HT/FT it is advised that the analyst review the images to verify if the temperature recorded as DT is truly due to melting or if it is a shape change caused by excessive shrinkage.

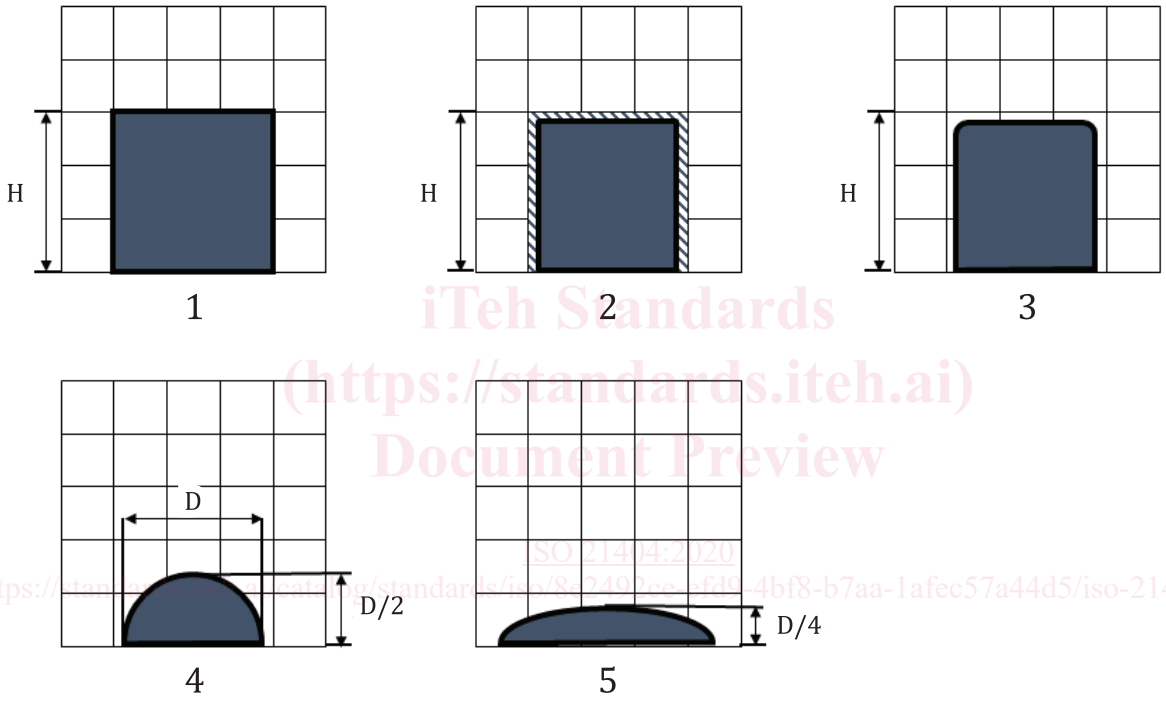
Note 2 to entry: Deformation temperature can be seen as rounding of the edges, smoothing of surfaces, expansion of the cylinder or general changing of the cylinder shape. If the test piece starts to swell or bubble without the edges being rounded, the temperature is registered as DT (since swelling and bubbling only occur when a fraction of the ash is melted).

Note 3 to entry: For computerized evaluation a shape factor change can be used to define the deformation temperature. For definition of shape factor see Annex A.

3.3
hemisphere temperature
HT
temperature at which the test piece forms approximately a hemisphere i.e. when the height is half of the base diameter

3.4
flow temperature
FT
temperature at which the ash is spread out over the supporting tile in a layer, the height of which is half of the height of the test piece at the hemisphere temperature as depicted in Figure 1.

Note 1 to entry: Half of the height of the test piece at the hemisphere temperature has been defined due to frequently occurring bubbling effects. This is especially important for automatic image evaluation.



Key
1 original shape at reference (ashing) temperature
2 SST, Shrinkage starting temperature
3 DT, Deformation temperature
4 HT, Hemisphere temperature
5 FT, Flow temperature

Figure 1 — Phases which can occur in the ash melting process

4 Principle

Ash from biofuel is prepared under controlled conditions of time and equipment specifications to a controlled temperature of (550 ± 10) °C. This ash is homogenized, and a test piece is made from the prepared ash. It is heated up at constant rate and is continuously observed while heated up. The temperatures at which characteristic changes of the shape occur are recorded. The characteristic temperatures are defined in Clause 3. Vivid images in Annex C show examples of the characteristic temperatures.

For some ashes produced at 550 °C it can be difficult to determine the deformation temperature due to liberation of carbon dioxide from carbonates in the ashes, creating strong shrinkage of the test pieces. Ashing at higher temperatures removes carbonates from the ash but also removes possible contents of low melting salts. For some purposes (as e.g. searching for glass melting problems regarding wood pellets) alternative ashing temperatures of (710 ± 10) °C or (815 ± 10) °C may be used provided it is specified in the test report.

5 Reagents

5.1 Ethanol, with a purity ≥95 %.

5.2 Gold wire, of diameter 0,5 mm or larger, or **gold plate,** of thickness 0,5 mm to 1,0 mm with a purity of 99,99 % or a certified melting point (e.g. 1 064 °C).

5.3 Nickel wire, of diameter 0,5 mm or larger, or **nickel plate,** of thickness 0,5 mm to 1,0 mm, with a purity of 99,9 % or a certified melting point (e.g. 1 455 °C).

NOTE Nickel is used for calibration in reducing atmosphere only.

5.4 Palladium wire, of diameter 0,5 mm or larger, or **palladium plate,** of thickness 0,5 mm to 1,0 mm with a purity of 99,9 % or a certified melting point (e.g. 1 554 °C).

5.5 Carbon dioxide, carbon monoxide, hydrogen or **ready mixture of carbon dioxide** and **carbon monoxide** with 55 % (*V*/*V*) to 65 % (*V*/*V*) carbon monoxide and 35 % (*V*/*V*) to 45 % (*V*/*V*) carbon dioxide or **ready mixture of hydrogen** and **carbon dioxide** with 45 % (*V*/*V*) to 55 % (*V*/*V*) hydrogen and 45 % (*V*/*V*) to 55 % (*V*/*V*) carbon dioxide.

6 Apparatus

6.1 Dishes for ashing made of inert material, such as platinum or graphite and of such size that the test portion loading does not exceed 1 g/cm^2 of bottom area can be used for all biomass material. Before the first use, the dish shall be heated to ashing temperature for 60 min and shall cool down to ambient temperature before use. The material of the dish shall not react with the sample or ash of the sample. When using dishes of other materials (e.g. porcelain) it shall be checked that no reaction with the ashes/biomass material occurs during ashing process, i.e. the ash should be a loose powder (no sintering or melt) and the surface of the dishes shall be intact after the ashing.

6.2 Furnace for ashing, which shall be 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.

NOTE For preparation of coal ashes according to ISO 1171, 5 to 10 air changes/min 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 on the ash melting results although an influence is expected. Biomass usually has a lower ash content and ash is of light weight in comparison to coal ash. This property 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 ash melting behavior can be valuable for a certain set-up.

6.3 Sample containers or bags, airtight, suitable for holding (1 to 10) g of ashed sample leaving minimum free air space e.g. LDPE zip -lip bags or max. 50 ml wide-mouth HDPE bottles.

6.4 Furnace for ash melting behaviour, electrically heated, which satisfies the following conditions:

a) it shall be capable of reaching the maximum temperature of at least 1 500 °C);

b) it shall provide an adequate zone of uniform temperature in which to heat the test piece(s);

c) it shall provide means of heating the test piece(s) at a uniform rate from 550 °C upwards;

d) it shall be capable of maintaining the required test atmosphere (see Clause 7) around the test piece(s);

e) it shall provide means of observing the change of shape of the test piece(s) during heating. The requirements for the operation/design of the furnace shall be such that a provision for observing the test in low light conditions is stipulated, be that provision one of illumination or camera sensitivity.

6.5 Pyrometer, comprised of a platinum/platinum-rhodium thermocouple.

6.6 Mould, of brass, stainless steel or other suitable material for preparing an upright cylinder of height 3 mm to 4 mm and with diameter equal to the height.

6.7 Hand press with spring pressure compression, to produce the test piece.

NOTE Manufacturers of ash fusion moulds (6.6) generally provide a hand press designed to be used with the mould. Manufacturer provided hand presses are considered suitable.

6.8 Support for the test piece, of such a material that it becomes neither distorted, not reacts with nor absorbs the ash during the determination.

Zirconium dioxide supports are suitable or a non-absorbent interface such as platinum foil can be used between the original support and the test piece. Supports of sintered alumina or fine textured mullite have shown to influence the result especially for FT due to reaction or absorption of liquid ash and should be avoided.

6.9 Flowmeters, for measuring the components of the reducing gases (see Clause 7) and/or for measuring the flow rate of the oxidizing gas.

6.10 Mortar and pestle made of agate or zirconium oxide or other adequate grinding instruments like ball mill made of abrasion resistant material.

6.12 Optical instrument, which enables the profile of the test piece to be observed throughout the determination by using a camera or video equipment.

7 Test atmosphere

An oxidizing or reducing atmosphere during ash melting test may be used depending on the condition within the combustion system and/or as requested. An oxidizing atmosphere is obtained with air or carbon dioxide introduced into the oven at a linear rate of flow past the test piece between 100 mm/min to 250 mm/min, calculated at ambient temperature. The reducing atmosphere is obtained by introducing a mixture of

a) 55 % (V/V) to 65 % (V/V) carbon monoxide with 35 % (V/V) to 45 % (V/V) carbon dioxide or

b) 45 % (V/V) to 55 % (V/V) hydrogen with 45 % (V/V) to 55 % (V/V) carbon dioxide into the furnace at the same flow rate as for oxidizing atmosphere.

NOTE The flow rate is not very critical, provided that in the case of reducing atmosphere it is sufficient to prevent any leakage of air into the furnace. However, the same flow rate level is recommended also for oxidizing atmosphere. For furnaces with larger diameter a flow around 400 mm/min for reducing atmosphere can be necessary. In all cases refer also to manufacturer instructions. The flow rate for rotameter adjustment can be calculated by multiplying the flow rate in mm/min with the inside cross-section area of the furnace tube and converting to units litres/min.

