SLOVENSKI STANDARD
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Trdna biogoriva - Določevanje taljenja pepela (ISO/DIS 21404:2019)

Solid biofuels - Determination of ash melting behaviour (ISO/DIS 21404:2019)

Biogene Festbrennstoffe - Bestimmung des Asche-Schmelzverhaltens (ISO/DIS 21404:2019)

Biocombustibles solides - Methode de détermination de la fusibilité des cendres (ISO/DIS 21404:2019)

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Solid biofuels — Determination of ash melting behaviour

Biocombustibles solides — Détermination du comportement à la fusion des cendres

ICS: 75.160.40; 27.190

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Foreword

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

Introduction

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

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

The test method is empirical. The ash used for the test is a homogeneous material, prepared from the fuel by ashing at 550 °C (other temperatures like 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 ISO 540:2008, DIN 51730:1998 and CEN/TS 15370-1. 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.

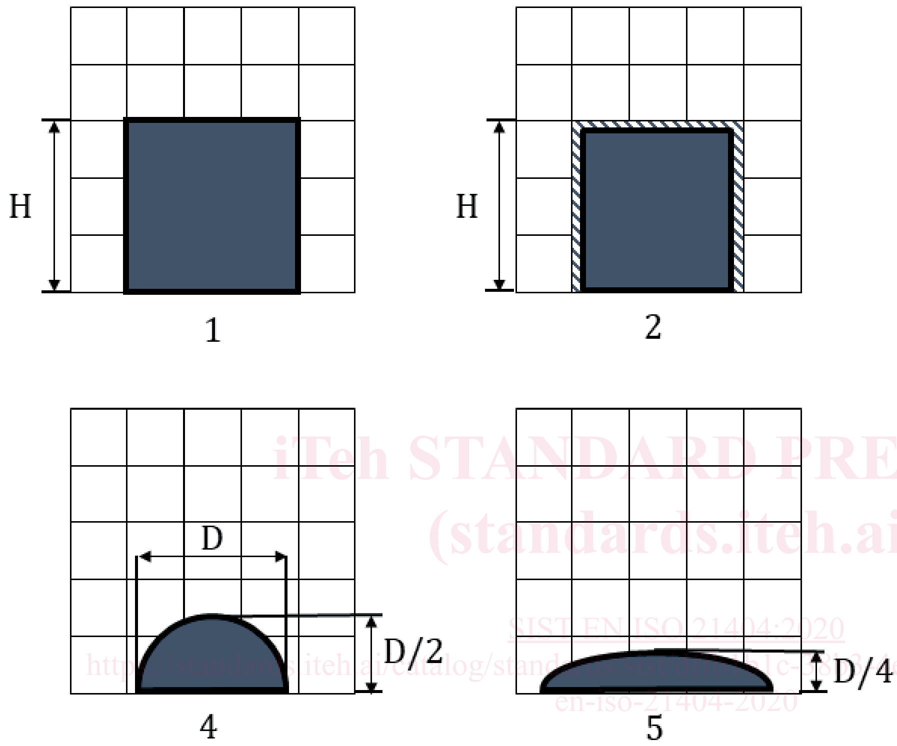
Note 1 to entry: Deformation temperature can be seen as rounding of the edges, smoothing of surfaces, expansion of the cylinder or general changing of the cylinder shape. If the test piece starts to swell or bubble without the edges being rounded, the temperature is registered as DT (since swelling and bubbling only occur when a fraction of the ash is melted).

Note 2 to entry: For computerized evaluation a shape factor change may 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.

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. Be aware that this definition is different to other standards.



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 may 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 continuously observed. The temperatures at which characteristic changes of the shape occur are recorded. The characteristic temperatures are defined in Clause 3. In informative Annex C vivid images show examples of the characteristic temperatures.

For some ashes produced at 550 °C it may 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) higher ashing temperatures as (710 ± 10) °C or (815 ± 10) °C may be used if 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. 1064 °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. 1455 °C).

Nickel is used for 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. 1554 °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 minutes. 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 SO2 and CO2 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 ISO 1171 5 – 10 air changes/min are required to eliminate reaction of SO2 and CO2 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 may cause the ash to be blown from the ashing crucible which may limit air exchange possibilities. Furthermore, a homogeneous temperature distribution in the furnace is also critical and should be balanced with the influence of air exchange. A sensitivity analysis of variation of these parameters on the result of ash melting behavior may be valuable for a certain set-up.

6.3 sample containers or bags, airtight, suitable for holding (1- 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 at which the properties of the ash are to be determined (a temperature of 1500 °C or more may be required);

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