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Hard coal and coke — Determination of ash fusibility

Houille et coke — Détermination de la fusibilité des cendres

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 540 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 540:1995), which has been technically revised.

Introduction

The method for determination of the fusibility temperatures of coal ash and coke ash described in this International Standard provides information about the fusion and melting behaviour of the composite inorganic constituents of the ash at high temperatures. The standard method is based on the “Seger Cone” method, which is well known in the ceramic industry, the use of which predates the year 1900. The conditions of the test, as well as basic studies on the influence of ash chemistry and of gas composition on ash fusibility temperatures (which have led to the standardization of the method), arose from the pioneering work of Fieldner, Hall and Field [1].

In the laboratory, the ash used for the test is a homogeneous mixture prepared from a representative sample of the coal or coke, and the determination is performed at a controlled rate of heating in either a reducing or an oxidizing atmosphere. In contrast, under industrial conditions, the complex processes of combustion and fusion involve heterogeneous mixtures of particles, heating rates (that can be several orders of magnitude greater than those used in the standard test) and variable gas composition.

During the first quarter of the 20th century, laboratory, pilot-scale and field studies were undertaken to establish that the ash fusibility test can provide a reasonable indication of the propensity of ash to form fused deposits (referred to as “clinker”) in stoker and other fuel-bed type furnaces (Nicholls and Selvig [2]). Subsequently, the test has been used as a general indicator of the tendency for ash to fuse on heating and of ash slagging propensity in pulverized coal-fired furnaces.

Hard coal and coke — Determination of ash fusibility

1 Scope

This International Standard specifies a method of determining the characteristic fusion temperatures of ash from coal and coke.

NOTE Descriptors: fossil fuels, solid fuels, ash, ashes, tests, high temperature tests, determination, and fusibility.

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 1171, *Solid mineral fuels — Determination of ash*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
deformation temperature
DT
temperature at which the first signs of rounding, due to melting, of the tip or edges of the test piece occur

NOTE Shrinkage or distortion of the test piece, or rounding of cracks and fins, are not criteria for deformation and should be ignored if the tip and edges remain sharp. However, for some solid mineral fuels, the temperature at which the test piece shrinkage begins can be of interest and should be reported as a feature noted during the determination.

3.2
sphere temperature
ST
in the case of pyramidal and truncated-cone test pieces, the temperature at which the height is equal to the width of the base, and in the case of cubical or cylindrical test pieces, the temperature at which the edges of the test pieces become completely round with the height remaining unchanged

3.3
hemisphere temperature
HT
temperature at which the test piece forms approximately a hemisphere, i.e. when the height becomes equal to half the base diameter

3.4
flow temperature
FT
temperature at which the ash melt is spread out over the supporting tile in a layer, the height of which is one-third of the height of the test piece at the hemisphere temperature

4 Principle

A test piece made from the ash is heated under standard conditions and continuously observed. The temperatures at which characteristic changes of shape occur are recorded. The characteristic temperatures are defined in Clause 3. (See also Figures 2, 3 and 4.)

Although the determination is usually performed in a reducing atmosphere, additional information can sometimes be obtained by performing a further determination in an oxidizing atmosphere. In general, the reducing atmosphere in 7.1 gives the lowest characteristic temperatures.

5 Reagents

5.1 Dextrin, 100 g/l solution.

Dissolve 10 g of dextrin in 100 ml of water.

5.2 Petroleum jelly.

5.3 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 % and a melting point of 1 064 °C.

5.4 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 % and a melting point of 1 455 °C.

5.5 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 % and a melting point of 1 554 °C.

5.6 Carbon dioxide.

5.7 Hydrogen or **carbon monoxide**.

6 Apparatus

6.1 Furnace, 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 determined (a temperature of 1 500 °C or more can be required).

NOTE Some furnaces can have a practical upper operating temperature, e.g. 1 480 or 1 540 °C, due to the type of heating elements used in their manufacture.

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 815 °C upwards.

d) It shall be capable of maintaining the required test atmosphere (see 7.1) around the test piece(s).

e) It shall provide a means of observing the change of shape of the test piece(s) during heating.

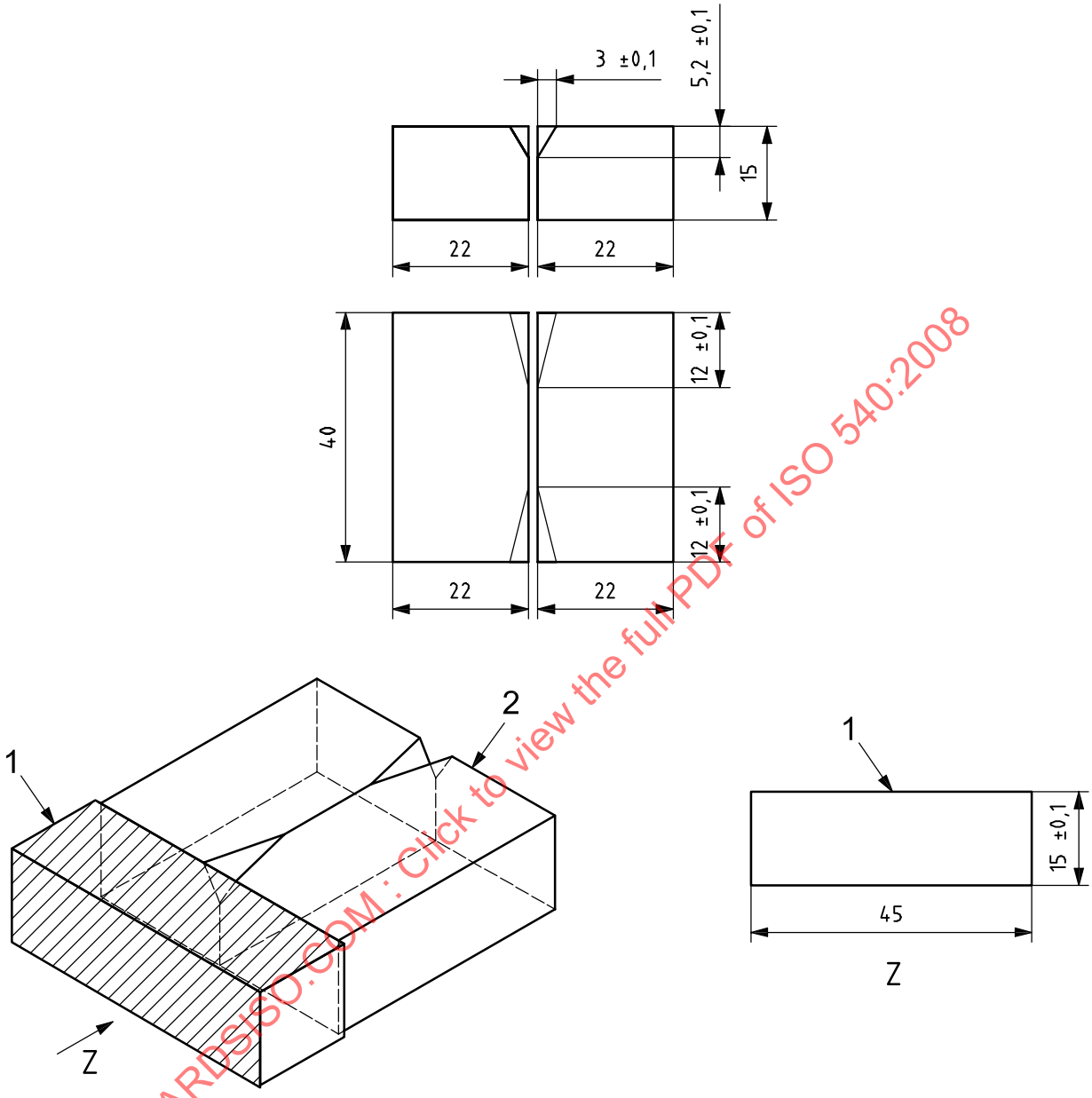
It is recommended to provide a facility for inserting, between the end window of the furnace and the optical viewing instrument, a piece of cobalt-blue or similar glass to protect the retina of the operator from radiation emitted at elevated temperatures.

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

The thermocouple is positioned so that the thermo-junction is on the longitudinal axis in the centre of the zone of uniform temperature.

6.3 Mould, of brass, stainless steel, or other suitable material, for preparing the test piece. (See example in Figure 1.)

Dimensions in millimetres



Key

1 base plate
2 mould(s)

Figure 1 — An example of a mould that is suitable for making a pyramidal specimen

6.4 Support for the test piece, of such a material that the support does not either become distorted or react with or absorb the ash during the determination. Supports of sintered alumina or fine-textured mullite are generally satisfactory, but difficulties can arise with individual ashes, in which case a non-absorbent interface such as platinum foil can be used between the original support and the test piece.

6.5 Flowmeters, two, for measuring the components of the reducing gases (see 7.1); it is not necessary to measure the flow rate when using an oxidizing gas.

If the flowmeter contains a liquid, this liquid shall be a non-volatile oil.

6.6 Agate mortar and **pestle**.

6.7 Test sieve, of aperture 0,075 mm (or less) and diameter of approximately either 100 mm or 200 mm, complete with lid and receiver.

6.8 Optical instrument, which enables the profile of the test piece to be observed throughout the determination.

The relative dimensions of the profile can be conveniently assessed by using a graticule.

Additional use of photographic equipment such as a camera or video equipment is optional but recommended.

7 Test conditions

7.1 Test atmosphere

The reducing atmosphere is obtained by introducing into the furnace one of the following mixtures of gases at a minimum linear rate of flow past the test piece of 400 mm/min, calculated at ambient room temperature; the rate is not critical, provided that it is sufficient to prevent any leakage of air into the furnace:

a) 55 volume % to 65 volume % carbon monoxide with 35 volume % to 45 volume % carbon dioxide; or

b) 45 volume % to 55 volume % hydrogen with 45 volume % to 55 volume % carbon dioxide.

NOTE 1 If a mixture of CO/CO_2 is used to produce the reducing atmosphere, ensure that the contents are totally mixed in accordance with manufacturer's instructions and that the temperature of the cylinder is maintained above the critical temperature at which CO_2 can liquefy and separate.

NOTE 2 Ashes rich in iron oxide can react with any oxygen present in the furnace, resulting in poor repeatability and reproducibility of characteristic temperatures.

An oxidizing atmosphere is obtained with air or carbon dioxide; the rate of flow is not critical.

WARNING — When using the reducing atmospheres given above, the gases emerging from the furnace contain a proportion of carbon monoxide. It is essential, therefore, to ensure that these gases are vented to the outside atmosphere, preferably by means of a hood or an efficient fan system. If hydrogen is used in the reducing atmosphere, great care shall be taken to prevent an explosion occurring, by purging the furnace with carbon dioxide both prior to the introduction of the hydrogen and after the hydrogen supply is shut off.

7.2 Shape of test piece

The test piece shall have sharp edges to facilitate observation.

The mass of the test piece shall be such as to ensure equalization of the temperature within the test body. Hence, dimensions that are too large shall be avoided.

The following shapes are acceptable:

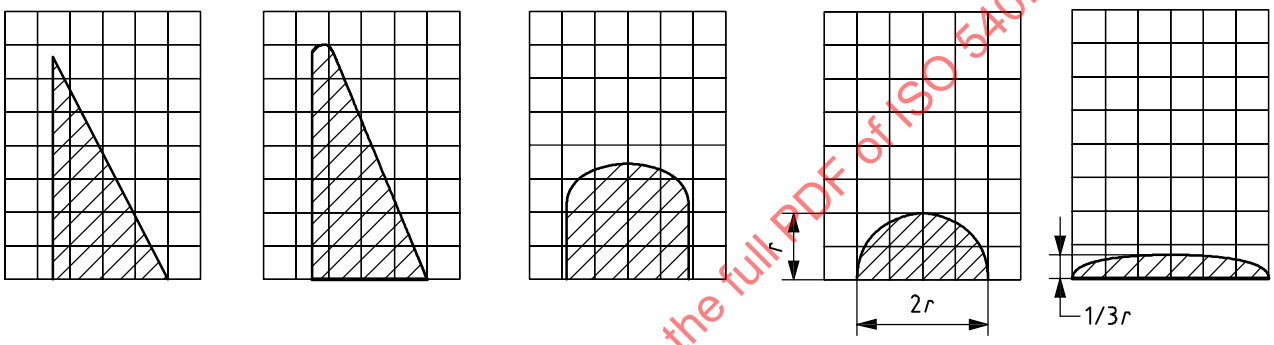
a) pyramid, the base of which is an equilateral triangle; the height shall not exceed 12 mm and shall be two to three times the length of the side of the base (see Figure 2);

b) cube of side 3 mm to 7 mm; (see Figure 3);

c) upright cylinder of height 3 mm to 9 mm and with diameter equal to the height (see Figure 3);

d) truncated cone of height 4 mm and with diameters 3 mm at the base and 1,5 mm at the top (see Figure 4).

If, when using the pyramidal test piece, the degree of bending becomes severe enough to preclude the direct observation of the dynamic state of the test piece at any time during the test, another shape should be used.

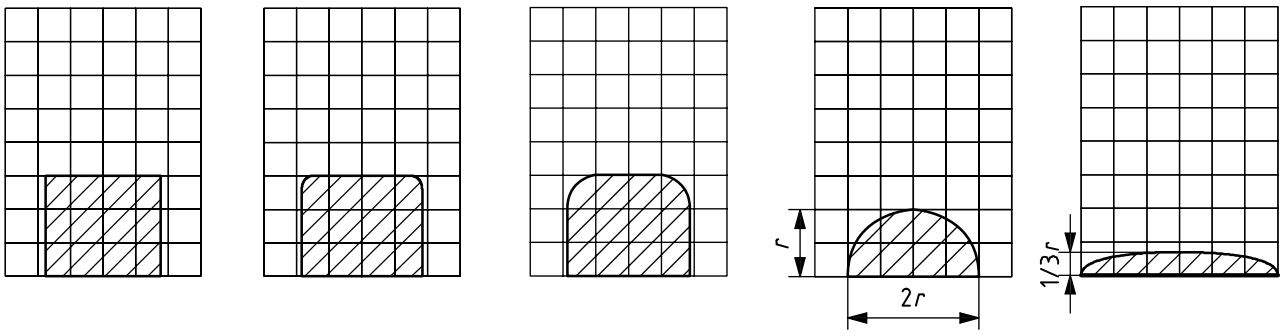


Key

1 original
2 deformation
3 sphere
4 hemisphere
5 flow

r = Radius of the hemisphere.

Figure 2 — Characteristic shapes of the pyramidal test piece



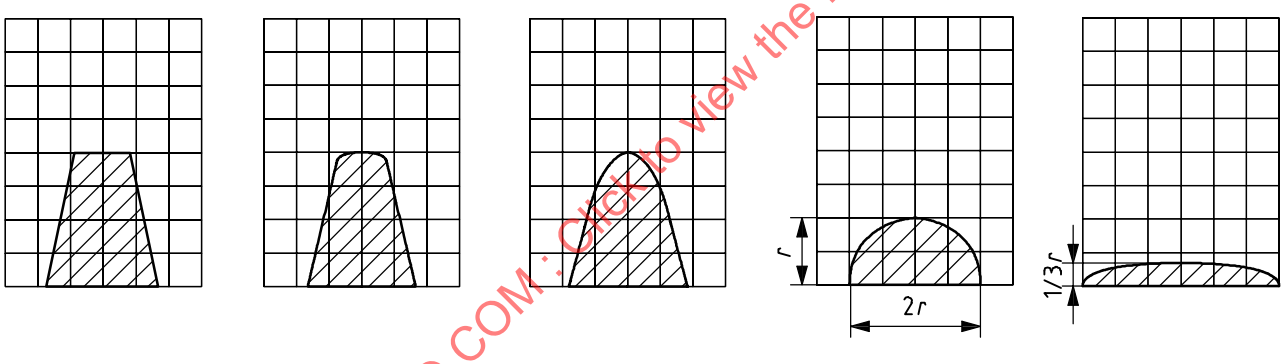
1 2 3 4 5

Key

1 original
2 deformation
3 sphere
4 hemisphere
5 flow

r = Radius of the hemisphere.

Figure 3 — Characteristic shapes of the cylindrical or cubical test piece



1 2 3 4 5

Key

1 original
2 deformation
3 sphere
4 hemisphere
5 flow

r = Radius of the hemisphere.

Figure 4 — Characteristic shapes of the truncated-cone test piece

8 Calibration check

Check the pyrometer regularly under routine test conditions by observation of the melting point of gold (5.3) and, if possible, the melting point of palladium (5.5). Test the reducing atmosphere by observing the melting point of nickel (5.4).

If the observed melting points for gold or palladium differ by more than 10 °C from the melting points given in 5.3 and 5.5, readjust or recalibrate.

NOTE 1 An alternative to the observations of the melting points of gold and palladium is to check the pyrometer using a thermocouple certified by a recognized reference laboratory or with a calibration that is traceable back to a standard reference laboratory.

If the observed melting point for nickel differs by more than 10 °C from the melting point given in 5.4, it can be due to oxidation of nickel caused by an insufficiently reducing atmosphere. Examine the apparatus for leakages, control the flow rate and the quality of the gases, and recheck the melting point of nickel.

NOTE 2 The correct melting point of nickel is not a guarantee that the composition of the reducing atmosphere is correct, as the fusibility is not likely to be affected until the deviations are considerable.

9 Preparation of the test piece

Prepare the ash according to the method specified in ISO 1171. Ensure that the incineration is complete.

Grind the ash in an agate mortar (6.6) until the maximum particle size is less than 0,075 mm. To maximize the number of particles in the peak of the pyramid, grinding to less than 0,063 mm is recommended.

Moisten a sufficient quantity of the prepared ash with demineralized water or, if necessary, with an adhesive dextrin solution (5.1), and make the mixture into a paste. To facilitate the removal of the test piece, the mould (6.3) may first be coated with a thin layer of petroleum jelly (5.2).

Fill the mould uniformly and completely with the prepared paste of ash so that the edges and tip of the specimen are sharp. Allow the test piece to visually dry before attempting to remove it from the mould. If the ash paste dries too quickly, it is acceptable to moisten it slightly with additional water or solution.

After removal, mount the specimen onto its support (6.4), using a thin layer of paste of the prepared ash to effect adhesion, taking care not to damage the tip or edges. The specimen(s) should be examined with the optical instrument (6.8) and those that do not have sharp edges and tips should be discarded.

Allow the test piece to dry, and remove any organic matter by heating the test piece slowly up to a temperature of about 815 °C. If preferred, this preliminary heating may be performed in the furnace (6.1) used for the test.

10 Procedure

Transfer the test piece on its support (6.4) to the furnace (6.1), and adjust the composition and flow rate of the atmosphere, taking into account the warning in 7.1. To minimize the risk of explosion when a reducing atmosphere is used, purge the furnace with carbon dioxide before admitting hydrogen.

The specimen shall be positioned on the tile so that, when inserted into the furnace, its vertical face is parallel to the longitudinal axis of the inspection opening. Further, the specimen shall be placed as close as possible to the end of the thermocouple sheath. Ensure that the thermocouple is in the correct position.

Raise the temperature of the furnace to a point below the expected deformation temperature, so that the temperature interval between the point and the expected temperature exceeds 150 °C.

If using a camera or video camera, focus on the specimen so that all parts of the specimen(s) are in sharp focus for the duration of the test. Adjust the focus if required during the test.

After a period of 10 min, raise the temperature at a uniform rate within the range of 3 °C/min to 7 °C/min; for small test pieces, a rate up to 10 °C/min is satisfactory. Commence observing or photographing the specimen and recording shapes at intervals of temperature change not greater than 20 °C until the maximum temperature of the furnace is reached or the flow temperature of the specimen has been attained.

Record the temperatures at which the characteristic changes of shape occur. If the characteristic shapes for the specimen lie between two successive frames using photographic or video techniques, the temperature should be recorded as the average temperature of the two consecutive frames. With some ashes, difficulties can be encountered owing to such effects as blistering, distortion, shrinkage, swelling, non-wetting of the support (caused by high surface tension) and bursting of internal gas bubbles. In such cases, it is desirable to record these phenomena and possibly repeat the experiment using a different type of support.

Where photographic equipment is used, a standard grid shall be placed over the viewing screen to assist in interpretation of characteristic shapes.

If a hydrogen/carbon dioxide atmosphere is used, turn off the hydrogen and continue the flow of carbon dioxide for at least 30 s, and then turn off the carbon dioxide flow.

Remove the specimen and tile from the furnace, allow cooling, and examine for evidence of chemical reaction between the ash and tile, which can cause difficulties in obtaining the characteristic shapes.

Process any film and examine the negatives or transparencies at magnification equivalent to a minimum of 10 times the original specimen height. Where films are used, results shall be based on the examination of negatives or transparencies, not of prints.

11 Precision of the method

11.1 Repeatability limit

The results of independent duplicate determinations, performed on the same day in the same laboratory by the same operator using the same apparatus on the same preparation of ash, shall not differ by more than the values given in Table 1.

11.2 Reproducibility limit

The means of the results of duplicate determinations, performed in each of two different laboratories on representative portions taken from the same sample after the last stage of preparation as specified in ISO 1171, shall not differ by more than the values given in Table 1.

Table 1 — Maximum acceptable differences between results

Fusibility of ash	All atmospheres	
	Repeatability °C	Reproducibility °C
Deformation temperature, DT [a]	30	80
Sphere temperature, ST	30	60
Hemisphere temperature, HT	30	60
Flow temperature, FT	30	80

[a] If the sphere temperature is not reached, the precision on deformation temperature might not be achievable.

Although the use of different profiles is permitted in this Standard, the precision data reported in Table 1 have been calculated from results obtained using the pyramid profile only and no precision data are reported for other profiles.