International Standard 540

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Solid mineral fuels — Determination of fusibility of ash — High-temperature tube method

Combustibles minéraux solides — Détermination de la fusibilité des cendres — Méthode du tube à haute température

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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International Standard ISO 540 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*.

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Australia	Iran	South Africa, Rep. of
Austria	Italy	Spain
Canada	Japan	Sweden
Chile	Netherlands	Switzerland
Czechoslovakia	New Zealand	Turkey
Denmark	Philippines	United Kingdom
France	Poland	USSR
Germany, F. R.	Portugal	Yugoslavia
India	Romania	

The member body of the following country had expressed disapproval of the document on technical grounds :

Belgium

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Solid mineral fuels — Determination of fusibility of ash — High-temperature tube method

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1 Scope and field of application

This International Standard specifies a method of determining the characteristic fusion temperatures of ash from all solid mineral fuels.

2 Reference

ISO 1171, *Solid mineral fuels — Determination of ash.*

3 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 the following :

Deformation temperature (symbol A)

The temperature at which the first signs of rounding of the tip or edges of the test piece occur (see note 1).

Hemisphere temperature (symbol B)

The temperature at which the test piece forms approximately a hemisphere, i.e. when the height becomes equal to half the base diameter as visually observed.

Flow temperature (symbol C)

The temperature at which the ash 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.

Although the determination is usually carried out in a reducing atmosphere, additional information can sometimes be obtained by carrying out a further determination in an oxidizing atmosphere (see note 2).

NOTES

1 Shrinkage or distortion of the test piece should be ignored if the tip and edges of the test piece remain sharp.

2 In general, the reducing atmospheres recommended give the lowest characteristic temperatures.

4 Test conditions

4.1 Shape of test piece

The test piece shall have sharp edges to facilitate observation and shall not bend during the determination.

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

The following shapes are acceptable :

a) pyramid, the base of which is an equilateral triangle and the height between two and three times the side of the base, but not exceeding 19 mm;

b) cube of side 3 to 7 mm;

c) right cylinder of height 3 to 9 mm and diameter 3 to 9 mm, the height being equal to the diameter.

4.2 Test atmosphere

The reducing atmosphere (see the warning) is conveniently 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 temperature (see the note) :

a) 60 ± 5 % (*V*/*V*) carbon monoxide with 40 ± 5 % (*V*/*V*) carbon dioxide

b) 50 ± 5 % (*V*/*V*) hydrogen with 50 ± 5 % (*V*/*V*) carbon dioxide

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

WARNING — When using the reducing atmospheres given above, the gases emerging from the furnace will 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 against explosion occurring.

NOTE — The rate of gas flow is not critical, provided that it is sufficient to prevent any leaking of air into the furnace. The rate of 400 mm/min is a minimum which has been determined experimentally.

5 Apparatus

5.1 Furnace, preferably 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 1 500 °C or higher may be required for many ashes);

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

c) it shall provide means of heating the test piece at a uniform rate within the range of 3 to 7 °C/min;

d) it shall be possible to maintain around the test piece an atmosphere of composition within the desired limits;

e) it shall provide means of observing the change of shape of the test piece during heating.

5.2 Pyrometer, comprising a platinum-rhodium/platinum thermocouple, the accuracy of which shall be checked from time to time (see the notes in 5.5).

5.3 Mould, for preparing the test piece.

5.4 Support, for the test piece.

The support shall be of such a material that it neither becomes distorted, nor reacts with nor absorbs the ash, during the determination.

While supports of sintered alumina, fine-textured mullite or platinum on a refractory support are generally satisfactory, difficulties may arise with individual ashes and only experience can show which support is the best for use in such circumstances.

5.5 *Optical instrument*

An instrument which enables the profile of the test piece to be observed throughout the determination. The relative dimensions of the profile can conveniently be assessed by the use of a graticule (see note 1).

NOTES

1 With the largest size of pyramidal test piece an optical pyrometer may be used, and in this case the optical instrument may be dispensed with.

2 The accuracy of the pyrometer may conveniently be checked by observing the melting points of gold (m.p. 1 063 °C), nickel (m.p. 1 452 °C) and palladium (m.p. 1 552 °C). It should be noted that if nickel is used, a 100 % hydrogen atmosphere is required. Gold and palladium may be melted in the same atmosphere as that used for the ash fusion test.

6 Preparation of the test piece

Prepare the ash according to the method specified in ISO 1171. The operator shall ensure that the incineration is complete.

Grind the ash in an agate mortar until the maximum particle size is less than 76 µm.

Moisten a sufficient quantity of the prepared ash with water, or if necessary with an adhesive solution (for example dextrin, starch or gum arabic), make into a paste and press into the mould (5.3). To facilitate the removal of the test piece, the mould may first be coated with a thin layer of petroleum jelly.

Allow the test piece to dry, mount it on its support (5.4), and remove any organic matter present by heating it slowly in air up to a temperature of about 815 °C.

NOTE — If preferred, the preliminary heating of the test piece in air may be carried out in the furnace used for the test.

7 Procedure

Transfer the test piece on its support (5.4) to the furnace (5.1), previously heated to a temperature of 815 °C, and adjust the composition and flow rate of the atmosphere. Raise the temperature at a uniform rate within the range of 3 to 7 °C/min (see the note) and record the temperatures at which the characteristic changes of shape occur. With some ashes difficulties may be encountered owing to such effects as blistering, distortion, shrinkage, swelling, non-wetting of the support and bursting of internal gas bubbles, and in such cases it is desirable to record these phenomena and possibly repeat the experiment using a different type of support.

NOTE — The slower rate of heating is preferable for the larger test pieces; with the smaller test pieces, a rate of temperature rise up to 10 °C/min may sometimes be satisfactory.

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8 Precision of the method

Fusibility of ash	Maximum acceptable differences between results obtained	
	In the same laboratory (Repeatability)	In different laboratories (Reproducibility)
Deformation temperature (A)	30 °C	(see 8.2)
Hemisphere temperature (B)	30 °C	
Flow temperature (C)	50 °C	

8.1 Repeatability

The results of duplicate determinations, carried out at different times 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 above amounts.

8.2 Reproducibility

No values for reproducibility can be quoted for determinations carried out in different laboratories since insufficient evidence is available on which to base such values.[1)]

9 Test report

The test report shall contain the following information :

a) an identification of the product tested;

b) the reference of the method used;

c) the deformation temperature (symbol A), rounded to the nearest 10 °C;

d) the hemisphere temperature (symbol B), rounded to the nearest 10 °C;

e) the flow temperature (symbol C), rounded to the nearest 10 °C;

f) the size and shape of the test piece;

g) the composition of the atmosphere;

h) the nature of the support;

j) any unusual features noted during the determination;

k) any operation not included in this International Standard, or regarded as optional.

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1) Further work in the United Kingdom makes it possible to recommend the following figures which, if adopted, would necessitate an appropriate modification in 8.2 :

Reproducibility : A 80 °C; B 50 °C; C 80 °C.

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