INTERNATIONAL STANDARD

ISO 540

Third edition
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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

Reference number
ISO 540:1995(E)

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.

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.

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

This third edition cancels and replaces the second edition (ISO 540:1981), which has been technically revised.

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

1 Scope

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

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1171:1981, *Solid mineral fuels — Determination of ash.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 deformation temperature (abbreviation: DT): The temperature at which the first signs of rounding, due to melting, of the tip or edges of the test piece occur.

NOTE 1 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 could be of interest and should be reported as a feature noted during the determination.

3.2 sphere temperature (abbreviation: 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 (abbreviation: HT): The 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 (abbreviation: FT): 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.

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 1 to 3).

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. In general, the reducing atmospheres recommended give 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.

NOTE 2 The hydrogen or carbon monoxide shall be sufficiently free from oxygen (see clause 8).

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 to be determined (a temperature of 1 500 °C or more may be required);

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 means of observing the change of shape of the test piece(s) during heating.

NOTE 3 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 glass or similar to protect the retina of the operator from radiation emitted at elevated temperatures.

6.2 Pyrometer, comprising a platinum/platinum-rhodium thermocouple.

6.3 Mould, of brass, stainless steel or other suitable material, for preparing the test piece.

6.4 Support for the test piece, of such a material that it becomes neither distorted, nor reacts with nor absorbs the ash during the determination. Supports of sintered alumina or fine-textured mullite are generally satisfactory, but difficulties may arise with individual ashes, in which case a non-absorbent interface such as platinum foil may 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 oxidizing gas.

NOTE 4 If the flowmeter contains a liquid, this shall be a non-volatile oil.

6.6 Agate mortar and pestle.

6.7 Test sieve, of aperture 0,075 mm and diameter 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 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 temperature; the rate is not critical, provided that it is sufficient to prevent any leakage of air into the furnace.

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.

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 will contain a proportion of carbon monoxide; it is essential, therefore, to ensure that these gases are vented to the outside atmos-

phere, 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 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:

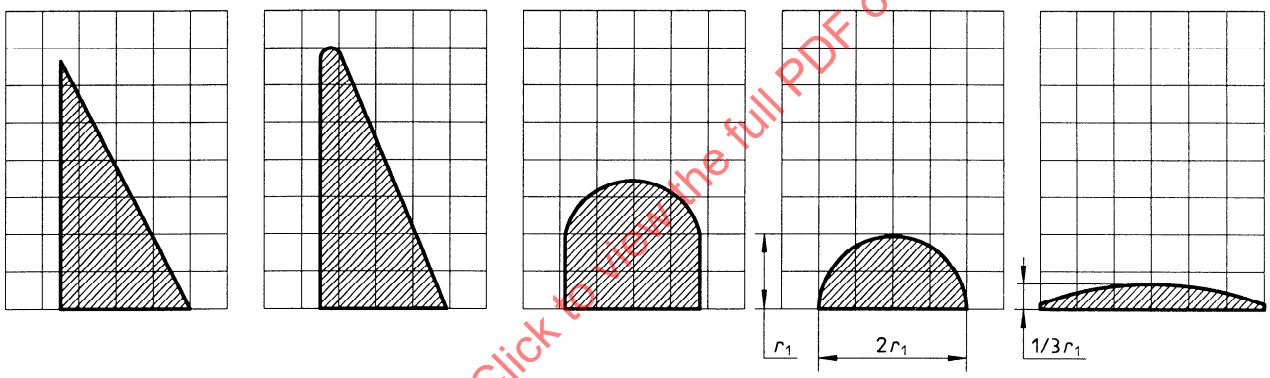
a) pyramid, the base of which is an equilateral triangle; the height shall not exceed 19 mm and shall be two to three times the length of the side of the base (see figure 1);

b) cube of side 3 mm to 7 mm (see figure 2);

c) upright cylinder of height 3 mm to 9 mm and with diameter equal to the height (see figure 2);

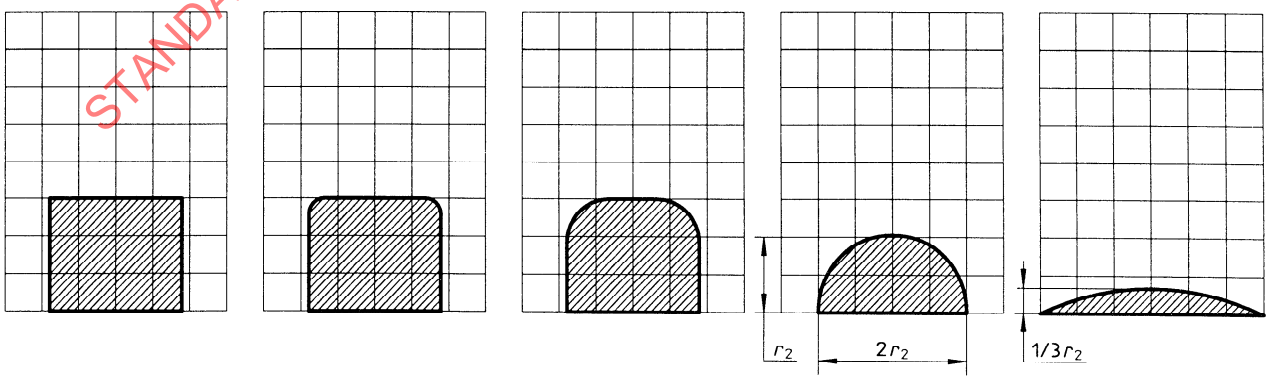
d) truncated cone of height 4 mm and with diameters 3 mm at the base and 1,5 mm at the top (see figure 3).

NOTE 5 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.



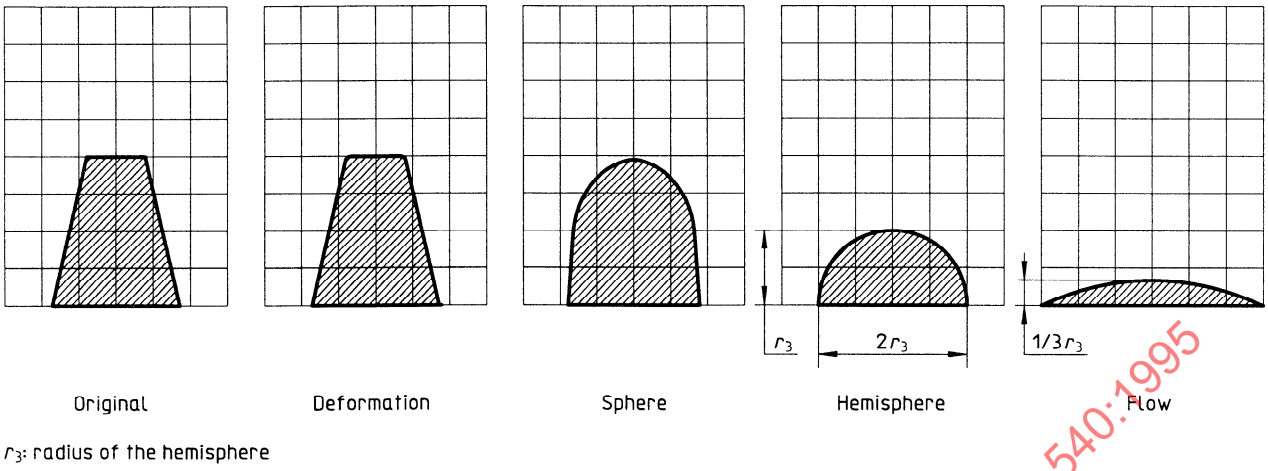
r_1: radius of the hemisphere

Figure 1 — Characteristic shapes of the pyramidal test piece



r_2: radius of the hemisphere

Figure 2 — Characteristic shapes of the cylindrical or cubical test piece



r_3: radius of the hemisphere

Figure 3 — Characteristic shapes of the truncated-cone test piece

8 Calibration check

Check the pyrometer regularly under routine test conditions by observations 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 6 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 which 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 give 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 7 The correct melting point of nickel is not a guarantee that the composition of the reducing atmosphere is correct, as deviations should be considerable before the fusibility is affected.

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.

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

Allow the test piece to dry, mount it on its support (6.4), and remove any organic matter by heating it slowly in air up to a temperature of about 815 °C. If preferred, this preliminary heating may be carried out 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.

Raise the temperature to a point below the expected deformation temperature, so that the temperature interval between the point and the expected deformation temperature exceeds 150 °C. Then proceed to raise the temperature at a uniform rate within the range of 3 °C/min to 7 °C/ min; for small test pieces, which are common in heating microscopes with tube diameters of about 20 mm, a rate up to 10 °C/min will be satisfactory.

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 (caused by high surface tension) and bursting of internal gas bubbles, and in such cases it is desirable to record these phenomena and possibly