International Standard

ISO 1014

Coke — Determination of true relative density, apparent relative density and porosity

Coke — Détermination de la densité relative vraie, de la densité relative apparente et de la porosité

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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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1014 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

ISO 1014 was first published in 1975. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

Coke — Determination of true relative density, apparent relative density and porosity

1 Scope and field of application

This International Standard specifies methods of

— determining the true relative density of coke, crushed to 212 µm, in relation to water[1]

— determining the apparent relative density of coke, i.e. the ratio of the mass of a volume of dry coke to the mass of an equal volume of water

— calculating the porosity of the coke.

2 Reference

ISO 687, *Coke — Determination of moisture in the analysis sample.*

3 True relative density

3.1 Principle

The mass of water displaced by a known mass of dry coke, ground to pass a sieve of nominal size of openings 212 µm, is determined using a pyknometer. Air is displaced by boiling during the determination. Air-free distilled water[1] is specified, as the use of organic liquids, or of water with wetting agents added, has been found to give variable results. Thermostatic control of the temperature is essential since a difference of 1 °C can cause an error of about 0,012 in the result.

3.2 Apparatus

3.2.1 Pyknometer, 50 ml capacity

3.2.2 Water bath, with stirrer, thermostatically controlled to maintain a desired temperature θ °C to within ±1 °C.

3.2.3 Two wash bottles, each containing about 50 ml of air-free distilled water. One wash bottle is kept hot (80 to 90 °C) and the other is left in the water bath (3.2.2).

3.2.4 Reflux air condenser: a glass tube about 1 m long, of the same external diameter as the neck of the pyknometer (3.2.1) with a short length of rubber tubing for attaching it to the latter.

3.2.5 Glycerol bath: a suitable vessel in which sufficient glycerol can be heated for the lower two-thirds of the pyknometer (3.2.1) to be immersed.

3.2.6 Balance, accurate to 0,1 mg.

3.3 Preparation of sample

The coke used for the determination is the analysis sample, ground to pass a sieve of nominal size of openings 212 µm. Before commencing the determination, mix the sample thoroughly for at least 1 min, preferably by mechanical means.

3.4 Procedure

Clean the pyknometer (3.2.1) using a mixture of potassium dichromate and sulfuric acid, rinse thoroughly and fill with air-free distilled water. Insert the stopper and immerse the pyknometer up to the neck in the water bath (3.2.2) at θ °C for 1 h. The value of θ should be about 5 °C above the ambient temperature. At the end of 1 h, remove the blob of water from the top of the stopper with a piece of filter paper, remove the pyknometer from the water bath, rapidly cool under cold running water, dry, allow to stand beside the balance (3.2.6) for 30 min and weigh with an accuracy of ±0,1 mg. Empty the pyknometer and dry the neck.

Dry a portion of the coke sample for 1 h at 105 to 110 °C (see the note), cool, then weigh about 5 g of the dry coke to the nearest 0,1 mg and transfer it completely to the pyknometer. Wash down any coke adhering to the neck or side of the pyknometer with air-free distilled water, making up the volume to about 25 ml. Attach the air condenser (3.2.4) to the neck of the pyknometer with the rubber tubing and immerse the pyknometer in the glycerol bath (3.2.5). Heat the bath so that the water in the pyknometer begins to boil vigorously. Wash down any scum of coke with a few millilitres of the hot, air-free distilled water. After boiling for 30 min, remove the pyknometer from the glycerol bath, detach the air condenser and allow the pyknometer to cool. Fill the pyknometer with air-free distilled water at θ °C, insert the stopper and immerse the pyknometer up to the neck in the water bath at θ °C for 1 h, compensating for any contraction of the liquid by the addition of air-free distilled water and ensuring that air bubbles are not trapped, either below the stopper or in the capillary. At the end of 1 h, remove the blob of water from the top of the stopper, remove the pyknometer from the water bath, cool, dry, allow to stand beside the balance for 30 min and weigh as before.

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1) It should be noted that "true relative density" varies according to the displacement liquid used.

NOTE — Some reactive cokes are hygroscopic and with such materials it is permissible to omit the drying procedure; in this case, a moisture determination is carried out at the same time on a separate portion of the analysis sample (see ISO 687) and the mass of coke taken is corrected accordingly.

3.5 Expression of results

3.5.1 Calculation

The true relative density of the coke, d, is given by the equation

$$d = \frac{m_1}{m_1 + m_2 - m_3}$$

where

m_1 is the mass, in grams, of dry coke;

m_2 is the mass, in grams, of the pyknometer filled with water;

m_3 is the mass, in grams, of the pyknometer and coke, filled with water.

The result, preferably the mean of duplicate determinations (see 3.5.2), shall be reported to the nearest 0,01.

3.5.2 Precision

3.5.2.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator using the same apparatus on representative portions taken from the same analysis sample, shall not differ by more than 0,03.

3.5.2.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories on representative portions taken from the same analysis sample, shall not differ by more than 0,05.

4 Apparent relative density

4.1 General

Experimental work has shown that the difficulties in the determination of the apparent relative density of coke, due to water draining out of large pores after immersion in water (to determine the amount of water which has entered the porous structure), may be overcome by limiting this drainage period to 10 s. A simple reproducible method is thus obtained which gives results agreeing with more complex methods, such as filling the external pores with gelatine gel.

4.2 Principle

The volume of a large amount of coke is determined by displacement in water; dividing the mass of the dried coke by the mass of an equal volume of water gives the apparent relative density.

4.3 Apparatus

4.3.1 Cage, 0,03 m³ capacity, made of galvanized iron wire of about 12 mm mesh, fitted with a lid of the same material and a fastening device.

4.3.2 Tank, approximately 0,25 m³ capacity, deep enough to immerse the cage (4.3.1) completely and fitted with a tap for emptying.

NOTE — The tank should contain sufficient water (about 250 litres) for the increase in temperature of the water after the immersion of the hot coke not to exceed 20 °C.

4.3.3 Weighing machine, capable of weighing, to within 1 g, a mass not greater than 2 kg. The machine shall be supported firmly on a wooden support above the tank (4.3.2). A wire stirrup passed over the beam of the scale shall pass through a hole in the wooden support and end in two hooks to fit on the sides of the cage (4.3.1). Alternatively, a suitable dial machine may be used.

4.3.4 Drainage tray, of galvanized iron sheet, slightly larger than the base of the cage (4.3.1) and 13 mm deep.

4.3.5 Platform scales, maximum capacity 25 kg, accurate to 25 g.

4.3.6 Drying oven, large enough to accommodate the cage (4.3.1).

4.4 Sample

The sample shall be representative of the coke[1] and more than sufficient in volume to carry out the determination in duplicate.

4.5 Procedure

Weigh the dry empty cage (4.3.1) on the platform scales (4.3.5). Balance the weighing machine (4.3.3) with its stirrup, suspend the cage from the stirrup, immerse completely in water in the tank (4.3.2) and weigh. Remove the cage, allow it to drain for 10 s, place it on the drainage tray (4.3.4) and re-weigh on the platform scales. Fill the cage with coke and dry in the oven (4.3.6) until constant in mass (see note 1). Weigh the hot coke and cage on the platform scales. Suspend the full cage from the stirrup and immerse completely in water (see note 2). Agitate the cage after 1 min to remove air bubbles. After a further 2 min, record the mass of the cage immersed in water, remove the cage from the tank and allow to drain for 10 s (see note 3). At the end of this time place the cage on the drainage tray and re-weigh on the platform scales.

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1) Material passing a 25 mm sieve should be removed.

NOTES

1 The time for drying 0,03 m³ of coke of the size concerned should be determined by separate tests. Overnight drying at 105 °C is usually most convenient but the time can be shortened by drying at 200 °C.

2 The coke should be immersed in the tank whilst still hot to facilitate displacement of air from the surface. The final weighing after drying serves as a check to confirm complete dryness.

3 Certain cokes, for example those which contain a large number of small pores, may require a longer drainage period and for such cokes a drainage period of 30 s is advised.

4.6 Expression of results

4.6.1 Calculation

The apparent relative density of the coke, d_A, is given by the equation

$$d_A = \frac{m_7 - m_4}{(m_9 - m_6) - (m_8 - m_5)}$$

where

m_4 is the mass, in grams, of the dry, empty cage in air;

m_5 is the mass, in grams, of the empty cage in water;

m_6 is the mass, in grams, of the empty cage after draining plus drainage tray;

m_7 is the mass, in grams, of the cage plus dry coke in air;

m_8 is the mass, in grams, of the cage plus coke in water;

m_9 is the mass, in grams, of the cage plus coke after draining plus drainage tray.

The result, preferably the mean of duplicate determinations (see 4.6.2), shall be reported to the nearest 0,01.

4.6.2 Precision

4.6.2.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus on representative portions from the same bulk sample, shall not differ by more than 0,02.

4.6.2.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories on representative portions taken from the same bulk sample, shall not differ by more than 0,03.

5 Porosity

5.1 Principle

The porosity is calculated from the true relative density (see clause 3) and the apparent relative density (see clause 4) of the coke.

5.2 Expression of results

The porosity of the coke, expressed as a percentage by volume, is given by the formula

$$\frac{d - d_A}{d} \times 100$$

where

d is the true relative density of the coke;

d_A is the apparent relative density of the coke.

The result shall be reported to the nearest 1 %.

6 Test report

The test report shall include the following information:

a) identification of the product tested;

b) the reference of the method used;

c) the parameter, the results and the method of expression used;

d) any unusual features noted during the determination;

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