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 institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 1014 and found it technically suitable for transformation. International Standard ISO 1014 therefore replaces ISO Recommendation R 1014-1969 to which it is technically identical.

ISO Recommendation R 1014 was approved by the Member Bodies of the following countries :

Australia	Germany	Portugal
Austria	Greece	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Canada	Israel	Switzerland
Chile	Japan	Turkey
Czechoslovakia	Korea, Rep. of	United Kingdom
Denmark	Netherlands	U.S.A.
Egypt, Arab Rep. of	New Zealand	U.S.S.R.
France	Poland	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1014 into an International Standard.

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 0,2 mm, in relation to water;

— 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 0,2 mm sieve, is determined using a density bottle. Air is displaced by boiling during the determination. Air-free distilled water 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 Density bottle, 50 ml capacity.

3.2.2 Water bath, with stirrer, thermostatically controlled to maintain a desired temperature t °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 (see 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 density bottle, 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 density bottle 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 0,2 mm aperture. Before commencing the determination, mix the sample thoroughly for at least 1 min, preferably by mechanical means.

3.4 Procedure

Clean the density bottle using a mixture of potassium dichromate and sulphuric acid, rinse thoroughly and fill with air-free distilled water. Insert the stopper and immerse the bottle up to the neck in the water bath at t °C for 1 h. The value of t 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 bottle from the water bath, rapidly cool under cold running water, dry, allow to stand beside the balance for 30 min and weigh with an accuracy of ± 0,1 mg. Empty the bottle and dry the neck.

Dry a portion of the coke sample for 1 h at 105 to 110 °C (see note below), cool, then weigh about 5 g of the dry coke to the nearest 0,1 mg and transfer it completely to the density bottle. Wash down any coke adhering to the neck or side of the bottle with air-free distilled water, making up the volume to about 25 ml. Attach the air condenser to the neck of the bottle with the rubber turbing and immerse the bottle in the glycerol bath. Heat the bath so that the water in the bottle 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 bottle from the glycerol bath, detach the air condenser and allow the bottle to cool. Fill the bottle with air-free distilled water at t °C, insert the stopper and immerse the bottle up to the neck in the water bath at t °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 bottle from the water bath, cool, dry, allow to stand beside the balance for 30 min and weigh as before.

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

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

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

where

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

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

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

The result, preferably the mean of duplicate determinations (see 3.6), shall be reported to the nearest 0,01.

3.6 Precision of the method

True relative density	Maximum acceptable difference between results	
	Repeatability	Reproducibility
	0,03	0,05

3.6.1 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 the above value.

3.6.2 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 the above value.

4 APPARENT RELATIVE DENSITY

4.1 Introduction

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 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. 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. 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 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.

4.4 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 on the platform scales. Balance the weighing machine with its stirrup, suspend the cage from the stirrup, immerse completely in water and weigh. Remove the cage, allow it to drain for 10 s, place it on the drainage tray and re-weigh on the platform scales. Fill the cage with coke and dry in the oven 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.

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.

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

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

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

$$d_A = \frac{M_0 - m_0}{(M_2 - m_2) - (M_1 - m_1)}$$

where

m_0 is the mass, in grams, of the dry, empty cage in air;

m_1 is the mass, in grams, of the empty cage in water;

m_2 is the mass, in grams, of the empty cage after draining plus drainage tray;

M_0 is the mass, in grams, of the cage plus dry coke in air;

M_1 is the mass, in grams, of the cage plus coke in water;

M_2 is the mass, in grams, of the cage plus coke after draining plus drainage tray.

The result, preferably the mean of duplicate determinations (see 4.7) shall be reported to the nearest 0,01.

4.7 Precision of the method

Apparent relative density	Maximum acceptable differences between results	
	Repeatability	Reproducibility
	0,02	0,03

4.7.1 *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 the above value.

4.7.2 *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 the above value.

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, 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 particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

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

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