

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1014

DETERMINATION OF THE TRUE RELATIVE DENSITY,
THE APPARENT RELATIVE DENSITY
AND THE POROSITY OF COKE

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1014, *Determination of the true relative density, the apparent relative density and the porosity of coke*, was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led, in 1966, to the adoption of two Draft ISO Recommendations.

In April 1967, these two Draft ISO Recommendations (Nos. 1147 and 1148) were circulated to all the ISO Member Bodies for enquiry. They were approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Drafts.

The two Drafts were combined by the ISO/TC 27 Secretariat into a single document and submitted by correspondence to the ISO Council, which decided, in March 1969, to accept it as an ISO RECOMMENDATION.

DETERMINATION OF THE TRUE RELATIVE DENSITY,
THE APPARENT RELATIVE DENSITY
AND THE POROSITY OF COKE

1. TRUE RELATIVE DENSITY

1.1 Scope

Section 1 of this ISO Recommendation describes a method for the determination of the true relative density of coke, crushed to 0.2 mm, in relation to water.

1.2 Principle

The mass of water displaced by a known mass of dry coke, ground to pass a 0.2 mm sieve, is determined in 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.

1.3 Apparatus

1.3.1 *Density bottle*, 50 ml capacity.

1.3.2 *Water bath*, with stirrer, thermostatically controlled to maintain a desired temperature t °C to within ± 1 °C.

1.3.3 *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 clause 1.3.2).

1.3.4 *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.

1.3.5 *Glycerol bath* : a suitable vessel in which sufficient glycerol can be heated for the lower two-thirds of the density bottle to be immersed.

1.3.6 *Balance*, accurate to 0.1 mg.

1.4 Procedure

Clean the density bottle with potassium dichromate-sulphuric acid mixture, 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 $t^{\circ}\text{C}$ for 1 hour. The value of t should be about 5°C above the ambient temperature. At the end of 1 hour, 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 minutes and weigh with an accuracy of ± 0.1 mg. Empty the bottle and dry the neck.

Mix the analysis sample of coke, ground to pass a 0.2 mm sieve, thoroughly for at least 1 minute, preferably by mechanical means.

Dry a portion of the coke sample for 1 hour 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 tubing 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 minutes, 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 $t^{\circ}\text{C}$, insert the stopper and immerse the bottle up to the neck in the water bath at $t^{\circ}\text{C}$ for 1 hour, compensating for any contraction of the liquid by 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 hour, 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 minutes and weigh as before.

NOTE. — Some reactive coke 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 Recommendation R 687, *Determination of moisture in the analysis sample of coke*) and the mass of coke taken is corrected accordingly.

1.5 Expression of results

The true relative density of the coke is calculated from the following formula :

$$\frac{m}{m + m_1 - m_2}$$

where

m is the mass, in grammes, of dry coke;

m_1 is the mass, in grammes, of the density bottle filled with water;

m_2 is the mass, in grammes, of the density bottle and coke, filled with water.

The result, preferably the mean of duplicate determinations (see clause 1.6), should be reported to the nearest 0.01.

1.6 Precision of the method

True relative density	Maximum acceptable difference between results obtained	
	in the same laboratory (Repeatability)	in different laboratories (Reproducibility)
	0.03	0.05

1.6.1 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, should not differ by more than the above value.

1.6.2 Reproducibility

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

2. APPARENT RELATIVE DENSITY

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 seconds. A simple reproducible method is thus obtained which gives results agreeing with more complex methods, such as filling the external pores with gelatine gel.

2.1 Scope

Section 2 of this ISO Recommendation describes a method of 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.

2.2 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.

2.3 Sample

The sample should be representative of the coke* and more than sufficient in volume to carry out the determination in duplicate.

2.4 Apparatus

2.4.1 *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.

2.4.2 *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) so that the increase in temperature of the water after the immersion of the hot coke does not exceed 20 °C.

2.4.3 *Weighing machine*, capable of weighing, to within 1 g, a mass not greater than 2 kg. The machine is supported firmly on a wooden support above the tank. A wire stirrup passed over the beam of the scale passes through a hole in the wooden support and ends in two hooks to fit on the sides of the cage. Alternatively, a suitable dial machine may be used.

2.4.4 *Drainage tray*, of galvanized iron sheet slightly larger than the base of the cage and 13 mm deep.

2.4.5 *Platform scales*, maximum capacity 25 kg, accurate to 25 g.

2.4.6 *Drying oven*, large enough to accommodate the cage.

2.5 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 seconds, 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 minute to remove air bubbles. After a further 2 minutes, record the mass of the cage immersed in water, remove the cage from the tank and allow to drain for 10 seconds (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.
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 seconds is advised.

* Material passing a 25 mm sieve should be removed.

2.6 Expression of results

The apparent relative density of the coke is calculated from the following formula :

$$\frac{M - m}{(M_2 - m_2) - (M_1 - m_1)}$$

where

- m is the mass, in grammes, of the dry, empty cage in air;
- m_1 is the mass, in grammes, of the empty cage in water;
- m_2 is the mass, in grammes, of the empty cage after draining plus drainage tray;
- M is the mass, in grammes, of the cage plus dry coke in air;
- M_1 is the mass, in grammes, of the cage plus coke in water;
- M_2 is the mass, in grammes, of the cage plus coke after draining plus drainage tray.

The result, preferably the mean of duplicate determinations (see clause 2.7) should be reported to the nearest 0.01.

2.7 Precision of the method

Apparent relative density	Maximum acceptable differences between results obtained	
	in the same laboratory (Repeatability)	in different laboratories (Reproducibility)
	0.02	0.03

2.7.1 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, should not differ by more than the above amount.

2.7.2 Reproducibility

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

3. POROSITY

3.1 Scope

Section 3 of this ISO Recommendation describes a method of calculating the porosity of coke.

3.2 Principle

The porosity is calculated from the true relative density (see section 1) and the apparent relative density (see section 2) of the coke.

3.3 Expression of results

The porosity of the coke, as a percentage, is calculated from the following formula :

$$\frac{D - AD}{D} \times 100$$

where

- D is the true relative density of the coke;
- AD is the apparent relative density of the coke.

The result should be reported to the nearest 1 %.