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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

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC *27*, *Coal and coke*, Subcommittee SC 3, *Coke*.

This third edition cancels and replaces the second edition (ISO 1014:1985), which has been technically revised. It also incorporates the Technical Corrigendum ISO 1014:1985/Cor 1:1994.

The main changes compared to the previous edition are as follows:

— the normative references (see Clause 2) have been updated;

— the mandatory Terms and definitions clause (Clause 3) has been added and subsequent clauses have been renumbered;

— general and technical revision.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies methods for

a) determining the true relative density of coke, crushed to <212 µm, in relation to water;

NOTE "True relative density" varies according to the displacement liquid used.

b) 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;

c) calculating the porosity of the coke.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

ISO 18283, *Hard Coal and Coke — Manual sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 True relative density

4.1 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 is specified. Thermostatic control of the temperature is essential since a difference of 1 °C can cause an error of about 0,012 in the result.

4.2 Apparatus

4.2.1 Pyknometer, 50 ml capacity, Figure 1.

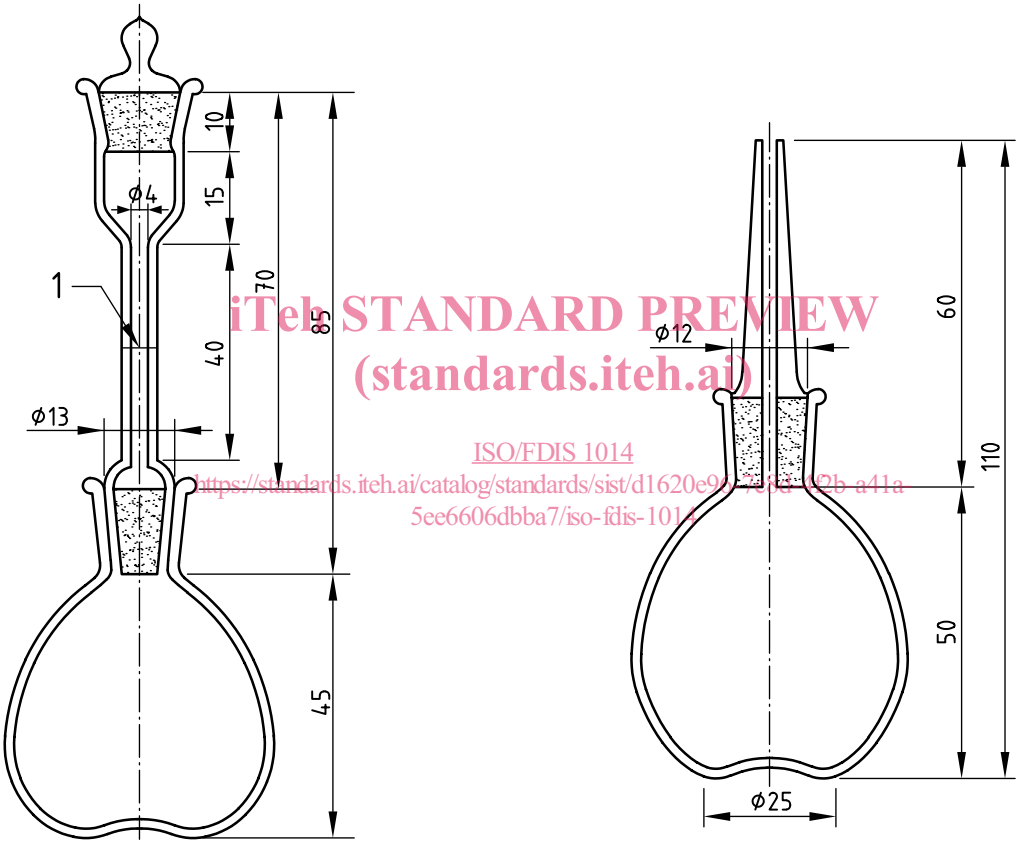
4.2.2 Water bath, with stirrer, thermostatically controlled to maintain a desired temperature 0 °C to within ±1 °C.

4.2.3 Two wash bottles, each containing about 50 ml of air free distilled water. One wash bottle is kept hot (80 °C to 90 °C) and the other is left in the water bath (4.2.2).

4.2.4 Reflux air condenser: a glass tube about 1 m long, of the same external diameter as the neck of the pyknometer (4.2.1) with a short length of rubber tubing for attaching it to the latter.

4.2.5 Glycerol bath: a suitable vessel in which sufficient glycerol can be heated for the lower two-thirds of the pyknometer (4.2.1) to be immersed.

4.2.6 Balance, accurate to 0,1 mg.



Key

1 marked line

Figure 1 — Example of pyknometer

4.3 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 (see ISO 13909-6, ISO 18283 and ISO 3310-2). Before commencing the determination, mix the sample thoroughly for at least 1 min, preferably by mechanical means.

4.4 Procedure

4.4.1 Clean the pyknometer (4.2.1), fill with air-free distilled water. Insert the stopper and immerse the pyknometer up to the neck in the water bath (4.2.2) 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 to approximately ambient temperature under cold running water, dry, allow to stand beside the balance (4.2.6) for 30 min and determine the mass of the pyknometer with an accuracy of ±0,1 mg. Empty the pyknometer and dry the neck.

4.4.2 Dry a portion of the coke sample for 1 h at 105 °C to 110 °C (see 4.4.3), cool to ambient temperature, 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 (4.2.4) to the neck of the pyknometer with the rubber tubing and immerse the pyknometer in the glycerol bath (4.2.5). 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 at ambient temperature. After the water has boiled 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 determine the mass of the pyknometer as before.

4.4.3 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.

4.5 Expression of results

4.5.1 Calculations

The true relative density of the coke, ρ, is given by the following Formula (1):

$$\rho = \frac{m_1}{m_1 + m_2 - m_3} \tag{1}$$

where

m_1 is the mass, in grams, of the 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 4.5.2), shall be reported to the nearest 0,01.

4.5.2 Precision

4.5.2.1 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.

4.5.2.2 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.

5 Apparent relative density

5.1 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.

5.2 Principle

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

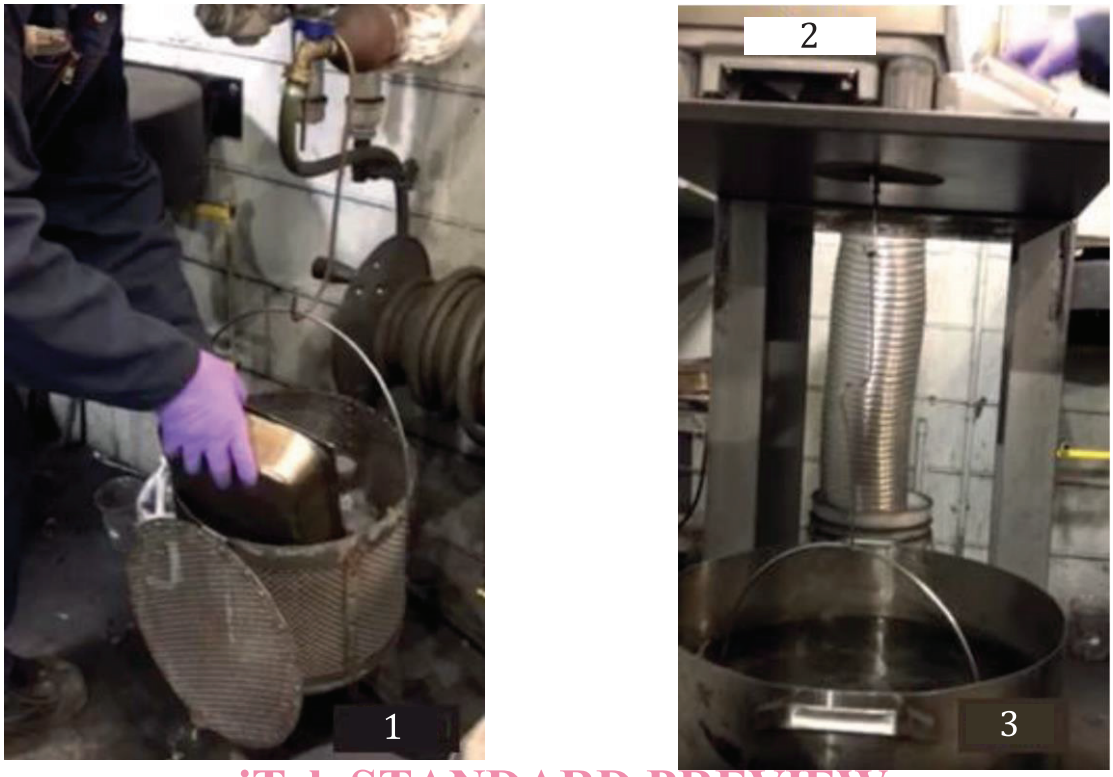
5.3 Apparatus

5.3.1 Cage, 0,03 m^3 capacity, made of galvanized iron wire or metallic mesh with hole size about 12 mm, fitted with a lid of the same material and a fastening device.

5.3.2 Water tank, deep enough to immerse the cage (5.3.1) completely and fitted with a tap for emptying. An elliptical tank (see Figure 2) or rectangular cross-sectioned container (see Figure 3) are both allowed.

The tank or container is approximately 560 mm in length, 280 mm in width, and a minimum of 330 mm in height, provided with a spout consisting of a short 13 mm nipple extending horizontally from the container about 270 mm above the bottom.

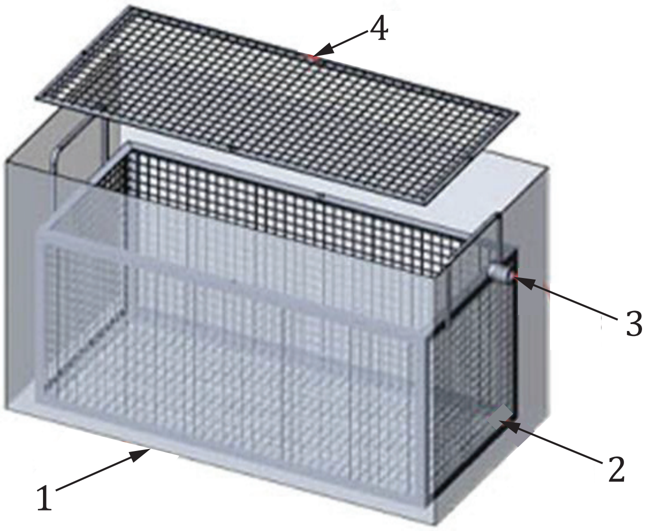
An elliptical tank made of steel plate of 2 mm minimum thickness with inside diameter of 330 mm, 330 mm minimum height shall be used.



Key

1 cage
2 balance
3 tank

Figure 2 — Elliptical tank and iron wire cage for determination of coke apparent density



Key

1 ASG container
2 ASG basket
3 spout
4 basket lid

Figure 3 — Rectangular cross-sectioned container and metallic mesh cage for determination of coke apparent density

5.3.3 Mass determination machine, capable of mass determination, to within 1 g, a mass up to 2 kg. The machine shall be supported firmly on a wooden support above the tank (5.3.2). 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 (5.3.1). Alternatively, a suitable dial machine may be used.

5.3.4 Drainage tray, of galvanized iron sheet, slightly larger than the base of the cage (5.3.1) and 13 mm deep.

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

5.3.6 Drying oven, large enough to accommodate the cage (5.3.1).

5.4 Sample

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

When the mass of lump coke of about 25 mm or more in the gross sample is less than 40 kg, the sampled number of pieces of increment should be increased so that the mass becomes 40 kg or more. The gross sample taken in accordance with ISO 13909-6 or ISO 18283 shall be screened with the sieve of hole size of 25 mm, in accordance with ISO 3310-2. The coke lumps remaining on this sieve shall be collected, and a one-time sample of 10 kg or more shall be taken by the increment reduction method.

Dry the coke to constant mass at a temperature of 105 °C to 200 °C. Determine the mass of the coke when cool, after shaking and brushing off any adhering dust.

