BRITISH STANDARD

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Methods for

Analysis and testing of coal and coke —

Part 108: Tests special to coke —

Section 108.5 Determination of density and porosity

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Solid Mineral Fuels Standards Policy Committee (SFC/-) to Technical Committee SFC/3, upon which the following bodies were represented:

British Cement Association
British Coal Corporation
British Gas plc
British Steel Industry
Electricity Industry in United Kingdom
GAMBICA (BEAMA Ltd.)
Institute of Petroleum
Power Generation Contractors' Association (BEAMA Ltd.)

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Contents

Foreword

This Section of BS 1016 has been prepared under the direction of the Solid Mineral Fuels Standards Policy Committee. Part 108 is a revision of the 1980 edition of BS 1016-13, which is withdrawn. This Section replaces clauses **6**, **7** and **8** of BS 1016-13:1980, from which the principal changes are as follows.

a) A test portion of about 5 g (instead of about 2 g) is now specified for the determination of true relative density.

b) A time of 1 min (instead of 10 s) is now specified for draining the empty cage, as well as being specified for draining the full cage, during the determination of apparent relative density.

Part 108 is a further Part numbered under a scheme for rationalizing and restructuring BS 1016. The new series, when complete, will begin with Part 100, which will include a general introduction. The earlier series of Parts is as follows, with the new Part numbers (which will be given to revisions when they are published) in parentheses.

— *Part 1: Total moisture of coal* (Part 101);

— *Part 2: Total moisture of coke* (Part 102);

— *Part 6: Ultimate analysis of coal* (Part 106);

— *Part 7: Ultimate analysis of coke* (Part 106);

— *Part 8: Chlorine in coal and coke* (Part 106);

— *Part 9: Phosphorus in coal and coke* (Part 106);

— *Part 10: Arsenic in coal and coke* (Part 106);

— *Part 11: Forms of sulphur in coal* (Part 106);

— *Part 14: Analysis of coal ash and coke ash* (Part 114);

— *Part 15: Fusibility of coal ash and coke ash* (Part 113);

— *Part 16: Methods for reporting results* (Part 100);

— *Part 17: Size analysis of coal* (Part 109);

— *Part 18: Size analysis of coke* (Part 110);

— *Part 20: Determination of Hardgrove grindability index of hard coal* (Part 112);

— *Part 21: Determination of moisture-holding capacity of hard coal* (Part 103).

The following Parts in the new series have been published.

— *Part 104: Proximate analysis;*

— *Part 105: Determination of calorific value;*

— *Part 107: Caking and swelling properties of coal;*

— *Part 108: Tests special to coke;*

— *Part 111: Determination of abrasion index of coal.*

Part 108 is divided into six Sections as follows.

— *Section 108.1: Determination of shatter indices;*

— *Section 108.2: Determination of Micum and Irsid indices;*

— *Section 108.3: Determination of bulk density (small container);*

— *Section 108.4: Determination of bulk density (large container);*

— *Section 108.5: Determination of density and porosity;*

— *Section 108.6: Determination of critical air blast value.*

This Section is related to ISO 1014:1985, published by the International Organization for Standardization (ISO). The principal differences are as follows.

a) ISO 1014 specifies the use of both a weighing machine and platform scales for the determination of apparent relative density, whereas this Section of BS 1016 specifies the use of the same machine for all weighing operations during that determination.

b) ISO 1014 specifies draining times of 10 s for the determination of apparent relative density, whereas this Section of BS 1016 specifies 1 min.

WARNING. This British Standard does not necessarily detail all the precautions necessary to comply with the requirements of the Health and Safety at Work etc. Act 1974 [1] or the Control of Substances Hazardous to Health Regulations, 1988 [2]. Attention should be paid to any appropriate precautions and the method should be operated only by trained personnel.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This Section of BS 1016 describes methods for the determination of the true relative density of coke and the apparent relative density of coke and for the calculation of porosity.

2 References

2.1 Normative references

This Section of BS 1016 incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this Section of BS 1016 only when incorporated in it by updating or revision.

2.2 Informative references

This Section of BS 1016 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Definitions

For the purposes of this Section of BS 1016 the following definitions apply.

3.1
true relative density

the ratio of the mass of a sample of dry coke ground to pass a 212 µm test sieve to the mass of an equal volume of water at a specified temperature

3.2
apparent relative density

the ratio of the mass of dry coke to the mass of a volume of water equal to the apparent volume of the coke at a specified temperature

3.3
porosity

the ratio of the volume of the voids within a piece of coke to its apparent volume

4 Determination of true relative density

4.1 Principle

The mass of de-aerated water displaced by a known mass of dry coke is determined in a pyknometer at a controlled temperature.

4.2 Reagent

4.2.1 *Water*, conforming to grade 3 of BS 3978:1987, de-aerated by boiling.

4.3 Apparatus

4.3.1 *Oven*, capable of being maintained at a temperature within the range 105 °C to 110 °C.

4.3.2 *Balance*, capable of weighing to the nearest 0.1 mg.

4.3.3 *Pyknometer*, of capacity 50 ml, conforming to BS 733-1:1983.

4.3.4 *Water bath*, with stirrer and thermostatic control capable of maintaining a temperature of t °C to within ± 0.1 °C.

NOTE The value of t should be about 5 °C above the ambient temperature.

4.3.5 *Wash bottle*, containing about 100 ml of the water (**4.2.1**).

4.3.6 *Reflux air condenser*, comprising a glass tube about 1 m long, of the same external diameter as the neck of the pyknometer (**4.3.3**), fitted with a short length of rubber tubing for attaching it to the pyknometer.

4.3.7 *Glycerol bath*, consisting of a suitable vessel in which sufficient glycerol can be heated for the lower two-thirds of the pyknometer (**4.3.3**) to be immersed.

4.4 Preparation of test sample

The coke used for the determination is the general analysis sample (see BS 1017-2:1960), ground to pass a 212 µm test sieve conforming to BS 410:1986. Immediately before weighing out the test portion (see **4.5.1**), mix the test sample for not less than 1 min, preferably by mechanical means.

4.5 Procedure

4.5.1 *Test portion*

Heat a portion of the test sample (**4.4**) in a suitable dish in the oven (**4.3.1**) for about 1 h at a temperature within the range 105 °C to 110 °C. Allow to cool and then weigh the dish to the nearest 0.1 mg using the balance (**4.3.2**). Reheat the dish in the oven at the same temperature for not less than 10 min, cool and reweigh to the nearest 0.1 mg. Repeat the operations of heating for not less than 10 min, cooling and weighing until the difference between successive weighings does not exceed 1 mg.

NOTE Some reactive cokes are hygroscopic and with such cokes the drying operations described above may be omitted. In this case, the moisture content of the general analysis sample should be determined by the method described in BS 1016-104.2:1991, at the same time as its density is determined, and the mass of the test portion for the latter determination should be corrected accordingly.

Weigh, to the nearest 0.1 mg, about 5 g of the dry coke using the balance (**4.3.2**).

4.5.2 *Determination*

Clean the pyknometer (**4.3.3**) using a degreasing agent. Rinse it thoroughly with the water (**4.2.1**) and then fill it also with the water. Insert the stopper and immerse the pyknometer up to the neck in the water bath (**4.3.4**), maintained at a temperature of $t \pm 0.1$ °C.

NOTE The value of t should be about 5 °C above the ambient temperature.

After 1 h, remove any water from the top of the stopper with a piece of filter paper. Remove the pyknometer from the water bath, cool it rapidly under running cold water, dry, and allow it to stand beside the balance (**4.3.2**) for 30 min. Weigh the pyknometer to the nearest 0.1 mg using the balance. Then empty the pyknometer and dry the neck.

Transfer the test portion (**4.5.1**) quantitatively to the pyknometer. Wash down any coke adhering to the neck or upper part of the pyknometer using water from the wash bottle (**4.3.5**) and make up the volume to about 25 ml with the water. Attach the air condenser (**4.3.6**) to the neck of the pyknometer with the rubber tubing and immerse the pyknometer in the glycerol bath (**4.3.7**). Heat the bath so that the water in the pyknometer begins to boil vigorously to eliminate air. Wash down any scum of coke with a few millilitres of water from the wash bottle. 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 water from the wash bottle at t °C, insert the stopper and immerse the pyknometer up to the neck in the water bath maintained at a temperature of $t \pm 0.1$ °C, compensating for any contraction of the liquid in the pyknometer by the addition of water from the wash bottle and ensuring that air bubbles are not trapped either below the stopper or in the capillary. After 1 h, remove any 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, then weigh to the nearest 0.1 mg using the balance.

4.5.3 *Number of determinations*

Carry out two determinations, as described in **4.5.2**, using separate test portions (**4.5.1**).

4.6 Expression of results

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

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

where

m_1 is the mass of the test portion (in g);

m_2 is the mass of the pyknometer filled with water at t °C (in g);

m_3 is the mass of the pyknometer, plus test portion, filled with water at t °C (in g).

Calculate the result as the mean of the two determinations. Report the result to the nearest 0.01.

4.7 Precision

4.7.1 *Repeatability*

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator using the same apparatus on two representative portions taken from the same test sample, should not differ by more than 0.02.

4.7.2 *Reproducibility*

The means of the results of duplicate determinations, carried out in each of two different laboratories on representative portions taken from the same sample after the final stage of sample preparation, should not differ by more than 0.03.

5 Determination of apparent relative density

5.1 Principle

The mass of water displaced by a test portion of coke is determined by weighing the coke in air and in water.

5.2 Apparatus

5.2.1 *Cage*, of capacity 0.03 m^3, made of corrosion-resistant wire mesh of about 12 mm aperture size, fitted with a lid of the same material and a fastening device.

5.2.2 *Tank*, of capacity approximately 0.3 m^3, deep enough to immerse the cage completely and fitted with a tap for emptying.

NOTE The tank should contain sufficient water (about 250 l) to ensure that the increase in temperature of the water after the immersion of the hot coke does not exceed 20 K.

5.2.3 *Weighing machine*, with a capacity of not less than 25 kg, capable of weighing to the nearest 1 g.

NOTE The weighing machine may be fitted with a tare bar or a suspension hook; alternatively, a suitable dial or digital machine may be used.

The machine shall be supported firmly above the tank. A wire stirrup shall be provided for fixing to the weighing machine and shall terminate in two hooks for attachment to the sides of the cage.

5.2.4 *Tray*, 25 mm deep and slightly larger in area than the base of the cage, made of suitable corrosion-resistant material.

5.2.5 *Oven*, large enough to contain the cage and capable of being maintained at a temperature within the range 105 °C to 110 °C.

5.3 Preparation of test sample

Take a sample for physical testing in accordance with BS 1017-2:1960.

5.4 Procedure

NOTE 1 It is recommended that duplicate determinations are carried out.

Weigh the dry empty cage (**5.2.1**) to the nearest 1 g using the weighing machine (**5.2.3**). Fix the stirrup to the weighing machine and balance it. Suspend the cage from the stirrup, immerse it completely in the water in the tank (**5.2.2**) and weigh to the nearest 1 g. Remove the cage from the tank, allow it to drain for 1 min, place it on the tray (**5.2.4**) and reweigh.

Fill the cage with coke and dry in the oven (**5.2.5**) for about 8 h at a temperature within the range 105 °C to 110 °C. Weigh the hot coke and cage to the nearest 1 g using the weighing machine. Return the cage to the oven, continue drying at the same temperature for 15 min and then reweigh. Repeat the operations of drying for 15 min and weighing until the difference between successive weighings does not exceed 10 g. Suspend the full cage from the stirrup and immerse completely in the water in the tank whilst still hot.

NOTE 2 Hot immersion facilitates the removal of air bubbles.

Agitate the cage after 1 min to remove air bubbles. After a further 2 min, record the mass of the cage immersed in water to the nearest 1 g, using the weighing machine, remove the cage from the tank and allow it to drain for 1 min. Place the cage on the tray and weigh to the nearest 1 g.

5.5 Expression of results

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 of the dry empty cage in air (in g);

m_5 is the mass of the empty cage in water (in g);

m_6 is the mass of the empty cage, after draining, plus the tray (in g);

m_7 is the mass of the cage, plus dry coke, in air (in g);

m_8 is the mass of the cage, plus coke, in water (in g);

m_9 is the mass of the cage, plus coke, after draining, plus the tray (in g).

If duplicate determinations are carried out (see note 1 of **5.4**), calculate the result as the mean of the two determinations. Report the result to the nearest 0.01.

5.6 Precision

5.6.1 *Repeatability*

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator using the same apparatus on two representative portions taken from the same test sample, should not differ by more than 0.02.

5.6.2 *Reproducibility*

The means of the results of duplicate determinations, carried out in each of two different laboratories on representative portions taken from the same sample after the final stage of sample preparation, should not differ by more than 0.03.

6 Calculation of porosity

The porosity of the coke, p, expressed as a percentage by volume, is given by the equation:

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

where

d is the true relative density of the coke;

d_A is the apparent relative density of the coke.

Report the result to the nearest 1 % (*V*/*V*).

7 Test report

The test report shall include the following:

a) the identification of the sample;

b) the reference to the method used, i.e. BS 1016-108.5:1992;

c) the results expressed in accordance with **4.6**, **5.5** and clause **6**;

d) any unusual features noted during the determination;

e) any operation not specified in this standard or regarded as optional.

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List of references (see clause 2)

Normative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 410:1986, *Specification for test sieves.*
BS 733, *Pyknometers.*
BS 733-1:1983, *Specification.*
BS 1016, *Methods for analysis and testing of coal and coke.*
BS 1016-104, *Proximate analysis.*
BS 1016-104.2:1991, *Determination of moisture content of the general analysis sample of coke.*
BS 1017, *Methods for sampling of coal and coke.*
BS 1017-2:1960, *Sampling of coke.*
BS 3978:1987, *Specification for water for laboratory use.*

Informative references

ISO standards publications

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO), Geneva. (All publications are available from BSI Sales.)

ISO 1014:1985, *Coke — Determination of true relative density, apparent relative density and porosity*[1)].

Other references

[1] GREAT BRITAIN. Health and Safety at Work etc. Act 1974. London: HMSO.
[2] GREAT BRITAIN. Control of Substances Hazardous to Health Regulations, 1988. London: HMSO.

[1)] Referred to in the foreword only.

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