BRITISH STANDARD

BS 6043-2.12: 1994

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 2: Electrode coke —

Section 2.12 Determination of particle size distribution of fine coke

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/24, upon which the following bodies were represented:

Aluminium Federation
British Ceramic Research Ltd.
Chemical Industries Association
Institute of Petroleum
Refractories Association of Great Britain

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The following BSI references relate to the work on this standard:
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Amendments issued since publication

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Contents

Foreword

This Section of BS 6043 has been prepared under the direction of the Chemicals Standards Policy Committee. BS 6043 provides methods of sampling and test for carbonaceous materials used in aluminium manufacture. The standard is published in four Parts, each Part being divided into Sections.

Part 2 comprises a series of methods of test for electrode coke as follows:

Section	Subject	Identical with
2.1	Sampling	ISO 6375
2.2	Ash content of green and calcined cokes	ISO 8005
2.3	Ash analysis (AAS)	—
2.4	Ash analysis (XRF)	—
2.5	Apparent density and porosity	—
2.6	Density (xylene method)	ISO 8004
2.7	Oil content (gravimetric method)	ISO 6997
2.8	Oil content (extraction method)	ISO 8723
2.9	Sieve analysis	—
2.10	Electrical resistivity	—
2.11	Volatile matter content	—
2.12	Particle-size distribution of fine coke (laser diffraction method)	—

It is essential to use BS 3406-1:1986 in conjunction with this Section of BS 6043.

This British Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

This standard describes a method of test only, and should not be used or quoted as a specification defining the characteristics of a product. Reference to this Section should indicate that the method of test used in accordance with BS 6043-2.12:1994.

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 and ii, pages 1 and 2, 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.

1 Scope

This Section of BS 6043 describes a method for the determination of the particle size distribution of fine calcined petroleum coke powders by a laser diffraction procedure. The method is applicable to particle size analysers capable of measuring particle size distributions in the approximate range 1 μm to 500 μm.

2 Normative references

This Section of BS 6043 incorporates, by dated or undated reference, provisions from other publications. These normative references are made at the appropriate places in the text and the cited publications are listed on the inside back cover. For dated references, only the edition cited applies; any subsequent amendments to or revisions of the cited publication apply to this Section of BS 6043 only when incorporated in the reference by amendment or revision. For undated references, the latest edition of the cited publication applies, together with any amendments.

3 Principle

A representative sample of the petroleum coke powder is sieved to remove any oversize particles which would fall outside the measurement range of the laser diffraction equipment, and the amounts of material passing through and retained on the sieve are weighed and recorded. A representative subsample of the material which passes through the sieve is dispersed at a suitable concentration in methanol and is passed through the beam of a monochromatic laser light source. The light scattered by the particles in the near-forward direction is recorded by a multi-element detector and the numerical values relating to the scattering pattern are passed to a computer memory. The stored data representing the scattering pattern are transformed, using an adequate optical model and mathematical procedure to yield the proportions of the total volume present in a discrete number of size classes to form a volumetric particle size distribution.

4 Reagent

4.1 *Methanol,* general purpose reagent grade.

WARNING. Refer to the reagent supplier's Health and Safety data sheets for the precautions which are to be taken for the safe use of methanol.

NOTE It is essential to use methanol for all reference purposes. However, for routine purposes dispersion media other than methanol may be used.

5 Apparatus

5.1 *Ordinary laboratory apparatus*

5.2 *Laser-diffraction particle size analyser,* capable of measuring the particle size distribution of powder, having a measurement range which includes the range of the petroleum coke to be tested and having a sample dispersion unit, a sample cell, and data handling facilities.

NOTE The sample cell of the particle size analyser may be of the static or flow-through types.

5.3 *Test sieve,* conforming to BS 410:1986, having a nominal aperture size slightly smaller than the upper limit of the measurement range of the particle size analyser (**5.2**), typically 500 μm, and having an appropriate lid and receiver.

5.4 *Microscope,* with associated light source, suitable for examining dispersions by transmitted light at a nominal linear magnification of approximately × 500.

5.5 *Ultrasonic bath*

6 Sampling and preparation of test sample

Prepare a representative test sample of approximately 10 g of the coke in accordance with clause **13** or clause **14** of BS 3406-1:1986. Sieve the test sample using the test sieve (**5.3**) to separate any particles which are larger than the upper limit of the size range of the laser particle size analyser (**5.2**). Weigh the material retained on the sieve and set it aside. Record the mass. Carefully brush the sieve to remove any fine particles which may be clinging to the mesh or elsewhere. Add this material to the coke which passed through the sieve, weigh the combined undersize material then mix it thoroughly and use this material for the particle size analysis.

7 Procedure

7.1 Setting up

Follow the manufacturer's instructions for setting up the laser diffraction particle size analyser (**5.2**) for the expected range of particle sizes.

7.2 Sample dispersion

Disperse an appropriate amount of the test sample (see clause **6**) in the methanol (**4.1**) in the test cell in accordance with the instrument manufacturer's instructions. In the first instance disperse the sample solely by stirring and after the recommended time has elapsed withdraw a small amount of the dispersion by pipette and examine it using the microscope (**5.4**) for the presence of agglomerates. If agglomerates are present subject the dispersion in the instrument to ultrasonic treatment for 1 min then repeat the microscopic examination. Repeat the ultrasonic treatment until agglomeration is no longer detected under the microscope, but take care to use the minimum period of ultrasonic treatment which achieves this aim.

7.3 Measurement

Measure the particle size distribution of the coke in accordance with the instrument manufacturer's instructions, producing the test result in the form of a table showing the cumulative percentage of undersize material at each of the size points in the range for which the instrument was set up. If the option exists, also produce the test result in graphical form as a curve showing the cumulative percentage of undersize material (as ordinate) against particle size (as abscissa). Perform a duplicate measurement using a new portion of the test sample (see clause **6**). Express the test result as the average of the two sets of measurements.

In the event that the test result shows that the particle size range for which the instrument was set up is inappropriate, select an appropriate size range and repeat the measurement. If the instrument does not have an appropriate range include a comment to this effect in the test report (see clause **8**).

8 Test report

The test report shall include the following information:

a) a complete identification of the sample;

b) a reference to this British Standard, i.e. BS 6043-2.12:1994;

c) the amount of oversize material, expressed as a percentage by mass, removed by sieving prior to the determination;

d) the manufacturer's name and the model identification of the laser diffraction particle size analyser (**5.2**);

e) the type of sample cell (**5.2**) used;

f) the particle size distribution measurement result (see **7.3**) expressed as a table showing the cumulative percentage of undersize material at each of the particle-size points associated with the instrumental range used;

g) a graphical representation of the particle size distribution if this is available as an instrumental output option;

h) any unusual features noted during the determination;

i) any operation not included in this British Standard or regarded as optional.

List of references (see clause 2)

Normative references

BSI publications

BRITISH STANDARDS INSTITUTION, London

BS 410:1986, *Specification for test sieves.*
BS 3406, *Methods for determination of particle size distribution.*
BS 3406-1:1986, *Guide to powder sampling.*

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W4 4AL