

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 1: Electrode pitch —

Section 1.11 Determination of the
atomic C/H ratio of the
quinoline-insoluble material

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

This British Standard, having been prepared under the direction of the Chemicals Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 15 June 1994

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The following BSI references relate to the work on this standard:
Committee reference CIC/24
Draft for comment 93/505980 DC

ISBN 0 580 22854 1

Amendments issued since publication

Amd. No.	Date	Comments

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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 1 comprises a series of methods of test for electrode pitch as follows.

Section	Subject	Identical with
1.1	Sampling	ISO 6257
1.2	Water content (Dean and Stark method)	ISO 5939
1.3	Softening point (ring and ball method)	ISO 5940
1.4	Content of toluene-insoluble material	ISO 6376
1.5	Content of quinoline-insoluble material	ISO 6791
1.6	Coking value	ISO 6998
1.7	Density	—
1.8	Ash	ISO 8006
1.9	Sulfur content	ISO 9055
1.10	Volatile matter content (gas chromatographic method)	—
1.11	Atomic C/H ratio of quinoline-insoluble material	—
1.12	Mesophase content	—
1.13	Sulfur content (Routine method)	—

Other international methods of test for electrode pitch are under consideration and, subject to approval by the United Kingdom, will be published as British Standards as they become available.

It is essential to use BS 6043-1.1 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 state that the method of test used is in accordance with BS 6043-1.11:1994.

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.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, 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 atomic carbon/hydrogen ratio of quinoline-insoluble material isolated from coal-tar pitches. A procedure for the isolation of the quinoline-insoluble material in a form suitable for the analysis is described.

NOTE The isolation procedure is not an alternative to BS 6043-1.5:1983; the latter is to be used in all cases where the amount of quinoline-insoluble matter present in a pitch is to be determined.

2 References

2.1 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 Part 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.

2.2 Informative references

This Section of BS 6043 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 Principle

The quinoline-insoluble material is isolated from a finely powdered sample of the coal-tar pitch by digestion with hot quinoline followed by pressure-filtration through a membrane filter. The isolated material is washed on the filter with hot quinoline then with hot toluene and dried.

The carbon and hydrogen contents of the isolated quinoline-insoluble material are determined by combustion of a weighed aliquot portion of that material in a stream of oxygen and trapping and weighing the carbon dioxide and water which are formed. The atomic carbon/hydrogen ratio of the quinoline-insoluble material is calculated from its carbon and hydrogen contents and the relative atomic masses of carbon and hydrogen.

4 Isolation of quinoline-insoluble material for analysis

4.1 Reagents

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 quinoline and toluene.

4.1.1 Quinoline, of purity at least 97 %, freshly distilled, boiling between 235 °C and 237 °C at 0.1013 MPa. The distilled quinoline should be stored between 5 °C and ambient temperature in a stoppered bottle of dark glass and be used within two weeks; after this time it should be redistilled or discarded.

4.1.2 Toluene, of general purpose reagent grade.

4.1.3 Nitrogen or other inert gas, supplied via a two-stage pressure regulator with a maximum outlet pressure of 0.45 MPa.

4.2 Apparatus

WARNING. Before using pressure filtration equipment ensure that it is provided with adequate safety devices and that the equipment has been tested to ensure compliance with relevant Health and Safety legislation.

4.2.1 Ordinary laboratory apparatus

4.2.2 Thermostatically controlled electric hotplate, capable of maintaining liquids in beakers at 70 °C to 80 °C.

4.2.3 Pressure filtration apparatus, accepting filter discs of 47 mm diameter and designed for safe operation at up to 120 °C with applied gas pressures of up to 1 MPa. A typical pressure filtration apparatus is shown in Figure 1.

4.2.4 Membrane filter discs, of polytetrafluoroethylene (PTFE), 47 mm in diameter and having a nominal pore size of 0.2 µm.

4.2.5 Hand-held hot air blower, of general-purpose laboratory type, typically of 500 W power rating and giving an air exit temperature of approximately 300 °C.

4.2.6 Test sieve, of 212 µm nominal aperture, complying with BS 410:1986, and having a lid and receiver.

4.2.7 Watch glass, approximately 70 mm in diameter, dried at 105 °C to 110 °C for 1 h and cooled in a desiccator.

4.2.8 Measuring cylinder, of borosilicate glass, of 50 ml capacity with pouring spout, complying with BS 604:1982.