BRITISH STANDARD

BS 6043-1.5: 1983
ISO 6791:1981

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 1: Electrode pitch —

Section 1.5 Determination of content of quinoline-insoluble material

[ISO title: Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of content of quinoline-insoluble material]

UDC 665.775:669.713.7:543.868

Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

Association of Fatty Acid Distillers
British Tar Industry Association*
Chemical Industries Association
Consumer Standards Advisory Committee of BSI
Department of Health and Social Security
Department of Industry (Laboratory of the Government Chemist)
Fertiliser Manufacturers' Association Ltd.
Ministry of Agriculture, Fisheries and Food
Ministry of Defence
National Sulphuric Acid Association
Paintmakers' Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Royal Society of Chemistry
Soap and Detergent Industry Association
Standardization of Tar Products Tests Committee*

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the Technical Committee entrusted with the preparation of this British Standard:

Aluminium Federation
British Ceramic Research Association
Institute of Petroleum

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on
31 May 1983

The following BSI references relate to the work on this standard:
Committee reference CIC/24
Draft for comment 80/50463 DC

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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee to provide methods of sampling and test for carbonaceous materials used in the production of aluminium. The standard will be published in two Parts, each Part being divided into Sections. The two Parts are:

— *Part 1: Electrode pitch*;

— *Part 2: Electrode coke*.

Initially, it is proposed that Part 1 will comprise the following Sections:

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 ....[a]
1.7	Density	ISO ....[a]
1.8	Ash content	ISO ....[a]
1.9	Sulphur content	ISO ....[a]

[a] In course of preparation and referred to for information purposes only.

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

This Section is identical with ISO 6791:1981 "*Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of content of quinoline-insoluble material*", published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "British Standard".

Cross-reference

International Standard	Corresponding British Standard
ISO 6257:1980	BS 6043 *Methods of sampling and test for carbonaceous materials used in aluminium manufacture* Part 1 *Electrode pitch* Section 1.1:1981 *Sampling* (Identical)

Additional information. This standard describes methods of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Section should state that the method of test used is in accordance with BS 6043-1.5:1982.

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 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 and field of application

This International Standard specifies a conventional gravimetric method for the determination of the content of quinoline-insoluble material in pitch used in the production of aluminium.

2 Reference

ISO 6257, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling.*

3 Principle

Extraction of a test portion with quinoline, at a specified temperature and for a specified period. Filtration and weighing of the residue.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade.

4.1 *Quinoline,* freshly distilled, boiling between 235 and 237 °C at 1 013 mbar[1)].

WARNING — Risk of poisoning by inhalation or swallowing. Irritating to skin and eyes. Prevent inhalation of vapour and contact with skin, eyes and clothing.

4.2 *Toluene*

WARNING — Highly flammable. Harmful by inhalation. Keep away from sources of ignition. No smoking. Do not empty into drains. Take precautionary measures against static discharges.

4.3 *Filter aid,* diatomaceous silica type, free from organic compounds, and chemically neutral.

The particle sizes shall be within the range 1 to 40 µm and the average particle size shall be within the range 6 to 10 µm.

Dry the material in the oven (**5.4**), controlled at 105 to 110 °C, for 1 h. Allow to cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. Store the dried material in a desiccator.

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Water bath,* capable of being controlled at 70 to 80 °C.

5.2 *Hartley funnel,* or equivalent, having a disc diameter of 70 mm (a suitable type is shown in the Figure).

5.3 *Glass fibre filter pads,* of diameter 70 mm, with retention equal to 98 % for particles of size 1,2 µm.

NOTE A larger funnel (**5.2**) and pad (**5.3**), for example of diameter 120 mm, may be used, if available, as this largely obviates the need to transfer any residue from the walls of the funnel to the pad during the determination (**7.3**).

5.4 *Electric oven,* capable of being controlled at 105 to 110 °C.

6 Sample preparation

Sample in accordance with ISO 6257. Prepare the test sample immediately before the determination.

If it is sufficiently hard, grind the whole of the test sample, using a mortar and pestle, to a particle size not exceeding 200 µm.

If the pitch is too soft for grinding, melt and mix the sample, ensuring that its temperature does not exceed 150 °C and that the melting period does not exceed 10 min.

NOTE For soft pitches, the material for the test portion may be taken from the molten sample.

7 Procedure

WARNING — Attention is drawn to the dangers involved in the use of quinoline and toluene (see WARNINGS to clauses 4.1 and 4.2). Carry out all operations involving the use of these products in an efficiently ventilated fume cupboard.

7.1 Test portion

Weigh, to the nearest 0,005 g, approximately 1 g of the test sample.

7.2 Filter preparation

Heat one of the filter pads (**5.3**) in the oven (**5.4**), controlled at 105 to 110 °C, for 1 h. Transfer the pad to a desiccator, allow to cool to ambient temperature and weigh to the nearest 0,005 g. Repeat the process of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. Fit the pad into the clean and dry Hartley funnel (**5.2**).

[1)] 1 mbar = 100 Pa

7.3 Determination

Transfer the test portion (**7.1**) quantitatively to a 100 ml beaker. Add approximately 1 g of the dried filter aid (**4.3**), weighed to the nearest 0,005 g, and 25 ml of the quinoline (**4.1**). Stir the contents of the beaker to break up any lumps and cover the beaker with a watch glass.

Partially immerse the beaker in the water bath (**5.1**), controlled at 70 to 80 °C, together with another suitable beaker containing about 100 ml of the quinoline. Bring the temperature of the contents of each beaker to 70 to 80 °C and continue heating for 15 to 20 min, occasionally stirring the contents of the beaker containing the test portion.

Fit the assembled Hartley funnel and filter pad (see **7.2**) into the neck of a filter flask. Moisten the pad with a little of the hot quinoline and apply suction. As soon as filtration is complete, start filtering the dissolved test portion and any residue without unduly disturbing the solution, transferring about 2 ml of this suspension at a time to the filter pad and allowing each portion to filter completely before making the next addition. Between each addition, maintain the temperature of the solution remaining in the beaker at 70 to 80 °C by means of the heated water bath. When filtration is complete, rinse the beaker five times with approximately 5 ml portions of the hot quinoline from the other beaker, so as to transfer all remaining insoluble matter to the pad. Wash the residue on the filter pad with further 5 ml portions of the hot quinoline until the washings are clear. About ten washings are usually necessary.

Wash the beaker which had contained the test portion with ten approximately 5 ml portions of the toluene (**4.2**), allowing each washing to pass completely through the filter pad before making the next addition. During the entire filtering operation, ensure that as little as possible of the solution and residue is allowed to come into contact with the walls of the funnel.

Release the suction, remove the filter pad with the residue and, after transferring to it any solid remaining on the walls of the funnel, place in the oven (**5.4**), controlled at 105 to 110 °C, for about 1 h. Transfer from the oven to the desiccator and allow to cool to ambient temperature. Weigh to the nearest 0,005 g. Repeat the operations of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,005 g. A drying time of 2 to 3 h is usually sufficient.

8 Expression of results

8.1 Method of calculation

The content of quinoline-insoluble material, expressed as a percentage by mass, is given by the formula

$$\frac{m_3 - m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (**7.1**);

m_1 is the mass, in grams, of the dried filter pad (**5.3**);

m_2 is the mass, in grams, of the filter aid (**4.3**) added;

m_3 is the mass, in grams, of the dried filter pad, the filter aid and the residue.

8.2 Precision

Repeatability: 1 %

Reproducibility: 1,50 %

9 Test report

The test report shall include the following particulars:

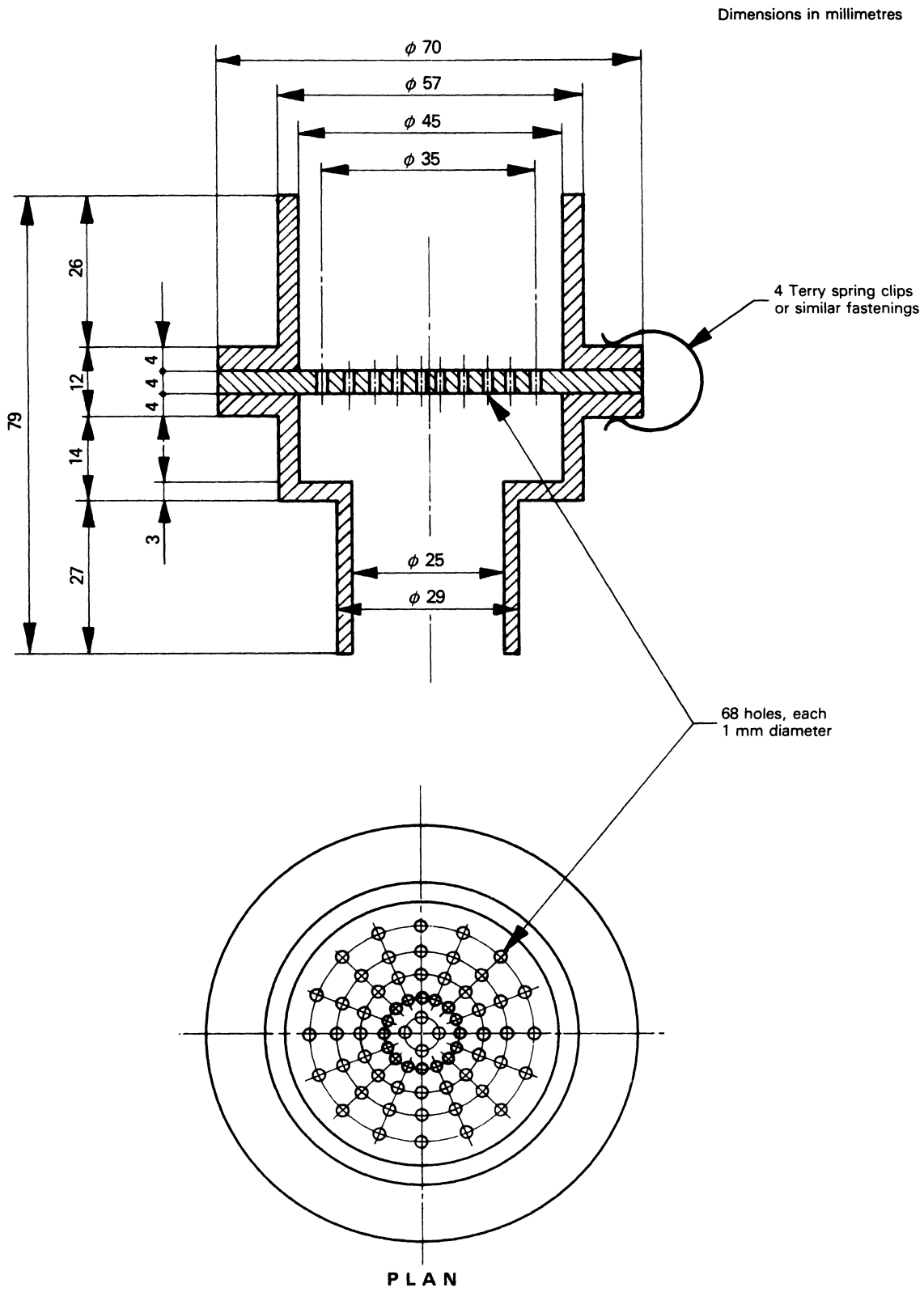
a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.



Material : Aluminium or other suitable metal

Finish : Fine machine all over polished bores and mating surfaces, i.e. flanges

Figure — Details of Hartley funnel

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Publications referred to

See national foreword.

BSI — British Standards Institution

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Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

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