BRITISH STANDARD

BS 6043-1.4: 1981
ISO 6376:1980

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 1: Electrode pitch —

Section 1.4 Determination of content of toluene-insoluble material

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

UDC 665.775:669.713.7:543.86

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*
Chemical Society, Analytical Division*
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
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
Royal Society of Chemistry

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board and comes into effect on 30 October 1981

The following BSI references relate to the work on this standard:
Committee reference CLC/24
Draft for comment 79/54055 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*" and 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[a]
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.

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 6376:1980 "*Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of content of toluene-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 especially drawn to the following.

The comma has been used throughout 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-references

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)

The Technical Committee has reviewed the provisions of ISO 383, ISO 565 and ISO 4793, to which reference is made in clauses **5** and **6**, and has decided that they are acceptable for use in conjunction with this standard.

Related British Standards for ISO 383, ISO 565 and ISO 4793 are BS 572, BS 410 and BS 1752 respectively. ISO 4797, to which reference is made in **5.1**, is in course of preparation and, subject to approval by the UK, will be published as a British Standard without deviation.

Additional information. This standard prescribes 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 6043-1.4:1981.

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 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.

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1 Scope and field of application

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

NOTE The content of toluene-insoluble material is commonly called "sum of resins α + β".

2 Reference

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

3 Principle

Determination by weighing of the fraction of a test portion of pitch which is insoluble in toluene after boiling for a specified period.

4 Reagents and materials

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

4.1 *Toluene*

4.2 *Acetone*

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Conical flask,* of capacity 500 ml, of borosilicate glass, fitted with a ground glass socket (see ISO 4797).

5.2 *Reflux condenser,* effective length 300 mm, having a ground glass cone (see ISO 383) at its lower end, by means of which it can be fitted to the socket of the conical flask (**5.1**).

5.3 *Filter crucible,* of glass, of capacity about 30 ml, fitted with a sintered glass disc (see ISO 4793) of porosity grade P16 (pore size index 10 to 16 μm) and having the following approximate principal dimensions:

disc diameter: 30 mm

height (disc to upper rim): 35 mm.

5.4 *Electric oven,* capable of being controlled at a temperature between 105 and 110 °C.

6 Sampling

See ISO 6257.

In the case of hard pitches, grind the sample so that it passes through a sieve of nominal aperture size 200 μm (see ISO 565).

In the case of soft pitches, use the sample as received.

7 Procedure

WARNING — Toluene is toxic and highly flammable. Carry out all operations involving its use in an efficiently ventilated fume cupboard.

7.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the sample (see clause **6**).

7.2 Determination

Prepare the filter crucible (**5.3**) for the determination by heating it for about 1 h in the oven (**5.4**) controlled at a temperature between 105 and 110 °C, allowing it to cool to ambient temperature in a desiccator and weighing it to the nearest 0,001 g.

Place the test portion (**7.1**) in the conical flask (**5.1**). Add about 100 ml of hot (approximately 80 °C) toluene (**4.1**) and swirl to dissolve the test portion.

NOTE Dissolution before boiling of soft pitches that are liquid on transfer to the flask is particularly necessary to prevent the formation of droplets of pitch which are subsequently not readily soluble in the boiling toluene.

Fit the condenser (**5.2**) to the flask and start the water circulation. Bring the contents of the flask to a steady boil and continue boiling under reflux for about 30 min.

Stop the heating and remove the condenser. Using gentle suction, immediately filter the contents of the flask through the dried and weighed filter crucible (**5.3**). Rinse the flask (**5.1**) with about 10 ml of the hot (approximately 80 °C) toluene (**4.1**) and filter the washings through the filter crucible (**5.3**). When filtration is complete, repeat the rinsing and filtering operations with further 10 ml portions of the hot toluene until any residue in the flask is transferred to the filter crucible and the filtrate is colourless. Wash the filter crucible and its contents with approximately 10 ml of the acetone (**4.2**) and, when filtration is complete, repeat the operation with a further 10 ml portion of the acetone (**4.2**).

Treat the filter crucible and its contents as follows:

Heat for approximately 1 h in the oven (**5.4**), maintained at a temperature between 105 and 110 °C, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,001 g.

8 Expression of results

8.1 Method of calculation

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

$$\frac{(m_1 - m_2)}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the crucible containing the insoluble material;

m_2 is the mass, in grams, of the empty crucible.

8.2 Precision

Repeatability: 0,5 %

Reproducibility: 1 %

9 Test report

The test report shall contain the following information:

a) 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.

Publications referred to

See national foreword.

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