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BRITISH STANDARD

BS 6043-1.2:
1981
ISO 5939:1980

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 1: Electrode pitch —

Section 1.2 Determination of water content (Dean and Stark method)

[ISO title: Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of water content — Azeotropic distillation (Dean and Stark) method]

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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.
Hydrocarbon Solvents Association
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 Institute 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 31 December 1980

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The following BSI references relate to the work on this standard
Committee reference CIC/24
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Amendments issued since publication

Amd. No.	Date of issue	Comments

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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.*

This Section is identical with ISO 5939:1980 "*Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of water content — Azeotropic distillation (Dean and Stark) method*", published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication, without deviation, as a British Standard. 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. There are no corresponding British Standards for the following International Standards referred to in the text: ISO 5940[1)] and ISO 6257[1)]. Subject to final approval by the United Kingdom, ISO 5940[1)] and ISO 6257[1)] will be published, without alteration, as further Sections of BS 6043.

The technical committee has reviewed the provisions of ISO 383:1976, to which reference is made in **5.1.1**, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard for ISO 383:1976 is BS 572:1960 "*Interchangeable ground glass joints*".

The references to the ISO publications listed in the Annex are for information purposes only.

Additional information

Xylene. Analytical grades of xylene normally available in the United Kingdom, the boiling ranges of which may differ marginally from that of the products specified in **4.1**, are nevertheless suitable for use in this determination.

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 complies with the requirements of BS 6043-1.2:1981.

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 and ii, 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.

[1)] In course of preparation.

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1 Scope and field of application

This International Standard specifies an azeotropic distillation method for the determination of the water content of pitch used in the production of aluminium.

The method is applicable to pitches having water contents in the range 0,1 to 2 % (*m*/*m*).

NOTE Water contents in excess of 2 % (*m*/*m*) may be determined by means of this method by using a larger receiver.

2 References

ISO 383, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 5940, *Carbonaceaous materials for the production of aluminium — Pitch for electrodes — Determination of softening point by the ring and ball method.*

ISO 6257, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Sampling*[2)].

3 Principle

Heating the pitch under reflux with xylene. Collection in a receiver of the condensate of xylene and water and measurement of the volume of water which separates out in the graduated part of the receiver.

4 Reagent

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

4.1 *Xylene*, or any of its isomers, or a mixture of these isomers in any proportion, provided that the boiling range is between 130 and 140 °C. (See WARNING, clause **6**.)

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Dean and Stark apparatus*, comprising the following items:

5.1.1 *Distillation flask*, of glass, capacity 500 ml, having a ground-glass joint, complying with ISO 383 (see Figure 1) to fit the receiver (**5.1.2**).

5.1.2 *Graduated receiver*, of capacity 2 ml. The receiver shall be graduated at every 0,05 ml with a maximum error of ± 0,02 ml (see Figure 3).

NOTE For pitches having water contents in excess of 2 (*m*/*m*), use a receiver of 10 ml capacity, graduated at every 0,2 ml and having a maximum error of ± 0,06 ml.

5.1.3 *Reflux condenser*, connected to the receiver **5.1.2** (see Figure 2).

NOTE Before use, remove all traces of contamination from the graduated receiver and from the interior of the reflux condenser by washing them successively with, for example, chromic-sulphuric acid mixture, distilled water and acetone, and dry. Perfect cleanliness of the apparatus is essential to the success of the test.

6 Procedure

WARNING — Carry out the test in a well ventilated fume cupboard, to prevent the exposure of the operators to toxic xylene vapour.

6.1 Test portion

Weigh, to the nearest 0,1 g, 100 ± 0,5 g of the roughly crushed or melted sample (see ISO 6257) into the flask (**5.1.1**) of the Dean and Stark apparatus.

It is important to prevent loss of water when preparing the test portion. If the expected softening point of the sample is higher than 50 °C (see ISO 5940), make it brittle by cooling, crush it, and weigh it in such a way as to exclude extraneous moisture (see ISO 6257).

Melt softer samples using the minimum quantity of heat.

6.2 Determination

Add 100 ml of the xylene (**4.1**) to the distillation flask containing the test portion (**6.1**) and add a few glass beads to regulate boiling. Attach the flask to the Dean and Stark apparatus and circulate cold water through the jacket of the condenser. Heat the flask gently until the test portion has dissolved and then adjust the rate of heating so that the condensate falls from the end of the reflux condenser at a rate of 2 to 5 drops per second.

Continue the distillation until condensed water is no longer visible in any part of the apparatus except in the bottom of the graduated receiver and until the volume of water collected remains constant for 5 min. If a persistent ring of condensed water forms in the condenser tube, remove it by increasing the rate of distillation by a few drops per second for a few minutes.

When the carry-over of water is complete, allow the receiver and contents to cool to room temperature and read the volume of water to the nearest scale division, correcting for any certified error of the receiver.

[2)] At present at the stage of draft.

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7 Expression of results

7.1 Method of calculation

The water content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times 100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (**6.1**);

m_1 is the mass, in grams, of the water collected in the receiver.

NOTE The assumption that 1 ml of water weighs 1 g is sufficiently accurate for the purpose of this determination.

7.2 Precision

Repeatability: ± 0,1 g (absolute).

Reproducibility: ± 0,2 g (absolute).

8 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 Standards to which reference is made, or regarded as optional.

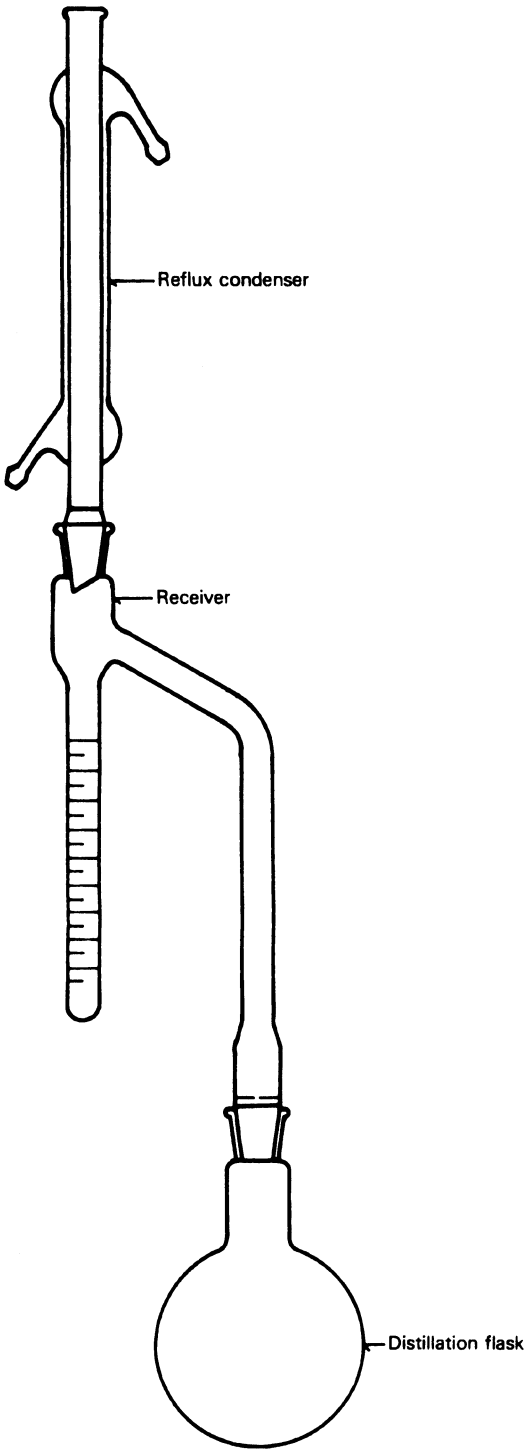

Figure 1 — Typical assembly of Dean and Stark apparatus (5.1)

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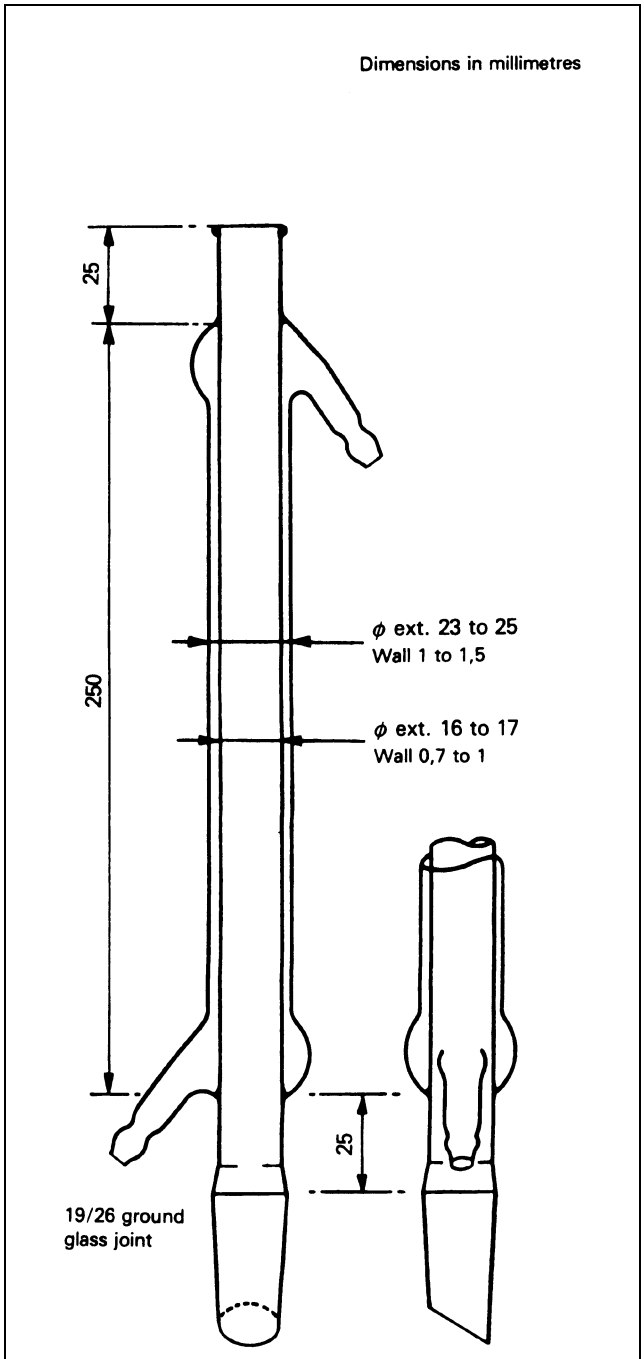

Figure 2 — Suitable form of reflux condenser (5.1.3)

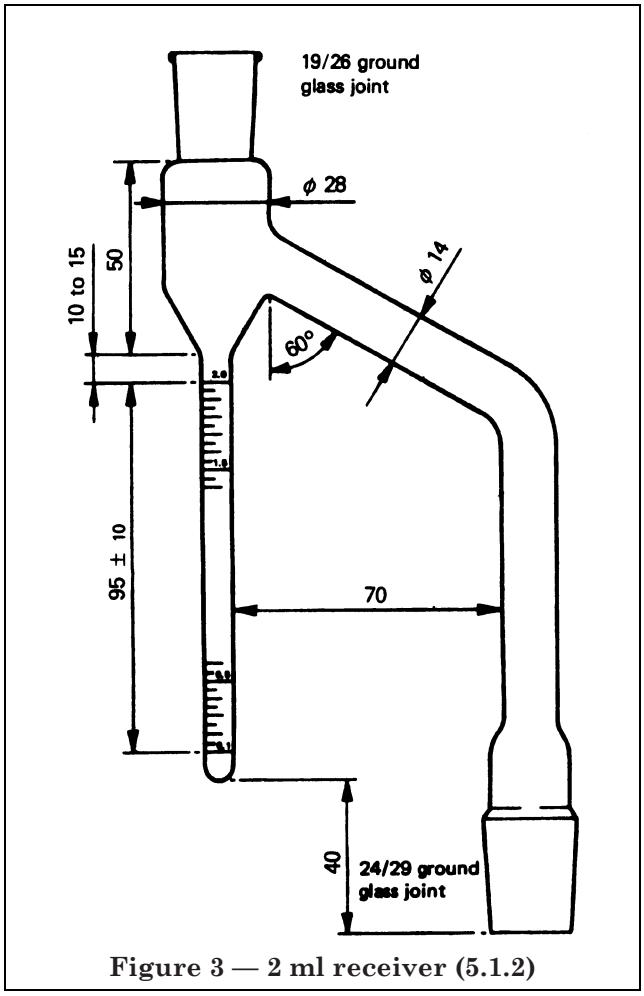

Figure 3 — 2 ml receiver (5.1.2)

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Annex ISO publications relating to the determination of water content in different products, using the azeotropic distillation (Dean and Stark) method

(This annex does not form part of the standard.)

ISO 348, *Hard coal — Determination of moisture in the analysis sample — Direct volumetric method.*

ISO 589, *Hard coal — Determination of total moisture.*

ISO 934, *Animal and vegetable fats and oils — Determination of water — Entrainment method.*

ISO 939, *Spices and condiments — Determination of moisture content — Entrainment method.*

ISO 1015, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 1897-2, *Phenol, o-cresol, m-cresol, p-cresol, cresylic acid and xylenols for industrial use — Methods of test — Part 2: Determination of water — Dean and Stark method.*

ISO 3733, *Petroleum products and bituminous materials — Determination of water — Distillation method.*

ISO 4318, *Surface active agents and soaps — Determination of water content — Azeotropic distillation method.*

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Publications referred to

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