BRITISH STANDARD

BS 6043-4.5: 1991

Methods of sampling and test for

Carbonaceous materials used in aluminium manufacture —

Part 4: Cold ramming pastes —

Section 4.5 Determination of water content of unbaked pastes

Confirmed November 2008

NOTE It is recommended that this Section be read in conjunction with the general information in BS 6043-4.0, published separately.

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 31 October 1991

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	Comments

Contents

Foreword

This Section of BS 6043 has been prepared under the direction of the Chemicals Standards Policy Committee to provide a method of test for cold ramming pastes used in the production of aluminium. There is no corresponding International Standard; however, it is intended, upon publication of this Section of BS 6043, that it be offered to the International Organization for Standardization (ISO) for possible adoption as an International Standard.

It is essential to use BS 6043-4.1 in conjunction with this Section of BS 6043.

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 is in accordance with BS 6043-4.5:1991.

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 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.

0 Introduction

The binders used in cold ramming pastes may be coal tar or bituminous in character or may alternatively comprise one or more resins. Certain types of resins evolve water as a result of the polymerization process; this British Standard determination is not suitable for application to ramming materials containing resin binders.

1 Scope

This Section of BS 6043 describes an azeotropic distillation method for the determination of the water content of unbaked cold ramming pastes used in aluminium manufacture. The method is applicable to cold ramming pastes made with coal tar or bituminous binders, having water contents in the range 0.1 % to 2 % (*m*/*m*). The method is not applicable to cold ramming pastes made with resin-based binders, e.g. phenol-formaldehyde or foam types.

NOTE 1 Water contents in excess of 2 % (*m*/*m*) may be determined by using a larger receiver.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

A sample of cold ramming paste is heated under reflux with xylene. The condensate of xylene and water is collected in a graduated receiver and the volume of water which separates out in the graduated part of the receiver is measured.

3 Reagent

WARNING. Refer to the reagent supplier's health and safety data sheets for the precautions which are to be observed for the safe use of the reagent.

3.1 *Xylene*, any of its isomers, or a mixture of these isomers in any proportion provided that the boiling range is between 130 °C and 140 °C, of recognized analytical grade.

4 Apparatus

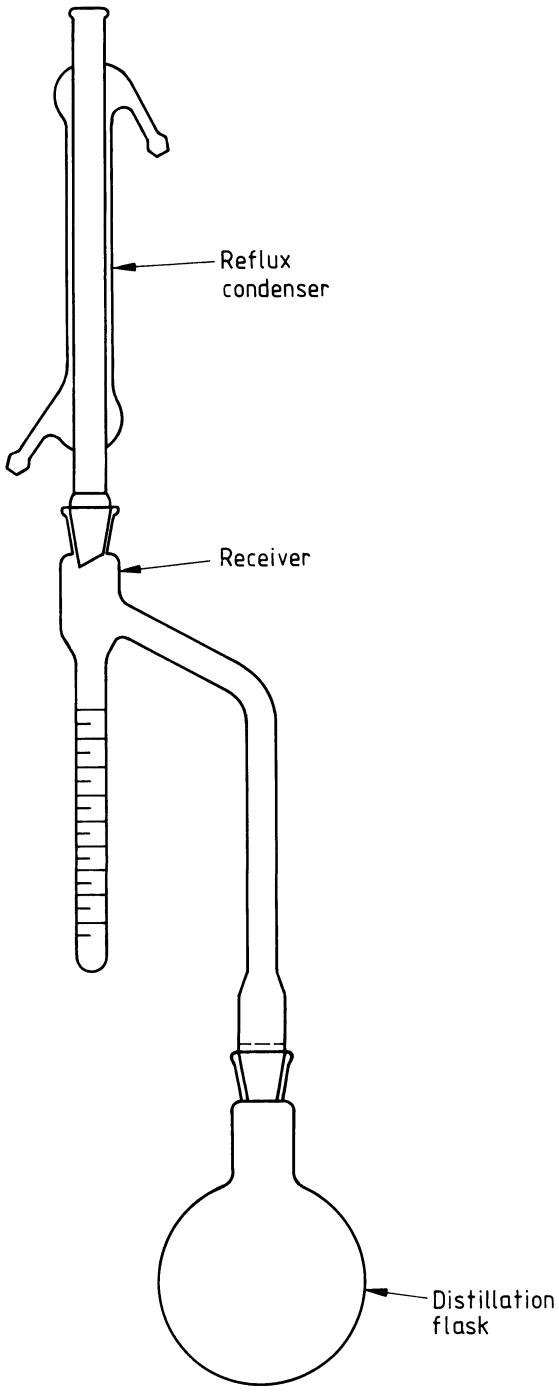
4.1 *Ordinary laboratory apparatus*

4.2 *Dean and Stark apparatus*, complying with type 1 of BS 756:1952, comprising the items described in **4.2.1** to **4.2.4**.

Before use, remove all traces of contamination from the graduated receiver (**4.2.2**) and from the interior of the reflux condenser (**4.2.3**) by washing them successively with, for example, a proprietary laboratory detergent, distilled water and acetone, then drying. Perfect cleanliness of the apparatus is essential.

NOTE This cleaning procedure ensures good water wetting characteristics.

4.2.1 *Distillation flask*, of glass, capacity 500 mL, with a ground-glass joint to fit the receiver (**4.2.2**) (see Figure 1).



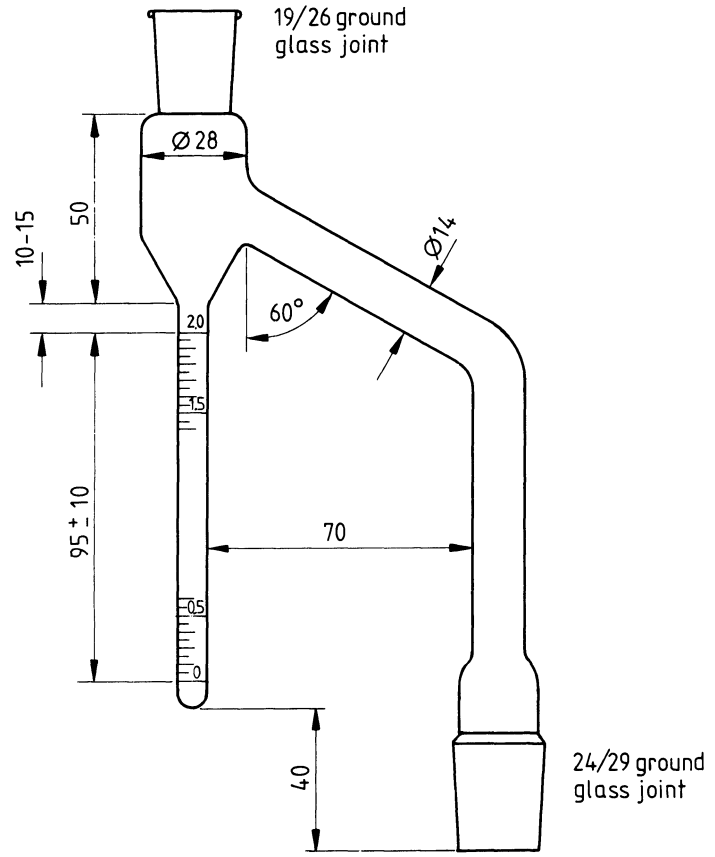
All dimensions are in millimetres.

Figure 1 — Typical assembly of Dean and Stark apparatus (4.2)

4.2.2 *Graduated receiver*, of capacity 2 mL, graduated at every 0.05 mL with a maximum error of ± 0.02 mL (see Figure 2). For cold ramming pastes with water contents in excess of 2 % (*m*/*m*) use a receiver of 10 mL capacity, graduated at every 0.2 mL with a maximum error of ± 0.06 mL.

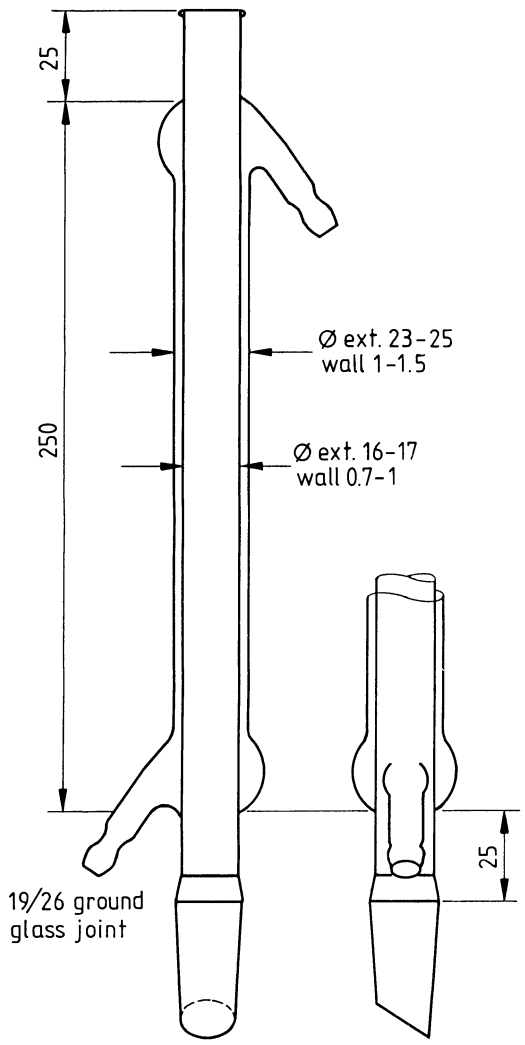
4.2.3 *Reflux condenser*, water-jacketted, with a ground-glass joint to fit the receiver (**4.2.2**) (see Figure 1 and Figure 3).

4.2.4 *Electrically-heated mantle*, or equivalent, to fit the distillation flask (**4.2.1**).



NOTE 1 All dimensions are in millimetres.
NOTE 2 This figure is equivalent to Figure 5 of BS 756:1952 and shows alternative connections to distillation vessel.

Figure 2 — 2 mL receiver (4.2)



NOTE 1 All dimensions are in millimetres.
NOTE 2 This figure is equivalent to Figure 3 of BS 756:1952.

Figure 3 — Typical reflux condenser (4.2.3)

5 Sampling

Prepare a representative laboratory sample of cold ramming paste in accordance with BS 6043-4.1.

6 Preparation of test sample

Take 100 ± 0.5 g from the laboratory sample (see clause **5**) of cold ramming paste in accordance with **5.5.3** of BS 6043-4.1:1991.

7 Procedure

Weigh the test sample (see clause **6**) to the nearest 0.1 g then transfer it quantitatively to the distillation flask (**4.2.1**). Add 100 mL of the xylene (**3.1**) to the distillation flask containing the test portion. Attach the flask to the receiver (**4.2.2**) and circulate cold water through the jacket of the condenser (**4.2.3**). Heat the flask gently until the test portion has dispersed in the xylene, then adjust the rate of heating so that the condensate falls from the end of the reflux condenser at a rate of two to five 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 collected water 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 its contents to cool to room temperature and read the volume of water to the nearest scale division.

8 Calculation and expression of results

Calculate the water content, W, as a percentage by mass (see note) from the following equation:

$$W = \frac{100\ M_2}{M_1}$$

where

M_1 is the mass of the test portion (in g);

M_2 is the mass of the water collected in the receiver (in g).

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

Express the result to the nearest 0.05 % (*m*/*m*); if condensate is seen in the receiver for a result which is less than 0.05 % (*m*/*m*), report the result as "trace — less than 0.05 % (*m*/*m*)".

9 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 6043-4.5:1991;

c) the results calculated and expressed in accordance with clause **8**;

d) any unusual features noted during the determination;

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

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Publication(s) referred to

BS 756, *Specification for Dean and Stark apparatus.*
BS 6043, *Methods of sampling and test for carbonaceous materials used in aluminium manufacture.*
BS 6043-4, *Cold ramming pastes.*
BS 6043-4.0, *General introduction*[1)].
BS 6043-4.1, *Methods of sampling.*

[1)] Referred to on the front cover only.

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