BRITISH STANDARD

BS 4140-4:
1986
ISO 804:1976

Methods of test for

Aluminium oxide —

Part 4: Preparation of sample solution by alkaline fusion

[ISO title: Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion]

NOTE It is recommended that this Part be read in conjunction with the general information given in BS 4140-0 "*General introduction*" which is issued separately.

UDC 546.623 – 31:620.1

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
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The Committees responsible for this British Standard are shown in Part 0.

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

Contents

National foreword

This Part of BS 4140 is identical with ISO 804:1976 "*Aluminium oxide primarily used for the production of aluminium — Preparation of solution for analysis — Method by alkaline fusion*", published by the International Organization for Standardization (ISO).

The method supersedes clause **4** of BS 4140:1967. Parts 1 to 7 of this standard collectively supersede BS 4140:1967, which is withdrawn.

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.

In British Standards it is current practice to use the symbol "L" for litre (and its submultiples) rather than "l".

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

Cross-references

International Standard	Corresponding British Standard
	BS 4140 *Methods of test for aluminium oxide*
ISO 802:1976	Part 1:1986 *Preparation and storage of test samples* (Identical)
ISO 2927:1973	Part 20:1980 *Sampling* (Identical)

NOTE The other International Standards listed in the Annex are for information only. Their correspondence with British Standards is summarized in BS 4140-0 "*General introduction*".

Additional information. In **6.2** and **6.4.1** it is preferable in the UK to use the fusion mixture containing boric acid for solutions subsequently to be used for the determination of silica (see BS 4140-5) as the results are found to be more reliable.

In the UK it is preferable to use polypropylene beakers in place of the polyethylene type referred to in **6.3**.

NOTE *Textual error*. In line 1 of clause **3** "protion" should read "portion".

This standard prescribes methods of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Part should indicate that the method of preparation complies with BS 4140-4:1986.

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 Scope and field of application

This International Standard specifies a method for the dissolution, by alkaline fusion, of aluminium oxide primarily used for the production of aluminium, in order to obtain a principal solution (solution P) for certain, determinations.

2 References

ISO 802, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples.*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling.*

3 Principle

Alkaline fusion of a test protion

— either with a mixture of sodium carbonate and boric acid,

— or with a mixture of sodium carbonate and sodium tetraborate.

Dissolution of the melt in an excess of nitric acid so that the final pH of the solution is approximately 1, after dilution to 500 ml, or approximately 0,4, after dilution to 250 ml.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 *Sodium carbonate*, anhydrous.

4.2 *Boric acid* (H_3BO_3).

4.3 *Sodium tetraborate*, anhydrous ($Na_2B_4O_7$).

4.4 *Aluminium oxide*, extra pure.

4.5 *Nitric acid*, approximately 8 N solution.

Dilute 540 ml of nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m*/*m*) solution, with water and dilute to 1 000 ml.

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Platinum dish*, flat bottomed, of diameter approximately 70 mm and depth approximately 35 mm, fitted with a platinum lid.

5.2 *Electric furnace*, capable of being controlled at 500 ± 50 °C.

5.3 *Electric furnace*, capable of being controlled between 1 000 and 1 025 °C.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, exactly 5 g of the test sample, dried at 300 °C (see **3.3** of ISO 802).

6.2 Fusion of test portion

Weigh into the platinum dish (**5.1**)

— either 12 g of the sodium carbonate (**4.1**) and 4 g of the boric acid (**4.2**),

— or 10,3 g of the sodium carbonate (**4.1**) and 3,3 g of the sodium tetraborate (**4.3**).

Mix thoroughly. Add the test portion (**6.1**) and carefully mix the whole, preferably with a platinum spatula. Cover the dish with its lid and place it in the electric furnace (**5.2**), controlled at 500 ± 50 °C, taking care to isolate it from the floor of the furnace by means of a support that cannot cause introduction of impurities. Maintain at 500 ± 50 °C until the reaction subsides. Then transfer the dish to the electric furnace (**5.3**), controlled between 1 000 and 1 025 °C, taking care to isolate it, as before, from the floor of the furnace.

Keep the dish in the furnace for 30 min. Ensure that a temperature of between 1 000 and 1 025 °C is maintained for a minimum of 20 min.

6.3 Preparation of the principal solution

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish, heating gently until dissolution of the melt.

After slight cooling, transfer the contents of the dish to a polyethylene beaker of suitable capacity containing 50 ml of the nitric acid solution (**4.5**).

NOTE In the cases where dissolution of the melt in nitric acid prior to obtaining the principal solution is unsuitable for the determination of certain elements, the dissolution should be made in an alternative appropriate acid which should be stated in the method of test of the element.

Dissolve any residue still adhering to the walls of the dish (consisting essentially of iron(III) oxide, calcium oxide and titanium oxide) with 20 ml of the nitric acid solution and transfer the solution obtained to the polyethylene beaker. Carefully wash both the dish and the lid with hot water and transfer the washings to the polyethylene beaker.

By careful washing, transfer the contents of the polyethylene beaker to a glass beaker. Heat for a few minutes at a temperature close to the boiling point until any remaining aluminium hydroxide is completely dissolved. Allow to cool slightly. When the solution is lukewarm, transfer quantitatively to a 250 or 500 ml one-mark volumetric flask, depending on the content of the elements to be determined. Allow to cool, dilute to the mark and mix.

NOTE When the solution obtained is opalescent, prepare a new solution, taking care to grind the aluminium oxide so as to obtain a particle size smaller than approximately 50 μm.

6.4 Preparation of the blank test solution

Prepare a blank test solution as indicated in **6.4.1** or **6.4.2**, proceeding, in accordance with the instructions given in the International Standard relating to the determination to be carried out, in the presence or absence of extra-pure aluminium oxide (**4.4**).

6.4.1 *Blank test solution containing extra-pure aluminium oxide*

Follow the same procedure as used for the test portion (see **6.2** and **6.3**), using exactly 5 g of extra-pure aluminium oxide (**4.4**), weighed to the nearest 0,001 g.

6.4.2 *Blank test solution free from extra-pure aluminium oxide*

Weigh into the same platinum dish (**5.1**)

— either 12 g of the sodium carbonate (**4.1**) and 4 g of the boric acid (**4.2**),

— or 10,3 g of the sodium carbonate (**4.1**) and 3,3 g of the sodium tetraborate (**4.3**).

Mix carefully. Cover the dish with its lid and place in the electric furnace (**5.2**), controlled at 500 ± 50 °C, taking care to isolate the dish from the floor of the furnace. Maintain at 500 ± 50 °C until the reaction subsides. Then transfer the dish to the electric furnace (**5.3**), controlled at a temperature of between 1 000 and 1 025 °C, taking care to isolate it, as before, from the floor of the furnace. Keep the dish in the furnace for a maximum of 5 min.

Remove the dish from the furnace and allow to cool in air. Add boiling water to the dish, heating gently until dissolution of the melt.

After slight cooling, transfer the contents of the dish to a polyethylene beaker of suitable capacity containing 30 ml of the nitric acid solution (**4.5**). Wash carefully both the dish and the lid with hot water and transfer the washings to the polyethylene beaker. Transfer carefully, by washing, the contents of the polyethylene beaker to a glass beaker. Heat for a few minutes at a temperature close to the boiling point. Allow to cool slightly and transfer quantitatively to either a 250 or a 500 ml one-mark volumetric flask, according to requirements.

Place 36,7 ml of the nitric acid solution (**4.5**) in the platinum dish and evaporate almost to dryness. Add a small amount of hot water to the dish. Add 3,3 ml of the nitric acid solution. Heat if necessary, and after cooling, transfer the solution quantitatively to the one-mark volumetric flask to which the contents of the glass beaker have previously been transferred. Allow to cool, dilute to the mark and mix.

NOTE This technique of partial evaporation is used to ensure the same pH values, both in the blank test and in the principal solution, in the presence of the same quantities of impurities introduced.

Annex ISO publications relating to aluminium oxide primarily used for the production of aluminium

ISO 802, *Preparation and storage of test samples.*

ISO 803, *Determination of loss of mass at 300 °C (conventional moisture).*

ISO 804, *Preparation of solution for analysis — Method by alkaline fusion.*

ISO 805, *Determination of iron content — 1,10-Phenanthroline photometric method.*

ISO 806, *Determination of loss of mass at 1 000 and 1 200 °C.*

ISO 900, *Determination of titanium content — Diantipyrylmethane photometric method.*

ISO 901, *Determination of absolute density — Pyknometer method.*

ISO 902, *Measurement of the angle of repose.*

ISO 903, *Determination of untamped density.*

ISO 1232, *Determination of silica content — Reduced molybdosilicate spectrophotometric method.*

ISO 1617, *Determination of sodium content — Flame emission spectrophotometric method.*

ISO 1618, *Determination of vanadium content — N-Benzoyl-N-phenylhydroxylamine photometric method.*

ISO 2069, *Determination of calcium content — Flame atomic absorption method.*

ISO/R 2070, *Determination of calcium content — Spectrophotometric method using naphthalhydroxamic acid.*

ISO 2071, *Determination of zinc content — Flame atomic absorption method.*

ISO/R 2072, *Determination of zinc content — PAN photometric method.*

ISO 2073, *Preparation of solution for analysis — Method by hydrochloric acid attack under pressure.*

ISO 2828, *Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method.*

ISO 2829, *Determination of phosphorus content — Reduced phosphomolybdate spectrophotometric method.*

ISO 2865, *Determination of boron content — Curcumin spectrophotometric method.*

ISO 2926, *Particle size analysis — Sieving method.*

ISO 2927, *Sampling.*

ISO 2961, *Determination of an adsorption index.*

ISO 3390, *Determination of manganese content — Flame atomic absorption method.*

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Publications referred to

See national foreword.

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