BS 4140 : Part 6 : 1980
ISO 900-1977
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British Standard Methods of test for

# Aluminium oxide

Part 6. Determination of titanium content

[ISO title : Aluminium oxide primarily used for the production of aluminium—Determination of titanium content—Diantipyrylmethane photometric method]

Méthodes d'essai de l'oxyde d'aluminium
Partie 6. Dosage du titane

Prufverfahren fur Aluminiumoxyd
Teil 6. Bestimmung des Titangehalts

NOTE. It is recommended that this Part be read in conjunction with the general information given in BS 4140 : Part 0 'General introduction' which is ussued separately and is free on request.

## National foreword

This Part of BS 4140 is identical with ISO 900 'Aluminium oxide primarily used for the production of aluminium — Determination of titanium content — Diantipyrylmethane photometric method', which replaces the tiron photometric method specified in ISO/R 900. The latter method corresponds to that specified in BS 4140 : 1967, clause **6**, the withdrawal of which is currently under discussion.

**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 British Standards identical with ISO 802 and ISO 804 referred to in the text. However, a related British Standard for both ISO 802 and ISO 804 is BS 4140 : 1967 'Methods of test for aluminium oxide'. The relevant clauses in that standard for the specific clauses referred to in the ISO text are as follows.

| ISO 802 | BS 4140 | Content |
|---|---|---|
| 3.3 | 2.3 | Preparation and storage of laboratory samples |
| 4.5 | 4.2 (3) | Preparation of sample solution |
| 6.1, 6.2 and 6.3 | 4.3.1 | |
| 6.4.2 | 4.3.2 | |

NOTE. Both of the above British Standard methods are to be revised and it is expected that they will be published as separate Parts of BS 4140, identical with ISO 802 and ISO 804 respectively.

The other international standards listed in the annex are for information only. Their correspondence with British Standards is summarized in BS 4140 : Part 0 'General introduction'.

## Additional information

*Water.* Water complying with the requirements of clause **4** is specified in BS 3978 'Water for laboratory use'.

WARNING *di*potassium hexafluorotitanate (**4.8.1**) and *di*potassium titanyl dioxalate dihydrate (**4.8.2**) are both toxic and irritant, particularly in the form of dust. Avoid breathing the dust and prevent contact with the eyes and skin.

*Printing error.* In line 7 of the note to **4.8.2**, 'pryosulphate' should read 'pyrosulphate'.

**This standard prescribes methods of test only, and should not be used or quoted as a specification defining limits of purity. Reference to the standard should indicate that the methods of test used comply with the requirements of BS 4140.**

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a diantipyrylmethane photometric method for the determination of the titanium content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a titanium content, expressed as titanium dioxide ($TiO_2$), equal to or greater than 0,001 % (*m/m*).

## 2 REFERENCES

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

ISO 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis – Method by alkaline fusion.*

## 3 PRINCIPLE

Alkaline fusion of a test portion and extraction of the fused mass with sulphuric acid solution. Formation of the titanium-diantipyrylmethane complex in approximately 4,6 N sulphuric acid solution.

Photometric measurement of the coloured complex at a wavelength of approximately 420 nm.

## 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** ($H_3BO_3$), or

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

**4.3 Sulphuric acid,** approximately 8 N solution.

**4.4 Sulphuric acid,** approximately 18 N solution.

**4.5 Ascorbic acid,** 30 g/l solution.

Use a freshly prepared solution.

**4.6 Copper(II) sulphate** pentahydrate ($CuSO_4.5H_2O$), 50 g/l solution.

**4.7 Diantipyrylmethane,** 50 g/l hydrochloric acid solution.

Dissolve 5 g of 4,4′-methylenediantipyrine (diantipyrylmethane) ($C_{23}H_{24}N_4O_2$) in 100 ml of approximately 1 N hydrochloric acid solution.

**4.8 Titanium,** standard solution corresponding to 0,400 g of titanium dioxide ($TiO_2$) per litre.

Prepare the solution by either of the following two methods :

**4.8.1** In a platinum dish of suitable capacity, weigh, to the nearest 0,000 1 g, 0,601 5 g of *di*potassium hexafluorotitanate ($K_2TiF_6$) previously dried at about 50 °C and cooled in a desiccator. Moisten the product with a few drops of water and then add 15 ml of sulphuric acid solution, $\rho$ approximately 1,84 g/ml. Evaporate carefully almost to dryness in a well-ventilated fume cupboard. Repeat the operation until the fluorine is completely expelled, using each time 5 to 6 ml of the same sulphuric acid solution. Finally, add 3 ml of the same sulphuric acid solution and heat until the residue is completely dissolved.

Allow to cool, immerse the dish and its contents in a beaker containing 95 ml of water and 5 ml of the same sulphuric acid solution and heat on a boiling water bath until the solution is completely clear.

Remove the platinum dish, wash it carefully with water, transfer the solution and the washings quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains the equivalent of 0,400 mg of $TiO_2$.

**4.8.2** Weigh, to the nearest 0,000 1 g, 0,886 5 g of *di*potassium titanyl dioxalate dihydrate [$K_2TiO(C_2O_4)_2.2H_2O$] and place in a Kjeldahl flask of capacity about 100 ml. Add 0,80 g of ammonium sulphate and 10 ml of sulphuric acid solution, $\rho$ approximately 1,84 g/ml. Heat carefully until the reaction subsides and then boil for 10 min.