BRITISH STANDARD

BS 4993-2:
1974

Methods of test for

Aluminium fluoride for industrial use —

Part 2: Determination of iron content

Confirmed
December 2011

UDC 661.862.361:546.623′161:543.432:546.72

Co-operating organizations

The Chemicals Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

British Steel Industry
Chemical Industries Association*
Department of Health and Social Security
Department of Trade and Industry
Fertilizer Manufacturers' Association Limited
Ministry of Agriculture, Fisheries and Food
National Sulphuric Acid Association
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association

The industrial organization marked with an asterisk in the list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Aluminium Federation
Society for Analytical Chemistry
British Ceramic Research Association
Society of Chemical Industry

This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on 28 February 1974

The following BSI references relate to the work on this standard:
Committee reference CIC/24
Drafts for comment/approval 71/53613 and 73/50651

ISBN 0 580 08095 1

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Foreword

This British Standard has been prepared under the authority of the Chemicals Industry Standards Committee in order to provide methods for the analysis of aluminium fluoride.

For some years the United Kingdom has participated in the work of preparing methods of test applicable to aluminium fluoride for industrial use, organized by Sub-Committee 7 "Alumina and related compounds" of Technical Committee 47 "Chemistry" of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as parts of this British Standard.

This part is based on the International Standard ISO 2368, "*Aluminium fluoride for industrial use — Determination of iron content — 1,10-phenanthroline photometric method*", modified to take into account the comments made by the United Kingdom during its development.

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 be in a form of words indicating that the methods of test used conform to BS 4993.

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

This Part of BS 4993 specifies a photometric method for the determination of the iron content of aluminium fluoride for industrial use.

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

2 Field of application

The method is applicable to the determination of iron contents, expressed as Fe_2O_3, greater than 0.020 %.

3 Principle

A test portion is dissolved by either alkaline or acid fusion, and preliminary reduction of iron (III) is carried out by means of hydroxylammonium chloride.

The iron (II)-1,10-phenanthroline complex is formed in a buffered medium (pH value between 3.5 and 4.2) and measured photometrically at a wavelength of 510 nm.

4 Reagents

The reagents used shall be of recognized analytical reagent quality. Water complying with the requirements of BS 3978 shall be used throughout.

4.1 *Sodium carbonate,* anhydrous.

4.2 *Boric acid,* (H_3BO_3).

4.3 *Potassium pyrosulphate,* ($K_2S_2O_7$), finely crushed.

4.4 *Nitric acid,* approximately 8N solution.

Dilute 540 ml of nitric acid, ρ 1.40 g/ml, approximately 68 % (m/m) solution, with water and dilute to 1 000 ml.

4.5 *Hydrochloric acid,* approximately 6N solution.

Dilute 515 ml of hydrochloric acid, ρ 1.18 g/ml, approximately 38 % (m/m) solution, with water and dilute to 1 000 ml.

4.6 *Hydroxylammonium chloride,* 10 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water and dilute to 1 000 ml.

4.7 *1,10-phenanthroline,* 2.5 g/l solution.

Dissolve 2.5 g of 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$) in water and dilute to 1 000 ml.

4.8 *Buffer solution.* Dissolve 272 g of sodium acetate trihydrate in approximately 500 ml of water and filter the solution.

Add 240 ml of glacial acetic acid, approximately 17.4N, and dilute to 1 000 ml.

4.9 *Sodium acetate,* 500 g/l solution.

Dissolve 50 g of sodium acetate trihydrate in water, dilute to 100 ml and filter.

4.10 *Acetic acid,* dilute solution.

Dilute 500 ml of glacial acetic acid, approximately 17.4N, with water and dilute to 1 000 ml.

4.11 *Standard iron solution,* corresponding to an Fe_2O_3 content of 0.200 g/l.

Dissolve a weighed quantity of high purity iron, of known iron content, in 20 ml of sulphuric acid (1 + 3), the mass of iron being such as will give 0.2000 g of iron (III) oxide (Fe_2O_3). Cool the solution, transfer it quantitatively to a 1 000 ml one-mark volumetric flask (BS 1792), dilute to the mark and mix.

1 ml of this standard solution contains 0.200 mg of Fe_2O_3.

4.12 *Standard iron solution,* corresponding to an Fe_2O_3 content of 0.010 g/l.

Transfer 50.0 ml of the standard solution (**4.11**) to a 1 000 ml one-mark volumetric flask (BS 1792), dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of Fe_2O_3.

Prepare this solution immediately before use.

4.13 *Indicator paper,* covering the pH range 3.5 to 4.2 at intervals of 0.2 units.

5 Apparatus

Ordinary laboratory apparatus and the following are required.

5.1 *Platinum dish,* flat-bottomed, approximately 80 mm in diameter and approximately 35 mm deep, fitted with a platinum lid.

5.2 *Electric furnace,* capable of being controlled at 550 ± 25 °C.

5.3 *Electric furnace,* capable of being controlled at 750 ± 25 °C.

5.4 *pH meter,* fitted with a glass electrode. Essential requirements are given in BS 2586, and in BS 3145 or BS 3422.

5.5 *Spectrophotometer,* or

5.6 *Photoelectric absorptiometer*

6 Procedure

6.1 Test portion. Weigh, to the nearest 0.001 g, 1 g of the dried test sample[1)].

6.2 Blank test

6.2.1 *Alkaline fusion.* Weigh 12 g of the sodium carbonate (**4.1**) and 4 g of the boric acid (**4.2**) into the platinum dish (**5.1**). Mix thoroughly, preferably with a platinum spatula. Cover the dish with its lid and place it in the electric furnace (**5.2**), controlled at 550 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support which avoids the risk of contamination. Maintain at 550 ± 25 °C until the reaction subsides.

Transfer the dish to the electric furnace (**5.3**), controlled at 750 ± 25 °C, isolating it, as before, from the furnace floor, and allow to remain for a maximum of 5 min.

Remove the dish from the furnace and allow to cool in the atmosphere. Add boiling water to the dish and heat gently until the contents are completely dissolved.

After cooling slightly, transfer the contents of the dish to a beaker of suitable capacity containing 20 ml of the nitric acid (**4.4**). Carefully wash the dish and lid with 18 ml of the nitric acid (**4.4**) and then with hot water, collecting the washings in the beaker, and simmer gently for a few minutes until completely dissolved.

Allow to cool slightly and transfer quantitatively to a one-mark volumetric flask (BS 1792) of the same capacity as that used for the preparation of the test solution (**6.4.1**). After cooling, dilute to the mark and mix thoroughly.

6.2.2 *Acid fusion.* Weigh 10 g of the potassium pyrosulphate (**4.3**) into the platinum dish (**5.1**). Cover the dish with its lid and place in the electric furnace (**5.3**), controlled at 750 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support which avoids the risk of contamination. Maintain at 750 ± 25 °C for a maximum of 10 min.

Remove the dish from the furnace and allow to cool in the atmosphere. Add 10 ml of water and 10 ml of the hydrochloric acid (**4.5**) to the dish. Rinse the lid and walls of the dish with hot water and simmer gently until the contents are completely dissolved.

Allow to cool slightly and transfer the solution quantitatively to a one-mark volumetric flask (BS 1792) of the same capacity as that used for the preparation of the test solution (**6.4.1**). After cooling, dilute to the mark and mix thoroughly.

6.3 Preparation of calibration curve

6.3.1 *Preparation of the standard matching solutions for photometric measurements with a 1 cm cell.* Into each of a series of eight 100 ml one-mark volumetric flasks transfer respectively the volumes of the standard iron solution (**4.12**) shown in the following table.

Volume of standard iron solution (4.12)	Corresponding mass of Fe_2O_3
ml	µg
0[a]	0
1.0	10
2.5	25
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250

[a] Compensation solution.

Add to each flask an amount of water sufficient to dilute to approximately 50 ml, then add 5 ml of the hydroxylammonium chloride solution (**4.6**), 5 ml of the 1,10-phenanthroline solution (**4.7**) and 25 ml of the buffer solution (**4.8**). Dilute to the mark and mix thoroughly.

6.3.2 *Photometric measurements.* After not less than 10 min, carry out the photometric measurements with the spectrophotometer (**5.5**), at a wavelength of 510 nm, or with the photoelectric absorptiometer (**5.6**), fitted with a suitable filter, after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart.* Prepare a calibration chart having, for example, the Fe_2O_3 contents, in milligrams per 100 ml of standard matching solutions, as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution*

6.4.1.1 *Alkaline fusion.* Weigh 12 g of the sodium carbonate (**4.1**) and 4 g of the boric acid (**4.2**) into the platinum dish (**5.1**). Mix thoroughly, preferably with a platinum spatula. Add the test portion (**6.1**) to the mixture and mix thoroughly. Cover the dish with its lid and place it in the electric furnace (**5.2**), controlled at 550 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support which avoids the risk of contamination.

[1)] Methods for the preparation and storage of test samples of aluminium fluoride are in course of preparation and will be published as a part of this standard.

Maintain at 550 ± 25 °C until the reaction subsides (about 30 min).

Transfer the dish to the electric furnace (**5.3**), controlled at 750 ± 25 °C, isolating it, as before, from the furnace floor. Keep the dish in the furnace for 30 min, making sure that the temperature of 750 ± 25 °C is maintained for at least 20 min.

Remove the dish from the furnace and allow to cool in the atmosphere. Add boiling water to the dish and heat gently until the contents are completely dissolved.

After cooling slightly, transfer the contents of the dish to a beaker of suitable capacity containing 20 ml of the nitric acid solution (**4.4**).

Dissolve the residue (which consists essentially of iron (III) oxide) adhering to the walls of the dish in 18 ml of the nitric acid (**4.4**) and carefully wash the dish and its lid with hot water, collecting the washings in the beaker.

Simmer the solution for a few minutes until completely dissolved. Allow to cool slightly and transfer quantitatively to a one-mark volumetric flask of either 250 ml or 500 ml capacity, according to the iron content to be determined.

After cooling, dilute to the mark and mix thoroughly.

6.4.1.2 *Acid fusion.* Weigh 10 g of the potassium pyrosulphate (**4.3**) into the platinum dish (**5.1**). Add the test portion (**6.1**) and mix carefully with a platinum spatula. Cover the dish with its lid and place in the electric furnace (**5.3**), controlled at 750 ± 25 °C, taking care to isolate it from the floor of the furnace by means of a support which avoids the risk of contamination. Maintain for 30 min at 750 ± 25 °C.

Remove the dish from the furnace and allow to cool in the atmosphere. Add 10 ml of water and 10 ml of the hydrochloric acid (**4.5**) to the dish. Rinse the lid and walls of the dish with hot water and simmer gently until the solid is completely dissolved. Allow to cool slightly and transfer the solution quantitatively to a one-mark volumetric flask of either 250 ml or 500 ml capacity, according to the iron content to be determined.

After cooling, dilute to the mark and mix.

6.4.2 *Colour reaction*

6.4.2.1 *Colour development.* Take an aliquot portion of the test solution (**6.4.1**) containing between 50 µg and 250 µg of Fe_2O_3 and place it in a 100 ml one-mark volumetric flask. Dilute to approximately 50 ml with water. Add 5 ml of the hydroxylammonium chloride solution (**4.6**), 5 ml of the 1,10-phenanthroline solution (**4.7**) and 25 ml of the buffer solution (**4.8**). Check the pH value of the solution using either the indicator paper (**4.13**) or the pH meter (**5.4**). This value should be between **3.5** and **4.2**; if not, adjust the pH value by slowly adding the required volume of the sodium acetate solution (**4.9**) or the acetic acid (**4.10**), as appropriate. Stir after each addition.

Dilute to the mark and mix.

6.4.2.2 *Photometric measurement.* After not less than 10 min carry out the photometric measurements on the sample solution and blank solution, following the procedure described in **6.3.2**, after having adjusted the instrument to zero absorbance against water.

7 Calculation

By reference to the calibration chart (see **6.3.3**), read the iron contents corresponding to the values of the photometric measurements.

Iron content, expressed as a percentage by mass of Fe_2O_3, is given by the formula:

$$(m_1 - m_2) \times \frac{D}{10 \times m_0}$$

where

D is the ratio of volume of sample solution to volume of aliquot portion taken for the colour reaction;

m_0 is the mass of the test portion (g);

m_1 is the mass of iron (III) oxide (Fe_2O_3) determined in the aliquot portion of the sample solution (mg);

m_2 is the mass of iron (III) oxide (Fe_2O_3) determined in the aliquot portion of the blank test solution (mg).

8 Note on procedure

Generally, the aliquot portion of the blank solution after treatment for photometric measurement shows a slight coloration. In this case, it is advisable to use it as the compensation solution.

In this case, the formula for calculation becomes:

$$m_1 \times \frac{D}{10 \times m_0}$$

where the symbols have the same meaning as those given in clause **7**.

Publications referred to

This standard makes reference to the following British Standards:

BS 1792, *One-mark volumetric flasks.*

BS 2586, *Glass electrodes for the measurement of pH.*

BS 3145, *Laboratory potentiometric pH meters.*

BS 3422, *Laboratory deflection pH meters.*

BS 3978, *Water for laboratory use.*

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