

Methods of test for Cryolite

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- British Steel Industry
- Chemical Industries Association*
- Department of Health and Social Security
- Department of Trade and Industry*
- Department of Trade and Industry — Laboratory of the Government Chemist*
- 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 Government departments and industrial organization marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

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

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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 natural and artificial cryolite.

In the drafting of this standard, account has been taken of methods adopted by the International Organization for Standardization (ISO), in the preparation of which the United Kingdom has been an active participant. It is intended to provide further methods, in the form of addenda to this standard, as the work of Technical Committee 47 — Chemistry, and, in particular, ISO/TC 47 Sub-committee 7 — Alumina and related compounds, advances.

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 5050.

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 14, 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 British Standard describes methods of test for natural and artificial cryolite having a NaF : AlF_3 ratio of between 3 and 1.7.

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

2 Preparation and storage of test samples

2.1 Introduction. This clause is based on ISO/R 1619, modified to take into account the comments made by the United Kingdom during its development.

The method described is intended for the preparation of two test samples from a laboratory sample which shall be representative of the material under test, the two samples being:

- a) a crude sample for moisture determination and certain physical tests, and
- b) a ground and dried analytical sample for other tests.

2.2 Preparation of crude sample. Thoroughly mix 3 kg of the laboratory sample and place approximately 300 g of it in an airtight container of such a capacity that it is nearly filled by the sample.

2.3 Preparation of analytical sample

2.3.1 Principle. The sample is ground, sieved until the whole passes through a 0.125 mm aperture sieve, well mixed and dried at 110 °C.

2.3.2 Procedure. Sieve approximately 100 g of the laboratory sample using a 0.125 mm aperture sieve, complying with the requirements of BS 410 and constructed of a material that cannot cause introduction of the impurities that are being determined. Select the sieve material in relation to the nature of the impurities to be determined. Grind the material remaining on the sieve in a corundum mortar and sieve again. Add the sieved part previously obtained and mix carefully. Repeat the crushing, sieving and mixing operations until none of the material remains on the sieve.

Place the sample thus prepared in a platinum dish and dry for 2 h in an electric oven, ventilated by convection and controlled at 110 ± 2 °C. Then allow to cool in a desiccator containing fresh phosphorus pentoxide and keep the dried sample in an airtight container of a capacity such that it is nearly filled by the sample.

3 Determination of silica content

3.1 Introduction. This clause is based on ISO/R 1620, modified to take into account the comments made by the United Kingdom during its development.

3.2 Principle. The sample is fused with a mixture of sodium carbonate and boric acid. The fused mass is dissolved in excess nitric acid so that the pH of the resultant solution is between 0.3 and 0.5 after dilution to 250 ml. The silicon is complexed with sodium molybdate in acid solution and the complex reduced. The absorbance of the reduced silicomolybdate complex is measured at a wavelength of about 815 nm.

The method is applicable provided that the P_2O_5 ¹⁾ content does not exceed 0.02 %.

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

NOTE Water produced by ion-exchange may not be suitable, because it may have too high a silica content.

3.3.1 Sodium carbonate, anhydrous.

3.3.2 Boric acid

3.3.3 Ascorbic acid, 20 g/l solution, freshly prepared.

3.3.4 Tartaric acid, 100 g/l solution.

3.3.5 Nitric acid, approximately 8N solution.

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

3.3.6 Sulphuric acid, approximately 16N solution. Carefully add 450 ml of sulphuric acid solution, ρ approximately 1.84 g/ml, about 96 % (m/m) solution, to about 500 ml of water. Cool and dilute to 1 000 ml.

3.3.7 Sodium molybdate, 195 g/l solution.

Dissolve 19.5 g of sodium molybdate dihydrate, $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$, in hot water, cool, dilute with water to 100 ml and mix. Store in a polyethylene bottle. If necessary, filter before use.

3.3.8 Stock silica solution, corresponding to 0.5 g of SiO_2 per litre.

¹⁾ The preparation of a method for the determination of phosphorus is being undertaken, and will be included in an addendum to this standard.

Into a platinum crucible weigh, to the nearest 0.001 g, 0.500 g of finely ground quartz, previously heated at 1 000 °C for 1 h and cooled in a desiccator. Add 5 g of the anhydrous sodium carbonate (3.3.1) to the crucible, mix thoroughly and heat until its contents are completely fused. Allow to cool, add hot water to the crucible and use gentle heat to complete solution of the contents. Transfer to a polyethylene beaker and allow to cool. Transfer to a 1 000 ml one-mark volumetric flask (BS 1792), dilute with water to the mark and mix. Transfer without delay to a polyethylene bottle.

3.3.9 Standard silica solution, corresponding to 5 mg of SiO₂ per litre.

Pipette 50.0 ml of the stock silica solution (3.3.8) into a 1 000 ml one-mark volumetric flask (BS 1792), dilute with water to the mark and mix. Pipette 50.0 ml of this solution into a 250 ml one-mark volumetric flask (BS 1792), dilute with water to the mark and mix. This solution shall be freshly prepared.

1 ml of this diluted solution $\equiv$ 5 µg of SiO₂.

3.4 Apparatus. Ordinary laboratory apparatus and the following are required.

3.4.1 pH meter, with glass and calomel electrode assembly. Essential performance requirements are given in BS 2586, BS 3145 and BS 3422.

3.4.2 Photoelectric absorptiometer or spectrophotometer, with 1 cm and 4 cm cells.

3.4.3 One-mark volumetric flask, 250 ml capacity, complying with the requirements of BS 1792.

3.4.4 Eight one-mark volumetric flasks, 100 ml capacity, complying with the requirements of BS 1792.

3.4.5 Platinum dish, approximately 100 mm diameter and 45 mm depth, fitted with a platinum lid.

3.5 Procedure

3.5.1 Preparation of calibration graph

3.5.1.1 Preparation of the blank solution.

Weigh 30 g of the sodium carbonate (3.3.1) and 10 g of the boric acid (3.3.2) into the platinum dish (3.4.5) and mix thoroughly. Cover the dish with its lid, place in an electric furnace, controlled at 550 ± 25 °C, and allow it to remain at this temperature until the reaction has ended. Transfer the dish to an electric furnace, controlled at 750 ± 25 °C, making sure that this temperature is maintained for 5 min.

Remove the dish and allow it to cool in air. Add about 125 ml of boiling water to the dish, heating gently until the material is dissolved. After cooling slightly, transfer the contents of the dish to a polyethylene beaker, of suitable capacity, containing 50 ml of the nitric acid solution (3.3.5), and carefully wash the dish and lid, first with 45 ml of the nitric acid solution and then with water, transferring the washings to the polyethylene beaker. Carefully transfer the contents of the beaker, by washing, to a glass beaker. Simmer gently for several minutes. Cool and transfer to the 250 ml one-mark volumetric flask (3.4.3) and dilute to the mark.

3.5.1.2 Colour development. Transfer 20 ml of the blank solution (3.5.1.1) into a beaker and add 15 ml of water, 15 ml of the standard silica solution (3.3.9) and 5 ml of the sodium molybdate solution (3.3.7). Mix thoroughly and, using the pH meter (3.4.1), adjust the pH of the solution to between 0.85 and 0.90 by the addition of the nitric acid solution (3.3.5). This pH value should relate to a solution volume of 65 ml. Note the volume of nitric acid solution added to adjust the pH value. Discard this solution.

Into a series of six of the 100 ml one-mark volumetric flasks (3.4.4), transfer 20 ml of the blank solution (3.5.1.1) and the volume of nitric acid solution determined above. Transfer amounts of the standard silica solution (3.3.9) equivalent to 0 µg (reagent blank) to 125 µg of silica, increasing by stages of 25 µg, and treat each solution in the following manner.

Dilute to 60 ml with water and then add 5 ml of the sodium molybdate solution (3.3.7). Mix thoroughly and allow to stand for 15 min to 25 min at 20 °C to 25 °C. Then add 5 ml of the tartaric acid solution (3.3.4), 11 ml of the sulphuric acid solution (3.3.6) and finally 2 ml of the ascorbic acid solution (3.3.3). Mix, dilute to the mark with water and mix again.

3.5.1.3 Photometric measurements. After an interval of 10 min to 40 min, measure the absorbance of each solution against water at the wavelength of maximum absorption (approximately 815 nm), using 4 cm cells, and prepare a calibration graph.

NOTE For samples of higher silica content, where it is necessary to use 1 cm cells in the determination, a calibration graph covering the appropriate range should be prepared using 1 cm cells.