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Cryolite (natural and artificial) — Determination of aluminium content — 8-hydroxyquinoline gravimetric method

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FOREWORD

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India
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Cryolite (natural and artificial) — Determination of aluminium content — 8-hydroxyquinoline gravimetric method

1 SCOPE

This International Standard specifies a gravimetric method, using 8-hydroxyquinoline, for the determination of aluminium content of natural and artificial cryolite.

2 FIELD OF APPLICATION

The method can be applied to the determination of the aluminium present in natural and artificial cryolite, provided that the Fe_2O_3 content does not exceed 0,15 %.

NOTE — The method can also be applied to both natural and artificial products with molar ratio of NaF to AlF_3 between approximately 3 and 1,7.

3 REFERENCE

ISO/R 1619, *Cryolite (natural and artificial) — Preparation and storage of test samples.*

4 PRINCIPLE

Solution of a test portion by acid fusion using potassium pyrosulphate and recovery by hydrochloric acid. Separation of interfering elements by precipitation of aluminium, with ammonium benzoate, in an acetic reducing medium. Dissolution of the precipitate and reprecipitation of aluminium as aluminium tri(quinolin-8 oxide) in an acetic buffer medium. Filtration, washing, drying of the precipitate at a temperature of 130 °C and weighing.

5 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

5.1 Potassium pyrosulphate ($K_2S_2O_7$), finely ground.

5.2 Hydrated sodium sulphite ($Na_2SO_3.7H_2O$).

5.3 Sulphuric acid, ρ 1,84 g/ml approximately, 96 % (*m/m*) solution.

5.4 Hydrochloric acid, ρ 1,19 g/ml approximately, 38 % (*m/m*) solution.

5.5 Ammonia, ρ 0,97 g/ml approximately, 6,7 % (*m/m*) solution.

5.6 Reducing buffer solution.

Dissolve 50 g of hydroxylammonium chloride and 50 g of ammonium chloride in a little water, add 50 ml of glacial acetic acid, ρ approximately 1,05 g/ml, dilute to 1 000 ml and mix.

5.7 Ammonium benzoate, 100 g/l solution.

Dissolve 100 g of ammonium benzoate in tepid water, add 0,001 g of thymol and, after cooling, dilute to 1 000 ml and mix.

5.8 Ammonium benzoate, acetic acid wash solution.

Dilute 100 ml of the ammonium benzoate solution (5.7) with 900 ml of water, add 20 ml of glacial acetic acid, ρ approximately 1,05 g/ml, and mix.

5.9 Hydrochloric acid, approximately ρ 1,05 g/ml approximately 10 % (*m/m*) solution.

5.10 Tartaric acid, 500 g/l solution.

5.11 Ammonia, ρ 0,90 g/ml approximately, 27,8 % (*m/m*) solution.

5.12 Acetic acid, ρ 1,01 g/ml (1,7 N approximately).

Dilute 100 ml of glacial acetic acid, ρ approximately 1,05 g/ml, with water, dilute to 1 000 ml and mix.

5.13 8-hydroxyquinoline (quinolin-8-ol), 20 g/l acetic acid solution.

Dissolve 20 g of 8-hydroxyquinoline (quinolin-8-ol) in 80 ml of glacial acetic acid, ρ approximately 1,05 g/ml, dilute to 1 000 ml and mix.

Store the solution in a dark glass bottle.

5.14 Ammonium acetate, 600 g/l solution.

5.15 Bromophenol blue, 2 g/l ethanolic solution.

Dissolve 0,20 g of bromophenol blue in 95 % (*V/V*) ethanol, dilute to 100 ml with the same ethanol and mix.

5.16 Neutral red, 0,5 g/l ethanolic solution.

Dissolve 0,05 g of neutral red in 95 % (*V/V*) ethanol, dilute to 100 ml with the same ethanol and mix.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Platinum dish, diameter approximately 75 mm, depth approximately 30 mm, fitted with a platinum lid.

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

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,1 mg, 0,500 g of the dried test sample (see 2.3 of ISO/R 1619).

7.2 Determination

7.2.1 *Preparation of the test solution.*

Weigh into the platinum dish (6.1) 10 g of the potassium pyrosulphate (5.1). Add the test portion (7.1) and carefully mix with a platinum spatula. Cover the dish with its lid and place in the electric furnace (6.2), controlled at a temperature of 750 ± 25 °C, taking care to isolate the dish from the floor of the furnace by means of a support from which there is no risk of introducing impurities. Maintain the temperature at 750 ± 25 °C for 30 min. Withdraw the dish from the furnace and leave to cool in air. Add 5 ml of the sulphuric acid solution (5.3) to the dish and heat on a hot plate until evolution of white sulphuric acid fumes ceases. Then add 10 ml of the hydrochloric acid solution (5.4) to the dish. Heat at a temperature near to boiling point until solution is complete, at the same time taking care to rinse the lid and the walls of the dish with warm water, collecting the whole quantity in the dish. Leave to cool slightly and transfer the solution quantitatively into a 250 ml one-mark volumetric flask.

After cooling, dilute to the mark and mix.

7.2.2 *Separation of aluminium from interfering elements*

Take 50,0 ml of the test solution (7.2.1) and place this in a beaker of suitable capacity (250 ml, for example). To the solution add 40 ml of water, 2 or 3 drops of the bromophenol blue solution (5.15) and neutralize with the ammonia solution (5.5) until the indicator turns violet. Then add 20 ml of the buffer solution (5.6) and 20 ml of the ammonium benzoate solution (5.7). Heat the solution to boiling point, while stirring, and simmer for 5 min, then filter through a medium-grade filter paper. Wash the beaker and the precipitate eight to ten times with the boiling wash solution (5.8) and discard the filtrate.

7.2.3 *Precipitation of aluminium tri(quinolin-8-oxide)*

Dissolve the precipitate on the filter by means of a boiling solution, prepared by mixing 50 ml of the hydrochloric acid solution (5.9) and 10 ml of the tartaric acid solution (5.10), added in small portions. Wash the filter with warm water and collect the solution and washing water in the beaker used for the first precipitation (see 7.2.2). Transfer the solution quantitatively into a beaker of suitable capacity (400 ml, for example) and add 1 g of the sodium sulphite (5.2), a few drops of the neutral red solution (5.16) and, slowly and cautiously, the ammonia solution (5.11) until the indicator turns yellow. Dilute the solution to approximately 200 ml and then heat to about 70 °C. Add the acetic acid solution (5.12) until the indicator turns red, then, while stirring, 40 ml of the 8-hydroxyquinoline solution (5.13) and finally 50 ml of the ammonium acetate solution (5.14). Leave to precipitate at approximately 70 °C for 30 min.

7.2.4 *Filtration, washing, drying and weighing of the aluminium tri(quinolin-8-oxide)*

Filter the precipitate on a sintered glass crucible (porosity between 3 and 15 μm), previously weighed after drying in an oven at 130 °C and cooled in a desiccator, applying a slight suction. Wash six to eight times with portions of 10 to 15 ml of tepid (60 to 70 °C) water. Dry at 130 °C for 2 h in an oven ventilated by convection.

Remove the crucible from the oven, place it in a desiccator and, after cooling to ambient temperature, weigh it. Check that the mass is constant.

8 EXPRESSION OF RESULTS

Aluminium content, expressed as Al, is given, as a percentage by mass, by the formula.

$$\frac{m_1 \times 0{,}058\,7 \times D \times 100}{m_0} = 5{,}87 \times D \frac{m_1}{m_0}$$

where

D is the ratio between the volume of the test solution (7.2.1) and the aliquot portion taken for the determination (7.2.2);

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the weighed aluminium tri(quinolin-8-oxide);

0,058 7 is the conversion factor from aluminium tri(quinolin-8-oxide) to aluminium.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

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