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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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International Standard ISO 2367 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in November 1971 by the Member Bodies of the following countries :

Austria	Israel	Switzerland
Belgium	Italy	Thailand
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France	Portugal	U.S.S.R.
Germany	Romania	Yugoslavia
Hungary	South Africa, Rep. of	
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The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

India
Poland

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.

$$\frac{m_1 \times 0,0587 \times D \times 100}{m_0} = 5,87 \times D \frac{m_1}{m_0}$$

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);

0,0587 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.

