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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
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The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

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 ($\text{K}_2\text{S}_2\text{O}_7$), finely ground.

5.2 Hydrated sodium sulphite ($\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$).

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.