TECHNICAL REPORT

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Cryolite, natural and artificial — Conventional test for evaluation of free fluorides content

Cryolithe, naturelle et artificielle — Essai conventionnel pour l'évaluation de la teneur en fluorures libres

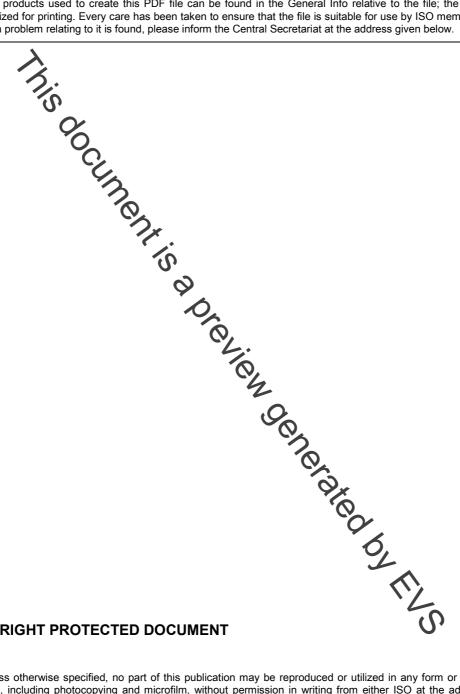


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Foreword

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In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an international Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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ISO/TR 4277 was prepared by Technical Committee ISO/TC 226, Materials for the production of primary aluminium.

This first edition cancels and replaces ISO 4277:1977, which it constitutes a minor revision.

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ISO 4277:1977 was withdrawn in 2004.

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Cryolite, natural and artificial — Conventional test for evaluation of free fluorides content

1 Scope

This Technical Report describes a conventional test for the evaluation of the free fluorides content of natural, artificial and recovered evolite.

This method is applicable to products having free fluorides content greater than 0,15 % (mass fraction) of AIF₃ or 0,4 % (mass fraction) of NAF.

2 Principle

A test portion is sintered with a known quantity of sodium fluoride at 790 \pm 20 °C for 20 minutes. Under these conditions, aluminium fluoride in excess of that required for the stoichiometric formula AIF $_3$ ·3NaF reacts with some of the sodium fluoride to form cryolite.

The ground sintered mass is extracted with boiling water and the solution is acidified with hydrochloric acid solution to a pH less than 3,7, followed by the ation of the excess sodium fluoride with standard volumetric thorium nitrate solution in the presence of alizarios, as indicator.

3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

- **3.1 Sodium fluoride**, anhydrous, dried at about 120 °C to constant mass in a platinum crucible and cooled in a desiccator.
- **3.2 Gelatine**, 3 % freshly prepared solution.
- **3.3 Hydrochloric acid**, approximately 0,1 N solution.
- 3.4 Sodium fluoride, 4,20 g/l standard solution (corresponding to 0,1 N)

Weigh, to the nearest 0,001 g, 4,20 g of the sodium fluoride (3.1). Transfer quantitatively to a 1 000 ml one-mark volumetric flask containing a little water and, after dissolution, dilute to the mark and mix. Transfer the solution to a suitable plastics bottle.

1 ml of this solution contains 4,20 mg of NaF.

3.5 Thorium nitrate, 0,1 N standard volumetric solution.

Weigh, to the nearest 0,001 g, 13,805 g of thorium nitrate tetrahydrate [Th(NO₃)₄·4H₂O], transfer quantitatively to a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.