TECHNICAL REPORT

ISO/TR 4277

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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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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, of which it constitutes a minor revision.

Introduction

This Technical Report was published in order to retain the method specified in ISO 4277:1977 in a publicly available standard.

ISO 4277:1977 was withdrawn in 2004.

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 cryolite.

This method is applicable to products having free fluorides content greater than 0,15 % (mass fraction) of AlF_3 or 0,4 % (mass fraction) of NaF.

2 Principle

A test portion is sintered with a known quantity of sodium fluoride at 790 ± 20 °C for 20 minutes. Under these conditions, aluminium fluoride in excess of that required for the stoichiometric formula $AlF_3 \cdot 3NaF$ 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 titration of the excess sodium fluoride with standard volumetric thorium nitrate solution in the presence of alizarin S 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 $[Th(NO_3)_4 \cdot 4H_2O]$, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.