International Standard 4193

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Aluminium and aluminium alloys — Determination of chromium content — Flame atomic absorption spectrometric method

Aluminium et alliages d'aluminium — Dosage du chrome — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Romania
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The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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Aluminium and aluminium alloys — Determination of chromium content — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the chromium content of aluminium and its alloys.

The method is applicable to products having chromium (Cr) contents between 0,003 and 0,6 % (m/m).[1)]

2 Principle

Dissolution of a test portion in hydrochloric acid and hydrogen peroxide. Aspiration of the solution into an air-acetylene or, preferably, dinitrogen oxide acetylene flame, and comparison of the absorbance of resonance energy of chromium by the test solution (wavelength of 357,9 nm normally) with that of standard solutions.

3 Reagents

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

3.1 Aluminium, extra pure (purity 99,99 %), free from chromium.

3.2 Hydrochloric acid, ϱ approximately 1,1 g/ml, about 20 % (m/m) or approximately 6 mol/l solution.

Dilute 500 ml of hydrochloric acid, ϱ approximately 1,19 g/ml, about 38 % (m/m) or approximately 12 mol/l solution, with 500 ml of water.

3.3 Hydrogen peroxide, about 30 % (m/m) solution.

3.4 Sulphuric acid, ϱ approximately 1,48 g/ml, about 58 % (m/m) or approximately 9 mol/l solution.

While stirring and cooling, add 50 ml of sulphuric acid, ϱ approximately 1,84 g/ml, about 96 % (m/m) or approximately 18 mol/l solution, to 40 ml of water. Again cool, dilute to the mark in a 100 ml volumetric flask, and mix.

3.5 Hydrofluoric acid, ϱ approximately 1,13 g/ml, about 40 % (m/m) solution.

3.6 Nitric acid, ϱ approximately 1,4 g/ml, about 68 % (m/m) or approximately 15 mol/l solution.

3.7 Aluminium, 20 g/l solution.

Weigh, to the nearest 0,01 g, 20 g of previously pickled, extra pure aluminium (3.1), place it in a 1 000 ml beaker and cover with a watch glass. Add, in small portions, 600 ml of the hydrochloric acid solution (3.2) and, if necessary, a drop of metallic mercury to assist the attack. If necessary, warm gently to aid the dissolution, and then add a few drops of the hydrogen peroxide solution (3.3). After cooling, quantitatively transfer the solution thus obtained to a 1 000 ml volumetric flask, dilute to the mark and mix.

3.8 Chromium, standard solution corresponding to 0,5 g of Cr per litre.

Weigh, to the nearest 0,001 g, 1,414 g of extra pure potassium dichromate ($\geqslant$ 99,9 % $K_2Cr_2O_7$), previously dried at 140 °C, and transfer it to a 400 ml tall-form beaker. Dissolve in 20 ml of water and 10 ml of the hydrochloric acid solution (3.2). Add, drop by drop, 10 ml of the hydrogen peroxide solution (3.3) and allow the solution to stand at room temperature until the yellow colour has completely disappeared (up to a half or a whole day, for instance) indicating that the chromium has been totally reduced to the trivalent state. Heat gently, without boiling, in order to decompose the remaining hydrogen peroxide. Transfer the solution to a 1 000 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,5 mg of chromium.

3.9 Chromium, standard solution corresponding to 0,025 g of Cr per litre.

Transfer 25,0 ml of the standard chromium solution (3.8) to a 500 ml volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,025 mg of chromium.

1) The method may also be used for higher chromium contents [up to 1,5 % (m/m) for instance].