
INTERNATIONAL STANDARD



3981

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Aluminium and aluminium alloys — Determination of nickel — Atomic absorption spectrophotometric method

Aluminium et alliages d'aluminium — Dosage du nickel — Méthode par spectrophotométrie d'absorption atomique

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FOREWORD

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International Standard ISO 3981 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*, and was circulated to the member bodies in November 1975.

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

Austria	Japan	Spain
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The member body of the following country expressed disapproval of the document on technical grounds :

Australia

Aluminium and aluminium alloys — Determination of nickel — Atomic absorption spectrophotometric method

1 SCOPE

This International Standard specifies a method for the determination of nickel in aluminium and its alloys by atomic absorption spectrophotometry.

2 FIELD OF APPLICATION

The method is applicable to nickel (Ni) contents between 0,005 and 3 % (*m/m*).

3 PRINCIPLE

Dissolution of a test portion in hydrochloric acid medium. Atomization of this solution into an air-acetylene flame of the atomic absorption apparatus.

Comparison of the absorption of the resonance energy of nickel (wavelength normally 232,0 nm) with that of calibration solutions.

4 REAGENTS

During the analysis, use only reagents of analytical grade and only distilled or de-ionized water.

4.1 Hydrochloric acid, ρ about 1,1 g/ml, solution about 20 % (*m/m*).

Dilute 500 ml of hydrochloric acid, ρ about 1,19 g/ml, solution about 38 % (*m/m*), with 500 ml of water.

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

4.3 Sulphuric acid, ρ about 1,48 g/ml, solution about 58 % (*m/m*).

While stirring and cooling, add 50 ml of sulphuric acid, ρ about 1,84 g/ml, solution about 96 % (*m/m*), to 40 ml of water. Cool again; then dilute to volume in a 100 ml volumetric flask and mix.

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

4.5 Nitric acid, ρ about 1,4 g/ml, solution about 68 % (*m/m*).

4.6 Aluminium, 20 g/l base solution.

Weigh, to the nearest 0,01 g, 20 g of aluminium free from nickel, previously pickled, and transfer to a 1 000 ml conical flask (for example). Add, in small portions, 600 ml of the

hydrochloric acid solution (4.1) and if necessary a drop of metallic mercury to facilitate the attack. If necessary, warm gently to assist the dissolution, then add some drops of the hydrogen peroxide solution (4.2), and boil for some minutes to remove the excess of hydrogen peroxide. After cooling, transfer the solution thus obtained quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

50 ml of this solution contains 1 g of aluminium and 30 ml of hydrochloric acid solution (4.1).

5 ml of this solution contains 0,1 g of aluminium and 3 ml of hydrochloric acid solution (4.1).

4.7 Nickel, standard solution corresponding to 0,125 g of Ni per litre.

Transfer $1 \pm 0,000$ 1 g of pure nickel (purity not less than 99,8 %) to a 400 ml beaker (for example). Add 50 ml of the hydrochloric acid solution (4.1) and cover with a watch glass. Then add some drops of the hydrogen peroxide solution (4.2) and warm gently, if necessary, until the nickel is completely dissolved. Then boil for some minutes to remove the excess of hydrogen peroxide, cool, make up to volume in a 1 000 ml volumetric flask and mix.

Transfer 25,0 ml of this solution to a 200 ml volumetric flask; make up to volume and mix.

1 ml of this standard solution contains 0,125 mg of Ni.

4.8 Nickel, standard solution corresponding to 0,025 g of Ni per litre.

Transfer 20,0 ml of the standard nickel solution (4.7) to a 100 ml volumetric flask; make up to volume with water and mix.

1 ml of this standard solution contains 0,025 mg of Ni.

5 APPARATUS

Usual laboratory equipment and :

5.1 Burette, graduated in 0,05 ml.

5.2 Atomic absorption spectrophotometer, fitted with a burner supplied by compressed air, and acetylene.

5.3 Nickel hollow-cathode lamp.