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# INTERNATIONAL STANDARD



# 3979

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## Aluminium and aluminium alloys — Determination of nickel — Spectrophotometric method using dimethylglyoxime

*Aluminium et alliages d'aluminium — Dosage du nickel — Méthode spectrophotométrique à la diméthylglyoxime*

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International Standard ISO 3979 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 :

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# Aluminium and aluminium alloys – Determination of nickel – Spectrophotometric method using dimethylglyoxime

## 1 SCOPE

This International Standard specifies a spectrophotometric method using dimethylglyoxime for the determination of nickel in aluminium and aluminium alloys.

## 2 FIELD OF APPLICATION

The method is applicable to nickel (Ni) contents between 0,001 and 3 % (m/m).

## 3 PRINCIPLE

Attack of a test portion by a solution of hydrochloric acid, in the presence of a small quantity of iron, to bring about the precipitation of the nickel, and filtration of the residue.

Dissolution of the residue, which contains all the nickel, by an acid mixture and complexing of the interfering elements by the addition, at a given pH value, of tartaric acid, hydroxylammonium chloride and sodium thiosulphate.

Formation of the nickel-dimethylglyoxime complex, extraction of this complex by chloroform and passage of the nickel of the nickel-dimethylglyoxime complex into aqueous solution by treatment with dilute hydrochloric acid.

Oxidation of the nickel with bromine water and development of the colour by the addition of dimethylglyoxime in an ammoniacal medium.

Photometric measurement of the coloured complex at the maximum of the absorption curve (wavelength about 445 nm).

## 4 REAGENTS

Throughout the analysis use only reagents of analytical grade and only distilled water or water of equivalent purity.

**4.1 Chloroform** ( $\text{CHCl}_3$ ),  $\rho$  about 1,48 g/ml.

**4.2 Nitric acid**,  $\rho$  about 1,40 g/ml, 68 % (m/m) solution or about 15 N.

**4.3 Hydrofluoric acid**,  $\rho$  about 1,05 g/ml, solution about 40 % (m/m).

**4.4 Ammonia solution**,  $\rho$  about 0,90 g/ml, 27 % (m/m) solution or about 14,4 N.

**4.5 Ammonia solution**,  $\rho$  about 0,99 g/ml, 0,85 % (m/m) solution or about 0,5 N.

Dilute 35 ml of ammonia solution (4.4), make up the volume to 1 000 ml and mix.

**4.6 Sulphuric acid**,  $\rho$  about 1,48 g/ml, 58 % (m/m) solution or about 18 N.

Add to about 400 ml of water, while stirring and cooling, 500 ml of sulphuric acid solution,  $\rho$  about 1,84 g/ml [solution about 96 % (m/m)].

After cooling, make up the volume to 1 000 ml and mix.

**4.7 Hydrochloric acid**,  $\rho$  about 1,1 g/ml, 20 % (m/m) solution or about 6 N.

Dilute 500 ml of hydrochloric acid solution,  $\rho$  about 1,19 g/ml, solution about 38 % (m/m), with water, make up the volume to 1 000 ml and mix.

**4.8 Hydrochloric acid**,  $\rho$  about 1,03 g/ml, 7,1 % (m/m) solution or about 2 N.

Dilute 170 ml of hydrochloric acid solution,  $\rho$  about 1,19 g/ml, 38 % (m/m) solution or about 12 N, with water, make up the volume to 1 000 ml and mix.

**4.9 Hydrochloric acid**,  $\rho$  about 1,01 g/ml, 1,81 % (m/m) solution or about 0,5 N.

Dilute 250,0 ml of hydrochloric acid solution (4.8), make up the volume to 1 000 ml and mix.

**4.10 Tartaric acid**, 300 g/l solution.

Dissolve 300 g of tartaric acid ( $\text{C}_4\text{H}_6\text{O}_6$ ) in water and heat to assist dissolution. Filter, if necessary, through a medium textured filter and collect the filtrate in a 1 000 ml volumetric flask. Wash with water and, after cooling to ambient temperature, make up to volume and mix.

**4.11 Acid mixture**

Mix, in a conveniently sized receptacle, 200 ml of the nitric acid solution (4.2) with 50 ml of the hydrochloric acid solution (4.7).