# INTERNATIONAL STANDARD ISO 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 :

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# 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,** $\rho$ about 1,1 g/ml, solution about 20 % (*m/m*).

Dilute 500 ml of hydrochloric acid, $\rho$ 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,** $\rho$ about 1,48 g/ml, solution about 58 % (*m/m*).

While stirring and cooling, add 50 ml of sulphuric acid, $\rho$ 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,** $\rho$ about 1,13 g/ml, solution about 40 % (*m/m*).

**4.5 Nitric acid,** $\rho$ 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 ± 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.**

## 6 SAMPLING

### 6.1 Laboratory sample1)

### 6.2 Test sample

Chips 1 mm thick or less shall be obtained by milling or drilling.

## 7 PROCEDURE

### 7.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample (6.2).

### 7.2 Establishment of the calibration curve

#### 7.2.1 *Preparation of the reference solutions*

#### 7.2.1.1 NICKEL CONTENTS BETWEEN 0,005 AND 0,3 %

To a series of ten 100 ml volumetric flasks transfer the volumes of the standard nickel solutions (4.7 and 4.8) shown in table 1, using the burette (5.1). Then add to each flask 50 ml of the aluminium solution (4.6), make up to volume and mix.

TABLE 1

| **Standard nickel solution (4.8)** | **Corresponding mass of nickel** | **Corresponding mass of aluminium** | **Nickel in sample** |
|---|---|---|---|
| ml | mg | g | % (*m/m*) |
| 0* | 0 | 1 | 0 |
| 2 | 0,05 | 1 | 0,005 |
| 4 | 0,10 | 1 | 0,010 |
| 12 | 0,30 | 1 | 0,030 |
| 20 | 0,50 | 1 | 0,050 |
| **Standard nickel solution (4.7)** | | | |
| ml | | | |
| 8 | 1,00 | 1 | 0,100 |
| 12 | 1,50 | 1 | 0,150 |
| 16 | 2,00 | 1 | 0,200 |
| 20 | 2,50 | 1 | 0,250 |
| 24 | 3,00 | 1 | 0,300 |

* Blank test of the reagents used for the calibration curve.

NOTE — If necessary, appropriately amplify the signal of the spectrophotometer for the first four terms of the curve, for example.

#### 7.2.1.2 NICKEL CONTENTS BETWEEN 0,25 AND 3 %

To a series of eight 100 ml volumetric flasks transfer the volumes of the standard nickel solution (4.7) shown in table 2, using the burette (5.1). Then add to each flask 5 ml of the aluminium solution (4.6), make up to volume and mix.

TABLE 2

| **Standard nickel solution (4.7)** | **Corresponding mass of nickel** | **Corresponding mass of aluminium** | **Nickel in sample** |
|---|---|---|---|
| ml | mg | g | % (*m/m*) |
| 0* | 0 | 0,1 | 0 |
| 2 | 0,25 | 0,1 | 0,25 |
| 4 | 0,50 | 0,1 | 0,50 |
| 8 | 1,00 | 0,1 | 1,00 |
| 12 | 1,50 | 0,1 | 1,50 |
| 16 | 2,00 | 0,1 | 2,00 |
| 20 | 2,50 | 0,1 | 2,50 |
| 24 | 3,00 | 0,1 | 3,00 |

* Blank test of the reagents used for the calibration curve.

#### 7.2.2 *Spectrophotometric measurements and plotting of the calibration curve*

Atomize the reference solutions into the flame and measure the intensity of the non-absorbed radiations at a wavelength of 232,0 nm, for example. Then plot the calibration curve.

### 7.3 Determination

#### 7.3.1 *Preparation of the test solution*

Transfer the test portion (7.1) to a 250 ml beaker, for example, and cover. Add about 30 to 40 ml of water, then, in small portions, 30 ml of the hydrochloric acid solution (4.1), warming gently, if necessary, to complete the dissolution. Add some drops of the hydrogen peroxide solution (4.2) and heat for about 10 min to remove the excess of hydrogen peroxide. Filter if necessary.

NOTES

1 If it is difficult to attack the sample with hydrochloric acid solution alone, add a drop of metallic mercury.

2 For silicon contents greater than 1 % proceed as follows :

Transfer the filter containing the silicon to a platinum crucible and ignite it, taking care that it does not inflame; then heat at about 550 °C. After cooling, add 2 ml of the sulphuric acid solution (4.3), 5 ml of the hydrofluoric acid solution (4.4) and, drop by drop, the nitric acid solution (4.5) until a clear solution is obtained (about 1 ml). Evaporate to dryness and heat again, at about 700 °C, for some minutes, to volatilize the silicon completely. After cooling, bring the non-volatile matter into solution with the least possible quantity of the hydrochloric acid solution (4.1), filter if necessary and add this filtrate quantitatively to the previous filtrate.

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1) The sampling of aluminium and aluminium alloys will form the subject of a future International Standard.

#### 7.3.1.1 NICKEL CONTENTS BETWEEN 0,005 AND 0,3 %

Transfer solution (7.3.1) quantitatively to a 100 ml volumetric flask, make up to volume and mix.

Use calibration curve 7.2.1.1.

#### 7.3.1.2 NICKEL CONTENTS BETWEEN 0,25 AND 3 %

Transfer solution (7.3.1) quantitatively to a 1 000 ml volumetric flask, make up to volume and mix.

Use calibration curve 7.2.1.2.

### 7.3.2 *Spectrophotometric measurements*

Measure the absorption of the test solution and of the blank test solution respectively following the same procedure used for the standard solutions.

## 8 EXPRESSION OF RESULTS

By means of the calibration curve, determine the quantity of nickel corresponding to the spectrophotometric measurements of the test solution and of the blank test solution.

The nickel (Ni) content is given, as a percentage by mass, by the formula

$$\frac{(m_1 - m_2) \times R}{m_0 \times 10}$$

where

$m_0$ is the mass, in grams, of the test portion (1 g);

$m_1$ is the mass, in milligrams, of nickel found in the test solution submitted to the spectrophotometric readings;

$m_2$ is the mass, in milligrams, of nickel found in the blank test solution;

$R$ is the ratio between the volume of the dilution of the test portion and the volume of the standard solutions taken (100 ml).

## 9 CONFIDENCE INTERVAL OF RESULTS

[Under study.]

## 10 TEST REPORT

The test report shall include the following information :

a) the reference of the method used;

b) the results and the form in which they are expressed;

c) any particular details noted during the test;

d) any operations not included in this International Standard or any optional operations.