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Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 1: Determination of cobalt, chromium, copper, iron and manganese

Alliages de nickel — Analyse par spectrométrie d'absorption atomique dans la flamme —
Partie 1: Détermination du cobalt, du chrome, du cuivre, du fer et du manganèse

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 155, *Nickel and nickel alloys*.

This second edition of ISO 7530-1 cancels and replaces ISO 7530-1:1990, ISO 7530-2:1990, ISO 7530-3:1990, ISO 7530-4:1990, ISO 7530-5:1990, and ISO 7530-6:1990, which have been technically revised.

ISO 7530 consists of the following parts, under the general title *Nickel alloys — Flame atomic absorption spectrometric analysis*:

- *Part 1: Determination of cobalt, chromium, copper, iron and manganese*
- *Part 7: Determination of aluminium content*
- *Part 8: Determination of silicon content*
- *Part 9: Determination of vanadium content*

Introduction

This part of ISO 7530 describes five flame atomic absorption spectrometric methods for the determination of cobalt, chromium, copper, iron, and manganese in nickel alloys.

Although the methods are described independently, it is possible to determine more than one element on a single test solution by adjustment of the sample weight and initial and subsequent dilutions.

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 1: Determination of cobalt, chromium, copper, iron and manganese

1 Scope

This part of ISO 7530 describes flame atomic absorption spectrometric methods for the determination of cobalt, chromium, copper, iron, and manganese in nickel alloys which can be dissolved in the nitric-hydrochloric acids mixture specified.

For each element, the method is applicable to the content range between 0,01 % and 4 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

3 Principle

Dissolution of a test portion in a nitric–hydrochloric acids mixture, evaporation of acids excess, and dissolution of the salts.

Addition of an ionization suppressant if necessary and dilution of the solution to a known volume.

Nebulization of the test solution after suitable dilution, if necessary, into an air/acetylene or a nitrous oxide/acetylene flame of an atomic absorption spectrometer.

Measurement of the absorption of the energy of the resonance line from the spectrum of the element being determined and comparison with that of calibration solutions of the same element.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Nitric acid, (HNO_3) ρ_{20} = 1,41 g/ml.

4.2 Nitric acid solution, 1 + 1.

Add 500 ml of nitric acid (4.1) to 500 ml of water.

4.3 Hydrochloric acid, (HCl) ρ_{20} = 1,18 g/ml.

4.4 Hydrochloric acid solution, 1 + 1.

Add 500 ml of hydrochloric acid (4.3) to 500 ml of water.

4.5 Nitric-hydrochloric acids mixture.

CAUTION — This acid mixture is highly corrosive and unstable. Noxious gas (chlorine) is liberated on standing. It shall be prepared and used in a fume cupboard and shall not be kept in a closed container.

Carefully mix one part of nitric acid (4.2) and three parts of hydrochloric acid (4.3). This mixture is not stable and should be prepared only as needed.

4.6 Strontium chloride solution.

Transfer 113,5 g of strontium chloride hexahydrate ($SrCl_2 . 6H_2O$) into a 600 ml beaker and dissolve in 400 ml of hot water (50 °C to 60 °C). Cool, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix.

4.7 Hydrogen peroxide, 300 g/l solution.

4.8 Cobalt standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of cobalt (Co ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid solution (4.4), cover with a watch glass, and heat gently until the cobalt is completely dissolved. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, add 35 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of cobalt.

4.9 Cobalt standard solution, 0,025 g/l.

Transfer 5,0 ml of cobalt standard solution (4.8) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

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

4.10 Chromium standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of chromium (Cr ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4), cover with a watch glass, and heat to complete dissolution. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, add 35 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of chromium.

4.11 Chromium standard solution, 0,100 g/l.

Transfer 20,0 ml of chromium standard solution (4.10) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of chromium.

4.12 Copper standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of copper (Cu ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 50 ml of nitric acid (4.2), cover with a watch glass, and allow to stand until the reaction ceases. Heat to complete dissolution, boil to remove nitrogen oxides, and evaporate just to dryness. Cool, add 25 ml of hydrochloric acid (4.3), and evaporate just to dryness. Add again 25 ml of hydrochloric acid (4.3) and repeat the evaporation. Dissolve the salts in 50 ml of hydrochloric acid (4.4) and heat if necessary. Cool, transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

1 ml of this solution contains 1,000 mg of copper.

4.13 Copper standard solution, 0,100 g/l.

Transfer 10,0 ml of copper standard solution (4.12) into a 100 ml one-mark volumetric flask and add 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of copper.

4.14 Copper standard solution, 0,025 g/l.

Transfer 5,0 ml of copper standard solution (4.12) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

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

4.15 Iron standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of iron (Fe ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4) and cover with a watch glass. Heat to initiate the reaction and complete dissolution. Cool to about 50 °C, cautiously add 1 ml of hydrogen peroxide (4.7), and bring to the boil to oxidize the iron. Cool, transfer into a 1 000 ml one-mark volumetric flask, and add 35 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of iron.

4.16 Iron standard solution, 0,050 g/l.

Transfer 10,0 ml of iron standard solution (4.15) into a 200 ml one-mark volumetric flask and add 10 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,050 mg of iron.

4.17 Manganese standard solution, 1,000 g/l.

Weigh 1 g ± 0,001 g of manganese (Mn ≥ 99,9 %) and transfer it into a 400 ml beaker. Add 30 ml of hydrochloric acid (4.4) and 2 ml of nitric acid (4.1). Cover with a watch glass and heat to initiate the reaction and complete dissolution. Add 0,5 ml of hydrogen peroxide (4.7). Cool, transfer into a 1 000 ml one-mark volumetric flask, and add 50 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

1 ml of this solution contains 1,000 mg of manganese.

4.18 Manganese standard solution, 0,100 g/l.

Transfer 10,0 ml of manganese standard solution (4.17) into a 100 ml one-mark volumetric flask and add 2,5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,100 mg of manganese.

4.19 Manganese standard solution, 0,010 g/l.

Transfer 5,0 ml of manganese standard solution (4.17) into a 500 ml one-mark volumetric flask and add 12,5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,010 mg of manganese.

5 Apparatus

All volumetric glassware shall be class A in accordance with ISO 385, ISO 648, or ISO 1042 as appropriate.

Ordinary laboratory apparatus and the following.

5.1 Atomic absorption spectrometer.

WARNING — The manufacturer's recommendations should be closely followed and particular attention is drawn to the following safety points.

a) The explosive nature of acetylene and regulations concerning its use.

b) The need to shield the eyes of the operator from ultraviolet radiation by means of tinted glass.

c) The need to keep the burner clear of deposits because a badly clogged burner can cause a flashback.

d) The need to ensure that the liquid trap is filled with water.

e) The need to spray distilled water between the test solutions, blank solution, and/or calibration solutions.

The atomic absorption spectrometer used shall be checked according to the instrument performance parameters given in Annex A.

The instrument shall be equipped with burners suitable for both an air/acetylene and a nitrous oxide/acetylene flame.

The instrument should be suitable for operating with single or multi-element element hollow cathode lamps powered at currents recommended by the manufacturer.

6 Sampling and sample preparation

Sampling and sample preparation shall be carried out by normal agreed procedures or, in case of dispute, by appropriate National or International Standards.

The laboratory sample normally is in the form of millings or drillings and no further preparation of the sample is necessary.

If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned with high purity acetone and then dried in air.

If the laboratory sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffling.

7 Procedure

7.1 Test portion

Weigh to the nearest 1 mg, approximately 1,0 g of the test sample.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

7.3 Dissolution

Transfer the test portion (7.1) into a 600 ml beaker. Add 20 ml of the nitric–hydrochloric acids mixture (4.5) and cover with a watch glass. Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, some adjustment of the acids mixture may be required. Add hydrochloric acid (4.3) in 1 ml increments and continue heating to dissolve the sample.

Using a low heat, evaporate the solution just to dryness. Do not bake. Cool to about 50 °C, add 25 ml of hydrochloric acid (4.3), and evaporate again to dryness. Add a new 25 ml of hydrochloric acid (4.3) portion and repeat the evaporation.

NOTE Some alloys having high copper contents can be easily dissolved in nitric acid solution (4.2). For some alloys, an acid mixture containing 30 ml hydrochloric acid (4.3) and 2 ml of nitric acid (4.1) is more effective.

If sample inhomogeneity is suspected, a larger mass of sample may be taken for analysis. However, an aliquot portion corresponding to a 1 g sample shall be taken from this test solution and processed in accordance with the procedure given.

7.4 Determination of cobalt

7.4.1 Preparation of the test solution

Cool the solution (7.3) to about 50 °C, add 5 ml of hydrochloric acid (4.3), 20 ml of water, and heat to dissolve the salts. Cool to room temperature.

Transfer the solution quantitatively into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

7.4.1.1 Cobalt contents between 0,01 % and 0,10 %

Transfer 25 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 4 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

7.4.1.2 Cobalt contents between 0,1 % and 1 %

Transfer 2,5 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

7.4.1.3 Cobalt contents between 1 % and 4 %

Transfer 5 ml of the test solution (7.4.1) into a 100 ml one-mark volumetric flask, dilute to the mark with water, and mix well.

Transfer 10 ml of this solution into a 100 ml one-mark volumetric flask, add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3), dilute to the mark with water, and mix well.

7.4.2 Preparation of the calibration solutions

7.4.2.1 Cobalt contents between 0,01 % and 0,10 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 1. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Table 1 — Calibration for cobalt contents between 0,01 % and 0,10 % percentage (mass fraction)

Cobalt standard solution volume (4.9) (ml)	Corresponding cobalt mass (mg)	Corresponding content of cobalt in the sample percentage (mass fraction)
0	0	0
1	0,025	0,01
2	0,050	0,02
4	0,100	0,04
6	0,150	0,06
8	0,200	0,08
10	0,250	0,10

7.4.2.2 Cobalt contents between 0,1 % and 1 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 2. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.

Table 2 — Calibration for cobalt contents between 0,1 % and 1 % percentage (mass fraction)

Cobalt standard solution volume (4.9) (ml)	Corresponding cobalt mass (mg)	Corresponding content of cobalt in the sample percentage (mass fraction)
0	0	0
1	0,025	0,1
2	0,050	0,2
4	0,100	0,4
6	0,150	0,6
8	0,200	0,8
10	0,250	1,0

7.4.2.3 Cobalt contents between 1 % and 4 %

Into each of a series of 100 ml one-mark volumetric flasks, introduce the volumes of cobalt standard solution (4.9) shown in Table 3. Add 4 ml of strontium chloride solution (4.6) and 5 ml of hydrochloric acid (4.3). Dilute to the mark with water and mix well.