
**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*

get full document from standards.iteh.ai



Sample Document

get full document from standards.iteh.ai



COPYRIGHT PROTECTED DOCUMENT

© ISO 2015, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	1
5 Apparatus	4
6 Sampling and sample preparation	4
7 Procedure	5
7.1 Test portion	5
7.2 Blank test	5
7.3 Dissolution	5
7.4 Determination of cobalt	5
7.4.1 Preparation of the test solution	5
7.4.2 Preparation of the calibration solutions	6
7.5 Determination of chromium	7
7.5.1 Preparation of the test solution	7
7.5.2 Preparation of the calibration solutions	8
7.6 Determination of copper	9
7.6.1 Preparation of the test solution	9
7.6.2 Preparation of the calibration solutions	9
7.7 Determination of iron	11
7.7.1 Preparation of the test solution	11
7.7.2 Preparation of the calibration solutions	12
7.8 Determination of manganese	13
7.8.1 Preparation of the test solution	13
7.8.2 Preparation of the calibration solutions	13
7.9 Adjustment of atomic absorption spectrometer	15
7.10 Spectrometric measurements	15
7.11 Calibration curves	16
7.12 Expression of results	16
7.13 Precision	16
7.13.1 General	16
7.13.2 Precision for cobalt	17
7.13.3 Precision for chromium	18
7.13.4 Precision for copper	18
7.13.5 Precision for iron	18
7.13.6 Precision for manganese	18
8 Test report	19
Annex A (normative) Procedures for checking the instrumental criteria	20
Bibliography	23

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

Sample Document

get full document from standards.iteh.ai

Sample Document

get full document from standards.iteh.ai

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) $\rho_{20} = 1,41$ 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) $\rho_{20} = 1,18$ 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 ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) 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 $1 \text{ g} \pm 0,001 \text{ g}$ of cobalt ($\text{Co} \geq 99,9 \%$) 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 $1 \text{ g} \pm 0,001 \text{ g}$ of chromium ($\text{Cr} \geq 99,9 \%$) 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 $1\text{ g} \pm 0,001\text{ g}$ of copper ($\text{Cu} \geq 99,9\%$) 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 $1\text{ g} \pm 0,001\text{ g}$ of iron ($\text{Fe} \geq 99,9\%$) 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 $50\text{ }^{\circ}\text{C}$, 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 $1\text{ g} \pm 0,001\text{ g}$ of manganese ($\text{Mn} \geq 99,9\%$) 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.