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# Nickel alloys — Determination of chromium content — Potentiometric titration method with ammonium iron(II) sulfate

*Alliages de nickel — Détermination du chrome — Méthode par titrage potentiométrique avec du sulfate de fer(II) et d'ammonium*



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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

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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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This document was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

This second edition cancels and replaces the first edition (ISO 7529:1989), which has been technically revised with the following changes:



— the scope has been modified;

— Table 1 has been modified.

 

# Nickel alloys — Determination of chromium content — Potentiometric titration method with ammonium iron(II) sulfate

## 1 Scope

This document specifies a potentiometric titration method for the determination of chromium content in nickel alloys which do not contain insoluble carbides and which have a vanadium content less than a mass fraction of 0,2 %. The method is applicable to chromium contents between a mass fraction of 5 % to a mass fraction of 22 %.

Vanadium, which can be present as an impurity in the alloy, will give a positive bias interference. However, at a level of a mass fraction of 0,2 %, this bias is equivalent to a mass fraction of 0,068 % chromium, which is about half the reproducibility of the method.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

## 4 Principle

Dissolution of a test portion in a nitric/hydrochloric acids mixture, and evaporation to fumes of sulfuric acid.

Dissolution of the salts in water and oxidation of chromium to chromium(VI), with ammonium peroxydisulfate using silver nitrate as a catalyst.

Removal of excess peroxydisulfate by boiling, and reduction of manganese(VII) with hydrochloric acid.

Titration of chromium(VI) with ammonium iron(II) sulfate using potentiometric end-point detection.

## 5 Reagents

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

**5.1 Hydrochloric acid**, $\rho_{20}$ = 1,19 g/ml.

**5.2 Hydrochloric acid**, $\rho_{20}$ = 1,19 g/ml, diluted 1 + 3.

**5.3 Nitric acid**, $\rho_{20}$ = 1,41 g/ml.

**5.4 Sulfuric acid**, $\rho_{20}$ = 1,84 g/ml, diluted 1 + 1.

**5.5 Silver nitrate** ($AgNO_3$), 15 g/l solution.

**5.6 Ammonium peroxydisulfate** [$(NH_4)_2S_2O_8$].

**5.7 Nitric/hydrochloric acids**, mixture.

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

Carefully mix 25 ml of nitric acid (5.3) and 75 ml of hydrochloric acid (5.1).

This mixture is not stable and shall be prepared just before use.

**5.8 Potassium dichromate,** standard solution, $c(1/6\ K_2Cr_2O_7)$ = 0,100 mol/l.

Dissolve exactly 4,903 g of potassium dichromate ($K_2Cr_2O_7$, 99,95 % minimum purity), previously dried at 105 °C for 1 h, in 500 ml of water.

Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

**5.9 Ammonium iron(II) sulfate,** standard solution, $c[(NH_4)_2Fe(SO_4)_2]$ = 0,1 mol/l.

**5.9.1 Preparation**

Dissolve 40 g of ammonium iron(II) sulfate hexahydrate [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$] in 400 ml of water. Add slowly, with constant stirring, 100 ml of sulfuric acid (5.4).

Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

**5.9.2 Standardization**

Add, using a burette, 40,0 ml of the potassium dichromate solution (5.8) to 200 ml of water in a 400 ml tall-form beaker. Add 10 ml of sulfuric acid (5.4), 5 ml of the silver nitrate solution (5.5) and 5 ml of hydrochloric acid (5.2). Titrate this solution potentiometrically with the ammonium iron(II) sulfate solution (5.9), as specified in 8.2.

The real concentration, $c$, of the ammonium iron(II) sulfate solution (5.9), expressed in moles of iron per litre, is given by Formula (1):

$$\frac{V_1}{V_2} \times 0{,}1 \qquad (1)$$

where

$V_1$ is the volume, in millimetres, of the potassium dichromate solution (5.8) taken for the standardization (= 40,0 ml);

$V_2$ is the volume, in millimetres, of ammonium iron(II) sulfate solution (5.9), used for the titration.