INTERNATIONAL STANDARD

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

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

Reference number
ISO 7529 : 1989 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7529 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Annex A of this International Standard is for information only.

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

1 Scope

This International Standard specifies a potentiometric titration method for the determination of 1 % (*m/m*) to 25 % (*m/m*) chromium content in nickel alloys which do not contain insoluble chromium carbides, and which have a vanadium content of less than 0,2 % (*m/m*). Typical compositions of some nickel alloys are given in annex A.

Vanadium, which may be present as an impurity in the alloy, will give a positive bias interference. However, at a level of 0,2 % (*m/m*) this bias is equivalent to 0,068 % (*m/m*) chromium, which is about half the reproducibility of the method. See clause 9.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1 : General requirements.*

ISO 648 : 1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Principle

Dissolution of a test portion in a nitric/hydrochloric acid 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) by hydrochloric acid.

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

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 Hydrochloric acid, ϱ_{20} = 1,18 g/ml.

4.2 Hydrochloric acid, ϱ_{20} = 1,18 g/ml, diluted 1 + 3.

4.3 Nitric acid, ϱ_{20} = 1,41 g/ml.

4.4 Sulfuric acid, ϱ_{20} = 1,83 g/ml, diluted 1 + 1.

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

4.6 Ammonium peroxydisulfate [$(NH_4)_2S_2O_8$].

4.7 Nitric/hydrochloric acid, 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 cupboard and shall not be kept in a closed container.

Carefully mix 25 ml of nitric acid (ϱ_{20} = 1,41 g/ml) and 75 ml of hydrochloric acid (ϱ_{20} = 1,18 g/ml). This mixture is not stable and shall be prepared just before use.

4.8 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.