INTERNATIONAL STANDARD

ISO 11400

First edition
1992-09-15

Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method

Nickel, ferronickel et alliages de nickel — Dosage du phosphore — Méthode par spectrométrie d'absorption moléculaire au phosphovanadomolybdate

Reference number
ISO 11400:1992(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11400 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Sub-Committee SC 4, *Analysis of nickel alloys*.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method

1 Scope

This International Standard specifies a molecular absorption spectrometric method for the determination of the phosphorus content in nickel, ferronickel and nickel base alloys in the range of 0,000 5 % (*m*/*m*) to 0,05 % (*m*/*m*).

Arsenic, chromium, hafnium, niobium, silicon, tantalum, titanium and tungsten interfere, but the interferences can be avoided by complexation or volatilisation (for Cr). The lowest phosphorus content [0,000 5 % (*m*/*m*)] can only be reached in samples with low contents of the interfering elements.

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 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 mixture of nitric and hydrochloric acids. Fuming with perchloric acid in a PFA or PTFE beaker and removal of chromium as volatile chromylchloride.

Complexation of silicon and the refractory elements with fluoride ions.

Conversion of phosphorus to phosphovanadomolybdic acid in a perchloric and nitric acid solution.

Extraction of phosphovanadomolybdic acid into 4-methyl-2-pentanone with citric acid present to complex arsenic.

4 Reagents

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

Verify by blank tests (7.6) that the relevant reagents are free from phophorus. Lots giving high blank values are unsuitable and should not be used. The blank value should be below 0,000 5 % (*m*/*m*), calculated for 1 g of sample.

4.1 Nitric acid, ρ_{20} = 1,41 g/ml, diluted 1 + 4.

4.2 Hydrofluoric acid, [40 % (*m*/*m*)], ρ_{20} = 1,14 g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water and seek medical advice.

4.3 Citric acid, solution.

Dissolve 500 g of citric acid monohydrate ($H_8C_6O_7.H_2O$) in water, dilute to 1 000 ml and mix.

4.4 4-methyl-2-pentanone (methyl isobutyl ketone).