

INTERNATIONAL STANDARD

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Nickel alloys — Determination of phosphorus content — Molybdenum blue molecular absorption spectrometric method

*Alliages de nickel — Dosage du phosphore — Méthode par
spectrométrie d'absorption moléculaire au bleu de molybdène*



Reference number
ISO 9388:1992(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 9388 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Sub-Committee SC 4, *Analysis of nickel alloys*.

Annex A of this International Standard is for information only.

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Nickel alloys — Determination of phosphorus content — Molybdenum blue molecular absorption spectrometric method

1 Scope

This International Standard specifies a molecular absorption spectrometric method for the determination of the phosphorus content of nickel alloys in the range 0,001 % (m/m) to 0,025 % (m/m). Typical compositions of some nickel alloys are given in annex A.

Chromium(III) and silica cause interference which is eliminated in the procedure. 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 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 nitric and hydrochloric acids. Removal of hydrochloric acid by fuming with sulfuric acid, and oxidation of chromium(III) to chromium(VI) with ammonium peroxydisulfate.

Precipitation of iron(III) phosphate and redissolution of the precipitate in a hydrofluoric-nitric acid mixture.

Addition of boric, tartaric, and sulfamic acids to complex free fluoride, eliminate arsenic interference, and remove oxides of nitrogen.

Formation and extraction of molybdophosphoric acid into isobutyl alcohol, reduction of the heteropoly acid to molybdenum blue and back-extraction into an aqueous phase.

Measurement of the absorbance of the aqueous solution at 700 nm.

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/hydrochloric acid, mixture.

WARNING — This acid mixture is highly corrosive and unstable. Noxious gases (chlorine) are 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 ($\rho_{20} = 1,41$ g/ml) and 125 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml). This mixture should be prepared just before use.

4.2 Hydrofluoric acid, $\rho_{20} = 1,15$ g/ml, 48 % (m/m) HF.

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

Slowly, and with constant stirring, add 100 ml of sulfuric acid to 100 ml of water.