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Nickel, ferronickels and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method

Nickel, ferronickels et alliages de nickel — Détermination de la teneur en phosphore — Méthode spectrophotométrique au phosphovanadomolybdate

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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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11400:1992), which has been technically revised. The main changes are as follows:

- the title has been reworded;
- Clause 2 has been updated;
- Clause 3 has been added and the subsequent clauses have been renumbered;
- Clause 4 (the former Clause 3) has been partially reworded;
- in Clause 5 (the former Clause 4): the references of several reagents have been added;
- Clause 6 (the former Clause 5) has been partially reworded, namely 6.1;
- Clause 8 (the former Clause 7) has been partially rearranged regarding the numbering of the paragraphs;
- in 8.4 (the former 7.7), Table 2 has been added;
- 8.5, "Check samples" has replaced 7.8, "Number of determinations";
- the Bibliography has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

INTERNATIONAL STANDARD **ISO 11400:2021(E)**

Nickel, ferronickels and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method

1 Scope

This document specifies a spectrophotometric method for the determination of the phosphorus content in nickel, ferronickels and nickel alloys between 0,000 5 % (by mass) and 0,05 % (by mass).

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

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 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:

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

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

4 Principle

Dissolution of a test portion in a mixture of nitric and hydrochloric acids. Fuming with perchloric acid in a perfluoroalkoxy copolymer (PFA) or polytetrafluoroethylene (PTFE) beaker and removal of chromium as volatile chromylchloride.

Complexation of silicon and refractory elements with hydrofluoric acid.

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

After addition of citric acid to complex arsenic, extraction of phosphovanadomolybdic acid with 4-methyl-2-pentanone.

Spectrophotometric measurement at a wavelength of 355 nm.

5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity (see ISO 3696).

Verify by blank tests (see 8.2) that all reagents are free from phosphorus. Lots giving high blank values are unsuitable and should not be used. The blank value should be below 0,000 5 % (by mass), calculated for 1 g of test portion.

5.1 Nitric acid, ρ_{20} approximately 1,41 g/ml.

5.2 Nitric acid solution, 1 + 4.

Carefully add 100 ml of nitric acid (5.1) to 400 ml of water and mix.

5.3 Hydrochloric acid, ρ_{20} approximately 1,19 g/ml.

5.4 Hydrofluoric acid, ρ_{20} approximately 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, apply a topical gel containing 2,5 % (by mass) calcium gluconate, and seek immediate medical treatment.

5.5 Perchloric acid, ρ_{20} approximately 1,67 g/ml.

WARNING — Perchloric acid vapour can cause explosions in the presence of ammonia, nitrous fumes or organic material in general. All evaporations shall be carried out in fume cupboards suitable for use with perchloric acid.

5.6 Citric acid, 500 g/l.

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

5.7 4-methyl-2-pentanone (methyl isobutyl ketone).

5.8 Hexaammonium heptamolybdate, 150 g/l.

Dissolve 15 g of hexaammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$] with water, dilute to 100 ml with water and mix.

Prepare a fresh solution each day. If high and unstable blank values are recorded, it is possible there is a problem with the reagent used. In such a case, switch to another lot.

5.9 Ammonium metavanadate, 2,5 g/l.

Dissolve 2,5 g of ammonium metavanadate (NH_4VO_3) with water, dilute to 1 000 ml with water and mix.

5.10 Sodium nitrite, 50 g/l.

Dissolve 50 g of sodium nitrite ($NaNO_2$) with water, dilute to 1 000 ml with water and mix.

5.11 Fluoro-boric acid.

Add 75 g of boric acid (H_3BO_3) into 600 ml of hot water in a plastic beaker. Add 50 ml of hydrofluoric acid (5.4), dilute to 1 000 ml with water and stir until the boric acid is dissolved.

Keep the solution in a plastic bottle.

The solution should be gently heated if the boric acid tends to crystallize.