
**Nickel alloys — Determination
of titanium content —
Diantipyrylmethane molecular
absorption method**

*Alliages de nickel — Détermination de la teneur en titane —
Méthode par spectrophotométrie d'absorption moléculaire au
diantipyrylméthane*

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 11433:1993), which has been technically revised in order to incorporate the Amendment ISO 11433:1993/Amd 1:2013.

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.

Nickel alloys — Determination of titanium content — Diantipyrylmethane molecular absorption method

1 Scope

This document specifies a molecular absorption spectrophotometric method for the determination of titanium content in nickel alloys.

The method is applicable to titanium contents between 0,3 % (mass fraction) and 5,0 % (mass fraction).

Molybdenum, if present in the alloy, can cause a high bias in the reported titanium value to the extent of 0,001 % Ti for every 1,0 % Mo.

NOTE 1 Evidence exists that extension of this method is possible for titanium contents down to 0,05 % (mass fraction).

NOTE 2 Modifications in the general method allow the determination of titanium in alloys containing tungsten and/or tantalum.

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*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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 <http://www.electropedia.org/>

4 Principle

Dissolution of a test portion with hydrochloric and nitric acids.

Elimination of hydrochloric and nitric acids by evaporation to fumes in the presence of sulphuric acid.

Formation of a yellow complex with diantipyrylmethane.

Spectrophotometric measurement of the absorption of the coloured complex at a wavelength of about 390 nm.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and grade 2 water as specified in ISO 3696.

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

5.2 Hydrochloric acid solution, 1 + 1.

Add 500 ml of hydrochloric acid (5.1) to 500 ml of water and mix.

5.3 Sulphuric acid solution, 1 + 1.

Slowly, and with constant stirring and cooling, add 100 ml of sulphuric acid, $\rho_{20} = 1,84$ g/ml, to 100 ml of water, allow to cool and mix.

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

5.5 Ammonium hydroxide, $\rho_{20} = 0,88$ g/ml.

5.6 Potassium hydrogen sulfate (KHSO_4).

5.7 Ascorbic acid, 100 g/l.

Dissolve 20 g of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in water, dilute to 200 ml and mix.

Prepare this solution immediately before use.

5.8 Oxalic acid, 50 g/l.

Dissolve 10 g of oxalic acid dihydrate $[(\text{COOH})_2 \cdot 2\text{H}_2\text{O}]$ in water, dilute to 200 ml and mix.

5.9 Diantipyrylmethane, 20 g/l.

Dissolve 5 g of diantipyrylmethane monohydrate ($\text{C}_{23}\text{H}_{24}\text{N}_4\text{O}_2 \cdot \text{H}_2\text{O}$) in water containing 25 ml of hydrochloric acid (5.2), dilute to 200 ml and mix.

5.10 Sodium chloride, 234 g/l.

Dissolve 117 g of sodium chloride (NaCl) in water, dilute to 500 ml and mix.

5.11 Titanium standard solution prepared with titanium, 500 mg/l.

Weigh $0,1 \text{ g} \pm 0,001 \text{ g}$ of pure titanium (99,99 % purity) and transfer into a 250 ml beaker.

Add 50 ml of sulfuric acid, $\rho_{20} = 1,84$ g/ml, diluted 1 + 3, cover with a watch glass and heat gently to assist dissolution.

Oxidize the titanium by adding nitric acid (5.4) dropwise until the blue colour is just cleared. Avoid an excess of nitric acid, which will cause the titanium to precipitate.

Cool to room temperature, transfer into a 200 ml volumetric flask, dilute to volume with sulfuric acid, $\rho_{20} = 1,84$ g/ml, diluted 1 + 9, and mix.

1 ml of this standard solution contains 0,5 mg of titanium.