

TECHNICAL REPORT

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Nickel alloys — Determination of niobium and tantalum — Ion exchange method

*Alliages de nickel — Dosage du niobium et du tantale — Méthode par
échange d'ions*



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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 main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11434, which is a Technical Report of type 1, was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Subcommittee SC 4, *Analysis of nickel alloys*.

Originally, this ion exchange method for the determination of niobium and tantalum contents was part of a composite ion exchange procedure which also included determinations of molybdenum, titanium and tungsten contents. Because of the very complexity of the lengthy composite procedure, it was decided to rewrite the method in the present form covering niobium and tantalum only. The interlaboratory precision test-

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ing of the method provided sets of results from only three participating laboratories, which was considered insufficient for statistical evaluation and sub-committee support. Since the results were good and represented a considerable amount of work, it was resolved to publish the method in the form of a Technical Report of type 1.

Nickel alloys — Determination of niobium and tantalum — Ion exchange method

1 Scope

This Technical Report describes an ion exchange separation method for the determination of niobium in the range of 0,3 % (m/m) to 5 % (m/m) and of tantalum in the range of 0,01 % (m/m) to 5 % (m/m) in nickel alloys. The elements normally found in nickel alloys do not interfere.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report 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*.

3 Principle

Pass a solution of the sample in a hydrochloric-hydrofluoric acid mixture through an ion exchange column packed with a strongly basic ion-exchange resin. Remove all elements other than niobium and tantalum by washing.

Separate niobium and tantalum from the resin bed by first selectively eluting niobium and then follow with the elution of tantalum.

Determine the eluted niobium and tantalum gravimetrically as the pentoxides, if present at high levels.

For small contents of tantalum, use coprecipitation with zirconium as cupferrates, ignite to oxides and fuse with potassium pyrosulfate. Dissolve the melt and measure the tantalum spectrophotometrically with pyrogallol at a wavelength of 400 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 Hydrofluoric acid, $\rho_{20} = 1,13$ g/ml, 40 % (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.

4.2 Hydrochloric acid, $\rho_{20} = 1,18$ g/ml, diluted 1 + 4.

4.3 Nitric acid, $\rho_{20} = 1,41$ g/ml, diluted 1 + 4.

4.4 Sulfuric acid, $\rho_{20} = 1,84$ g/ml, diluted 1 + 19.

4.5 Anion exchange resin, strongly basic 100 to 200 mesh (8 % cross-linked polystyrene-divinyl benzene matrix).

4.6 Hydrofluoric acid (5,4 mol/l)-hydrochloric acid (3,0 mol/l), solution.

Add 1 200 ml of hydrofluoric acid (4.1) to 1 250 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml), dilute to 5 litres with water and mix. Prepare and store in a plastics bottle.