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Nickel alloys — Determination of tantalum — Inductively coupled plasma optical emission spectrometric method

Alliages de nickel — Détermination du tantale — Méthode par spectrométrie d'émission optique avec source à plasma induit par haute fréquence

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Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Principle **1**

5 Reagents **2**

6 Apparatus **3**

7 Sampling and sample preparation **3**

8 Procedure **4**
8.1 Test portion 4
8.2 Preparation of test solution, T_{TA} 4
8.3 Optimization of the spectrometer 4
8.4 Pre-determination of the test solution 5
8.5 Preparation of calibration solutions for bracketing, $K_{I,Ta}$ and $K_{h,Ta}$ 5
8.6 Measurement of test solutions 6

9 Expression of results **6**
9.1 Method of calculation 6
9.2 Precision 6
9.2.1 Laboratory tests 6
9.2.2 Wavelength for measurement 6
9.2.3 Statistical evaluation 6
9.3 Trueness 7

10 Test report **7**

Annex A (normative) **Checking the performance of an inductively coupled plasma optical emission spectrometer** **8**

Annex B (informative) **Notes on the test programme** **10**

Bibliography **11**

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).

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

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 tantalum — Inductively coupled plasma optical emission spectrometric method

1 Scope

This document specifies an inductively coupled plasma optical emission spectrometric method for the determination of tantalum contents between 0,1 % and 5 % in nickel alloys.

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 in a mixture of hydrofluoric, hydrochloric, nitric and phosphoric acid and fuming after addition of perchloric acid. Addition of hydrofluoric acid and, if desired, of an internal reference element and dilution of the solution to known volume. Nebulization of the solution into an inductively coupled plasma optical emission spectrometer and measurement of the intensity of the emitted light from tantalum, and, where appropriate, from the internal reference element, simultaneously.

An example of the wavelength for tantalum is given in Table 1.

The method uses a calibration based on a very close matrix-matching of the calibration solutions to the sample and bracketing between 0,75 and 1,25 of the approximate content of tantalum in the sample to be analysed. The content of all elements in the sample has, therefore, to be approximately known. If the contents are not known, the sample has to be analysed by some semi-quantitative method. The advantage of this procedure is that all possible interferences from the matrix will be compensated, which will result in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed matrixes. All possible interferences shall be kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected wavelengths.

The line corresponding to 240,06 nm has been investigated. If other lines are used, they shall be carefully checked. The wavelength for the internal reference element should be selected carefully. The

use of scandium at 363,07 nm is recommended. This wavelength is interference-free for the elements and contents generally found in nickel alloys.

Table 1 — Example of wavelength for tantalum

Element	Wavelength	Interferences
Tantalum	240,06	Co - Fe - Hf

NOTE The use of an internal reference element is not essential since no relevant differences between laboratories operating with or without internal reference elements were found when the precision test was carried out (see 9.2.2).

5 Reagents

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

5.1 Hydrofluoric acid, HF, ρ = 1,14 g/ml, or ρ = 1,17 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 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

5.2 Hydrochloric acid, HCl, ρ = 1,19 g/ml.

5.3 Nitric acid, HNO_3, ρ = 1,40 g/ml.

5.4 Phosphoric acid, H_3PO_4, ρ = 1,70 g/ml.

5.5 Perchloric acid, $HClO_4$, ρ = 1,54 g/ml, or ρ = 1,67 g/ml.

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general. The use of fume hoods (water scrubbed) when using perchloric acid is highly recommended.

5.6 Internal reference element solution, 100 mg/l.

Choose a suitable element to be added as internal reference and prepare a 100 mg/l solution.

5.7 Tantalum standard solution, 10 g/l.

Weigh, to the nearest 0,001 g, 1 g of tantalum [minimum 99,9 % (mass fraction)], place it in a beaker (6.1), cover with an appropriate lid and dissolve it in a mixture of 10 ml of hydrofluoric acid (5.1) and 10 ml of nitric acid (5.3).

Cool and transfer quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 10 mg of tantalum.

5.8 Tantalum standard solution, 1 g/l.

Weigh, to the nearest 0,000 5 g, 0,1 g of tantalum [minimum 99,9 % (mass fraction)], place it in a beaker (6.1), cover with an appropriate lid and dissolve it in a mixture of 10 ml of hydrofluoric acid (5.1) and 10 ml of nitric acid (5.3).

