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

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

Reference number
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Tel. + 41 22 749 01 11
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Foreword

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ISO 22725 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Subcommittee SC 3, *Analysis of nickel, ferronickel and nickel alloys*.

INTERNATIONAL STANDARD **ISO 22725:2007(E)**

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

1 Scope

This International Standard specifies an inductively coupled plasma atomic emission spectrometric method for the determination of the mass fraction of tantalum between 0,1 % and 5 % in nickel alloys.

2 Normative references

The following referenced documents are indispensable for the application 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:—1) , *Laboratory glassware — Single volume pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

3 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 atomic emission spectrometer and measurement of the intensity of the emitted light from tantalum, and eventually from the internal reference element, simultaneously.

An example of the analytical line 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 of the mass fractions between 0,75 and 1,25 of the approximate concentration of tantalum in the sample to be analysed. The concentration of all elements in the sample has, therefore, to be approximately known. If the concentrations 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 automatically compensated, which will result in high accuracy. This is most important for spectral interferences,

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1) To be published. (Revision of ISO 648:1977)

which can be severe in very highly alloyed metals. 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 analytical lines.

The line corresponding to 240,06 nm has been carefully investigated. If other lines are used, they shall be carefully checked. The analytical line for the internal standard should be selected carefully. The use of Scandium at 363,07 nm is recommended. This line is interference-free for the elements and concentrations generally found in nickel alloys.

Table 1 — Example of analytical line for tantalum

Element	Analytical line nm	Interferences
Tantalum	240,06	Fe - Hf

NOTE The use of an internal standard is not essential since no relevant differences between laboratories operating with or without internal standards were found.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade and only grade 2 water as specified in ISO 3696:1987.

4.1 Hydrofluoric acid, HF, 40 % (mass fraction), ρ = 1,14 g/ml, or 50 % (mass fraction), 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.

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

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

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

4.5 Perchloric acid, $HClO_4$, 60 % (mass fraction) ρ = 1,54 g/ml or 70 % (mass fraction), ρ = 1,67 g/ml.

4.6 Internal standard solution, 100 mg/l.

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

4.7 Tantalum standard solution, 10 g/l.

Weigh, to the nearest 0,000 5 g, 1 g of high-purity tantalum [minimum 99,9 % (mass fraction)], place it in a beaker and dissolve it in a mixture of 10 ml of hydrofluoric acid (4.1) and 10 ml of nitric acid (4.3).

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

NOTE 1 ml of this solution contains 10 mg of tantalum.

4.8 Tantalum standard solution, 1 g/l.

Weigh, to the nearest 0,000 5 g, 0,1 g of high-purity tantalum [minimum 99,9 % (mass fraction)], place it in a beaker and dissolve it in a mixture of 10 ml of hydrofluoric acid (4.1) and 10 ml of nitric acid (4.3).

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

NOTE 1 ml of this solution contains 1 mg of tantalum.

4.9 **Tantalum standard solution**, 100 mg/l.

Using a calibrated pipette (or burette), transfer 10 ml of the tantalum standard solution (4.8) into a calibrated 100 ml one-mark volumetric flask. Add 10 ml of hydrofluoric acid (4.1) and 10 ml of nitric acid (4.3). Dilute to the mark with water and mix.

NOTE 1 ml of this solution contains 0,1 mg of tantalum.

4.10 **Standard solutions of interfering elements**.

Prepare standard solutions for each element whose mass fraction is higher than 1 % in the test sample. Use pure metal or chemical substances with mass fractions of tantalum less than 10 µg/g.

5 Apparatus

All volumetric glassware shall be class A and calibrated, in accordance with ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus and the following.

5.1 **Polytetrafluoroethylene (PTFE) beakers or perfluoroalkyoxy-polymer (PFA)** beakers with a graphite base.

5.2 **Polypropylene volumetric flasks**, of capacity 100 ml, calibrated in accordance with ISO 1042.

5.3 **Atomic emission spectrometer (AES)**.

The spectrometer shall be equipped with an inductively coupled plasma (ICP) and a nebulization system resistant to hydrofluoric acid. The ICP-AES used will be satisfactory if, after optimising in accordance with 7.3, it meets the performance criteria given in this subclause.

The spectrometer can be either a simultaneous or a sequential one. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal standard line, it can be used with the internal reference method. If the sequential spectrometer is not equipped with this arrangement, an internal reference cannot be used and an alternative method without an internal standard should be applied.

5.3.1 **Practical resolution of the sequential spectrometer**.

Calculate the bandwidth (full width at half maximum), in accordance with A.2 (see Annex A), for the analytical line used, including the line for internal reference. The bandwidth shall be less than 0,030 nm.

5.3.2 **Short-term stability**.

Calculate the standard deviation of ten measurements of the absolute intensity or intensity ratio corresponding to tantalum and to the internal standard, by using the most concentrated calibration solution for tantalum in accordance with A.3. The relative standard deviation shall not exceed 0,4 %.

5.3.3 **Background equivalent concentration**.

Calculate the background equivalent concentration (BEC) in accordance with A.4, for the analytical line using a solution containing only the analyte element. The maximum values of BEC obtained should be 0,8 mg/l.

6 Sampling and sample preparation

6.1 Sampling and preparation of the laboratory sample shall be carried out by the normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample is normally in the form of millings or drillings and no further mechanical preparation is necessary.

6.3 The laboratory sample shall be cleaned by washing with pure acetone and drying in air.

6.4 If brazed alloy tools are used in the preparation of the laboratory sample then the sample shall be further cleaned by pickling in 15 % (mass fraction) nitric acid for a few minutes. It shall then be washed several times with distilled water, followed by washing in acetone and drying in air.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,000 5 g, 0,25 g of the test sample.

7.2 Preparation of test solution, T_{Ta}

A PTFE or PFA beaker (5.1) should be used when using HF (4.1).

7.2.1 Place the test portion in a PTFE or PFA beaker with a graphite base.

7.2.2 Add 5 ml of HF (4.1), 30 ml of HCl (4.2), 3 ml of HNO_3 (4.3) and allow the dissolution of the sample to take place overnight at room temperature. Then add 2,5 ml of H_3PO_4 (4.4). If necessary, heat to complete dissolution. Add 7,5 ml of $HClO_4$ (4.5) and heat until the perchloric acid starts to fume. Continue to fume for 2 to 3 min.

NOTE 5 ml of H_3PO_4 (4.4) and 5 ml of $HClO_4$ (4.5) can also be used, instead of 2,5 ml of H_3PO_4 (4.4) and 7,5 ml of $HClO_4$ (4.5).

7.2.3 Cool the solution and add 10 ml of water to dissolve the salts. Some residues may remain undissolved. In this case, add 2 ml of HF (4.1) and heat gently for about 20 min until the residues dissolve completely.

NOTE The following is an alternative dissolution procedure for 7.2.2 and 7.2.3.

Add 30 ml of HCl (4.2), 3 ml of HNO_3 (4.3) and 5 ml of H_3PO_4 (4.4), or 20 ml of HCl (4.2), 10 ml of HNO_3 (4.3) and 5 ml of H_3PO_4 (4.4). Let the dissolution begin at room temperature. If necessary, heat to complete dissolution. Add 2 ml of HF (4.1) and 5 ml of H_2SO_4 (ρ = 1,84g/ml) and heat until sulfuric acid starts to fume. Cool the solution and add 10 ml of water to dissolve the salts. Heat gently until the residues dissolve completely.

7.2.4 Cool the solution to room temperature and transfer the solution quantitatively to a 100 ml volumetric polypropylene flask. If the internal standard solution (4.6) is used, add, with a calibrated pipette, 10 ml of this internal standard.

7.2.5 Dilute to the mark with water and mix. Proceed as quickly as possible to the measurement.

7.3 Optimisation of spectrometer

7.3.1 Start the ICP-AES and let it run for at least 30 min for stabilisation before taking any measurements.

7.3.2 Optimise the instrument in accordance with the manufacturer's instructions.

7.3.3 Prepare the software to measure the intensity, mean value and relative standard deviation of the selected analytical lines.

7.3.4 If an internal standard is used, prepare the software to calculate the ratio between analyte intensity and internal standard intensity. The intensity of the internal standard shall be measured simultaneously with the analyte intensity.

7.3.5 Check the instrument performance requirements given in 5.3.1 to 5.3.3.

7.4 Pre-determination of the test solution

Prepare a calibration solution K_x, matrix matched to the test sample solution as follows.

7.4.1 Using a calibrated pipette (or burette) and one 100 ml volumetric polypropylene flask (5.2) marked K_x, prepare a calibration solution K_x, corresponding to the estimated mass fraction of tantalum in the sample, in percent, as indicated in Table 2.

Table 2 — Pre-determination of the test solution

Estimated mass fraction %	K_x equivalent mass fraction %	K_x identification	Tantalum standard solution	Standard solution volume ml
0,10 – 1,0	1,0	K_1	4.8	2,5
1,0 – 5,0	5,0	K_5	4.8	12,5

7.4.2 In this volumetric flask K_x, add volumes of standard solutions (4.10) necessary to match the sample matrix to be tested, for each element whose mass fraction is above 1 %.

The matrix should be matched to the nearest percent.

7.4.3 Add 2,5 ml of H_3PO_4 (4.4) and 7,5 ml of $HClO_4$ (4.5) and 10 ml of the internal standard solution (4.6). Dilute with water and mix.

7.4.4 Also prepare a zero member solution, K_0, prepared in the same way as the calibration solution K_x omitting tantalum.

7.4.5 Measure the absolute intensities (I_0 and I_x) for the solutions K_0 and K_x.

7.4.6 Measure the absolute intensity I_{TTa} for the test solution T_{Ta}.

7.4.7 Calculate the approximate mass fraction of tantalum, w_{TTa}, in percent, in the test solution, by means of the following formula:

$$w_{\mathrm{TTa}} = \frac{I_{\mathrm{TTa}}\ (w_x - w_0)}{I_x - I_0}$$

7.5 Preparation of calibration solutions for bracketing, $K_{\mathrm{l,Ta}}$ and $K_{\mathrm{h,Ta}}$

For each test solution T_{Ta} prepare two matrix matched calibration solutions, $K_{\mathrm{l,Ta}}$ and $K_{\mathrm{h,Ta}}$, with the tantalum concentrations in $K_{\mathrm{l,Ta}}$ slightly below, and in $K_{\mathrm{h,Ta}}$ slightly above, the concentration in the unknown test solution as follows:

7.5.1 Using calibrated pipettes (or a burette), add tantalum standard solution (4.8) or (4.9) to one PTEF or PFA beaker marked $K_{\mathrm{l,Ta}}$ so that the mass fraction of tantalum $w_{\mathrm{l,Ta}}$, in percent, is approximately $K_{\mathrm{TTa}} \times 0{,}75 < K_{1,\mathrm{Ta}} < K_{\mathrm{TTa}} \times 0{,}95$. Select $K_{1,\mathrm{Ta}}$ in such a way to take an easy volume with a calibrated pipette.

7.5.2 Using calibrated pipettes (or burette), add tantalum standard solution (4.8) or (4.9) to one PTEF or PFA beaker marked $K_{h,Ta}$ so that the mass fraction of tantalum $w_{h,Ta}$, in percent, is approximately $K_{TTa} \times 1{,}05 < K_{h,Ta} < K_{TTa} \times 1{,}25$. Select $K_{h,Ta}$ in such a way as to take an easy volume with a calibrated pipette.

7.5.3 Add, to the calibration solutions $K_{1,Ta}$ and $K_{h,Ta}$, all matrix elements whose mass fractions are above 1 % in the test sample solution, using the appropriate amount of standard solutions (4.10) to match the equivalent mass fraction of matrix to the nearest percent.

7.5.4 Proceed as specified in 7.2.2 to 7.2.5.

7.6 Measurement of test solutions

Measure the absolute or ratioed intensity of the analytical line of the lowest calibration solution $K_{1,Ta}$ firstly, then of the test sample solution, T_{Ta} and finally the highest calibration solution $K_{h,Ta}$. Repeat this sequence three times and calculate the mean intensities $I_{l,Ta}$ and $I_{h,Ta}$ for the low and high calibration solution and I_{TTa} for the test solution respectively.

8 Expression of results

8.1 Method of calculation

Calculate the mass fraction of tantalum, w_{Ta}, in percent, in the test solution T_{Ta}, by means of the formula:

$$w_{Ta} = w_{l,Ta} + \frac{(I_{Ta} - I_{l,Ta})(w_{h,Ta} - w_{l,Ta})}{I_{h,Ta} - I_{l,Ta}}$$

8.2 Precision

8.2.1 Laboratory tests

Ten laboratories from seven countries participated in an inter-laboratory test programme under the auspices of ISO/TC 155/SC 3/WG 8, involving three determinations of tantalum at eight levels. Each laboratory did two determinations under the repeatability conditions defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration and a minimum period of time. The third determination was done on a different day using the same apparatus with a different calibration.

8.2.2 Wavelength for measurements

Concerning the wavelength taken for measurements which have been statistically evaluated, all the laboratories operated at 240,06 nm. No relevant difference between laboratories operating with or without an internal standard was found.

8.2.3 Statistical analysis

A statistical analysis was done in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3. Results from one laboratory were rejected as a consistent outlier.

This evaluation also shows that the performances of the method were not satisfactory enough for tantalum levels lower than 0,1 % on one hand and higher than 5 % on the other hand.

Results of the evaluation were used to calculate the smoothed values for repeatability r, and within-laboratory reproducibility, R_w, and between-laboratory reproducibility, R, for the mass fractions of tantalum between 0,1 % and 5 %, which are given in Table 3.

Table 3 — Repeatability and reproducibility limits

Mass fraction of tantalum %	Repeatability limit r	Within laboratory reproducibility limit R_W	Between laboratory reproducibility limit R
0,1	0,001 4	0,004 6	0,011 3
0,2	0,002 8	0,008 2	0,020 3
0,5	0,007 0	0,017 5	0,044 1
1,0	0,013 9	0,031 3	0,079 4
2,0	0,027 6	0,055 7	0,142 8
5,0	0,068 4	0,119 7	0,310 3

8.3 Trueness

The determined mean mass fractions in the test samples (see Annex B) are given in Table 4 together with the accepted values. Two of the values are certified. Comparing both sets of values allow the conclusion that trueness is satisfactory.

Table 4 — Evaluation of trueness

Sample No.	Name	Accepted value	Value found
		% (mass fraction)	
8-1-Ta [a]	ETI 569 [b]	0,020	0,009 0
8-2-Ta	ETI 673 [b]	0,141	0,138 8
8-3-Ta	MBH 211X 11224	0,316	0,320 9
8-4-Ta	ETI 596 [b]	1,19	1,231 2
8-5-Ta	ETI 597 [b]	2,30	2,368 6
8-6-Ta	MBH 219X 1867	3,41	3,460 1
8-7-Ta [a]	ETI 2042 [b]	7,92	7,986 2
8-8-Ta [a]	ETI 1868 [b]	8,89	8,921 8

[a] Samples for which the precision data were unsatisfactory.

[b] Not certified.

9 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis or of the test report;

b) the method used by reference to this International Standard;

c) the results and the unit in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard, or any optional operation which may have influenced the results.

Annex A
(normative)

Checking the performance of an ICP instrument

A.1 Introduction

A joint working group (ISO/TC 47/SC 1) involving representatives from ISO/TC 47, ISO/TC 17 and ISO/TC 155 was formed in 1995 to establish guidelines for inductively coupled plasma spectrometry. The project reached the stage of a committee draft (ISO/CD 12235) but the work was not completed. This annex is abstracted from this committee draft and was used in the tests in this International Standard.

A.2 Resolution of a spectrometer

The resolution of a spectrometer can be defined as the wavelength difference, $\Delta\lambda$, between two lines which can still just be observed separately. In practice, the parameter FWHM (Full Width at Half Maximum) is used as a resolution measure.

Ideally, the resolution should be of the same order as the physical line width in ICP OES [2] spectra, i.e. 2 to 5 pm (1 pm = 10^{-12} m). In practice, however, the observed width of the emission lines and, consequently, the resolution will often be determined by the bandwidth (r_{bp}) of the spectrometer being used. As long as broadening resulting from aberrations can be neglected, this bandwidth is given by:

$$r_{\mathrm{bp}} = \mathrm{LMH} = (\mathrm{d}\lambda/\mathrm{d}x)(b_{\mathrm{i}} + b_{\mathrm{o}})/2$$

where

b_{i} and b_{o} are the widths of the entrance slit and exit slit respectively;

$\mathrm{d}\lambda/\mathrm{d}x$ is the reciprocal linear dispersion which is given by:

$$\mathrm{d}\lambda/\mathrm{d}x = \mathrm{d}(\cos\beta)/nL$$

in which

L is the focal length of the spectrometer;

n is the order number;

d is the reciprocal of the grove density in the grating;

β is the diffraction angle.

Normally, commercial spectrometers present resolutions in the range of 4 to 30 pm. A good resolution is of great importance to cope with the frequent spectral interferences which occur in ICP OES. Since a line with a wavelength in the second order will have the same diffraction angle β as a line with a wavelength 2λ in the first order, a spectrometer shall either have an order-sorting possibility or an optical filter to avoid an order overlap.

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[2] OES: Optical Emission Spectroscopy.

A.3 Evaluating the short- and long-term stability

The evaluation of the short-term stability consists of measuring the repeatability standard deviation of the ICP emission spectrometer.

A series of 10 consecutive intensity measurements of the (high) multi-element calibration solution is carried out using the typical integration time for the system. The average I_{avrg} and standard deviation S_{I} of the 10 measurements are calculated, as well as the relative standard deviation RSD_I, in percent, using the formula:

$$\mathrm{RSD}_I = (S_{\mathrm{I}}/I_{\mathrm{avrg}}) \times 100$$

In ICP AES, for solutions with concentrations of at least twice the BEC, RSD_I-values between 0,3 and 1,0 % are generally accepted. Multi-element calibration solutions may be used for measurement at various analytical lines present in simultaneous optics.

Long-term stability assessment is a measurement of the instrument drift. This is only required if the ICP emission spectrometer is set up to work for long intervals of time. It consists of carrying out the same short-term stability tests at specific intervals of time, 15 min to 1 h, and plotting the deviation of the average found for every short-term test against time. Deviations of more than 2 % per hour must not be accepted. In case the instrument is not able to perform better, during analysis the control calibration solution should be measured more often and the mean results of the test sample solutions should be recalculated by interpolation between two consecutive control calibration solutions.

A.4 Evaluating the Background Equivalent Concentration

The Background Equivalent Concentration (BEC) is used as a measure for instrument sensitivity. (Since the analyte signal is usually on a relatively high background, including the background intensity is suggested rather than the sensitivity by itself.) It is calculated as follows:

$$\mathrm{BEC} = (I_{\mathrm{BG}}/I_{\mathrm{net}}) \times c_{\mathrm{A}}$$

where

I_{BG} is the intensity of the background;

I_{net} is the intensity of the analyte (overall intensity minus intensity of the background);

c_{A} is the concentration of analyte that yields I_{net}.

The BEC values for the elements to be analysed can be found in wavelength tables (usually part of the instrument software). The smaller its numerical value is, the better the BEC.

Annex B
(informative)

Notes on the test programme

B.1 Composition of samples used in the test programme

The chemical compositions of the samples used in the test programme are given, in percent (mass fraction), in Table B.1. For the test programme, the samples were identified as 8-1-Ta to 8-8-Ta. All mass fractions, except for tantalum, are approximate values.

Table B.1 — Chemical composition of test samples

Values in percent (mass fraction)

Sample No.	Ta	C	Si	Mn	Ni	Cr	Mo	W	Al	Co	Ti	Fe	Nb	Zr
ETI 569	0,020	0,06	0,04	2,2	(56)	17	—	—	—	18	2,4	4,7	—	—
ETI 673	0,141	0,05	0,25	0,10	(51)	18	2,2	0,06	0,40	0,30	1,3	20	6,0	—
MBH 211X 11224	0,316	0,02	0,25	0,09	(70)	14	4,0	—	6,0	0,30	1,3	0,50	2,9	0,10
ETI 596	1,19	0,12	—	—	(66)	19	1,3	3,6	—	7,5	—	—	1,2	—
ETI 597	2,30	0,15	0,20	0,08	(66)	16	2,6	2,3	—	10	—	0,30	0,60	—
MBH 219X 1867	3,41	0,12	0,15	0,20	(59)	7,2	6,0	0,60	7,9	11	2,5	1,8	0,10	0,50
ETI 2042	7,92	< 0,01	—	—	(65)	7,4	1,9	5,4	5,0	6,5	1,2	—	—	—
ETI 1868	8,89	< 0,01	0,10	—	(68)	7,9	—	9,9	5,4	—	—	—	—	—

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