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**Methods for ICP emission
spectrometric analysis of tantalum**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Society of Newer Metals (JSNM)/ Japanese Standards Association (JSA), with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS H 1699 : 1999** is revised and **JIS H 1682 : 2002** is withdrawn, and they are replaced with this Standard.

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Methods for ICP emission spectrometric analysis of tantalum

1 Scope This Japanese Industrial Standard specifies the quantitative method by ICP emission spectrometric analysis of aluminium, calcium, chrome, copper, iron, magnesium, manganese, molybdenum, niobium, nickel, titanium, tungsten and silicon contained in tantalum.

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS H 1680 *Tantalum—General rules for chemical analysis*

JIS K 0050 *General rules for chemical analysis*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS Z 8402-2 *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*

3 General matters The general matters common to analysis method shall be as specified in JIS H 1680, JIS K 0050, JIS K 0116, JIS K 0557, and JIS Z 8402-2.

4 Classification of analysis methods The quantitative method of each element contained in tantalum shall be as specified in one of the following.

- a) **ICP emission spectrometric analysis** This is a method of dissolving a sample with hydrofluoric acid and nitric acid and measuring directly impurity elements excluding silicon by using an ICP emission spectrometer equipped with a hydrofluoric acid-resistant sample chamber ⁽¹⁾, with a sample containing hydrofluoric acid and nitric acid. The analyte elements and quantitative range shall be as specified in table 1.

Note⁽¹⁾ : A hydrofluoric acid-resistant sample chamber shall be made of such materials that protect an analyzer and components contacting sample solution from corrosion by hydrofluoric acid.

- b) **Anion-exchange separation ICP emission spectrometric analysis** This is a method of dissolving a sample with hydrofluoric acid and nitric acid, separating tantalum by using the anion-exchange separation method, removing hydrofluoric acid and nitric acid with the sulfuric acid white fuming process, and measuring impurity elements excluding silicon by using an ICP emission spectrometer equipped with a usual quartz/glass sample chamber. The analyte elements and quantitative range shall be as specified in table 2.

- c) **Quantitative method of silicon by ICP emission spectrometric analysis** In order to prevent vaporization of silicon, this is a method of dissolving a sample with hydrofluoric acid and nitric acid under existence of sodium nitrate, and measuring directly silicon by using an ICP emission spectrometer equipped with a hydrofluoric acid-resistant sample chamber ⁽¹⁾, with a sample containing hydrofluoric acid and nitric acid. The quantitative range of silicon shall be as specified in table 3.

Table 1 The analyte elements and quantitative range for ICP emission spectrometric analysis

Analyte elements	Quantitative range % (mass fraction)	Analyte elements	Quantitative range % (mass fraction)
Aluminium	0.001 0 to 0.020	Manganese	0.000 5 to 0.020
Calcium	0.000 5 to 0.020	Molybdenum	0.000 5 to 0.050
Chrome	0.000 5 to 0.050	Niobium	0.001 0 to 0.20
Copper	0.000 5 to 0.050	Nickel	0.000 5 to 0.050
Iron	0.000 5 to 0.050	Titanium	0.000 5 to 0.020
Magnesium	0.000 5 to 0.020	Tungsten	0.003 0 to 0.10

Table 2 The analyte elements and quantitative range for anion-exchange separation ICP emission spectrometric analysis

Analyte elements	Quantitative range % (mass fraction)	Analyte elements	Quantitative range % (mass fraction)
Aluminium	0.000 2 to 0.020	Manganese	0.000 1 to 0.020
Calcium	0.000 2 to 0.020	Molybdenum	0.000 3 to 0.020
Chrome	0.000 3 to 0.050	Niobium	0.000 3 to 0.020
Copper	0.000 1 to 0.020	Nickel	0.000 1 to 0.020
Iron	0.000 8 to 0.020	Titanium	0.000 1 to 0.020
Magnesium	0.000 1 to 0.020	Tungsten	0.000 4 to 0.020

Table 3 The quantitative range of silicon for ICP emission spectrometric analysis

Analyte element	Quantitative range % (mass fraction)
Silicon	0.000 5 to 0.020

5 ICP emission spectrometric analysis Dissolve a sample with hydrofluoric acid and nitric acid and spray it into the argon plasma in the ICP emission spectrometer. Then, measure the light emission intensity of the analyte elements.

5.1 Reagents The reagents shall be as follows ⁽²⁾.

Note ⁽²⁾ : The reagents specified in JIS K 0116, 5.4.2 (Reagents) may be used.

- a) Nitric acid
- b) Nitric acid (1+1)
- c) Hydrochloric acid (1+1, 1+9)

- d) **Hydrofluoric acid (1+1)**
- e) **Tantalum to create a calibration curve** Use tantalum which has on tantalum content of 99.9 % (mass fraction) or more and is free from analyte elements. Alternatively, use tantalum whose content of analyte elements is known and is as lower than that of the sample as possible.
- f) **Standard aluminium solution A (Al : 100 µg/ml)** Measure and take 0.100 g of aluminium [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 20 ml of hydrochloric acid (1+1) to it, heat it gradually, and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard aluminium solution A.
- g) **Standard aluminium solution B (Al : 10 µg/ml)** Dilute standard aluminium solution A [f] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard aluminium solution B.
- h) **Standard calcium solution A (Ca : 100 µg/ml)** Dry calcium carbonate at 110 °C for 3 h. Cool it down in a desiccator to room temperature. Measure and take it exactly 0.250 g, transfer it into a beaker (200 ml), cover it with a watch glass, add 10 ml of hydrochloric acid (1+1) gradually to it, dissolve it, heat it, and expel carbon dioxide. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard calcium solution A.
- i) **Standard calcium solution B (Ca : 10 µg/ml)** Dilute standard calcium solution A [h] exactly 10 times with of water when needed to obtain necessary quantity. It shall be taken as standard calcium solution B.
- j) **Standard chrome solution A (Cr : 100 µg/ml)** Measure and take 0.100 g of chrome [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 30 ml of hydrochloric acid (1+1) to it, heat it gradually, and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard chrome solution A.
- k) **Standard chrome solution B (Cr : 10 µg/ml)** Dilute standard chrome solution A [j] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard chrome solution B.

- l) **Standard copper solution A (Cu : 100 µg/ml)** Measure and take 0.100 g of copper [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 20 ml of nitric acid (1+1) to it, heat it gradually, and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard copper solution A.
- m) **Standard copper solution B (Cu : 10 µg/ml)** Dilute standard copper solution A [l)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard copper solution B.
- n) **Standard iron solution A (Fe : 100 µg/ml)** Measure and take 0.100 g of iron [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 20 ml of nitric acid (1+1) to it, heat it gradually and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard iron solution A.
- o) **Standard iron solution B (Fe : 10 µg/ml)** Dilute standard iron solution A [n)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard iron solution B.
- p) **Standard magnesium solution A (Mg : 100 µg/ml)** Measure and take 0.100 g of magnesium [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 50 ml of water to it, pour 20 ml of hydrochloric acid (1+1) gradually thereto, heat it gradually and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard magnesium solution A.
- q) **Standard magnesium solution B (Mg : 10 µg/ml)** Dilute standard magnesium solution A [p)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard magnesium solution B.
- r) **Standard manganese solution A (Mn : 100 µg/ml)** Measure and take 0.100 g of manganese [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 20 ml of hydrochloric acid (1+1) to it, heat it gradually, and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water, and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard manganese solution A.

- s) **Standard manganese solution B (Mn : 10 µg/ml)** Dilute standard manganese solution A [r)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard manganese solution B.
- t) **Standard molybdenum solution A (Mo : 100 µg/ml)** Measure and take 0.100 g of molybdenum [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add to it 20 ml of hydrochloric acid (1+1) and a small quantity of nitric acid (1+1), heat it gradually and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard molybdenum solution A.
- u) **Standard molybdenum solution B (Mo : 10 µg/ml)** Dilute standard molybdenum solution A [t)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard molybdenum solution B.
- v) **Standard niobium solution A (Nb : 100 µg/ml)** Measure and take 0.100 g of niobium [99.9 % (mass fraction) or more], transfer it into a platinum dish (No. 100) or a tetrafluoroethylene resin beaker (100 ml), add to it 20 ml of hydrofluoric acid (1+1) and several drops of nitric acid, heat it and dissolve it. Cool it down to ordinary temperature, use water and transfer the solution into a 1 000 ml polyethylene volumetric flask ⁽³⁾. Dilute it with water up to the marked line. It shall be taken as standard niobium solution A.
- Note ⁽³⁾ : Before use, calibrate it according to **JIS K 0050, 9.3.2** (Volumetric flask). It is allowable to use a flask made of other materials that are free from contamination of analytical components caused by corrosion and that is calibrated.
- w) **Standard niobium solution B (Nb : 10 µg/ml)** Dilute standard niobium solution A [v)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard niobium solution B.
- x) **Standard nickel solution A (Ni : 100 µg/ml)** Measure and take 0.100 g of nickel [99.9 % (mass fraction) or more], transfer it into a beaker (200 ml), cover it with a watch glass, add 10 ml of nitric acid (1+1) to it, heat it gradually and dissolve it. Cool it down to ordinary temperature, wash the undersurface of watch glass and the inner wall of beaker with water and remove the watch glass. Use water and transfer the solution into a 1 000 ml volumetric flask. Dilute it with water up to the marked line. It shall be taken as standard nickel solution A.
- y) **Standard nickel solution B (Ni : 10 µg/ml)** Dilute standard nickel solution A [x)] exactly 10 times with water when needed to obtain necessary quantity. It shall be taken as standard nickel solution B.