

JIS

JAPANESE INDUSTRIAL STANDARD

Methods for determination of palladium in titanium alloys

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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in titanium alloys

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1. Scope This Japanese Industrial Standard specifies the methods for determination of palladium in titanium alloys.

Remarks: The following standards are cited in this Standard.

JIS B 0601 Definitions and designation of surface roughness

JIS H 1611 General rule for chemical analysis of titanium

JIS K 0050 General rules for chemical analysis

JIS K 0116 General rules for emission spectrochemical analysis

JIS K 0119 General rules for X-ray fluorescence spectrometric analysis

JIS R 6251 Abrasive cloths

JIS R 6252 Abrasive papers

JIS Z 8402 General rules for permissible tolerance of chemical analyses and physical tests

2. General matter General matter common to the chemical analysis shall be in accordance with JIS H 1611, JIS K 0116 and JIS K 0119.

3. Classification of determination methods The methods for determination of palladium shall be in accordance with one of the following methods.

(1) Dimethylglyoxime extraction absorptiometry This method shall be applied to the sample of 0.01 % (m/m) or over to and excluding 0.30 % (m/m) in palladium content.

(2) Inductively coupled plasma emission spectrochemical analysis This method shall be applied to the sample of 0.05 % (m/m) or over to and excluding 0.40 % (m/m) in palladium content.

(3) Fluorescent X-ray analysis This method shall be applied to the sample of 0.05 % (m/m) or over to and excluding 0.25 % (m/m) in palladium content.

4. Dimethylglyoxime extraction absorptiometry

4.1 Summary Decompose a sample with sulfuric acid and hydrofluoric acid. After oxidizing titanium or the like with nitric acid, generate white fume of sulfuric acid. Add dimethylglyoxime, extract the dimethylglyoxime nickel complex to be generated with benzene, measure the absorbance.

4.2 Reagents The reagents shall be as follows.

(1) Nitric acid (1 + 1)

- (2) Hydrofluoric acid
- (3) Sulfuric acid (1 + 1)
- (4) Dimethylglyoxime solution Dissolve 1 g of dimethylglyoxime in 100 ml of ethanol (99.5).
- (5) Benzene
- (6) Reference palladium solution (25 $\mu\text{gPd/ml}$) Weigh out 0.500 g of palladium [99.9 % (m/m) min.], transfer into a beaker (300 ml), add 20 ml of hydrochloric acid (1 + 1) and 30 ml of nitric acid (1 + 1), cover with a watch glass, and decompose by heating. After cooling to ordinary temperature, wash the bottom surface of the watch glass and the inner wall of the beaker with water, and remove the watch glass. Transfer the solution into a 500 ml volumetric flask by using water, dilute to the marked line with water to make stock solution (1 mgPd/ml). Dilute a necessary quantity of the stock solution accurately 40 times with water at each time of use to make the reference palladium solution.

4.3 Weighing out amount of sample Weigh out 0.50 g of a sample to the nearest 1 mg.

4.4 Operation

4.4.1 Preparation of sample solution Weigh out a sample, transfer tetrafluoroethylene resin (hereafter referred to as the "PTFE") into a beaker (200 ml), add 25 ml of sulfuric acid (1 + 1) and 2 ml of hydrofluoric acid, cover with a PTFE watch glass, and decompose by heating ⁽¹⁾. Drip 4 ml of nitric acid (1 + 1). After heating for several minutes, wash the bottom surface of the watch glass with water, and remove the watch glass. Concentrate by heating gently, raise temperature gradually, and generate white fume of sulfuric acid. After allowing to cool to room temperature, wash the inside wall of the beaker with a small quantity of water. After stirring sufficiently, generate white fume of sulfuric acid by heating again. After allowing to cool to room temperature, dissolve salts by adding little by little about 50 ml of water. Add 20 ml of sulfuric acid (1 + 1). After cooling to ordinary temperature, transfer the solution into a 250 ml volumetric flask by using water, and dilute to the marked line with water.

Note ⁽¹⁾ When the sample can not be decomposed, decompose by additionally applying hydrofluoric acid.

4.4.2 Extraction Carry out extraction in accordance with the following procedure.

- (1) Take aliquot 25.0 ml from the solution obtained in 4.4.1 into a separatory funnel (200 ml), and make up the amount of solution to about 100 ml with water.
- (2) Add 5 ml of dimethylglyoxime solution [4.2 (4)]. After mixing by shaking, add 20.0 ml of benzene, mix by violently shaking for 2 min. After separating into two layers by standing still, discard the aqueous phase of the lower layer.