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JAPANESE INDUSTRIAL STANDARD

Methods for determination of tungsten in zirconium and zirconium alloys

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Methods for determination of tungsten
in zirconium and zirconium alloys

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1. Scope This Japanese Industrial Standard specifies the methods for determination of tungsten in zirconium and zirconium alloys.

Remarks: The following standards are cited in this Standard.

JIS H 1650 General rules for chemical analysis of zirconium and zirconium alloys

JIS K 0116 General rules for emission spectrochemical analysis

2. General matters General matters common to the methods for chemical analyses shall be in accordance with JIS H 1650 and JIS K 0116.

3. Classification of determination methods The methods for determination of tungsten shall be in accordance with either of the following methods.

- (1) Thiocyanic acid extraction-absorptiometry: This method shall be applied to the sample of 0.001% (m/m) or over to and excluding 0.015% (m/m) tungsten content.
- (2) Inductively coupled plasma emission spectrochemical method: This method shall be applied to the sample of 0.001% (m/m) or over to and excluding 0.015% (m/m) tungsten content.

4. Thiocyanic acid extraction absorptiometry

4.1 Summary Decompose a sample with sulfuric acid and hydrofluoric acid, and generate white fume of sulfuric acid by heating. After reducing tungsten with titanium trichloride, add potassium thiocyanate, and extract tungsten thiocyanate complex generated into butyl acetate. After washing an organic phase with oxalic acid, measure the absorbance by using a photoelectric photometer.

4.2 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid
- (2) Nitric acid
- (3) Hydrofluoric acid
- (4) Sulfuric acid (1 + 1)
- (5) Titanium trichloride solution: Add 40 ml of hydrochloric acid to 20 ml of titanium trichloride solution [20% (m/m) or over content], and make up the quantity of solution to 100 ml with water. Prepare it at each use.
- (6) Ammonium fluoride solution (400 g/l)
- (7) Potassium thiocyanate solution (200 g/l)