

JIS

JAPANESE INDUSTRIAL STANDARD

Methods for Determination of Copper in Zirconium and Zirconium Alloys

JIS H 1657—1985

Translated and Published

by

Japanese Standards Association

Translation without guarantee
In the event of any doubt arising, the original
standard in Japanese is to be evidence



JAPANESE INDUSTRIAL STANDARD

J I S

Methods for Determination of Copper
in Zirconium and Zirconium Alloys

H 1657-1985

1. Scope

This Japanese Industrial Standard specifies the methods for determination of copper in zirconium and zirconium alloys.

2. General Matters

General matters common to analysis method shall be in accordance with JIS H 1650.

3. Classification of Methods

The determination method of copper shall be in accordance with one of the following methods:

- (1) Ion-exchange separation atomic absorption method This method applies to the sample of 0.0002 % or more in copper content.
- (2) Extraction separation atomic absorption method This method applies to the sample of 0.0002 % or more in copper content.
- (3) Diethyldithiocarbamic acid absorptiometry This method applies to the method of 0.002 % or more in copper content.

Applicable Standard:

JIS H 1650-General Rules for Chemical Analysis of Zirconium and
Zirconium Alloys

4. Ion-Exchange Separation Atomic Absorption Method

4.1 Summary A sample is decomposed with hydrofluoric acid and nitric acid and copper is adsorbed by passing it through the column of ion-exchange. Then, zirconium is let to flow out. The copper is eluted with hydrochloric acid, added with nitric acid and perchloric acid and organic matters are decomposed by evaporation to dryness. Thereafter, the residue is dissolved with nitric acid and the absorbance of copper is measured by using an atomic absorption photometer.

4.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1+2, 1+120)
- (2) Nitric acid
- (3) Nitric acid (1+1)
- (4) Hydrofluoric acid (1+1, 1+9, 1+25) Store in a polyethylene container.
- (5) Perchloric acid (60 %)
- (6) Standard copper solution (10 µgCu/ml) Weigh out 0.100 g of copper (99.9 % min.), add 10 ml of nitric acid (1+1) and decompose by heating. After cooling to ordinary temperature, transfer into a measuring flask of 1000 ml and dilute up to the marked line with water to make the stock solution. Accurately dilute a necessary amount of this stock solution 10 times at each use to make the standard copper solution.

4.3 Apparatus The apparatus shall be, as a rule, as follows:

- (1) Atomic absorption photometer
- (2) Ion-exchange column The absorbent cotton unfastened with water is loosely packed into the polyethylene tube (250 mm in length and 10 mm in inner diameter) one end of which is thinned to a thickness of approx. 5 mm, and approx. 10 ml of the strong acid cation exchange resin swelled with water (74 to 149 µm and 1.9 meq/ml or more in exchange capacity) is made slurry state to be flowed thereinto. Then, it is sedimented. After regulating the flow rate of effluent to be 1.0 to 1.5 ml per minute by adjusting the packing method of absorbent cotton, 50 ml of hydrofluoric acid (1+25) is preliminarily flowed.

4.4 Weighed Out Amount of Sample The weighed out amount of sample shall be 1.0 g.

4.5 Operation The determination operation shall be carried out according to the following procedures: