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JAPANESE INDUSTRIAL STANDARD

**Methods for Determination of
Manganese in Zirconium and
Zirconium Alloys**

JIS H 1652—1985

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Methods for Determination of Manganese in
Zirconium and Zirconium Alloys

H 1652-1985

1. Scope

This Japanese Industrial Standard specifies the methods for determination of manganese 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 methods for determination of manganese shall be in accordance with either one of the following methods:

- (1) Ion-exchange separation atomic absorption method This method applies to samples of 0.0002 % min. in manganese content.
- (2) Extraction separation atomic absorption method This method applies to samples of 0.0002 % min. in manganese content.
- (3) Permanganate absorptiometry This method applies to samples of 0.001 % min. in manganese 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 manganese is adsorbed by passing it through the column of ion-exchange. Then, zirconium is let to flow out. Manganese is eluted with hydrochloric acid, added with nitric acid and perchloric acid and organic matters are decomposed by its evaporation to dryness. Thereafter, the residue is dissolved with nitric acid and the absorbance of manganese 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) They are stored in polyethylene containers.
- (5) Perchloric acid (60 %)
- (6) Standard manganese solution (10 µg Mn/ml) 0.100 g manganese (99.9 % min.) is weighed out, added with 10 ml of nitric acid (1 + 1) and decomposed by heating. After cooling it to ordinary temperature, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water to make the stock solution. Necessary amount of this stock solution is accurately diluted 10 times with water at each use to make the standard manganese solution.

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

- (1) Atomic absorption photometer
- (2) Column of ion-exchange The absorbent cotton unfastened with water is loosely packed into the polyethylene tube (250 mm in length and 10 mm in inside diameter) one end of which is thinned to a thickness of about 5 mm and about 10 ml of strong acid cation exchange resin swelled with water (74 to 149 µm and 1.9 meq/ml min. 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.