

JIS

JAPANESE INDUSTRIAL STANDARD

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

JIS H 1654—1989

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Methods for Determination of Iron in
Zirconium and Zirconium Alloys

H 1654-1989

1. Scope

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

2. General Matters

Matters related to the analysis method in general shall be in accordance with JIS H 1650.

3. Classification of Methods

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

- (1) 1, 10-Phenanthroline Absorptiometry This method applies to the sample 0.02 wt % or over to and excluding 0.30 wt % in iron content.
- (2) Thiocyanate Absorptiometry This method applies to the sample 0.02 wt % or over to and excluding 0.3 wt % in iron content.
- (3) Inductively Coupled Plasma Emission Spectrochemical Method
This method applies to the sample 0.02 wt % or over to and excluding 0.3 wt % in iron content.

4. 1, 10-Phenanthroline Absorptiometry

4.1 Summary Decompose the sample with a mixture of hydrofluoric acid and boric acid, reduce iron by adding diammonium hydrogen citrate and L(+)-ascorbic acid, generate 1, 10-phenanthroline iron complex by adding 1, 10-phenanthroline, and then measure the absorbance using a photoelectric photometer.

Applicable Standards:

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

JIS H 1655-Methods for Determination of Nickel in Zirconium and Zirconium Alloys

JIS H 1656-Methods for Determination of Chromium in Zirconium and Zirconium Alloys

JIS H 1659-Methods for Determination of Tin in Zirconium Alloys

JIS K 8005-Standard Substances for Volumetric Analysis

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4.2 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid (1 + 4)
- (2) Nitric acid
- (3) Solution of hydrofluoric acid and boric acid After dissolving by adding little by little 50 g of boric acid to 100 ml of hydrofluoric acid, store it in a polyethylene bottle.
- (4) Aqueous ammonia (1 + 1)
- (5) Diammonium hydrogen citrate solution (300 g/l)
- (6) L(+)- ascorbic acid solution (20 g/l) It shall be prepared at each use.
- (7) 1, 10-phenanthroline solution Dissolve 2 g of 1, 10-phenanthroline (monohydrate) in hot water, cool to room temperature, and dilute to 1 l with water.
- (8) Standard iron solution (20 µg Fe/ml) Dissolve 0.200 g of iron (99.5 wt % or over) in 20 ml of nitric acid (1 + 1) by heating, cool to ordinary temperature, transfer to a 1000-ml volumetric flask using water and dilute up to the mark to make a stock solution (200 µg Fe/ml). Dilute one necessary volume of this stock solution to accurately ten volumes at each use to take it as the standard iron solution.

4.3 Weighed-out Amount of Sample The weighed-out amount of sample shall be 2.0 g.

4.4 Operation

4.4.1 Preparation of Sample Solution Prepare the sample solution in the following procedure:

- (1) Weigh out the sample, transfer it to a polyethylene beaker (100 ml), cover it with a polyethylene watch glass and decompose it by adding 20 ml of water and 10 ml of the solution of hydrofluoric acid and boric acid [4.2 (3)]⁽¹⁾. When there is insoluble residue, decompose it by adding several drops of nitric acid.
- (2) After cooling to ordinary temperature, wash the bottom face of the watch glass with water, remove the watch glass, transfer the solution to a 200-ml volumetric flask and dilute it up to the mark with water.

Note (1) The progressive rate of decomposition varies according to the thickness of sample. If necessary, decompose it by heating on a water bath.

4.4.2 Coloration The coloration shall be carried out in the following procedure.

- (1) Take a definite amount⁽²⁾ of the solution obtained in 4.4.1 (2) into a beaker (100 ml).