

JIS

JAPANESE INDUSTRIAL STANDARD

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

H 1655-1989

1. Scope

This Japanese Industrial Standard specifies the method for determination of nickel contained in zirconium or 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 method for nickel shall be in accordance with one of the following methods:

- (1) Dimethylglyoxime Extraction Absorptiometry This method applies to the sample containing nickel 0.001 wt % or over to and excluding 0.08 wt %.
- (2) Dimethylglyoxime Absorptiometry This method applies to the sample containing nickel 0.03 wt % or over to and excluding 0.08 wt %.
- (3) Inductively Coupled Plasma Emission Spectrometry This method applies to the sample containing nickel 0.01 wt % or over to and excluding 0.1 wt %.

4. Dimethylglyoxime Extraction Absorptiometry

4.1 Summary Decompose the sample with the mixed acid of hydrofluoric acid and boric acid, add diammonium hydrogencitrate, and adjust its pH using aqueous ammonia. Generate dimethylglyoxime nickel complex by adding dimethylglyoxime, and extract it with chloroform. Measure its absorbance using a photoelectric photometer.

4.2 Reagents The reagents shall be as follows:

- (1) Nitric acid
- (2) Solution of hydrofluoric acid and boric acid Dissolve 50 g of boric acid little by little in 100 ml of hydrofluoric acid and store it in a polyethylene bottle.
- (3) Aqueous ammonia (1 + 1)

Applicable Standards: See page 9.

- (4) Sodium sulfate (anhydrous)
- (5) Diammonium hydrogencitrate solution (300 g/l)
- (6) Dimethylglyoxime solution Dissolve 1 g of dimethylglyoxime in ethanol (95), and dilute it with ethanol (95) to make 100 ml in total.
- (7) Chloroform
- (8) Standard nickel solution (5 μg Ni/ml) Dissolve 0.200 g of nickel (99.9 wt % or over) in 10 ml of nitric acid (1 + 1) by heating, allow to cool down to room temperature, transfer the solution into a 1000-ml volumetric flask while rinsing the beaker with water, and dilute it with water up to the marked line to take as a stock solution (200 μg Ni/ml). At each time of use, dilute one required volume with water by exactly 40 volumes to prepare a standard nickel solution.

4.3 Amount of Sample The amount of the sample to be weighed out shall be 2.0 g.

4.4 Operation

4.4.1 Preparation of Sample Solution The preparation of sample solution shall be carried out as follows:

- (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 [4.2 (2)] ⁽¹⁾. If there is some insoluble residue, decompose it by adding several drops of nitric acid.
- (2) After cooling it down to room temperature, remove the watch glass while rinsing its lower surface with water, transfer it into a 200-ml volumetric flask while rinsing the beaker with water and dilute with water up to the marked line.

Notes ⁽¹⁾ The progressing rate of decomposition varies according to the thickness of sample. If necessary, decompose the sample by heating in water bath.

4.4.2 Extraction of Complex The extraction of complex shall be carried out as follows:

- (1) Take a definite aliquot ⁽²⁾ of the solution prepared in 4.4.1 (2) into a beaker (100 ml).
- (2) Dilute the aliquot with water up to about 50 ml, add 10 ml of diammonium hydrogencitrate solution, and adjust its pH to 9.0 ± 0.5 ⁽³⁾ using aqueous ammonia (1 + 1).
- (3) Transfer the solution into a separatory funnel (200 ml) while rinsing the beaker with water, add 1 ml of dimethylglyoxime solution [4.2 (6)], and dilute the solution with water up to about 100 ml.