

# JIS

## JAPANESE INDUSTRIAL STANDARD

### Methods for Determination of Tin in Zirconium Alloys

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In the event of any doubt arising,  
the original Standard in Japanese is to be final authority.

## JAPANESE INDUSTRIAL STANDARD

J I S

## Methods for Determination of Tin in Zirconium Alloys H 1659-1989

1. Scope

This Japanese Industrial Standard specifies the methods for determination of tin contained zirconium alloys.

2. General Matters

The general requirement of the analysis method shall be in accordance with JIS H 1650.

3. Classification of Methods

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

- (1) Potassium iodate titration method after aluminum reduction This method applies to the sample containing from 0.5 wt % or over to and excluding 3 wt % of tin.
- (2) 8-quinolinol extraction absorptiometry This method applies to the sample containing from 0.01 wt % or over to and excluding 3 wt % of tin.
- (3) Inductively coupled plasma emission spectrometry This method applies to the sample containing from 0.5 wt % or over to and excluding 3 wt % of tin.

4. Potassium Iodate Titration Method after Aluminum Reduction

4.1 Summary Decompose the sample with hydrochloric acid and the mixed acid of hydrofluoric acid and boric acid, add aluminum, heat, reduce tin and then titrate it with a potassium iodate standard solution.

4.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1)
- (3) Solution of hydrofluoric acid and boric acid Add 50 g of boric acid little by little to 100 ml of hydrofluoric acid to dissolve and store in a polyethylene bottle.
- (4) Aluminum sheet Those corresponding to 1100 or 1200 of JIS H 4000 shall be used.
- (5) Dry ice
- (6) Potassium iodide
- (7) Sodium hydrogencarbonate solution (saturated, approximately 100 g/l)

Applicable Standards: See page 11.

- (8) Standard tin solution (1 mg Sn/ml) Weigh out 0.500 g of tin (99.9 wt % or over) to a beaker (300 ml) containing a piece of platinum, add 100 ml of hydrochloric acid (1 + 1) to decompose by heating to the extent of not boiling while allowing the tin the piece of platinum to contact each other. After cooling down to room temperature, transfer the solution to a 500-ml volumetric flask while rinsing the beaker with hydrochloric acid (1 + 5), and dilute the solution with hydrochloric acid (1 + 5) up to the marked line.
- (9) Potassium iodate standard solution Take 1 g of sodium hydroxide and 10 g of potassium iodide in a beaker (300 ml), and add 200 ml of water to dissolve them. Dissolve 0.60 g of potassium iodate, and transfer this solution to a 1000-ml volumetric flask to dilute with water up to the marked line.

The amount of tin corresponding to 1 ml of this solution shall be calculated as follows.

Take accurately 20 ml of the standard tin solution [(8)] in an Erlenmeyer flask (300 ml), add 100 ml of hydrochloric acid (1 + 1) and 5 ml of mixed solution [(3)] of hydrofluoric acid and boric acid, heat the solution up to 40 to 50°C, and hereafter carry out the procedure of 4.5.2 (2) to 4.5.3. Calculate the amount of tin corresponding to 1 ml of potassium iodate standard solution from the formula given below. The blank test shall be carried out as follows. Take 100 ml of hydrochloric acid (1 + 1) and 5 ml of the mixed acid of hydrofluoric acid and boric acid [(3)] into an Erlenmeyer flask (300 ml), and heat it up to 40 to 50°C. Hereafter carry out the procedure of 4.5.2 (2) to 4.5.3, provided that about 1 g of potassium iodide should be added before titration.

$$f = 0.001 \times \frac{20}{V_1 - V_2}$$

where,  $f$ : amount of tin corresponding to 1 ml of potassium iodate standard solution (g)

$V_1$ : volume of potassium iodate standard solution consumed for the test of standard tin solution (ml)

$V_2$ : volume of potassium iodate standard solution consumed for the blank test (ml)

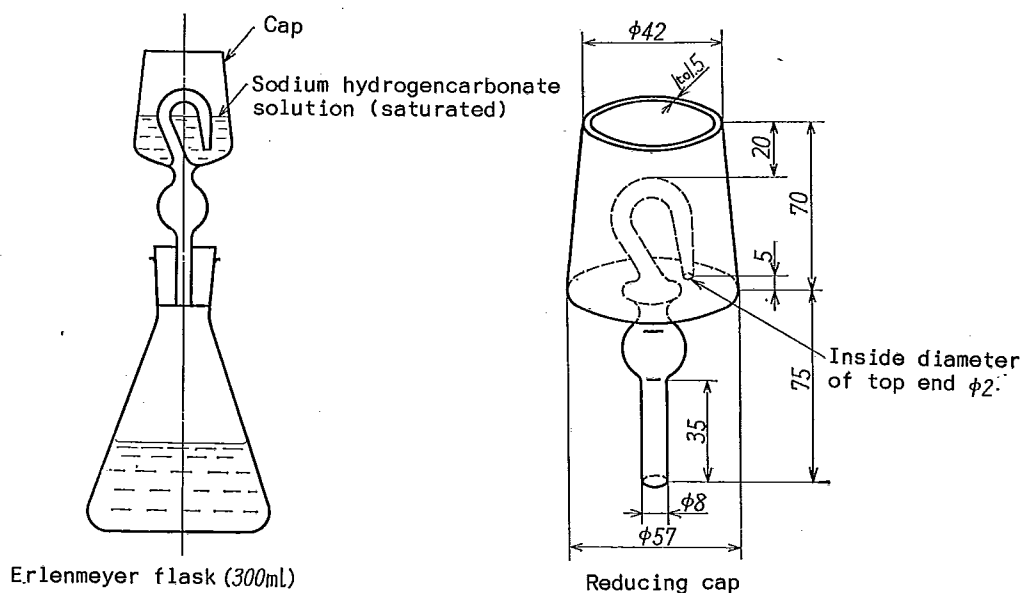
- (10) Starch solution Add a small amount of water to 1 g of starch (soluble), mix with kneading, and dissolve it by adding it to 100 ml of hot water while agitating the solution. Prepare this solution for each use, as a rule.

#### 4.3 Apparatus The following apparatus shall, as a rule, be used.

- (1) Reducing Apparatus This shall be as given in Figure. The reducing cap shall be so mounted that its lower end does not protrude from the rubber stopper.

Figure. Example of Reducing Apparatus

Unit: mm



4.4 Amount of Sample Weighed Out The amount of sample to be weighed out shall be specified in the Table below.

Table. Amount of Sample Weighed Out

| Tin content                  | wt % | Amount of sample weighed out g |
|------------------------------|------|--------------------------------|
| 0.5 or over to and excl. 1.0 |      | 2.0                            |
| 1.0 or over to and excl. 3   |      | 1.0                            |

#### 4.5 Operation

4.5.1 Decomposition of Sample Weigh out the sample, transfer it into an Erlenmeyer flask (300 ml), add about 50 ml of water, 70 ml of hydrochloric acid and 5 ml of the mixed solution of hydrofluoric acid and boric acid [4.2 (3)], decompose them by heating, and allow to cool.

4.5.2 Reduction of Tin The reduction of tin shall be carried out in the following procedure.

- (1) Heat the solution obtained in 4.5.1 up to the temperature of 40 to 50°C.
- (2) Immediately after adding 1 g of aluminum sheet [4.2 (4)], attach the reducing cap [4.3 (1)], and add about 50 ml<sup>(1)</sup> of sodium hydrogencarbonate solution (saturated).