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Methods for determination of bismuth in aluminium and aluminium alloys

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for revision of Japanese Industrial Standard submitted by the Japan Aluminium Association (JAA)/the Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law. Consequently **JIS H 1364 : 1971** is revised, and the method for determination of bismuth in the former **JIS H 1364** is replaced by this Standard.

Though in **JIS H 1364 : 1971**, the method for determination of lead and the method for determination of bismuth were specified together, as a result of the revision they are separately specified in the following individual standards.

JIS H 1364 *Methods for determination of bismuth in aluminium and aluminium alloys*

JIS H 1366 *Methods for determination of lead in aluminium and aluminium alloys*

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Methods for determination of bismuth in aluminium and aluminium alloys

1 Scope This Japanese Industrial Standard specifies the methods for determination of bismuth in aluminium and aluminium alloys.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS H 1306 *Methods for atomic absorption spectrometric analysis of aluminium and aluminium alloys*

JIS H 1307 *Methods for inductively coupled plasma emission spectrometric analysis of aluminium and aluminium alloys*

JIS H 1351 *General rules for chemical analysis of aluminium and aluminium alloy*

JIS P 3801 *Filter paper (for chemical analysis)*

3 General requirements General requirements common to the analysis shall conform to JIS H 1351.

4 Classification of determination methods The methods for determination of bismuth shall conform to the appropriate one among those specified in the following:

- a) **Electrolytic separation disodium ethylenediamine tetraacetate titrimetric method** This method is applied to the sample of a bismuth content of 0.1 % (m/m) up to and including 1.0 % (m/m).
- b) **Thiourea absorptiometry** This method is applied to the sample of a bismuth content of 0.1 % (m/m) up to and including 1.0 % (m/m).
- c) **Bromide and methyltriethylammonium bromide extraction atomic absorption spectrometry** This method is applied to the sample of a bismuth content of 0.003 % (m/m) up to and including 0.1 % (m/m).
- d) **Atomic absorption spectrometry** This method is applied to the sample of a bismuth content of 0.1 % (m/m) up to and including 1.0 % (m/m).
- e) **Inductively coupled plasma emission spectrometric analysis** This method is applied to the sample of a bismuth content of 0.01 % (m/m) up to and including 1.0 % (m/m).

5 Electrolytic separation disodium ethylenediamine tetraacetate titrimetric method

5.1 Summary After decomposing a sample by a sodium hydroxide solution, make it acid by adding nitric acid. Carry out electrolysis by using a platinum electrode, and after decomposing bismuth, lead, copper and the like with nitric acid which are deposited on an anode and a cathode, adjust the concentration of acid of the solution.

Partially take a specific amount of the solution and titrate it with disodium ethylenediamine tetraacetate (hereafter referred to as "EDTA2Na") standard solution by using Xylenol Orange (hereafter referred to as "XO") as an indicator.

5.2 Reagents The reagents shall be as follows.

- a) **Nitric acid**
- b) **Nitric acid (1+1)**
- c) **Sodium hydroxide solution** Dissolve 200 g of sodium hydroxide in water, and make 1 000 ml with water. Reserve the solution in a polyethylene bottle, and use the supernatant.
- d) **Hydrogen peroxide (1+9)**
- e) **0.01 mol/L EDTA2Na standard solution** Dissolve 3.72 g of disodium ethylenediamine tetraacetate dihydrate in water, dilute accurately to 1 000 ml with water, and reserve it in a polyethylene bottle.

Obtain the bismuth equivalent amount of this solution according to the following procedure.

Weigh out 1.000 g of bismuth [99.9 % (m/m) or higher], transfer it into a beaker (200 ml), cover it with a watch glass, add 30 ml of nitric acid (1+1), and decompose it by gently heating. After cooling to ordinary temperature, rinse the bottom surface of the watch glass and the inner wall of the beaker with water, remove the watch glass, transfer the solution to a one-mark volumetric flask of 500 ml by using water, and dilute to the mark with water. Accurately take 20 ml from this solution into a beaker (300 ml). After diluting to about 100 ml with water, carry out hereafter the operation according to the procedure of 5.5.2, titrate it with 0.01 mol/L EDTA2Na standard solution, and obtain the bismuth equivalent amount according to the following formula.

$$f_{\text{Bi}} = \frac{0.04}{V_1 - V_2}$$

where, f_{Bi} : bismuth equivalent amount of 0.01 mol/L EDTA2Na standard solution of 1 ml (g)

0.04: bismuth amount in standard bismuth solution of 20 ml (g)

V_1 : used amount of 0.01 mol/L EDTA2Na standard solution (ml)

V_2 : used amount of 0.01 mol/L EDTA2Na standard solution in blank test (ml)

- f) **XO solution** Dissolve 0.1 g of Xylenol Orange in water, and dilute it to 100 ml with water.

5.3 Apparatus and device The apparatus and device shall be as follows.

- a) **Cylindrical platinum electrode** For both the anode and cathode, the cylindrical platinum electrode specified in Attached Fig. 1 shall be used.
- b) **Semi-circular watch glass** For the semi-circular watch glass, the watch glass whose shape is similar to that given in Attached Fig. 2 shall be used.