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Methods for determination of lead in aluminium and aluminium alloys

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee, as the result of proposal for establishment 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. By this establishment, **JIS H 1364 : 1971** is revised, and the method for determination of lead in the former **JIS H 1364** is replaced with 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 this 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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In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

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Methods for determination of lead in aluminium and aluminium alloys

1 Scope This Japanese Industrial Standard specifies the methods for determination of lead 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 edition 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 lead 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 lead content of 0.1 % (m/m) up to and including 1.0 % (m/m).
- b) **Bromide and methyltriethylammonium bromide extraction atomic absorption spectrometry** This method is applied to the sample of a lead content of 0.003 % (m/m) up to and including 0.1 % (m/m).
- c) **Atomic absorption spectrometry** This method is applied to the sample of a lead content of 0.1 % (m/m) up to and including 1.0 % (m/m).
- d) **Inductively coupled plasma emission spectrometric analysis** This method is applied to the sample of a lead 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 with sodium hydroxide solution, make the solution acid by adding nitric acid. Carry out electrolysis by using a platinum electrode. Decompose bismuth, lead, copper and the like with nitric acid, which are deposited on an anode and a cathode. Partially take a specific amount of the solution, and adjust the pH with sodium acetate. Thereafter, mask the copper by adding thiourea, 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, 1+3)**
- c) **Sodium hydroxide solution** Dissolve 200 g of sodium hydroxide in water, and dilute to 1 000 ml with water. Reserve this solution in a polyethylene bottle, and use the supernatant.
- d) **Hydrogen peroxide (1+9)**
- e) **Sodium acetate solution (300 g/L)**
- f) **Thiourea solution (100 g/L)**
- g) **0.01 mol/L EDTA2Na standard solution** Dissolve 3.72 g of disodium EDTA dihydrate in water, dilute accurately to 1 000 ml with water, and reserve in a polyethylene bottle.

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

- 1) **Standardization of bismuth equivalent amount:** Weigh out 1.000 g of bismuth [99.9 % (*m/m*) or higher], transfer into a beaker (200 ml), cover with a watch glass, add 30 ml of nitric acid (1+1), and decompose by gently heating. After cooling to ordinary temperature, wash the bottom surface of the watch glass and the inner wall of the beaker with water, remove the watch glass, transfer the solution into a one-mark volumetric flask of 500 ml by using water, and dilute to the mark with water to make standard bismuth solution. Sample accurately 20 ml from the solution into a beaker (300 ml). After diluting to about 100 ml with water, add 0.5 ml of XO solution [5.2 h)], mix by shaking sufficiently, titrate with 0.01 mol/L EDTA2Na standard solution [5.2 g)], and deem the point at which the reddish purple color changes to yellow color as the end point. Obtain the bismuth equivalent amount from the used amount of 0.01 mol/L EDTA2Na standard solution [5.2 g)] according to the following formula.

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

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

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

- 2) **Standardization of lead equivalent amount:** Weigh out 1.000 g of lead [99.9 % (*m/m*) or higher], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of nitric acid (1+3), decompose by gently heating, and expel nitrogen oxide by further heating. After cooling to ordinary temperature,