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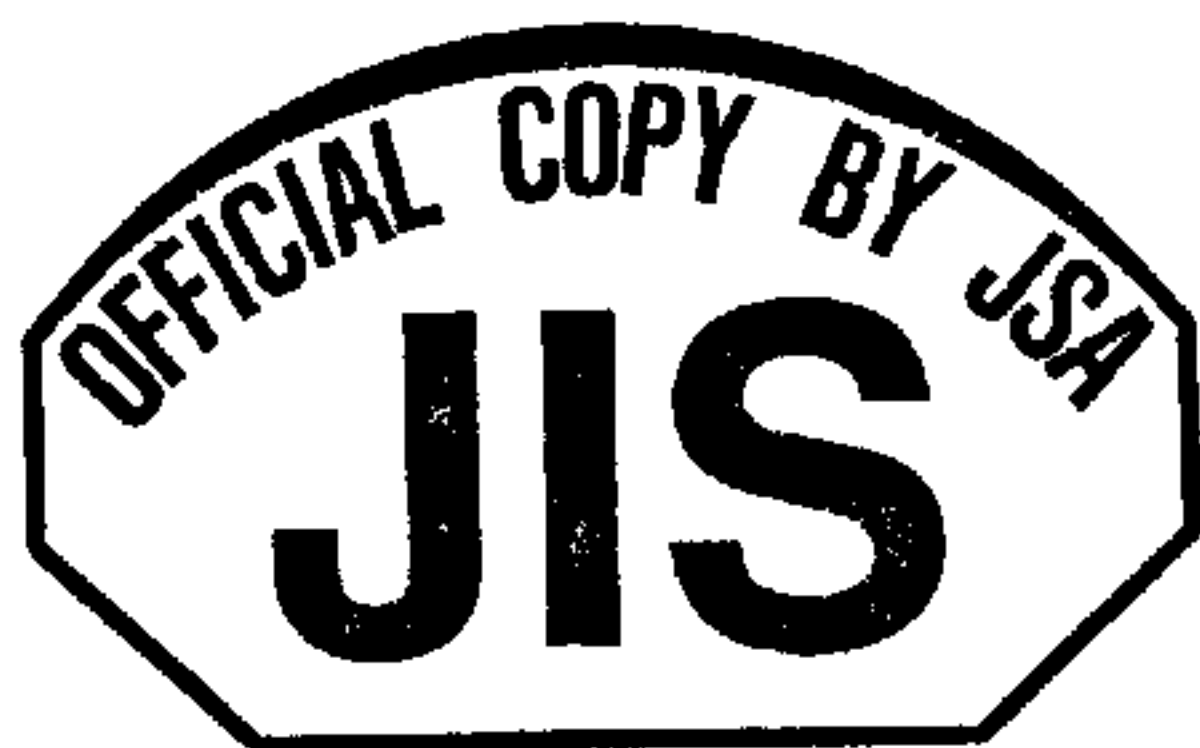
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JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of
Die Casting Zinc Alloy

JIS

H 1551-1964
(Reaffirmed: 1983)



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J I S

Methods for Chemical Analysis of H 1551-1964
Die Casting Zinc Alloy (Reaffirmed: 1983)

1. General Rules

1.1 Scope This Japanese Industrial Standard specifies the methods for chemical analysis of zinc alloy ingot for die casting specified in JIS H 2201 and zinc alloy die castings specified in JIS H 5301.

1.2 General Matters General matters common to the methods for chemical analysis shall be in accordance with JIS K 0050.

1.3 Sampling and Treating of Sample

1.3.1 The cutting of sample shall be carried out in accordance with the following each item.

- (1) When chips are taken from a cast sample or a product sample, the sample shall be bored at the central part, near both ends, etc. thereof, and shall be penetrated by boring vertically that surface, or otherwise the cutting shall be carried out by other methods so that the chipped sample can completely have the same quality as that of product.
- (2) When a chipping sample is taken by boring or the like, after preliminarily cleaning a drill and other tools by using alcohol or the like, the sample surface shall be cleaned and drilled by giving force to a degree not to oxidize chips without using oils and other lubricants.
- (3) When the chipping sample is of string state, after cutting it to about 30 mm max. by using clean scissors, they shall be sufficiently mixed.
- (4) All the chipped sample shall be completely collected to prepare the sample. After carefully removing iron scraps or the like by using a strong magnet, they are sufficiently mixed.

To prevent oxidation, the chipping sample shall be put in a suitable vessel and sealed to prepare the analysis sample.

1.3.2 The prepared sample is transferred onto an evaporating dish, put into the desiccator put with desiccating agent and let to stand for 1 hour min. Thereafter, it is weighed out.

1.3.3 For weighing out of the analysis sample, a chemical balance is used.

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1.4 Instruments

1.4.1 For measurement at a high temperature, a thermocouple shall, as a rule, be used. When the thermocouple is difficult to be used, an optical pyrometer may be used.

1.4.2 For the optical pyrometer, a filter photoelectric photometer or a photoelectric spectrophotometer shall be used.

1.4.3 The filter photoelectric photometer shall be equipped with at least 4 pieces of filters capable of obtaining monochromatic light as far as possible within a range of the lowest limit 400 to 700 mμ in wavelength.

1.4.4 Unless otherwise specified when the absorbance is measured, water shall be used for contrast solution.

1.4.5 General matters concerning polarographic method shall be in accordance with JIS K 0111.

1.5 Reagents For the reagents to be used for absorptiometry, cares shall be especially taken about its purity. When there is a fear to contain harmful impurities, they shall be preliminarily necessarily verified.

1.6 Blank Test When analyzed, the analysis value shall be connected by carrying out a blank test about reagents only over all the operation. However, when operations corresponding to a blank test are especially included in the analytical operation of absorptiometry or the like, this rule does not apply.

1.7 Number of Times for Analysis Concerning the same sample, the analytical operation shall, as a rule, be carried out not less than two times.

1.8 Expression of Analytical Results The analytical value shall be expressed by percentage and the rounding off of the numerical value shall be carried out as follows:

Alloy constituents other than zinc and impurities shall be obtained to the place next to a specified one and rounded off to the specified place in accordance with JIS Z 8401.

1.9 Investigation of Analytical Results It shall be confirmed by verification analysis or other suitable methods that the obtained analytical value satisfies the precision and bias within an estimated range.

2. Method for Determination of Aluminium

2.1 The method for determination of aluminium shall be in accordance with volumetric method.

2.2 Volumetric Method

2.2.1 Summary After decomposing a sample with hydrochloric acid and hydrogen peroxide water, pH is regulated at about 4. Aluminium is precipitated by adding ammonium benzoate and filtered.

The precipitate is dissolved with hydrochloric acid, added with disodium ethylenediaminetetraacetate (EDTA) standard solution and pH is regulated at 5.2 to 5.5. The solution is back-titrated with zinc standard solution and aluminium is determined.

2.2.2 Reagents and Their Preparation Methods The reagents to be used for operation in the text and their preparation methods shall be as follows:

- (1) Hydrochloric acid (1+1, 1+11)
- (2) Nitric acid (1+1)
- (3) Sulfuric acid (1+1)
- (4) Aqueous ammonia (1+1, 1+14)
- (5) Hydrogen peroxide water (3 w/v%)
- (6) Hydroxylamine hydrochloride solution (10 w/v%)
- (7) Ammonium benzoate solution (10 w/v%) 100 g ammonium benzoate is added with a suitable amount of water and dissolved by heating. After cooling, it is diluted to 1 l with water. Further, it may be prepared with benzoic acid and aqueous ammonia.
- (8) Buffer solution 83 ml hydrochloric acid is diluted with about 700 ml water and added with 94 g ammonium acetate. After dissolving it, the solution is diluted to 1 l with water. pH of this solution is of about 4.
- (9) Ammonium benzoate washing liquid 10 g ammonium benzoate is added with 5 ml acetic acid and water to 1 l and dissolved by heating.
- (10) Hexamine solution (30 w/v%) 300 g hexamethylenetetramine is dissolved with water to make 1 l of the solution.
- (11) M/100 aluminium standard solution 0.270 g refined aluminium metal (99.99% min. in purity) is weighed out and decomposed by adding 10 ml sodium hydroxide solution (20 w/v%). After slightly acidifying by using hydrochloric acid (1+1), it is accurately diluted to 1 l by adding water.

- (12) M/100 zinc standard solution 0.6537 g special class zinc metal (99.99% min. in purity) is weighed out, added with 10 ml hydrochloric acid (1+1), dissolved by heating and concentrated. After removing free hydrochloric acid as far as possible, it is accurately diluted to 1 l by adding water.
- (13) M/100 disodium ethylenediaminetetraacetate (EDTA) standard solution 3.723 g EDTA (2 hydrate) is dissolved with water, accurately diluted to 1 l and stored in a polyethylene vessel. The titer of this standard solution is obtained as follows:

25 ml M/100 zinc standard solution is accurately taken into a beaker (200 ml in capacity), and neutralized by dropping aqueous ammonia (1+14) utilizing 5 to 6 drops 2.5 - dinitrophenol solution as indicator. Thereafter, it is added with 4 ml hydrochloric acid (1+11) and 5 ml hexamine solution (30 w/v%) and diluted to about 100 ml with water. It is added with 0.7 to 0.8 ml Xylenol Orange solution, titrated by using M/100 EDTA standard solution until the colour of solution turns yellow from red and its titer is obtained from the following formula:

$$F = \frac{25}{V}$$

where F : titer of M/100 EDTA standard solution

V : used amount of M/100 DETA standard solution (ml)

- (14) 2.5-dinitrophenol solution 0.1 g 2.5-dinitrophenol is dissolved with 50 ml ethyl alcohol (95 v/v%) and diluted to 100 ml by adding water.
- (15) Xylenol Orange solution 0.1 g Oxylenol Orange is dissolved with 50 ml ethyl alcohol (95 v/v%) and diluted to 100 ml by adding water.

2.2.3 Weighing out Amount of Sample It shall be 0.2 g.

2.2.4 Operation The determination operation shall be carried out in accordance with the following procedures:

- (1) Weigh out a sample into a beaker (300 ml in capacity)⁽¹⁾, cover it with a watch glass and add 10 ml hydrochloric acid (1+1). When the violent reaction has been ended, decompose a great portion of sample by quietly heating it, add 5 ml hydrogen peroxide water (3 v/v %), mix them by shaking and completely dissolve copper or the like. Boil the solution and decompose excess hydrogen peroxide.

- (2) Add aqueous ammonia (1+1). When the precipitates of aluminium or the like are slightly generated, dissolve these precipitates by dropping hydrochloric acid (1+1) and dilute it to about 150 ml with water.

Add 10 ml hydroxylamine hydrochloride solution (10 w/v%) and 20 ml buffer solution and boil them for 2 to 3 minutes. Thereafter, it is gradually added with 20 ml warm ammonium benzoate solution (10 w/v%) ⁽²⁾. When boiling starts by heating it again, transfer it onto a water bath and let it stand still for about one hour.

- (3) Filter it by using a filter paper (class 5 B). After washing it 4 to 5 times with warm ammonium benzoate washing liquid ⁽³⁾, transfer it into the original beaker by washing with warm water, pour 10 ml warm hydrochloric acid (1+1) from above the filter paper, receive the filtrate with the original beaker, wash it 4 to 5 times with warm water and dissolve the precipitate by heating. Pour this warm solution from above the original filter paper, dissolve the precipitate adhered to the filter paper and wash it 4 to 5 times with warm water.
- (4) Receive the filtrate and washings with a beaker (300 ml in capacity), accurately add 35 ml M/100 EDTA standard solution, add 5 to 6 drops of 2.5 - dinitrophenol solution as indicator and neutralize it with aqueous ammonia (1+1). Thereafter, boil it for 2 to 3 minutes. Cool it to room temperature, add 4 ml hydrochloric acid (1+1) and 5 ml hexamine solution (30 w/v%) ⁽⁴⁾ and dilute it to about 150 ml with water. Add 0.7 to 0.8 ml Xylenol Orange solution thereto and titrate it by using M/100 zinc standard solution until the colour of solution turns faint red from yellow.
- (5) Separately, weigh out 0.19 g special zinc metal into a beaker (300 ml in capacity) as reference sample, and decompose it by using hydrochloric acid (1+1) and hydrogen peroxide water (3 v/v%) in accordance with 2.2.4 (1), as appropriate, accurately add 30 ml M/100 aluminium standard solution, hereafter operate it in accordance with the procedures on and after 2.2.4 (2) and obtain a corrected value ⁽⁵⁾.

- (6) Calculate the content of aluminium from the following formula:

$$\text{Aluminium (\%)} = \frac{(V_1 - V_2 - V_3) \times 0.0002698}{W} \times 100$$

where V_1 : additional amount of M/100 EDTA standard solution (ml)

V_2 : used amount of M/100 zinc standard solution (ml)

V_c : corrected value (ml)

W : weighing out amount of sample (g)

Notes (1) The separation of zinc, copper, etc. can be carried out by the following mercury cathode electrolysis method:

Weigh out 2 g a sample into a beaker (300 ml in capacity), dissolve it by adding 20 ml nitric acid (1+1), add 15 ml sulfuric acid (1+1) and generate white fume of sulfuric acid near to dryness by heating. After standing to cool, add 50 ml warm water, dissolve soluble salts by warming, transfer it into a mercury cathode electrolyser (refer to Attached Fig. 1), dilute the amount of solution to about 100 ml, utilize a platinum anode, electrolyze it by a current density of about 0.1 A/cm² and separate zinc, copper, etc.

Separate the remainder electrolyte from the mercury layer, transfer it into a measuring flask of 250 ml, accurately dilute it to 250 ml by using water, accurately take 25 ml therefrom into a beaker (300 ml in capacity), hereafter carry out the operation of 2.2.4 (4) and determine aluminium.

- (2) When added at a time, or otherwise when intensively boiled after addition, the precipitate sometimes overflows in a foamy state.
- (3) When the temperature of ammonium benzoate washing liquid is low, crystal is deposited on a filter paper. Therefore, warm solution shall be used.
- (4) Suitable pH is of 5.2 to 5.5. When pH does not become within this range, a pH meter is used and pH is regulated by dropping hydrochloric acid (1+11) or aqueous ammonia (1+14).
- (5) The correction value by a reference sample shall be obtained from the following formula:

$$V_c = V_1 - V_2 - V_3$$

where V_c : correction value (ml)

V_1 : additional amount of M/100 EDTA standard solution (ml)

V_2 : used amount of M/100 zinc standard solution (ml)

V_3 : additional amount of M/100 aluminium standard solution (ml)

3. Method for Determination of Magnesium

3.1 The method for determination of magnesium shall be in accordance with volumetric method.

3.2 Volumetric Method

3.2.1 Summary A sample is decomposed with nitric acid, added with triethanolamine-sodium hydroxide solution and potassium cyanide solution and magnesium is filtered by precipitation. After dissolving the precipitate with nitric acid, it is added with buffer solution and potassium cyanide solution, titrated with EDTA standard solution utilizing Eriochrome Black T as indicator and magnesium is determined.

3.2.2 Reagents and Their Preparation Methods The reagents to be used for operation in the text and their preparation methods shall be as follows:

- (1) Nitric Acid (1+1, 1+20)
- (2) Sodium hydroxide solution (30 w/v%)
- (3) Potassium cyanide solution (40 w/v%)
- (4) Triethanolamine solution (50 v/v%)
- (5) Triethanolamine - sodium hydroxide washing liquid 20 ml triethanolamine solution (50 v/v%) is added with 20 ml sodium hydroxide solution (30 w/v%) and diluted to 1 l with water.
- (6) Buffer solution 70 g ammonium chloride is dissolved with 750 ml aqueous ammonia and water and diluted to 1 l with water, pH of this solution is of 10.
- (7) Standard magnesium solution 0.500 g magnesium (99.9% min. in purity, class 1 metal of JIS H 2150 or special class reagent equivalent product of JIS K 8875) is weighed out, added with water and a small amount of hydrochloric acid and decomposed by heating. After cooling, it is transferred into a measuring flask of 1 l, diluted up to the marked line with water and mixed by shaking to prepare the stock solution. When used, it is accurately diluted 10 times with water to prepare the standard magnesium solution. 1 ml of this solution contains 5 µg magnesium.
- (8) M/100 disodium ethylenediaminetetraacetate (EDTA) standard solution 3.723 g EDTA (2 hydrate) is dissolved with water, accurately diluted to 1 l and stored in a polyethylene vessel. The standardization of the titer of this solution shall be carried out as follows: