

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for atomic absorption
spectrometric analysis of
aluminium and aluminium alloys**

JIS H 1306—1992

Translated and Published

by

Japanese Standards Association

Printed in Japan

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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 atomic absorption spectrometric
analysis of aluminium and aluminium alloys

H 1306-1992

1. Scope This Japanese Industrial Standard specifies the methods for atomic absorption spectrometric analysis of aluminium and aluminium alloys.

Remarks 1. The following standards are cited in this Standard:

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

JIS K 0050 General rules for chemical analysis

JIS K 0121 General rules for atomic absorption spectrochemical analysis

2. Corresponding International Standards to this Standard are given as follows:

ISO 3256:1977 Aluminium and aluminium alloys - Determination of magnesium - Atomic absorption spectrophotometric method

ISO 3980:1977 Aluminium and aluminium alloys - Determination of copper - Atomic absorption spectrophotometric method

ISO 3981:1977 Aluminium and aluminium alloys - Determination of nickel - Atomic absorption spectrophotometric method

ISO 4192:1981 Aluminium and aluminium alloys - Determination of lead content - Flame atomic absorption spectrometric method

ISO 4193:1981 Aluminium and aluminium alloys - Determination of chromium content - Flame atomic absorption spectrometric method

ISO 5194:1981 Aluminium and aluminium alloys - Determination of zinc content - Flame atomic absorption spectrometric method

2. General matter General matter common to the chemical analysis shall be in accordance with JIS H 1351, JIS K 0050, and JIS K 0121.

3. Determining elements and determining ranges The determining elements and the determining ranges shall be as given in Table 1.

Table 1. Determining elements and determining ranges

Determining element	Determining range % (m/m)
Iron	0.005 or over to and excl. 1.5
Copper	0.005 or over to and excl. 5.0
Manganese	0.005 or over to and excl. 1.5
Zinc	0.005 or over to and excl. 6.0
Magnesium	0.005 or over to and excl. 5.0
Chromium	0.01 or over to and excl. 0.5
Nickel	0.005 or over to and excl. 3.0
Bismuth	0.1 or over to and excl. 1.0
Lead	0.1 or over to and excl. 1.0

4. Method for determination of iron

4.1 Summary After decomposing a sample with hydrochloric acid and hydrogen peroxide, atomize the solution into the air-acetylene flame of an atomic absorption photometer, and measure the absorbance.

4.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 1)
- (2) Nitric acid (1 + 1)
- (3) Hydrofluoric acid
- (4) Aluminium Aluminium of which the purity is as high as possible without containing iron, or aluminium of which the iron content is known and is as low as possible.
- (5) Hydrogen peroxide
- (6) Tin solution Weigh out 0.1 g of tin [99.9 % (m/m) or over], transfer into a beaker (200 ml), add 30 ml of hydrochloric acid (1 + 1), and decompose by heating at 50°C to 80°C making platinum contact therewith. After cooling to ordinary temperature, make up to 100 ml in the quantity of solution with hydrochloric acid (1 + 5).
- (7) Nickel solution Weigh out 0.1 g of nickel [99.9 % (m/m) or over], transfer into a beaker (200 ml), add 10 ml of nitric acid (1 + 1), and decompose by gently heating. After cooling to ordinary temperature, make up to 100 ml in the quantity of solution with water.

- (8) Standard iron solution A (1000 $\mu\text{gFe/ml}$) Weigh out 1,000 g of iron [99.9 % (m/m) or over], 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, and remove the watch glass. Transfer the solution into a 1000 ml volumetric flask by using water, and dilute to the mark with water to make the standard iron solution A.
- (9) Standard iron solution B (100 $\mu\text{gFe/ml}$) Dilute a necessary quantity of standard iron solution A [(8)] accurately 10 times with water every service, to make the standard iron solution B.

4.3 Weighing out quantity of sample The weighing out quantity of a sample shall be 1.00 g.

4.4 Operation

4.4.1 Preparation of sample solution Prepare sample solution in accordance with the following procedure:

- (1) Weigh out a sample, and transfer into a beaker (300 ml).
- (2) Cover with a watch glass, add 30 ml of hydrochloric acid (1 + 1), and decompose by gently heating⁽¹⁾. Add 1 ml of hydrogen peroxide to this solution⁽²⁾, and decompose the sample thoroughly by heating⁽³⁾⁽⁴⁾. After cooling to ordinary temperature, wash the bottom surface of the watch glass and the inner wall of the beaker with water, and remove the watch glass. Transfer the solution into a 100 ml volumetric flask by using water, and dilute to the mark with water.
- (3) Partially take the solution into a 100 ml volumetric flask in accordance with Table 2 corresponding to the iron content in the sample, and dilute to the mark with water⁽⁵⁾.

Table 2. Aliquot quantity

Iron content % (m/m)	Aliquot quantity ml
0.1 or over to and excl. 0.5	50.0
0.5 or over to and excl. 1.5	20.0

Notes (1) Where a sample is difficult to be decomposed, add 2 ml of tin solution [4.2 (6)] or 2 ml of nickel solution [4.2 (7)].

(2) Since metallic copper occasionally remains where the copper content is high, further add hydrogen peroxide.

(3) Where insoluble substances are observed, filter with filter paper (class 5B). After washing with warm water, join the filtrate and washings to be reserved. Transfer the filter paper and the insoluble substances into a platinum crucible (No.20), raise gradually temperature, and heat at 700°C to 800°C. After

incinerating the filter paper thoroughly, allow to cool. Add 5 ml of nitric acid (1 + 1), add little by little about 3 ml of hydrofluoric acid, and decompose the insoluble substances thoroughly. After drying up by further heating, add several drops of hydrochloric acid (1 + 1), dissolve the salts, and join the solution to the filtrate and washings which are reserved by using a small quantity of water.

- (⁴) Since a lower value is occasionally indicated when hydrogen peroxide remains, heat sufficiently.
- (⁵) Where the content of iron in the sample is under 0.1 % (m/m), do not perform the operation of (3).

4.4.2 Measurement of absorbance Atomize a portion of the solution obtained in (2) or (3) of 4.4.1 into the air-acetylene flame of the atomic absorption photometer of which the zero point is regulated by using water, and measure the absorbance at 248.3 nm or 372.0 nm wavelength.

4.5 Blank test The absorbance of the solution to which the standard iron solution to be obtained in the operation of 4.6 is not added, shall be taken as the absorbance of the blank test.

4.6 Preparation of working curve Weigh out each 1.00 g of aluminium [4.2 (4)], transfer into several beakers (300 ml), and add standard iron solution stepwise in accordance with Table 3 corresponding to the iron content in the sample. Hereinafter, perform the same operation as for the sample in accordance with the procedure of 4.4.1 (2) to 4.4.2 in parallel with the sample, prepare the relation curve between the obtained absorbance and the quantity of iron(⁶) added as the standard iron solution, and translate said relation curve in parallel therewith so as to pass through the original point to make the working curve.

Note (⁶) Where the operation of 4.4.1 (3) is performed, the quantity (g) of iron added as the standard iron solution in the aliquot solution.

Table 3. Additive quantity of standard iron solution

Iron content % (m/m)	Standard iron solution to be used	Additive quantity of standard iron solution ml
0.005 or over to and excl. 0.1	B [4.2 (9)]	0 to 10.0
0.1 or over to and excl. 0.5	A [4.2 (8)]	0 to 5.0
0.5 or over to and excl. 1.5	A [4.2 (8)]	0 to 15.0

4.7 Calculation The calculation shall be in accordance with either of the following provisions:

- (1) For no aliquot taken in 4.4.1 Obtain the quantity of iron from the absorbances obtained in 4.4.2 and 4.5 and the working curve prepared in 4.6, and calculate the content of iron in the sample from the following formula.

$$\text{Iron \% (m/m)} = \frac{A_1 - (A_2 - A_3)}{m} \times 100$$

where, A_1 : detected quantity of iron in sample solution (g)
 A_2 : detected quantity of iron in blank test solution (g)
 A_3 : quantity of iron contained in 1.00 g of aluminium [4.2 (4)] (g)
 m : weighing out quantity of sample (g)

- (2) For aliquot taken in 4.4.1 Obtain the quantity of iron from the absorbances obtained in 4.4.2 and 4.5 and the working curve prepared in 4.6, and calculate the content of iron in the sample from the following formula.

$$\text{Iron \% (m/m)} = \frac{(A_1 - A_2) \times \frac{100}{B} + A_3}{m} \times 100$$

where, A_1 : detected quantity of iron in aliquot sample solution (g)
 A_2 : detected quantity of iron in aliquot blank test solution (g)
 B : aliquot quantity of sample solution and blank test solution (ml)
 A_3 : quantity of iron contained in 1.00 g of aluminium [4.2 (4)] (g)
 m : weighing out quantity of sample (g)

5. Method for determination of copper

5.1 Summary After decomposing a sample with hydrochloric acid and hydrogen peroxide, atomize the solution into the air-acetylene flame of an atomic absorption photometer, and measure the absorbance.

5.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 1)
- (2) Nitric acid (1 + 1)
- (3) Hydrofluoric acid
- (4) Aluminium Aluminium of which the purity is as high as possible without containing copper or aluminium of which the copper content is known and is as low as possible.
- (5) Hydrogen peroxide
- (6) Tin solution As described in 4.2 (6).

6
H 1306-1992

- (7) Nickel solution As described in 4.2 (7).
- (8) Standard copper solution A (1000 $\mu\text{gCu/ml}$) Weigh out 1.000 g of copper (99.9 % (m/m) or over), 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, and remove the watch glass. Transfer the solution into a 1000 ml volumetric flask by using water, and dilute to the mark with water to make the standard copper solution A.
- (9) Standard copper solution B (500 $\mu\text{gCu/ml}$) Dilute a necessary quantity of standard copper solution A [(8)] accurately two times with water every service to make the standard copper solution B.
- (10) Standard copper solution C (100 $\mu\text{gZn/ml}$) Dilute only necessary quantity of standard copper solution A [(8)] accurately 10 times with water at each time of use to make the standard copper solution C.

5.3 Weighing out quantity of sample The weighing out quantity of a sample shall be as given in Table 4 according to the content of copper in the sample.

Table 4. Weighing out quantity of sample

Copper content % (m/m)	Weighing out quantity of sample g
0.005 or over to and excl. 1.5	1.00
1.5 or over to and excl. 5.0	0.20

5.4 Operation

5.4.1 Preparation of sample solution Prepare sample solution in accordance with the following procedure:

- (1) Weigh out a sample, and transfer into a beaker (300 ml).
- (2) Cover with a watch glass, add 30 ml of hydrochloric acid (1 + 1), and decompose by gently heating⁽⁷⁾. Add 1 ml of hydrogen peroxide to this solution⁽²⁾, and decompose the sample thoroughly by heating⁽³⁾⁽⁴⁾. After cooling to ordinary temperature, wash the bottom surface of the watch glass and the inner wall of the beaker with water, and remove the watch glass. Transfer the solution into a 100 ml volumetric flask when the copper content in the sample is under 1.5 % (m/m) and into a 200 ml volumetric flask when it is 1.5 % (m/m) or over by using water, and dilute to the mark with water.
- (3) Take the aliquot of the solution in accordance with Table 5 according to the content of copper in the sample, transfer into the volumetric flask as given in Table 5, and dilute up to the mark with water⁽⁸⁾.

Table 5. Aliquot quantity

Copper content % (m/m)	Aliquot quantity ml	Volumetric flask ml
0.1 or over to and excl. 0.5	50.0	100
0.5 or over to and excl. 1.5	20.0	100
1.5 or over to and excl. 5.0	20.0	200

Notes (7) Where the sample is difficult to be decomposed, add 2 ml of tin solution [5.2 (6)] or 2 ml of nickel solution [5.2 (7)].

(8) Where the content of copper in the sample is under 0.1 % (m/m), do not carry out the operation of (3).

5.4.2 Measurement of absorbance Atomize a portion of the solution obtained in (2) or (3) of 5.4.1 into the air-acetylene flame of the atomic absorption photometer of which the zero point is regulated by using water, and measure the absorbance at 324.8 nm or 327.4 nm wavelength.

5.5 Blank test The absorbance of the solution without addition of standard copper solution to be obtained in the operation of 5.6 shall be taken as the absorbance of the blank test.

5.6 Preparation of working curve Weigh out aluminium [5.2 (4)] so that the weighing out quantity given in Table 4 according to the copper content in a sample is equalized to the mass to the nearest 10 mg, transfer into several beakers (300 ml), and add stepwise standard copper solution in accordance with Table 6 according to the copper content in the sample. Hereinafter, perform the same operation as for the sample in accordance with the procedure of 5.4.1 (2) to 5.4.2 in parallel with sample, prepare the relation curve between the obtained absorbance and the quantity of copper⁽⁹⁾ added as standard copper solution, and translate the relation curve so as to pass through the original point in parallel with itself to make the working curve.

Note (9) The quantity (g) of copper added as standard copper solution in the aliquot solution where the operation of 5.4.1 (3) is performed.

Table 6. Additive quantity of standard copper solution

Copper content % (m/m)	Standard copper solution to be used	Additive quantity of standard copper solution ml
0.005 or over to and excl. 0.1	C [5.2 (10)]	0 to 10.0
0.1 or over to and excl. 0.5	A [5.2 (8)]	0 to 5.0
0.5 or over to and excl. 1.5	A [5.2 (8)]	0 to 15.0
1.5 or over to and excl. 5.0	B [5.2 (9)]	0 to 20.0