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JAPANESE INDUSTRIAL STANDARD

Methods for inductively coupled plasma emission spectrometric analysis of aluminium and aluminium alloys

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In the event of any doubt arising,
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Methods for inductively coupled plasma emission
spectrometric analysis of aluminium
and aluminium alloys

H 1307-1993

1. Scope This Japanese Industrial Standard specifies the methods for inductively coupled plasma emission spectrometric analysis of aluminium and aluminium alloys.

Remarks: The following standards are cited in this Standard.

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

JIS K 0050 General rules for chemical analysis

JIS K 0116 General rules for emission spectrochemical analysis

JIS Z 2611 General rules for photoelectric emission spectrochemical analysis of metal materials

JIS Z 8402 General rules for permissible tolerance of chemical analyses and physical tests

2. General items General items common to the chemical analyses shall be in accordance with JIS H 1351, JIS K 0050, JIS K 0116, JIS Z 2611 and JIS Z 8402.

3. Classification of the chemical analyses The chemical analysis shall be in accordance with either of the following methods.

- (1) Method A This method shall be applied to the samples of not less than 99.0 % (m/m) in aluminium content, and its determining elements and determination ranges shall be as given in Table 1.
- (2) Method B This method shall be applied to the samples of under 99.0 % (m/m) in aluminium content, and its determining elements and determination ranges shall be as given in Table 2.

Table 1. Determining elements and determination ranges of method A

Determining elements	Determination ranges % (m/m)
Copper	0.002 or over up to and incl. 0.25
Iron	0.002 or over up to and incl. 1.0
Manganese	0.002 or over up to and incl. 0.1
Magnesium	
Zinc	
Titanium	
Chromium	
Vanadium	

Table 2. Determining elements and determination ranges of method B

Determining elements	Determination ranges % (m/m)
Copper	0.01 or over up to and incl. 6.0
Iron	0.02 or over up to and incl. 1.5
Manganese	0.01 or over up to and incl. 2.0
Magnesium	0.01 or over up to and incl. 12.0
Zinc	0.01 or over up to and incl. 8.0
Nickel	0.01 or over up to and incl. 3.0
Titanium	0.01 or over up to and incl. 0.5
Chromium	
Lead	0.01 or over up to and incl. 1.0
Bismuth	
Zirconium	0.01 or over up to and incl. 0.5
Tin	
Vanadium	0.01 or over up to and incl. 0.25
Boron	

4. Method A

4.1 Summary After decomposing a sample with hydrochloric acid and hydrogen peroxide, atomize the solution into the argon plasma of an inductively coupled plasma (hereafter referred to as the "ICP") emission spectrometric apparatus, and measure the light emission intensity of a determining element.

4.2 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid (1 + 1)
- (2) Hydrofluoric acid
- (3) Sulfuric acid (1 + 1)
- (4) Hydrogen peroxide
- (5) Aluminium Aluminium of which the purity is as high as possible without containing the determining elements or aluminium having the known contents of the determining elements which are as low as possible.
- (6) Yttrium solution (1 mgY/ml) Weigh out 1.270 g of diyttrium trioxide [99.9 % (m/m) or over], transfer into a beaker (200 ml), add 50 ml of hydrochloric acid (1 + 1), and decompose by heating. After cooling to ordinary temperature, transfer into a 1000 ml volumetric flask by using water, and dilute to the mark with water.
- (7) Nickel solution Weigh out 0.1 g of nickel [99.9 % (m/m) or over], transfer into a beaker (200 ml), add 30 ml of nitric acid (1 + 1), and decompose by heating gently. After cooling to ordinary temperature, make up the volume to 100 ml with water.
- (8) Tin solution Weigh out 0.1 g of tin [99.9 % (m/m) or over], transfer on a platinum dish (No. 100), add 30 ml of hydrochloric acid, and decompose by heating at 50°C to 80°C. After cooling to ordinary temperature, make up the volume to 100 ml with water.
- (9) Standard copper solution A (100 µgCu/ml) Weigh out 0.100 g of copper [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of nitric acid (1 + 1), and decompose by heating gently. 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 standard copper solution A.
- (10) Standard copper solution B (10 µgCu/ml) This solution shall be made by dilution of standard copper solution A [(9)] accurately ten times with water for the required quantity just before use.
- (11) Standard iron solution A (500 µgFe/ml) Weigh out 0.500 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 heating gently. 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 standard iron solution A.

- (12) Standard iron solution B (100 $\mu\text{gFe/ml}$) This solution shall be made by dilution of standard iron solution A [(11)] accurately five times with water for the required quantity just before use.
- (13) Standard manganese solution A (100 $\mu\text{gMn/ml}$) Weigh out 0.100 g of manganese [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of hydrochloric acid (1 + 1), and decompose by heating gently. 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 standard manganese solution A.
- (14) Standard manganese solution B (10 $\mu\text{gMn/ml}$) This solution shall be made by dilution of standard manganese solution A [(13)] accurately ten times with water for the required quantity just before use.
- (15) Standard magnesium solution A (100 $\mu\text{gMg/ml}$) Weigh out 0.100 g of magnesium [99.9 % (m/m) or over], transfer into a beaker (300 ml), cover with a watch glass, and add 50 ml of water. Thereafter, add 20 ml of hydrochloric acid (1 + 1) little by little, and decompose by heating gently. 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 standard magnesium solution A.
- (16) Standard magnesium solution B (10 $\mu\text{gMg/ml}$) This solution shall be made by dilution of standard magnesium solution A [(15)] accurately ten times with water for the required quantity just before use.
- (17) Standard zinc solution A (100 $\mu\text{gZn/ml}$) Weigh out 0.100 g of zinc [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of hydrochloric acid (1 + 1), and decompose by heating gently. 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 standard zinc solution A.
- (18) Standard zinc solution B (10 $\mu\text{gZn/ml}$) This solution shall be made by dilution of standard zinc solution A [(17)] accurately ten times with water for the required quantity just before use.
- (19) Standard titanium solution A (100 $\mu\text{gTi/ml}$) Weigh out 0.100 g of titanium [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 80 ml of hydrochloric acid (1 + 1), and decompose by heating gently. After cooling to ordinary temperature, wash the bottom surface of the watch glass and the inner wall of the beaker with hydrochloric acid (1 + 1), and remove the watch glass. Transfer the solution into a 1000 ml volumetric flask by using hydrochloric acid (1 + 1), and dilute to the mark with hydrochloric acid (1 + 1) to make standard titanium solution A.
- (20) Standard titanium solution B (10 $\mu\text{gTi/ml}$) This solution shall be made by dilution of standard titanium solution A [(19)] accurately ten times with hydrochloric acid (1 + 1) for the required quantity just before use.

- (21) Standard chromium solution A (100 $\mu\text{gCr/ml}$) Weigh out 0.100 g of chromium [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of hydrochloric acid (1 + 1), and decompose by heating gently. 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 standard chromium solution A.
- (22) Standard chromium solution B (10 $\mu\text{gCr/ml}$) This solution shall be made by dilution of standard chromium solution A [(21)] accurately ten times with water for the required quantity just before use.
- (23) Standard vanadium solution A (100 $\mu\text{gV/ml}$) Weigh out 0.100 g of vanadium [99.9 % (m/m) or over], transfer into a beaker (200 ml), cover with a watch glass, add 20 ml of mixed acid (three pts vol of hydrochloric acid and one pt vol of nitric acid), decompose by heating gently, and evaporate near to dryness. Add 20 ml of hydrochloric acid, and dissolve the salts by heating gently. 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 standard vanadium solution A.
- (24) Standard vanadium solution B (10 $\mu\text{gV/ml}$) This solution shall be made by dilution of standard vanadium solution A [(23)] accurately ten times with hydrochloric acid (1 + 50) for the required quantity just before use.

4.3 Apparatus and measuring conditions The apparatus and measuring conditions shall be as follows.

- (1) ICP emission spectrometric apparatus The ICP emission spectrometric apparatus having a function which conforms to reference for reproducibility of (3) shall be used.
- (2) Selection of analytical line For the analytical line used for measuring the light emission intensity of a determining element, select, as a rule, the analytical line of a wavelength which does not receive spectral interference of a coexistent element. When it is difficult to select the analytical line which does not receive the spectral interference, preliminarily obtain the spectral overlap coefficient of the coexistent element to the determining element.

The method to obtain the spectral overlap coefficient shall be as follows.

- (a) Weigh out 0.500 g of aluminium [4.2 (5)], transfer into a beaker (200 ml), and add the standard solution of a coexistent element (*j*) which generates spectral overlap at the measuring wavelength of a determining element (*i*) so that the quantity of the coexistent element (*j*) becomes a certain appropriate quantity (for example, the maximum coexistent quantity when 0.500 g of an analysis object sample is weighed out.). Hereafter operate in accordance with the procedure of (2), (3) of 4.5.1 and 4.5.2 (1) or the procedure of (2), (3) of 4.5.1 and 4.5.2 (2)(¹).

- (b) Weigh out 0.500 g of aluminium [4.2 (5)], and transfer into a beaker (200 ml). Hereafter, operate in parallel with the operation of (a) according to the procedure of (2), (3) of 4.5.1 and 4.5.2 (1) or the procedure of (2), (3) of 4.5.1 and 4.5.2 (2)⁽¹⁾.
- (c) Operate in parallel with the operation of (a) according to the procedure of (a) to (c) of 4.7 (1) or the procedure of (a), (b) of 4.7 (2), and prepare the working curve of a determining element (*i*).
- (d) Calculate a spectral overlap coefficient from the following formula.

$$l_j = \frac{a_i - b_i}{m_j}$$

where, l_j : spectral overlap coefficient of coexistent element (*j*) to determining element (*i*)

a_i : quantity of apparent determining element (*i*) obtained from the working curve prepared in (c) and the light emission intensity or light emission intensity ratio obtained in (a) (g)

b_i : quantity of apparent determining element (*i*) obtained from the working curve prepared in (c) and the light emission intensity or light emission intensity ratio obtained in (b) (g)

m_j : quantity of coexistent element (*j*) joined in (a) (g)

Note ⁽¹⁾ The light emission intensity shall be measured with the wavelength of the analytical line of the determining element (*i*).

- (3) Selection of measuring conditions The measuring conditions (an analytical line, a solution atomization condition, a photometric condition, etc.) shall be set for each determining element so as to conform to both of the following two references for reproducibility.
- (3.1) Reference for reproducibility in lower limit area of determination In operation according to the following procedure, the standard deviation of ten determined values obtained for each determining element shall be not more than the upper limit value of the standard deviation given in Table 3.
 - (a) Weigh out 0.500 g of aluminium [4.2 (5)], transfer into a beaker (200 ml), and add the standard solution of a determination element to be examined on the reference for reproducibility according to Table 3. Thereafter, operate according to the procedure of (2), (3) of 4.5.1 and 4.5.2 (1) (a) or the procedure of (2), (3) of 4.5.1 and 4.5.2 (2) (a).
 - (b) Weigh out 0.500 g of aluminium [4.2 (5)], transfer into a beaker (200 ml), and operate in parallel with the operation of (a) according to the procedure of (2), (3) of 4.5.1 and 4.5.2 (1) (a) or the procedure of (2), (3) of 4.5.1 and 4.5.2 (2) (a).
 - (c) Carry out the operation of 4.5.2 (1) (b) or 4.5.2 (2) (b) by using the solution obtained in (a) and (b), and measure the light emission intensity of the determining element (*i*).

- (d) Operate in parallel with the operation of (a) according to the procedure of 4.7 (1) (a) and (b) or the procedure of 4.7 (2) (a).
- (e) Carry out the operation of 4.7 (1) (c) or 4.7 (2) (b) in parallel with the operation of (c) by using the solution obtained in (d), and prepare the working curve of the determining element (*i*).
- (f) Obtain the quantity of the determining element (*i*) from the light emission intensity obtained in (c) and the working curve prepared in (e), and calculate the determined value of the determining element (*i*) (content rate converted to the content rate in aluminium) to four places of decimals according to the following formula.

$$C_i = \frac{A_i - B_i}{0.500} \times 100$$

where, C_i : determined value of determining element (*i*)
[% (m/m)]

A_i : quantity of determining element (*i*) obtained from the light emission intensity obtained in (c) by using the solution prepared in (a) and the working curve prepared in (e) (g)

B_i : quantity of determining element (*i*) obtained from the light emission intensity obtained in (c) by using the solution prepared in (b) and the working curve prepared in (e) (g)

- (g) Obtain ten determined values by repeating the operation of (c), (e) and (f) ten times, and calculate the standard deviation.
- (3.2) Reference for reproducibility in upper limit area of determination In operation according to the following procedure, the standard deviation of ten determined values obtained for each determining element shall be not more than the upper limit value of the standard deviation given in Table 4.
- (a) Weigh out 0.500 g of aluminium [4.2 (5)], transfer into a beaker (200 ml), and add the standard solution of a determining element to be examined on the reference for reproducibility according to Table 4. Thereafter, operate according to the procedure of (2), (3) of 4.5.1 and 4.5.2 (1) (a) or the procedure of (2), (3) of 4.5.1 and 4.5.2 (2) (a).
 - (b) Operate according to (3.1) (b).
 - (c) Operate according to (3.1) (c).
 - (d) Operate according to (3.1) (d).
 - (e) Operate according to (3.1) (e).
 - (f) Operate according to (3.1) (f).
 - (g) Operate according to (3.1) (g).