

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for chemical analysis
of cadmium metal**

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In the event of any doubt arising,
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Methods for chemical analysis
of cadmium metal

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1. Scope

This Japanese Industrial Standard specifies the methods of chemical analysis of lead, copper, zinc, and iron in cadmium metal specified in JIS H 2113.

However, the method for photoelectric emission spectrochemical analysis shall be as specified in JIS H 1163.

Remarks: Applicable standards to this Standard are shown as follows:

- JIS H 0301 General Rules for Tests and Inspection of Base Metals
- JIS H 1163 Method for Photoelectric Emission Spectrochemical Analysis of Cadmium Metal
- JIS H 2113 Cadmium Metal
- JIS K 0050 General Rules for Chemical Analysis
- JIS K 0115 General Rules for Molecular Absorptiometric Analysis
- JIS K 0116 General Rules for Emission Spectrochemical analysis
- JIS K 0121 General Rules for Atomic Absorption Spectrochemical Analysis
- JIS Z 8401 Rules for Rounding off of Numerical Values

2. General matters

General matters common to chemical analysis shall be in accordance with JIS K 0050, JIS K 0115, JIS K 0116 and JIS K 0121.

3. Sampling and treating of sample

3.1 Sampling The sampling shall be as follows.

- (1) The sampling of an analytical sample shall be, as a rule, in accordance with 2.2 of JIS H 0301. However, the sample drilled off with a drill is cut to approx. 5 mm or under by using clean scissors.

- (2) In the case where the specification of (1) can not be applied to the sampling of the analytical sample, the sampling method shall be determined as agreed upon between the parties concerned with delivery.

3.2 Treatment of sample The treatment of a sample shall be as follows:

- (1) In order to prevent contamination due to foreign matters or the like, put the analytical sample into a glass container with a suitable cap, hermetically stopper, and reserve.
- (2) In the case where oil or the like are feared to stick to the surface of an analytical sample, preliminarily wash the sample with ethanol, acetone, etc. and dry.
- (3) In the case where the analytical sample is used for quantitative analysis, preliminarily take a necessary quantity into a beaker, add hydrochloric acid (1 + 10) to the extent that the test piece is sunk, and boil by heating for approx. 5 min. Otherwise, heat at approx. 80°C for approx. 30 min, and dissolve the mixed zinc, iron, etc. After washing with water, wash successively with ethanol and acetone, and dry.

3.3 Weighing out of sample The weighing out of a sample shall be as follows.

- (1) When an analytical sample is weighed out, take care so that the sample represents average composition.
- (2) For weighing out of the analytical sample, use, as a rule, a chemical balance and read to the nearest 1 mg.

4. Arrangement of analytical value

4.1 Number of analyses As a rule, perform chemical analysis repetitively two times at the same analytical laboratory.

4.2 Blank test On analysis, perform a blank test, and correct the measured value.

4.3 Expression of analytical value Express an analytical value by mass percentage and round off the numerical value as follows.

- (1) Calculate the contents of lead, copper, zinc, and iron to the following second right place from the lowest place of the numerical value specified in JIS H 2113, and round off to the next right place from the lowest place of the numerical value specified in JIS H 2113 according to JIS Z 8401.

- (2) For the content of cadmium, subtract the sum of the contents of respective elements calculated by (1) from 100, and omit the numerical value following right from the lowest place specified in JIS H 2113.

5. Method for determination of lead

5.1 Classification of determination method The method for determination of lead shall be in accordance with any one of the following methods.

- (1) Atomic absorption method This method shall be applied to the sample of 0.0005 wt % or over up to and including 0.05 wt % in lead content. However, in the case where cadmium having less lead content than that of the sample [5.2.2 (2)] can not be obtained, this method can not be applied.
- (2) Inductively coupled plasma emission spectrometry This method shall be applied to the sample of 0.0002 wt % or over up to and including 0.05 wt % in lead content. However, in the case where cadmium having less lead content than that of the sample [5.3.2 (2)], this method can not be applied.
- (3) Ion exchange separation atomic absorption method This method shall be applied to the sample of 0.0001 wt % or over up to and including 0.01 wt % in lead content.
- (4) Ion exchange separation inductively coupled plasma emission spectrometry This method shall be applied to the sample of 0.0001 wt % or over up to and including 0.01 wt % in lead content.

5.2 Atomic absorption method

5.2.1 Summary After decomposing a sample with nitric acid, spray solution into the air and acetylene flame of an atomic absorption photometer, and measure its absorbance.

5.2.2 Reagents The reagents shall be as follows:

- (1) Nitric acid (1 + 1, 1 + 3),
- (2) Cadmium (99.99 wt % or over) Its lead content is known and less than the lead content in a sample (¹).
- (3) Standard lead solution A (50 µg Pb/ml) Decompose 0.100 g of lead (99.9 wt % or over) with 20 ml of nitric acid (1 + 3). After cooling to ordinary temperature, transfer into a 1000 ml volumetric flask by using water, and dilute to the mark with water to make stock solution (100 µg Pb/ml). Dilute a necessary quantity of this stock solution accurately two times with water each service to make standard lead solution A.

- (4) Standard lead solution B (10 μg Pb/ml) Dilute a necessary quantity of stock solution (100 μg Pb/ml) of (3) accurately 10 times each service to make standard lead solution B.

Note (1) The lead content is obtained by determination by the ion exchange separation atomic absorption method of 5.4 or the ion exchange separation inductively coupled plasma emission spectrometry of 5.5.

5.2.3 Weighing out quantity of sample The weighing out quantity of a sample shall be 5.0 g.

5.2.4 Operation

5.2.4.1 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 50 ml of nitric acid (1 + 3). After violent reaction ends, decompose thoroughly by heating gently. Successively gently boil, and expel nitrogen oxide. After cooling to ordinary temperature, wash the bottom surface of the watch glass with water, and remove the watch glass.
- (3) Transfer this solution into a 100 ml volumetric flask by using water, and dilute to the mark with water (2).

Note (2) In the case where the quantity of lead in this solution is 1000 μg or over, partially take 10.0 ml into a 50 ml volumetric flask from this solution. After adding 5 ml of nitric acid (1 + 1), dilute to the mark with water.

5.2.4.2 Measurement of absorbance Spray a portion of the solution obtained in 5.2.4.1 (3) into the air and acetylene flame of the atomic absorption photometer of which the zero point is adjusted by using water, and measure the absorbance at 217.0 nm or 283.3 nm in wavelength.

5.2.5 Blank test A blank test is not performed.

5.2.6 Preparation of working curve The preparation of a working curve shall be in accordance with either of the following cases.

- (1) In the case where partially taking is not performed in 5.2.4.1 (3)
 - (a) Weigh out each 5.00 g of cadmium [5.2.2 (2)] several times, and transfer each into a beaker (300 ml).
 - (b) Perform the operation of 5.2.4.1 (2), and transfer the solution into a 100 ml volumetric flask by using water.

- (c) Add stepwise various quantities (0 to 1000 µg as lead) of standard lead solution A [5.2.2 (3)] and standard lead solution B [5.2.2 (4)] to those solutions, and dilute to the mark with water.
 - (d) Spray a portion of those solutions into the air and acetylene flame of the atomic absorption photometer of which the zero point is adjusted by using water, measure the absorbance at 217.0 nm or 283.3 nm in wavelength parallel to the sample, and prepare the relation curve between the obtained absorbance and the quantity of lead to make a working curve.
- (2) In the case where partially taking is performed in 5.2.4.1 (3)
- (a) Weigh out 5.00 g of cadmium [5.2.2 (2)], and transfer into a beaker (300 ml).
 - (b) Perform the operation of 5.2.4.1 (2), transfer the solution into a 100 ml volumetric flask by using water, and dilute to the mark with water.
 - (c) Partially take each 10.0 ml of this solution into several 50 ml volumetric flasks. After adding 5 ml of nitric acid (1 + 1), add stepwise 0 to 25.0 ml of standard lead solution B [5.2.2 (4)] (0 to 250 µg as lead), and dilute to the mark with water.
 - (d) Spray a portion of those solutions into the air and acetylene flame of the atomic absorption photometer of which the zero point is adjusted by using water, measure the absorbance at 217.0 nm or 283.3 nm in wavelength parallel to the sample, and prepare the relation curve between the obtained absorbance and the quantity of lead to make a working curve.

5.2.7 Calculation The calculation shall be in accordance with either of the following cases.

- (1) In the case where partially taking is not performed in 5.2.4.1 (3) Obtain the quantity of lead from the absorbance obtained in 5.2.4.2 and the working curve prepared in 5.2.6 (1), and calculate the content of lead in a sample from the following formula.

$$\text{Lead wt \%} = \frac{A_1 + A_2}{m} \times 100$$

where,

- A_1 : detection quantity of lead in sample solution (g)
- A_2 : quantity of lead contained in 5.00 g of cadmium used for preparation of working curve (g)
- m : weighing out quantity of sample (g)

- (2) In the case where partially taking is performed in 5.2.4.1 (3)
Obtain the quantity of lead from the absorbance obtained in
 5.2.4.2 and the working curve prepared in 5.2.6 (2), and
 calculate the content of lead in a sample from the following
 formula.

$$\text{Lead wt \%} = \frac{A_3 + \left(A_4 \times \frac{1}{10} \right)}{m \times \frac{1}{10}} \times 100$$

where, A_3 : detection quantity of lead in aliquot
 sample solution (g)

A_4 : quantity of lead contained in 5.00 g of
 cadmium used for preparation of working
 curve (g)

m : weighing out quantity of sample (g)

5.3 Inductively coupled plasma emission spectrometry

5.3.1 Summary After decomposing a sample with nitric acid, spray
 the solution in the argon plasma of an inductively coupled plasma emission
 spectroscope, and measure its light emission intensity.

5.3.2 Reagents The reagents shall be as follows.

- (1) Nitric acid (1 + 1, 1 + 3).
- (2) Cadmium As specified in 5.2.2 (2).
- (3) Standard lead solution A (100 µg Pb/ml) Take stock solution
 (100 µg Pb/ml) of 5.2.2 (3) as standard lead solution A.
- (4) Standard lead solution B (10 µg Pb/ml). As specified in
 5.2.2 (4).

5.3.3 Weighing out quantity of sample The weighing out quantity of
 a sample shall be 5.0 g.

5.3.4 Operation

5.3.4.1 Preparation of sample solution Prepare sample solution in
 accordance with the following procedure.

- (1) Operate in accordance with 5.2.4.1 (1) and (2).
- (2) Transfer this solution into a 100 ml volumetric flask by using
 water, and dilute to the mark with water.

5.3.4.2 Measurement of light emission intensity Spray a portion of the solution obtained in 5.3.4.1 into the argon plasma of an inductively coupled plasma emission spectroscope, and measure light emission intensity at 220.353 nm in wavelength ⁽³⁾.

Note ⁽³⁾ When precision and accuracy are confirmed, other wavelength may be used for measurement.

Further, for an apparatus capable of using higher order spectral lines, the higher order spectral lines may be used. For an apparatus equipped with a background correcting mechanism, the background correcting mechanism may be used.

5.3.5 Blank test A blank test is not performed.

5.3.6 Preparation of working curve Prepare a working curve in accordance with the following procedure.

- (1) Weigh out each 5.00 g of cadmium [5.3.2 (2)] several times, and transfer each into a beaker (300 ml).
- (2) Carry out the operation of 5.2.4.1 (2), and transfer the solution into a 100 ml volumetric flask by using water.
- (3) Add stepwise various quantities of standard lead solution A [5.3.2 (3)] and standard lead solution B [5.3.2 (4)] (0 to 2.5 mg as lead) to those solutions, and dilute to the mark with water.
- (4) Spray a portion of those solutions into the argon plasma of an inductively coupled plasma emission spectroscope, measure the light emission intensity at 220.353 nm in wavelength parallel to the sample ⁽³⁾, and prepare the relation curve between the obtained light emission intensity and the quantity of lead to be taken as a working curve.

5.3.7 Calculation Obtain the quantity of lead from the light emission intensity obtained in 5.3.4.2 and the working curve prepared in 5.3.6, and calculate the content of lead from the following formula.

$$\text{Lead wt \%} = \frac{A_1 + A_2}{m} \times 100$$

where

A_1 : detection quantity of lead in sample solution (g)

A_2 : quantity of lead contained in 5.00 g of cadmium used for preparation of working curve (g)

m : weighing out quantity of sample (g)

5.4 Ion exchange separation atomic absorption method

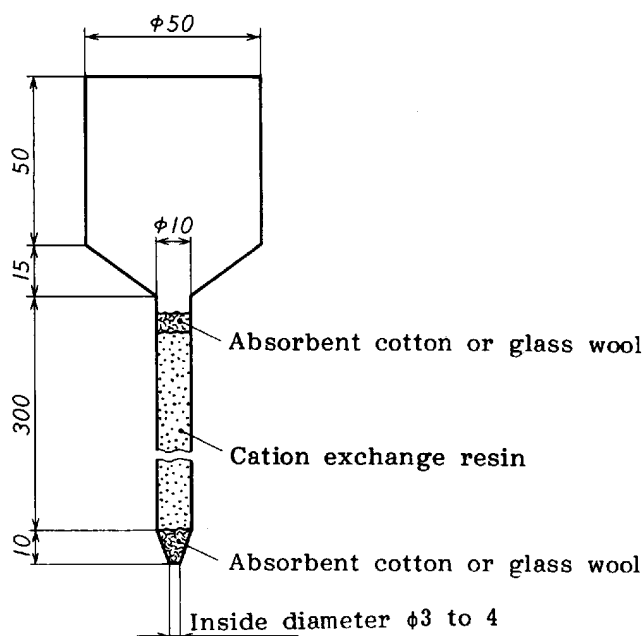
5.4.1 Summary Decompose a sample with hydrochloric acid and nitric acid. After making it a syrupy state by heating, add hydrochloric acid, dry up, and dissolve salts with a small quantity of hydrochloric acid and water. Adsorb lead by passing the solution through a cation exchange column, and make cadmium flow out. Elute lead by passing hydrochloric acid. After concentrating the eluate by heating, spray into the air and acetylene flame of an atomic absorption photometer, and measure its absorbance.

5.4.2 Apparatus For the apparatus, a cation exchange column is used, and its example is given in Figure 1. The preparation shall be as follows.

Gently pack absorbent cotton or glass wool loosened with water by a thickness of approx. 5 to 10 mm into the bottom part of the glass tube as given in Figure 1, and make approx. 18 ml of strong acid cation exchange resin swollen with water (74 to 149 μm in particle size and 1.9 milli equivalent/ml or over in exchange capacity) flow into in a slurry state. After the resin has been precipitated, gently pack absorbent cotton or glass wool loosened with water by a thickness of approx. 5 mm thereon. After regulating the flow rate of effluent to 1 to 2 ml per min by adjustment or the like of packing of absorbent cotton or glass wool, pass successively 50 ml of sodium hydroxide solution (10 g/l) and 50 ml of water, and reserve the resin as a NaR form.

Fig. 1. Example of cation exchange column

Unit: mm



5.4.3 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid (1 + 1, 1 + 3, 1 + 5, 1 + 16)
- (2) Mixed acid (hydrochloric acid 10, nitric acid 2, and water 10)
- (3) Standard lead solution A (25 $\mu\text{g Pb/ml}$) Dilute a necessary quantity of the stock solution of 5.2.2 (3) (100 $\mu\text{g Pb/ml}$) accurately 4 times with water each service to make standard lead solution A.
- (4) Standard lead solution B (10 $\mu\text{g Pb/ml}$) As specified in 5.2.2 (4).

5.4.4 Weighing out quantity of sample The weighing out quantity of a sample shall be 5.0 g.

5.4.5 Operation

5.4.5.1 Preparatory operation Pass successively 50 ml of hydrochloric acid (1 + 5), and 50 ml of water through a cation exchange column (5.4.2).

5.4.5.2 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, and add 40 ml of mixed acid [5.4.3 (2)]. After violent reaction ends, decompose thoroughly by gently heating. Make a syrupy state by successively heating. After standing to cool, wash the bottom surface of a watch glass with water, and remove the watch glass.
- (3) Add 20 ml of hydrochloric acid (1 + 1), thoroughly dry up by heating at approx. 200°C.
- (4) After standing to cool, add 1 ml of hydrochloric acid (1 + 1) and approx. 50 ml of water. After dissolving salts by heating, cool to room temperature.

5.4.5.3 Separation of lead Separate lead in accordance with the following procedure.

- (1) Pass the solution obtained in 5.4.5.2 (4) through the cation exchange column for which preparatory operation of 5.4.5.1 is ended. Then, wash the inner wall of a beaker two times with 10 ml of hydrochloric acid (1 + 16). After passing through the column each time, further pass 80 ml of hydrochloric acid (1 + 16) (⁴), make cadmium flow out, and discard the effluent.

- (2) pass successively 100 ml of hydrochloric acid (1 + 3) ⁽⁵⁾ through the column, and elute lead. Receive the eluate with a beaker (200 ml).
- (3) After concentrating the eluate to approx. 10 ml by heating, cool to ordinary temperature, transfer into a 25 ml volumetric flask ⁽⁶⁾ by using water, and dilute to the mark with water.

- Notes ⁽⁴⁾ Since the required quantity of hydrochloric acid (1 + 16) varies occasionally according to a column to be used or the like, confirm an elution curve, and vary the quantity to be used when differs.
- ⁽⁵⁾ Since the necessary quantity of hydrochloric acid (1 + 3) varies occasionally according to a column to be used or the like, confirm the elution curve, and vary the quantity to be used when differs.
- ⁽⁶⁾ In the case where the quantity of lead in this solution is 250 µg or over, transfer into a 50 ml volumetric flask by using water, and dilute to the mark with water.

5.4.5.4 Measurement of absorbance Spray a portion of the solution obtained in 5.4.5.3 (3) into the air and acetylene flame of the atomic absorption photometer of which the zero point is adjusted by using water, and measure the absorbance at 217.0 nm or 283.3 nm in wavelength.

5.4.6 Blank test Perform the same operation as that for a sample parallel to the sample without using the sample.

5.4.7 Preparation of working curve Prepare a working curve in accordance with the following procedure.

- (1) Take stepwise various quantities of standard lead solution A [5.4.3 (3)] and standard lead solution B [5.4.3 (4)] (0 to 250 µg as lead) into several 25 ml volumetric flasks ⁽⁷⁾.
- (2) Add 10 ml of hydrochloric acid (1 + 1) to those solutions, and dilute to the mark with water.
- (3) Spray a portion of those solutions into the air and acetylene flame of the atomic absorption photometer of which the zero point is adjusted by using water, measure the absorbance at 217.0 nm or 283.3 nm in wavelength parallel to the sample, prepare the relation curve between the obtained absorbance and the quantity of lead, and transfer the relation curve parallelly so as to pass the original point, and make a working curve.

Note ⁽⁷⁾ In the case where Note ⁽⁶⁾ in 5.4.5.3 is applied, deal out stepwise 0 to 20.0 ml of standard lead solution A [5.4.3 (3)] (0 to 500 µg as lead) to several 50 ml volumetric flasks.

5.4.8 Calculation Obtain the quantity of lead from the absorbance obtained in 5.4.5.4 and 5.4.6 and the working curve prepared in 5.4.7, and calculate the content of lead in a sample from the following formula.

$$\text{Lead wt \%} = \frac{A_1 - A_2}{m} \times 100$$

where, A_1 : detection quantity of lead in sample solution (g)

A_2 : detection quantity of lead in blank test solution (g)

m : weighing out quantity of sample (g)

5.5 Ion exchange separation inductively coupled plasma emission spectrometry

5.5.1 Summary Decompose a sample with hydrochloric acid and nitric acid. After making a syrupy state by heating, add hydrochloric acid, dry up, and dissolve salts in a small quantity of hydrochloric acid and water. Adsorb lead by passing the solution through a cation exchange column, and make cadmium flow out. Elute lead by passing hydrochloric acid. After concentrating the eluate by heating, spray into the argon plasma of an inductively coupled plasma emission spectroscope, and measure its light emission intensity.

5.5.2 Apparatus For the apparatus, the cation exchange column of 5.4.2 shall be used.

5.5.3 Reagents The reagents shall be as follows.

- (1) Hydrochloric acid (1 + 1, 1 + 3, 1 + 5, 1 + 16)
- (2) Mixed acid (hydrochloric acid 10, nitric acid 2, and water 10)
- (3) Standard lead solution A (50 µg Pb/ml) As specified in 5.2.2 (3)
- (4) Standard lead solution B (10 µg Pb/ml) As specified in 5.2.2 (4)

5.5.4 Weighing out quantity of sample The weighing out quantity of a sample shall be 5.0 g.

5.5.5 Operation

5.5.5.1 Preparatory operation The preparatory operation shall be as specified in 5.4.5.1.

5.5.5.2 Preparation of sample solution Sample solution shall be prepared as specified in 5.4.5.2.