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JAPANESE INDUSTRIAL STANDARD

Methods for Chemical
Analysis of White Metal

JIS H 1501—1975

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Japanese Standards Association



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JAPANESE INDUSTRIAL STANDARD

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Methods for Chemical Analysis of
White MetalH 1501-1975
(Reaffirmed: 1984)1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of chemical components (tin, antimony, copper, lead, zinc, iron, aluminium, arsenic and bismuth) specified in JIS H 5401.

2. General Matters

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

3. Sampling and Handling of Sample

3.1 Sampling The sampling and the preparation method of sample shall be in accordance with JIS H 5401.

3.2 Handling of Sample The handling of sample shall be as follows:

- (1) The sample shall be put in a glass vessel with a suitable cover or the like, sealed and stored in order to prevent the contamination due to foreign matters or the like.
- (2) When it is feared that oil or the like are adhered to the surface of sample, the surface shall be preliminarily cleaned with pure alcohol or ether and removed of the oil or the like.

Applicable Standards:

JIS H 5401-White Metal

JIS K 0050-General Rules for Chemical Analysis

JIS K 0115-General Rules for Molecular Absorptiometric Analysis

JIS K 8005-Standard Substances for Volumetric Analysis

JIS Z 8401-Rules for Rounding off of Numerical Values



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3.3 Weighing of Sample The weighing of sample shall be carried out as follows:

- (1) When a sample is weighed out, the sample shall be carefully and sufficiently mixed by stirring to represent its average composition.
- (2) For weighing out of sample, a chemical balance shall, as a rule, be used and the amount near a specified amount shall be weighed out to necessary places of figures referring to the designated number of figures of analysis value.

4. Expression of Analysis Value and Cautions on Operation

4.1 Expression of Analysis Value The analysis value shall be expressed by percentage, calculated to the next figure of the significant lowest place of the numerical value specified in JIS H 5401 and rounded off in accordance with JIS Z 8401.

4.2 Cautions on Analysis Operation The cautions on analysis operation shall be as follows:

- (1) The same sample shall be analyzed not less than two times and the results shall be confirmed.
- (2) A blank test shall be carried out over all the operation in analysis and the measured value shall be corrected.

5. Method for Determination of Tin

5.1 Classification of Method The method for determination of tin shall be in accordance with iodine titrimetric method.

5.2 Iodine Titrimetric Method

5.2.1 Summary A sample is decomposed with sulfuric acid, joined by water and hydrochloric acid and boiled. Thereafter, it is joined by hydrochloric acid, reducing lead, antimony chloride, etc. and stoppered by a capped rubber stopper. After reducing it by heating, it is quickly titrated with sodium iodine standard solution utilizing starch as indicator.

5.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Sulfuric acid
- (3) Sodium hydrogencarbonate solution (saturated, approx. 9 w/v %)
- (4) Antimony trichloride solution 5 g antimony trichloride is dissolved with 500 ml hydrochloric acid (1 + 1) by heating.
- (5) N/10 iodine standard solution 30 g potassium iodide and 12.8 g iodine are weighed out, transferred into a beaker (300 ml), joined by 50 ml cold water and iodine is completely dissolved by mixing them with shaking. Thereafter, it is diluted to 150 ml and filtered by using a sintered glass filter (17 G 4). Thereafter, it is diluted to 1 l with water and put in a brown bottle to be stored. Though 1 ml this solution corresponds to approx. 0.006 g tin, a fixed amount of tin metal Class 1 is used and standardized at each service as follows:

Method for standardization Weigh out 1.000 g tin metal Class 1, transfer it into a tall beaker (300 ml), add 20 ml sulfuric acid, cover it with a watch glass, decompose it by ignition and successively ignite to vaporize the sulfur adhered to the watch glass and the inside wall of beaker. After cooling, wash the watch glass and the inside wall of beaker by using water and hydrochloric acid (1 + 1), dissolve soluble salts by adding 50 ml hydrochloric acid (1 + 1), transfer it into a measuring flask of 250 ml and dilute it up to the marked line by using hydrochloric acid (1 + 1). Otherwise decompose 1.000 g tin with 50 ml hydrochloric acid on a water bath by utilizing a platinum plate as a catalyst, transfer it into a measuring flask of 250 ml and dilute it up to the marked line by using hydrochloric acid (1 + 1) to prepare the standard tin solution.

Accurately take 25 ml this solution, transfer it into an Erlenmeyer flask (500 ml) and dilute it to 200 ml with water. Thereafter, operate it in accordance with the procedures of 5.2.5 (3) to (5) and calculate the amount of tin equivalent to 1 ml from the following formula:

$$f = \frac{0.1}{V_1 - V_2}$$

where f : amount of tin equivalent to 1 ml N/10 iodine standard solution (g)

V_1 : used amount of N/10 iodine standard solution (ml)

V_2 : blank test value (ml)

- (6) N/30 iodine standard solution 15 g potassium iodide and 4.3 g iodine are weighed out, transferred into a beaker (300 ml), joined by 50 ml cold water and mixed by shaking. After completely dissolving iodine, it is diluted to 150 ml and filtered by using a sintered glass filter (17 G 4). Thereafter, it is diluted to 1 l with water and put in a brown bottle to be stored. Though 1 ml this solution corresponds to approx. 0.002 g tin, a fixed amount of tin metal Class 1 is used and standardized at each service as follows:

Method for standardization Accurately take 10 ml standard tin solution [prepared in accordance with (5)], titrate it in the same way as that in (5) and calculate the amount of tin equivalent to 1 ml from the following formula:

$$f = \frac{0.04}{V_1 - V_2}$$

where f : amount of tin equivalent to 1 ml N/30 iodine standard solution (g)

V_1 : used amount of N/30 iodine standard solution (ml)

V_2 : blank test value (ml)

- (7) **Starch solution** 0.1 g starch (soluble) is made muddy with a small amount of cold water and poured into hot water of 100 ml while mixing them by stirring. It is used after standing to cool. This solution is prepared at each service and the solution which turned red brown due to iodine shall not be used.

5.2.3 **Apparatus** Those given in Attached Figs. 1, 2 and 3 shall, as a rule, be used for the apparatus.

- (1) Though assay lead is conveniently used as the reducing lead, a nickel cylinder as given in Attached Fig. 1 may be used instead of the reducing lead and antimony trichloride. The pure nickel plate of approx. 1.5 mm in thickness is used for the nickel cylinder.
- (2) For the capped rubber stopper, the one manufactured by using the hard glass of 1 to 1.5 mm in thickness as given in Attached Fig. 2 is used.

Further, the apparatus which reduces a sample solution by passing carbon dioxide as given in Attached Fig. 3 may be used.

5.2.4 **Weighing out Amount of Sample** A sample shall be weighed out in accordance with Table 1 according to the content of tin.

Table 1

Content of tin %	Weighing out amount of sample g	Concentration of iodine standard solution
Under 9	1	N/30
9 and over, up to 20	1	N/10
20 and over, up to 35	0.5	N/10
35 and over, up to 55	0.3	N/10
55 and over, up to 65	0.2	N/10

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

- (1) Weigh out a sample, transfer it into an Erlenmeyer flask (500 ml), add 15 ml sulfuric acid and decompose it by heating.
- (2) After standing to cool, add 50 ml water and 10 ml hydrochloric acid, vaporize arsenic by boiling and dilute the solution to 200 ml with water.

- (3) Add 40 ml hydrochloric acid, 10 g reducing lead and 10 ml antimony trichloride solution, stopper it by a capped rubber stopper, put 50 ml water into the cap and reduce it by boiling it quietly for about 10 min.
- (4) Let it stand to cool for a while and reflux the water in the cap into the Erlenmeyer flask. Thereafter, add 50 ml sodium hydrogen-carbonate and completely reduce tin by heating it again for 30 min.
- (5) Cool this solution to 10°C or less, remove the rubber stopper and wash the inside wall of Erlenmeyer flask and the bottom end of rubber stopper with the cleaning water ⁽¹⁾ preliminarily prepared. After adding 10 ml starch solution as indicator, titrate it at approx. 10°C by using iodine standard solution (refer to Table 1).

5.2.6 Calculation The content of tin in sample shall be calculated from the following formula:

$$\text{Tin (\%)} = \frac{f \times V}{W} \times 100$$

where f : amount of tin equivalent to 1 ml iodine standard solution (g)

V : used amount of iodine standard solution (ml)

W : weighed out amount of sample (g)

Note ⁽¹⁾ Expel air from water by boiling and cool the water while passing carbon dioxide to be used for the cleaning water. When oxygen is mixed in carbon dioxide, dissolve 45 g cuprous chloride with 300 ml hydrochloric acid (1 + 1) and add a small amount of metalline copper turning chips thereto. Thereafter, tightly stopper the solution, let it stand for 24 h or more and remove oxygen therefrom. Then, use the solution.

Remark: The operation in the case of using the apparatus in which a sample is reduced by passing carbon dioxide as given in Attached Fig. 3, shall be as follows:

After operating the sample in accordance with the procedures of 5.2.5 (1) to (2), add 40 ml hydrochloric acid, 10 g reducing lead and 10 ml antimony chloride solution, assemble the apparatus, heat them for about 30 min while passing carbon oxide and completely reduce tin. After standing to cool, cool it to 10°C or less in running water, extract a glass bar (e), insert the mouth of cleaning bottle, wash the inside wall of Erlenmeyer flask and the bottom end of rubber stopper with cleaning water ⁽¹⁾ and add 10 ml starch solution as indicator. Thereafter, insert the top of the burette containing iodine standard solution into the hole of (e) and titrate the solution at approx. 10°C. Hereafter, calculate the content of tin in accordance with 5.2.6.

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6. Method for Determination of Antimony

6.1 Classification of Method The method for determination of antimony shall be in accordance with potassium permanganate titrimetric method.

6.2 Potassium Permanganate Titrimetric Method

6.2.1 Summary A sample is decomposed with sulfuric acid and potassium sulfate by heating, joined by water and hydrochloric acid, boiled and cooled by further adding water. Thereafter, it is titrated by using potassium permanganate standard solution.

6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Sulfuric acid
- (3) Potassium sulfate
- (4) Sodium sulfite
- (5) N/20 potassium permanganate standard solution 1.58 g potassium permanganate is dissolved with 1 l water, put into a flask and quietly boiled. Thereafter, it is let to stand in a dark place for one night. The supernatant is filtered with a sintered glass filter (17 G 4) (it shall not be washed with water before and after filtration), put into a brown bottle and stored in a dark place. Though 1 ml of this solution corresponds to approx. 0.003 g antimony, it shall be standardized as follows:

Method for standardization Weigh out 0.600 g antimony metal extra class, transfer it into an Erlenmeyer flask (500 ml) and operate it in accordance with the procedure of 6.2.4 (1). After standing to cool, dissolve soluble salts by carefully adding 50 ml water and 50 ml hydrochloric acid and cool it. Thereafter, transfer it into a measuring flask of 250 ml and dilute it up to the marked line by using hydrochloric acid (1 + 1).

Separately take 25 ml of this solution, add 100 ml water and 20 ml sulfuric acid, quietly boil it for about 5 min, dilute it to approx. 250 ml by adding water, cool it to approx. 10°C, operate it in accordance with 6.2.4 (3) and calculate the amount of antimony equivalent to 1 ml from the following formula:

$$f = \frac{0.06}{V_1 - V_2}$$

where f : antimony equivalent to 1 ml N/20 potassium permanganate standard solution (g)

V_1 : used amount of N/20 potassium permanganate standard solution (ml)

V_2 : blank test value (ml)

- (6) N/20 potassium bromate standard solution 1.3918 g potassium bromate which is recrystallized two times with water and dried at 180°C to constant weight, is dissolved with water. Thereafter, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water. 1 ml of this solution corresponds to 0.003044 g antimony.

- (7) Methyl Orange solution (0.1 w/v %)

6.2.3 Weighing out Amount of Sample A sample shall be weighed out in accordance with Table 2 according to the content of antimony.

Table 2

Content of antimony %	Weighing out amount of sample g
Under 10	1
10 min.	0.5

Remark: As for lead group alloys (Classes 7 to 10), 0.3 g shall be weighed out.

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

- (1) Weigh out a sample, transfer it into an Erlenmeyer flask (500 ml), add 20 ml sulfuric acid and 4 g potassium sulfate, at first gradually heat them and decompose them by gradually raising temperature. Thereafter, remove sulfur dioxide ⁽²⁾ by successively heating ⁽¹⁾ it for 10 to 30 min.
- (2) After standing to cool, carefully add 50 ml water, wash the inside wall of Erlenmeyer flask with water, add 15 ml hydrochloric acid ⁽³⁾ thereto, quietly boil it ⁽⁴⁾ for 5 min (for 10 min when the content of iron or arsenic is much) and dilute it to approx. 250 ml by adding water ⁽⁵⁾. Thereafter, cool it to approx. 10°C.
- (3) Gradually titrate it with N/20 potassium permanganate standard solution ⁽⁶⁾ with keeping it at approx. 10°C.

6.2.5 Calculation The content of antimony in sample shall be calculated from the following formula:

$$\text{Antimony (\%)} = \frac{f \times V}{W} \times 100$$

where f : amount of antimony equivalent to 1 ml N/20 potassium permanganate standard solution (g)

V : used amount of N/20 potassium permanganate standard solution (ml)

W : weighed out amount of sample (g)

- Notes (1) When heated at a too high temperature in that case, a low value is liable to be obtained and therefore, cares shall be necessarily taken for that.
- (2) Sulfur dioxide remains, potassium permanganate solution being consumed and a high value is sometimes obtained. Therefore, it is necessary to avoid that by successive heating after decomposition.
- (3) Though the amount of hydrochloric acid should preferably be 15 ml for tin group alloy, use 25 ml for lead group alloy, lastly dilute total volume to approx. 400 ml with water and titrate it.
- (4) When heating is carried out for a long time under a state acidified with hydrochloric acid, antimony (III) is oxidized by air and a low value is obtained (when copper coexists, this phenomenon is especially remarkable).
- (5) When plenty of the precipitate of lead sulfate exists in that case, the amount of antimony is liable to become high. Therefore, it is necessary to dissolve the precipitate as far as possible by mixing them with shaking immediately after boiling for 1 min.
- (6) The titration shall be gradually carried out. When rapidly carried out, the titre is consumed by 0.2 to 0.3 ml in excess due to its reaction with hydrochloric acid.

Remark: As for Class 10 which has arsenic as alloy constituent, the operation shall be carried out as follows: After decomposing a sample in accordance with 6.2.4 (1), let it stand to cool. Thereafter, add 10 ml water, 75 ml hydrochloric acid and 1 g sodium sulfite and evaporate it by quietly boiling it until the volume becomes 60 ± 5 ml. Dilute it to 300 ml by adding hot water thereto and violently pass air for 1 min. Thereafter, add Methyl Orange solution to this hot solution as indicator, titrate it by using N/20 potassium bromate standard solution and calculate the content of antimony from the following formula:

$$\text{Antimony (\%)} = \frac{f \times V}{W} \times 100$$

where f : amount of antimony equivalent to 1 ml N/20 potassium bromate standard solution (g)

V : used amount of N/20 potassium bromate standard solution (ml)

w : weighed out amount of sample (g)

7. Method for Determination of Copper

7.1 Classification of Method The method for determination of copper shall be as specified in either of the following methods:

- (1) Sodium thiosulfate titrimetric method
- (2) Electrogravimetric method This method applies to the sample of under 3 % in lead content.

7.2 Potassium Thiosulfate Titrimetric Method

7.2.1 Summary A sample is decomposed with hydrochloric acid and hydrogen peroxide water by heating, joined by tartaric acid, alkalized with sodium hydroxide, joined by grape sugar and copper is precipitated by boiling. The precipitate is dissolved with nitric acid-bromine mixed solution. After expelling bromine, it is made ammoniacal. The iodine liberated by adding potassium iodide to this solution is titrated with sodium thiosulfate standard solution.

7.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Sulfuric acid
- (3) Nitric acid-bromine mixed solution Approx. 25 ml sulfuric acid (1 + 1) is joined by 5 ml bromine solution (saturated).
- (4) Aqueous ammonia (1 + 1, 1 + 50)
- (5) Sodium hydroxide solution (40 w/v %)
- (6) Hydrogen peroxide water (30 %)
- (7) Potassium iodide
- (8) Acetic acid
- (9) Tartaric acid
- (10) Grape sugar
- (11) N/20 sodium thiosulfate standard solution Approx. 13 g sodium thiosulfate (5 hydrate) and approx. 0.2 g sodium carbonate (anhydrous) are weighed out, dissolved with the water containing no carbonic acid and the amount of solution is diluted to approx. 1 l. It is put in a brown bottle, tightly stoppered and let stand for at least 5 days. Thereafter, it is used. Though 1 ml of this solution corresponds to approx. 0.003 g copper, it shall be standardized as follows:

Method for standardization Weigh out 0.100 g pure copper, transfer it into an Erlenmeyer flask (300 ml), add 2 ml water, 3 ml nitric acid and 1 ml sulfuric acid, decompose them by heating on a water bath and evaporate it until white fume of sulfuric acid is generated (to a extent that it is solidified after cooling).

Add 20 ml water thereto, decompose it by heating, add aqueous ammonia (1 + 1) and rather excessively add it until the solution turns blue. Thereafter, boil it until ammonia odor disappears, add 2 ml acetic acid and cool it. Add approx. 3 g potassium iodide, titrate the liberated iodine with N/20 sodium thiosulfate standard solution, add approx. 5 ml starch solution (0.1 w/v %) as indicator when the solution turns faint yellow, continue to titrate it and allow the point at which blue has disappeared to be the end point. Calculate the amount of copper equivalent to 1 ml from the following formula:

$$f = \frac{0.100}{V_1 - V_2}$$

where f : amount of copper equivalent to 1 ml N/20 sodium thiosulfate standard solution (g)

V_1 : used amount of N/20 sodium thiosulfate standard solution (ml)

V_2 : blank test value (ml)

(12) Phenolphthalein solution (0.1 w/v % alcohol solution)

(13) Starch solution (0.1 w/v %) 0.1 g starch (soluble) is made muddy with a small amount of cold water and poured into the hot water of 100 ml while mixing them by stirring. Thereafter, it is boiled for a while, let stand to cool and used. This solution shall be prepared at each service.

7.2.3 Weighing out Amount of Sample 1 g of the sample shall be weighed out.

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

- (1) Weigh out a sample, transfer it into a tall beaker (300 ml), cover it with a watch glass, add 50 ml hydrochloric acid and quietly heat it. After the reaction becomes slow, add little by little hydrogen peroxide water, heat them and decompose the sample.
- (2) After decomposition, boil it for about 5 min, wash the watch glass and the inside wall of beaker with water, add approx. 10 g tartaric acid and dissolve it. Thereafter, let it stand to cool.
- (3) Neutralize the solution with sodium hydroxide solution utilizing phenolphthalein solution as indicator. After further adding 15 ml in excess, add approx. 3 g, grape sugar, boil them for about 10 min and completely precipitate copper. After standing still for about 30 min, filter them by using a fine filter paper and sufficiently wash the precipitate with warm water.
- (4) Dissolve the precipitate on the filter paper and the precipitate in the beaker by using the nitric acid-bromine mixed warm solution, receive it with a tall beaker (300 ml) and sufficiently wash it with warm water.