

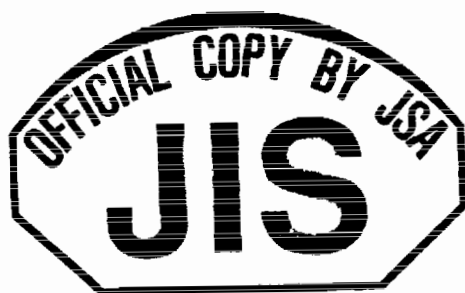
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This standard was revised in 6, 1995

JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of Lead Metal

JIS H 1121-1974
(Reaffirmed: 1983)



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Methods for Chemical Analysis of Lead Metal H 1121-1974
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1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of the chemical components (silver, copper, bismuth, antimony, arsenic, tin, iron and zinc) specified in JIS H 2105.

2. General Matters

General matters common to the methods for chemical analysis shall be as specified in JIS K 0050, JIS K 0115 and JIS K 0121.

3. Sampling and Handling of Sample

3.1 Sampling of Sample

- (1) The sampling method shall be, as a rule, as specified in JIS H 0301. However, the sample chipped with a drill shall be cut to about 5 mm max. by using clean scissors.
- (2) When it is impossible to employ the sampling method in accordance with the above specification, it shall be subjected to the agreement between the parties concerned.

3.2 Handling of Sample

- (1) The analysis sample shall be put into a glass vessel with a suitable cover and tightly sealed and stored in order to prevent contamination due to foreign matters and the like.
- (2) When there is a fear that the analysis sample is soiled by oils or the like on the surface thereof, the sample shall be preliminarily cleaned with pure alcohol and ether, and oils or the like shall be removed.

Applicable Standards:

JIS H 0301-General Rules for Tests and Inspection of Base Metals

JIS H 2105-Pig Lead

JIS K 0050-General Rules for Chemical Analysis

JIS K 0115-General Rules for Absorptiometric Analysis

JIS K 0121-General Rules for Atomic Absorption Spectrochemical Analysis

JIS Z 8401-Rules for Rounding off of Numerical Values

JIS Z 8801-Test Sieves

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- (3) When a sample is mechanically contaminated with iron content from the used apparatus or the like, or otherwise there is such a fear in preparation of the sample, the sample used for determination of iron content shall be preliminarily treated as follows.

Take a necessary amount of the analysis sample into a beaker, add hydrochloric acid (1+1) to an extent that the test piece is sunk, boil it by heating for 5 minutes, or otherwise heat it at about 80°C for 30 minutes and dissolve the iron content adhering to the surface or mixed. Thereafter, wash it with water, further clean it with pure alcohol and ether and dry it.

3.3 Weighing of Sample

- (1) When an analysis sample is weighed out, ensure that the sample is sufficiently mixed represent the average composition.
- (2) For weighing out an analysis sample, a chemical balance shall, as a rule, be used.

4. Expression of Analysis Value and Cautions on Operation

4.1 Expression of Analysis Value The analysis value shall be expressed by percentage, which shall be calculated to the next place of the significant lowest place of the numerical value specified in JIS H 2105 and rounded off in accordance with JIS Z 8401.

4.2 Cautions on Analytical Operation

- (1) The same sample shall be analyzed not less than two times to confirm the results.
- (2) When analyzed, a blank test shall be carried out through the whole operation and the measured value shall be corrected.
- (3) When absorbance is measured in atomic absorptiometry, the air-acetylene flame shall be used, unless otherwise specified. However, if precision and accuracy are verified, the air-hydrogen and other flames may be used.

5. Method for Determination of Silver

5.1 Classification of Methods The method for determination of silver shall be carried out according to one of the following methods:

- (1) Dry assay method This method shall be applied to the sample of 0.00005 % min. in silver content.
- (2) Dithizone copper absorptiometry This method shall be applied to the sample of 0.0001 % min. in silver content.

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- (3) Atomic absorption method This method shall be applied to the sample of 0.0002 % min. in silver content.

5.2 Dry-Assay Method

5.2.1 Summary A sample is transferred onto a scorifier, mixed with a small amount of the equally-composed mixture of assaying silicon dioxide and borax glass, put into the preheated muffle furnace and fused. Thereafter, greater part of the sample is slagged by blowing a small amount of air into it. It is cast into an iron mould and about 30 g of lead button is prepared. It is cupelled and the mass of the obtained silver particle is weighed by using an assay balance.

5.2.2 Reagents The reagents shall be as follows:

- (1) Silicon Dioxide for Assay Sodium dioxide (95 % min.) is ground and passed through a sieve of 149 μm (equivalent to 100 mesh) of JIS Z 8801 to be used. However, the silver content thereof shall be necessarily confirmed before using.
- (2) Borax Glass For the borax glass, after roasting sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) on a clean iron plate, it is fused in a graphite crucible or an iron crucible and let to flow on the clean iron plate. After cooling, it is ground and passed through a sieve of 420 μm (equivalent to 35 mesh) of JIS Z 8801 to be used.

5.2.3 Weighing-out Amount of Sample 100 g of the sample shall be weighed out to the nearest 0.1 g.

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

- (1) Weigh out a sample onto a scorifier⁽¹⁾, spread about 1 g of the equivalent mixture of assaying silicon dioxide and borax glass, put it into the muffle furnace preheated at about 1000°C, close the door and sufficiently fuse it by heating.
- (2) After about 5 minutes, slightly open the door, lower the temperature to about 900°C by making air flow in, prevent the excess corrosion of scorifier and at the same time, slag the great portion of the sample.
- (3) Close the door several minutes after the disappearance of the iron shot, raise the temperature to about 950°C and take out the scorifier 5 minutes after. Gently rotate the contents thereof several times, make lead particles gather and quietly pour it into the central part of the iron mould⁽²⁾.

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- (4) After cooling, take out lead button⁽³⁾, hammer it by using a hammer and tweezers and make it form a cube removing the slag as much as possible.
- (5) Quickly put the lead button into the cupel⁽⁴⁾ preheated at about 850° C for 5 minutes in the muffle furnace, close the door, raise the temperature thereof to about 820°C by heating, treat about 30 g of lead button and regulate draft so that cupellation is completed in about 45 min.⁽⁵⁾ Cover the sample, the cupellation of which is completed with the preheated cupel, quietly take it out and let it stand to cool.
- (6) After cooling, take out silver particle, completely remove the adhering bone ash by using a brush, make it of thin sheet by lightly striking it with the hammer and weigh it by using an assay balance.

5.2.5 Calculation The content of silver in sample shall be calculated from the following formula⁽⁶⁾:

$$\text{Silver (\%)} = \frac{w_1 + w_2}{W} \times 100$$

where w_1 : mass of silver particle (g)

w_2 : correction amount (g)

W : weighed out amount of sample (g)

- Notes ⁽¹⁾ the clayish scorifier of about 25 mm in height, 60 mm in inside diameter and 30 ml in capacity shall, as a rule, be used.
- ⁽²⁾ The iron mould having the cone shaped round bottom hole of 40 mm in diameter and 25 mm in depth shall be used. However, before use, it shall be preliminarily cleaned, heated at about 100°C, coated with a small amount of machine oil or iron oxide red and sufficiently dried.
- ⁽³⁾ The amount of lead button shall be 30 g. When its amount is too much, it shall be reduced to about 30 g by repeating the procedures of 5.2.4 (1) to (3).
- ⁽⁴⁾ The mass of cupel shall be about 50 g in general. The cupel naturally dried for about 3 months at least after manufacture shall be used. The pure bone ash containing no carbonate, which is ground and passed through a sieve of 297 µm (equivalent to 48 mesh) of JIS Z 8801, shall be used. The cupel is manufactured by adding about 8 % water thereto. However, 20 % max. cement can be mixed therewith.
- ⁽⁵⁾ The end point of cupellation shall be the time when special brilliancy disappears.

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Note (⁶) Because a substantial amount of silver is lost when this method is applied, silver shall be recovered by assaying the slag and the cupel after cupellation and the correction shall be carried out.

5.3 Dithizone Copper Absorptiometry

5.3.1 Summary After decomposing a sample with nitric acid, a fixed amount of solution is separately taken and diluted with water. Thereafter, it is joined by dithizone copper carbon tetrachloride and mixed by shaking. When the copper of dithizone copper complex is substituted with silver, the absorbance of the remaining dithizone copper complex is measured.

5.3.2 Reagents The reagents shall be as follows:

- (1) Nitric acid (1+4)
- (2) Dithizone copper carbon tetrachloride solution 0.500 g of copper (99.9 % min.) is decomposed with 10 ml of nitric acid (1+1) and 10 ml of sulfuric acid (1+3) and evaporated by heating to dryness. After standing to cool, it is dissolved with water, transferred into a measuring flask of 500 ml and diluted up to the marked line with water. 1 ml of this solution (equivalent to 1 mg as copper) is taken into a separatory funnel (100 ml) and given 0.5 ml of sulfuric acid (1+3) and 20 ml of water. Then, it is joined by 50 ml of dithizone carbon tetrachloride solution (dithizone 5 mg/100 ml)(¹), mixed by violently shaking and separated into two layers by standing still. Thereafter, the carbon tetrachloride layer is transferred into another separatory funnel (100 ml), given about 20 ml of water, mixed by shaking and washed. It is separated into two layers by standing still and the carbon tetrachloride layer is transferred into a clean vessel. It is diluted by adding about 2 times amount of carbon tetrachloride(²). This solution is prepared at each service.
- (3) Carbon tetrachloride If necessary, it is mixed with calcium oxide, redistilled and refined.
- (4) Standard silver solution (1 µg Ag/ml) 0.100 g of silver (99.9 % min.) is decomposed with 20 ml of nitric acid (1+1) and cooled. Thereafter, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water. This solution is put into a brown bottle to be stored. A necessary amount thereof is accurately diluted to 100 times with water at each service to prepare the standard silver solution.

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Notes (1) 50 mg of dithizone is dissolved with 100 ml of carbon tetrachloride, washed by extraction with aqueous ammonia (1+10) and hydrochloric acid (1+10) and refined by repetition of this operation to prepare the stock solution. It is diluted to 10 times with carbon tetrachloride at each service.

(2) The absorbance at near 540 nm in wavelength of this solution shall be 0.6 to 0.7.

5.3.3 Weighing-out Amount of Sample 10 g of the sample shall be weighed out to the nearest 10 mg.

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

- (1) Weigh out a sample into a beaker (300 ml), cover it with a watch glass, add 70 ml of nitric acid (1+4) and decompose it by quietly heating. Thereafter, evaporate it by heating and concentrate it near to dryness⁽³⁾.
- (2) After standing to cool, dissolve it by adding about 30 ml of water, transfer it into a measuring flask of 100 ml and dilute it up to the marked line with water. Separately take accurately 20 ml⁽⁴⁾ from this solution into a separatory funnel (100 ml).
- (3) Dilute it to 50 ml by adding water⁽⁵⁾, accurately add 10 ml of dithizone copper carbon tetrachloride solution thereto, mix them by violently shaking for about two minutes and separate it into two layers by standing still.
- (4) Remove the water content of the carbon tetrachloride layer⁽⁶⁾, take a portion thereof into the absorption cell of a photometer, measure the absorbance at near 540 nm in wavelength and obtain the absorbance of sample solution by subtracting it from the absorbance of the dithizone copper carbon tetrachloride solution at the same wavelength.

5.3.5 Calculation The amount of silver shall be obtained from the working curve prepared in 5.3.6 and the content of silver in sample shall be calculated from the following formula:

$$\text{Silver (\%)} = \frac{A}{W \times B} \times 100$$

where A: detected amount of silver in aliquot sample solution (g)
 W: weighed-out amount of sample (g)
 B: aliquot ratio of sample solution

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5.3.6 Preparation of Working Curve Stepwise take various amounts of standard silver solution (0 to 15 μg as silver) into several separatory funnels (100 ml), add 3 ml of nitric acid (1+4), hereinafter operate it in the same way as that of the sample in accordance with the procedures on and after 5.3.4 (3), prepare the relation curve between the obtained absorbance and the amount of silver and allow it to be the working curve.

- Notes (³) When lead nitrate starts to be deposited in concentration by heating, the sample solution is transferred to a place at a low temperature (about 150°C), continuously concentrated, taken down from the heat source and let to stand to cool. Just at that time, it should preferably become of a state concentrated to dryness.
- (⁴) When the content of silver is large, the aliquot amount shall be lessened to be 5 to 15 μg in silver.
- (⁵) pH of the aqueous solution at that time becomes 0.8 to 1.0.
- (⁶) The carbon tetrachloride layer herein shall be dehydrated by using absorbent cotton or a filter paper.

5.4 Atomic Absorption Method

5.4.1 Summary After decomposing a sample with nitric acid, diluted to a fixed amount with water and the absorbance is measured by using an atomic absorption photometer.

5.4.2 Reagents The reagents shall be as follows:

- (1) Nitric acid (1+4)
- (2) Lead (without gold, silver and lead) Lead of 0.00002 % max. in silver is used.
- (3) Standard silver solution (20 μg Ag/ml) 0.100 g of silver (99.9 % min.) is decomposed with 20 ml of nitric acid (1+1). After cooling, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water. This solution is put into a brown bottle to be stored. Only a necessary amount thereof is accurately diluted to 5 times with water to prepare the standard silver solution.

5.4.3 Weighing out Amount of Sample 10 g of the sample shall be weighed out to the nearest 10 mg.

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

- (1) Weigh out a sample into a beaker (300 ml), cover it with a watch glass, add 70 ml of nitric acid (1+4) and decompose by quietly heating.

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- (2) After cooling, transfer it into a measuring flask of 100 ml and dilute it up to the marked line with water⁽¹⁾(²).
- (3) Measure the absorbance at 328.1 nm in wavelength by using an atomic absorption photometer.

5.4.5 Calculation The amount of silver shall be obtained from the working curve prepared in 5.4.6 and the content of silver in sample shall be calculated from the following formula:

$$\text{Silver (\%)} = \frac{A}{W \times B} \times 100$$

where A : detected amount of silver in aliquot sample solution (g)

W : weighed-out amount of sample (g)

B : aliquot ratio of sample solution.

5.4.6 Preparation of Working Curve Weigh out 10.00 g of lead into several beakers (300 ml), cover them with watch glasses and decompose each of them by adding 70 ml of nitric acid (1+4) thereto. After cooling, transfer it into a measuring flask of 100 ml, stepwise add various amounts of standard silver solution (0 to 300 µg as silver) thereto and dilute it up to the marked line with water⁽³⁾. Hereinafter, operate it in the same way as that of the sample in accordance with the procedure of 5.4.4 (3), prepare the relation curve between the absorbance measured in parallel with the sample and the amount of silver and allow it to be the working curve.

- Notes (1) When the content of silver is large, the sample solution is separately taken into a measuring flask of 100 ml so as to be 50 to 300 µg in silver and diluted up to the marked line with water.
- (2) In the case of generating the turbidity of metastannic acid or the like, the sample solution is filtered with a dry filter paper (class 5 C).
- (3) When the aliquot operation of Note (1) is carried out, the same amount as the aliquot amount of sample solution is separately taken into several measuring flasks of 100 ml, stepwise joined by various amounts of standard silver solution (0 to 300 µg as silver) and diluted up to the marked line with water.

6. Method for Determination of Copper

6.1 Division of Methods The method for determination of copper shall be in accordance with either one of the following methods.

- (1) Neocuproine absorptiometry This method shall be applied to the sample of 0.0001 % min. in copper content.

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- (2) Atomic absorption method This method shall be applied to the sample of 0.0001 % min. in copper content.

6.2 Summary After decomposing a sample with nitric acid, it is mixed with ammonium acetate and hydroxylamine hydrochloride. Thereafter, pH is regulated with aqueous ammonia and neocuproine is added thereto. The generated neocuproine copper complex is extracted with chloroform and the absorbance is measured.

6.2.2 Reagents The reagents shall be as follows:

- (1) Nitric acid (1+4)
- (2) Sulfuric acid (1+3)
- (3) Aqueous ammonia (1+1)
- (4) Hydroxylamine hydroxide solution (10 % w/v)
- (5) Acetic acid solution 10 g of acetic acid (monohydrate) is dissolved with water to 100 ml.
- (6) Ammonium acetate solution (30 % w/v)
- (7) Neocuproine solution 0.1 g of neocuproine is dissolved with 100 ml of ethyl alcohol (95 % v/v).
- (8) Chloroform
- (9) Standard copper solution (2.5 µg Cu/ml) 0.100 g of copper (99.9 % min.) is decomposed with 20 ml of nitric acid (1+1). After cooling, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water. Only a necessary amount is accurately diluted to 40 times with water at each service to prepare the standard copper solution.

6.2.3 Weighing-out Amount of Sample 10 g of the sample shall be weighed out to the nearest 10 mg.

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

- (1) Weigh out a sample into a beaker (300 ml), cover it with a watch glass, add 70 ml of nitric acid (1+4) and decompose it by quietly heating.
- (2) After cooling, transfer it into a measuring flask of 100 ml, dilute it up to the marked line with water and separately take accurately 20 ml⁽¹⁾ from this solution into a beaker (100 ml).
- (3) After adding 10 ml of ammonium acetate solution and 5 ml of hydroxylamine hydrochloride solution thereto, dilute the amount of solution to about 50 ml with water and regulate pH at 5 to 6 with aqueous ammonia (1+1).

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- (4) Transfer it into a separatory funnel (100 ml) and add 2 ml of neocuproine solution. Thereafter, accurately add 10 ml of chloroform, mix them by violently shaking for about one minute and separate it into two layers by standing still.
- (5) Remove the water content of the chloroform layer⁽²⁾, take a portion thereof into the absorption cell of a photometer and measure the absorbance at near 460 nm in wavelength.

6.2.5 Calculation The amount of copper shall be obtained from the working curve prepared in 6.2.6 and the content of copper in sample shall be calculated from the following formula:

$$\text{Copper (\%)} = \frac{A}{W \times B} \times 100$$

where A : detected amount of copper in aliquot sample (g)

W : weighed-out amount of sample (g)

B : aliquot ratio of sample solution

6.2.6 Preparation of Working Curve Stepwise take various amounts of standard copper solution (0 to 50 µg as copper) into several beakers (100 ml), add 5 ml of nitric acid (1+4), hereinafter operate it in the same way as that of the sample in accordance with the procedures on and after 6.2.4 (3), prepare the relation curve between the obtained absorbance and the amount of copper and allow it to be the working curve.

Notes (1) When the content of copper is large, the aliquot amount shall be so lessened as to be 10 to 50 µg in copper

(2) The chloroform layer shall be dehydrated by using absorbent cotton or a filter paper

Remark: When the contents of bismuth and antimony are high, the operation shall be carried out as follows:

Weigh out 10 g of the sample into a beaker (300 ml), cover it with a watch glass, add 70 ml of nitric acid (1+4) and decompose it by quietly heating. After cooling, transfer it into a measuring flask of 250 ml and dilute it to about 200 ml with water. Thereafter, add 30 ml of sulfuric acid (1+3) and dilute it up to the marked line with water. After the precipitate of lead sulfate has settled, separately take accurately 50 ml from that supernatant into a beaker (100 ml), add 5 ml of citric acid solution and 5 ml of hydroxylamine hydrochloride solution and control pH at 5 to 6 by using aqueous ammonia (1+1). Hereinafter, operate it in accordance with the procedures on and after 6.2.4 (4).