

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for Chemical
Analysis of Copper Phosphorus
Brazing Filler Metals**

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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 Chemical Analysis of
Copper Phosphorus Brazing Filler Metals

Z 3903-1988

1. Scope

This Japanese Industrial Standard specifies the methods for determination of phosphorus, silver, copper, lead, tin and iron specified in JIS Z 3264.

2. General Matters

The general matters common to the methods of chemical analysis shall be in accordance with JIS K 0050, JIS K 0115 and JIS K 0121.

3. Sampling Method and Treatment of Sample

3.1 Sampling and Preparation of Sample Sampling and preparation of the sample shall be as follows:

- (1) In the sampling and preparation of the sample, a fair representation of the average quality of the sample shall be ensured, and segregation, contamination, etc. shall be cautiously warded off.
- (2) The sampling and preparation of the sample shall be in accordance with JIS Z 3900.

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

- (1) In weighing out the sample, stir sufficiently to attain the fair average composition of the sample, and ascertain that no foreign substance has mingled in.
- (2) In weighing out the sample, a chemical balance shall be used and be read to the nearest 0.1 mg.

Applicable Standards:

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 K 8005-Standard Substances for Volumetric Analysis

JIS Z 3264-Copper Phosphorus Brazing Filler Metal

JIS Z 3900-Methods for Sampling of Precious Brazing Filler Metals

JIS Z 8401-Rules for Rounding off of Numerical Values

4. Arrangement of Analysis Value

4.1 Number of Times of Analysis The same sample shall be analyzed not less than two times to enhance the reliability of the results.

4.2 Expression of Analysis Value An analysis value shall be expressed as follows:

- (1) The analysis value shall be expressed by mass percentage.
- (2) Each of the analysis values of an alloy component shall be calculated to two places of decimals, and then two or more of them shall be averaged and rounded off to one place of decimal in accordance with JIS Z 8401.
- (3) Each of the analysis values of impurities shall be calculation to three places of decimals, and then two or more of them shall be averaged and rounded off to two places of decimals in accordance with JIS Z 8401.

5. Blank Test

For the analysis, a blank test shall be conducted throughout the operations and measured values shall be corrected.

6. Determination of Phosphorus

6.1 Classification of Methods Determination of phosphorus shall be in accordance with either one of the following:

- (1) Magnesium phosphate separation-disodium ethylenediaminetetraacetate titration
- (2) Phosphorus vanadomolybdic acid absorptiometry

6.2 Magnesium Phosphate Separation-Disodium Ethylenediaminetetraacetate Titration

6.2.1 Principle Decompose the sample with nitric acid to precipitate ammonium magnesium phosphate, add hydrochloric acid, oxidize phosphorus by heating, add citric acid, neutralize with aqueous ammonium, and mix with stirring while cooling in water by adding magnesium chloride solution. Allow to stand, the precipitates filter, and dissolve in hydrochloric acid after washing with dilute aqueous ammonium. Add a definite amount of disodium ethylenediaminetetraacetate (hereinafter referred to as "EDTA") standard solution, further add aqueous ammonia in excess, and titrate with zinc standard solution, using Eriochrome Black T (hereinafter referred to as "EBT") as indicator, until the solution becomes reddish purple distinctly.

6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 3)
- (3) Nitric acid (1 + 1, 1 + 50)

- (4) Aqueous ammonia
- (5) Aqueous ammonia (5 + 100)
- (6) Magnesium chloride solution: Dissolve 50 g of magnesium chloride (6 hydrate) and 100 g of ammonium chloride in 800 ml of water. Add aqueous ammonia to make alkaline, using phenolphthalein solution (1 g/l ethanol solution) as indicator, and allow to stand for about 48 h. Filter precipitates, when generated, slightly acidify the filtrate by dripping hydrochloric acid, and then add water to dilute to 1 l. Regulate the pH of this solution to 5 to 6 using aqueous ammonia and hydrochloric acid, with *p*-nitrophenol solution (2 g/l) as indicator, and store.
- (7) Citric acid solution: Dissolve 30 g of citric acid (1 hydrate) in water to dilute to 100 ml.
- (8) 0.02 mol/l zinc standard solution: Weigh out 1.3074 g of zinc (JIS K 8005), dissolve it in as small an amount as possible of hydrochloric acid (1 + 1), cool, transfer into a 1000-ml volumetric flask using water, and dilute with water up to the marked line.
- (9) 0.02 mol/l EDTA standard solution: Dissolve 7.45 g of disodium ethylenediaminetetraacetate (2 hydrate) in water to 1000 ml. Carry out standardization as follows:

Take 25 ml of the 0.02 mol/l EDTA standard solution accurately, add 10 ml of ammonium chloride solution (250 g/l) and 0.1 ml of EBT solution as indicator, dilute to about 100 ml with water, add aqueous ammonia (1 + 1) drop by drop until the colour of solution turns blue, titrate with 0.02 mol/l zinc standard solution, and take the point when the colour of solution has changed to reddish purple as the end point. Calculate the factor from the following equation.

$$F = \frac{V}{25}$$

where F : factor of the 0.02 mol/l EDTA standard solution

V : consumption of the 0.02 mol/l zinc standard solution (ml)

- (10) EBT solution: After dissolving 0.3 g of EBT in 15 ml of ethanol (95 vol %), add 15 ml of triethanolamine.

6.2.3 Mass of Sample to Be Weighed Out 0.20 g of the sample shall be weighed out⁽¹⁾.

Note (1) Although 0.20 g of the sample is to be weighed out, if segregation is anticipated, 1.0 g or 2.0 g of the sample is decomposed with the nitric acid (1 + 1) and transferred to a 250 ml volumetric flask using water, and diluted up to the marked line with water. A definite amount (equivalent to 0.20 g) is separated from this solution, and 10 ml of nitric acid (1 + 1) is added.

6.2.4 Procedure The determination procedure shall be made in the following order:

- (1) Weigh out the sample and transfer to a conical beaker (300 ml), add 10 ml of the nitric acid (1 + 1), cover with a watch glass, and heat quietly to dissolve.
- (2) Add 10 ml of the hydrochloric acid to this solution, and boil for about 5 min gently. Wash the bottom face of watch glass and the inside walls of beaker with water.
- (3) Cool the solution down to about 40°C, add 2 ml of citric acid solution, and then neutralize with the aqueous ammonia to dissolve the once generated copper hydroxide. Add carefully until it presents blue colour of copper ammine.
- (4) Add 10 ml of magnesium chloride solution, and cool in water while sufficiently agitating for about 3 min by rubbing the walls of beaker with a glass rod having rubber tube (policeman). Add 10 ml of aqueous ammonia, mix with shaking sufficiently, and allow to stand for about 2 h at room temperature.
- (5) After standing, filter this using filter paper (Class 5 C), and wash sufficiently the precipitate and filter paper with aqueous ammonia (5 + 100) until chloride ions fade away. Further wash them three times. Drip the warm hydrochloric acid (1 + 3) on the filter paper to dissolve the precipitate into the beaker (500 ml) and wash the filter paper sufficiently with warm water.
- (6) Add a definite amount of 0.02 mol/l EDTA standard solution correctly to this solution, add warm water at about 80°C to make approximately 250 ml in liquid quantity, add 3 to 5 drops of the EBT solution as indicator, and add aqueous ammonia carefully until the solution presents blue colour. Then, add 20 ml in excess⁽²⁾.
- (7) Titrate this solution with zinc standard solution while carefully and sufficiently agitating, and take the point when the blue colour of the solution has changed to fresh purplish red as the end point.

Note (2) In this case pH of the solution indicates about 9.5, and it can be maintained at 9 and upwards until the completion of the titration.

6.2.5 Calculation Phosphorus content in the sample shall be calculated from the following formula.

$$\text{Phosphorus wt \%} = \frac{(V_1 - V_2) \times 0.0006195}{m} \times 100$$

where V_1 : consumption of the 0.02 mol/l EDTA standard solution (ml)
 V_2 : consumption of the 0.02 mol/l zinc standard solution (ml)
 m : mass of the sample weighed out (g)