

JIS

JAPANESE INDUSTRIAL STANDARD

Methods of Chemical Analysis for
Phosphor Copper Ingots

JIS H 1552-1976
(Reaffirmed: 1984)

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1. General Rules

1.1 Scope This Japanese Industrial Standard specifies the methods of chemical analysis for chemical constituents (copper, phosphorus, iron, lead, tin) specified in JIS H 2501.

1.2 General items common to the chemical analysis of phosphorus copper ingot shall be in accordance with JIS H 1012.

1.3 Method of Taking Chemical Analysis Sample

1.3.1 Method of taking cast ingot in the case of taking sample necessary for chemical analysis test shall be subjected to the agreement between the parties concerned.

1.3.2 After properly and perfectly removing adhering substance from the surface of cast ingot sampled in accordance with 1.3.1, serve the ingot for the chemical analysis test.

1.3.3 The sample obtained in accordance with 1.3.2 shall be crushed quickly to the size of 0.5 to 3.0 mm in diameter by using an iron mortar or a suitable device. Carefully remove iron powder and so on using a powerful magnet, and after keeping in a desiccator, weigh it out.

2. Method for Copper Determination

2.1 Summary After dissolving the sample in nitric acid, heat and evaporate, make it into the syrup state and add warm water and a small amount of nitric acid to dissolve the soluble salt. Add sulfuric acid to electrolyze it (the residual liquid of electrolysis may be used for the determination of phosphorus or iron).

Applicable Standards:

JIS H 1012-General Rules for Chemical Analysis of Copper Products
and Copper Alloys

JIS H 2501-Phosphor Copper Metal

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2.2 Operations Weigh out the sample of 1 g into a beaker for analysis (refer to Appendix Fig. 1) and cover with a watch glass, heat and dissolve adding 15 mm of nitric acid (1 + 1). After washing the lower surface of watch glass and inner wall of the beaker, further, heat and make it a syrup state. Add 3 ml of nitric acid (1 + 1) and 50 ml of warm water and heat, and dissolve soluble salt ⁽¹⁾. Add to the solution 10 ml of sulfuric acid (1 + 1), after making approx. 150 ml with water, use cylindrical platinum cathode (refer to Appendix Fig. 2) and spiral platinum anode (refer to Appendix Fig. 3) and cover with two pieces of semicircular watch glasses (refer to Appendix Fig. 4), electrolyze by passing electric current of 0.3 to 0.5 A for one night at room temperature. After finishing electrolysis, wash the lower surface of watch glass, inner wall of the beaker and surface of part of handle of electrode exposed on the surface of liquid with the washing bottle, let the level of electrolysis of liquid rise approx. 5 mm with the washing water, and continue electrolyzing further for approx. 30 min, until no more copper is educed on the handle newly immersed in the liquid. Then pull up the electrodes while passing the electric current and washing both electrodes (residual electrolyte may be used for the determinatiion for phosphorus or iron).

After washing with water first, then with ethyl alcohol, dry quickly at approx. 30 min, weigh the mass. Calculate copper content from the increment according to the following formula:

$$\text{Copper (\%)} = \frac{\text{Mass of deposited copper (g)}}{\text{Sample (g)}} \times 100$$

Note ⁽¹⁾ If precipitates exist, after standing still in warm place for 1 to 2 h, filter quickly with a fine filter paper containing a small quantity of pulp, and wash with warm nitric acid (2 + 100).

3. Method for the Determination of Phosphorus

The method for the determination of phosphorus shall be carried out according to either gravimetric analysis or titration.

3.1 Gravimetric Method

3.1.1 Summary Dissolve the sample with nitric acid, after oxidizing phosphorus with perchloric acid, neutralize with aqueous ammonia and make acidic with nitric acid, let the phosphorus precipitate by adding ammonium nitrate and ammonium molybdate, and after dissolving this into aqueous ammonia, let generate precipitation with mixture of magnesia. Filter the precipitate, and after heating strongly, weigh the mass as magnesium pyrophosphate.

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3.1.2 Operations Weigh 1g of the sample into a beaker (300 ml), add 20 ml of nitric acid (1 + 1) and dissolve gently by heating. Then heat and evaporate adding 20 ml of perchloric acid, continue heating for approx. 5 min after generating white fume. After cooling, add 100 ml of warm water, transfer the solution into a 250 ml measuring flask, and after cooling, dilute it up to the marked line with water.

Separately take 25 ml of this solution into a beaker (500 ml), after neutralizing with aqueous ammonia and make it acidic with nitric acid (1+ 1), and add excessively 10 ml. Add 10 g of ammonium nitrate, keep at approx. 60°C, add 100 ml of ammonium molybdate solution warmed previously at approx. 60°C. After mixing enough by stirring for five min, keep the temperature at approx. 50°C, leave to stand still for approx. 2 h and let the precipitate to settle.

Filter the precipitate with a fine filter paper containing a small quantity of pulp for filter paper as ingredients. After washing with nitric acid (1 + 100) till copper ion fades out, then wash with nitric acid (1 + 500) several times, dissolve in 20 ml of aqueous ammonia (1 + 1), and wash the filter paper with aqueous ammonia (1 + 20) several times.

Make the solution weak acidic with hydrochloric acid, keep the quantity of liquid not more than 60 ml, add 20 ml of mixture of magnesia ⁽³⁾, and make alkaline by adding aqueous ammonia while cooling in water, mix violently for 5 min or more to generate precipitation, at last add 10 ml of aqueous ammonia and mix enough, and after approx. 2 h filter with a fine filter paper.

Wash the precipitate with aqueous ammonia (1 + 20) until chlorine ion fades out, transfer into a porcelain crucible together with the filter paper, after drying incinerate at low temperature, finally ignite at 1050 to 1100°C to reach a constant mass, after leaving to cool in a desiccator, weigh out magnesium pyrophosphate, and calculate the phosphorus content from the following formula:

$$\text{Phosphorus (\%)} = \frac{\text{Mg}_2\text{P}_2\text{O}_7(\text{g}) \times 0.2787}{\text{Sample (g)} \times \frac{1}{10}} \times 100$$

Notes ⁽²⁾ For ammonium molybdate solution, dissolve 40 g of ammonium molybdate with 300 ml of water and 80 ml of aqueous ammonia, after cooling, add this solution little by little into 60 ml of nitric acid (1 + 1) by agitating to mix. This solution is used after filtration at every time of use.

⁽³⁾ For mixture of magnesia, dissolve 50 g of magnesium chloride and 100 g of ammonium chloride into 800 ml of water, make alkaline adding aqueous ammonia using phenolphthalein solution as indicator, and leave for two days and nights. When precipitation develops, filter the solution and make weak acidic with chloric acid and amount to 1 l with water. Preserve this solution while adjusting pH between 5 to 6 using aqueous ammonia and hydrochloric acid with *p*-nitrophenol as indicator.