

JIS

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JAPANESE INDUSTRIAL STANDARD

Methods for Determination of
Copper in Ores

JIS M 8121-1982



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Methods for Determination of
Copper in Ores

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1. Scope

This Japanese Industrial Standard specifies the methods for determination of copper in ores. However, it does not apply to the ores to which the methods for determination of copper are specified in other Japanese Industrial Standards.

2. General Matters

General matters common to the analytical method shall be in accordance with JIS K 0050, JIS K 0115 and JIS K 0121.

3. Methods for Sampling and Preparation of Sample

3.1 Sampling and Preparation of Sample The sampling and preparation of sample shall be in accordance with JIS M 8101 and JIS M 8083.

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

- (1) In the case of weighing out a sample, the sample shall be taken care of showing the mean composition by sufficiently mixing the sample with stirring and it shall be confirmed that there is no mixing of foreign substances.

Applicable Standards: See page 30.

JAPANESE STANDARDS ASSOCIATION

- (2) The sample shall be put into the air bath conditioned at $105 \pm 5^{\circ}\text{C}$ and dried. It shall be taken out from the air bath every 2 h and cooled to ordinary temperature in a desiccator. The drying shall be repeated until the decreased amount due to drying becomes not more than 0.1 % per 2 h. However, the drying condition (temperature, time, etc.) for the sample easy to change its quality due to containing sulfides or the like, shall be in accordance with the agreement between the parties concerned.
- (3) The weighing out the sample, the specified sample shall generally be weighed out to the place of 0.1 mg by use of chemical balance.

4. Method for Expression of Analytical Value and Cautions on Operation

4.1 Method for Expressing Analytical Value The method for expression of analytical value shall be as follows:

- (1) The analytical value shall be expressed by percentage and rounded off to 3 places of decimals according to JIS Z 8401.
- (2) The analysis shall repeatedly be carried out 2 times in the same laboratory. When the difference between these analyses is less than the in-room tolerance, hereinafter referred to as the "tolerance", the mean value shall be obtained and reported by rounding off it to 2 places of decimals according to JIS Z 8401.
- (3) When the difference between the analytical values repeated 2 times is not less than its tolerance, the analysis shall again be repeated 2 times.

JAPANESE STANDARDS JIS M 8121

(4) The tolerance shall be as shown in Table 1.

Table 1. Tolerance⁽¹⁾

Method of determination	Division	Unit: %
		In the same laboratory for analysis (repetition)
Electrogravimetry	20 and over	0.100
	0.1 to 1 excl.	0.015
Titration method of sodium thiosulfate	1 to 5 excl.	0.030
	5 to 15 excl.	0.080
	15 to 40 excl.	0.150
	0.5 to 1 excl.	0.030
Absorptiometry of ammonia	1 to 5 excl.	0.090
	5 to 10 excl.	0.150
	0.01 to 0.2 excl.	0.006
Atomic absorption method	0.2 to 1 excl.	0.040
	1 to 3 excl.	0.055

Note ⁽¹⁾ When 2 analytical values cover 2 divisions, the tolerance of the division corresponding to the mean value of the 2 analytical values shall be applied.

4.2 Cautions on Analytical Operation For analysis, blank test shall be carried out through the whole operation and the measured value shall be corrected.

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5. Method of Determination

5.1 Classification of Methods The method for determination of copper in ores shall be in accordance with either one of the following methods:

- (1) Electrogravimetry This method applies to the sample of copper content of not less than 20 %.
- (2) Titration Method of Sodium Thiosulfate This method applies to the sample with copper content of 0.1 % up to 40 % excl.
- (3) Absorptiometry of Ammonia This method applies to the sample with copper content of 0.5 % up to 10 % excl.
- (4) Atomic Absorption Method This method applies to the sample with copper content of 0.01 % up to 3 % excl.

5.2 Electrogravimetry

5.2.1 Summary Resolve the sample with nitric acid and sulfuric acid, and concentrate it by heating until white fume of sulfuric acid is generated. After dissolving soluble salt with water, coprecipitate arsenic and antimony together with iron hydroxide by adding aqueous ammonia and filter it. Concentrate the filtrate by heating. After regulating the amount of solution, add sulfuric acid and nitric acid, and electrolyze it by passing low electric current. After completion of electrodeposition, wash out the cathode with water and ethanol, and then weigh the amount of electrodeposited copper with drying.

In addition to this, dissolve precipitates such as iron hydroxide in hydrochloric acid. After diluting it to a certain amount, obtain the copper content absorbed in the precipitates with atomic absorption method and add this to the amount of deposited copper.

JAPANESE STANDARDS ASSOCIATION

5.2.2 Reagent The reagent shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1, 1 + 3, 1 + 19)
- (3) Nitric acid
- (4) Nitric acid (1 + 1)
- (5) Hydrofluoric acid
- (6) Hydrobromic acid
- (7) Sulfuric acid (1 + 1, 1 + 4)
- (8) Aqueous ammonia
- (9) Aqueous ammonia (1 + 100)
- (10) Bromine
- (11) Ammonium sulfate
- (12) Solution of sodium chloride (0.05 w/v %)
- (13) Solution of ammonium iron (III) sulfate
Dissolve 4.3 g of ammonium (12-hydrate) of iron (III) sulfate in 5 ml of sulfuric acid (1 + 1) and water, and dilute it to 100 ml. Allow 1 ml of this solution to contain about 5 mg of iron.
- (14) Ethanol (99.5 v/v %)
- (15) Standard copper solution (10 µgCu/ml) After dissolving 1.000 g of copper (not less than 99.9 %) with 20 ml of nitric acid (1 + 3), add 5 ml of sulfuric acid (1 + 1) and concentrate it by heating until white fume of sulfuric acid is sufficiently generated. After leaving it cooled, add about 50 ml of water, and dissolve it by heating. After cooling, transfer it into a measuring flask of 1000 ml, and dilute it to the marked line with water. Take this solution as the stock solution. Every time when this stock solution is used, accurately dilute the required amount of it to 100 times and make this be the standard copper solution.

JAPANESE STANDARDS ASSOCIATION

5.2.3 Apparatus and Devices The apparatus and devices shall be as follows:

- (1) Cylindrical Platinum Cathode For the cylindrical platinum cathode, that shown in Fig. 1 shall generally be used.
- (2) Spiral Platinum Anode For the spiral platinum anode, that shown in Fig. 2 shall generally be used.

Fig. 1. Cylindrical Platinum Cathode

Unit: mm

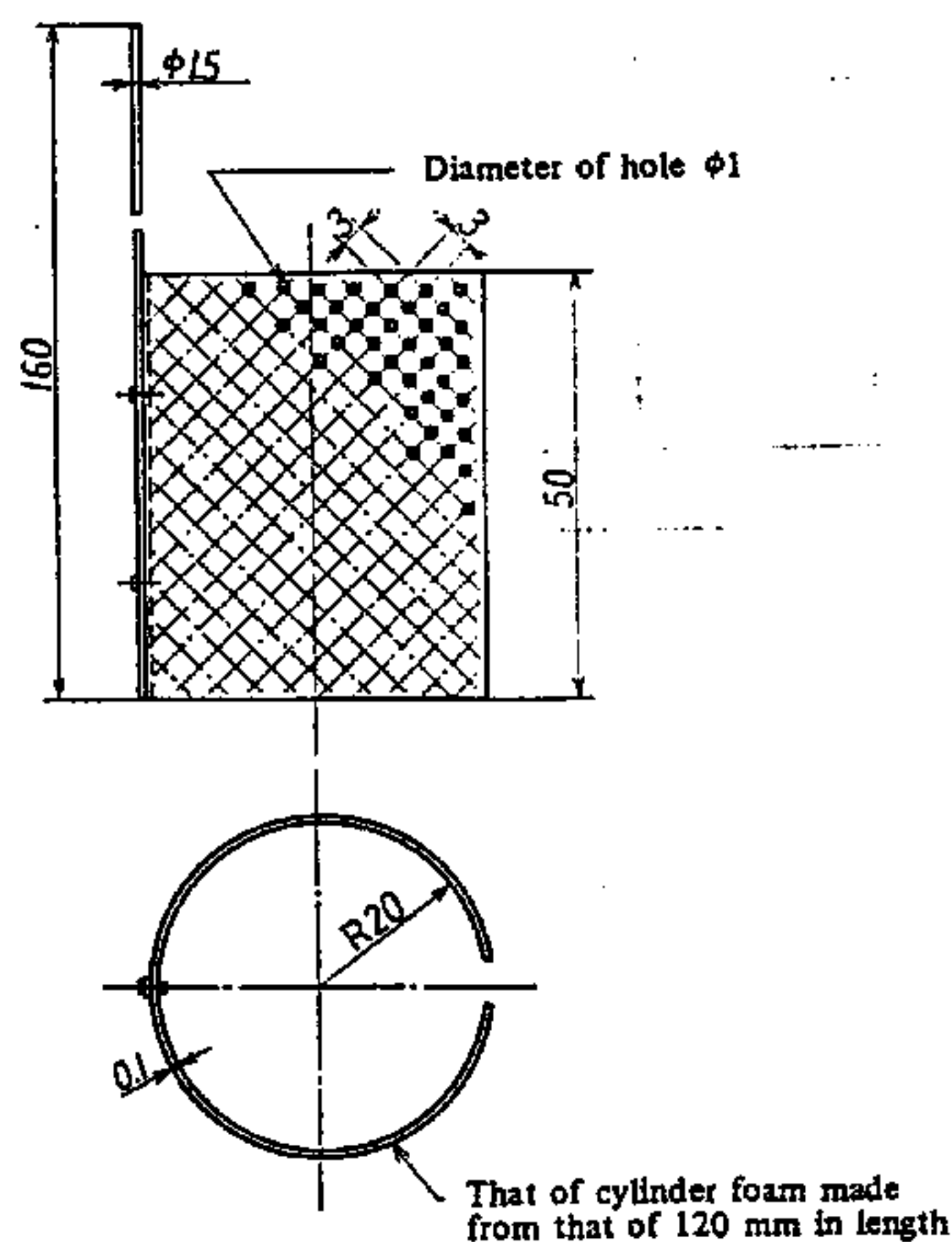
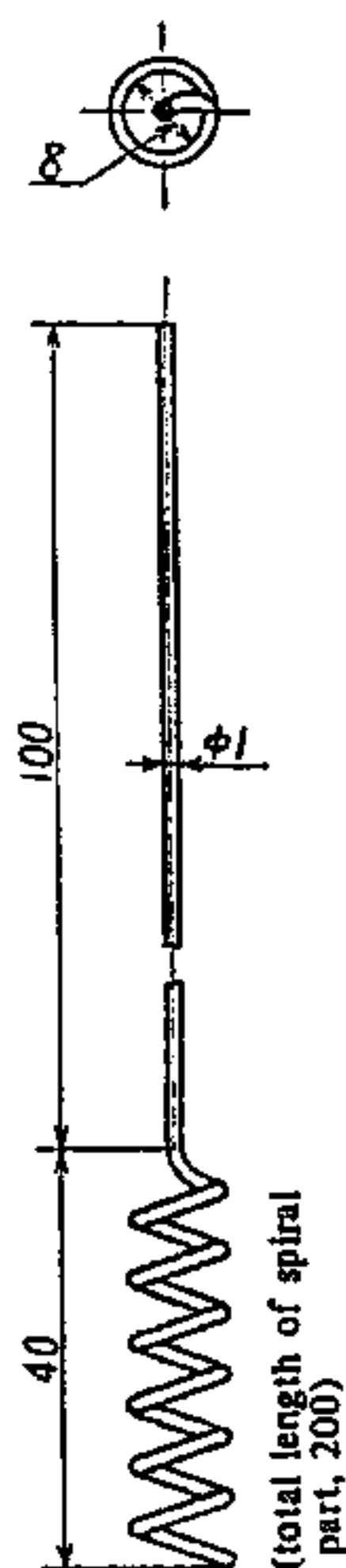


Fig. 2. Spiral Platinum Anode

Unit: mm

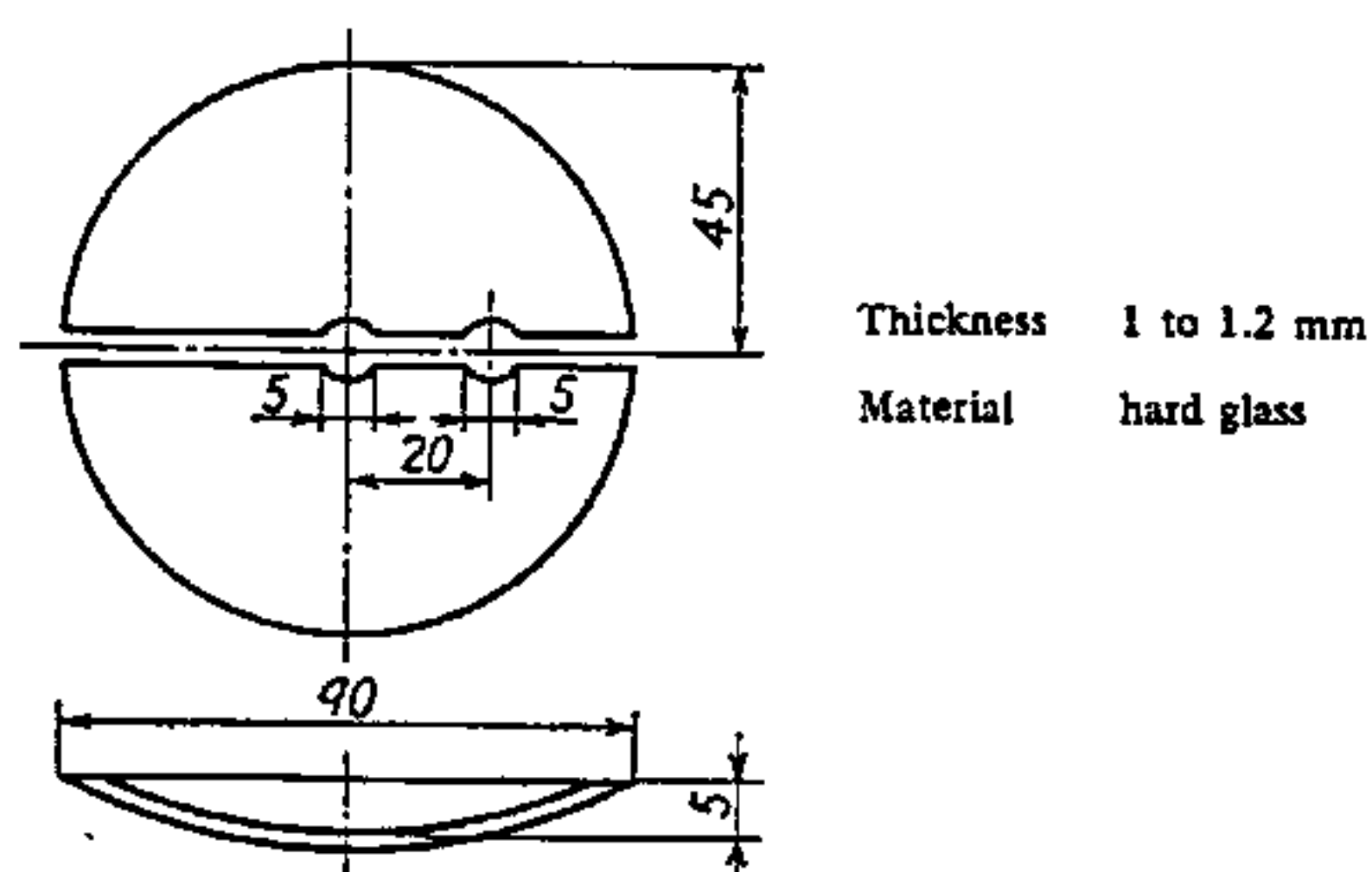


JAPANESE STANDARDS ASSOCIATION

- (3) Semi-circular Watch Glass For the semi-circular watch glass, that shown in Fig. 3 shall generally be used.

Fig. 3. Watch Glass of Semi-circular Form

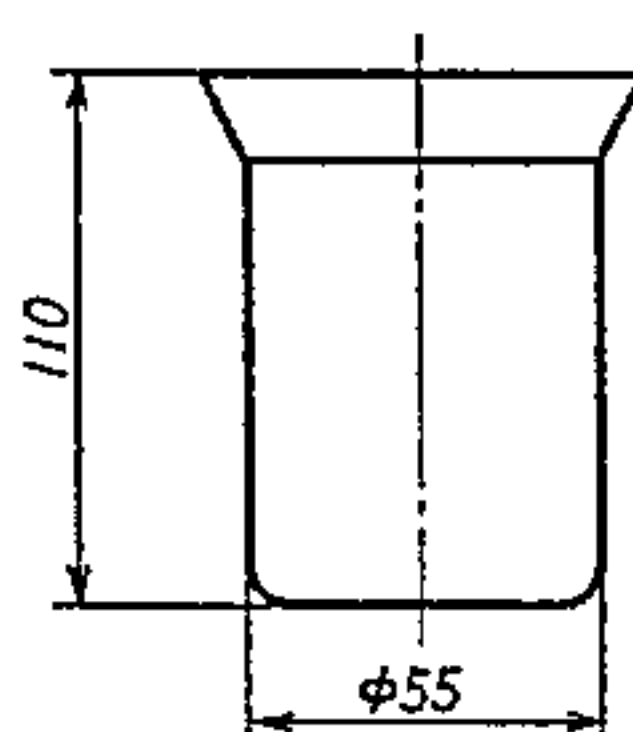
Unit: mm



- (4) Electrolysis Beaker For the electrolysis beaker, that of 300 ml in capacity given in Fig. 4 or in JIS R 3503, shall generally be used.

Fig. 4. Electrolysis Beaker

Unit: mm



- 5.2.4 Weighed Out Amount of Sample For sampling, weigh out 2 g of the sample.

JAPANESE STANDARDS ASSOCIATION

5.2.5 Operation The operation of determination shall be carried out by the following procedures:

- (1) Weigh out the sample, and put it into a beaker (300 ml). Cover it with a watch glass, and add 15 to 20 ml of sulfuric acid (1 + 1) and 20 to 30 ml of nitric acid⁽²⁾. Resolve it by heating slowly. After the end of violent reaction, wash the watch glass and the inner wall of beaker with a little amount of water, and concentrate⁽³⁾ it by heating again. Let white fume of sulfuric acid be sufficiently generated⁽⁴⁾⁽⁵⁾.
- (2) After leaving it cooled, add about 150 ml of water, and dissolve soluble salt by heating⁽⁶⁾⁽⁷⁾. After leaving it cooled for a short time, add a little amount of aqueous ammonia until the once precipitated basic copper salt dissolves during mixing this solution⁽⁸⁾. Further, add excessively aqueous ammonia of about 20 ml. After boiling it for about 5 minutes by heating, filter it by use of a filter paper (Grade 5 A), and receive the filtrate with a beaker (500 ml). Several times wash it with warm aqueous ammonia (1 + 100).
- (3) Wash the precipitate and put it into the original beaker. After dissolving the precipitate of hydroxide by gradually adding 15 ml of warm sulfuric acid (1 + 4) little by little from over the filter paper, sufficiently wash the filter paper with warm water. Receive this solution in the original beaker to which the main precipitate is transferred by washing. After making the amount of solution about 100 ml by adding water, add aqueous ammonia during mixing with stirring until the precipitate of iron hydroxide is slightly generated. Further, add excessively 5 ml of it.

After boiling it for about 5 min, filter it by use of a filter paper (Grade 5 A) and receive the filtrate in the original beaker (500 ml). Several times wash it with warm aqueous ammonia (1 + 100) and stock the precipitate.

JAPANESE STANDARDS ASSOCIATION

- (4) After concentrating⁽⁹⁾ the amount of solution in the beaker (500 ml) to about 150 ml of solution amount by heating, add 10 ml of sulfuric acid (1 + 1), and concentrate the amount of solution to about 100 ml by continuously heating further. Wash this solution and transfer to the electrolysis beaker. Add 5 ml of nitric acid (1 + 1) and make the amount of solution about 150 ml by adding water⁽¹⁰⁾.
- (5) Using the cylindrical platinum cathode and the spiral platinum anode which mass are known, cover the solution sample with 2 semi-circular watch glasses, and make the temperature of solution at 15 to 30°C. After electrolyzing it for about 18 h⁽¹¹⁾⁽¹²⁾ by passing the electric current of about 0.25 A, wash the bottom surface of watch glass, the inner wall of electrolysis beaker and the electrode portion exposed over the solution surface with water, and raise the surface of electrolytic solution by about 5 mm by washings and further continue to electrolyze it for about 1 h⁽¹³⁾.
- (6) Slowly pull up the both electrodes during washing while electric current is passed, and as quickly as possible dip it into fresh water. Take off the cathode, and after washing it by moving up and down several times, dip it into ethanol. Remove water by moving it up and down several times, and dry it in an air bath of about 80°C as quickly as possible. Immediately thereafter, put it into a desiccator and leave it cooled for about 30 min.
- (7) By use of the chemical balance used for weighing the mass of platinum cathode before the electrodeposition of copper, weigh the mass of the platinum cathode deposited with copper.