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JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of
Brass Brazing Filler Metals

JIS Z 3902—1984

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Methods for Chemical Analysis of
Brass Brazing Filler Metals

Z 3902-1984

1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of chemical components (copper, tin, iron, nickel, lead, aluminium, manganese, silver, phosphorus, and silicon) specified in JIS Z 3262.

2. General Matters

General matters common to methods for chemical analysis shall be in accordance with JIS K 0050, JIS K 0115 and JIS K 0121.

3. Sampling Method and Handling Method of Analytical Sample3.1 Sampling and Preparation of Analytical Sample

3.1.1 In sampling and preparation of analytical sample, average quality of total samples shall be represented and cares shall be specially taken about segregation, contamination, etc.

3.1.2 The sampling and preparation of analytical sample shall be carried out according to 5. of JIS H 0321 and 2.2 of JIS H 0301, as appropriate, not less than 2 (one when unit melting amount is very little) pieces of about 100 g cast sample shall be taken per one melting, not less than 2 times amount of chips required for analysis be manufactured by chipping this sample and it shall be made the analytical sample by sufficiently mixing.

3.1.3 The chipping method of cast sample shall be carried out according to the following procedure:

- (1) Drills and other tools shall be cleaned with ethanol and the like.
- (2) The surface of sample shall be cleaned by removing oil and other deposits adhered thereto.
- (3) The central part or parts near both ends of sample shall be penetrated by drilling it at right angles thereto from one side surface, or it shall be drilled until the drill reaches the center part from both surfaces at least. When these drillings can not be carried out, it shall be chipped out with a suitable method.

Applicable Standards: See page 40.

- (4) The size of chipping sample shall be made about not more than 5 mm in length by drilling to the degree so as chips not to become too thick. If chips are of string shape, they shall be cut to about 5 mm max. with clean scissors.

Further, cares shall be taken so that the abrasion powder of drill is not mixed with the chips at that time. Furthermore, water and the like shall not be added to cool the sample.

3.1.4 When the sampling and preparation of analytical sample cannot be in accordance with the above-mentioned specifications, they shall be subjected to the agreement between the parties concerned.

3.2 Weighing Out of Analytical Sample

3.2.1 When the analytical sample is weighed out, cares shall be taken so that the average composition is represented by sufficiently mixing and it shall be further confirmed that foreign matters are not mixed.

3.2.2 For weighing out the analytical sample, a chemical balance shall be used and it shall be read to the place of 0.1 mg.

4. Cautions on Analytical Operation

4.1 The analysis shall generally be carried out not less than 2 times about the same sample.

4.2 In analysis, blank tests shall be carried out through whole operation and the content rate shall be corrected.

5. Expression of Analytical Results

The analytical value shall be expressed by percentage and rounded to the place specified in JIS Z 3262 in accordance with JIS Z 8401 by averaging not less than 2 analytical values. However, regarding zinc content, chemical components excepting zinc shall be calculated according to the above-mentioned procedure and thereafter the remainder of 100 subtracted by the sum of respective contents shall be taken as the zinc content.

6. Copper Determination Method

6.1 Classification of Methods The determination method of copper shall be carried out according to electrogravimetry.

6.2 Electrogravimetry

6.2.1 Summary The sample shall be decomposed with nitric acid, electrolysis be carried out after adding water and sulphuric acid and the mass of copper deposited on the cathode shall be measured.

6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 9)
- (2) Nitric acid (1 + 1, 1 + 50, 1 + 100)
- (3) Sulphuric acid (1 + 1, 1 + 50)
- (4) Ethanol (95 v/v %)

6.2.3 Apparatus and Devices For the apparatus and devices, the following shall generally be used.

- (1) Electrolytic beaker (refer to Attached Fig. 1)
- (2) Platinum electrode A (refer to Attached Fig. 2)
- (3) Platinum electrode B (refer to Attached Fig. 3)
- (4) Semicircular watch glass (refer to Attached Fig. 4)

6.2.4 Weighing Out Amount of Sample For the sample, 1 g shall be weighed out.

6.2.5 Operation The determining operation shall be carried out according to the following procedures:

- (1) Weigh out the sample, transfer it into a tall beaker (300 ml), cover it with a watch glass, add 10 ml nitric acid (1 + 1), slowly decompose it by heating and sweep out nitrogen oxide by boiling. Thereafter, wash the bottom surface of watch glass and the inner wall of beaker with water.
- (2) Make the amount of liquid about 100 ml by adding water thereto, and add little by little 10 ml sulphuric acid (1 + 1) while mixing with stirring ⁽¹⁾.
- (3) Transfer the solution into an electrolytic beaker and dilute it to about 150 ml with water. Thereafter, make the platinum electrode A, of which the mass is preliminarily measured, a cathode, use the platinum electrode B as anode, cover them with 2 pieces of semicircular watch glasses and carry out electrolysis for one night applying 0.3 to 0.5 A current thereto at room temperature.
- (4) Wash the bottom surface of watch glass, the inner wall of beaker and the part of electrode stem exposed above the liquid surface with a little amount of water, raise the surface of electrolyte by about 5 mm with that washing water and further continue to carry out the electrolysis for about 30 min.
- (5) When no more copper is deposited at the stem part of cathode freshly dipped in the electrolyte, pull up both electrodes while washing them with water with current applied, at last dip them into fresh water quickly and remove the cathode.
- (6) Wash the cathode at first with water and then with ethanol (95 v/v %), quickly dry it in air bath at about 80°C and measure the mass after leaving it cooled as it is in a desiccator for about 30 min.

Note ⁽¹⁾ When the precipitate of lead sulphate is generated in that case, filter it using a filter paper (Class 5 B) and sufficiently wash it with sulphuric acid (1 + 50).

- Remarks 1. When the sample contains silicon, operate according to the procedure of 6.2.5 (1). Thereafter, make it syrupy state by further evaporating it by heating, add 10 ml of nitric acid (1 + 1) and 50 ml of warm water thereto, and dissolve soluble salts by heating. Thereafter, filter it using a filter paper (Class 5 B) and sufficiently wash it using warm nitric acid (1 + 50). Hereinafter, follow the operation of Remark 4. However, when it contains tin together with silicon, follow the operation of Remark 2.
2. When the sample contains tin, operate according to Remark 1. and dissolve soluble salts. Thereafter, further heat it until it boils and quietly leave it at warm place for 1 to 2 h. Quickly thereafter, filter it with the filter paper (Class 5 B) containing a small amount of filter paper pulp and sufficiently wash it using warm nitric acid (1 + 50). Hereinafter, follow the operation of Remark 4. [the filtrate in the determination of tin of 7.2.5 (2) can be used].
3. When the sample contains silver, operate according to Remark 1. and dissolve soluble salts. Thereafter, make the amount of liquid about 150 ml by adding water, drop hydrochloric acid (1 + 9) while mixing it with stirring and generate the precipitate of silver chloride. After the precipitate is not generated, further add 1 ml thereof in excess, quickly heat it for about 5 min and boil it. After cooling it in running water, filter it using a filter paper (Class 5 B) and sufficiently wash it at first with nitric acid (1 + 100) and then with cold water. Hereinafter, follow the operation of Remark 4. (the filtrate that the silver chloride in 13.2.4 is filtered can be used).
4. When the operation follows Remarks 1. to 3., add 15 ml of sulphuric acid (1 + 1) to respective filtrates, evaporate them by heating until white fume of sulphuric acid is generated, add about 50 ml of water after cooling and dissolve soluble salts [when the precipitate of lead sulphate is found, remove it according to the operation of Note (1)]. Add 5 ml nitric acid (1 + 1) and hereinafter, carry out the electrolysis according to the procedure in 6.2.5 (3) and thereafter.

6.2.6 Calculation The content of copper in sample shall be calculated from the following formula:

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

where w : mass of copper deposited on cathode (g)

W : weighed out amount of sample (g)

7. Tin Determination Methods

7.1 Classification of Methods The determination method of tin shall be in accordance with either one of the following:

- (1) Potassium iodate titration method
- (2) Atomic absorption method

7.2 Potassium Iodate Titration Method

7.2.1 Summary The sample is decomposed with nitric acid, evaporated to dryness and tin is made metastannic acid. Thereafter, soluble salts are dissolved with warm water and nitric acid and filtered. After the precipitate is decomposed with nitric acid and sulphuric acid, white fume of sulphuric acid is generated. Thereafter, hydrochloric acid is added and tin is reduced with antimony trichloride and lead (or nickel) using a cup put with sodium hydrogen carbonate. It is titrated with potassium iodate standard solution by using starch as an indicator.

7.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Nitric acid
- (3) Nitric acid (1 + 1, 1 + 50)
- (4) Sulphuric acid
- (5) Lead [It is 99.99 % min. in purity and lead equivalent to Extra Class of JIS H 2105 or granular lead for assay of JIS K 8701].
- (6) Nickel cylinder or nickel wire It is 99.8 % min. in purity (that equivalent to Class 3 of JIS H 4502) and shall generally be of shape A or B of Attached Fig. 5. This cylinder or wire can be repeatedly used.
- (7) Carbon dioxide When it contains oxygen, 45 g of cuprous chloride is dissolved in 300 ml of hydrochloric acid (1 + 1) and added with a small amount of metallic copper chips. The gas shall be refined by passing the gas through the solution left as it is for not less than 24 h after being sealed.
- (8) Antimony trichloride solution It is prepared by dissolving 5 g of antimony trichloride in 500 ml hydrochloric acid (1 + 1) by heating.
- (9) Sodium hydrogencarbonate solution (saturated)
- (10) Standard tin solution (4 mg/Sn/ml) 1.000 g of tin (it is not less than 99.90 % in purity and equivalent to Class 1 A of JIS H 2108) is weighed out, transferred into a beaker (300 ml), added with 20 ml sulphuric acid, covered with a watch glass and decomposed by heating. Thereafter, it is successively heated and the sulphur adhered to the inner walls of watch glass and beaker

are vaporized. After cooling, the inner walls of the watch glass and beaker are washed using water and hydrochloric acid (1 + 1), and soluble salts are dissolved by adding 50 ml of hydrochloric acid (1 + 1). The solution is transferred into a measuring flask of 250 ml and diluted up to the marked line with hydrochloric acid (1 + 1). Otherwise, 1.000 g of tin is decomposed with 50 ml hydrochloric acid on water bath using platinum plate as catalyst, transferred into a measuring flask of 250 ml and may be diluted up to the marked line with hydrochloric acid (1 + 1).

- (11) Potassium iodate standard solution 1.784 g of potassium iodate is dissolved in 100 ml of the water in which 0.5 g of sodium hydroxide and 5 g of potassium iodide are dissolved and diluted to 1000 ml with water.

Though this solution is equivalent to about N/20, the standardization shall be carried out as follows:

Accurately 25 ml standard tin solution is sampled, put into the Erlenmeyer flask (500 ml) of reducing apparatus and added with 200 ml of water and 50 ml of hydrochloric acid. Hereinafter, the operation is carried out according to the procedure of 7.2.5 (4) to (6) and the corresponding amount of tin is obtained from the following formula:

$$f = \frac{0.100}{V}$$

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

0.100: amount of tin in 25 ml of standard tin solution (g)

V : used amount of potassium iodate standard solution (ml)

- (12) Iodine standard solution 20 g of potassium iodide and 6.4 g of iodine are weighed out, transferred into a beaker (300 ml), added with about 50 ml of cold water and iodine is completely dissolved by mixing with shaking. It is diluted to about 150 ml with water and filtered using a glass filter (Buchner Type, 17 G 4). Thereafter, it is made 1000 ml using water and stored in a brown bottle. Though 1 ml of this solution is equivalent to about 3 mg of tin, the standardization is carried out at every use with standard tin solution. The standardization method is in accordance with (11) potassium iodate standard solution, as appropriate.
- (13) Starch solution It is prepared by a method wherein 1 g of starch (soluble) or starch is made muddy by adding a little amount of water, added into 100 ml hot water while mixing it with stirring and boiled for about 1 min. Thereafter, it is left cooled as it is and insoluble matters is filtered. This solution is prepared at every use and that turning red brown by iodine shall not be used.

7.2.3 Apparatus For the reducing apparatus, that given in Attached Fig. 6 shall generally be used and otherwise that given in Fig. 7 may be used.

7.2.4 Weighing Out Amount of Sample For the sample, 1 g shall be weighed out ⁽²⁾.

7.2.5 Operation The determining operation shall be carried out according to the following procedure:

- (1) Weigh out the sample, transfer it into a tall beaker (300 ml), cover it with a watch glass, add 15 ml of nitric acid (1 + 1) ⁽³⁾ and decompose it by heating. Wash the bottom surface of watch glass and the inner surface of beaker with water, further carefully heat it on water bath and evaporate it to dryness.
- (2) Add 5 ml of nitric acid and about 50 ml of warm water, dissolve soluble salts by boiling and leave it quiet at warm place for 1 to 2 h. Immediately thereafter, filter it with filter paper (Class 5 C) containing a small amount of pulp and sufficiently wash it using warm nitric acid (1 + 50).
- (3) Put the precipitate together with the filter paper into the original beaker, add 15 ml of nitric acid and 10 ml of sulphuric acid, cover it with a watch glass, carefully decompose it by heating and sufficiently generate white fume of sulphuric acid by further successively heating it.

If the decomposition of organic matter is insufficient, further add 10 ml of nitric acid after cooling and repeat the decomposition thereof by heating. After cooling, wash the watch glass and the inner surface of beaker with water, remove the watch glass, further evaporate it by heating and sufficiently generate white fume of sulphuric acid.

- (4) After leaving it cooled as it is, dissolve salts by adding 200 ml of water and 50 ml of hydrochloric acid, transfer the solution into an Erlenmeyer flask (500 ml) and add 10 ml of antimony trichloride solution and 10 g of lead, otherwise add nickel cylinder or wire. Equip the Erlenmeyer flask with a rubber stopper attached with the cap of Attached Fig. 6, add about 50 ml of sodium hydrogencarbonate solution to the cap and reduce the tin by quietly boiling it for about 30 min.
- (5) Remove the heat source of Erlenmeyer flask and leave it cooled as it is for about 5 min. Because the sodium hydrogencarbonate solution in the cap reversely flows into the Erlenmeyer flask, when there is any fear of absorbing air due to the decrease of solution, add the solution thereto. Then, dip the Erlenmeyer flask into water and cool it until it becomes not more than 10°C.
- (6) Remove the rubber stopper together with the cap ⁽⁴⁾ and immediately clean the lower end of rubber stopper and the inner wall of Erlenmeyer flask with the preliminarily prepared washing water ⁽⁵⁾. Thereafter, add 5 ml of starch solution as an indicator, titrate it with potassium iodate standard solution ⁽⁶⁾ and allow the point where the colour of solution turns blue purple to be the end point.

7.2.6 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 : mass of tin equivalent to 1 ml potassium iodate standard solution (g)

V : used amount of potassium iodate standard solution (ml)

W : weighed out amount of sample (g)

Notes (2) The sample shall be weighed out so that the content of tin becomes not less than 20 mg as far as possible.

(3) When the weighed out amount of sample is 2 to 3 g, 25 to 30 ml shall be added.

(4) When nickel is used as a reducing agent, the nickel cylinder or nickel wire is taken out and the rubber stopper and the like together with that shall be washed with water.

(5) For the washing water, air shall be swept off and the water cooled with passing carbon dioxide shall be used.

(6) Instead of potassium iodate standard solution, iodine standard solution may be used.

Remark: When the apparatus of Attached Fig. 7 is used for the reducing apparatus, the operation shall be carried out as follows:

Operate according to the procedure of 7.2.5 (1) to (3) and sufficiently generate white fume of sulphuric acid.

After leaving it cooled as it is, add 200 ml of water and 50 ml of hydrochloric acid and dissolve salts. Thereafter, put it into the Erlenmeyer flask (a) of reducing apparatus, add 10 ml of antimony trichloride solution and 10 g of lead, or nickel cylinder or wire, set a stopper rubber (b) and reduce the tin by boiling it for about 30 min while passing carbon dioxide.

After cooling it to not more than 10°C with running water while passing carbon dioxide, stop the feed of carbon dioxide and close the rubber tubes fit into both ends of carbon dioxide introducing conduit (c) and exhausting conduit (d) with a pinch cock. Take out the glass rod (e), add 5 ml of starch solution as an indicator through the hole (f), further quickly insert the tip of burette into the hole (f) and titrate it with potassium iodate standard solution (6). When the end point comes near, remove the rubber stopper (b), wash the inner wall of Erlenmeyer flask (a), the lower end of rubber stopper (b) and the glass tube with washing water (5), continue to titrate it and allow the point where the colour of solution turns blue purple to be the end point.

7.3 Atomic Absorption Method

7.3.1 Summary The sample is decomposed with the mixed acid of nitric acid and hydrofluoric acid and added with boric acid. Thereafter, its volume is made constant and the absorbancy is measured with an atomic absorption photometer.

7.3.2 Reagents The reagents shall be as follows:

- (1) Boric acid solution (5 w/v %)
- (2) Mixed acid It is prepared by adding 25 ml of hydrofluoric acid to 1 l of nitric acid (2 + 1).
- (3) Copper (It is not less than 99.96 % in purity and equivalent to electrolytic cathode copper specified in JIS H 2121).
- (4) Zinc (It is not less than 99.99 % in purity and equivalent to Extra Class of JIS H 2107).
- (5) Standard tin solution (5 mg Sn/ml) It is prepared by a method wherein 1.250 g of tin (99.90 % min. and equivalent to Class 1 A of JIS H 2108) is weighed out, transferred into a polyethylene beaker (200 ml), added with 10 ml of nitric acid (2 + 1) and 2 ml of hydrofluoric acid and quietly decomposed on water bath by heating. After cooling it to ordinary temperature, it is transferred into a polyethylene measuring flask (⁷) of 250 ml and diluted up to the marked line with water.

7.3.3 Weighing Out Amount of Sample For the sample, 1 g shall be sampled.

7.3.4 Operation The determining operation shall be carried out according to the following procedures:

- (1) Weigh out the sample, transfer it into a polyethylene beaker (100 ml), add 10 ml of mixed acid, leave it as it is at room temperature and decompose it (⁸). After ending the decomposition, add 10 ml of boric acid solution.
- (2) After cooling it to ordinary temperature, transfer it into a measuring flask of 100 ml and dilute it up to the marked line with water.
- (3) Atomize this solution into the flame of air and acetylene and measure the absorbancy near 286.3 nm wavelength using an atomic absorption photometer.

7.3.5 Calculation The amount of tin shall be obtained from the working curve prepared in 7.3.6 and the content of tin in the sample shall be calculated from the following formula:

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

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

W: weighing out amount of sample (g)

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7.3.6 Preparation of Working Curve Weigh out the copper and zinc contained in sample by the almost same rate as that of sample content so that all mass becomes 1 g, make this unit group, weigh out several groups, transfer respective groups into polyethylene beakers (100 ml), add 10 ml of mixed acid, leave it as it is at room temperature and decompose it ⁽⁸⁾. Stepwise add 0 to 10.0 ml of standard tin solution (0 to 0.05 g as tin) and further add 10 ml boric acid. Hereinafter, operate according to the procedures of 7.3.4 (2) and (3), prepare the relationship curve of the absorbancy measured in parallel with sample and the amount of tin and allow it to be the working curve.

Notes ⁽⁷⁾ The polyethylene measuring flask (polypropylene measuring flask may be used) shall be corrected in accordance with 11. (4) of JIS K 0050 to be used.

⁽⁸⁾ When the room temperature and the liquid temperature of mixed acid are low, the decomposition shall be carried out by heating it at not less than 20°C.

Remark: The operation of 7.3.4 (1) may be carried out as follows: Weigh out the sample, transfer it into a polyethylene measuring flask ⁽⁷⁾ of 100 ml, add 10 ml of mixed acid and decompose it by leaving it as it is at room temperature ⁽⁸⁾. After the decomposition has completed, cool it to ordinary temperature and dilute it up to the marked line with water. Hereinafter, operate according to the procedure of 7.3.4 (3) and determine the tin. Allow the working curve in this case to be prepared using this operation.

8. Iron Determination Methods

8.1 Classification of Methods The determination method of iron shall be in accordance with either one of the following:

- (1) Sulphosalicylic acid absorptiometry
- (2) Atomic absorption method (Method A) This method shall apply to the sample of BCuZn-0 and BCuZn-4 not containing tin.
- (3) Atomic absorption method (Method B) This method shall apply to the sample of BCuZn-3 and BCuZn-5 containing tin.

8.2 Sulphosalicylic Acid Absorptiometry

8.2.1 Summary The sample is decomposed with nitric acid, added with aqueous ammonia in excess, copper, zinc, nickel, silver, etc. being dissolved as complex, and at the same time iron and the like are filtered by precipitating them. The precipitates are dissolved with hydrochloric acid and the pH is regulated using aqueous ammonia and nitric acid. Thereafter, it is coloured by adding sulphosalicylic acid and its absorbancy is measured.

8.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 3, 1 + 50)
- (2) Nitric acid (1 + 1, 1 + 2, 1 + 100)

- (3) Aqueous ammonia (1 + 1, 1 + 20)
- (4) Sulphosalicylic acid solution It is prepared by a method wherein after dissolving 10 g of sulphosalicylic acid with about 80 ml of water, the pH is made 2.0 to 2.5 by aqueous ammonia (1 + 1) and it is diluted to 100 ml with water.
- (5) Standard iron solution (100 µg Fe/ml) It is prepared by a method wherein 0.100 g of iron (99.5 % min.) is decomposed with 15 ml of hydrochloric acid (1 + 1) by heating, and carefully added little by little with 2 ml of hydrogen peroxide water (1 + 1). Thereafter, excessive hydrogen peroxide is decomposed by boiling. After cooling it, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water.
- (6) Thymol Blue test paper After dipping a good filter paper into the ethanol solution of Thymol Blue (0.1 w/v %), it is dried and used.

8.2.3 Weighing Out Amount of Sample For the sample, 1 g shall be weighed out ⁽⁹⁾.

8.2.4 Operation The determining operation shall be carried out according to the following procedures:

- (1) Weigh out the sample, transfer it into a beaker (300 ml), cover it with a watch glass, add 15 ml of nitric acid (1 + 1) and decompose it by slowly heating it. Wash the lower surface of watch glass and the inner wall of beaker with water and sweep out nitrogen oxide by successively heating it ⁽¹⁰⁾. Thereafter, dilute it to about 60 ml with water.
- (2) Make it strong ammoniac solution by adding aqueous ammonia (1 + 1) until the once generated precipitates of copper, zinc, etc. are dissolved, and boil it for 1 to 2 min by heating. After leaving it quietly as it is for a while, filter it using a filter paper (Class 5 B) and sufficiently wash it with warm aqueous ammonia (1 + 20).
- (3) Set the original beaker under the funnel and dissolve the precipitate by pouring as small amount of warm hydrochloric acid (1 + 3) as possible from over the filter paper. Thereafter, wash it with warm hydrochloric acid (1 + 50) several times ⁽¹¹⁾.
- (4) Make the amount of solution about 60 ml by adding water to the filtrate and washings, and make it almost neutralized by adding aqueous ammonia (1 + 1). Thereafter, make the pH 2.0 to 2.5 ⁽¹²⁾ using nitric acid (1 + 2) and aqueous ammonia (1 + 1).
- (5) After cooling it to ordinary temperature, transfer it into a measuring flask of 100 ml, add 3 ml of sulphosalicylic acid solution, dilute it up to the marked line with water and make it coloured by sufficiently mixing with shaking.
- (6) Take a portion of this solution into the absorption cell of photometer and measure the absorbancy near 545 nm wavelength.

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8.2.5 Calculation The amount of iron shall be obtained from the working curve prepared in 8.2.6 and the content of iron in sample shall be calculated from the following formula:

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

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

W: weighed out amount of sample (g)

8.2.6 Preparation of Working Curve Stepwise take 0 to 20.0 ml of standard iron solution (0 to 2.0 mg as iron) into several beakers (200 ml), operate according to the procedures of 8.2.4 (4) to (6), prepare the relation curve between the obtained absorbancy and the amount of iron and allow it to be the working curve.

Notes ⁽⁹⁾ The sampling amount shall be regulated so that the content of iron becomes not more than 2 mg as far as possible.

⁽¹⁰⁾ When precipitates of silicon, tin, etc. are observed in that case, after leaving it at warm place for about 1 h, it is immediately filtered with the filter paper (Class 5 B) added with a small amount of filter paper pulp, and sufficiently washed using warm nitric acid (1 + 100). Thereafter, it is made about 60 ml by evaporating the filtrate and washings by heating.

⁽¹¹⁾ When the precipitate of silver chloride is observed in the filtrate, it is filtered by using the original filter paper and sufficiently washed with warm hydrochloric acid (1 + 50). Thereafter, it is made about 60 ml by evaporating the filtrate and washings by heating.

Further, when the filtrate presents the colouration of copper ion, it is reprecipitated by carrying out the operation of 8.2.4 (2), and filtered and washed.

⁽¹²⁾ Thymol Blue test paper can be used. In that case, the pH shall be made to the order where the colour of test paper turns light red.

8.3 Atomic Absorption Method (Method A)

8.3.1 Summary The sample is decomposed with nitric acid and made of a fixed volume. Thereafter, the absorbancy shall be measured using an atomic absorption photometer.

8.3.2 Reagents The reagents shall be as follows:

- (1) Nitric acid (1 + 1)
- (2) Copper [in accordance with 7.3.2 (3)]
- (3) Zinc [in accordance with 7.3.2 (4)]