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JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of Palladium Brazing Filler Metals

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Methods for Chemical Analysis of
Palladium Brazing Filler Metals

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1. Scope

This Japanese Industrial Standard specifies the methods for determination of palladium, silver, copper, manganese, nickel, and lead specified in JIS Z 3267.

2. General Matters

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

3. Sampling and Treating of Analytical Sample3.1 Sampling Method

3.1.1 A sample shall be taken in accordance with JIS Z 3900.

3.1.2 The sample shall be made to represent mean quality, and cares shall be especially taken for segregation, contamination, etc. therefore.

3.2 Weighing of Sample

3.2.1 When a sample is weighed out, cares shall be taken for the sample to represent average composition by sufficiently mixing by stirring and further, no mixing with foreign matter shall be confirmed.

3.2.2 The sample shall be weighed out to the nearest 0.1 mg by using a chemical balance.

4. Arrangement of Analytical Values

4.1 Number of Times of Analyses The same sample shall, as a rule, be analyzed not less than two times.

4.2 Blank Test When a sample is analyzed, a blank test shall be performed through all operations and the content shall be corrected.

Applicable Standards: See page 18.

4.3 Indication of Analytical Value An analytical value shall be expressed by mass percentage. Palladium, silver, copper, manganese and nickel shall be rounded off to one place of decimal, and lead to two places of decimals in accordance with JIS Z 8401 after averaging not less than two analytical values.

5. Classification by Analytical Sample and Procedures of Analysis

Since the methods for chemical analysis of palladium brazing filler metals are in accordance with a systematic analytical method, the analytical sample of palladium brazing filler metals shall be classified into four types, and the sequence of analysis thereof shall be as given in the following table.

Table. Classification by Analytical Sample and Sequence of Analysis

Classification by analytical sample		Class of palladium brazing filler metals	Sequence of analysis
Type	Component system		
(1)	Pd-Ag-Cu system	B Pd-1,2,4,6	Silver determination - filtrate (A) - (6.2.4) palladium determination - filtrate (B) - (7.2.4.1) copper determination (8.2.4)
	Pd-Ag system	B Pd-7	Silver determination - filtrate (A) - (6.2.4) palladium determination (7.2.4.1)
(2)	Pd-Ag-Mn system	B Pd-9,10	Silver determination - filtrate (A) - (6.2.4) palladium determination - filtrate (B) - (7.2.4.1) manganese determination (9.2.3.1 or 9.3.3)
(3)	Pd-Cu-Mn-Ni system	B Pd-12	Palladium determination - filtrate (B) - (7.2.4.2) copper determination - electrolytic residual (8.2.4) liquid - manganese determination - sample (9.2.3.2) solution (D) - nickel determination (10.2.3.2)
(4)	Pd-Mn-Ni system	B Pd-11	Palladium determination - filtrate (B) - (7.2.4.2) manganese determination - sample solution (C) - (9.2.3.1) nickel determination (10.2.3.1)
	Pd-Ni system	B Pd-14	Palladium determination - filtrate (B) - (7.2.4.2) nickel determination (10.2.3.1)

6. Method for Determination of Silver

6.1 Determination Method The method for determination of silver shall be in accordance with silver chloride gravimetric method. This method applies to types (1) and (2).

6.2 Silver Chloride Gravimetric Method

6.2.1 Summary Decompose a sample with nitric acid, and make palladium the ammine complex by adding aqueous ammonia in excess. Thereafter, acidify slightly by using acetic acid and deposit silver chloride by adding hydrochloric acid. Filter the generated silver chloride by using a sintered-glass filter. After drying, weigh the mass thereof [Take the filtrate and washings as a filtrate (A), and use it for determination of palladium.].

6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 9)
- (2) Nitric acid (1 + 1, 1 + 100)
- (3) Aqueous ammonia (1 + 1)
- (4) Acetic acid

6.2.3 Weighing-out Amount of Sample 0.5 g of a sample shall be weighed out.

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

- (1) Decomposition of Sample Weigh out a sample, transfer to a conical beaker (300 ml), add 20 ml of nitric acid (1 + 1), cover with a watch glass, and decompose by heating.
- (2) Generation of Silver Chloride Precipitate Wash the bottom face of a watch glass and the inner wall of a beaker, add water to the amount of liquid about 150 ml, and slightly alkalify by adding aqueous ammonia (1 + 1). Thereafter, add acetic acid (1 + 1) until the solution is slightly acidified. Add little by little hydrochloric acid (1 + 9) with mixing by stirring the solution, and generate precipitates of silver hydrochloride. After no precipitate becomes to be generated, further add 1 ml thereof in excess, sufficiently stir to mix, wash the bottom face of the watch glass and the inner wall of the beaker with water, and gently boil by heating for about 5 min.
- (3) Filtration and Washing of Precipitates After standing as it is in a dark place for one night, filter by using a sintered-glass filter (1 G 4) which has been preliminarily made constant mass, and sufficiently wash initially with nitric acid (1 + 100) and then with cold water [Take the filtrate and washings as filtrate (A) and use it for determination of palladium of types (1) and (2).].

- (4) Drying and Weighing of Precipitate Dry the precipitate together with the sintered-glass filter in an air bath at about 130°C for about 1 h, and let stand to cool in a desiccator for about 1 h. Thereafter, weigh the mass, and repeat this operation to constant mass.

6.2.5 Blank Test Use only reagents, and operate in parallel to a sample in accordance with the procedures on and after 6.2.4 (1). Use the filtrate and washings for the blank test of 7.2.5.

6.2.6 Calculation Calculate the content of silver in a sample from the following formula:

$$\text{Silver wt\%} = \frac{(m_1 - m_2) \times 0.7526}{m_0} \times 100$$

where m_1 : mass of silver chloride obtained in 6.2.4 (g)

m_2 : mass obtained in 6.2.5 (g)

m_0 : weighing out amount of sample (g)

7. Method for Determination of Palladium

7.1 Determination Method The method for determination of palladium shall be in accordance with dimethylglyoxime palladium gravimetric method. This method applies to the types of (1) and (2) and the types of (3) and (4).

7.2 Palladium Dimethylglyoxime Gravimetric Method

7.2.1 Summary For the types of (1) and (2), add hydrochloric acid to the filtrate (A) obtained in 6.2.1 and boil. Thereafter, dilute with water. For the types of (3) and (4), decompose a sample with aqua regia, evaporate by heating to a sirupy state, add hydrochloric acid, and remove nitric acid by repeating this operation. Then, add hydrochloric acid, and dilute with water.

Add dimethylglyoxime to the solution obtained by either of those operations, precipitate palladium complex, filter by using a sintered-glass filter, and dry. Thereafter, weigh the mass [Take the filtrate washings as filtrate (B), and use for determination of copper or manganese, and nickel.].

7.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1)
- (3) Aqua regia (3 parts hydrochloric acid and one part nitric acid)
- (4) Dimethylglyoxime solution Dissolve 1.0 g of dimethylglyoxime in 100 ml of ethanol (95 vol %).
- (5) Ethanol (95 vol %)

7.2.3 Weighing-out Amount of Sample For the types of (1) and (2), use all the amount of filtrate (A), and for the types of (3) and (4), weigh out 0.5 g of a sample.

7.2.4 Operation

7.2.4.1 For Types of (1) and (2) For the types of (1) and (2), perform the determining operation in accordance with the following procedures:

- (1) **Hydrochloric Acid Treatment** Add 10 ml of hydrochloric acid to the solution (A) obtained in 6.2.4 (3), and gently boil by heating for 5 to 10 min. Thereafter, dilute to about 700 ml with water.
- (2) **Generation of Palladium Dimethylglyoxime Precipitate** Add dimethylglyoxime solution [7.2.2 (4)] by a rate of 3.0 ml per 0.01 g in palladium content with mixing the solution ⁽¹⁾. After heating at 70 to 80°C for about 20 min, allow to stand in a warm place for about 2 h.
- (3) **Filtration and Washing of Precipitate** Filter the precipitate by using a sintered-glass filter (2 G 4) which has been preliminarily made constant mass, wash sufficiently with warm water [Take the filtrate and washings as filtrate (B), and use for determination of copper of type (1) and manganese of type (2).], and further wash once with ethanol.
- (4) **Drying and Weighing of Precipitate** Dry the precipitate together with the sintered-glass filter in an air bath at about 110°C for about 1 h, and allow to stand to cool in a desiccator for about 1 h. Thereafter, weigh the mass, and repeat this operation to constant mass ⁽²⁾.

Notes ⁽¹⁾ Though the required calculated amount of dimethylglyoxime solution is 2.2 ml per 0.01 g of palladium, it is in excess required for complete precipitation.

⁽²⁾ Since palladium dimethylglyoxime is slightly dissolved in water, when it is required to enhance precision, perform a parallel test by using a known synthetic sample, and correct a measured value of palladium.

7.2.4.2 For Types of (3) and (4) Perform the determining operation for the types of (3) and (4) in accordance with the following procedures:

- (1) **Decomposition of Sample** Weigh out a sample, transfer to a beaker (200 ml), add 20 ml of aqua regia, cover with a watch glass, and decompose by heating. Thereafter, gently evaporate by heating to a sirupy state.
- (2) **Denitration** Add 10 ml of hydrochloric acid, and evaporate by heating again to a sirupy state. Repeat this operation 3 or 4 times.