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JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of Nickel Brazing Filler Metals

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Methods for Chemical Analysis of
Nickel Brazing Filler MetalsZ 3905-1976
(Reaffirmed: 1980)1. Scope

This Japanese Industrial Standard specifies methods for chemical analysis of composition of chrome, boron, silicon, iron, carbon and phosphorus specified in JIS Z 3265.

2. General Matters

General matters common to chemical analysis shall conform to JIS K 0050.

3. Method of Sampling and Treatment of Analytical Samples3.1 Sampling and Preparation of Analytical Samples

3.1.1 In selecting and preparing analytical samples, endeavor to make them represent the average quality of the entire samples and take care to preclude segregation and contamination.

3.1.2 Sampling and preparation of samples shall be made in conformity with 5. of JIS H 0321 and 2.2 of JIS H 0301. Take 2 or more cast samples from each melt (or one sample if the melt is exceptionally small). Take approx. 100 g of melt sample, whittle out of it twice the amount of chips needed for analysis and, by mixing it well, make it the final analytical sample.

3.1.3 The method of shaving cast samples shall be as follows:

- (1) The drill and other tools to be used shall be washed clean with ethanol, etc.
- (2) Remove traces of oil and other adherents from the surface of cast sample.
- (3) Drill the central portion of the sample to penetrate or the vicinity of either end of the sample from both faces as far as the core, or otherwise use some suitable methods to obtain shavings.
- (4) Make the size of the sample for shaving 5 mm or under so as not to make the chips too thick. In case the chips come out in the form of long strips, cut the sample with clean scissors to the length not exceeding 5 mm.

Care shall be taken then to prevent drill scrapings from mingling with the chips, and no water shall be poured for the purpose of cooling.

3.1.4 When sampling and preparation of analytical samples cannot conform to the above prescription, the matter shall be decided by agreement between the parties concerned.

Applicable Standards: See page 18.

3.1.5 As for the method of collection and preparation of wire-form or tape-form materials containing bonding substances, the matter shall be decided by agreement between the parties concerned.

3.2 Weighing of Analytical Sample

3.2.1 When analytical sample is to be weighed out, care shall be taken to mix the sample so adequately that the sample represents the average composition and to ascertain that no impurities are contained.

3.2.2 In weighing analytical samples, use a chemical balance and read to the nearest 0.1 mg.

4. Notes on Analytical Operation

4.1 Analysis shall, as a rule, be carried out two or more times on the same sample.

4.2 Before performing analysis, blank tests shall be carried out for the entire operations to make corrections on percentage of content.

5. Rounding off Analyzed Values

The analyzed value shall be represented by the average of values of two or more analyses rounded-off according to JIS Z 8401 to the unit specified in JIS Z 3265.

6. Determination of Chromium

6.1 Division of Methods The method for determination of chromium shall be the ammonium persulfate oxidation titrimetric method.

6.2 Ammonium Persulfate Titrimetric Method

6.2.1 Gist of Procedure Dissolve the sample with aqua regia, add sulfuric acid, phosphoric acid and hydrofluoric acid and evaporate to generate white fume of sulfuric acid, and after cooling, dissolve soluble salts with water, add silver nitrate as catalizer, and further add ammonium persulfate to oxidize chromium to change into dichromate. Decompose permanganate concurrently produced by adding hydrochloric acid, and next add manganese sulfate. After cooling, reduce the dichromate by adding an excess amount of ferrous ammonium sulfate standard solution and back-titrate the excess ferrous ammonium sulfate with potassium permanganate standard solution.

6.2.2 Reagents The reagents shall be as given in the following:

- (1) Hydrochloric acid (1 + 3)
- (2) Perchloric acid
- (3) Hydrofluoric acid
- (4) Sulfuric acid (1 + 1, 1 + 10, 1 + 50)
- (5) Phosphoric acid

- (6) Aqua regia (chloric acid 3, nitric acid 1)
- (7) Silver nitrate solution (0.5 w/v %)
- (8) Ammonium persulfate solution (20 w/v %)
- (9) Potassium pyrosulfate
- (10) Potassium permanganate solution (2 w/v %)
- (11) Manganous sulfate solution Dissolve 10 g MnSO_4 (4 to 6 hydrate) in 100 ml of water.
- (12) N/10 ferrous ammonium sulfate standard solution Dissolve 40 g ferrous ammonium sulfate crystal $[\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ in approx. 300 ml of water, and add 60 ml sulfuric acid (1 + 1), transfer the solution into a 1000 ml volumetric flask, dilute with water to the marked line. The standardization of this solution shall be checked for each use. If the N/10 is not exactly N/10, calculate the factor and correct the volume of ferrous ammonium sulfate standard solution used for the chromium equivalent as follows: Take accurately 25 ml ferrous ammonium sulfate standard solution in a beaker, add 25 ml of water and 0.25 ml of o-phenanthroline solution of (14), and next titrate with potassium permanganate standard solution of (13) until light green colour appears, and calculate the factor of ferrous ammonium sulfate standard solution for correcting potassium permanganate solution from the following formula:

$$F_1 = \frac{V_1 \times F_2}{V_2}$$

where F_1 : factor of N/10 ferrous ammonium sulfate standard solution

V_1 : volume of N/10 potassium permanganate standard solution (ml)

V_2 : volume of N/10 aliquot (25 ml) of ferrous ammonium sulfate solution

F_2 : factor of N/10 potassium permanganate standard solution

- (13) N/10 potassium permanganate standard solution (3.161 g KMnO_4/l)
The preparation, standardization and preservation method of this solution shall conform to 2. (22) of JIS K 8006.
- (14) o-phenanthroline solution Weigh out 3.0 g of o-phenanthroline (1-hydrate) and dissolve it in 200 ml of water containing 2.0 g of ferrous ammonium sulfate standard solution (6-hydrate).

6.2.3 Weighing-Out Amount of Sample Weigh out 0.2 g of sample.

6.2.4 Procedure The determining procedure shall be taken in the following order:

- (1) Transfer the sample to a beaker (500 ml) and cover with a watch glass, add 20 ml of aqua regia, heat gently until decomposition ceases, add 20 ml of sulfuric acid (1 + 1), 10 ml of phosphoric acid and 1 to 2 ml of hydrofluoric acid⁽¹⁾ and evaporate to generate white fume of sulfuric acid, letting it continue fuming for 3 to 4 min⁽²⁾.
- (2) After cooled, add approximately 150 ml of warm water, and heat until salts are solved⁽³⁾, add 10 ml of silver nitrate solution and 20 to 25 ml of ammonium persulfate solution, boil the solution for 3 to 5 min to oxidize chromium into dichromic acid. If manganese content is very small and the colour of permanganate does not develop, add the potassium permanganate solution to produce crimson colouring. Boil the solution until ammonium persulfate is decomposed, add approximately 5 ml of hydrochloric acid (1 + 3) to decompose permanganate. When the permanganic colour is not destroyed by boiling, or if a precipitate of manganese dioxide remains, add 2 to 3 ml more of hydrochloric acid (1 + 3) and boil to the complete dissolution. Next, add 5 ml of manganous sulfate solution⁽⁴⁾ and boil for 2 to 3 min and expel completely the chlorine gas produced.
- (3) Cool the beaker to below room temperature by using cold water, dilute the solution with water to approximately 300 ml, and add 0.25 ml of *o*-phenanthroline solution and a measured volume of N/10 ferrous ammonium sulfate solution until the sample solution begins to show brownish colour, then approximately 5 ml in excess, immediately titrate with N/10 potassium permanganate standard solution, and make it the end point when the brownish colour turns light green.

6.2.5 Calculation The chromium content shall be calculated from the following formula:

$$\text{Chromium (\%)} = \frac{(V_1 - V_2) \times 0.001734}{W} \times 100$$

where V_1 : volume of N/10 ferrous ammonium sulfate standard solution used (ml)

V_2 : volume of N/10 potassium permanganate standard solution required for titration (ml)

W : weighed-out mass of sample (g)

Notes ⁽¹⁾ In the case of sample containing no silicon, it is not necessary to add hydrofluoric acid.

⁽²⁾ Caution shall be exercised against too long a duration of a thick white fume of sulfuric acid because it may cause difficulties to the next dissolution.

- (³) When the residue of sample is coloured owing to imperfect decomposition, filter the precipitate using filter paper (Class 5 B) with a small quantity of filter paper pulp, and wash with sulfuric acid (1 + 50). Preserve the filtrate and washing water as main sample solution. Transfer the paper and residue to a platinum crucible, and after drying, ignite to incinerate at high temperature to the ash. Add approximately 10 times the volume of potassium pyrosulfate and melt by heating. After cooling, dissolve the salt with a small quantity of warm water and sulfuric acid (1 + 10), and then join it with the main sample solution.
- (⁴) According to the direct titration method prescribed in the following Remark, the addition of manganous sulfate solution may be omitted.

Remark: The titration may be conducted by direct titration method, and the procedure shall be as follows:

After performing the procedure shown in 6.2.4 (1) and (2), cool the solution to below room temperature with cold water. Dilute the solution with water to approximately 300 ml, add 0.25 ml of *o*-phenanthroline solution as indicator and titrate with N/10 ferrous ammonium sulfate standard solution, taking the time the colour of solution turns brown as the end point.

In this case, the chromium content shall be calculated from the following formula:

$$\text{Chromium (\%)} = \frac{V \times 0.001734}{W} \times 100$$

where V : volume of N/10 ferrous ammonium sulfate standard solution used (ml)

W : weighed-out mass of sample (g)

7. Determination of Boron

7.1 Division of Methods Determination of boron shall be conducted by the mercury cathode electrolytic separation-sodium hydroxide titration method.

7.2 Mercury Cathode Electrolytic Titration Method

7.2.1 Gist of Procedure Dissolve the sample by heating with addition of weak solution of sulfuric acid, and oxidize iron and other compositions by adding hydrogen peroxide. Further, oxidize with potassium permanganate, and then decompose the generated manganese dioxide by addition of hydrogen dioxide solution. After cooling, separate nickel, chromium and iron by the method of mercury cathode electrolysis. Boil the electrolyte to expel carbon dioxide, then adjust the pH value of the solution, add mannitol and titrate with sodium hydroxide standard solution, and allow the time when it returns to the original pH to be the end point.