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JAPANESE INDUSTRIAL STANDARD

Methods of Chemical Analysis
for Nickel Chromium
Electric Heating Material

JIS

H 1412-1976
(Reaffirmed: 1984)



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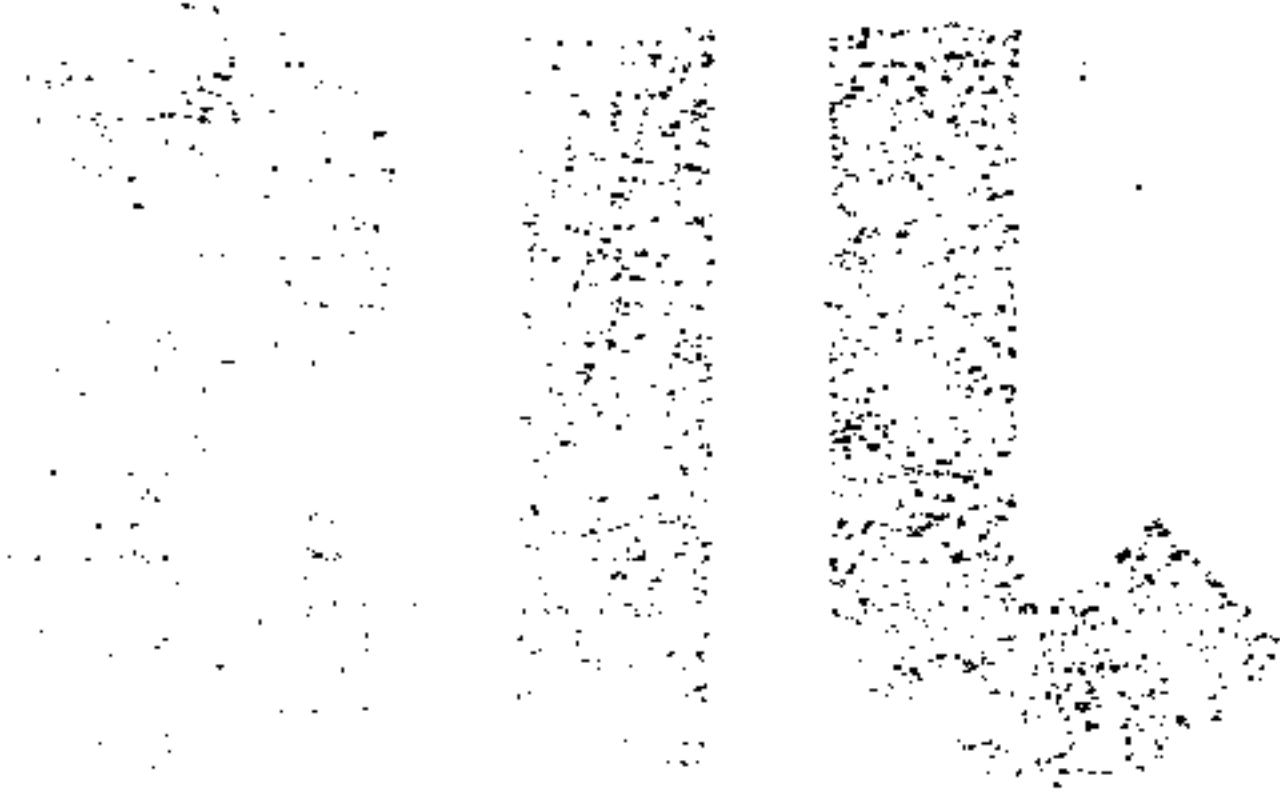
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J I S

Methods of Chemical Analysis
for Nickel Chromium
Electric Heating Material

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1. Scope

This Japanese Industrial Standard specifies the methods of chemical analysis for chemical composition (nickel, chromium, manganese, silicon, iron, cobalt and carbon) of nickel chromium electric heating wires and ribbons specified in JIS C 2520.

2. General Matters

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

3. Sampling and Treating of Sample

3.1 The sampling shall be in accordance with 2.3 of JIS H 0321.

3.2 When a cast sample is taken, to obtain the sample representing its average material quality, not less than two samples per one fusion (one sample in the case of especially small quantity of one fusion amount) shall be taken. The cast sample shall be carefully especially free from segregation to obtain the same material quality as that of product as completely as possible.

Applicable Standards:

JIS C 2520-Electric Heating Wires and Ribbons

JIS H 0321-General Rules for Inspection of Non-ferrous Metal
Materials

JIS H 2107-Zinc Metal

JIS K 0050-General Rules for Chemical Analysis

JIS K 0115-General Rules for Absorptiometric Analysis

JIS K 8005-Primary Standard Substances for Volumetric
Determination

JIS K 8006-Fundamental Articles on Volumetric Determinations

JIS Z 8401-Rules for Rounding off of Numerical Values

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3.3 The chipping of sample shall be as follows:

- (1) In the case where there are adhering substances or the like on the surface of sample, they shall be removed by using an emery paper or the like and the surface shall be cleaned.
- (2) Drills and other tools shall be cleaned by using alcohol or the like.
- (3) When a sample is chipped out from a cast sample, the sample shall be pierced by drilling vertically from one side surface of the central part and parts near both ends, or otherwise the sample shall be drilled from both surfaces until the drilling reaches its core part at least. Otherwise, the chipping of sample shall be in accordance with other suitable methods.
- (4) When a sample is chipped out from product samples such as wires, ribbons, plates, etc., they shall be chipped out in a size suitable of the analytical operation by using a drill or other suitable tools.
- (5) Because the surface of chipped piece is occasionally oxidized by the generation of heat when drilled, drilling is carried out by giving the pressure and number of revolution of a degree not to oxidize it to the drill. In that case, it is prohibited to use oils or other lubricants, or otherwise to pour water or the like for cooling. Further, care shall be taken so that chipped pieces are not mixed with the wearing powder of drill.
- (6) The size of chipped piece shall not be too thick and its length shall be not more than about 5 mm.

3.4 Treating of Sample

- (1) All the chipped out samples (generally, not lower than 50 g), are collected, from which the mixed iron powder or the like are carefully removed by using a strong magnet, and at least sufficiently mixed to be the sample for chemical analysis.
- (2) When the sample is difficult to be sampled in accordance with above-mentioned specifications, it may be otherwise determined according to the agreement between the purchaser and the manufacturer.
- (3) The sample shall be put into a desiccator and let to stand for not less than 1 h. Thereafter, it shall be weighed out.

3.5 Weighing of Sample The weighing of sample shall be as follows:

- (1) When a sample is weighed out, care shall be taken so that average chemical composition may be represented by sufficiently mixing the sample.

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- (2) For weighing out of sample, a chemical balance shall, as a rule, be used and an amount near a specified amount shall be weighed out to a necessary place referring to the indicated number of places of analytical value.

4. Expression of Analytical Value and Caution on Operation

4.1 Expression of Analytical Value The analytical value shall be expressed as follows:

- (1) The analytical value is expressed by percentage.
- (2) The nickel of NCH 1 shall be calculated as the remainder for which total sum of the calculated values of chemical components excepting nickel specified in JIS C 2520 is subtracted from 100 and rounded off to integral value in accordance with JIS Z 8401 to be indicated.
- (3) Each chemical component excepting (2) shall be calculated to the places next to the place specified in JIS C 2520 and indicated by rounding off it to the specified place in accordance with JIS Z 8401.

4.2 Cautions on Analytical Operation The cautions on analytical operation shall be as follows:

- (1) The same sample shall be analyzed not less than two times and the results shall be confirmed.
- (2) When analyzed, a blank test shall be carried out over all operations and the measured value shall be corrected.

5. Method for Determination of Nickel

5.1 Classification of Methods The method for determination of nickel shall be as follows:

- (1) Gravimetric method
- (2) Titrimetric method

5.2 Gravimetric Method

5.2.1 Summary A sample is decomposed with perchloric acid by heating and silicic acid is filtered (the filtrate in the method for determination of silicon may be used). A fixed amount is separately taken therefrom, added with tartaric acid and ammonium chloride, made alkaline with aqueous ammonia and nickel is precipitated by adding dimethylglyoxime. Thereafter, the precipitate is filtered, washed and its mass is measured.

5.2.2 Reagents The reagents shall be as follows:

- (1) Perchloric acid

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- (2) Sulfuric acid (1+10)
- (3) Aqueous ammonia
- (4) Ammonium chloride solution (25 w/v %)
- (5) Sodium pyrosulfate
- (6) Tartaric acid solution (25 w/v %)
- (7) Dimethylglyoxime solution 1.0 g of dimethylglyoxime is dissolved in 100 ml ethyl alcohol, or otherwise dissolved with 100 ml of sodium hydroxide solution (1 w/v %).

5.2.3 Weighing out Amount of Sample 1.0 g of the sample shall be weighed out.

5.2.4 Operation The determination operation shall be carried out in accordance with the following procedures:

- (1) Weigh out a sample, transfer it into an electrolytic beaker (300 ml), cover it with a watch glass, add 20 ml of perchloric acid, decompose it by heating, oxidize chromium by successively heating it ⁽¹⁾, continue to heat it until red chromic acid appears and further heat it for 15 to 20 min.
- (2) After cooling, add about 100 ml of warm water, dissolve salts by agitation, immediately filter insoluble matters by using a filter paper (Class 5 B) and sufficiently wash it with warm water ⁽²⁾. Transfer the filtrate into a measuring flask of 500 ml after cooling, and dilute it up to the marked line with water. Accurately take separately a fixed amount ⁽³⁾⁽⁴⁾ of this solution into a beaker (500 ml) and dilute it to about 150 ml with water.
- (3) Add 10 ml of tartaric acid solution and 20 ml of ammonium chloride solution thereto, make it weak alkaline by adding aqueous ammonia and dilute the amount of solution to about 250 ml by adding warm water.
- (4) Heat this solution at about 90°C, add dimethylglyoxime solution at a rate of 7 ml per 10 ml of anticipated content of nickel, precipitate nickel, further add 5 ml in excess and sufficiently mix them. Thereafter, let it stand for about 10 min and precipitate nickel.
- (5) Filter the precipitate by using the sintered-glass filter (Type G 3) the mass of which is preliminarily made constant weight and sufficiently wash it with warm water. Thereafter, dry it at 120 to 130°C for about 1 h, let it stand to cool in a desiccator to room temperature, weigh its mass and repeat this operation until it becomes constant weight.

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5.2.5 Calculation The content of nickel in sample shall be calculated from the following formula:

$$\text{Nickel (\%)} = \frac{w \times 0.2032}{W \times B} \times 100$$

where,

w : mass of nickel dimethylglyoxime (g)

W : weighed amount of sample (g)

B : aliquot ratio of sample solution

- Notes (1) The sample is heated at a liquid temperature of about 200°C with a hot plate or the like.
- (2) For the sample decomposed incompletely with acid, insoluble residue together with the filter paper are transferred into a platinum crucible and the filter paper is incinerated by ignition. Thereafter, it is mixed with a suitable amount of sodium pyrosulfate and fused. After standing to cool, it is dissolved in 10 ml of sulfuric acid (1+10), joined to the main solution and hereinafter treated according to the body as appropriate.
- (3) 50 ml in the case of gravimetric method and 25 ml in the case of titrimetric method are accurately taken separately.
- (4) The filtrate in the method for determination of silicon may be used. In that case, the equivalent amount of 0.1 g for gravimetric method and that of 0.05 g for titrimetric method are separately taken.

5.3 Titrimetric method

5.3.1 Summary A sample is decomposed with perchloric acid by heating and silicic acid is filtered (the filtrate in the method for determination of silicon may be used). A fixed amount is separately taken therefrom, added with tartaric acid and ammonium chloride, made alkaline with aqueous ammonia and nickel is precipitated by adding dimethylglyoxime. Thereafter, the precipitate is filtered and heated. It is added with EDTA and EBT and titrated with zinc standard solution.

5.3.2 Reagents The reagents shall be as follows:

- (1) 5.2.2(1) to (7) are used.
- (2) Hydrochloric acid (1+1, 1+10)
- (3) Aqueous ammonia (1+1)

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- (4) M/50 zinc standard solution 1,308 g of metallic zinc (not less than 99.99 %, equivalent metal of extra grade of JIS H 2107) is weighed out, decomposed with as little quantity of hydrochloric acid (1+1) as possible by heating and cooled. Thereafter, it is transferred into a measuring flask of 1000 ml and diluted up to the marked line with water.
- (5) M/50 disodium ethylenediaminetetraacetate (EDTA) standard solution 7.45 g of disodium ethylenediaminetetraacetate (2 hydrate) is dissolved with water and accurately diluted to 1 l. The standardization of titer shall be carried out as follows.

50 ml of M/50 EDTA standard solution is separately taken, added with 10 ml of ammonium chloride solution (25 w/v %) and 0.1 ml of EBT indicator and the amount of solution is diluted to about 100 ml with water. Thereafter, it is dropped with aqueous ammonia (1+1) until the solution turns blue, titrated with M/50 of zinc standard solution and the point where the blue of solution turns reddish purple is made to be the end point. The titer of M/50 EDTA standard solution shall be calculated from the following formula:

$$F = \frac{V}{50}$$

where,

F: titer of M/50 EDTA standard solution

V: used amount of M/50 zinc standard solution (ml)

- (6) Eriochrome Black T (EBT) indicator 0.5 g of Eriochrome Black T and 4.5 g of hydroxylamine hydrochloride are dissolved with ethyl alcohol and diluted to 100 ml. This indicator can be used for about 6 months.

5.3.3 Weighing out Amount of Sample 1 g of the sample shall be weighed out.

5.3.4 Operation The determination operation shall be carried out in accordance with the following procedures:

- (1) Operate the sample in accordance with 5.2.4 (1) to (4).
- (2) Filter the precipitate by using a filter paper (Class 5 A) and sufficiently wash it with warm water. Wash down the precipitate on the filter paper into the original beaker by pouring warm water and 10 ml of hot hydrochloric acid (1+1), wash the filter paper several times with warm water and warm hydrochloric acid (1+10) and dissolve the precipitate by quietly heating it.

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- (3) After accurately adding M/50 EDTA standard solution at a rate of 10 ml per 10 mg of anticipated content of nickel to this solution, further add its 5 ml in excess, mix them 2 to 3 times by shaking, add 0.1 ml of EBT as indicator, drop aqueous ammonia (1+1) until the solution turns blue ⁽¹⁾(²), immediately titrate it with M/50 zinc standard solution and allow the point where the solution turns reddish purple to be the end point.

5.3.5 Calculation The content of nickel in sample shall be calculated from the following formula:

$$\text{Nickel (\%)} = \frac{(V_1 \times F - V_2) \times 0.001174}{W \times B} \times 100$$

where,

V_1 : used amount of M/50 EDTA standard solution (ml)

F : titer of M/50 EDTA standard solution

V_2 : used amount of M/50 zinc standard solution (ml)

W : weighed-out amount of sample (g)

B : aliquot ratio of sample solution

Notes ⁽¹⁾ pH in that case shall be necessarily regulated at about 8.0.

⁽²⁾ The temperature of solution in that case should be necessarily kept at about 30 to 40°C.

6. Method for Determination of Chromium

6.1 Classification of Methods The method for determination of chromium shall be in accordance with the titrimetric method of ammonium persulfate oxidation.

6.2 Ammonium Persulfate Oxidation Titrimetric Method

6.2.1 Summary A sample is decomposed with aqua regia, added with sulfuric acid and phosphoric acid, evaporated by heating and white fume of sulfuric acid is generated. After standing to cool, salts are dissolved with water, added with silver nitrate as catalyst and chromium is oxidized by further adding ammonium persulfate.

Permanganic acid is decomposed by adding hydrochloric acid thereto, and then it is added with manganese sulfate. After cooling, chromic acid is reduced by adding ammonium ferrous sulfate standard solution slightly in excess and excess ammonium ferrous sulfate is titrated with potassium permanganate standard solution.

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6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1+3)
- (2) Perchloric acid
- (3) Sulfuric acid (1+1)
- (4) Phosphoric acid
- (5) Aqua regia Hydrochloric acid and nitric acid are mixed by a rate of 3 to 1.
- (6) Mixed acid (water 13, sulfuric acid 2 and nitric acid 5)
- (7) Silver nitrate solution (0.5 w/v %)
- (8) Potassium permanganate solution (2 w/v %)
- (9) Manganese sulfate solution 10 g of manganese sulfate (4 to 6 hydrate) is dissolved with 100 ml of water.
- (10) N/10 ammonium ferrous sulfate standard solution 40 g of crystalline ammonium ferrous sulfate (6 hydrate) is dissolved in about 300 ml water, added with 60 ml sulfuric acid (1+1), transferred into a measuring flask of 1000 ml and diluted up to the marked line with water.

The titer of this solution shall be standardized with N/10 potassium permanganate standard solution at each use. If it is not accurately N/10, the titer to N/10 is obtained and the used amount of ammonium ferrous sulfate standard solution should be necessarily corrected when the amount of chromium is calculated, that is, 25 ml of ammonium ferrous sulfate standard solution prepared herein is accurately taken, added with 25 ml of water and 0.25 ml of o-phenanthroline solution, titrated with potassium permanganate standard solution prepared in the following (11) until it turns light green and the titer of ammonium ferrous sulfate standard solution shall be obtained from the following formula:

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

where,

F_1 : titer of ferrous ammonium sulfate standard solution

V_1 : used amount of potassium permanganate standard solution

V_2 : aliquot amount of ferrous ammonium sulfate standard solution (25 ml)

F_2 : titer of potassium permanganate standard solution

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- (11) N/10 potassium permanganate standard solution 3.16 g of potassium permanganate is dissolved in about 1050 ml of water and quietly boiled for 1 to 2 h by using a flask. Thereafter, it stands as it is in a dark place for one night and the supernatant is filtered with a sintered-glass filter (G 4) (it shall not be washed with water before and after that). It is put into the brown bottle washed with steam for 30 min and stored in dark place. The standardization of this solution shall be carried out as follows:

2 to 2.5 g of sodium oxalate (JIS K 8005) which is dried at 150 to 200°C for 45 to 60 min and cooled in a desiccator is accurately weighed out into a beaker (500 ml) and dissolved by adding about 150 ml of water. Thereafter, it is transferred into a measuring flask of 250 ml and diluted up to the marked line together with the water with which the beaker is washed. Then, 25 ml is separately taken into a beaker (500 ml) by using a pipet, added with 200 ml of water and 10 ml of sulfuric acid and the temperature of solution is made to be at 25 to 30°C. Prepared potassium permanganate standard solution is dropped by wholly opening the cock of buret to about 2 ml before the titration required amount of prepared potassium permanganate standard solution with slowly mixing this sodium oxalate solution by using a stirring bar. After faint red has disappeared, the solution is heated at $57.5 \pm 2.5^\circ\text{C}$ {K}, carefully titrated successively agitating it with the stirring bar and the point where faint red is kept is made to be the end point. The titer of potassium permanganate standard solution shall be calculated from the following formula:

$$F = \frac{G_1 \times \frac{1}{10}}{V \times G_2}$$

where,

F: titer of potassium permanganate standard solution

G₁: weighed-out amount of sodium oxalate (standard reagent) (g)

V: consumed amount of potassium permanganate standard solution (ml)

G₂: the amount of sodium oxalate corresponding to accurate 1 ml of N/10 potassium permanganate standard solution (0.006702 g)

Further, 200 ml of water and 10 ml of sulfuric acid are taken into another beaker (500 ml) and warmed at $57.5 \pm 2.5^\circ\text{C}$ {K}. It is corrected by carrying out a blank test.

- (12) o-Phenanthroline solution 3 g of o-phenanthroline (1 hydrate) is weighed out and dissolved in 200 ml of water containing 2.0 g of ammonium ferrous sulfate.

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6.2.3 Weighing-Out Amount of Sample 0.2 g of the sample shall be weighed out.

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

- (1) Weigh out a sample, transfer it into a beaker (500 ml), add 20 ml of aqua regia ⁽¹⁾ and decompose it by heating. Thereafter, add 20 ml of sulfuric acid and 10 ml of phosphoric acid, evaporate it by heating and generate white fume of sulfuric acid for 3 to 4 min ⁽²⁾.
- (2) After standing to cool, add about 150 ml of warm water, warm it and dissolve salts. Thereafter, add 10 ml of silver nitrate and 20 to 25 ml of ammonium persulfate solution, oxidize chromium to dichromic acid by boiling them for 5 to 7 min ⁽³⁾ and decompose ammonium persulfate. Thereafter, decompose permanganic acid by adding about 5 ml of hydrochloric acid (1+3) ⁽⁴⁾. Then, add about 5 ml of manganese sulfate solution ⁽⁵⁾, boil them for 2 to 3 min and completely remove the generated chlorine.
- (3) After cooling it to not higher than ordinary temperature by using cold water, dilute the solution to about 300 ml by adding water, add 0.25 ml of o-phenanthroline solution as indicator, accurately add N/10 ammonium ferrous sulfate standard solution so that after the sample solution starts to turn reddish brown ⁽⁶⁾, its amount further becomes 5 ml degree in excess, immediately titrate the sample solution with N/10 potassium permanganate standard solution and allow the point where its reddish brown turns light green to be the end point.

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

$$\text{Chromium (\%)} = \frac{(V_1 \times F_1 - V_2 \times F_2) \times 0.001734}{W} \times 100$$

where,

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

F_1 : titer of N/10 ammonium ferrous sulfate standard solution

V_2 : used amount of N/10 potassium permanganate standard solution (ml)

F_2 : titer of N/10 potassium permanganate standard solution

W : weighed out amount of sample

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Notes (1) Instead of the process wherein a sample is treated with aqua regia and added with sulfuric acid, the sample may be treated as follows:

Weigh out a sample, transfer it into an Erlenmeyer flask (500 ml), add 10 ml of perchloric acid and 10 ml of phosphoric acid and decompose it by heating to the degree that white fume due to perchloric acid is generated. Keep heating temperature at 200°C, further continue to heat it for 3 to 4 min and oxidize a great portion of chromium to chromic acid. After cooling it a little, dissolve salts by adding about 150 ml of water and boil it for about 1 min. Thereafter, add 15 ml of mixed acid and about 10 ml of silver nitrate solution. Then, treat it according to the procedures on and after the addition of ammonium persulfate solution of 6.2.4 (2) as appropriate, and determine chromium.

(2) Because, if thick white fume of sulfuric acid is generated for a long time, the subsequent dissolving operation becomes difficulty, care shall be taken therefor.

(3) In the case where the content of manganese is very little, and the colouration of permanganic acid does not appear, the sample solution is let to turn red by dropping potassium permanganate solution.

(4) When the colouration of permanganic acid or the precipitate of manganese dioxide remains in the solution, after further adding 2 to 3 ml of hydrochloric acid (1+3), completely decompose them by boiling.

(5) In the case of carrying out direct titration only with ammonium ferrous sulfate standard solution, the addition of manganese sulfate solution can be omitted.

(6) The point where the sample solution turns reddish brown, may be made the end point, too. The content of chromium in that case shall be calculated from the following formula:

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

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

F : titer of N/10 ammonium ferrous sulfate standard solution

W : weighed-out amount of sample (g)

7. Method for Determination of Manganese

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

- (1) Titrimetric method
- (2) Absorptiometry

7.2 Titrimetric Method

7.2.1 Summary A sample is decomposed with perchloric acid and chromium is oxidized by further continuing to heat it. Thereafter, it is added with sodium chloride and chromium is removed as chromyl chloride. It is added with mixed acid and ammonium persulfate and manganese is oxidized to permanganic acid. Then, the sample solution is titrated with sodium arsenite standard solution.

7.2.2 Reagents The reagents shall be as follows:

- (1) Nitric acid
- (2) Nitric acid (2+100)
- (3) Perchloric acid
- (4) Aqua regia Hydrochloric acid and nitric acid are mixed by a ratio of 3 to 1.
- (5) Mixed acid 150 ml of sulfuric acid is added into 435 ml of water and cooled. Thereafter, it is mixed with 250 ml of nitric acid, 150 ml of phosphoric acid and 15 ml of silver nitrate solution (20 w/v %).
- (6) Sodium chloride
- (7) Ammonium persulfate solution (20 w/v %) This solution shall be prepared at each use.
- (8) Sodium pyrosulfate

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- (9) Standard manganese solution (0.2 mg Mn/ml) 0.100 g of electrolytic manganese (not less than 99.9 %) is weighed out into a beaker (200 ml), added with 50 ml of sulfuric acid (1+4), decomposed by heating and cooled. Thereafter, it is transferred into a measuring flask of 500 ml and diluted up to the marked line with water.
- (10) Sodium arsenite standard solution 0.5 g of arsenic trioxide (As_2O_3) (standard reagent of JIS K 8005) is accurately weighed out into a beaker (200 ml), added with 20 ml of sodium hydroxide solution (4 w/v %) and about 100 ml of water, decomposed by heating and cooled. Thereafter, it is transferred into a measuring flask of 1000 ml. It is made slightly acid by adding sulfuric acid (1+35) utilizing phenolphthalein as indicator, added with 20 ml of sodium hydrogencarbonate solution (5 w/v %) thereto and diluted up to the marked line with water. The method for determination of manganese equivalent amount of this standard solution shall be as follows.

Pure nickel of the same amount as that of nickel contained in the sample is weighed out into an Erlenmeyer flask (500 ml) and decomposed with 10 ml of nitric acid (1+1) by heating. Thereafter it is accurately added with a fixed amount of standard manganese solution, hereinafter titrated according to the procedure on and after 7.2.4 (3) and the manganese equivalent amount per 1 ml of sodium arsenite standard solution shall be calculated from the following formula:

$$f = 0.0002 (\text{g}) \times \frac{V_1}{V_2}$$

where,

f : manganese equivalent amount per 1 ml of sodium arsenite standard solution (g)

V_1 : used amount of standard manganese solution (ml)

V_2 : used amount of sodium arsenite standard solution (ml)

7.2.3 Weighing-out Amount of Sample The sample shall, as a rule, be weighed out according to the content of manganese in accordance with the following Table.

Table

Manganese content %	Weighing-out amount g
Up to 0.05	1.0
0.05 and over, up to 0.5	0.5
Up to and incl. 0.5	0.2

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7.2.4 Operation The determination operation shall be carried out in accordance with the following procedures:

- (1) Weigh out a sample, transfer it into a beaker (300 ml), cover it with a watch glass, add 25 ml of perchloric acid for a sample of not heavier than 0.5 g, and 35 ml for 1 g and decompose it by quietly heating ⁽¹⁾.
- (2) Generate thick white fume of perchloric acid on a hot plate and sufficiently oxidize it until the solution turns orange. Add little by little 0.5 to 1.0 g of sodium chloride thereto, repeat the operation until the generation of brown fume stops and vaporize chromium as chromyl chloride. After removing chromium, generate white fume, again.
- (3) After cooling, transfer it into an Erlenmeyer flask (500 ml), add about 150 ml of warm water, boil it for several minutes, remove liberated chlorine and add 15 ml of mixed acid.
- (4) Dilute it to about 250 ml with warm water, start to boil it by heating, simultaneously add little by little 10 ml of ammonium persulfate solution, boil it until small bubbles become large bubbles, completely decompose ammonium persulfate and at the same time completely oxidize manganese to permanganic acid. Thereafter, immediately cool it to not higher than 25°C in running water. After cooling, immediately titrate it with sodium arsenite standard solution.

7.2.5 Calculation The content of manganese in sample shall be calculated from the following formula:

$$\text{Manganese (\%)} = \frac{V \times f}{W} \times 100$$

where,

V : used amount of sodium arsenite standard solution (ml)

f : manganese equivalent amount per 1 ml of sodium arsenite standard solution (g)

W : weighed-out amount of sample (g)

Note (1) When the sample is difficult to be decomposed with perchloric acid, decompose it by using a suitable amount of aqua regia and add perchloric acid. Thereafter, operate it according to the procedures on and after 7.2.4 (2) and determine manganese.

Further, for the sample which is incompletely decomposed with acid, filter the residue, wash it with warm nitric acid (2+100), transfer the residue together with filter paper into a platinum crucible and incinerate the filter paper. Thereafter, mix it with sodium pyrosulfate and fuse them. After cooling it, dissolve it with warm water and mix it with main solution.