

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for Chemical
Analysis of Chromium Metal**

JIS G 1323—1989

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JAPANESE INDUSTRIAL STANDARD

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Methods for Chemical Analysis of
Chromium Metal

G 1323-1989

1. Scope

This Japanese Industrial Standard specifies the methods for determination of chromium, carbon, phosphorus, sulfur, iron and aluminium in chromium metal.

Remark: The units and numerical values given in { } in this Standard are based on the traditional units and are appended for informative reference.

2. General Matters

General matters common to the methods for chemical analysis shall be in accordance with JIS G 1301.

3. Method for Determination of Chromium

3.1 Method for Determination The method for determination shall not be in accordance with chemical analysis and after determination of impurities, the residue, be taken.

3.2 Calculation The chromium content in the sample shall be calculated to two places of decimals according to the following formula, and the rest shall be discarded.

$$\text{Chromium wt\%} = 100 - (C + Si + P + S + Fe + Al)$$

where, C: carbon content (wt %)

Si: silicon content (wt %)

P: phosphorus content (wt %)

S: sulfur content (wt %)

Fe: iron content (wt %)

Al: aluminium content (wt %)

Applicable Standards:

JIS G 1301-General Rules for Chemical Analysis of Ferroalloys

JIS Z 2615-General Rules for Determination of Carbon in Metallic Materials

JIS Z 2616-General Rules for Determination of Sulfur in Metallic Materials

4. Method for Determination of Carbon

4.1 Division of Methods for Determination The method for determination of carbon shall be in accordance with one of the following:

- (1) Combustion - Neutralization Titrimetric Method This method applies to the sample of 0.05 wt % or over up to and excluding 0.1 wt % in carbon content.
- (2) Combustion - Conductometric Method This method applies to the sample of 0.001 wt % or over in carbon content.
- (3) Combustion - Coulometric Method This method applies to the sample of 0.001 wt % or over in carbon content.
- (4) Combustion - Infrared Absorptiometric Method This method applies to the sample of 0.001 wt % or over in carbon content.

4.2 Combustion - Neutralization Titrimetric Method

4.2.1 Summary Heat the sample in an oxygen gas flow, oxidize the carbon sufficiently to make carbon dioxide, after collecting this together with oxygen into a burette, introduce into a certain amount of sodium hydroxide standard solution to absorb the carbon dioxide, and thereafter titrate the excess sodium hydroxide by sulfuric acid standard solution.

4.2.2 Reagents Reagents shall be in accordance with 6.4.2 of JIS Z 2615.

4.2.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.2.4 Apparatus The apparatus shall be in accordance with 6.4.3 of JIS Z 2615.

4.2.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Allow the sample to be 1.0 g. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the amount most suitable for weighing-out amount of sample and combustion apparatus to mix thoroughly or cover on the sample.

Note ⁽¹⁾ High purity iron, tin, tungsten, etc.

4.2.6 Operation

4.2.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.4.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

Notes ⁽²⁾ Correct by taking precautions to the difference between the temperature indicating of pyrometer and the combustion tube inside temperature.

- (3) Those mean the conditions to be determined depending upon the specifications of apparatus used such as anode current, grid current, etc. of high frequency oscillator.

4.2.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.4.5 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace The operation shall be in accordance with Remark 2. of 6.4 of JIS Z 2615.

4.2.7 Blank Test The blank test shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.4.6 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace By using a crucible put-in with the ignition improver of the same amount as the amount of added sample without putting-in the sample, carry out the operation of 4.2.6.2 (2).

Further, in the case of using an ignition improver not to generate induction current by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon content included in the added iron or the like to take it as the blank test value.

4.2.8 Calculation The calculation shall be in accordance with 6.4.7 of JIS Z 2615.

4.3 Combustion - Conductometric Method

4.3.1 Summary Heat the sample in an oxygen gas flow to oxidize sufficiently the carbon to carbon dioxide, allow to absorb into a certain amount of alkali solution, and measure the change of conductivity of alkali solution before and after absorption.

4.3.2 Reagents Reagents shall be in accordance with 6.6.2 of JIS Z 2615.

4.3.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.3.4 Apparatus The apparatus shall be in accordance with 6.6.3 of JIS Z 2615 (4).

Note (4) Instead of tubular electric resistance heating furnace, a high frequency induction heating furnace may be used. In the case of using a high frequency induction heating furnace, for example the boat and boat cover are read as crucible and cover, instead of them.

4.3.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing amount of sample the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.3.6 Operation

4.3.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.6.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

4.3.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.6.5 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace

(a) Place a crucible containing the sample and an ignition improver on a receiving stand and close the combustion tube. After substituting the air in the tube by feeding-in oxygen at the designated flow rate, actuate the high frequency induction heating furnace.

(b) When the indicating value of recorder or indicator increases gradually and a definite value is indicated, read out the indicated value and cut the switch of high frequency to take out the crucible.

4.3.7 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.6.6 of JIS Z 2615.

(2) In the Case of Using High Frequency Induction Heating Furnace By using a crucible put-in with only ignition improver of the same amount as the amount added to the sample without putting-in the sample, carry out the operation of 4.3.6.2 (2).

Further, in the case of using such ignition improver that induction electric current is not generated by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon amount contained in the added iron or the like to take it as the blank test value.

4.3.8 Calculation The calculation shall be in accordance with 6.6.7 of JIS Z 2615.

4.4 Combustion - Coulometric Method

4.4.1 Summary Heat the sample in oxygen gas flow to oxidize sufficiently the carbon to make carbon dioxide, allow to absorb in weak alkaline barium salt solution made at a definite pH, and measure the quantity of electricity required for returning the pH reduced by absorption to the original pH by electrolysis of barium salt solution.

4.4.2 Reagents Reagents shall be in accordance with 6.7.2 of JIS Z 2615.

4.4.3 Devices and Materials Devices and materials shall be in accordance with 5. of JIS Z 2615.

4.4.4 Apparatus The apparatus shall be in accordance with 6.7.3 of JIS Z 2615 (4).

4.4.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount of sample the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable (1) from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.4.6 Operation

4.4.6.1 Preparatory Operation The preparatory operation shall be in accordance with 6.7.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

4.4.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 6.7.5 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace

- (a) Place a crucible containing the sample and ignition improver on a receiving stand, and close the combustion tube. After substituting the air in the tube by feeding-in oxygen at the designated flow rate, actuate the high frequency induction heating furnace and at the same time return the indicating value to zero.
- (b) When the indicating value of indicator increases gradually and a definite value is indicated, read out the indicated value and cut the switch of high frequency to take out the crucible.

4.4.7 Blank Test The blank test shall be carried out according to either one of the following:

- (1) In the Case of Using Tubular Electric Resistance Heating Furnace
The blank test shall be in accordance with 6.7.6 of JIS Z 2615.
- (2) In the Case of Using High Frequency Induction Heating Furnace
By using a crucible put-in with only ignition improver of the same amount as the amount added to the sample without putting-in the sample, carry out the operation of 4.4.6.2 (2).

Further, in the case of using such ignition improver, that the induction electric current is not generated by high frequency current, add 0.5 to 1.0 g of iron or the like of known carbon content and as lower as possible in carbon content to carry out, and correct the carbon amount contained in the added iron or the like to take it as the blank test value.

4.4.8 Calculation The calculation shall be in accordance with 6.7.7 of JIS Z 2615.

4.5 Combustion - Infrared Absorptiometric Method (Integral Method)

4.5.1 Summary Heat the sample in oxygen gas flow to oxidize sufficiently the carbon to make carbon dioxide, feed this together with oxygen into the infrared absorption cell, and measure the infrared absorption amount due to carbon dioxide.

4.5.2 Devices and Materials Devices and materials shall be in accordance with 5. and 6.9.2 of JIS Z 2615.

4.5.3 Apparatus The apparatus shall be in accordance with 6.9.3 of JIS Z 2615 (5).

Note (5) A tubular electric resistance heating furnace may be used.

4.5.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount the most suitable amount (usually 0.5 to 1.0 g) to the using apparatus. Select the ignition improver most suitable (1) from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing-out amount of sample and combustion apparatus and mix thoroughly or cover on the sample.

4.5.5 Operation

4.5.5.1 Preparatory Operation The preparatory operation shall be in accordance with 6.9.4 of JIS Z 2615.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1400 to 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

4.5.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace

- (a) Insert a boat containing the sample and ignition improver into the middle part of combustion tube, and feed oxygen at a designated flow rate to the apparatus to be used.
- (b) When the indicating value of indicator is increased gradually and a definite value is indicated, read out the indicating value.

(2) In the Case of Using a High Frequency Induction Heating Furnace The operation shall be in accordance with 6.9.5 of JIS Z 2615.

4.5.6 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.7.6 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace The blank test shall be in accordance with 6.9.6 of JIS Z 2615.

4.5.7 Calculation The calculation shall be in accordance with 6.9.7 of JIS Z 2615.

4.6 Combustion - Infrared Absorptiometric Method (Circulation Method)

4.6.1 Summary Heat the sample in a circulating oxygen gas flow at a definite pressure in a definite volume, oxidize carbon to carbon dioxide and carbon monoxide, feed together with the excess oxygen into the infrared absorption detector of circulation loop, and measure respectively infrared absorption amounts of carbon dioxide and carbon monoxide.

4.6.2 Devices and Materials Devices and materials shall be in accordance with 5. and 6.9.2 of JIS Z 2615.

4.6.3 Apparatus The apparatus shall be in accordance with 5. and 6.10.2 of JIS Z 2615 ⁽⁵⁾.

4.6.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver Make the weighing-out amount the most suitable amount (usually 0.5 to 1.0 g) to the apparatus to be used. Select the ignition improver most suitable ⁽¹⁾ from those indicated in 5. (13) of JIS Z 2615, add the most suitable amount to the weighing out amount of sample and combustion apparatus mix thoroughly or cover on the sample.

4.6.5 Operation

4.6.5.1 Preparatory Operation The preparatory operation shall be in accordance with 6.10.3 of JIS Z 2615.

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Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion furnace at 1400 to 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

4.6.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace

- (a) Insert a boat containing the sample and ignition improver into the middle part of combustion tube and feed oxygen at a designated flow rate to the apparatus to be used.
- (b) When the indicating value of indicator is increased gradually and a definite value is indicated, read out the indicating value.

(2) In the Case of Using a High Frequency Induction Heating Furnace The operation shall be in accordance with 6.10.4 of JIS Z 2615.

4.6.6 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 6.7.6 of JIS Z 2615.

(2) In the Case of Using a High Frequency Induction Heating Furnace The blank test shall be in accordance with 6.10.5 of JIS Z 2615.

4.6.7 Calculation The calculation shall be in accordance with 6.10.6 of JIS Z 2615.

5. Determination Method for Silicon

5.1 Determination Method The determination method for silicon shall be in accordance with silicon dioxide gravimetric method.

5.2 Silicon Dioxide Gravimetric Method

5.2.1 Summary Decompose the sample with hydrochloric acid, and carry out white fume treatment with perchloric acid to make silicon the insoluble silicic acid. After dissolving soluble salts with water, filter the precipitates. Ignite the precipitates to weigh the mass, thereafter treat with hydrofluoric acid to volatilize silicon dioxide and weigh its mass.

5.2.2 Reagents Reagents shall be as follows:

- (1) Hydrochloric acid (1 + 5, 1 + 10)
- (2) Perchloric acid (60 wt %)

- (3) Hydrofluoric acid
- (4) Sulfuric acid (1 + 3)
- (5) Hydrogen peroxide

5.2.3 Weighing-Out Amount of Sample The weighing-out amount of sample shall be 3.0 g.

5.2.4 Operation The determination operation shall be carried out according to the following procedures:

5.2.4.1 Decomposition of Sample and Dehydration Treatment of Silicic Acid Weigh out the sample, transfer to a beaker (300 ml), cover with a watch glass, and add hydrochloric acid (1 + 5) 60 ml to decompose by heating. Add perchloric acid 40 ml, evaporate by heating, and in the state where the vapour of perchloric acid back flows along the inside wall of beaker heat for 15 to 20 min.

5.2.4.2 Filtration Washing After allowing to cool with solution obtained in 5.2.4.1, add warm water approximately 150 ml to dissolve soluble salts, and add hydrogen peroxide 10 to 20 ml little by little to reduce chromium. After boiling ⁽⁶⁾ for approximately 1 min, remove the watch glass by washing with water, immediately filter the precipitates by using filter paper (Class 5 B), transfer the precipitates adhered on the inside wall of beaker by rubbing with policeman (glass rod with rubber) onto filter paper, at first wash with warm hydrochloric acid (1 + 10) five times, and then wash with warm water several times.

Note ⁽⁶⁾ If boils by heating for a long time, there is a fear that the insoluble silicic acid becomes again soluble, therefore carry out the dissolution in as short time as far as possible, and take so care as to carry out immediately the following filtration washing.

5.2.4.3 Incineration and Weighing The incineration and weighing shall be carried out according to the following procedures:

- (1) Transfer the precipitates obtained in 5.2.4.2 together with the filter paper into a platinum crucible (No. 30). After drying, heat gradually to incinerate the filter paper.
- (2) Ignite at approximately 1100°C for approximately 30 min, allow to stand to cool to room temperature in a desiccator and weigh the mass. Repeat this operation until it becomes a constant quantity.

5.2.4.4 Hydrofluoric - Acid Treatment and Weighing The hydrofluoric-acid treatment and weighing shall be carried out according to the following procedures:

- (1) Add 2 or 3 drops of sulfuric acid (1 + 3) to residue in a platinum crucible obtained in 5.2.4.3 to wet, and add hydrofluoric acid 3 to 5 ml to heat carefully. Volatilize silicon dioxide and sulfuric acid.

- (2) Ignite at approximately 1100°C for 30 min, allow to stand to cool to room temperature in a desiccator and weigh its mass. Repeat this operation until becoming constant quantity.

5.2.5 Blank Test By using only reagent, operate concurrently with the sample according to procedures of 5.2.4.1 to 5.2.4.4.

5.2.6 Calculation Calculate the silicon content in sample according to the following formula:

$$\text{Silicon wt\%} = \frac{[(m_1 - m_2) - (m_3 - m_4)] \times 0.4674}{m_0} \times 100$$

- where, m_1 : mass obtained in 5.2.4.3 (2) relating to sample (g)
- m_2 : mass obtained in 5.2.4.4 (2) relating to sample (g)
- m_3 : mass obtained in 5.2.4.3 (2) in blank test (g)
- m_4 : mass obtained in 5.2.4.4 (2) in blank test (g)
- m_0 : weighed out quantity of sample (g)

6. Determination Method of Phosphorus

6.1 Division of Determination Methods The determination method of phosphorus shall be in accordance with either one of the following:

- (1) Molybdenum Blue Absorptiometry This method applies to the sample less than 0.08 wt % in phosphorus content.
- (2) Molybdophosphoric Acid Extraction Separation Molybdenum Absorptiometry This method applies to the sample less than 0.03 wt % in phosphorus content.

6.2 Molybdenum Blue Absorptiometry

6.2.1 Summary Decompose the sample with perchloric acid to generate white fume. After reducing chromium, iron, etc. with sodium hydrogen sulfite, add coloration reagent containing hexa-ammonium hepta-molybdate and hydrazinium sulfate (2+), heat to generate phosphorus molybdenum blue and measure its absorbance.

6.2.2 Reagents Reagents shall be as follows:

- (1) Perchloric acid (60 wt %)
- (2) Sodium hydrogensulfite solution (100 g/l)
- (3) Color reagent solution Mix following A solution 25 ml, B solution 10 ml and water 40 ml at the time of use.

A solution: Dissolve hexa-ammonium hepta-molybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ 20 g in water 300 ml, add sulfuric acid (1 + 1) 650 ml to this, and after cooling, dilute with water to 1 l.

B solution: Hydrazinium sulfite (2 +) solution (1.5 g/l)

- (4) Standard phosphorus solution (100 μg P/ml) Dry potassium dihydrogen phosphate (KH_2PO_4) at 110°C, allow to stand to cool in a desiccator to room temperature, weigh out that made constant quantity 0.4394 g and dissolve in water. Transfer the solution by using water into a one mark volumetric flask of 1000 ml and dilute with water up to the mark.

6.2.3 Weighing-Out Amount of Sample The weighing-out amount of sample shall be 1.0 g.

6.2.4 Operation

6.2.4.1 Preparation of Sample Solution The preparation of sample solution shall be carried out according to the following procedures:

- (1) Weigh out the sample, transfer into a beaker (300 ml), cover with a watch glass, add perchloric acid 20 ml, heat to dissolve, generate white fume, and in the state where the inside of beaker becomes transparent and the vapour of perchloric acid back flows along the internal wall of beaker, heat approximately for 10 min. After standing to cool, remove the watch glass by washing with water, add warm water approximately 100 ml to dissolve soluble salts, filter by using filter paper (Class 5 A) into a one mark volumetric flask of 250 ml, and wash with warm water 4 or 5 times.
- (2) After cooling to ordinary temperature, dilute with water to the mark.

6.2.4.2 Coloration Separately take 25 ml accurately from the solution obtained in 6.2.4.1, transfer into a one mark volumetric flask of 100 ml, add sodium hydrogensulfite solution 10 ml, mix by shaking, and heat in boiling water bath until the color of solution becomes clear blue-green. Next, add color reagent solution [6.2.2 (3)] 25 ml, mix by shaking, and heat in boiling water bath for 40 min. After cooling to ordinary temperature in flow water, dilute with water up to the mark.

6.2.4.3 Measurement of Absorbance Take a part of solution obtained in 6.2.4.2 into an absorption cell (10 mm, made of glass) of photometer and taking water as the contrast solution measure the absorbance near 825 nm in wavelength.

6.2.5 Blank Test Using only reagent, operate concurrently with the sample according to the procedures of 6.2.4.1 to 6.2.4.3.

6.2.6 Preparation of Working Curve Take stepwise standard phosphorus solution [6.2.2 (4)] 0 to 8.0 ml (as phosphorus 0 to 800 μg) into several beakers (100 ml), and cover with watch glass. Add respectively perchloric acid 5 ml, heat to generate white fume, and in the state where the inside of beaker becomes transparent and the vapour of perchloric acid back flows

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along the internal wall of beaker, heat approximately 10 min. After cooling, wash the watch glass with water to remove, add warm water approximately 50 ml, and transfer by using water into a one mark volumetric flask of 250 ml. Hereinafter, operate according to the procedures of 6.2.4.1 (2) to 6.2.4.3, prepare the relation curve between the obtained absorbance and the phosphorus quantity, and travel in parallel the relation curve so as to pass the origin to take it as the working curve.

6.2.7 Calculation Obtain the quantity of phosphorus from the absorbance to be obtained from the absorbance obtained in 6.2.4 by subtracting the absorbance obtained in 6.2.5 and the working curve prepared in 6.2.6 and calculate the phosphorus content in sample according to the following formula:

$$\text{Phosphorus wt\%} = \frac{A}{m \times B} \times 100$$

where, A: detection quantity of phosphorus in separately taken sample solution (g)

m: weighing-out quantity of sample (g)

B: aliquot ratio of sample solution and blank test solution

6.3 Molybdophosphoric-Acid Extraction Separation Molybdenum Blue Absorptiometry

6.3.1 Summary Decompose the sample with perchloric acid to generate white fume. After reducing chromium with sodium hydrogensulfite, add hexa-ammonium hepta-molybdate, extract the generated molybdophosphoric acid complex with mixed solvent, back extract the molybdenum blue generated by reducing this with tin (II) chloride into water phase, and measure its absorbance.

6.3.2 Reagents Reagents shall be as follows:

- (1) Perchloric acid (60 wt %)
- (2) Sodium hydrogensulfite solution (100 g/l)
- (3) Hexa-ammonium hepta-molybdate solution (80 g/l) Dissolve hexa-ammonium hepta-molybdate tetrahydrate 8 g in water and dilute with water to 100 ml.
- (4) Tin (II) chloride solution Dissolve tin (II) chloride dihydrate 5 g in hydrochloric acid 160 ml, dilute with water to 500 ml and take it as the stock solution. Dilute this stock solution with water to five times at each use.
- (5) Mixed solvent Add 1-butanol (*n*-butyl alcohol) 1 to chloroform 3, shake sufficiently to mix.
- (6) Standard phosphorus solution (20 µg P/ml) Dry potassium dihydrogenphosphate (KH_2PO_4) at 110°C, stand to cool in a desiccator to ordinary temperature to make constant quantity, weigh out its 0.4394 g, dissolve in water transfer into a one mark volumetric flask 1000 ml, dilute with water up to the mark and take it as the standard stock solution (100 µg P/ml).

At each service dilute this stock solution with water accurately by five times and take it standard phosphorus solution.

6.3.3 Weighing-Out Amount of Sample The weighing-out amount of sample shall be 1.0 g.

6.3.4 Operation

6.3.4.1 Preparation of Sample Solution The preparation of sample solution shall be carried out according to the following procedures:

- (1) Weigh out the sample, transfer into a beaker (300 ml), cover with a watch glass, add perchloric acid 20 ml, heat to decompose, generate white fume and in the state where the inside of beaker becomes transparent, and vapour of perchloric acid back flows along the inside wall of beaker, heat approximately 10 min. After standing to cool, wash the watch glass with water to remove, add warm water approximately 100 ml to dissolve soluble salts, by using filter paper (Class 5 A) filter into a one mark volumetric flask of 250 ml, and wash with warm water four or five times.
- (2) After cooling to ordinary temperature, dilute with water up to the mark.

6.3.4.2 Extraction of Phosphorus Separately take 25 ml from the solution obtained in 6.3.4.1 (2), transfer to a separatory funnel (100 ml), add perchloric acid 5 ml ⁽⁷⁾, then while mixing by shaking the sodium hydrogensulfite until the solution indicates green color, further add its excess 1 ml, thereafter add hexa-ammonium hepta-molybdate solution [6.3.2 (3)] 5 ml and mix by shaking. Then, add mixed solvent [6.3.2 (5)] 20 ml to mix by shaking violently for 2 min, after standing still for a while to separate into two layers, transfer the organic phase of lower layer into other separatory funnel (100 ml) and store. Again add to water phase the mixed solvent 5 ml, mix by shaking violently for approximately 30 s, stand still for a while to separate into two layers, combine the organic phase of lower layer with the previously stored organic phase and discard the upper layer.

Note ⁽⁷⁾ The most suitable concentration of perchloric acid at the time of extraction is 1 to 2 N, and by adding in addition of perchloric acid of 5 ml it becomes approximately 1.5 N.

6.3.4.3 Coloration Add to the organic phase obtained in 6.3.4.2 the tin (II) chloride solution [6.3.2 (4)] correctly 20 ml, mix by shaking violently for approximately 30 s, stand still for a while, and after separating into two layers, discard the lower layer ⁽⁸⁾.

Note ⁽⁸⁾ If leave contacting the both phases for a long time, the lower value appears, and therefore after separating into two layers by standing still, immediately discard the organic phase.

6.3.4.4 Measurement of Absorbance Take a part of colored solution obtained in 6.3.4.3 into an absorption cell (10 mm), and taking water as the contrast solution measure the absorbance near 700 nm in wavelength.

6.3.5 Blank Test By using only reagent, operate concurrently with the sample according to the procedures of 6.3.4.1 to 6.3.4.4.

6.3.6 Preparation of Working Curve Take the standard phosphorus solution [6.3.2 (6)] 0 to 15.0 ml (as phosphorus 0 to 300 µg) stepwise into several beakers (300 ml), and cover with watch glass. Respectively, add perchloric acid 15 ml, heat to generate white fume, and in the state where the inside of beaker becomes transparent and the vapour of perchloric acid back flows along the inside wall of beaker, heat for approximately 10 min. After standing to cool, wash the watch glass with water to remove, add warm water approximately 100 ml to dissolve soluble salts, transfer by using water into a one mark volumetric flask of 250 ml, and after cooling to ordinary temperature, dilute up to the mark with water. Hereinafter, operate according to the procedures of 6.3.4.1 (2) to 6.3.4.4, prepare the relation curve between the obtained absorbance and the phosphorus quantity, travel the relation curve in parallel so as to pass through the origin, and take it as the working curve.

6.3.7 Calculation Obtain the phosphorus amount from the absorbance to be obtained by subtracting the absorbance obtained in 6.3.5 from the absorbance obtained in 6.3.4.4 and the working curve prepared in 6.3.6, and calculate the phosphorus content in sample according to the following formula:

$$\text{Phosphorus wt\%} = \frac{A}{m \times B} \times 100$$

where, A : detection amount of phosphorus in aliquot sample solution (g)

m : weighed-out amount of sample (g)

B : aliquot ratio of sample solution and blank test solution

7. Determination Methods of Sulfur

7.1 Division of Determination Methods The determination method of sulfur shall be in accordance with one of the following:

- (1) Combustion - Neutralization Titrimetric Method This method applies to the sample not less than 0.005 wt % in sulfur content.
- (2) Combustion - Conductometric Method This method applies to the sample not less than 0.001 wt % in sulfur content.
- (3) Combustion - Coulometric Method This method applies to the sample not less than 0.001 wt % in sulfur content.
- (4) Combustion - Infrared Absorptiometric Method This method applies to the sample not less than 0.001 wt % in sulfur content.

7.2 Combustion - Neutralization Titrimetric Method

7.2.1 Summary Combust the sample in an oxygen gas flow, absorb the generated sulfur oxide in hydrogen peroxide water to make sulfuric acid and titrate this by sodium hydroxide standard solution.

7.2.2 Reagents Reagents shall be in accordance with 7.2 (2) of JIS Z 2616.

7.2.3 Devices and Materials Devices and materials shall be in accordance with 6. of JIS Z 2616.

7.2.4 Apparatus The apparatus shall be in accordance with 7.2 (3) of JIS Z 2616 (4).

7.2.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver The weighing-out amount of sample shall be 0.5 g. As to the ignition improver, select the most suitable one (1) from those shown in 6.12 of JIS Z 2616, add the most suitable amount for the weighing-out amount of sample and the combustion apparatus, mix thoroughly or cover on the sample.

7.2.6 Operation

7.2.6.1 Preparatory Operation The preparatory operation shall be in accordance with 7.2 (4) of JIS Z 2616.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency heating furnace.

7.2.6.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The operation shall be in accordance with 7.2 (5) of JIS Z 2616.

(2) In the Case of Using a High Frequency Induction Heating Furnace The operation shall be in accordance with Remark of 7.2 of JIS Z 2616.

7.2.7 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 7.2 (6) of JIS Z 2616.

(2) In the Case of Using a High Frequency Induction Heating Furnace By using a crucible containing only ignition improver of the same amount as the added amount to the sample without adding sample, carry out the operation according to Remark of 7.2 of JIS Z 2616.

Further, in the case where an ignition improver such as not to generate induction current by high frequency electric current is used, add 0.5 to 1.0 g of iron or the like of known sulfur content and as lower as possible in sulfur content to carry out the test, and correct the sulfur amount contained in added iron or the like to take it as the blank test value.

7.2.8 Calculation The sulfur content in sample shall be calculated according to the following formula:

$$\text{Sulfur wt\%} = \frac{(V_1 - V_2) \times f}{m} \times 100.$$

where, V_1 : using amount of 0.01 mol/l sodium hydroxide standard solution obtained in 7.2.6.2 (ml)

V_2 : using amount of 0.01 mol/l sodium hydroxide standard solution obtained in 7.2.7 (ml)

f : amount of sulfur corresponding to 1 ml of 0.01 mol/l sodium hydroxide standard solution (g)

m : weighing-out amount of sample (g)

7.3 Combustion - Conductometric Method

7.3.1 Summary Heat the sample in oxygen gas flow to oxidize sulfur to sulfur oxide, absorb this into a definite amount of hydrogen peroxide water of sulfuric acid to make sulfuric acid, and measure the variation of conductivity of acid solution before and after the absorption.

7.3.2 Reagents Reagents shall be in accordance with 7.5 (2) of JIS Z 2616.

7.3.3 Devices and Materials Devices and materials shall be in accordance with 6. of JIS Z 2616.

7.3.4 Apparatus The apparatus shall be in accordance with 7.5 (3) of JIS Z 2616 (4).

7.3.5 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver The weighing-out amount of sample shall be the amount (usually 0.5 to 1.0 g) most suitable for the apparatus to be used. Select the ignition improver most suitable (1) from those indicated in 6.12 of JIS Z 2616, add the amount most suitable for the weighing-out amount of sample and the combustion apparatus and mix thoroughly or cover on the sample.

7.3.6 Operation

7.3.6.1 Preparatory Operation The preparatory operation shall be in accordance with 7.5 (4) of JIS Z 2616.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

7.3.6.2 Determination Operation The determination operation shall be in accordance with 7.5 (5) of JIS Z 2616.

7.3.7 Blank Test The blank test shall be in accordance with 7.5 (6) of JIS Z 2616 (9).

Note (9) In the case of using a high frequency induction heating furnace, use a sample of known sulfur content and as lower as possible in sulfur content, and subtract the measured value corresponding to the known sulfur amount from the obtained measured value to take it as the blank test value.

7.3.8 Calculation The calculation shall be in accordance with 7.5 (7) of JIS Z 2616.

7.4 Combustion - Coulometric Method

7.4.1 Summary Burn the sample in oxygen gas flow, and absorb the generated sulfur oxide into hydrogen peroxide · sodium sulfate absorption solution set preliminarily at a definite pH value. Measure the quantity of electricity required for generating alkali required for neutralizing the hydrogen ions increased at that time by electrolysis.

7.4.2 Reagents Reagents shall be in accordance with 7.6 (2) of JIS Z 2616.

7.4.3 Apparatus The apparatus shall be in accordance with 7.6 (3) of JIS Z 2616 (5).

7.4.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver The weighing-out amount of sample shall be the most suitable amount for the apparatus to be used (usually 0.5 to 1.0 g). As to the ignition improver, select the most suitable one (1) from those indicated in 6.12 of JIS Z 2616, add the amount most suitable for the weighing-out amount of sample and the combustion apparatus, and mix thoroughly or cover on the sample.

7.4.5 Operation

7.4.5.1 Preparatory Operation The preparatory operation shall be in accordance with 7.6 (4) of JIS Z 2616.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

7.4.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace

- (a) Insert a boat containing sample and ignition improver into the middle part of combustion tube, and feed oxygen at the flow rate designated to the apparatus to be used.
- (b) When the indicating value of indicator increases gradually and has indicated a definite value, read out the indicating value.

(2) In the Case of Using a High Frequency Induction Heating Furnace The determination operation shall be in accordance with 7.6 (5) of JIS Z 2616.

7.4.6 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 7.2 (6) of JIS Z 2616.(2) In the Case of Using a High Frequency Induction Heating Furnace The blank test shall be in accordance with 7.6 (6) of JIS Z 2616.

7.4.7 Calculation The calculation shall be in accordance with 7.6 (7) of JIS Z 2616.

7.5 Combustion - Infrared Absorptiometric Method (Integral Method)

7.5.1 Summary Heat the sample in oxygen gas flow to a high temperature to oxidize sulfur to sulfur dioxide, feed this together with oxygen into infrared absorption detector, and measure the infrared absorption amount of sulfur dioxide.

7.5.2 Devices and Materials Devices and materials shall be in accordance with 6. of JIS Z 2616.

7.5.3 Apparatus The apparatus shall be in accordance with 7.7 (2) of JIS Z 2616 (5).

7.5.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver The weighing-out amount of sample shall be the amount most suitable for the apparatus to be used (usually 0.5 to 1.0 g). As to the ignition improver, select the most suitable that ⁽¹⁾ from those indicated in 6.12 of JIS Z 2616, add the most suitable amount for the weighing-out amount of sample and the combustion apparatus, and mix thoroughly or cover on the sample.

7.5.5 Operation

7.5.5.1 Preparatory Operation The preparatory operation shall be in accordance with 7.7 (3) of JIS Z 2616.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1450°C (2).

Further, in the case of using a high frequency induction heating furnace, set the conditions (3) relating to the high frequency induction heating furnace.

7.5.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace

- (a) Insert a boat containing the sample and ignition improver into the middle part of combustion tube and feed oxygen at a flow rate designated to the apparatus to be used.
- (b) When the indicating value of indicator increases gradually and a definite value is indicated, read out the indicating value.

(2) In the Case of Using a High Frequency Induction Heating Furnace The determination operation shall be in accordance with 7.7 (4) of JIS Z 2616.

7.5.6 Blank Test The blank test shall be carried out according to either one of the following:

(1) In the Case of Using a Tubular Electric Resistance Heating Furnace The blank test shall be in accordance with 7.2 (6) of JIS Z 2616.

(2) In the Case of Using a High Frequency Induction Heating Furnace The blank test shall be in accordance with 7.7 (5) of JIS Z 2616.

7.5.7 Calculation The calculation shall be in accordance with 7.7 (6) of JIS Z 2616.

7.6 Combustion - Infrared Absorptometric Method (Circulation Method)

7.6.1 Summary Heat the sample in circulating oxygen gas flow in a definite volume under a definite pressure to high temperature to oxidize sulfur to sulfur dioxide, feed together with excess oxygen into the infrared absorption detector of circulation loop, and measure the infrared absorption amount of sulfur dioxide.

7.6.2 Devices and Materials Devices and materials shall be in accordance with 6. of JIS Z 2616.

7.6.3 Apparatus The apparatus shall be in accordance with 7.8 (2) of JIS Z 2616 (5).

7.6.4 Weighing-Out Amount of Sample and Additive Amount of Ignition Improver The weighing-out amount of sample shall be the most suitable amount for the apparatus to be used (usually 0.5 to 1.0 g). As to the ignition improver, select the most suitable improver ⁽¹⁾ from those indicated in 6.12 of JIS Z 2616, add its most suitable amount for the weighing amount of sample and the combustion apparatus, and mix thoroughly or cover on the sample.

7.6.5 Operation

7.6.5.1 Preparatory Operation The preparatory operation shall be in accordance with 7.8 (3) of JIS Z 2616.

Further, in the case of using a tubular electric resistance heating furnace, keep the inside temperature of combustion tube at 1450°C ⁽²⁾.

Further, in the case of using a high frequency induction heating furnace, set the conditions ⁽³⁾ relating to the high frequency induction heating furnace.

7.6.5.2 Determination Operation The determination operation shall be carried out according to either one of the following:

(1) **In the Case of Using a Tubular Electric Resistance Heating Furnace**

- (a) Insert a boat containing the sample and ignition improver into the middle part of combustion tube, and feed oxygen at a designated flow rate to the apparatus to be used.
- (b) When the indicating value of indicator increases gradually and a definite value is indicated, read out the indicating value.

(2) **In the Case of Using a High Frequency Induction Heating Furnace** The operation shall be in accordance with 7.8 (4) of JIS Z 2616.

7.6.6 Blank Test The blank test shall be carried out according to either one of the following:

- (1) **In the Case of Using a Tubular Electric Resistance Heating Furnace** The blank test shall be in accordance with 7.2 (6) of JIS Z 2616.
- (2) **In the Case of Using a High Frequency Induction Heating Furnace** The blank test shall be in accordance with 7.8 (5) of JIS Z 2616.

7.6.7 Calculation The calculation shall be in accordance with 7.8 (6) of JIS Z 2616.

8. Determination Method of Iron

8.1 Determination Method The determination method of iron shall be in accordance with atomic absorption spectrophotometry.

8.2 Atomic Absorption Spectrophotometry

8.2.1 Summary Decompose the sample by perchloric acid, after carrying out the residue treatment, dilute with water to a definite amount, spray the solution into the air-acetylene flame of atomic absorption photometer, and measure the absorbance.

8.2.2 Reagents Reagents shall be as follows:

- (1) Perchloric acid (60 wt %)
- (2) Hydrofluoric acid
- (3) Sulfuric acid (1 + 1)
- (4) Sodium disulfate
- (5) Sodium disulfate solution (100 g/l)
- (6) Chromium solution (10 mg Cr/ml) Weigh out electrolysis chromium metal-1.00 g, transfer into a beaker (500 ml), cover with a watch glass, add perchloric acid 15 ml to decompose by heating, successively heat and generate the white fume approximately for 10 min. After standing to cool, wash the watch glass to remove, add warm water approximately 50 ml to dissolve salts, after cooling to ordinary temperature, transfer by using water into a one mark volumetric flask of 100 ml, and dilute with water to the mark.
- (7) Standard iron solution (100 µg Fe/ml) Weigh out iron (99.5 wt % or more) 0.100 g, transfer into a beaker (300 ml), cover with a watch glass, add hydrochloric acid (1 + 1) 30 ml to decompose by heating, add nitric acid (1 + 1) 2 ml and boil approximately 10 min. After cooling to ordinary temperature, wash the watch glass with water to remove, transfer by using water into a one mark volumetric flask of 1000 ml and dilute with water to the mark.

8.2.3 Weighing-Out Amount of Sample The weighing-out amount of sample shall be 1.0 g.

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

8.2.4.1 Decomposition of Sample Weigh out the sample, transfer into a beaker (300 ml), cover with a watch glass, add perchloric acid 15 ml, and at first heat slowly to decompose a large part of sample. Then, ignite, and since beginning of generation of perchloric acid white fume, further heat approximately for 10 min. After standing to cool, add warm water approximately 50 ml to dissolve the soluble salts. Wash the watch glass with water to remove, and filter by using filter paper (Class 5 A) into a beaker (200 ml). After washing with warm water 4 or 5 times, store the filtrate and washings as the main solution.

8.2.4.2 Treatment of Residue Transfer the residue obtained in 8.2.4.1 together with the filter paper into a platinum crucible (No. 30), dry to incinerate the filter paper at a low temperature, and thereafter ignite at 500 to 600°C. After standing to cool to room temperature, wet the residue with two or three drops of water, add two or three drops of surface acid (1 + 1) and 1 ml of hydrofluoric acid, and heat on sand bath to evaporate to dryness. After standing to cool, add sodium disulfate 1 g, and heat until turning to dark red color to fuse. After standing to cool, put a crucible into a beaker containing the stored main solution, heat to dissolve the melt. Remove the crucible from the beaker by washing with water. After cooling to ordinary temperature, transfer the solution into a one mark volumetric flask of 100 ml by using water and dilute to the mark. Separately take 10 ml from this solution, transfer into a one mark volumetric flask of 100 ml, and dilute with water to the mark.

8.2.4.3 Measurement of Absorbance Spray a part of solution obtained in 8.2.4.2 into air · acetylene flame of atomic absorption photometer the zero point of which is regulated by using water, and measure the absorbance near 248.3 nm in wavelength ⁽¹⁰⁾.

Note ⁽¹⁰⁾ An example of measuring conditions of absorbance of iron is shown in Table 1.

Table 1. Example of Measuring Conditions

Wavelength nm	Electric current of lamp mA	Using gas	
		Pressure	Flowing speed
248.3	10 to 15	Acetylene { 0.049 MPa 0.5 kgf/cm ² }	3 l/min
		Air { 0.177 MPa 1.8 kgf/cm ² }	13 l/min

8.2.5 Blank Test By using only reagent, operate concurrently with the sample according to procedures of 8.2.4.1 to 8.2.4.3.

8.2.6 Preparation of Working Curve Take various solution amounts of standard iron solution [8.2.2 (7)] 0 to 2.0 ml (0 to 2.0 mg as iron) into several one mark volumetric flasks of 100 ml, add respectively chromium solution [8.2.2 (6)] 10.0 ml and sodium disulfate solution 1.0 ml and dilute with water to the mark. Spray a part of solution into air · acetylene flame of atomic absorption photometer zero point of which is regulated by using water, and measure, concurrently with the sample, the absorbance near 248.3 nm in wavelength. Prepare the relation curve between the obtained absorbance and the iron amount, travel the relation curve in parallel so as to pass through the origin and take it as the working curve.

8.2.7 Calculation Obtain the iron amount from the absorbance to be obtained by subtracting the absorbance obtained in 8.2.5 from the absorbance obtained in 8.2.4.3 and the working curving prepared in 8.2.6, and calculate the iron content in sample according to the following formula:

$$\text{Iron wt\%} = \frac{A}{m \times B} \times 100$$

where, A : detecting amount of iron in the aliquot sample solution (g)

m : weighing-out amount of sample (g)

B : aliquot ratio of sample solution and blank test solution

9. Determination Method of Aluminium

9.1 Determination Method The determination of aluminium shall be in accordance with atomic absorption spectrophotometry.

9.2 Atomic Absorption Spectrophotometry

9.2.1 Summary Decompose the sample by perchloric acid, after treating the residue, dilute with water to a definite value, spray the solution into dinitrogen oxide-acetylene flame of atomic absorption spectrophotometer and measure the absorbance.

9.2.2 Reagents Reagents shall be as follows:

- (1) Perchloric acid (60 wt %)
- (2) Hydrofluoric acid
- (3) Sulfuric acid (1 + 1)
- (4) Electrolysis chromium metal
- (5) Sodium disulfate
- (6) Sodium disulfate (100 g/l)
- (7) Standard aluminium solution (500 $\mu\text{g Al/ml}$) Weigh out aluminium (not less than 99.9 wt %) 0.500 g, transfer into a beaker (200 ml), and add hydrochloric acid (1 + 1) 50 ml to decompose by heating. After cooling to ordinary temperature, transfer by using water into a one mark volumetric flask of 1000 ml, and dilute with water to the mark.

9.2.3 Weighing-Out Amount of Sample The weighing-out amount of sample shall be 1.0 g.

9.2.4 Operation The determination operation shall be carried out according to the following procedures:

9.2.4.1 Decomposition of Sample Weigh out the sample, transfer into a beaker (300 ml), cover with a watch glass, add perchloric acid 15 ml, and at first heat slowly to decompose a large part of sample. Then, ignite and since beginning of generating of perchloric acid white fume, further ignite approximately for 10 min. After standing to cool, add warm water approximately 50 ml to dissolve soluble salts. Wash the watch glass with water to remove, and filter the solution by using filter paper (Class 5 A) into a beaker (200 ml). After washing with warm water 5 to 6 times, store the filtrate and washings as the main solution.

9.2.4.2 Treatment of Residue Transfer the residue obtained in 9.2.4.1 together with the filter paper into a platinum crucible (No. 30), dry and after incinerating the filter paper at a low temperature, ignite at 500 to 600°C. After cooling to room temperature, wet the residue by two or three drops of water, add two or three drops of sulfuric acid (1 + 1) and hydrofluoric acid 1 ml, and heat on sand bath to evaporate to dryness. After standing to cool, add sodium disulfate 1 g, and heat until dark red color appears to fuse. After standing to cool, put the crucible into a beaker containing the stored main solution, and heat to dissolve the melt. Remove the crucible from the beaker by washing with water. After cooling to ordinary temperature, transfer the solution by using water into a one mark volumetric flask of 100 ml and dilute with water up to the mark.

9.2.4.3 Measurement of Absorbance Spray a part of solution obtained in 9.2.4.2 into dinitrogen oxide · acetylene flame of atomic absorption spectrophotometer the zero point of which is regulated by using water, and measure the absorbance at 309.2 nm (or 396.2 nm) in wavelength ⁽¹¹⁾.

Note ⁽¹¹⁾ The example of measuring conditions of absorbance of aluminium is shown in Table 2.

Table 2. Example of Measuring Conditions

Wavelength nm	Electric current of lamp mA	Using gas	
		Pressure	Flow rate
309.2 (or 396.2)	10 to 15	Acetylene { 0.049 MPa 0.5 kgf/cm ² }	4 l/min
		Di-nitrogen oxide { 0.177 MPa 1.8 kgf/cm ² }	6 l/min

9.2.5 Blank Test By using only reagent, operate concurrently with the sample according to the procedures of 9.2.4.1 to 9.2.4.3.

9.2.6 Preparation of Working Curve Take various amounts of standard aluminium solution [9.2.2 (7)] 0 to 6.0 ml (as aluminium 0 to 3.0 mg) into several beakers (300 ml), add respectively electrolysis chromium metal 1.0 g by weighing-out, cover with a watch glass, add perchloric acid 15 ml, heat to decompose, and successively heat to evaporate to generate white fume approximately for 10 min. After standing to cool, add warm water approximately 50 ml to dissolve the soluble salts. Remove the watch glass by washing with water, filter the solution by using filter paper (Class 5 A) into a one mark volumetric flask of 100 ml, and wash with warm water 5 to 6 times. Add to this the sodium disulfate solution 10.0 ml, and after cooling to ordinary temperature, dilute with water to the mark. Spray a part of solution into dinitrogen oxide · acetylene flame of atomic absorption spectrophotometer the zero point of which is regulated by using water, and measure, concurrently with the sample, the absorbance at 309.2 nm (or 396.2 nm) in wavelength. Prepare the relation curve between the obtained absorbance and the aluminium amount and travel in parallel so as the relation curve to pass through the origin and take it as the working curve. However, the measurement of absorbance shall be carried out in parallel with the sample solution.

9.2.7 Calculation Obtain the aluminium amount from the absorbance to be obtained by subtracting the absorbance obtained in 9.2.5 from the absorbance obtained in 9.2.4.3 and the working curve prepared in 9.2.6, and calculate the aluminium content in the sample according to the following formula:

$$\text{Aluminium wt\%} = \frac{A}{m} \times 100$$

where, A : detected amount of aluminium in sample (g)

m : weighing-out amount of sample (g)

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