

JIS

JAPANESE INDUSTRIAL STANDARD

**Method for Chemical Analysis
of Silicon Carbide Abrasives**

JIS R 6124—1987

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JAPANESE INDUSTRIAL STANDARD

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Method for Chemical Analysis of Silicon
Carbide Abrasives

R 6124-1987

1. Scope

This Japanese Industrial Standard specifies the method for chemical analysis and the X-ray fluorescence spectrometric analysis of silicon carbide abrasives.

Remark: The units and numerical values given in { } in this Standard are in accordance with the traditional system of units to be the Standard values.

2. Analysis Items

The analysis items specified in this Standard shall be as follows:

2.1 Method for Chemical Analysis

- (1) Iron soluble in hydrochloric acid (Fe_s)
- (2) Total silicon (Si_T)
- (3) Total iron (Fe_T)
- (4) Aluminum (Al)
- (5) Calcium oxide (CaO)
- (6) Magnesium oxide (MgO)
- (7) Total carbon (C_T)
- (8) Free carbon (C_F)
- (9) Silicon carbide (SiC)
- (10) Free silicic acid (SiO_{2F})
- (11) Free silicon (Si_F)

2.2 X-ray Fluorescence Spectrometric Analysis

- (1) Total iron (Fe_T)
- (2) Aluminum (Al)

Applicable Standards, Corresponding International Standard and
Reference Standards: See page 51.



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3. General Items

General items that are applied in common to the chemical analysis shall be in accordance with JIS K 0050, general items that are in common to the absorptiometry, with JIS K 0115, and general items that are in common to the X-ray fluorescence spectrometric analysis, with JIS K 0119.

The symbols for classes of silicon carbide abrasives shall conform to Table 1 in JIS R 6111.

4. Sample

The sample shall be taken from one inspecting unit according to JIS R 6003, and, after about 20 g of the sample that is put in a flat weighing bottle (60 mm x 30 mm) is dried in an air bath of $105 \pm 5^\circ\text{C}$ for 1 hour, the sample kept in a desiccator and stoppered tightly shall be supplied as a sample.

Further, the sample shall be treated in the state of original grit as it is in principle, but in the case of the sample for the determination of total carbon and free carbon whose grit size is coarser than that of No. 80, the sample shall be pulverized to such degree as passing through the sieve of $150 \mu\text{m}$ by using a steel mortar.

5. Rounding-off of Analysis Results

Analysis results shall be expressed by percentage, and be rounded off to the second decimal place in accordance with JIS Z 8401.

6. Determination of Iron Soluble in Hydrochloric Acid

6.1 Summary Boil the sample in hydrochloric acid by heating, and add hydroxylammonium chloride solution into the solution, in which iron has been eluted, to reduce ferric ion to ferrous ion. After coloring the solution by adding 1, 10-phenanthroline solution, measure the absorbance to determine the iron soluble in hydrochloric acid.

6.2 Reagents Reagents to be used shall be as follows:

- (1) Hydrochloric acid (1 + 1, 2 + 100)
- (2) Sodium acetate solution (50 % w/v %)
- (3) Hydroxylammonium chloride solution (0.5 % w/v %)
- (4) 1, 10-phenanthroline solution (0.2 % w/v %)
- (5) Standard iron solution (0.1 mg Fe/ml): Weigh 0.100 g Fe (99.5 % min.) into a 300 ml beaker, and, after dissolving it with adding 5 ml of nitric acid (1 + 1) and 5 ml of sulfuric acid (1 + 1), heat until white fume of sulfuric acid evaporates. Add 10 ml of nitric acid (1 + 6) and water to dissolve it, and transfer it to a 1 l transfer flask. Dilute it with water up to the marked line.

6.3 Photometer Either a photoelectric photometer or a photoelectric spectrophotometer shall be used.

6.4 Quantity of Sampling About 1.0 g of sample shall be taken.

6.5 Operation Determination operation shall be carried out according to the following procedures:

- (1) Weigh out accurately about 1.0 g of the sample to the nearest 0.1 mg into a 300 ml beaker and, after adding 50 ml of hydrochloric acid (1 + 1), boil it for 10 min.
- (2) After cooling, filter it through the filter paper (Class 5 B). Wash several times the residue with hydrochloric acid (2 + 100). Collect the filtrate and washings into a 250 ml transfer flask and add water up to the marked line, to mix by shaking.
- (3) Place correctly an aliquot of 25 ml from the solution into a 100 ml beaker.
- (4) To make pH⁽¹⁾ of this solution 3.5, add sodium acetate solution (50 w/v %) into this solution, and transfer it to a 100 ml transfer flask.
- (5) Add 5 ml of hydroxylammonium chloride solution (0.5 w/v %) and mix by shaking⁽²⁾.
- (6) Add 5 ml of 1, 10-phenanthroline solution (0.2 w/v %) and dilute with water up to the marked line to mix by shaking⁽²⁾.
- (7) 15 min later, place a part of the solution in 10 mm absorption cell, and measure its absorbance with reference to a blank test solution in the vicinity of 500 nm wavelength. Obtain iron content by using the working curve prepared in advance.
- (8) The content of iron soluble in hydrochloric acid shall be calculated according to the following formula:

$$Fe_s (\%) = \frac{m_2}{m_1 \times \frac{25}{250}} \times 100$$

where $Fe_s (\%)$: content percentage of iron soluble in hydrochloric acid (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of iron detected in an aliquot sample solution (g)

Notes ⁽¹⁾ To control pH, use a pH meter, or Bromophenol Blue (BPB) pH test paper.

⁽²⁾ It is permissible to add 10 ml of mixed solution which has been prepared in advance by mixing hydroxylammonium chloride solution and 1, 10-phenanthroline solution.

6.6 Preparation of Working Curve Place several parts of the iron standard solution from 0 to 10 ml (from 0 to 1.0 mg as Fe) stepwise into 100 ml beakers, and pour 50 ml of water respectively. Then carry out the operations in 6.5 (4) and thereafter, and prepare the working curve by plotting the relations between the iron contents and the absorbances.

7. Determination of Total Silicon

7.1 Summary Fuse the sample with sodium hydroxide and potassium nitrate by heating, and, after dissolving in hydrochloric acid, evaporate to dryness by heating to make silicon insoluble silicon dioxide. Then, after vaporizing the silicon by adding hydrofluoric acid, determine the content of total silicon from the weight decreased.

7.2 Reagents Reagents to be used shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1)
- (3) Sulfuric acid (1 + 1)
- (4) Hydrofluoric acid
- (5) Sodium hydroxide
- (6) Potassium nitrate

7.3 Devices and Apparatus Devices and apparatus to be used shall be as follows:

- (1) Platinum crucible with a cap For instance, No. 30 specified in JIS H 6201, hereinafter referred to as the "crucible".
- (2) Nickel crucible with a cap Capacity 30 ml.
- (3) Nickel beaker Capacity 500 ml.
- (4) Electric furnace The furnace capable of keeping at about 1000°C.
- (5) Thermometer This can measure the temperature of the furnace in (4).
- (6) Porcelain evaporating dish This dish is round bottom type of 150 mm diameter specified in JIS R 1302.

7.4 Quantity of Sampling About 0.5 g of sample shall be taken.

7.5 Operation Determination operation shall be carried out as follows:

- (1) Heat gently about 10 g of sodium hydroxide to dehydrate in a nickel crucible on a burner.
- (2) Weigh out accurately about 0.5 g of the sample to the nearest 0.1 mg in the crucible and, after adding further 4 g of potassium nitrate and put a cap on the crucible, heat it until becoming dark red to fuse it.
- (3) After cooling, transfer the fused substance together with the nickel crucible and cap into a 500 ml nickel beaker in which about 150 ml of water has been put, and dissolve the fused substance in the crucible completely by heating.

- (4) Draw out the crucible and cap from the beaker after rinsing completely with water, and transfer gradually the solution in the beaker to the porcelain evaporating dish in which 30 ml of hydrochloric acid has been placed previously.
- (5) Heat this solution on a water bath finally to evaporate to dryness while mixing by agitating. Moisten this with a little amount of hydrochloric acid, and then evaporate to dryness until there rises scarcely hydrochloride gas.
- (6) After adding 10 ml of hydrochloric acid (1 + 1) and 100 ml of hot water, dissolve soluble salts completely by heating, and immediately filter it through filter paper (Class 5 B). Wash the precipitate with warm water until there is recognized no chloride ion reaction.
- (7) Transfer the filtrate into a porcelain evaporating dish and evaporate to dryness similarly to the operation of (5). After heating it for 1 h at $115 \pm 5^\circ\text{C}$ in an air bath, carry out the operation of (6). Store the filtrate and use it when determining total iron.
- (8) Dry gradually the precipitate of (6) and (7) together with the filter paper in a crucible and, after complete dehydration, incinerate the filter paper at a low temperature.
- (9) Then ignite at about 1000°C in an electric furnace for 1 h and, after cooling sufficiently in a desiccator, weigh its mass.
- (10) After moistening the ash with several drops of sulfuric acid (1 + 1), add 2 or 3 ml of hydrofluoric acid, and heat it on a sand bath with close attention lest the content should scatter away.
- (11) When white fume of sulfuric acid is not noticed, ignite in the electric furnace at about 1000°C for 15 min, and, after cooling sufficiently in a desiccator, weigh its mass. Repeat this operation until constant weight can be obtained. Store the residue left in the platinum crucible and use it when total iron is to be determined.
- (12) The content of total silicon shall be calculated according to the following formula:

$$Si_T (\%) = \frac{(m_2 - m_3) \times 0.4674}{m_1} \times 100$$

where $Si_T (\%)$: content of total silicon (%)

m_1 : quantity of sample taken (g)

m_2 : quantity weighed in (9) (g)

m_3 : quantity weighed in (11) (g)

8. Determination of Total Iron

8.1 Summary In the solution stored in 7.5 (7), let iron and aluminum precipitate by adding aqueous ammonia and ammonium chloride, and dissolve the precipitate by adding warm hydrochloric acid. Reduce ferric ion to ferrous ion by adding hydroxylammonium chloride solution and, after coloring by 1, 10-phenanthroline solution, determine the content of total iron by measuring its absorbance.

8.2 Reagents Reagents to be used shall be as follows:

- (1) Hydrochloric acid (1 + 1, 1 + 2)
- (2) Sulfuric acid (1 + 1)
- (3) Aqueous ammonia (1 + 1)
- (4) Potassium disulfate
- (5) Ammonium chloride
- (6) Ammonium nitrate solution (2% w/v)
- (7) Sodium acetate solution (50% w/v)
- (8) Hydroxylammonium chloride solution (0.5% w/v)
- (9) 1, 10-phenanthroline solution (0.2% w/v)
- (10) Methyl Red solution (0.2% w/v) Dissolve 0.2 g of Methyl Red in 90 ml of ethyl alcohol, and dilute with water to make 100 ml.
- (11) Standard iron solution To be in accordance with 6.2 (5).

8.3 Photometer Either a photoelectric photometer or a photoelectric spectrophotometer shall be used.

8.4 Operation Determination operation shall be carried out as follows:

- (1) To the residue kept in the platinum crucible of 7.5 (11), add 1 to 2 g of potassium disulfate, and fuse them by red-heating. After cooling, dissolve completely the fused substance with a little amount of hydrochloric acid and warm water, and add this solution into the solution stored in 7.5 (7).
- (2) Add about 1 g of ammonium chloride and 2 to 3 drops of nitric acid to the solution obtained in (1), and boil for several min. Add 1 to 2 drops of Methyl Red solution to this solution and, after neutralizing by adding aqueous ammonia (1 + 1), furthermore add 2 to 3 drops excessively. After boiling the solution successively for about 1 min, transfer it on a water bath, and let it stand to get the sedimentation owing to the aggregation of precipitate.
- (3) Immediately filter the precipitate through filter paper (Class 5 B), and wash it with warm ammonium nitrate solution (2% w/v) several times.
- (4) Wash the precipitate down into the original beaker by using a jet of washing bottle, and dissolve the precipitate completely by adding 10 ml of warm hydrochloric acid (1 + 2) from above the filter paper, and wash the paper with warm water 2 to 3 times. Receive the hydrochloric acid and the washings to the beaker in which the precipitate has been put, and dissolve the precipitate completely by heating.

- (5) After cooling, transfer the solution into a 250 ml transfer flask, and add water up to the marked line, to mix by shaking. Carry out the following operation on an aliquot of this solution, and store the remainder to be used for the determination of aluminum.
- (6) Take accurately an aliquot of this solution from 5 to 50 ml into a 100 ml beaker.
- (7) Add sodium acetate solution (50% w/v) into this solution so as to make pH(1) 3.5, and then transfer to a 100 ml transfer flask.
- (8) Add 5 ml of hydroxylammonium chloride solution (0.5% w/v) to the solution, and mix them by shaking.
- (9) Add 5 ml of 1, 10-phenanthroline solution (0.2% w/v), and dilute with water up to the marked line to mix by shaking.
- (10) After 15 min, transfer the solution into a 10 mm absorption cell, and measure the absorbance with reference to a blank test solution in the vicinity of 500 nm wavelength. Obtain the content of total iron by the use of the working curve prepared in advance.
- (11) The content of total iron shall be calculated according to the following formula:

$$Fe_T (\%) = \frac{m_2}{m_1 \times \frac{v}{250}} \times 100$$

where Fe_T (%): content of total iron (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of total iron detected in an aliquot (g)

v : volume of aliquot taken from sample solution (ml)

8.5 Preparation of Working Curve The working curve prepared in 6.6 shall be used.

Remark: When only total iron is to be determined promptly, the following operation shall be carried out.

Weigh accurately about 1.0 g of the sample into a crucible (for instance, No. 30), and, after adding 15 ml of hydrofluoric acid, 1 ml of nitric acid, and 2 ml of sulfuric acid (1 + 1), heat it to evaporate and concentrate on a sand bath with a close attention lest the content should scatter away. When white fume of sulfuric acid begins to appear, let it continue for about 10 min.

After cooling, transfer the crucible as it is into the 300 ml beaker containing 20 ml of hydrochloric acid (1 + 1) and 100 ml of water, and boil for about 10 min to dissolve soluble components. Draw out the crucible after rinsing sufficiently with water. Then obtain the content of total iron according to the operations described in 6.5 (2) and thereafter.

9. Determination of Aluminum

9.1 Division of Determinations Either of the following shall be adopted.

(1) Chrome Azurol S Absorptiometry(2) EDTA Titration Method9.2 Determination of Aluminum by Chrome Azurol S Absorptiometry

9.2.1 Summary From the solution stored in 8.4 (5), separate the components such as iron by adding cupferron and chloroform, and determine the content of aluminum by measuring absorbance of the coloring brought about by adding Chrome Azurol S and hydroxylammonium chloride solution.

9.2.2 Reagents Reagents to be used shall be as follows:

- (1) Sulfuric acid (1 + 1)
- (2) Aqueous ammonia (1 + 1)
- (3) Hydroxylammonium chloride solution (10 % w/v)
- (4) Cupferron solution (6 % w/v) Dissolve 6 g of cupferron in 100 ml of water, and, when producing decomposed substances, filter the solution through filter paper (Class 5 A). Prepare this solution in each case of use.
- (5) Chrome Azurol S solution (0.1 % w/v)
- (6) Chloroform
- (7) Standard aluminum solution (0.1 mg Al/ml) Weigh out 0.100 g aluminum (99.9 % min.) into a 300 ml beaker, and dissolve it by heating after adding 50 ml of hydrochloric acid (1 + 1). After cooling, transfer into a 1 l transfer flask, and dilute up to the marked line with water. This solution contains 0.1 mg of aluminum per 1 ml.
- (8) Standard aluminum solution (0.01 mg Al/ml) Take 10 ml standard aluminum solution (0.1 mg Al/ml) into a transfer flask of 100 ml and dilute it with water up to the marked line.

9.2.3 Operation Determination operation shall be carried out as follows:

- (1) From the solution stored in 8.4 (5), take accurately an aliquot of 50 ml into a 100 ml separatory funnel.
- (2) Add 5 ml⁽³⁾ of sulfuric acid (1 + 1), 10 ml of cupferron solution (6 % w/v), and 15 ml of chloroform, and mix them by shaking violently to separate such component as iron.
- (3) After standing still for about 1 min, discard an organic layer. Next add 10 ml of chloroform to the water layer, and mix them by violently shaking for about 30 s to carry out separation again.
- (4) Repeat once more the operation of (3), and discard the organic layer. Transferring the water layer into a 200 ml beaker, heat it to eliminate chloroform by vaporization, and continue heating to let generate the white fume of sulfuric acid.

- (5) After cooling and adding a little amount of water, transfer it into a 100 ml transfer flask, and dilute up to the marked line with water.
- (6) Take an aliquot⁽⁴⁾ of certain volume in a 200 ml beaker, and make total volume 50 ml by adding water.
- (7) After adding 2 ml of Chrome Azurol S solution (0.1 w/v %)⁽⁵⁾ and 5 ml of hydroxylammonium chloride solution (10 w/v %), control pH to make 5.9 to 6.1 by using aqueous ammonia (1 + 1)⁽⁶⁾.
- (8) Immediately transfer it into a 100 ml transfer flask, and dilute up to the marked line with water, to mix by shaking.
- (9) After letting it stand still for about 10 min⁽⁷⁾, transfer to solution into a 10 mm absorption cell, and measure the absorbance with reference to a blank test solution in the vicinity of 550 nm wavelength. Obtain the content of aluminum by the use of the working curve prepared in advance.
- (10) The content of aluminum shall be calculated according to the following formula:

$$Al (\%) = \frac{m_2}{m_1 \times \frac{50}{250} \times \frac{v}{100}} \times 100$$

where $Al (\%)$: content of aluminum (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of aluminum detected in an aliquot (g)

v : volume of aliquot taken separately from sample solution in (6) (ml)

- Notes ⁽³⁾ The concentration of sulfuric acid used when extracting shall be not less than about 0.5 mol/l (1 N). In this case, that would become about 0.9 mol/l (1.8 N).
- ⁽⁴⁾ In the case of green silicon carbide abrasives (GC), 50 ml of an aliquot is taken separately, and in the case of black silicon carbide abrasives (C), 5 to 10 ml is taken separately.
 - ⁽⁵⁾ Coloring reagent itself indicates the absorbance in the vicinity of 550 nm wavelength. Therefore, an accurate adding is necessary.
 - ⁽⁶⁾ To control pH, use a pH meter, or the Bromocresol Purple (BCP) pH test paper.
 - ⁽⁷⁾ After controlling pH, the maximum absorbance will be shown in about 5 min, and the coloring is stable for about 20 min.

9.2.4 Preparation of Working Curve Place several parts of the standard aluminum solution (0.01 mg Al/ml) from 0 to 3.0 ml (from 0 to 0.03 mg as Al) stepwise into 200 ml beakers, and pour 10 drops of sulfuric acid (1 + 1) and water to make respectively total volume of 50 ml. Then carry out the operations in 9.2.3 (7) and thereafter, and prepare the working curve by plotting the relations between the contents of aluminum and the absorbances.

9.3 Determination of Aluminum by EDTA Titration Method

9.3.1 Summary To the solution stored in 8.4 (5), add excessively disodium ethylenediaminetetraacetate solution (EDTA solution) to control pH, and complete aluminum-EDTA chelate compound by boiling. After cooling, determine the content of aluminum by means of the titration with standard zinc solution by using Xylenol Orange as indicator.

9.3.2 Reagents Reagents to be used shall be as follows:

- (1) Hydrochloric acid (1 + 1)
- (2) Aqueous ammonia (1 + 1)
- (3) Hexamine
- (4) Paranitrophenol solution (0.2 % w/v)
- (5) Xylenol Orange solution (0.1 % w/v)
- (6) Ammonium acetate solution (25 % w/v)
- (7) 0.01 mol/l zinc standard solution: Weigh exactly 0.6537 g of zinc specified in JIS K 8005 (in cases where zinc surface may be oxidized, use the zinc dried at 110°C for 5 min after washing successively by 6 mol/l {6 N} hydrochloric acid, water, and acetone) into a 200 ml beaker, and dissolve by heating with adding about 50 ml of water, 15 ml of hydrochloric acid (2 + 1), and 5 drops of bromine water. After eliminating bromine by boiling, transfer it to a 1 l transfer flask, and dilute up to the marked line with water.
- (8) 0.01 mol/l EDTA solution: Weigh out 3.75 g of disodium ethylenediaminetetraacetate (EDTA) into a 1 l transfer flask, and, after dissolving in water, dilute up to the marked line with water.

9.3.3 Operation Determination operation shall be carried out as follows:

- (1) Out of the solution stored in 8.4 (5), take exactly 100 ml into a 300 ml beaker.
- (2) Add exactly 10 ml of 0.01 mol/l EDTA solution, and dilute up to about 150 ml with water.
- (3) After adding 2 or 3 drops of paranitrophenol solution (0.2 % w/v) as indicator, neutralize it with aqueous ammonia (1 + 1) till the color turns yellow, and immediately add 5 ml of hydrochloric acid (1 + 1).
- (4) Control its pH to make the value 3.0 to 3.5⁽⁸⁾ by adding ammonium acetate solution (25 % w/v), and boil for about 10 min.
- (5) After cooling, control its pH to make the value 5 to 8⁽⁹⁾ by adding about 10 g of hexamine.

- (6) After adding 4 or 5 drops of Xylenol Orange solution (0.1 w/v %) as indicator, titrate it with 0.01 mol/l standard zinc solution, and make it the end point when color turns from yellow to red. Carry out a blank test under the same operation.
- (7) The content of aluminum shall be calculated according to the following formula:

$$Al (\%) = \frac{0.0002698 \times (a-b)}{m \times \frac{100}{250}} \times 100 - [Fe_r (\%) \times 0.4831]$$

where $Al (\%)$: content of aluminum (%)

$Fe_r (\%)$: content of total iron (%)

m : quantity of sample taken (g)

a : volume of 0.01 mol/l standard zinc solution used, equivalent to 10 ml of 0.01 mol/l EDTA solution (ml)

b : volume of 0.01 mol/l standard zinc solution used to titrate excess EDTA solution (ml)

Notes (⁸) To control pH, use a pH meter, or the Bromophenol Blue (BPB) in pH test paper.

(⁹) To control pH, use a pH meter, or the Bromocresol Green (BCG) in pH test paper.

10. Determination of Calcium Oxide

10.1 Summary Place the sample in a crucible, and, after adding hydrofluoric acid, nitric acid and perchloric acid, heat them to dryness. Dissolve soluble salts in hydrochloric acid and warm water, and separate insoluble matter. By means of adding ammonium chloride and aqueous ammonia, separate such components as iron and aluminum, and titrate the filtrate with EDTA solution to determine calcium oxide by using as indicator 2-hydroxy-1-(2'-hydroxy-4'-sulfo-1'-naphthylazo)-3-naphthoic acid (NN).

10.2 Reagents Reagents to be used shall be as follows:

- (1) Nitric acid
- (2) Perchloric acid
- (3) Hydrofluoric acid
- (4) Hydrochloric acid (1 + 1, 2 + 1, 1 + 100)
- (5) Ammonium chloride solution (20 w/v %)
- (6) Aqueous ammonia (1 + 1, 1 + 100)
- (7) Potassium hydroxide solution (20 w/v %)
- (8) Hydroxylammonium chloride solution (1 w/v %)

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- (9) Potassium cyanide solution (10 % w/v)
- (10) NN diluted powder: Place 1.0 g of 2-hydroxy-1-(2'-hydroxy-4'-sulfo-1'-naphthylazo)-3-naphthoic acid as indicator and 50 g of potassium sulfate in a mortar, and pulverize to mix them well.
- (11) 0.01 mol/l EDTA solution: Weigh out 3.75 g of EDTA in a 1 l transfer flask, and, after dissolving it in water, dilute the solution up to the marked line with water. Carry out the standardization of this solution by using 0.01 mol/l standard zinc solution in accordance with 4.4 (5.3) in JIS K 8001, as appropriate.

10.3 Crucible A crucible is, for instance, No. 30.

10.4 Quantity of Sampling About 1.0 g of sample shall be weighed out.

10.5 Operation Determination operation shall be carried out as follows:

- (1) Weigh out accurately about 1.0 g of the sample to the nearest 0.1 mg in a crucible, and, after adding 10 ml of hydrofluoric acid, 1 ml of nitric acid and 3 ml of perchloric acid, heat them on a sand bath until no white fume of perchloric acid is generated any more.
- (2) After cooling, transfer the sample together with the crucible into the 300 ml beaker containing 5 ml of hydrochloric acid (1 + 1) and 150 ml of warm water and dissolve the soluble salts in the crucible by heating.
- (3) After drawing out the crucible from the beaker after washing sufficiently with water, filter the solution through filter paper (Class 5 B). Wash the residue with hydrochloric acid (1 + 100) several times, and then with warm water, until no reaction of chloride ion is recognized. Receive the filtrate and washings in a 300 ml beaker.
- (4) Make the solution slightly alkaline by adding 5 ml of ammonium chloride solution (20 % w/v) and aqueous ammonia (1 + 1). Heat gently to let the hydroxides of iron and aluminum precipitate. Filter it through filter paper (Class 5 B) into a 250 ml transfer flask, and wash the precipitate by using aqueous ammonia (1 + 100) several times. Dilute the filtrate and washings in the transfer flask with water up to the marked line and shake to mix. Carry out the following operation on an aliquot of the solution, and store the remainder for the determination of magnesium oxide.
- (5) Take accurately an aliquot of 100 ml into a 300 ml beaker, and control pH to make the value 12.6 to 13.3 by means of dropping of potassium hydroxide solution (20 % w/v) (10).
- (6) Add 5 ml of hydroxylammonium chloride solution (1 % w/v) and 5 ml of potassium cyanide solution (10 % w/v).
- (7) After adding about 0.1 g of NN diluted powder as indicator, titrate the solution with 0.01 mol/l EDTA solution, and make it the end point when color turns completely from red to blue. Carry out a blank test under the same operation.

- (8) The content of calcium oxide shall be calculated according to the following formula:

$$\text{CaO (\%)} = \frac{(a-b) \times f \times 0.000561}{m \times \frac{100}{250}} \times 100$$

where CaO (%): content of calcium oxide (%)

f : factor of 0.01 mol/l EDTA solution

a : volume of 0.01 mol/l EDTA solution used for titration of sample (ml)

b : volume of 0.01 mol/l EDTA solution used for titration of blank test (ml)

m : quantity of sample taken (g)

Note (¹⁰) To control pH, use a pH meter, or the Alkali Blue (ALB) or Poirrier's Blue (POB) in pH test paper.

11. Determination of Magnesium Oxide

11.1 Summary To the solution stored in 10.5 (4) add Eriochrome Black T as indicator, and titrate the solution with EDTA solution to determine magnesium oxide.

11.2 Reagents Reagents to be used shall be as follows:

- (1) Ammonium chloride-ammonia buffer solution (pH 10) Dissolve 70 g of ammonium chloride in the mixture of 570 ml of aqueous ammonia and about 200 ml of water, and then dilute it to make 1 l with water.
- (2) Hydroxylammonium chloride solution (1 w/v %)
- (3) Potassium cyanide solution (10 w/v %)
- (4) Eriochrome Black T solution (0.5 w/v %) Dissolve 0.5 g of Eriochrome Black T in 100 ml of methyl alcohol, and add 0.5 g of hydroxylammonium chloride. Store this solution in a brown bottle with tight stoppering.
- (5) 0.01 mol/l EDTA solution: To be in accordance with 10.2 (11)

11.3 Operation Determination operation shall be carried out as follows:

- (1) Out of the solution stored in 10.5 (4), take accurately an aliquot of 100 ml into a 300 ml beaker.
- (2) Control pH to make the value 10(¹¹), by adding ammonium chloride-ammonia buffer solution.
- (3) Add 5 ml of hydroxylammonium chloride solution (1 w/v %) and 5 ml of potassium cyanide solution (10 w/v %) to the solution.
- (4) After adding 2 or 3 drops of Eriochrome Black T solution (0.5 w/v %) as indicator, titrate the solution with 0.01 mol/l EDTA solution, and make it the end point when color turns completely from red to blue. Carry out a blank test under the same operation.

- (5) The content of magnesium oxide shall be calculated according to the following formula:

$$MgO (\%) = \frac{(A-B) \times f \times 0.000403}{m \times \frac{100}{250}} \times 100$$

where $MgO (\%)$: content of magnesium oxide (%)
 f : factor of 0.01 mol/l EDTA solution
 A : volume obtained by subtracting volume of 0.01 mol/l EDTA solution used for the titration of blank test (ml) from that used for sample titration (ml)
 B : value of ($a-b$) at titration for calcium oxide
 m : quantity of sample taken (g)

Note (¹¹) To control pH, use a pH meter, or Alizarin Yellow (AZY) in pH test paper.

12. Determination of Total Carbon

12.1 Division of Methods Either one of the following shall be adopted:

- (1) Combustion Volumetric Method
- (2) Coulometric Method

12.2 Determination of Total Carbon by Combustion Volumetric Method

12.2.1 Summary Let carbon component completely change to carbon dioxide by oxidation owing to the combustion of the sample with ignition improver in an oxygen flow, and measure the volume of all produced gas together with oxygen after collecting in a buret. After absorbing and eliminating the carbon dioxide, measure the volume of remaining gas and then determine the content of total carbon from the decreased volume of gas.

12.2.2 Reagents Reagents to be used shall be as follows:

- (1) Oxygen This shall be as specified in JIS K 1101.
- (2) Ignition improver(¹²)
- (3) Sodium chloride solution (26 w/v %)
- (4) Potassium hydroxide solution (33 w/v %)
- (5) Sulfuric acid (1 + 1)
- (6) Potassium permanganate solution (5 w/v %)
- (7) Methyl Red solution (0.2 w/v %) To be in accordance with 8.2 (10).

Note (¹²) Choose from the four: The mixture of 2 g of tin-and-lead alloy (1 + 13) and 1 g of copper wire, the mixture of 2 g of trilead tetraoxide and 0.2 g of quick lime, copper coated by tin or 1 g of metallic tin powder shall be used.

12.2.3 Devices and Apparatus Devices and apparatuses to be used shall be as follows (refer to Attached Fig. 1):

- (1) Oxygen Supplying Device This shall have the capacity of approximately 20 l.
- (2) Oxygen Cleaning Device This device cleans and dries oxygen gas by removing carbon dioxide, organic gases, etc. contained in original oxygen gas, and has a construction connecting⁽¹⁴⁾ in series such as the washing bottle (c) provided with chromic acid saturated sulfuric acid⁽¹³⁾, the tube or tower (d) packed with soda lime, soda asbestos or sodium hydroxide, and the washing bottle or tower (e) provided with sulfuric acid or active alumina.
- (3) Combustion Furnace The tubular type electric furnace (g) of about 30 mm in inside diameter and 300 mm to 350 mm in length shall be used, and have the ability to keep the temperature at 1350°C or higher along the length of about 150 mm near its center.
- (4) Porcelain Combustion Tube (h) The porcelain combustion tube such as CT 1 or CT 2 specified in JIS R 1307 shall be used, and asbestos wool is to be packed at its center back where a porcelain combustion boat is inserted.
- (5) Porcelain Combustion Boat The porcelain combustion boat such as CB 1 specified in JIS R 1306 shall be used, and it is necessary to pre-burn it in advance at the same temperature as that of test in oxygen flow.
- (6) Thermometer (i) Thermoelectric thermometer that can measure the temperature in the range from 1300 to 1400°C shall be used.
- (7) Gas Analyzer Gas analyzer shall be constructed by connecting in series such as the following:
 - (a) Absorption Bottle for Sulfur Dioxide (j) This is supplied with about 5 ml of acidic solution that is the mixture of 10 ml of potassium permanganate (5 % w/v) and 1 ml of sulfuric acid (1 + 1).
 - (b) Cooling Tube (k)
 - (c) Three Way Cock (l) One way is to be opened towards outside air.
 - (d) Three Way Cock (m)
 - (e) Buret (n) This is supplied with sodium chloride solution (26 % w/v) which has been made slightly acidic by dropping sulfuric acid to such degree as the added Methyl Red solution shows red color. The buret has about 800 ml in whole volume, 90 ml in graduated length, and 0.2 ml of the scale mark. The scale shall be graduated by taking 16°C and 760 mmHg {101.3 kPa} as standard.
 - (f) Leveling Bulb (o) The capacity is 850 ml.