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**Methods for chemical analysis of fine
silicon carbide powders for fine ceramics**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Advanced Industrial Science and Technology (AIST) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS R 1616 : 1994** is replaced with this Standard.

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Methods for chemical analysis of fine silicon carbide powders for fine ceramics

1 Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of fine silicon carbide powders used as the raw material for manufacture of fine ceramics.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

- JIS K 0050 *General rules for chemical analysis*
- JIS K 0115 *General rules for molecular absorptiometric analysis*
- JIS K 0116 *General rules for atomic emission spectrometry*
- JIS K 0127 *General rules for ion chromatographic analysis*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8007 *General rule for test methods of highly purified reagents*
- JIS R 6003 *Method for sampling of abrasive grains*
- JIS Z 8401 *Guide to the rounding of numbers*

3 General items

General items common to the analysis method shall be in accordance with JIS K 0050, JIS K 0115, JIS K 0116, JIS K 0127, JIS K 8001 and JIS K 8007.

4 Analysis items

Analysis items specified in this Standard shall be as follows.

- a) Total silicon (T.Si)
- b) Total carbon (T.C)
- c) Free silicon (F.Si)
- d) Free silicone dioxide (F.SiO₂)

- e) Free carbon (F.C)
- f) Aluminium (Al)
- g) Iron (Fe)
- h) Calcium (Ca)
- i) Magnesium (Mg)
- j) Oxygen (O)
- k) Nitrogen (N)
- l) Fluorine (F)
- m) Chlorine (Cl)

5 Sampling and handling of sample

5.1 Sampling

Take analytical samples in accordance with **JIS R 6003**.

5.2 Handling of sample

Put about 10 g of analytical sample into a flat-type weighing bottle (50 mm × 30 mm). Dry it in a air bath at $135\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h. Cool it as it is in a desiccator (desiccant : magnesium perchlorate for drying). Take out the analytical sample of necessary amount quickly from this ¹⁾.

Note ¹⁾ If a sample increases mass while weighing out, dry necessary amount whenever analysis is made. The difference of mass of a weighing bottle between before and after measuring shall be the mass of sample. This procedure is not applicable to measuring to a capsule.

5.3 Weighing method of sample

Weigh out the analytical sample of the required quantity to the nearest 0.1 mg using a chemical balance.

6 Obtaining analytical values

6.1 Number of analyses

Analyze the same component twice on different days.

6.2 Blank test

Upon analysis, perform a blank test to correct the measured values.

6.3 Indication of analytical values

The analytical values shall be indicated in % (mass fraction) in dry state, and rounded off as follows in accordance with **JIS Z 8401**.

- a) For the total silicon, total carbon and free carbon, round off the figure to the first decimal place.
- b) For the free silicon, free silicone dioxide, oxygen, nitrogen, fluorine and chlorine, round off the figure to the second decimal place.
- c) For the aluminium, iron, calcium and magnesium, round off the figure to the third decimal place.

6.4 Investigation and adoption of analytical values

- a) When the difference between two analytical values does not exceed the tolerance shown in table 1, the average value shall be reported.
- b) When the difference between two analytical values exceeds the tolerance shown in table 1, perform additional two analyses. When the difference does not exceed the tolerance, the average value thereof shall be reported. If the difference also exceeds the tolerance, the median of four analytical values shall be reported.

Table 1 Tolerances on analytical values

Unit : % (mass fraction)							
Component	T.Si	T.C	F.Si	F.SiO ₂	F.C	Al	Fe
Tolerance	0.20	0.36	0.026	0.036	0.12	0.003 2	0.002 6
Component	Ca	Mg	O	N	F	Cl	
Tolerance	0.001 5	0.000 3	0.094	0.023	0.005	0.002	

7 Determination method of total silicon

7.1 Classification of determination method

The total silicon shall be determined in either of the following methods.

- a) Dehydrated mass determination and ICP (inductively coupled plasma, hereafter referred to as "ICP") emission spectrometry combined method : This method shall apply to the sample of which the total content rate of silicon is 40 % (mass fraction) or over to and including 80 % (mass fraction).
- b) Agglomerated mass determination and ICP emission spectrometry combined method : This method shall apply to the sample of which the total content rate of silicon is 40 % (mass fraction) or over to and including 80 % (mass fraction).

7.2 Dehydrated mass determination and ICP emission spectrometry combined method

7.2.1 Summary

Fuse the sample with sodium carbonate and dissolve it with hydrochloric acid, dehydrate silicate by evaporating to dryness, dissolve soluble salts with hydrochloric acid and filtrate. Ignite and weigh the precipitate, add hydrofluoric acid to vaporize silicon dioxide, then ignite again and weigh it. Obtain the amount of primary amount of silicon dioxide from the weight loss. Obtain the dissolved silicon amount with ICP emission spectrometry by taking filtrate. Calculate the total silicon content rate from both.

7.2.2 Reagents

Reagents used in this Standard shall be the highest purity of products on the market, and as follows. Reagent solutions shall be preserved in plastic bottles. The standard solutions shall be the products on the market conforming to National Measurement Standards Provision System (Japan Calibration Service System) or the equivalent products, and those traceable to SI units shall be used.

- a) **Water** A3 or superior specified in table 1 of **JIS K 0557 : 1998**.
- b) **Hydrochloric acid**
- c) **Hydrochloric acid (1 + 1, 1 + 4, 1 + 50)**
- d) **Hydrofluoric acid**
- e) **Sulfuric acid (1 + 1, 1 + 4)**
- f) **Sodium carbonate (anhydrous)**
- g) **Standard silicon solution (1.0 mg Si/ml)**
- h) **Standard silicon solution (0.1 mg Si/ml)** Prepare by taking exactly 10 ml from the solution of g) in a 100 ml volumetric flask every time when necessary, diluting it with water up to the marked line and shaking to mix it.

7.2.3 Containers

For containers to be used for various operations, glass products or plastic products ²⁾ shall be used. Meters made of plastics shall be verified beforehand. Glass products shall not be used for other operation than that specified. For the washing and preservation of containers, remove contaminants, wash with water sufficiently and preserve by filling with water in accordance with (1), (2) and (3) of clause 6 of **JIS K 8007 : 1992**.

Note ²⁾ Products made of polyethylene, polypropylene, tetrafluoroethylene resin and the like are available.

7.2.4 Weighing amount of sample

The weighing amount of sample shall be 0.30 g.

7.2.5 Operation

The determination operation shall be according to the following procedure.

- a) Weigh out the sample in a platinum dish (for example, No. 75), add 2.0 g of sodium carbonate (anhydrous) and mix, and spread it uniformly over the bottom of dish. Keep the temperature (700 °C to 800 °C) where silicon carbide is not combusted and sodium carbonate is not fused, and after oxidizing silicon carbide as much as possible ³⁾, ignite at about 1 000 °C to fuse it completely.
- b) Cool it as it is by covering with a watch glass and add 20 ml of hydrochloric acid (1 + 1). Heat and dissolve it on a water bath, wash the watch glass with small amount of water and remove, then evaporate it to dryness. During this, use a glass rod with a flattened tip to crush the deposited salts into powders at times. Cool it as it is, add 5 ml of hydrochloric acid and leave it for about 1 min, add 20 ml of hot

water and heat it on a water bath for about 5 min to dissolve soluble salts.

- c) Filtrate the insoluble residue with filter paper (Class 5 B), and wash it with hot hydrochloric acid (1 + 50) several times and with hot water sufficiently. Receive the filtrate and washings in a 250 ml volumetric flask, cool it as it is, dilute it with water up to the marked line and shake to mix it.
- d) Put the precipitate of c) into a platinum crucible (for example, No. 30) with filter paper, add 5 drops of sulfuric acid (1 + 4), heat it by keeping not to catch fire and reduce the filter paper to ashes, then ignite it at $1\,100\,^{\circ}\text{C} \pm 25\,^{\circ}\text{C}$ for 60 min. After cooling it as it is in a desiccator, weigh out the mass. Repeat this operation until reaching the constant mass.
- e) Moisten silicon dioxide of d) with water, add 3 drops of sulfuric acid (1 + 1) and 10 ml of hydrofluoric acid, and evaporate it to dryness on a sand bath. Ignite it at $1\,100\,^{\circ}\text{C} \pm 25\,^{\circ}\text{C}$ for 5 min, cool it as it is in a desiccator and weigh out the mass. The amount of loss shall be the primary silicon dioxide.
- f) Take exactly 10 ml from the solution of c) in a 100 ml volumetric flask, add 5 ml of hydrochloric acid (1 + 4), dilute it with water up to the marked line, and shake to mix it.
- g) Spray a portion of the solution of f) into the argon plasma of ICP emission spectrometer and measure the emission intensity, for example, at 251.61 nm in wavelength ⁴⁾.

Notes ³⁾ When an electric furnace is used, see 12.2.5 a).

- ⁴⁾ For the determination of the dissolved silicon, molybdenum blue absorptiometry may be used. In this case, take exactly 10 ml from the solution of c) in a plastic beaker (100 ml), add 2 ml of hydrofluoric acid (1 + 9), leave it for 10 min as it is, add 50 ml of boric acid solution and 2 ml of hydrochloric acid (1 + 4), and dilute it with water up to about 70 ml. Add 3 ml of hexaammonium heptamolybdate solution, stir it and leave it for 10 min as it is. Add 5 ml of tartaric acid solution and 2 ml of L-ascorbic acid solution sequentially and stir them each time. Transfer it into a 100 ml volumetric flask, dilute it with water up to the marked line, shake to mix it and leave it for 30 min as it is. Take a portion of this solution in a cell (1 cm) of absorptiometer and measure the absorbance at the neighbourhood of 650 nm in wavelength by taking the water as the reference solution.

In this case, the reagents shall be as follows.

- 1) **Hydrofluoric acid (1 + 9)**
- 2) **Boric acid solution (40 g/L)**
- 3) **Hexaammonium heptamolybdate solution** Dissolve 50 g of hexaammonium heptamolybdate tetrahydrate in warm water, filtrate it if necessary, and make it 500 ml with water. When any molybdate is precipitated during preservation, prepare it newly.
- 4) **Tartaric acid solution (100 g/L)**
- 5) **L-ascorbic acid solution (100 g/L)** Preserve it at a cool and dark

place. When two weeks elapsed after preparation, it shall not be used.

7.2.6 Blank test

Perform operations of 7.2.5 without using the sample, provided that the fusing operation is omitted. Take the solution corresponding to 7.2.5 c) as the blank test solution.

7.2.7 Preparation of working curve

Take exactly 10 ml from the blank test solution of 7.2.6 in several 100 ml volumetric flasks and add 0 ml to 4 ml stepwise and exactly from the standard silicon solution respectively. For each, add 5 ml of hydrochloric acid (1 + 4) gradually, dilute it with water up to the marked line and shake to mix it. Perform operations of 7.2.5 g) by using these solutions for working curve, prepare the relation curve between the emission intensity obtained and the amount of silicon, translate it so as to pass the origin, and take it as the working curve ⁵⁾.

Note ⁵⁾ When using the molybdenum blue absorptiometry, it shall be as follows. Take exactly 0 ml to 3 ml stepwise from the standard silicon solution in several plastics beakers (100 ml) and add exactly 10 ml from the blank test solution respectively. For each, perform subsequent operations of addition of hydrofluoric acid (1 + 9) of Note ⁴⁾ of 7.2.5 g), prepare the relation curve between the absorbance obtained and the amount of silicon, translate it so as to pass the origin, and take it as the working curve.

7.2.8 Calculation

Obtain the amount of dissolved silicon from the emission intensities obtained in 7.2.5 g) and 7.2.6 and the working curve prepared in 7.2.7, and calculate the total silicon content rate in sample according to the following equation.

$$T.Si = \frac{[(m_1 - m_2) \times 0.4674 + (A_1 - A_2) \times 250/10]}{m} \times 100$$

where, $T.Si$: Total silicon content rate [% (mass fraction)]

m_1 : Amount of loss of 7.2.5 e) (g)

m_2 : Amount of loss of 7.2.6 (g)

A_1 : Amount of dissolved silicon in taken sample solution (g)

A_2 : Amount of dissolved silicon in taken blank test solution (g)

m : Weighing amount of sample (g)

7.3 Agglomerated mass determination and ICP emission spectrometry combined method

7.3.1 Summary

Fuse the sample with sodium carbonate and boric acid, dissolve it with hydrochloric acid, agglomerate silicate by adding polyethylene oxide and filtrate. Ignite and weigh the precipitate, add hydrofluoric acid to vaporize silicon dioxide, then ignite again and weigh it. Obtain the amount of primary amount of silicon dioxide from the weight loss.

Obtain the dissolved silicon amount with ICP emission spectrometry by taking filtrate. Calculate the total silicon content rate from both.

7.3.2 Reagents

Reagents shall be in accordance with 7.2.2 and as given below. The reagent of b) shall be preserved in a plastic bottle.

- a) **Boric acid**
- b) **Polyethylene oxide solution (0.5 g/L)** Dissolve 0.1 g of polyethylene oxide gradually in 200 ml of water by stirring. When two weeks elapsed after preparation, it shall not be used.
- c) **Cellulose powder**

7.3.3 Containers

In accordance with 7.2.3.

7.3.4 Weighing amount of sample

Same as 7.2.4.

7.3.5 Operation

The determination operation shall be according to the following procedure.

- a) Weigh out the sample in a platinum dish (for example, No. 75), add 2.0 g of sodium carbonate (anhydrous) and 0.3 g of boric acid and mix, and fuse it in accordance with 7.2.5 a) ^{3), 6)}.
- b) Cool it as it is by covering with a watch glass, then add 25 ml of hydrochloric acid (1 + 1). Heat and dissolve it on a water bath until it becomes jelly-like, remove the watch glass without washing, add 0.05 g of cellulose powder and 10 ml of polyethylene oxide solution, then stir and leave them for about 5 min.
- c) Filtrate the insoluble residue with filter paper (Class 5 B), wash the platinum dish and watch glass with hot hydrochloric acid (1 + 50) several times and with hot water sufficiently. Receive the filtrate and washings in a 250 ml volumetric flask, cool it as it is, dilute it with water up to the marked line and shake to mix it.
- d) Perform operations of d) and e) of 7.2.5 on the precipitate and filter paper of c) in the same way.
- e) Take exactly 10 ml from the solution of c) and perform operations of f) and g) of 7.2.5 in the same way ⁷⁾.

Notes ⁶⁾ When fusing time is too long, the fused substance becomes less soluble in hydrochloric acid.

⁷⁾ For the determination of dissolved silicon, molybdenum blue absorptiometry may be used. In this a case, perform operations of Note ⁴⁾ of 7.2.5 g) in the same way provided that the addition amount of hydrochloric acid (1 + 4) of 7.2.5 f) shall be 1 ml.

7.3.6 Blank test

Perform operations of 7.3.5 without using the sample, provided that the fusing operation is omitted. Take the solution corresponding to 7.3.5 c) as the blank test solution.

7.3.7 Preparation of working curve

Take exactly 10 ml from the blank test solution of 7.3.6 in several 100 ml volumetric flasks respectively, then perform subsequent operations of the addition of standard silicon solution of 7.2.7.

7.3.8 Calculation

In accordance with 7.2.8.

8 Determination method of total carbon

8.1 Classification of determination method

The total carbon shall be determined in either of the following methods.

- a) Combustion (resistive heating) – infrared ray absorption method : This method shall apply to the sample of which the content rate of total carbon is 0.01 % (mass fraction) or over to and including 35 % (mass fraction).
- b) Combustion (high-frequency heating) – infrared ray absorption method (or thermal conductivity method) : This method shall apply to the sample of which the content rate of total carbon is 0.01 % (mass fraction) or over to and including 35 % (mass fraction).

8.2 Combustion (resistive heating) – infrared ray absorption method

8.2.1 Summary

Burn the sample in oxygen flow by resistive heating with the fire firework work fuel. The generated carbon dioxide (and carbon monoxide) is sent to an infrared ray analyzer with oxygen. The change of infrared ray absorption quantity is measured.

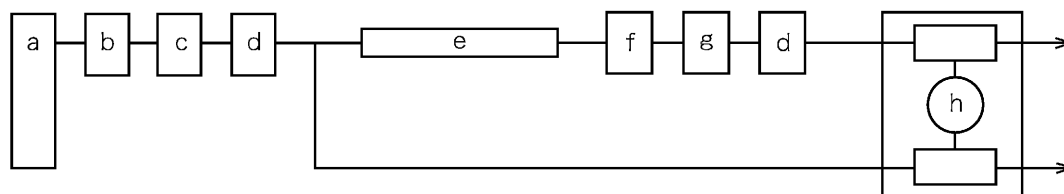
8.2.2 Material and reagent

Material and reagent shall be as follows.

- a) **Oxygen** 99.9 % (volume fraction) or over.
- b) **Firework fuel** Tin in chipped condition.
- c) **Combustion boat** In general, a porcelain combustion boat for chemical analysis, CB 1, 13.5 mm × 10 mm × 80 mm shall be used. It shall be heated without a sample at 1 350 °C in advance.
- d) **Calcium carbonate** 99.9 % (mass fraction) or over. It shall be heated at 500 °C to 550 °C for 2 h, and cooled in a desiccator as it is.

8.2.3 Apparatus

A carbon determination analyzer shall be used. It consists of an oxygen cleaner, sample burner, combustion gas refiner, gas measuring device, etc. The block diagram is shown in figure 1.



- | | |
|------------------------------------|--|
| a : oxygen container | b : oxidation tube with electric furnace |
| c : carbon dioxide absorption tube | d : dehydration tube |
| e : combustion tube | f : dust collecting tube |
| g : desulfurization tube | h : infrared ray analyzer |

Figure 1 Block diagram of carbon determination analyzer
Combustion (resistive heating) – Infrared ray absorption method

- a) **Oxygen cleaner** Consists of an oxidation tube with electric furnace (copper oxide), carbon dioxide absorption tube (sodium hydroxide grains for gas analysis), dehydration tube (magnesium perchlorate for drying), etc.⁸⁾.
- b) **Sample burner** Consists of a tubular electric furnace, porcelain combustion tube, etc.

The tubular electric furnace shall be capable of keeping the middle portion 150 mm or longer of the combustion tube at 1 350 °C.

- c) **Combustion gas refiner** Consists of a dust collecting tube (glass wool), desulfurization tube (manganese dioxide), oxidation tube with electric furnace (copper oxide), dehydration tube (magnesium perchlorate for drying), etc.⁹⁾.
- d) **Gas measuring device** Consists of an infrared ray analyzer for carbon dioxide and other components.

The infrared ray analyzer for carbon dioxide takes out the difference of infrared ray absorption amount by carbon dioxide between the sample cell and the reference cell as electric signals by means of its detector, converts into values corresponding to carbon quantity by a linearizing circuit and integrating circuit, and indicates on an integrating indicator ¹⁰⁾.

Notes ⁸⁾ Some apparatuses do not use the cleaner.

⁹⁾ Some apparatuses have no desulfurization tube nor oxidation tube.

¹⁰⁾ Some apparatuses measure separately the infrared ray absorption amount by carbon dioxide and that by carbon monoxide, convert both values into carbon amount, and obtain the resultant sum.

8.2.4 Weighing amount of sample

The weighing amount of sample shall be 0.10 g.

8.2.5 Operation

The determination operation shall be according to the following procedure.

- a) Switch on the apparatus. Raise the temperature of combustion tube to 1 350 °C, and wait for the gas measuring device to stabilize. Flow oxygen at the specified pressure and flow rate, and perform an airtightness test. The airtightness test and other detailed operation procedures shall follow the operation manual of the apparatus.
- b) Take the sample on the combustion boat and spread it over uniformly. Cover the sample uniformly with 2 g of tin, or mix the sample with tin and spread the mixture uniformly, or sandwich the sample with 1 g of tin each and spread it.
- c) Open the cock at the entrance of combustion tube. Insert the boat loaded with the sample and firework fuel to the middle of combustion tube. Close the cock immediately to ensure airtightness and flow oxygen. Read the value of integrating indicator (hereafter referred to as “integral value”) after the specified time.

8.2.6 Blank test

Perform operations of 8.2.5 without using the sample.

8.2.7 Calculation of calibration coefficient

Use 0.250 g of calcium carbonate or 0.100 g of silicon carbide sample whose total carbon content is known (sample for calibration). Perform operations of 8.2.5 and calculate the calibration coefficient by the following equations. When using calcium carbonate, tin need not be added. As the sample for calibration, use the one of which the time required for combustion is not greatly different from that of the analyte sample.

- a) When calcium carbonate is used

$$K = \frac{G \times 0.1200}{A_1 - A_2}$$

where, K : Calibration coefficient (g/integral value)
 G : Weighing amount of calcium carbonate (g)
 A_1 : Integral value of calcium carbonate
 A_2 : Integral value of 8.2.6

- b) When sample for calibration is used

$$K = \frac{G \times P / 100}{A_1 - A_2}$$

where, K : Calibration coefficient (g/integral value)
 G : Weighing amount of sample for calibration (g)
 P : Total carbon content rate of sample for calibration [% (mass fraction)]

A_1 : Integral value of sample for calibration
 A_2 : Integral value of 8.2.6

8.2.8 Calculation

Calculate the total carbon content rate in sample according to the following equation ¹¹⁾.

$$T.C = \frac{(A - A_2) \times K}{m} \times 100$$

where, $T.C$: Total carbon content rate [% (mass fraction)]

A : Integral value of 8.2.5 c)

A_2 : Integral value of 8.2.6

K : Calibration coefficient (g/integral value)

m : Weighing amount of sample (g)

Note ¹¹⁾ An apparatus available on the market memorizes automatically the blank test value, calibration coefficient, etc. If the weighing amount of sample is registered, the carbon content rate will be indicated.

8.3 Combustion (high-frequency heating) –infrared ray absorption method (or thermal conductivity method)

8.3.1 Summary

Burn the sample in oxygen flow by high-frequency heating with the firework fuel. The generated carbon dioxide is adsorbed to a trap¹²⁾. Next, the trap is heated to desorb carbon dioxide. Then, the carbon dioxide is sent to an infrared ray analyzer with oxygen and the change of infrared ray absorption amount is measured ¹³⁾.

Notes ¹²⁾ There is an apparatus measuring the change of absorption amount of infrared ray without trapping and fed directly to an infrared ray analyzer.

¹³⁾ For the apparatus for the thermal conductivity method, the carbon dioxide desorbed is fed to a thermal conductivity analyzer with oxygen and the change of thermal conductivity is measured.

8.3.2 Material and reagent

Material and reagent shall be as follows.

- a) **Oxygen** Same as 8.2.2 a).
- b) **Firework fuel** Copper, iron or tungsten in chipped condition.
- c) **Combustion crucible and receiving base** In general, a high-frequency crucible FC 2 26 mm and a receiving base FCB 1 shall be used.

8.3.3 Apparatus

A carbon determination analyzer shall be used. It consists of an oxygen cleaner, sample burner, combustion gas refiner, gas measuring device, etc. The block diagram is shown in figure 2.

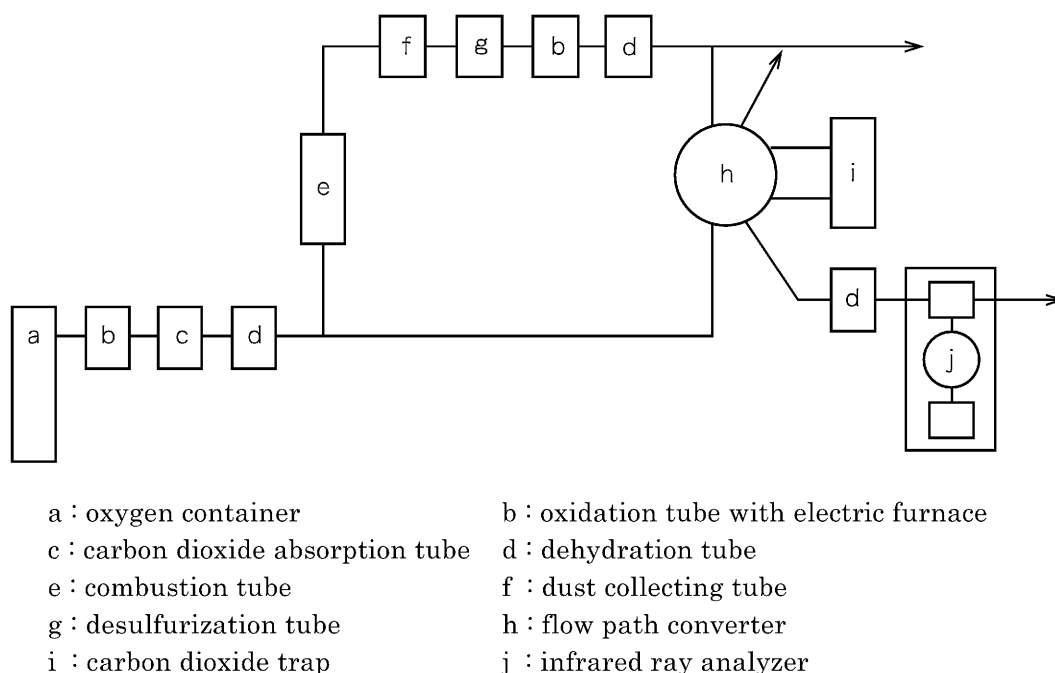


Figure 2 Block diagram of carbon determination analyzer
combustion (high-frequency heating) – Infrared ray ab-
sorption method

- a) **Oxygen cleaner** Same as 8.2.3 a).
b) **Sample burner** Consists of a high-frequency heating furnace, high-frequency transmitter, etc.

The high-frequency heating furnace consists of a heating copper coil, quartz glass combustion tube, etc. The combustion crucible shall be placed on the receiving base and maintained in the middle of the coil by a supporting bar.

- c) **Combustion gas refiner** Same as 8.2.3 c).
d) **Gas measuring device** Consists of a flow path converter, carbon dioxide trap (synthetic zeolite), infrared ray analyzer (or thermal conductivity analyzer), etc.

The flow path converter changes the oxygen flow path when carbon dioxide is trapped and released.

The carbon dioxide trap is housed in a thermostat and kept at 50 °C or lower at the time of trap, and at about 300 °C at the time of release.

When using the infrared ray analyzer, the difference of infrared ray absorption amount by carbon dioxide between the sample cell and the reference cell is taken out as electric signals by means of its detector, converted into values corresponding to carbon quantity by a linearizing circuit and integrating circuit, and indicated on an integrating indicator.

When using the thermal conductivity analyzer, the difference of electric resis-

tance by carbon dioxide between the sample cell and the reference cell is taken out as electric signals by means of its detector, converted into values corresponding to carbon quantity by a linearizing circuit and integrating circuit, and indicated on an integrating indicator.

8.3.4 Weighing amount of sample

Same as 8.2.4.

8.3.5 Operation

The determination operation shall be according to the following procedure.

- a) Switch on the apparatus and set various parts of apparatus at the specified conditions. Wait for the various parts of apparatus to stabilize and perform an airtightness test. The airtightness test and other detailed operation procedures shall follow the operation manual of the apparatus.
- b) Take the sample of specified amount in a combustion crucible. Add 1 g of copper and 1 g of iron, or add 1 g of copper and 1 g of tungsten. Place the combustion crucible on the receiving base, maintain it at the specified position by using a supporting bar and flow oxygen at the specified pressure and flow rate. Operate the high-frequency furnace for the specified period, send the combustion gas to the trap ¹⁴⁾ to make carbon dioxide adsorb.
- c) Change the oxygen flow path, heat the trap for the specified period to make carbon dioxide release, and send it to the infrared ray analyzer (or the thermal conductivity analyzer) with oxygen ¹¹⁾, and read the integral value.

Note ¹⁴⁾ The apparatus available on the market is capable of performing these operations automatically by setting a timer.

8.3.6 Blank test

Perform operations of 8.3.5 without using the sample.

8.3.7 Calculation of calibration coefficient

In accordance with 8.2.7.

8.3.8 Calculation

In accordance with 8.2.8.

9 Determination method of free silicon

9.1 Determination method

Free silicon shall be determined by the hydrogen gas generation – gas volume method. This method shall apply to the sample of which the content rate of free silicon is 0.01 % (mass fraction) or over to and including 1 % (mass fraction).