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**General rules for determination
of carbon in metallic materials**

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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 The Japan Society for Analytical Chemistry (JSAC)/ Japanese Standards Association (JSA) 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 Z 2615** : 2006 is replaced with this Standard.

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General rules for determination of carbon in metallic materials

1 Scope

This Japanese Industrial Standard specifies the general rules that are commonly applied to the determination of carbon in metallic materials.

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 0113 *General rules for methods of potentiometric, amperometric, coulometric, and Karl Fischer titrations*
- JIS K 0130 *General rules for electrical conductivity measuring method*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0212 *Technical terms for analytical chemistry (optical part)*
- JIS K 0213 *Technical terms for analytical chemistry (Electrochemistry part)*
- JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1101 *Oxygen*
- JIS K 8001 *General rule for test methods of reagents*
- JIS R 1306 *Porcelain combustion boats for chemical analysis*
- JIS R 1307 *Porcelain combustion tubes for chemical analysis*
- JIS Z 2616 *General rules for determination of sulfur in metallic materials*
- JIS Z 8402-1 *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*
- JIS Z 8402-2 *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- JIS Z 8402-3 *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*
- JIS Z 8402-4 *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

- JIS Z 8402-5 *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*
- JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*
- JIS Z 8801-1 *Test sieves — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0211**, **JIS K 0212**, **JIS K 0213** and **JIS K 0215** and the following apply.

3.1 lance

a carrier pipe which supplies oxygen efficiently to just above the sample for accelerating the combustion of sample

4 General matters

The general matters shall be as follows.

- a) General matters of chemical analysis shall be in accordance with **JIS K 0050**.
- b) General matters of electrical conductivity measuring method shall be in accordance with **JIS K 0130**.
- c) General matters of coulometric titration shall be in accordance with **JIS K 0113**.
- d) Water supplied for analysis shall be of the type and quality of A3 or A4 as defined in Annex 1 of **JIS K 0050** or clause 4 of **JIS K 0557**.
- e) All the reagents used shall be of the highest class specified in respective relevant **JIS** standards, if there is any, or be of an appropriate quality. Reagents not specified in **JIS** standards shall be of such a quality as not interfering with the analysis. For trace analysis, reagents of especially high purity shall be used.
- f) The apparatus and implement to be used shall satisfy the designated functions.

5 Analytical method

Sample is burnt by itself or with combustion improver in a stream of oxygen so that carbon in the sample is oxidized to produce carbon dioxide, which is then separated from other coexistent gases and determined. There are following analytical methods, each having a difference approach:

- a) **Gravimetric method** After sulfur oxide and water in combustion gas are removed, carbon dioxide is absorbed in soda lime, etc. whose mass increase is then measured. This method is, as a rule, applied to samples of carbon content not less than 0.05 % (mass fraction).
- b) **Gas volumetric method** After sulfur oxide in combustion gas is removed, carbon dioxide is collected in gas burette together with excess oxygen and its volume is measured. Then carbon dioxide is absorbed in sodium hydroxide, etc.

and the decrease in gas volume is obtained. This method is, as a rule, applied to samples of carbon content not less than 0.05 % (mass fraction).

- c) **Sulfuric acid back titration method** After sulfur oxide in combustion gas is removed, carbon dioxide is absorbed in a definite amount of sodium hydroxide solution, the excess alkali is titrated with sulfuric acid reference solution and the amount of titration is obtained. This method is, as a rule, applied to samples of carbon content not less than 0.005 % (mass fraction).
- d) **Electrical conductivity method** After sulfur oxide in combustion gas is removed, carbon dioxide is absorbed in a definite amount of sodium hydroxide solution and the change of electrical conductivity of sodium hydroxide solution before and after absorption is obtained. This method is, as a rule, applied to samples of carbon content not less than 0.001 % (mass fraction).
- e) **Coulometric method** After sulfur oxide in combustion gas is removed, carbon dioxide is absorbed in a definite amount of weakly alkaline solution. The solution is then electrolyzed until it returns to the original pH and the electrical quantity required for it is obtained. This method is, as a rule, applied to samples of carbon content not less than 0.001 % (mass fraction).
- f) **Thermal conductivity method** After sulfur oxide and water in combustion gas are removed, carbon dioxide is adsorbed in synthetic zeolite, and then desorbed by heating the collecting tube and introduced to a thermal conductivity cell by blowing oxygen. The change of thermal conductivity due to carbon dioxide is obtained. This method is, as a rule, applied to samples of carbon content not less than 0.001 % (mass fraction).
- g) **Infrared absorption method** After being removed of water, combustion gas is introduced to an infrared absorption cell and the amount of infrared absorption due to carbon dioxide is measured and integrated (integration method) or the maximum definite infrared absorption value is obtained (circulation method). This method is, as a rule, applied to samples of carbon content not less than 0.001 % (mass fraction).

6 Preparation of samples

6.1 Classification of samples

Samples dealt with in this Standard are classified as follows according to the pre-conditioned state.

Sufficient care shall be taken in preparing samples, especially to not allowing segregation of carbon, contamination or degeneration, so that the sample prepared will represent the average composition of the whole lot.

- a) Samples taken from molten metal
- b) Massive, cylindrical, linear, plate and foil type samples
- c) Sponge type samples
- d) Powdery samples.

6.2 Machine, tool and implement

The machine, tool and implement used for preparing samples shall be as follows.

- a) **Machine** Lathe, shaping machine, drilling machine, crusher, edge runner mill, stamp mill, compressor, etc.
- b) **Tool** Drill, cutting tool, cutting plier, cutting nippers, cutter, scissors, motar, hammer, etc.
- c) **Implement** Scoop, spoon, riffle sampler, sieve, stainless steel container, dies, punch, magnet, etc.

The machines, tools and implements shall be cleaned well before use. Especially, the parts which would come into contact with the sample shall be washed to be maintained clean so as not to contaminate samples. For cleaning, ethanol, acetone, diethylether, etc. shall be used while paying attention to exhaust.

6.3 Preparation of analytical samples from different forms of materials

6.3.1 Preparation from sample taken from molten metal

Preparation from sample taken from molten metal by casting or other method shall be as follows.

- a) Sample taken is removed of foreign matter, oxide, etc. adhered on the surface by means of an adequate machine such as drilling machine, and then continuously drilled to make shavings of thickness under 1 mm. These shavings are mixed well and then reduced, if necessary, to serve as an analytical sample. The pressure and rotational speed of drilling shall be such that the surfaces of resultant shavings are not oxidized. Lubricants such as oils and fats shall not be used. Water pouring for the purpose of cooling the sample shall not be performed. Further, care shall be taken so that the abraded powder of drill, etc. is not mixed in the shavings.
- b) Shavings in the state of cords, if produced, are made into smaller pieces by using an adequate tool such as a mortar or scissors, mixed well, and then reduced, if necessary, to serve as an analytical sample.

6.3.2 Preparation from massive, cylindrical, linear, plate and foil type samples

Preparation from massive, cylindrical, linear, plate and foil type samples shall be in accordance of one of the followings.

- a) **Where machining or cutting is suitable**
 - 1) For massive, cylindrical, linear and plate samples, preparation shall be according to **6.3.1 a)** or **b)**.
 - 2) For thin wire and foil type samples, after a new surface is washed with ethanol or diethylether, samples are cut into smaller pieces by means of scissors or other similar tool, and mixed well and reduced, if necessary, to serve as analytical sample.
- b) **Where crushing is suitable**
 - 1) For massive samples, the whole amount thereof is crushed to particles of most appropriate coarseness by using a crusher, stamp mill, mortar, etc., then they are reduced according to the increment reduction method, riffle sampler, etc.

Care shall be taken that loss of fragments or mixing of foreign matters does not occur during the course of crushing or reduction.

- 2) Crushing, mixing and reducing are repeated with care to maintain the average composition of the original sample, and the crushed sample is supplied for analysis.
- c) **Other cases** Preparation of samples that are difficult to machine, cut or crush shall be as follows.
 - 1) The mass of sample is smashed and a portion of some of the fractured surfaces is cracked to be collected, which are then repeatedly smashed, mixed and reduced while taking care so as to maintain the average composition of the original sample, to serve as an analytical sample.
 - 2) In the case of a sample mass of which the segregation is considered negligible, a portion of it is cracked to be collected, and then crushed to serve as an analytical sample.

6.3.3 Preparation from sponge type samples

Preparation from sponge type samples shall be as follows.

- a) A portion of the sample is taken such that the particle size is as uniform as possible. The taken portion is then put in a die, pressed by a punch inserted from above and moulded by applying suitable pressure by a compressor.
- b) The compressed mass is taken out and the shavings are made by means of a lathe or a drilling machine, etc. under such a pressure and at a rotational speed that the surfaces of resultant shavings will not be oxidized.

In the case of using a machine with which cutting in a stream of argon is possible so that oxidization can be avoided, the cutting speed should be increased.

- c) Shavings are collected in a stainless steel container, etc., and then smashed with a stainless steel rod or spoon, passed through a sieve whose nominal aperture is principle size 4 mm as specified in **JIS Z 8801-1**, and mixed well to serve as an analytical sample.

6.3.4 Preparation from powdery samples

Preparation from powdery samples shall be as follows.

- a) After mixing well, a portion of it is taken to serve as an analytical sample.
- b) If the powdery sample requires crushing, it is subjected to repeated crushing, mixing and reducing performed with care to maintain the average composition of the original sample, to serve as an analytical sample.

6.4 Handling of analytical sample

Handling of analytical samples shall be as follows.

- a) When it is possible for the surface of sample to be contaminated with oil, etc, in the course of preparation, it is washed with ethanol, diethylether, etc, before stored.

Further, when it is possible for iron powder, etc. to have been mixed during the course of preparation, if separation with a magnet or similar means is possible, it should be removed using a magnet.

- b) The analytical sample is, as a rule, stored in a hermetically sealed clean glass bottle. When the analytical sample of material which can be easily oxidized is to be preserved for a long period of time, air in the bottle is previously replaced with inert gas such as argon.

- c) When weighing out analytical sample from sample for analysis, the sample is mixed so as to maintain the average composition, and a clean stainless steel spoon or similar tool is used so that sample is not contaminated.

7 Classification of apparatus

7.1 Composition of apparatus

The apparatus in which the carbon in sample is oxidized to carbon dioxide can be largely separated into the carbon extraction part and the carbon dioxide determination part. Figure 1 shows an example of composition of apparatus for carbon determination method. The carbon extraction part consists of the oxygen purification part, sample combustion part and combustion gas purification part, and the carbon dioxide determination part consists of the carbon dioxide absorption part and carbon dioxide titration/measurement part. However, the composition of carbon dioxide determination part can vary according to the measuring system specified in clause 5.

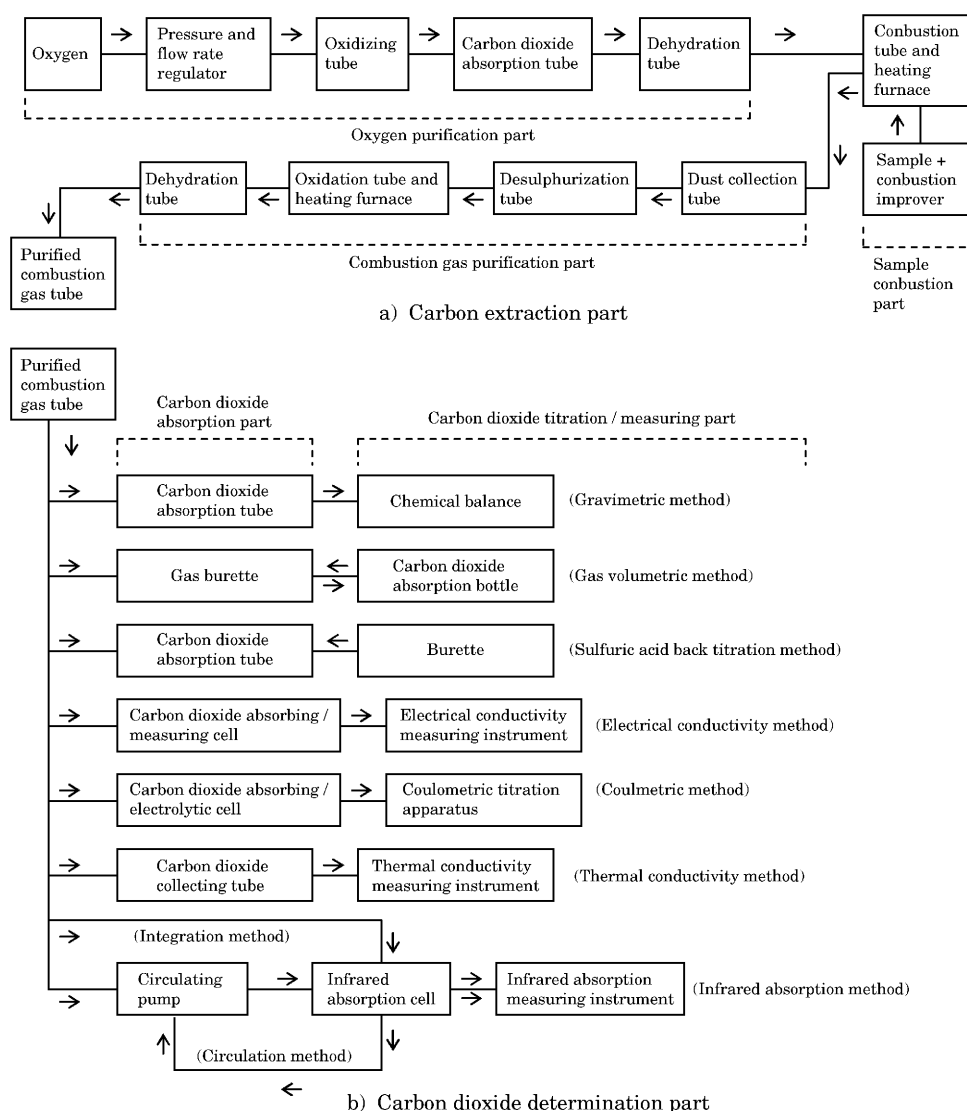


Figure 1 Examples of apparatus constitution for carbon determination method

7.1.1 Oxygen purification part

This part controls the pressure and flow rate of oxygen supplied from oxygen cylinder and removes the components to be the inhibitor of carbon determination such as carbon oxide, gas of organic matters, water, etc. contained in oxygen, and consists of an oxygen cylinder, pressure and flow rate regulator, oxidation tube, carbon dioxide absorption tube or carbon dioxide absorption column, dehydration tube or dehydration column, all connected in this order for use.

Moreover, in the case of a dry oxidation tube, the electric resistance heating furnace which heats the oxidation tube is used.

7.1.2 Sample combustion part

This part heats and burns the sample in a stream of oxygen and consists of a sample inserting part, combustion tube and heating furnace. For the combustion tube, there are those made of porcelain and those made of quartz glass, and for the heating furnace, there are those of the electric resistance heating system and those of the high frequency induction heating system. The inlet of the sample combustion part is connected to the oxygen purification part and the outlet is connected to the combustion gas purification part.

7.1.3 Combustion gas purification part

This part, in the case of determining the carbon dioxide in combustion gas, removes the component to be an inhibitor and consists of the dust collection tube, desulfurization tube, oxidation tube and dehydration tube. However, a part of these may be omitted depending on the measuring system. The inlet of combustion gas purification part is connected to the sample combustion part, and the outlet is connected to the carbon dioxide determination part.

7.1.4 Carbon dioxide determination part

This part determines the carbon dioxide in purified combustion gas. The composition of this part is different depending on the measuring system, and is as follows.

- a) **Gravimetric method** The part is composed of a carbon dioxide absorption tube for absorption of carbon dioxide in purified combustion gas and a protective tube [dehydration tube (or dehydration column) and tube (or carbon dioxide absorption column)] to prevent the intrusion of components from outside that can inhibit determination, such as carbon dioxide or water.
- b) **Gas volumetric method** The part is composed of a gas burette to measure the volume of purified combustion gas, an absorption bottle for absorption of carbon dioxide in combustion gas and a levelling bottle to take the combustion gas and send it to the absorption bottle.
- c) **Sulfuric acid back titration method** The part is composed of an absorption tube in which absorption of carbon dioxide in purified combustion gas and titration are carried out and an inspection trap to prevent the intrusion of carbon dioxide, etc. from the open air and to monitor the escaping carbon dioxide which has not been absorbed in the absorption bottle.
- d) **Electrical conductivity method** The part consists of a sample cell for absorption of carbon dioxide in purified combustion gas, attached with an electrode for measuring the change of electrical conductivity before and after absorption, and a reference cell, a thermostatic bath for maintaining both cells at a constant temperature, an electrical conductivity measuring circuit and an indicator or recorder.

- e) **Coulometric method** The part is composed of an absorption and electrolytic cell in which the carbon dioxide in purified combustion gas is absorbed and the electrolysis is carried out for returning the pH which has been reduced to the original value due to absorption, a coulometric measuring apparatus to measure the electrical quantity required for electrolysis and an indicator.
- f) **Thermal conductivity method** The part is composed of a collection tube to adsorb the carbon dioxide in purified combustion gas, a heating furnace to desorb the carbon dioxide collected, a sample cell and a reference cell to measure the change of thermal conductivity due to carbon dioxide, a thermostatic bath to maintain both cells at a constant temperature, a thermal conductivity measuring circuit and an indicator.
- g) **Infrared absorption method** The part is composed of an infrared absorption detector, an infrared absorption measuring circuit and an indicator for measuring the amount of infrared absorption due to carbon dioxide in purified combustion gas. For the measurement of the amount of infrared absorption, the integration method or circulation method is used. In the case of integration method, the combustion gas is released to the atmosphere through the absorption cell. In the circulation method, the circulating pump which circulates combustion gas is used between the infrared absorption cell and the combustion tube.

7.2 Selection of apparatus

The apparatus shall be in accordance with the determination method selected from those specified in 7.1.4 according to the working conditions such as the kind of analytical work, the carbon content of sample, etc.

For the selection, the following matters shall be considered:

- a) The type of sample (whether many types of samples are to be handled, or only those of the same metal type)
- b) The carbon content (a minute content or a wide range of measurement);
- c) The kind of analytical work (e.g. daily work analysis in a factory where many samples are analyzed, an analysis for research where several kinds of samples in relatively small numbers are analyzed, an analysis for research object where the apparatus is used for only a limited period of time)
- d) Sensitivity, accuracy, etc.

7.3 Setting of apparatus

For the setting of apparatus, the conditions suitable for respective apparatuses shall be satisfied. Generally, an apparatus should be installed in an environment satisfying the following conditions:

- a) There is no corrosive gas, especially no acidic gas and only a minor amount of dust;
- b) The humidity is not too high and temperature variation is small;
- c) Variation in voltage and frequency of supply power source is small;
- d) In the case of high frequency induction heating furnace, sufficient earthing is possible;
- e) There is only a minor amount of vibration.

7.4 Preliminary operation of apparatus

Prior to the determination operation, the preliminary operation is carried out using the analytical sample or a sample similar to the analytical sample to determine the conditions suitable for the apparatus to be used, such as the heating time for combustion of sample, the type and amount of combustion improver to be used, the flow rate and time of charging oxygen. The determination shall be carried out under thus determined conditions.

8 Implements and materials

The implements and materials shall be as follows.

8.1 Oxygen cylinder and pressure reducing valve The cylinder to supply oxygen for burning the sample shall be equipped with a pressure reducing valve for controlling the pressure and flow rate of oxygen. The two-step type pressure reducing valve is recommended.

8.2 Flow meter The flow meter is for monitoring the flow rate of oxygen supplied, and a rotor type meter of 0 ml/min to 4 000 ml/min should generally be used.

8.3 Oxidation tube There are dry type and wet type oxidation tubes.

- a) **Dry type oxidation tube** A tube stuffed with granular copper (II) oxide or platinum catalyst for oxidizing the organic matter, etc in oxygen, heated at 650 °C to 700 °C for the former, or at 700 °C to 750 °C for the latter. For those intended for oxidizing carbon monoxide, sulfur dioxide, etc. in combustion gas, the platinum catalyst heated at 350 °C is used.
- b) **Wet type oxidation tube or bottle** The tube or bottle containing chromic acid-sulfuric acid solution (saturated) or permanganic acid-sulfuric acid solution is used. The chromic acid-sulfuric acid solution (saturated) is prepared by dissolving chromium (VI) oxide in sulfuric acid (density: 1.82 g/cm³) to saturation. The permanganic acid-sulfuric acid solution is prepared by mixing at the rate of 1 ml of sulfuric acid (1 + 1) per 10 ml of potassium permanganate solution (50 g/L).

8.4 Carbon dioxide absorption tube or column It is a tube or a column stuffed with inorganic base materials such as silica gel or mica impregnated with sodium hydroxide, or granular sodium hydroxide or soda lime, used for the purpose of absorbing carbon dioxide in the oxygen passed through oxidation tube or preventing the intrusion of acidic gas such as carbon dioxide from the open air.

Sodium hydroxide, if used, shall be crushed to have newly exposed surfaces to improve the absorption of carbon dioxide

8.5 Dehydration tube or column The tube or column containing magnesium perchlorate, active alumina, phosphor (V) oxide, sulfuric acid (density: 1.84 g/cm³) etc. is used for removing moisture in oxygen or combustion gas or preventing the intrusion of moisture from the open air.

8.6 Combustion tube heating furnace The combustion tube heating furnace to be used shall be either the tubular electric resistance heating furnace or the high frequency induction heating furnace as described in the following.

- a) **Tubular electric resistance heating furnace**

- 1) The tubular electric resistance heating furnace consists of an electric resistance heating furnace and a combustion tube, and is exemplified in figure 2.

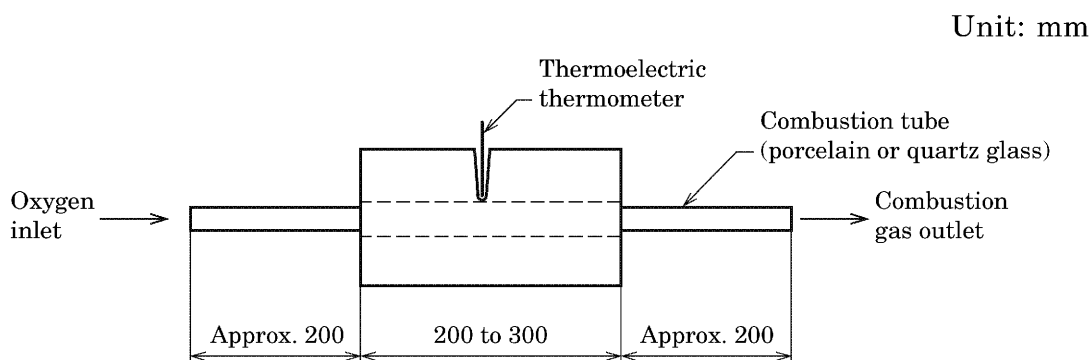


Figure 2 Example of tubular electric resistance heating furnace

- 2) The electric resistance heating furnace is a tubular furnace of 200 mm to 300 mm length, which performs heating by the electric resistance heating unit, and in which the temperature is regulated by controlling the electric current, and the part not less than 150 mm in length at the central part of the furnace can be maintained at a definite temperature of 1 200 °C to 1 450 °C.
- 3) The combustion tube of such a length as about 200 mm will protrude from each of the side surfaces of the furnace is inserted and the temperature is measured at immediately above the combustion tube at the centre of the furnace by using a thermoelectric thermometer. The indicated value of the thermoelectric thermometer is, as a rule, different from the temperature inside the combustion tube, therefore, the difference shall have been determined beforehand so that it can be used to calculate the temperature inside the combustion tube from the indicated value.
- 4) Quartz glass wool is stuffed in the rear central part of the combustion tube so as to extend about 50 mm.

If chlorine is contained in combustion gas, a copper net or silver net is stuffed in the rear of this.

- 5) For the connection of the combustion tube to oxygen purification part and the combustion tube to combustion gas purification part, a ground joint or heat resistant silicone rubber stopper, etc. is used.
- 6) In the case of inserting a combustion boat, for preventing entrance of air or for expelling the air entered, a sample inserting tube should be attached at the inlet part of the combustion tube. Figure 3 shows examples of four types of sample inserting tubes and the direction of gas flow at the time of heating of sample for respective types.

The inlet of the combustion tube may be opened if the excess oxygen can be released from this inlet and the intrusion of air in the tube can be prevented.

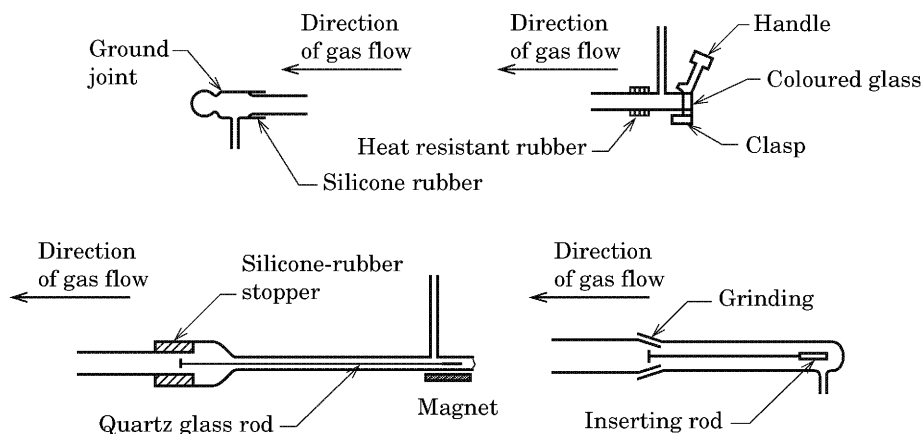


Figure 3 Examples of sample inserting tubes

- b) **High frequency induction heating furnace** The high frequency induction heating furnace consists of a quartz glass combustion tube (e.g. 30 mm to 44 mm in outside diameter, 26 mm to 37 mm in inside diameter, 200 mm to 220 mm in length), a heating coil wound around it (e.g. 35 mm to 55 mm in height, 4 to 5 is number of turns) and a high frequency oscillator to supply high frequency current. The analytical sample is put in the high frequency combustion crucible and with this combustion crucible held at about the centre of heating coil, high frequency current is transmitted and controlled to the temperature at which the sample is burnt and carbon is sufficiently oxidized. Some heating furnaces are equipped with a lance for blowing oxygen into the sample for accelerating combustion. Figure 4 shows an example of such a high frequency induction heating furnace.

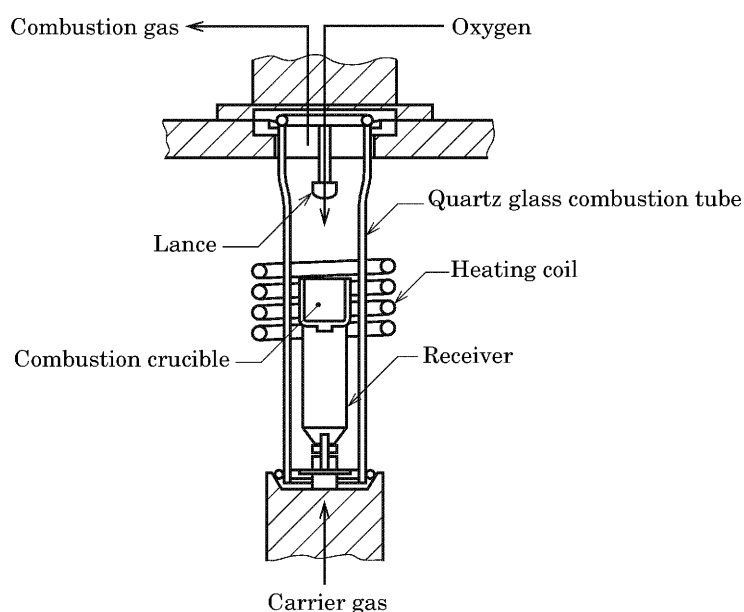


Figure 4 Example of high frequency induction heating furnace combustion tube

8.7 Dust collection tube The tube stuffed with filter of quartz glass wool, etc. for removing the combustion dust contained in combustion gas. In the case of stuffing quartz glass wool in the rear part of the combustion tube, this tube may be omitted.

8.8 Desulfurization tube or bottle It is a tube stuffed with manganese (IV) oxide or silver vanadate or a wet type oxidation tube or bottle in **8.3 b)**, used at ordinary temperature to oxidize and absorb sulfur dioxide in combustion gas. Instead of this, a desulfurization tube in which an oxidation tube stuffed with platinum foil, a heater for heating this and a sulfur trioxide collection tube stuffed with fine cellulose wool are combined may also be used. If this is used, the oxidation tube is heated at about 350 °C.

8.9 Porcelain combustion tube or quartz glass combustion tube The porcelain combustion tube used for tubular electric resistance heating furnace shall be CT0 or CT1 specified in **JIS R 1307** and those of which the service temperature is higher than that specified in **JIS R 1307** may be used. In the case of using a new combustion tube, pre-burning shall be carried out for the designated time by charging oxygen at the designated temperature. In the case of using a quartz glass combustion tube, it shall be the quartz glass tube suitable for the dimensions and performance of the furnace. For these combustion tubes, those having a shape and dimensions as designated by the apparatus to be used may be applied. In the case where the inner wall of a combustion tube is contaminated by iron oxide, etc. it shall be washed before use. When contamination is serious and can not be sufficiently removed, the tube shall be renewed.

8.10 Porcelain combustion boat and porcelain combustion cover The porcelain combustion boat used for tubular electric resistance heating furnace shall be CB1 specified in **JIS R 1306**. For the combustion boat, if necessary, a porcelain combustion boat cover is used. The combustion boat cover shall be CBC1 specified in **JIS R 1306**. For the combustion boat, combustion boat cover, etc., those having a shape and dimensions as designated by the apparatus to be used or those of which the service temperature is higher than that specified in **JIS R 1306** may be used. The combustion boat and combustion boat cover shall have been pre-burnt in air or in oxygen. When pre-burning many of them at one time, after leaving them to cool, preserve in a desiccator without coating of grease etc. They are handled with a pincette, etc. and shall not be touched directly by hand. A combustion boat or a combustion boat cover that has been preserved for an extended period of time should not be used since the blank test value can be high. In this case, pre-burning shall be carried out again.

8.11 High frequency porcelain combustion crucible For the high frequency porcelain combustion crucible and receiver to be used for high frequency induction heating furnace, those of a shape and dimensions as designated by the apparatus to be used or those suitable for analysis are used. If necessary, a lid may be applied on the combustion crucible. The combustion crucible, lid and receiver are pre-burnt and handled similarly to the combustion boat and combustion boat cover in **8.10** and preserved.

8.12 Oxygen Oxygen specified in **JIS K 1101** shall be used.

8.13 Combustion improver When the sample is heated to burn in a stream of oxygen, combustion improver is mixed with the sample or placed on top of the sample for promoting smooth oxidation reaction. When using a combustion improver,

that of the designated kind is added according to the designated procedures. Any one or a combination of several of the following materials should be used, the carbon content of each of which preferably being minimum. Those materials of which the carbon content (blank test value) can be reduced by heating at a high temperature shall be used after they have been pre-burnt.

- a) **Iron**, granular and 150 μm to 1 mm
- b) **Tin**, granular and 250 μm to 1 mm
- c) **Tungsten**, granular and 250 μm to 1 mm
- d) **Copper**, granular or in small pieces
- e) **Copper (II) oxide**, granular
- f) **Manganese (IV) oxide**, granular or powdery

9 Operation

9.1 Gravimetric method

9.1.1 Summary

Sample is burnt together with a combustion improver in a stream of oxygen to oxidize carbon to produce carbon dioxide. The produced carbon dioxide is then absorbed in soda lime or sodium hydroxide, the increase in mass of which is measured.

This method, which is a rather complicated and cumbersome procedure, is not suitable for determination of trace carbon.

9.1.2 Assembly of apparatus

The assembly of apparatus shall be based on 7.1 and in accordance with the following. Figure 5 shows an example of composition of an apparatus for gravimetric method. For the connection of the apparatus, glass pipe is used, the both ends of which are tightly connected and held with rubber tube or polyvinyl chloride tube.

9.1.2.1 Oxygen purification part It consists of an oxidation tube (c) containing chromic acid-sulfuric acid solution (saturated) [8.3 b)], a carbon dioxide absorption tube (d) stuffed with soda lime or granular sodium hydroxide and a dehydration column (e) stuffed with active alumina.

9.1.2.2 Sample combustion part The electric resistance heating furnace (f) is used. The outlet part of the combustion tube (g) is stuffed with quartz glass wool.

9.1.2.3 Combustion gas purification part Chromic acid-sulfuric acid solution (saturated) [8.3 b)] is stuffed in the desulfurization tube (i), quartz glass wool in the dust collection tube (j), and phosphor (V) oxide or magnesium perchlorate in the dehydration tube (k), respectively. In the case of the sample of sulfur content not less than 0.1 % (mass fraction), two gas washing bottles (i) containing chromic acid-sulfuric acid solution (saturated) [8.3 b)] are connected or the gas washing bottles containing permanganic acid-sulfuric acid solution [8.3 b)] are connected. It is recommended that phosphor (V) oxide is stuffed together with quartz glass wool in alternate layers or by mixing with shortly-cut quartz glass wool. The dehydration agent stuffed in dehydration tube, carbon dioxide absorption tube and protective tube shall be consistently either phosphor (V) oxide or magnesium perchlorate. For magnesium perchlorate, that intended for element analysis shall be used.