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**Coal and coke — Determination of
constituents**

ICS 73.040; 75.160.10

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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 Center for Coal Utilization, Japan (CCUJ) / 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 M 8813:1994** is replaced with this Standard.

This revision has been made based on **ISO 333:1996, Coal — Determination of nitrogen — Semi-micro Kjeldahl method** for the purposes of making it easier to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of an International Standard which is based on Japanese Industrial Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Coal and coke — Determination of constituents

Introduction The Japanese Industrial Standard has been prepared based on **ISO 333:1996**, *Coal — Determination of nitrogen — Semi-micro Kjeldahl method* and **ISO 334:1992**, *Solid mineral fuels — Determination of total sulfur — Eschka method* with some modifications of the technical contents.

Portions given sidelines or dotted lines are the matters modified from the original International Standards. A list of modifications with the explanations is given in annex 11 (informative).

1 Scope This Standard specifies the methods for determination of constituents of coal and coke. In this Standard, the determination of constituents means the method for determination of carbon, hydrogen, total sulfur, sulfur in ashes, nitrogen, phosphorus, oxygen, and carbon dioxide (as carbonate) of coal.

Remarks 1 This Standard may apply to the analysis of charcoal and briquets.

2 The International Standard corresponding to this Standard is as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and JIS are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21**.

ISO 333:1996, *Coal — Determination of nitrogen — Semi-micro Kjeldahl method* (MOD)

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

JIS H 6202	<i>Platinum dishes for chemical analysis</i>
JIS H 6203	<i>Platinum boats for chemical analysis</i>
JIS K 1101	<i>Oxygen</i>
JIS K 8001	<i>General rule for test methods of reagents</i>
JIS K 8085	<i>Ammonia solution</i>
JIS K 8116	<i>Ammonium chloride</i>
JIS K 8155	<i>Barium chloride dihydrate</i>
JIS K 8180	<i>Hydrochloric acid</i>
JIS K 8228	<i>Magnesium perchlorate</i>
JIS K 8230	<i>Hydrogen peroxide</i>
JIS K 8383	<i>Sucrose</i>
JIS K 8422	<i>Copper (II) oxide</i>
JIS K 8432	<i>Magnesium oxide</i>
JIS K 8550	<i>Silver nitrate</i>

- JIS K 8574 *Potassium hydroxide*
- JIS K 8576 *Sodium hydroxide*
- JIS K 8598 *Selenium*
- JIS K 8603 *Soda lime*
- JIS K 8625 *Sodium carbonate*
- JIS K 8863 *Boric acid*
- JIS K 8893 *Methyl orange*
- JIS K 8951 *Sulfuric acid*
- JIS K 8962 *Potassium sulfate*
- JIS M 0104 *Technical terms used in coal utilization*
- JIS M 8810 *Coal and coke — General rules for sampling, analysis and testing*
- JIS M 8811 *Coal and coke — Sampling and sample preparation*
- JIS M 8812 *Coal and coke — Methods for proximate analysis*
- JIS M 8815 *Methods for analysis of coal ash and coke ash*
- JIS P 3801 *Filter paper (for chemical analysis)*
- JIS R 1301 *Porcelain crucibles for chemical analysis*
- JIS R 1306 *Porcelain combustion boats for chemical analysis*
- JIS R 1307 *Porcelain combustion tubes for chemical analysis*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS R 3505 *Volumetric glassware*
- JIS Z 0701 *Silicagel desiccants for packaging*
- 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 8402-6 *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

3 Definitions For the purpose of this Standard, the definitions given in **JIS M 0104**, **JIS Z 8402-1** to **JIS Z 8402-4** and **JIS Z 8402-6** apply.

4 General requirements

4.1 Sample

4.1.1 For coal The air dried sample prepared in accordance with **JIS M 8811** shall be used (see clause 5 of **JIS M 8812**).

4.1.2 For coke The air dried sample prepared in accordance with **JIS M 8811** shall be used. In this case, it is not necessary to measure the moisture content of the air dried sample simultaneously.

4.1.3 For ashes The class 1 ash or the class 2 ash specified in **JIS M 8815** shall be used.

Remarks 1 Since the moisture content of the air dried sample stored being tightly stoppered does not almost change at least for about 7 days, it is not necessary to determine the moisture content in the air dried sample each time when the analysis of each constituent is performed.

2 Class 1 ash The ash in which a suitable amount of the air dried sample of coal and coke is incinerated ⁽¹⁾ in accordance with clause 6 of **JIS M 8812**. The class 1 ash shall be pulverized in an agate mortar until graininess is not felt in the tip of a finger (about not more than 75 μm), and further reignited at $815\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for 30 min, and then stored in a desiccator.

3 Class 2 ash The ash generated by combustion of coal or coke in the combustion appliances. The class 2 ash shall be pulverized in an agate mortar until graininess is not felt in the tip of a finger, and further may be incinerated completely provided that the effect is described clearly.

Note ⁽¹⁾ When the sample is incinerated, the thickness of sample layer, the draft rate and the rate of heating of an electric furnace shall strictly conform to the specification of clause 6 of **JIS M 8812**. In particular, in the case of a coal sample which contains much of sulfur content and calcium content, it should be noted that unless the specification of clause 6 of **JIS M 8812** is satisfied, the amount of sulfur content to be fixed in the process of incineration may increase noticeably.

4.2 Measuring instrument

4.2.1 Balance and weight When mass is measured, the balance and weight specified in **JIS M 8810** shall be used.

4.2.2 Volumeter When the volume of liquid is measured, the chemical volumeter specified in **JIS M 8810** shall be used.

4.2.3 Thermometer When temperature is measured, the thermometer specified in **JIS M 8810** shall be used.

4.3 Reagents and water The reagents and water shall be used as specified in **JIS M 8810** unless otherwise specified in each item.

In addition, the reagents which are highly toxic and the reagents which are inflammable and highly explosive are used, great attention should be given to handling and treatment.

4.4 Expression of analysis results

4.4.1 Basis of measurements and report value The expression of basis shall be as follows.

a) For the ultimate analysis result, 6 components of ash content ⁽²⁾, carbon, hydrogen, oxygen, sulfur ⁽³⁾, and nitrogen shall be expressed according to the anhydrous basis. However, the following components shall be expressed according to the following basis, respectively separately from the above-mentioned 6 components.

- Total sulfur and phosphorus For coal, according to the air dried basis or the anhydrous basis ⁽⁴⁾, and for coke, according to the anhydrous basis.
- Sulfur in ashes According to the anhydrous basis ⁽⁴⁾
- Carbon dioxide of carbonate type of coal According to the anhydrous basis ⁽⁴⁾

Notes ⁽²⁾ The numerical value of this ash content is obtained by converting the numerical value obtained in accordance with clause 6 of **JIS M 8812** into the anhydrous basis ⁽⁴⁾.

⁽³⁾ Since the sulfur content in coal and coke exists in a complicated form, the method for determining the sulfur content in coal and coke as the total sulfur content is specified in annex 2. Furthermore, the method for determination of sulfur content in ashes with regard to the sample obtained by incinerating the coal and coke sample with the specific method is specified in annex 3 [see note ⁽¹⁾].

⁽⁴⁾ For the moisture content for converting into the anhydrous basis, the average value (two decimal places) in 5.2.8, 5.3.9 or 5.4.9 of **JIS M 8812** shall be used.

b) Each above-mentioned basis may be expressed by converting into other basis as necessary. In this case, the abbreviated symbol of the converted basis shall be appended.

The conversion method and abbreviated symbol shall be in accordance with clause 8 and clause 9 of **JIS M 8810**.

4.4.2 Expression of results The expression of results shall be in accordance with clause 8 of **JIS M 8810**.

4.5 Tolerances The tolerances in the same laboratory and the tolerance in different laboratories of the ultimate analysis method shall be as given in table 1.

5 Method for determination of carbon and hydrogen In accordance with annex 1.

Information : The method for determination of carbon and hydrogen by the improved Liebig method is given in annex 8.

6 Method for determination of total sulfur In accordance with annex 2.

7 Method for determination of sulfur in ashes In accordance with annex 3.

8 Method for determination of nitrorogen In accordance with annex 4.

Information : The method for determination of nitrogen by the semi-micro Kjeldahl method is given in annex 10.

9 Method for determination of phosphorus In accordance with annex 6.

10 Method for determination of oxygen In accordance with annex 5.

Information : The method for direct determination of oxygen is given in annex 9.

11 Method for determination of carbon dioxide of carbonate In accordance with annex 7.

Table 1 Tolerance on ultimate analysis method

Unit : mass percentage (%)

Component (annex in which the applicable analysis method is specified)		Division	Tolerance in the same laboratory		Tolerance in different laboratories
			<i>n</i> = 2	<i>n</i> = 3	
Carbon (annex 1)		—	0.3	0.4	1.5
Hydrogen (annex 1)		—	0.15	0.18	0.70
Total sulfur (annex 2)	1.00 or under	0.04	0.05	0.07	
	Over 1.00 up to and incl. 2.00	0.07	0.08	0.12	
	Over 2.00	0.10	0.12	0.17	
Sulfur in ashes (annex 3)	1.00 or under	0.04	0.05	0.07	
	Over 1.00 up to and incl. 2.00	0.07	0.08	0.12	
	Over 2.00	0.10	0.12	0.17	
Nitrogen (annex 4)		—	0.05	0.06	0.1
Phosphorus (annex 6)	Molybdenum blue absorptio- metric method	0.100 or under	0.010	0.012	0.020
		Over 0.100 up to and incl. 0.200	0.030	0.036	0.030
		Over 0.200 up to and incl. 0.400	0.030	0.036	0.030
		Over 0.400 up to and incl. 0.600	0.030	0.036	0.070
		Over 0.600	0.060	0.072	0.070
	Phosphorus vanadomolyb- denum yellow absorptiometric method	0.100 or under	0.010	0.012	0.010
		Over 0.100 up to and incl. 0.200	0.030	0.036	0.050
		Over 0.200 up to and incl. 0.400	0.030	0.036	0.050
		Over 0.400 up to and incl. 0.600	0.030	0.036	0.050
		Over 0.600	0.060	0.072	0.220
	Titrimetric method	0.100 or under	0.010	0.012	0.030
		Over 0.100 up to and incl. 0.200	0.030	0.036	0.110
		Over 0.200 up to and incl. 0.400	0.030	0.036	0.110
		Over 0.400 up to and incl. 0.600	0.030	0.036	0.120
		Over 0.600	0.060	0.072	0.120
Carbon dioxide (annex 7)		—	0.06	0.07	0.10
Oxygen (annex 9) (Information)		—	0.30	0.36	—

Annex 1 (normative) Method for determination of carbon and hydrogen

1 Division of methods The determination of carbon and hydrogen shall be performed according to either of the following methods.

- a) Liebig method
- b) Sheffield high temperature method

2 Liebig method

2.1 Summary The sample is heated and burnt at about 800 °C in a low-speed oxygen flow, and carbon dioxide and steam which are generated are absorbed in each absorbent, respectively, and the increased amount is measured.

2.2 Reagents The reagents shall be as follows.

- a) Sulfuric acid As specified in **JIS K 8951**.
- b) Potassium hydroxide or sodium hydroxide Potassium hydroxide specified in **JIS K 8574** or sodium hydroxide specified in **JIS K 8576**.
- c) Copper gauze roll Aperture of about 500 µm, diameter of 15 mm to 20 mm (it shall fit in the inner wall of combustion tube).
- d) Oxygen As specified in **JIS K 1101** (oxygen made with the electrolytic method is not suitable).
- e) Copper (II) oxide The small bar, small tubular or granular copper (II) oxide specified in **JIS K 8422** (for analysis).
- f) Magnesium perchlorate (for analysis) Magnesium perchlorate specified in **JIS K 8228** (for ultimate analysis).
- g) Carbon dioxide absorbent ⁽¹⁾ A grain size of 1 mm to 3 mm (for analysis).

Note ⁽¹⁾ The carrier which consists mainly of magnesium silicate is mixed into the fused sodium hydroxide, and cooled, then those which are pulverized and selected in a particle size should be used.

- h) Lead (II) chromate A granular shape.
- i) Sucrose As specified in **JIS K 8383**.

2.3 Apparatus and preparation of apparatus The apparatus (see annex 1 figure 1) and its preparation shall be as follows.

- a) **Oxygen purification train** This apparatus is constructed of an empty bottle (a), a bottle (b) which contains sulfuric acid, a tube or a tower (c) which is filled with potassium hydroxide or sodium hydroxide, a tube or a tower (d) which is filled with carbon dioxide absorbent [2.2 g)] in the gas inlet side and magnesium perchlorate [2.2 f)] in the gas outlet side, which are connected in series, in order to purify oxygen.

Remarks : When oxygen contains combustible gas, the red-heated platinum narrow tube (about 800 °C), or the red-heated silica glass tube filled with copper (II) oxide [2.2 e)] (about 300 °C) or platinized asbestos (about 800 °C) shall be installed in front of the purification train.

- b) **Combustion assembly** The combustion furnace (e, f, g) shall consist of an electric furnace which heats the sample in the combustion tube at 800 °C ± 20 °C, heats copper (II) oxide at 800 °C ± 20 °C, and heats lead (II) chromate [2.2 h)] at 500 °C ± 20 °C, and is capable of controlling the heating temperature independently, respectively.

The combustion tube (h) of 15 mm to 20 mm in internal diameter and about 1 000 mm in length shall be made of borosilicate glass 1 or transparent silica glass, of which one end is extended or has the rubber stopper with a glass tube. The following various substances shall be filled into the tube one after another according to annex 1 table 1 and the rubber stopper with a glass tube shall be attached to the other end.

Instead of the rubber stopper, a glass ground joint may be used.

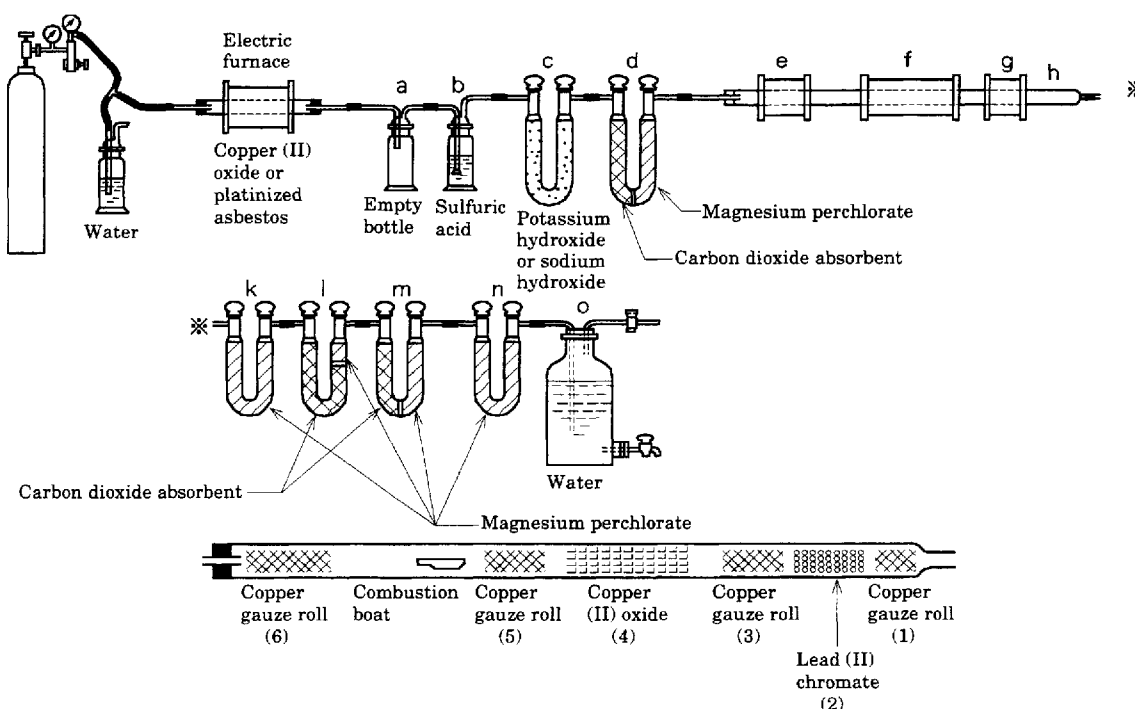
Annex 1 Table 1 Fillers of combustion tube (see annex 1 figure 1)

Fillers in combustion tube	Notes
(1) Copper gauze roll	Fill the copper gauze roll [2.2 c)] to the length of about 20 mm.
(2) Lead (II) chromate	Fill lead (II) chromate [2.2 h)] to the length of about 100 mm.
(3) Copper gauze roll	Fill the copper gauze roll [2.2 c)] to the length of about 20 mm.
(4) Copper (II) oxide	Fill copper (II) oxide [2.2 e)] which was red-heated beforehand to the length of 300 mm to 400 mm.
(5) Copper gauze roll	Fill the copper gauze roll [2.2 c)] to the length of about 20 mm.
(6) Copper gauze roll	Make the copper wire which is made convenient to pull out by bending its end as the core, and wound with a copper gauze on its outer part to make the length of about 100 mm and is oxidized beforehand.

- c) **Absorption train** The absorption train is constructed of a moisture absorber (k), a carbon dioxide absorber (l), a protective tube (m and n), and an absorber (o), which are connected in series, in order to absorb the combustion gas from the combustion tube.

The moisture absorber (k) is prepared by filling a U tube, etc. with magnesium perchlorate, the carbon dioxide absorber (l) is prepared by filling with carbon dioxide absorbent in the gas inlet side and a small amount of magnesium perchlorate in the gas outlet side, the protective tube (m) is prepared by filling with carbon dioxide absorbent in the gas inlet side and magnesium perchlorate in the gas outlet side, the protective tube (n) is prepared by filling with magnesium perchlorate, and the absorber (o) is for sucking the combustion gas from the combustion tube into the absorption train.

- d) **Combustion boat** As specified in JIS R 1306 or JIS H 6203.



Remarks : This figure indicates a gist of connection of each part and also indicates an example of shape.

Annex 1 figure 1 Apparatus for determination of carbon and hydrogen (Liebig method)

2.4 Weighing-out amount of sample Weigh out about 0.2 g of the sample to the nearest 0.1 mg.

2.5 Procedure (see annex 1 figure 1)

Warning on safe When performing the combustion procedure, use crucible scissors, etc. for handling the container heated at the high temperature and take care so as not to get burned. Furthermore, make an effort to prevent fire with careful attention to handling of superfluous oxygen exhaust.

2.5.1 Preparatory procedure The preparatory procedure shall be performed as follows.

- Check of apparatus** Check the apparatus before measurement, and confirm that each connection and the absorber are completely airtight.
- Heating** While flowing oxygen [2.2 d)] at a rate of 30 ml to 40 ml per min, heat the combustion tube to heat copper (II) oxide to $800\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$, lead (II) chromate to $500\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$, and flow as it is for 2 h.
- Measurement of amount of increase and decrease of absorber** Detach the moisture absorber (k), the carbon dioxide absorber (l) and the protective tube (m), and after allowing to cool, weigh the mass to the nearest 0.1 mg, obtain the mass change of each absorber, and repeat ⁽²⁾ the procedures of **b)** and **c)** until the value becomes less than 0.5 mg.

Note ⁽²⁾ The procedure to make the mass of absorber constant is required only for the first measurement of the day, and is not required for the second and subsequent times.

2.5.2 Main procedure The main procedure shall be performed as follows.

- a) **Weighing-out of sample** Move the weighed-out sample (2.4) into the combustion boat of which mass is known, and put it in a weighing tube with a ground stopper.
- b) **Attaching of absorber** While passing oxygen through gas-cleaning apparatus, preliminarily heat copper (II) oxide at $800\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$, and lead (II) chromate at $500\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$, and after weighing the mass of both absorbers to the nearest 0.1 mg, attach to the combustion tube.
- c) **Insertion of sample** Take out the combustion boat containing the sample from the weighing tube with a ground stopper, and rapidly insert it into a definite position of the combustion tube. Next enter a copper gauze roll at a position slightly separated from it and attach the combustion tube to the gas-cleaning apparatus.
- d) **Combustion** After attaching the apparatus, again flow oxygen, observe bubbles in clean bottle (b) containing sulfuric acid to adjust the flow rate of oxygen, and heat from the above-described copper gauze roll. Next, heat gradually the combustion boat from the feeding-in side of oxygen to evaporate the moisture, while taking care to the combustion degree of volatile components, gradually, advance the heating of combustion boat to copper (II) oxide side, and burn the sample completely. Let the final heating temperature be $800\text{ }^{\circ}\text{C} \pm 20\text{ }^{\circ}\text{C}$ and the heating time be 1 h to 1.5 h.

Carefully warm moisture collected at the tip of combustion tube, and feed completely into the moisture absorber.

- e) **Measurement of increased amount of absorber** Stop heating of the furnace (e), and after flowing oxygen further at the same flow rate for 0.5 h to 1 h, detach the both absorbers. Make the both ends airtight to wipe with a clean cloth, cool to room temperature, weigh the mass of both absorbers to the nearest 0.1 mg and obtain the respective increased amounts.

2.5.3 Discussion by sucrose Weigh out about 0.2 g of sucrose [2.2 i)] to the nearest 0.1 mg, perform the same procedures as those of 2.5.2 a) to e), calculate carbon and hydrogen according to the formula of 2.6, and when the difference between the average value of twice measurements not exceeding the tolerance of 2.8 and the theoretical composition value exceeds the value given in annex 1 table 2, review the apparatus and procedures.

**Annex 1 Table 2 Theoretical composition value
of sucrose and tolerance**

Unit : %

Composition	Theoretical composition value	Permissible deviation from theoretical composition value
Carbon	42.1	± 0.2
Hydrogen	6.48	± 0.10

Remarks 1 When the filler in the combustion tube is exchanged, the empty burning shall be carried out without fail.

- 2 At the time of measuring the mass of moisture absorber (k) and carbon-dioxide absorber (1), confirm that there is no change of the mass when the mass of protective tube (m) is weighed.

2.6 Calculation of measured value For the carbon and hydrogen content, calculate the numerical value obtained according to the following formula by rounding to one decimal place for carbon, and to two decimal places for hydrogen.

$$C = \frac{m_1}{m_2} \times 27.29 \times \frac{100}{100 - M_s}$$

$$H = \frac{m_2 - \left(m \times \frac{M_s}{100} \right)}{m} \times 11.19 \times \frac{100}{100 - M_s}$$

where, C : Carbon content in sample [mass percentage (%)]
 m_1 : Increased amount of carbon dioxide absorber (g)
 m : Weighing-out amount of sample (g)
 M_s : Moisture content in sample [mass percentage (%)]
 H : Hydrogen content in sample [mass percentage (%)]
 m_2 : Increased amount of moisture absorber (g)

2.7 Number of times of analysis The analysis shall be performed repeatedly twice in the same laboratory. When the difference between twice measured values is within the tolerance ($n = 2$) of annex 1 table 3, the average value of two values shall be treated according to 2.9. If the difference between twice measured values exceeds the tolerance ($n = 2$), further the one-time analysis shall be additionally performed. When the range of three-times measured values (the maximum value – the minimum value) is within the tolerance ($n = 3$), the average value of three values, and when it exceeds the tolerance ($n = 3$), the median value of the three values shall be treated according to 2.9, respectively.

2.8 Tolerance The tolerance of this method shall be given in annex 1 table 3.

Annex 1 table 3 Tolerance on method for determination of carbon and hydrogen (Liebig method)

Unit : mass percentage (%)

Composition	$n = 2$	$n = 3$
Carbon	0.3	0.4
Hydrogen	0.15	0.18

2.9 Report value The carbon and hydrogen contents shall be expressed according to the following procedures.

- a) For the carbon content, in the case of average value, round the numerical value to one decimal place to be expressed, and in the case of median value, express it as it is one decimal place.
- b) For the hydrogen content, in the case of average value, round the numerical value to two decimal places to be expressed, and in the case of median value, express it as it is two decimal places.

3 Sheffield high temperature method

3.1 Summary The sample is heated to about 1 350 °C to be burned in oxygen gas flow at a comparatively high speed, the generated carbon dioxide and steam are absorbed respectively in each absorbent, and the increased amounts are measured.

3.2 Reagents The reagents shall be as follows.

- a) Sulfuric acid As specified in **JIS K 8951**.
- b) Potassium hydroxide or sodium hydroxide Potassium hydroxide specified in **JIS K 8574** or sodium hydroxide specified in **JIS K 8576**.
- c) Silver gauze roll Put a silver core wire of about 2 mm in diameter into the centre of silver gauze roll of 710 µm to 590 µm in aperture, about 100 mm in length, and about 24 mm in diameter (it shall fit in the internal diameter of combustion tube) and make the one end a hook state.

Wash, before using, in a beaker containing boiling water for 5 min, next in a wide mouthed bottle containing aqueous ammonia specified in **JIS K 8085** for 2 min shaking thoroughly, at the end again wash with boiling water and dry sufficiently in an oven at about 200 °C.

This procedure, in the case of sample amount of 0.1 g, shall be performed for each measurement of about 10 times.

- d) Oxygen (Oxygen made by the electrolytic method is not suitable.) As specified in **JIS K 1101**.
- e) Magnesium perchlorate (for analysis) Magnesium perchlorate specified in **JIS K 8228** (for ultimate analysis).
- f) Calcium sulfate (anhydride)
- g) Carbon dioxide absorbent⁽¹⁾ 1 mm to 3 mm in grain size (for analysis).
- h) Sucrose As specified in **JIS K 8383**.

3.3 Apparatus The apparatus (see annex 1 figure 2.1 and annex 1 figure 2.2) shall be as follows.

3.3.1 Oxygen purification train The apparatus almost same as that of Liebig method in 2.3. However, because the gas flow rate is large, each reagent amount shall be increased and the apparatus for removing combustible gas shall be provided necessarily.

3.3.2 Combustion assembly

- a) **Electric furnace** The electric furnace (e) in which silicon carbide or platinum heating element is used, and which is capable of heating the sample at about 1 350 °C, and the electric furnace (f) is capable of heating the silver gauze roll at about 800 °C and further of independently adjusting the furnace temperature respectively.
- b) **Combustion tube** The combustion tube of class 1 or class 2 (30 mm × 24 mm × 750 mm to 800 mm) specified in **JIS R 1307** of which both ends are provided with borosilicate glass cap ground joint respectively.
- c) **Sample push bar** The opaque silica glass tube of 5 mm to 7 mm in external diameter and about 480 mm in length of which one end is sealed in with a small amount of iron pieces and the other end is pushed to be a circular plate of 10 mm to 15 mm in external diameter.
- d) **Magnet** A strong permanent magnet or electromagnet capable of pushing in the boat.
- e) **Combustion boat** The combustion boat of class 1 (16 mm × 12 mm × 80 mm) specified in **JIS R 1306**. It shall be empty-burned necessarily before using.

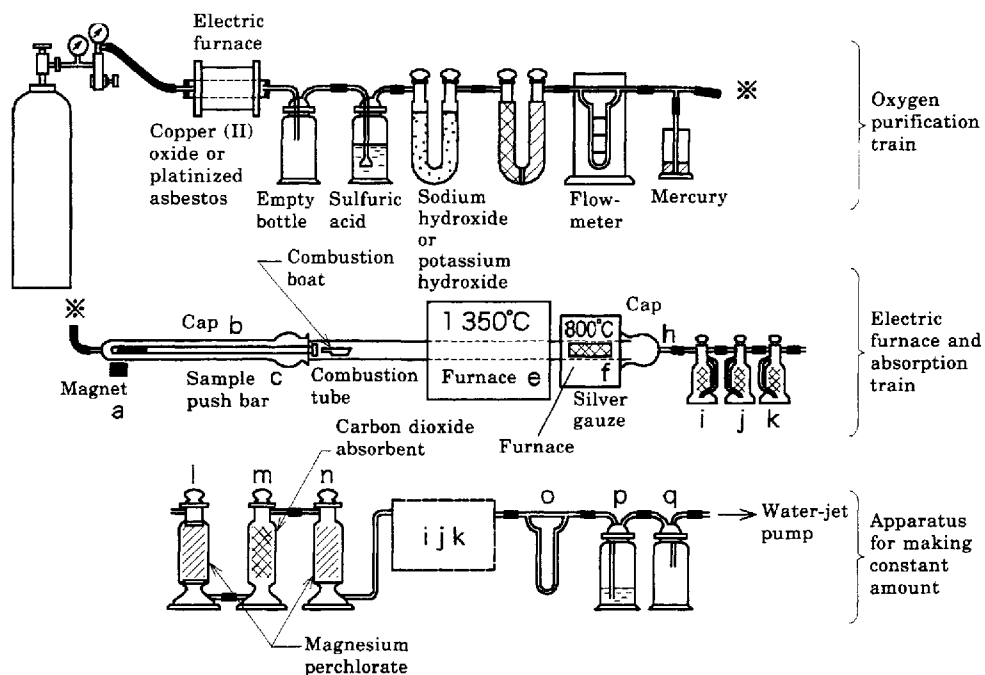
Remarks : Put the boat into a muffle furnace, open slightly the furnace lid to pass electric current, when the temperature is raised to about 1 350 °C, further leave as it is for 1 h, close the furnace lid to stop passing electric current and after standing to cool for a night, preserve in a desiccator. If when empty burning is carried out by using the furnace (e) in **a)**, while flowing oxygen, gradually push it into the highest temperature part and after leaving as it is for 20 min draw out to leave to cool.

3.3.3 Absorption train The absorption train in which moisture absorber (i), carbon dioxide absorber (j) and protective tube (k) are connected in series for the purpose of absorbing combustion gas from the combustion tube.

The moisture absorber is the one filled with magnesium perchlorate [3.2 e)], the carbon dioxide absorber is the one filled with carbon dioxide absorbent [3.2 g)] at the gas inlet side and with a small amount of magnesium perchlorate at the gas outlet side, and the protective tube is the one filled with carbon dioxide absorbent at gas inlet side and with magnesium perchlorate at the outlet side by each equivalent.

3.3.4 Apparatus for making constant amount This apparatus is the one in which moisture absorber, carbon dioxide absorber, and moisture absorber are connected in series.

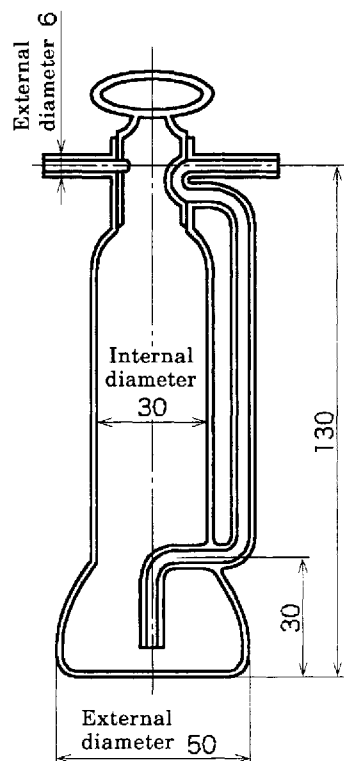
The moisture absorber is the one filled with magnesium perchlorate and the carbon dioxide absorber is the one filled with carbon dioxide absorbent.



Remarks : This figure indicates a gist of connection of each part and also indicates an example of shape.

Annex 1 Figure 2.1 Apparatus for determination of carbon and hydrogen (Sheffield high temperature method)

Unit : mm



Annex 1 Figure 2.2 Absorber for determination of carbon and hydrogen (Sheffield high temperature method)

3.4 Weighing-out amount of sample Weigh out 0.1 g to 0.5 g of the sample to the nearest 0.1 mg.

3.5 Procedures

Warning on safe When performing the combustion procedure, use crucible scissors, etc. for handling the container heated at the high temperature and take care not to burn. Furthermore, make an effort to prevent fire with careful attention to handling of superfluous oxygen exhaust.

3.5.1 Preparatory procedure The preparatory procedure shall be performed as follows.

- a) **Check of apparatus** Before measurement, check the apparatus to confirm that each connecting part and absorbers are airtight completely.
- b) **Heating** While flowing oxygen at a rate of 10 ml per min, raise the temperature to about 1 350 °C for furnace (e) and to about 800 °C for furnace (f).

Thereafter, detach the absorption train, weigh the mass to the nearest 0.1 mg to attach again, and flow oxygen at a rate of 300 ml per min for 10 min.

- c) **Making constant amount** Detach the absorption train, immediately attach to the apparatus for making constant amount, and flow air at a rate of 200 ml per min for 10 min. Thereafter, close the cock, after cooling, weigh the mass to the nearest 0.1 mg to obtain mass increase and decrease of each absorber and repeat the procedure of b) and c) until the value becomes less than 0.5 mg.

3.5.2 Main procedure The main procedure shall be performed as follows.

- a) **Weighing-out and insertion of sample** After the preparation is completed, move the weighed-out sample (3.4) into the combustion boat of which mass is known, detach the cap (b), insert into the end of combustion tube, and again attach the cap.
- b) **Combustion** While flowing oxygen at a rate of 300 ml per min, move the push bar (c) by the magnet (a), push-in the boat into a position at 350 °C to 400 °C, hold at this position normally for 5 min, or depending upon the sample for 5 min to 10 min to burn the volatile component completely. Then move the sample to the position at 1 350 °C and further burn for 5 min to 8 min.
- c) **Marking constant amount** Detach the absorption train, immediately attach to the apparatus for making constant amount, and flow air at a rate of 200 ml per min for 10 min. After cooling, weigh the mass to the nearest 0.1 mg to obtain the increased amount of each absorber.

3.5.3 Discussion by sucrose Weigh out about 0.2 g of sucrose [3.2 h)] to the nearest 0.1 mg, add 0.01 g to 0.02 g of calcium sulfate [3.2 f)], mix sufficiently, perform the same procedures as those of 3.5.2 a) to c), calculate carbon and hydrogen according to the formula of 3.6, and when the difference between the average value of twice measurements not exceeding the tolerance of 3.8 and the theoretical composition value exceeds the value of annex 1 table 4, the apparatus and the procedures shall be reviewed.

Annex 1 Table 4 Theoretical composition value of sucrose and tolerance

Unit : mass percentage (%)

Composition	Theoretical composition value	Permissible deviation from theoretical composition value
Carbon	42.1	± 0.2
Hydrogen	6.48	± 0.10

3.6 Calculation of measured value For the carbon and hydrogen contents, calculate the numerical value obtained according to the following formulae by rounding to one decimal place for carbon, and to two decimal places for hydrogen.

$$C = \frac{m_1}{m_2} \times 27.29 \times \frac{100}{100 - M_s}$$

$$H = \frac{m_2 - \left(m \times \frac{M_s}{100} \right)}{m} \times 11.19 \times \frac{100}{100 - M_s}$$

where C : Carbon content in sample [mass percentage (%)]
 m_1 : Increased amount of carbon dioxide absorber (g)
 m : Weighed-out amount of sample (g)
 M_s : Moisture mass percentage in sample (%)
 H : Hydrogen content in sample [mass percentage (%)]
 m_2 : Increased amount of moisture absorber (g)

3.7 Number of times of analysis The analysis shall be performed repeatedly twice in the same laboratory. When the difference between twice measured values is within the tolerance ($n = 2$) of annex 1 table 5, the average value of two values shall be treated according to **3.9**. If the difference between twice measured values exceeds the tolerance ($n = 2$), further the one-time analysis shall be additionally performed. When the range of three-times measured values (the maximum value — the minimum value) is within the tolerance ($n = 3$), the average value of three values, and when it exceeds the tolerance ($n = 3$), the median value of the three values shall be treated according to **3.9**, respectively.

3.8 Tolerance The tolerance of this method shall be given in annex 1 table 5.

Annex 1 Table 5 Tolerance on method for determination of carbon and hydrogen (Sheffield high temperature method)

Unit : mass percentage (%)

Composition	$n = 2$	$n = 3$
Carbon	0.3	0.4
Hydrogen	0.15	0.18

3.9 Report value The carbon and hydrogen contents shall be expressed according to the following procedures.

- a) For the carbon content, in the case of average value, round the numerical value to one decimal place to be expressed, and in the case of median value, express it as it is one decimal place.
- b) For the hydrogen content, in the case of average value, round the numerical value to two decimal places to be expressed, and in the case of median value, express it as it is two decimal places.

Annex 2 (normative) Method for determination of total sulfur

1 Division of methods The determination of total sulfur shall be performed according to either of the following methods.

- a) Eschka method
- b) High-temperature combustion method

2 Eschka method

2.1 Summary The sample is heated in air together with Eschka mixture, after total sulfur in the sample is fixed as sulfate, the mass as the precipitate of barium sulfate is measured.

2.2 Reagents The reagents shall be as follows.

- a) Hydrochloric acid As specified in **JIS K 8180**.
- b) Aqueous ammonia As specified in **JIS K 8085**.
- c) Barium chloride solution (85 g/L) Dissolve 100 g of barium chloride dihydrate specified in **JIS K 8155** in water to make 1 000 ml. After leaving as it is for not less than 12 h, filter to use.
- d) Silver nitrate solution (20 g/L) Prepare using silver nitrate specified in **JIS K 8550** and preserve by putting it in a brown bottle.
- e) Eschka mixture Mix sufficiently 2 parts of magnesium oxide ⁽¹⁾ specified in **JIS K 8432** and 1 part of sodium carbonate ⁽²⁾ specified in **JIS K 8625**, and preserve it being tightly stoppered.

Notes ⁽¹⁾ Magnesium oxide easily becomes basic magnesium carbonate by absorbing moisture and carbon dioxide in air during storage, and therefore, before preparing Eschka mixture, use it after heating at about 550 °C.

In addition, it is preferable that magnesium oxide to be used herein is not more than 0.13 g/ml in bulk density.

⁽²⁾ Before preparing Eschka mixture, use it after heating at about 700 °C.

- f) Methyl orange solution (1 g/L) Dissolve 0.1 g of methyl orange specified in **JIS K 8893** in 100 ml of hot water.

2.3 Apparatus and instrument The apparatus and instrument shall be as follows.

2.3.1 Electric furnace The electric furnace with good ventilation to which a vent cylinder is attached at the rear part, which is capable of adjusting at 800 °C ± 25 °C and has a large soaking zone ⁽³⁾.

Note ⁽³⁾ When the temperature distribution is measured under the exactly same condition as that under which the sample is heated to be oxidized, it refers to the range which can be maintained at 800 °C ± 25 °C.

2.3.2 Vessels

- a) **Sample heating vessel** The platinum dish (No. 35) specified in **JIS H 6202**. The porcelain crucible (type-B 30 ml) specified in **JIS R 1301** may be used ⁽⁴⁾.
- b) **Crucible for igniting barium sulfate** The porcelain crucible specified in **JIS R 1301** (type A 15 ml).

Note ⁽⁴⁾ The porcelain crucible is corroded by sodium carbonate and ash in sample, becomes impossible to be used in 2 or 3 times, and makes a blank test value larger, and therefore a platinum dish should be used.

2.4 Weighing-out amount of sample Weigh out about 1 g of the sample to the nearest 0.1 mg.

2.5 Procedures

2.5.1 Preparation of sample The preparation procedure of sample shall be performed as follows.

- a) Cover uniformly the bottom of sample-heating vessel [2.3.2 a)] with Eschka mixture [2.2 e)] of about 0.5 g.
- b) Move the weighed-out sample (2.4) into a distillation dish or other suitable vessel, add about 2.5 g of Eschka mixture to mix thoroughly, transfer into a sample-heating vessel [2.3.2 a)] and make it flat.
- c) Then, cover on it with about 1 g of Eschka mixture.

2.5.2 Heating oxidation The procedure of heating oxidation shall be performed as follows.

- a) Put the vessel prepared according to 2.5.1 into an electric furnace (2.3.1) which is made to be kept at room temperature.
- b) Open the door of furnace so slightly that air passes sufficiently, and raise the temperature to 800 °C in 1 h so that the first volatile components gradually evaporate.
- c) Heat at 800 °C ± 25 °C for 1.5 h.

2.5.3 Extraction of sulfate The procedure of extraction of sulfate shall be performed as follows.

- a) Transfer the heated mixture into a beaker (500 ml) containing 25 ml to 30 ml of water as much as possible.
- b) Wash the sample-heating vessel thoroughly with warm water of about 100 ml, and add the wash fluid to the content of beaker.
- c) Cover the beaker by a watch glass, and in order to accelerate dissolution while warming the content of beaker, add carefully hydrochloric acid (usually 14 ml to 17 ml is required) of such amount that the large part of solids is dissolved.
- d) In order to expel carbon dioxide, boil for 5 min.
- e) Filter it into a beaker (500 ml) with the filter paper class 5 B specified in **JIS P 3801**. By using about 20 ml of warm water each wash the filter paper and residue by four times.

2.5.4 Preparation before generation of barium sulfate precipitate The preparation procedure before generation of barium sulfate precipitate shall be performed as follows.

- a) Join the filtrate and washings, add 2 drops or 3 drops of methyl orange solution as indicator, and carefully add aqueous ammonia ⁽⁵⁾ until its colour turns to orange colour to neutralize⁽⁶⁾.
- b) Add 1 ml of hydrochloric acid.
- c) Add water to the solution to make about 250 ml in total volume.

Notes ⁽⁵⁾ Usually, the neutralization point is attained by addition of a small amount of aqueous ammonia, and therefore pipet or burette should be used.

⁽⁶⁾ If at this time insoluble precipitate is generated, add 1 drop or 2 drops (not to be more than it) of hydrochloric acid to redissolve the precipitate.

2.5.5 Generation of barium sulfate precipitate The procedure of generation of barium sulfate precipitate shall be performed as follows.

- a) Cover a beaker with a watch glass, heat it, and when the solution boils, weaken heating in such a degree that boiling occurs slightly.
- b) Add about 10 ml of barium chloride warm solution [2.2 c)] to the centre of hot solution stirring with a pipet ⁽⁷⁾.

Note ⁽⁷⁾ If a transfer pipet of 10 ml is used, 10 ml flows down in about 30 s.

2.5.6 Aging of barium sulfate precipitate Continue heating for 1 h in such a degree that the liquid temperature becomes slightly lower than the boiling point (for example on a warm bath) or stand still at room temperature for not less than 12 h.

2.5.7 Filtration of barium sulfate precipitate The procedure of filtration of barium sulfate precipitate shall be performed as follows.

- a) Filter ⁽⁸⁾ by using the filter paper (class 5 C) specified in **JIS P 3801**.
- b) Wash the precipitate according to the inclination method. Namely, leave the precipitate which was brought to completion of aging standing, and when the supernatant liquid is generated, gently flow out only the liquid content on the filter paper from the beaker. Or leave the beaker standing and flow out only the supernatant liquid. Repeat this procedure.
- c) Add a small amount of warm water to the precipitate remaining in the beaker to mix by stirring, thereafter leave it standing, and filter again the supernatant liquid by inclination. After repeating this procedure several times, transfer the precipitate on the filter paper.
- d) When transferring it on the filter paper, add a small amount of warm water, and flow out the precipitate together with the liquid content while mixing at times.
- e) Spray warm water on the inner wall of the beaker to wash down the precipitate as much as possible and make it flow down toward the bottom of filter paper.

In addition, the precipitate adhered to the inner wall of the beaker and the policeman with the policeman (a glass bar with a rubber tube) wet with water should be rubbed off to be filtered.

- f) The warm water to be used in the procedures of c) to e) shall not exceed 250 ml. Wash with warm water until washings of 20 ml do not indicate milky white colour by silver nitrate solution [2.2 d)] ⁽⁹⁾.

Notes (8) At the time of filtering, in the case of being sucked by a water-jet pump or the like, unless the sufficient reinforcement of filter paper is performed, the precipitate is frequently lost.

(9) The effective washing shall be performed so that chlorine ion may be lost within 250 ml of warm water. For example, the inclination method is also one method, but it is not effective for washing to pour new washings on washings in the funnel while the funnel contains washings.

2.5.8 Ignition of precipitate The procedure of ignition of precipitate shall be performed as follows.

- a) Transfer the wet filter paper into the crucible which is ignited preliminarily and made constant in mass.
- b) Slightly slide the cover to the position ⁽¹⁰⁾ at low temperature of the front part of electric furnace being kept at about 800 °C to enter the crucible, take out the cover when the filter paper is incinerated, gradually move the crucible toward the center of furnace, and heat at about 800 °C for 20 min.

Note ⁽¹⁰⁾ In order to prevent the loss of barium sulfate due to rapid and violent combustion of filter paper, at first, preferably insert in the low temperature part. In addition, if there is a suitable fire resistant plate, place the crucible on it to insert together into the electric furnace.

2.5.9 Measurement of mass of precipitate Cool the barium sulfate which has become constant in mass in a desiccator and weigh the mass to the nearest 0.1 mg.

2.6 Blank test Without using sample, perform exactly the same procedure as that of 2.5.1 to 2.5.9 not less than twice. Allow the average value to be the blank test value.

Remarks : For obtaining the mass of barium sulfate precipitate, instead of a filter paper, a sintered glass filter of crucible type (1G4) specified in **JIS R 3503** may be used. In this case, after filtering the precipitate, heat the filter at about 200 °C to make constant in mass and measure the mass by cooling. In the case of using the sintered glass filter, preliminarily filter barium sulfate precipitate and necessarily confirm that there is no leakage of precipitate.

In addition, the barium sulfate blocking the aperture of sintered glass filter can be detached by dissolution if it is filtered by suction with the hot solution of ethylene diaminetetraacetate tetrasodium (0.1 mol/L).

2.7 Calculation of measured value For the total sulfur content, calculate the numerical value obtained according to the following formulae by rounding to two decimal places.

a) **In the case of expressing according to the air dried basis**

$$S = \frac{m_1 - m_2}{m} \times 13.74$$

b) In the case of expressing according to the anhydrous basis

$$S = \frac{m_1 - m_2}{m} \times 13.74 \times \frac{100}{100 - M_s}$$

where S : Total sulfur content in sample [mass percentage (%)]
 m_1 : Mass of barium sulfate obtained in main procedure (g)
 m_2 : Mass of barium sulfate obtained in blank test (g)
 m : Weighed-out amount of sample (g)
 M_s : Moisture content in sample [mass percentage (%)]

2.8 Number of times of analysis The analysis shall be performed repeatedly twice in the same laboratory. When the difference between twice measured values is within the tolerance ($n = 2$) of annex 2 table 1, the average value of two values shall be treated according to **2.10**.

If the difference between twice measured values exceeds the tolerance ($n = 2$), further the one-time analysis shall be additionally performed. When the range of three-times measured values (the maximum value – the minimum value) is within the tolerance ($n = 3$), the average value of three values, and when it exceeds the tolerance ($n = 3$), the median value of the three values shall be treated according to **2.10**, respectively.

2.9 Tolerance The tolerance of this method shall be given in annex 2 table 1.

Annex 2 Table 1 Tolerance on method for determination of total sulfur by Eschka method

Unit : mass percentage (%)

Division of total sulfur content	$n = 2$	$n = 3$
1.00 or under	0.04	0.05
Over 1.00 up to and incl. 2.00	0.07	0.08
Over 2.00	0.10	0.12

2.10 Report value For the total sulfur content, in the case of average value, round the numerical value to two decimal places to be expressed, and in the case of median value, express it as it is two decimal places.

3 High temperature combustion method

3.1 Summary The sample is heated at about 1 350 °C in oxygen gas flow, oxidize total sulfur to gasify, after collecting this in hydrogen peroxide water, titrate by sodium hydroxide standard solution.

3.2 Reagents The reagents shall be as follows:

- Hydrogen peroxide water Dilute 35 ml of hydrogen peroxide specified in **JIS K 8230** with water to make 1 000 ml, and preserve in a brown bottle.

- b) Sodium hydroxide or potassium hydroxide Potassium hydroxide specified in **JIS K 8576** or sodium hydroxide specified in **JIS K 8574**.
- c) Oxygen
- d) Silica gel Silica gel desiccating agent type A class 1 for package specified in **JIS Z 0701**.
- e) Permanganic acid-sulfuric acid solution The solution mixed at a ratio of 1ml of sulfuric acid (1+1) to 10ml of potassium permanganate solutions (50 g/L).
- f) Mercuric oxycyanide solution (saturated) (required only when carrying out correction of chlorine) Dissolve about 7 g of mercuric oxycyanide in 500 ml of water, filter after mixing by shaking, add 0.01 mol/L sulfuric acid until it becomes faint alkaline and preserve in a brown bottle. This solution lapsed by not less than 4 days shall not be used.
- g) 0.1 mol/L or a 0.05 mol/L sodium hydroxide standard solution
 - 1) 0.1 mol/L sodium hydroxide standard solution In accordance with 4.5 (19.4) of **JIS K 8001**.
 - 2) 0.05 mol/L sodium hydroxide standard solution In accordance with 4.5 (19.5) of **JIS K 8001**.
- h) Methyl red methylene blue mixed liquor In accordance with 4.4 of **JIS K 8001**.
- i) Susrose As specified in **JIS K 8383**.

3.3 Apparatus and instrument The apparatus and instrument shall be as follows (see annex 2 figure 1).

3.3.1 Oxygen purification train The oxygen purification train ⁽¹¹⁾ shall be as follows.

Note ⁽¹¹⁾ In the case where the oxygen not containing such impurities that cause influence on the measured value, the oxygen purification train may be omitted.

- a) Empty bottle (a) The gas washing bottle specified in **JIS R 3503**.
- b) Bottle (b) containing permanganic acid-sulfuric acid solution [3.2 e)] The gas washing bottle specified in **JIS R 3503**.
- c) Tube or tower (c) containing sodium hydroxide or potassium hydroxide [3.2 b)]
The gas washing tower 50 mm specified in **JIS R 3503**.
- d) Tower (d) containing silica gel [3.2 d)] The gas washing tower 50 mm specified in **JIS R 3503**.
- e) Flowmeter (e) Float type or orifice-manometer type flowmeter.
- f) Safety devices (o, p) The gas washing bottle (o) specified in **JIS R 3503** and the measuring cylinder 100 ml (p) specified in **JIS R 3505**.

(o) is, between the oxygen cylinder and the empty bottle (a), and (p) is, between the flowmeter (e) and the combustion tube (g), attached through respective T tubes and put-in with water respectively.

3.3.2 Combustion assembly The combustion assembly shall be as follows.

- a) **Combustion furnace** (f) The tubular electric furnace of about 300 mm in length, and capable of heating the sample at about 1 350 °C. For the heating element, for example, the following dimensions are suitable :

Heat generating part diameter 8 mm, length 200 mm or 250 mm

Shank part diameter 15 mm, length 85 mm

- b) **Combustion tube** (g) Class 2 or class 1 combustion tube (30 mm × 24 mm × 600 mm) specified in **JIS R 1307** of which the one end has borosilicate glass cap with ground joint. The length of ground joint shall be not less than about 20 mm. Fix the combustion tube to the furnace (f) so that the end of combustion tube of cap side protrudes from the furnace wall by 25 mm to 30 mm, raise the maximum temperature in the tube to about 1 350 °C, and flow oxygen at a rate of 500 ml per min. At that time, the temperature of center of cap sphere part shall be 300 °C or higher.
- c) **Sample push bar** (i) Seal the one end of opaque silica glass tube of 5 mm to 7 mm in external diameter and wind spirally or collapse it by pushing to make a circular disc of about 15 mm in diameter to be about 500 mm in total length. Insert this into a glass tube fitted with rubber plug (j), and seal by rubber tube so as to make the bar be able to move back and forth airtightly.
- d) **Combustion boat** (k) The combustion boat of class 1 (16 mm × 20 mm × 80 mm) specified in **JIS R 1306**. Before use, it shall be empty-burned without fail.

Remarks 1 Instead of the apparatus in annex 2 figure 1, the apparatus of such structure as to press-in the sample by using a magnet specified in (d) may be employed.

- 2 Put the combustion boat into the muffle furnace, open the furnace cover slightly, heat, and when the temperature is raised to about 1 350 °C, further leave to stand for 1 h, and close the furnace cover, stop heating, and after allowing to cool for one night, preserve in a desiccator. When performing the empty burning using the furnace (f), push into the highest temperature section gradually flowing oxygen, pull out after leaving to stand for 20 min, and allow to cool.

3.3.3 Absorption train This absorption train is connected with the following devices in series for the purpose of collecting sulfur oxide in the gas generated by combustion.

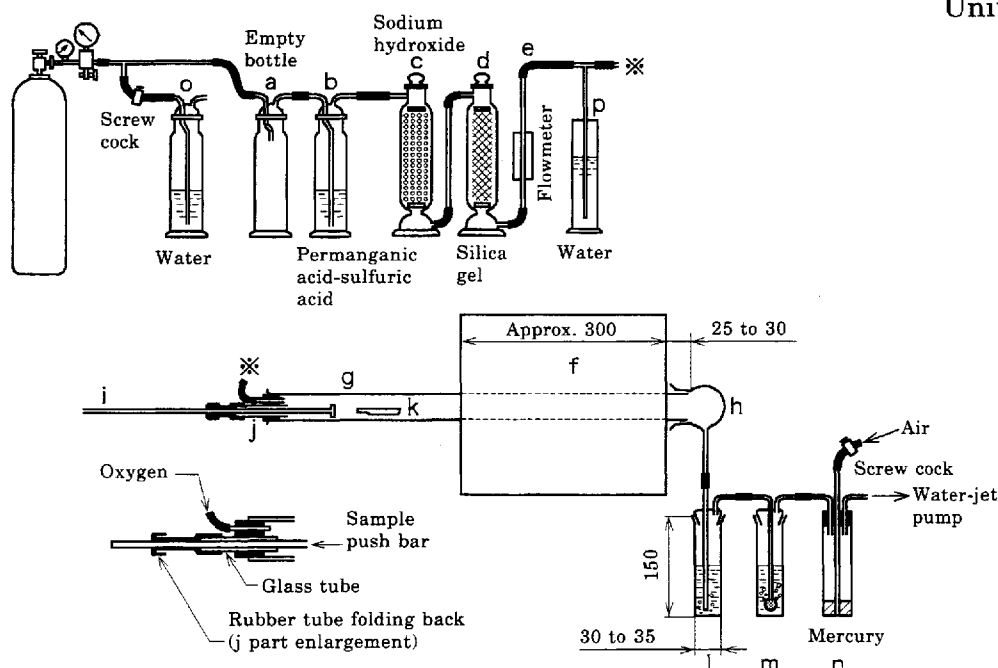
- a) **Absorption bottle** (l) The circular cylinder type bottle of 30 mm to 35 mm in diameter and about 150 mm in height.
- b) **Absorption bottle with filter glass plate** (m) The bottle of the same dimensions as that in a) and the glass filter plate (or sphere) of G2 (diameter, about 15 mm) specified in **JIS R 3503** is attached to it.
- c) **Gas flow rate adjuster containing a small amount of mercury** (n) The circular cylinder shape of about 30 mm in diameter and 150 mm in height.
- d) **Water-jet pump**

In addition, (l) and (m) should be prepared by not less than two sets, respectively.

Remarks : c) and d) may be omitted.

3.3.4 Titration instrument The semi-micro buret of 10 ml in capacity and sub-division mark 0.02 ml or 25 ml in capacity and subdivision mark 0.05 ml.

Unit : mm



Remarks : This figure indicates a gist of connection of each part and also indicates an example of shape.

Annex 2 figure 1 Apparatus for determination of total sulfur (high temperature combustion method)

3.4 Weighing-out amount of sample Depending upon the sulfur content of sample, weigh out 1 g to 0.25 g of sample to the nearest 0.1 mg as given in annex 2 table 2.

Annex 2 Table 2 Weighing-out amount of sample

Sulfur content Mass percentage (%)	Weighing-out amount of sample g
0.5 or under	1
Over 0.5 up to and incl. 3.0	0.5
Over 3.0	0.25

3.5 Procedures

Warning on safety When performing the combustion procedure, use crucible scissors, etc. for handling the container heated at the high temperature and take care so as not to get burned. Furthermore, make an effort to prevent fire with careful attention to handling of superfluous oxygen exhaust. In this test method, when chlorine is corrected, mercuric oxycyanide is used. In this case, since this may cause an environmental hazard, it is desirable to determine chlorides by the ion electrode method or the ion-chromatography.

3.5.1 Preparatory procedure The preparatory procedure shall be performed as follows.