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Contents

	Page
Introduction	1
1 Scope	1
2 Normative references	2
3 Terms and definitions	3
4 Classification	3
5 Quality	3
6 Test method	4
6.1 General requirements for testing apparatus	4
6.2 Sampling method	4
6.3 Test method for vapour pressure (measuring method)	8
6.4 Test method for vapour pressure (calculation method)	15
6.5 Test method for sulfur content (oxyhydrogen flame combustion— barium perchlorate precipitate titration method)	16
6.6 Test method for sulfur content (oxidative microcoulometry method)	25
6.7 Test method for sulfur content (ultraviolet fluorescence method)	33
6.8 Test method for density (measuring method)	42
6.9 Test method for density (calculation method)	47
6.10 Method of analyzing composition (gas chromatography method)	48
6.11 Copper strip corrosion test method	57
6.12 Precautions for testing	66
7 Designation of product	67
8 Marking	67
9 Precautions in manipulation	67
Annex JA (normative) Conversion table for density, mass and volume of liquefied petroleum gases	69
Annex JB (informative) Comparison table between JIS and corresponding International Standards	110

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 Japan LP Gas Association (JLPGA)/ Japanese Standard 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 K 2240:2007** is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

Liquefied petroleum gases

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 3993** published in 1984, the second edition of **ISO 4256** published in 1996, the second edition of **ISO 4257** published in 2001, the second edition of **ISO 6251** published in 1996, the first edition of **ISO 7941** published in 1988, the first edition of **ISO 8973** published in 1997 and the first edition of **ISO 9162** published in 1989 with some modifications of the technical contents.

The portions given sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JB. In addition, the conversion tables for density, mass, and volume of liquefied petroleum gases which are not described in the original Standards are given in Annex JA to supplement 6.8 in the main body.

1 Scope

This Standard specifies liquefied petroleum gases used as fuels for households, businesses, industries, automobiles and the like, and as raw materials for industries.

NOTE : The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 3993:1984 *Liquefied petroleum gas and light hydrocarbons—Determination of density or relative density—Pressure hydrometer method*

ISO 4256:1996 *Liquefied petroleum gases—Determination of gauge vapour pressure—LPG method*

ISO 4257:2001 *Liquefied petroleum gases—Method of sampling*

ISO 6251:1996 *Liquefied petroleum gases—Corrosiveness to copper—Copper strip test*

ISO 7941:1988 *Commercial propane butane—Analysis by gas chromatography*

ISO 8973:1997 *Liquefied petroleum gases—Calculation method for density and vapour pressure*

ISO 9162:1989 *Petroleum products—Fuels (class F)—Liquefied petroleum gases—Specifications* (overall evaluation: MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

WARNING The liquefied petroleum gases specified in this Standard, if not handled or used with adequate precautions measures, can cause dangers. This Standard does not purport to address all of the safety problems associated with the use of liquefied petroleum gases. It is the responsibility of the user of this Standard to determine rules of

operation in accordance with the related laws and regulations, and to take adequate preventive measures for safety and health practice prior to use.

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) listed below shall be applied.

- JIS B 7410 *Liquid-in-glass thermometers for testing of petroleum product*
- JIS B 7505-1 *Aneroid pressure gauges—Part 1: Bourdon tube pressure gauges*
- JIS B 8245 *Valves for liquefied petroleum gas cylinder*
- JIS C 1302 *Insulation resistance testers*
- JIS H 3100 *Copper and copper alloy sheets, plates and strips*
- JIS K 0114 *General rules for gas chromatography*
- JIS K 0512 *Hydrogen*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1101 *Oxygen*
- JIS K 6347-1 *Rubber hoses for liquefied petroleum gases (LPGs)—Part 1: Automobile, General equipment and home application—Specification*
- JIS K 8034 *Acetone (Reagent)*
- JIS K 8223 *Perchloric acid (Reagent)*
- JIS K 8230 *Hydrogen peroxide (Reagent)*
- JIS K 8355 *Acetic acid (Reagent)*
- JIS K 8680 *Toluene (Reagent)*
- JIS K 8809 *Potassium hydrogen phthalate (Reagent)*
- JIS K 8848 *Hexane (Reagent)*
- JIS K 8913 *Potassium iodide (Reagent)*
- JIS K 8962 *Potassium sulfate (Reagent)*
- JIS K 9501 *Sodium azide (Reagent)*
- JIS K 9551 *Barium perchlorate (Reagent)*
- JIS K 9703 *2,2,4-Trimethylpentane (Reagent)*
- JIS R 3505 *Volumetric glassware*
- JIS R 6111 *Artificial abrasives*
- JIS R 6251 *Abrasive cloths*
- JIS R 6252 *Abrasive papers*
- JIS Z 8401 *Guide to the rounding of numbers*
- JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results—Part 6: Use in practice of accuracy values*

3 Terms and definitions

For the purpose of this Standard, the following terms and definitions apply.

3.1 liquefied petroleum gases

liquefied fuel gases and raw material gases for industries whose principal components are propane, propylene, butane and butylene

3.2 pressure liquefied sample

liquid sample taken from a tank or a pipeline stored in a pressurized state

3.3 low temperature liquefied sample

liquid sample taken from a tank or a pipeline stored at a low temperature in a state close to atmospheric pressure

4 Classification

Liquefied petroleum gases shall be classified into two classes according to the use: Class 1 and Class 2, and Class 1 is further subdivided into No. 1 to No. 3, and Class 2 into No. 1 to No. 4 according to the composition as shown in table 1.

5 Quality

When liquefied petroleum gases are tested by the testing method of clause 6, the results shall meet the requirements specified in table 1.

NOTE : Liquefied petroleum gases are stipulated in the High Pressure Gas Safety Act that odorants shall be added except those used for manufacturing aerosol or gas lighters and other industrial purposes (as fuels and raw materials).

Table 1 Classification and quality

Classification		Composition mol %				Sulfur content mass fraction %	Vapour pressure (40 °C) MPa	Density (15 °C) g/cm ³	Copper strip corrosion (40 °C, 1h)	Principal uses			
		Ethane + ethylene	Propane + propylene	Butane + butylene	Buta- diene								
Class 1	No. 1	5 or under	80 or over	20 or under	0.5 or under	0.005 0 or under ^{b)}	1.53 or under	0.500 to 0.620	1 or under	Fuel for household Fuel for business			
	No. 2		60 or over to and excl. 80	40 or under									
	No. 3		Under 60	30 or over									
Class 2	No. 1	—	90 or over	10 or under	— ^{a)}		0.005 0 or under ^{b)}			1.55 or under	0.500 to 0.620	1 or under	Fuel for industries Raw material for industries Fuel for automobiles
	No. 2		50 or over to and excl. 90	50 or under									
	No. 3		Under 50	50 or over to and excl. 90									
	No. 4		10 or under	90 or over									
Test method		6.10				6.5, 6.6 or 6.7		6.3 or 6.4	6.8 or 6.9	6.11			

Table 1 (concluded)

For the sulfur content, vapour pressure, density test method, at least two of them are specified, however, in such a case, any one of the test methods may be used. However, for the vapour pressure and density test method, the measuring method (6.3 and 6.8) shall be used in principle. And the test method adopted shall be additionally mentioned in test result.

NOTE : "Butane + Butylene" means a mixture of iso-butane, *n*-butane, iso-butylene, 1-butylene, trans-2-butylene and cis-2-butylene. "Butadiene" represents 1,3-butadiene.

Notes ^{a)} In the case of the uses for automobiles, industries (as fuels and raw materials) and others, the content of butadiene shall not be detrimental to the using purposes.

^{b)} The specification under the condition before odorant, etc. is added.

6 Test method

6.1 General requirements for testing apparatus

A testing apparatus shall incur no danger under ordinary using conditions and shall have enough durability and a correct shape. It shall be assembled reliably and free from mechanical and electrical troubles.

The insulation resistance of the testing apparatus shall be 5 MΩ or over when measured between the supply terminals and the enclosure with the electrical circuits closed, by a 500 V insulation resistance tester specified in JIS C 1302. However, when it includes an electric heating circuit, 0.5 MΩ or over will suffice.

In the case where the inner capacity exceeds 100 ml, the vessel shall comply with the "Containers Safety Regulations, High Pressure Gas Safety Act". The above stated vessels shall be fitted with valves and safety valves having passed the "inspection of attachment parts" according to the "Containers Safety Regulations". In the case of expiring the inspection period for attachment parts, only those having passed the "reinspection of attachment parts" shall be mounted on the vessels.

6.2 Sampling method

6.2.1 Outline of sampling method

Wash the sample vessel with the sample in advance, take the liquid sample of about 80 % of the vessel capacity for the pressure liquefied sample, and about 70 % for the low temperature liquefied sample.

The sample shall be appropriately taken according to the apparatus so as to represent the quality of the liquefied petroleum gas under test.

6.2.2 Sampling apparatus

The construction of the apparatus shall be as shown in figure 1 and consist of a) and b).

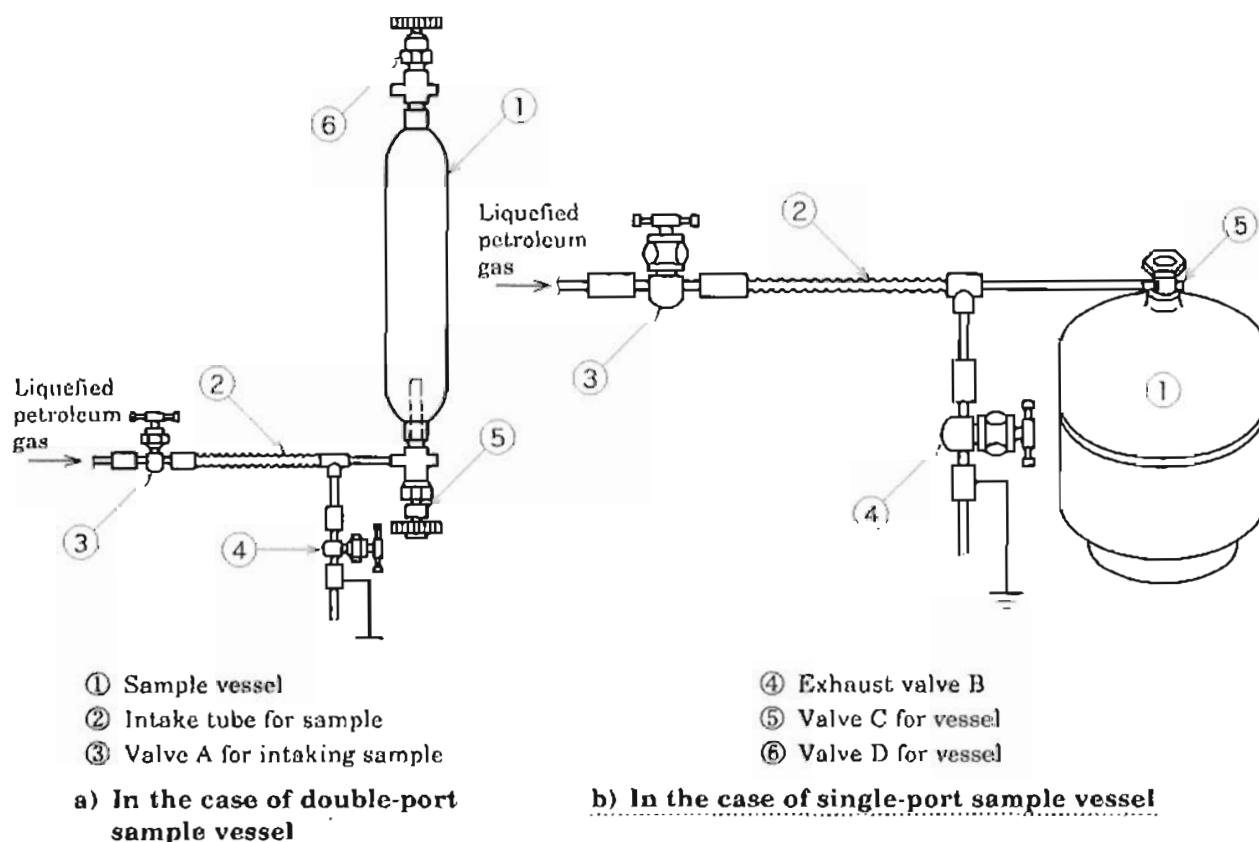


Figure 1 Sampling apparatus (example)

- a) **Intake tube for sample** The tube shall be flexible, made of stainless steel, aluminium alloy or copper to withstand the pressure of 7.85 MPa or over, and provided with the exhaust valve B as shown in figure 1. However, the copper intake tube shall not be used for the sample for determining sulfur content. Further, when a sample is introduced, earthing shall be performed in order to remove static electricity.
- b) **Sample vessel** The construction shall be as shown in figure 2, and the vessel shall be made of stainless steel, steel, or aluminium alloy of double-port or single-port type, which withstands the pressure of 3.04 MPa or over and can be connected to the intake tube for sample.

Vessel valves shall have the dimensions and the constructions specified in JIS B 8245.

Unit: mm

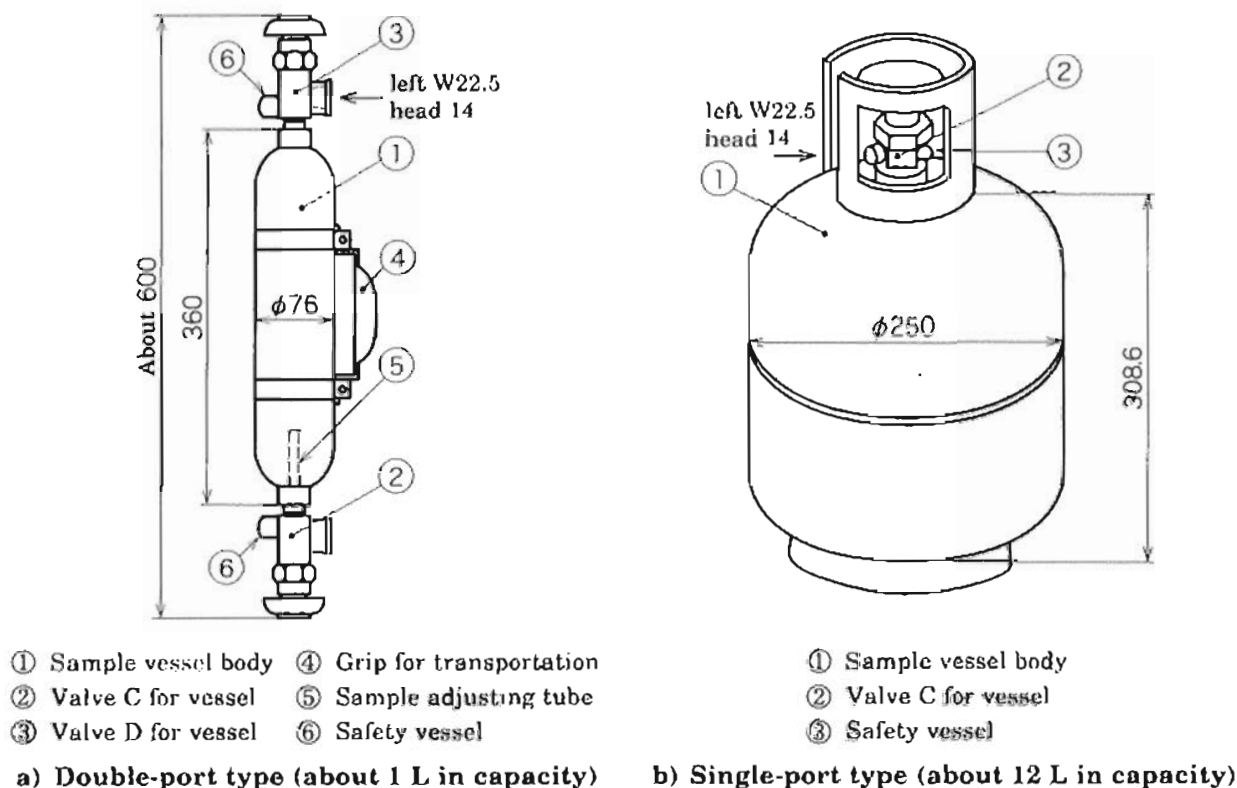


Figure 2 Sampling apparatus (example)

6.2.3 Preparation for sampling (refer to figure 1)

The preparation for sampling shall be as follows.

- Washing of new vessel** When using the new vessel, it should be washed.
- Washing of intake tube with sample** Previously displace contents of the intake tube for sample up to the connecting port of the sample vessel, by the liquefied petroleum gas, place the sample vessel vertically, and then tightly connect valve A for intaking sample with valve C for sample vessel using the intake tube for sample without leakage.

In the case of the double-port sample vessel, arrange the sample adjusting tube installed in the sample vessel or valve C to be positioned at the bottom, and in the case of the single port sample vessel, arrange valve C at the top.

In addition, keep valve A, valve B and valve C in the closed state, then open valve A, next open valve B, and close valve B after filling the intake tube for sample up with the sample.

- Washing of sample vessel with sample** Washing of the sample vessel of double-port type and single-port type with the sample shall be as follows.
 - Double-port sample vessel** Open valve C, and then introduce the sample to the lower portion by opening valve D gradually. Close valve C, release part of the sample in gaseous state through valve D, and close valve D. Then invert the sample vessel, release the remainder in liquid state through valve D, then close

valve D and return the sample vessel to the original position. Repeat this operation at least three times.

For the low temperature sample, cool the sample vessel nearly to the liquid temperature by this operation.

- 2) **Single-port sample vessel** After introducing the sample into the sample vessel by opening valve C, close valve A, and release part of the sample in gaseous state by opening valve B. Invert the sample vessel, and after releasing the remainder in liquid state through valve B, close valve B, and return the sample vessel to the original position. Repeat this operation at least three times, then close valve C, and open valve A. For the low temperature sample, cool the sample vessel nearly to the liquid temperature by this operation.

6.2.4 Procedure of sampling (refer to figure 1)

Fill the sample vessel with the sample by opening valve C. Close valve C and valve A, decrease the pressure in the intake tube for sample perfectly by opening valve B, then disconnect the sampling port, the intake tube for sample and the sample vessel. Check for leakage of the sample vessel visually and by sound. When leakage exists or valve C is mistakenly opened before the operation in 6.2.5, discard the sample.

NOTE : When the sample vessel is decompressed previously, it should be easier to take the sample.

6.2.5 Adjustment of sampling quantity

Immediately after taking the sample, adjust the sample quantity either to about 80 % of the capacity of the sample vessel for the ordinary temperature sample, or to about 70 % for the low temperature sample.

- a) **In the case of the vessel with sample adjusting tube** For the sample vessel provided with a sample adjusting tube, stand the sample vessel vertically with the sample adjusting tube at the top, and gradually open valve C on the sample adjusting tube side. In this case, the excess sample escapes in liquid state and, when the content reaches the specified quantity, the sample is converted into gaseous state. Therefore, immediately close valve C. When the sample escapes in gaseous state from the beginning, the sample shall be discarded.
- b) **In the case of the vessel without sample adjusting tube** Obtain the sample mass by subtracting the mass of empty sampling vessel from the mass of the sample vessel with sample. In the case of taking the sample to excess, adjust by releasing the sample gradually through the lower valve by inverting the sample vessel.

6.2.6 Marking of sample

A tag, a label, etc. on which the class of the sample taken, place, date, etc. are described shall be attached to the sample vessel.

6.2.7 Precautions for preservation of sample

When preserving the sample, tightly stopper the valve for vessel so that the sample taken does not leak and then transfer the vessel immediately to a cool place away from direct sunlight.

6.3 Test method for vapour pressure (measuring method)

6.3.1 Principle

Fill the sample chamber of the vapour pressure tester (6.3.2) with the sample, discard the sample in the lower sample chamber and put the tester in a thermostatic water bath kept at 40 °C. Read the indication of the pressure gauge when pressure becomes constant, and take it as the vapour pressure.

NOTE : This test method may also be applied to a test whose test temperature ranges from 35 °C to 42 °C.

6.3.2 Vapour pressure tester

The tester shall have the construction as shown in figure 3 and consist of the following.

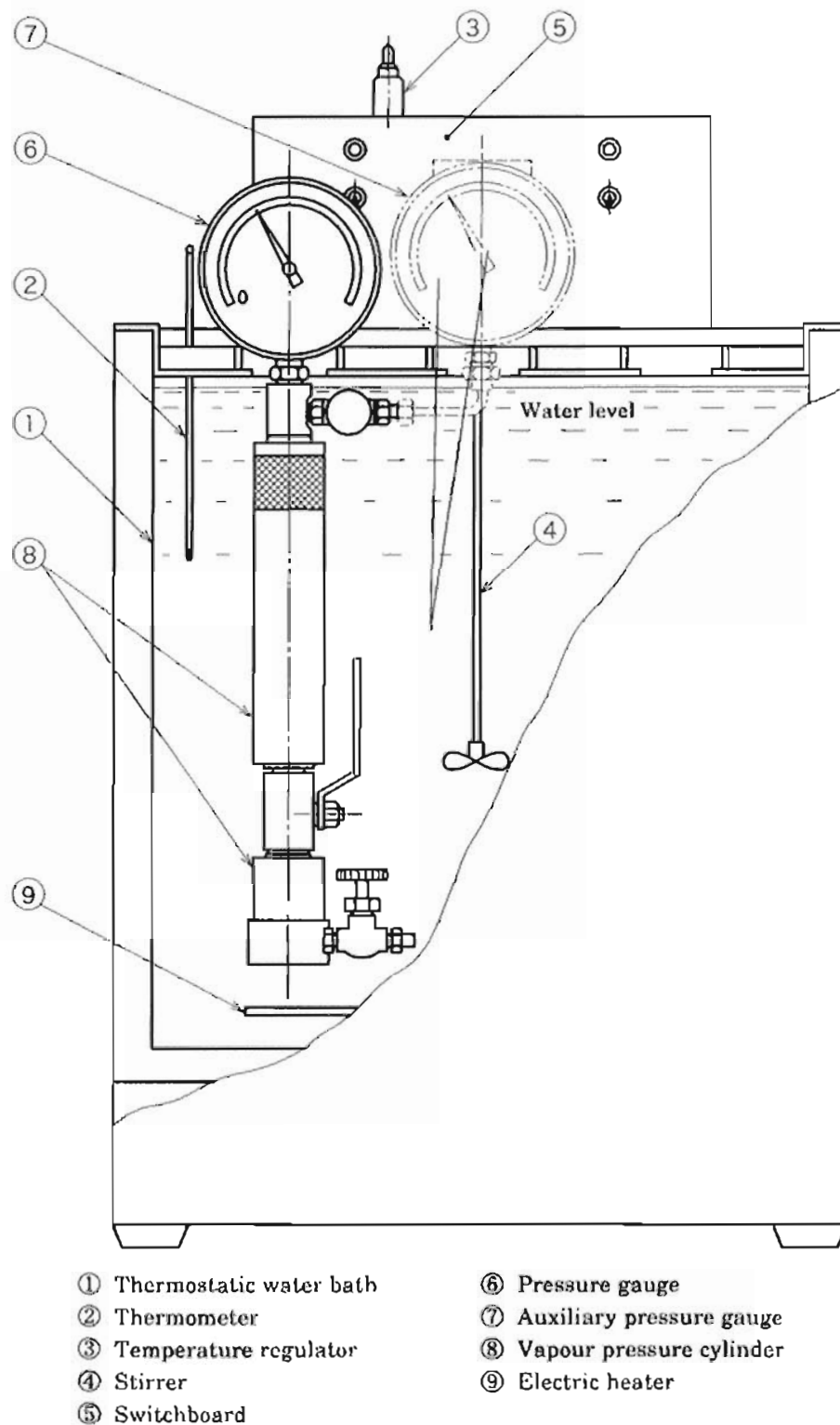


Figure 3 Vapour pressure tester (example)

- a) **Vapour pressure cylinder** The cylinder, having the shape as shown in figure 4 and consisting of the following 1) to 3), shall keep the airtightness for 3.43 MPa of internal pressure and shall withstand the pressure of 6.86 MPa or over. The cylinder shall be inspected based on "High Pressure Gas Safety Act" to be judged acceptable.

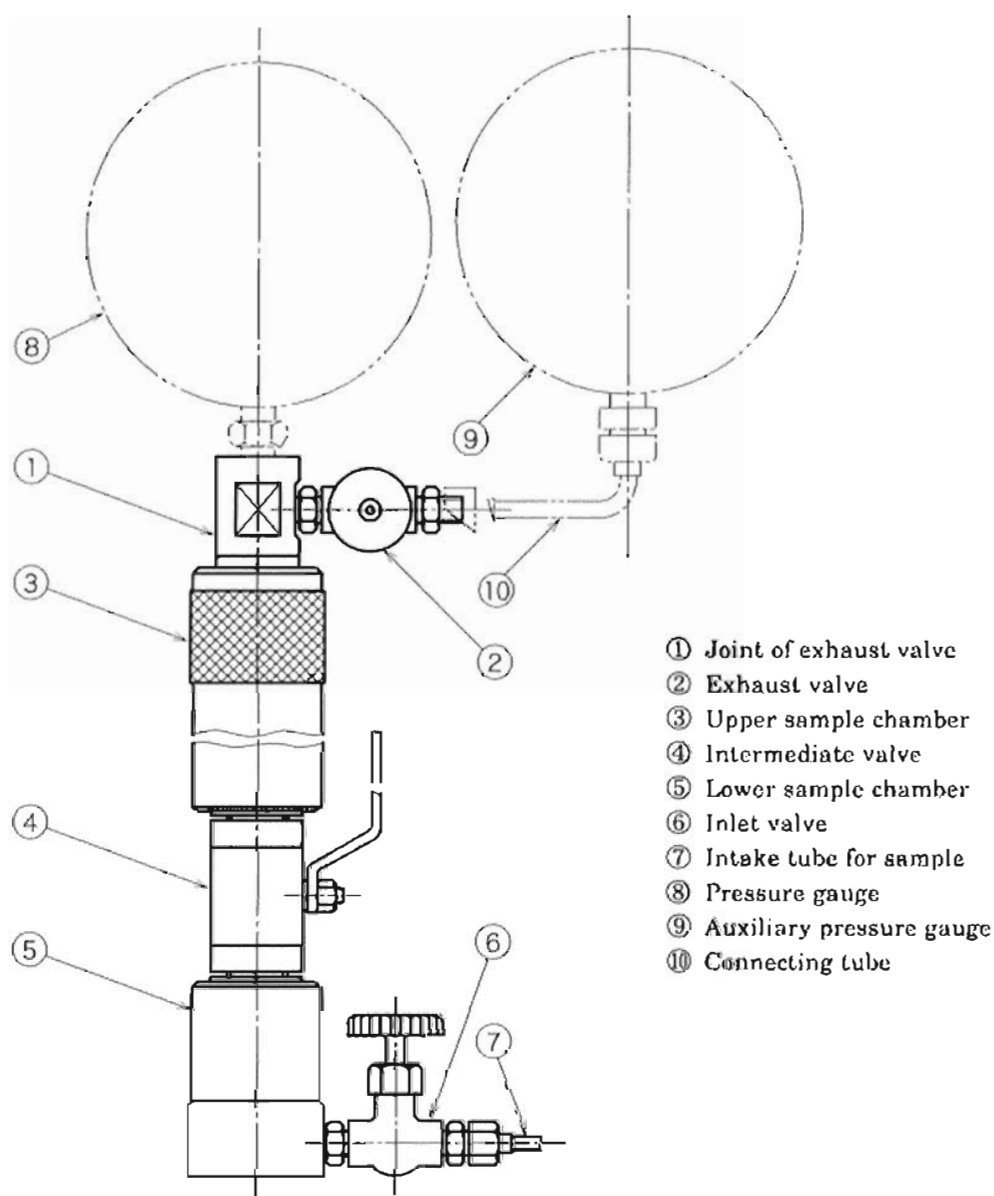


Figure 4 Vapour pressure cylinder (example)

- 1) **Upper sample chamber** The chamber shall be a pressure vessel whose shape and dimensions are as shown in figure 5, made of stainless steel (SUS 304) or a material with a corrosion resistance property equal or superior and further connectable with a joint of exhaust valve at the upper end and with an intermediate valve at the lower end. When it stands vertically, slight inclination is given to the inner surfaces of both ends so that the liquid sample in the sample chamber flows out perfectly.
- 2) **Lower sample chamber** The lower sample chamber shall be a pressure vessel whose shape and dimensions are as shown in figure 6, made of stainless steel and further connectable with an intermediate valve at the upper end and with an inlet valve in the bottom side.

In the lower sample chamber, there are $\frac{1}{3}$ and $\frac{1}{5}$ sample chambers, and either of them may be used. The volume ratio of lower sample chamber and upper sample chamber shall be 1:1.97 to 2.03 for the $\frac{1}{3}$ sample chamber and 1:3.94 to 4.05 for the $\frac{1}{5}$ sample chamber.

- 3) **Intermediate valve** The valve shall be of corrosion resistant metal and a straight way type as shown in figure 6, and the minimum inside diameter of the path shall be 10 mm or over.

Unit: mm

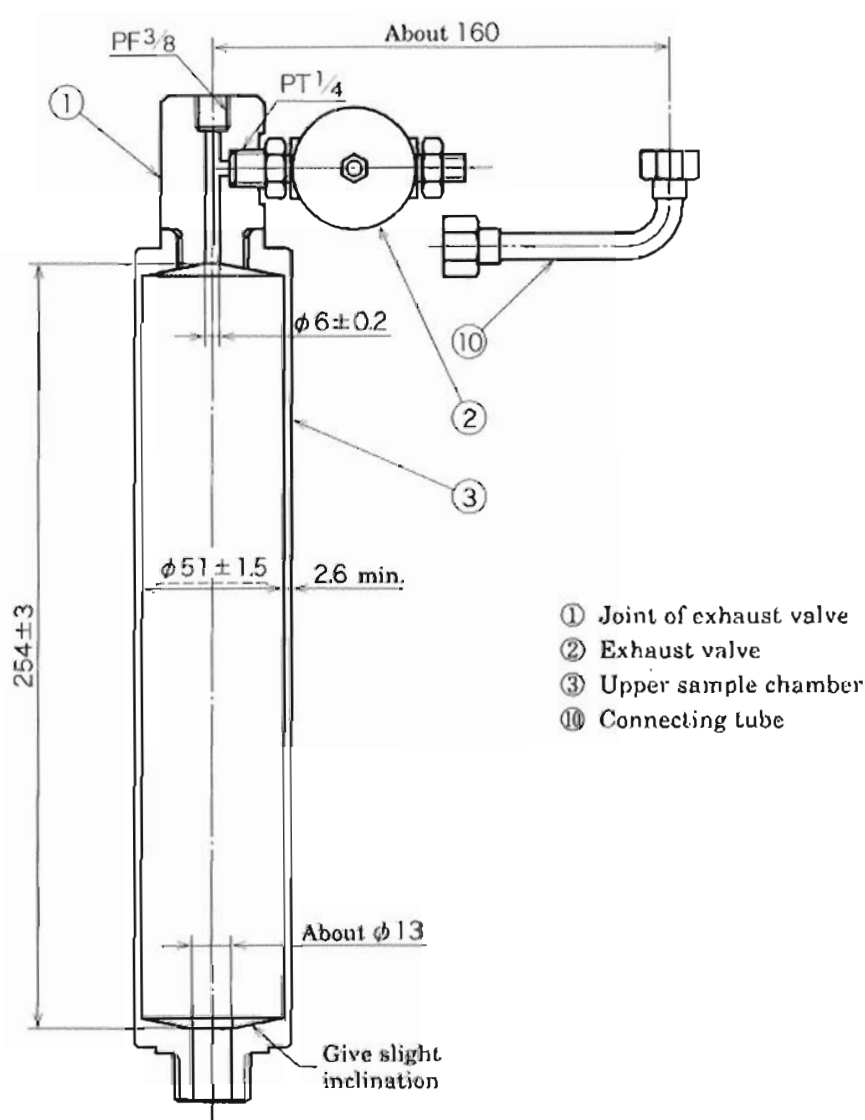


Figure 5 Upper sample chamber (example)

Unit: mm

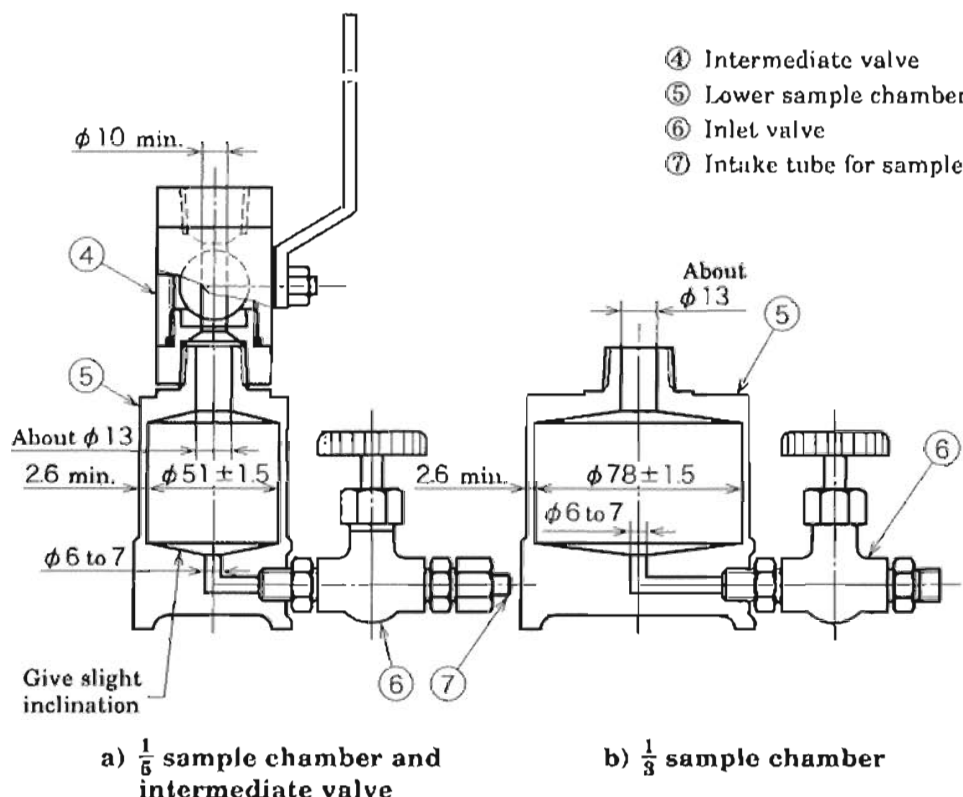


Figure 6 Lower sample chamber and intermediate valve (example)

- b) **Thermostatic water bath** The bath as shown in figure 3 shall be capable of immersing the vapour pressure cylinder to the top of the exhaust valve, and of keeping the bath temperature at $40\text{ }^{\circ}\text{C} \pm 0.1\text{ }^{\circ}\text{C}$.
- c) **Pressure gauge and others** The pressure gauge and others shall be as follows.
- 1) **Pressure gauge** That specified in **JIS B 7505-1** and used according to the prospected vapour pressure of the sample shown in table 2.

Table 2 Pressure gauge according to vapour pressure of sample

Unit: MPa

Vapour pressure of sample	Pressure gauge
0.7 or under	A ⑥ $\frac{3}{8} \times 150 \times 1.0\text{ MPa}$
0.6 to 1.8	A ⑥ $\frac{3}{8} \times 150 \times 2.5\text{ MPa}$
1.6 to 2.9	A ⑥ $\frac{3}{8} \times 150 \times 4.0\text{ MPa}$

- 2) **Intake tube for sample** The tube of about 6 mm in diameter shall be pressure resistant, corrosion resistant, flexible, and capable of being connected on one end to the valve of the sample vessel, and the other end shall be provided with a PF $\frac{1}{4}$ cap nut to connect to the inlet valve of the vapour pressure cylinder.
- 3) **Thermometer** VAP-2 specified in **JIS B 7410**.

6.3.3 Preparation of tester

Clean every part of the vapour pressure cylinder and the pressure gauge, dry with dried air, and then assemble the vapour pressure cylinder and the pressure gauge.

6.3.4 Procedure of test

The procedure of the test shall be as follows.

- a) **Displacement of gas in vapour pressure cylinder** The displacement of gas in vapour pressure cylinder shall be as follows.
 - 1) Open the inlet valve and intermediate valve, close the exhaust valve, immediately erect the vapour pressure cylinder upright, connect the inlet valve and the sample vessel with the intake tube for sample and open the valve (valve C) of the sample vessel. Then, open the exhaust valve, and fill the sample chamber with the liquid sample while exhausting the air or vapour from the sample chamber.
 - 2) Close the exhaust valve with the intake tube for sample attached, and then close the inlet valve. Immediately, invert the vapour pressure cylinder, open the exhaust valve, and keep this state until all the liquid sample is exhausted. When the pressure of the vapour pressure cylinder comes nearly equal to the atmospheric pressure, close the exhaust valve.
- b) **Sampling introduction into vapour pressure cylinder** The sample introduction into the vapour pressure cylinder shall be as follows.
 - 1) Return the inverted vapour pressure cylinder to the original position and open the inlet valve. When the pressure in the vapour pressure cylinder shows the same pressure as that in the sample vessel, immediately open the exhaust valve. When the sample does not come out in liquid state from the exhaust valve, repeat the procedure a). When the liquid sample comes out, immediately close the exhaust valve, the inlet valve and the sample vessel valve in this order, and disconnect the intake tube for sample.
 - 2) Keep the vapour pressure cylinder standing upright, immediately close the intermediate valve, open the inlet valve, and exhaust the sample from the lower sample chamber quickly. After exhausting the liquid sample from the lower sample chamber, close the inlet valve, open immediately the intermediate valve, and fill the lower sample chamber with the sample.
 - 3) In the case of using the $\frac{1}{3}$ lower sample chamber, move on to procedure c), and in the case of using the $\frac{1}{6}$ lower sample chamber, repeat the procedure of 2) once again.
- c) **Measurement of vapour pressure** The measurement of vapour pressure shall be as follows.
 - 1) After shaking the vapour pressure cylinder vigorously several times while inverting it, return it to the original position, and then immerse it into the thermostatic water bath previously kept at $40\text{ }^{\circ}\text{C} \pm 0.1\text{ }^{\circ}\text{C}$ up to the top of the exhaust valve of the cylinder.

- 2) Examine the existence of leakage during the immersion of the vapour pressure cylinder in the thermostatic water bath. When leakage is observed, stop the test immediately, and repeat the test from the procedure of 6.3.3.
- 3) Immerse the vapour pressure cylinder in the thermostatic water bath, take it out after a lapse of 5 min, invert and shake it vigorously and return it again to the water bath. In this case, this procedure shall be carried out quickly so that the vapour pressure cylinder and the sample are not cooled.

At an interval of 2 min or over, repeat the procedure of taking out the vapour pressure cylinder from the thermostatic water bath, inverting and shaking it vigorously and returning it to the bath. In this case, before taking out the cylinder from the bath, read the indication of the pressure gauge by tapping it gently. Repeat these procedures until two successive indications of the pressure gauge show the same value.

NOTE : Usually it takes 20 min to 30 min until the same value is obtained.

- 4) When two successive indications of the pressure gauge show the same value, read the value to two places of decimals. When there is doubt about indication of the pressure gauge due to shaking and the like, use the auxiliary pressure gauge, and correct by the following procedures.
 - 4.1) After completion of the measurement of 6.3.4 c) 4), connect the connecting tube to the exhaust valve under the condition of the vapour pressure cylinder is immersed in the thermostatic water bath with the pressure gauge attached.
 - 4.2) Attach the auxiliary pressure gauge previously corrected by the standard dead-weight pressure gauge to the connecting tube, and open the exhaust valve.
 - 4.3) After a lapse of 5 min, read the indications of both pressure gauges to two places of decimals and correct the value obtained in the measurement of 6.3.4 c) 4) with the value read on the auxiliary pressure gauge.

6.3.5 Result

Designate the indication of the pressure gauge or the value corrected by the auxiliary pressure gauge as the vapour pressure, and express it in MPa.

To convert the vapour pressure to the vapour pressure under standard atmospheric pressure, use the following formula.

$$P = P_1 - (P_0 - P_2)$$

where, P : vapour pressure under standard atmospheric pressure (MPa)

P_1 : vapour pressure (MPa)

P_0 : standard atmospheric pressure (MPa)

P_2 : atmospheric pressure at the time of measurement (MPa)

When the vapour pressure is measured at a temperature other than 40 °C, the test temperature shall be stated.

6.3.6 Precision

The tolerance (probability 0.95) on the test results obtained by this test method shall be as follows.

When the test results exceed the tolerance, they shall be treated according to the specification of **JIS Z 8402-6**.

- a) **Repeatability** When a person has tested twice continuously within a short time the same sample with the same tester in the same laboratory, the difference between the test results shall not exceed 0.02 MPa.
- b) **Reproducibility** Not specified.

6.4 Test method for vapour pressure (calculation method)

6.4.1 Principle

Calculate the vapour pressure of the sample from the composition of the sample and the vapour pressure of each hydrocarbon. The results obtained by this method may be used instead of the results carried out in 6.3 when the component of ethane plus ethylene is not more than 1.5 % (mol fraction). When the component of ethane plus ethylene exceeds 1.5 % (mol fraction), this may be used only when no difference for collation with the results carried out in 6.3 is confirmed.

6.4.2 Calculation method

Calculate the vapour pressure of the sample from the following formula by using the analytical values of components obtained by the method of analyzing composition in 6.10 and the vapour pressure of each component as given in table 3.

$$P_{40} = \Sigma \left(P_i \times \frac{C_i}{100} \right)$$

where, P_{40} : vapour pressure at 40 °C (MPa)

P_i : vapour pressure of component i at 40 °C given in table 3 (MPa)

C_i : content of component i by the method in 6.10 (mol fraction %)

Table 3 Vapour pressure of each hydrocarbon

Component i	Vapour pressure (40 °C) (MPa)
Ethane + ethylene ^{a)}	5.000
Propane	1.275
n-Butane	0.278
iso-Butane	0.427
n-Pentane	0.015
iso-Pentane	0.050
Propylene	1.542
1-Butylene	0.356
cis-2-Butylene	0.235
trans-2-Butylene	0.265
iso-Butylene	0.365
1,3-Butadiene	0.355
Note ^{a)} Since ethane and ethylene cannot be separated by the method of analyzing composition in 6.10, the vapour pressure of ethane is adopted as the component of ethane+ethylene.	

6.4.3 Result

Calculate the result to three places of decimals and round off to two places of decimals in accordance with **JIS Z 8401**.

6.5 Test method for sulfur content (oxyhydrogen flame combustion–barium perchlorate precipitate titration method)

6.5.1 Principle

Burn the sample in oxyhydrogen flame, allow the produced sulfur oxides to be absorbed in hydrogen peroxide solution to convert it to sulfuric acid. Concentrate or collect partially the absorbent solution, add the buffer solution and acetone to the solution, titrate the sulfate ion in the solution with standard barium perchlorate solution using dimethyl-sulfonazo III as the indicator, and determine the sulfur content in the sample. This method shall be applied to the sample whose sulfur content is 0.000 1 mass % or over.

This method is not applied to the sample whose halogen content is 0.010 0 mass % or over.

6.5.2 Tester for sulfur content

The tester shall consist of the following a) to c) (refer to figure 7).

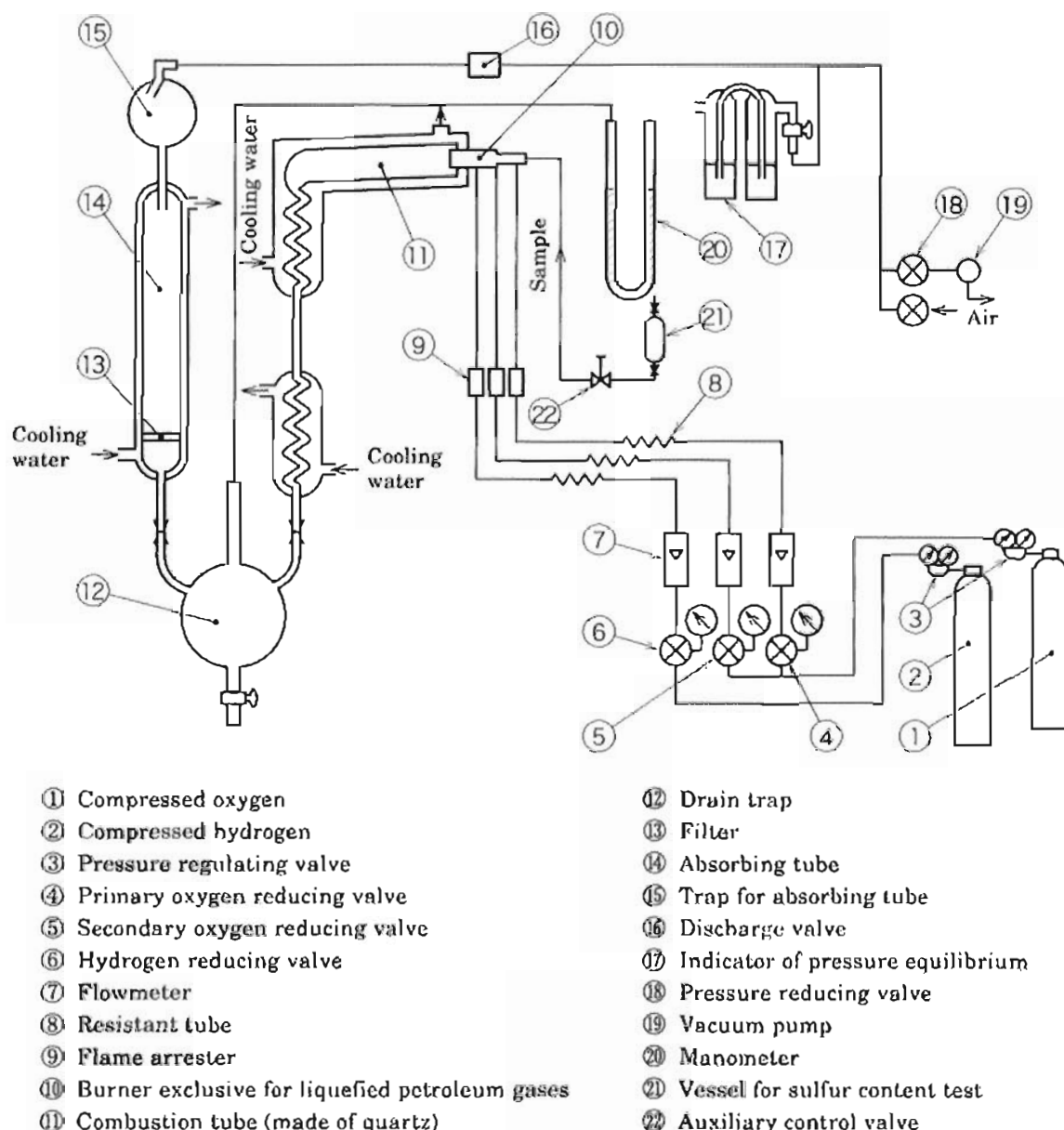


Figure 7 Tester for oxyhydrogen flame combustion—barium perchlorate precipitate titration method (example)

- a) **Oxyhydrogen flame combustion apparatus** The apparatus shall consist of a burner exclusive for liquefied petroleum gases (hereafter referred to as "burner"), a combustion tube, an absorbing tube and the like as shown in figure 7, and to ensure safety in operation, the following shall be provided.
- 1) **Discharge valve** Capable of releasing the combustion gas automatically in the air at the time of the power failure.
 - 2) **Flame arrester** Capable of preventing flame of the burner from backfiring to the conduit of oxygen or hydrogen.
 - 3) **Protecting board** Shall be mounted on the front of the apparatus.

b) **Vessel for sulfur content test and vapourizer**

- 1) **Vessel for sulfur content test** The construction of the vessel shall be as shown in figure 8. The vessel shall withstand the pressure of 3.04 MPa or over, be of double-port type or single-port type, made of stainless steel (SUS 304 or a material having corrosion resistant property equal or superior), with 90 ml to 99 ml in capacity, and be provided with a safety valve (2.06 MPa) and a needle valve.

Unit: mm

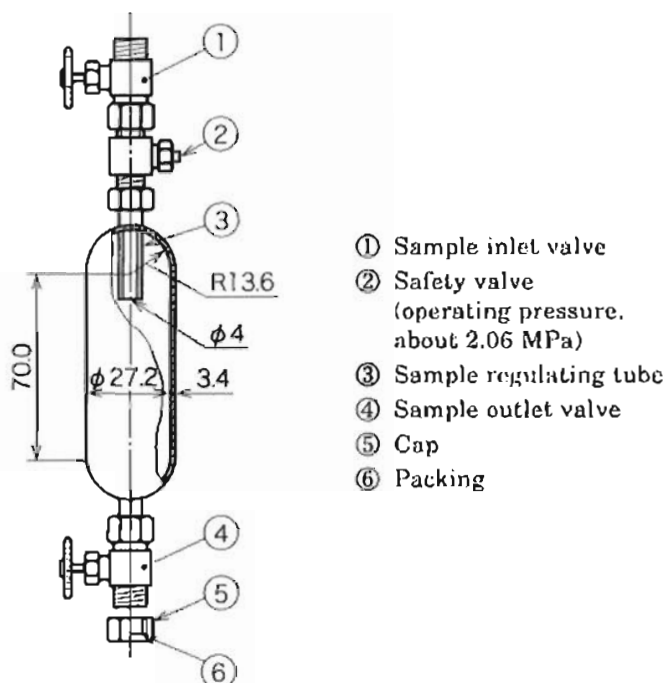


Figure 8 Vessel for sulfur content test (example)

- 2) **Vapourizer** The vapourizer shall be a connecting tube for joining the vessel for sulfur content test and the burner provided with an auxiliary control valve having an airtightness for 1.82 MPa of internal pressure and a withstanding pressure of 3.04 MPa or over, made of stainless steel (SUS 304 or a material having corrosion resistant property equal or superior) (refer to figure 9).

In necessary for combustion, use an appropriate water bath (60 °C to 80 °C) for heating the auxiliary control valve.

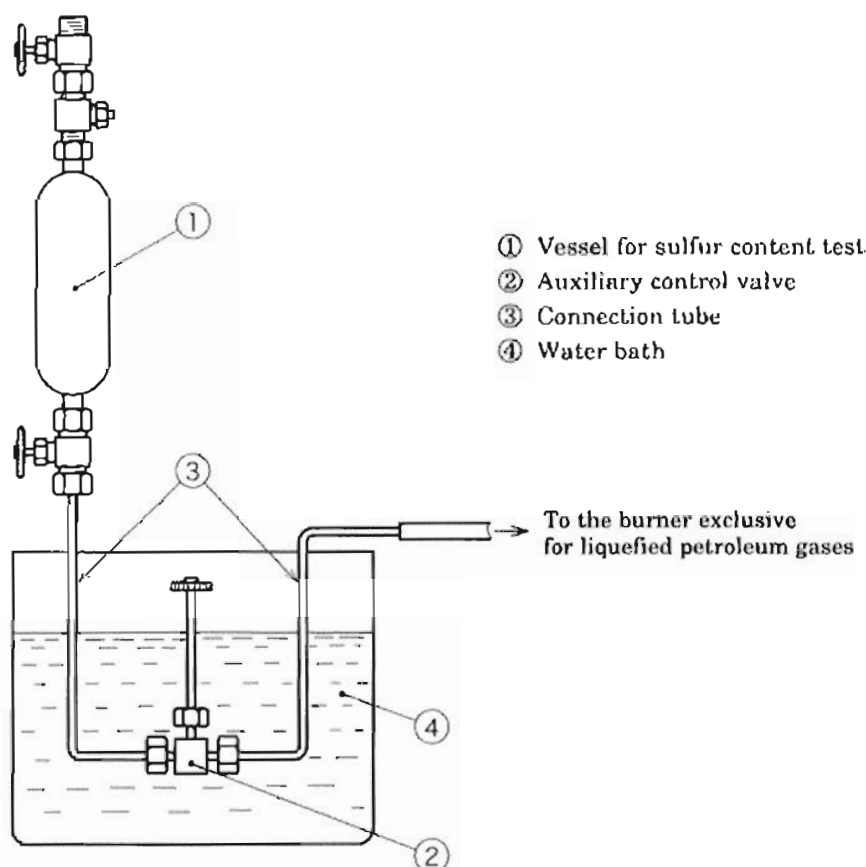


Figure 9 Sample vapourizer system diagram (example)

c) **Vacuum pump and others**

- 1) **Vacuum pump** The pump shall be an oilless vacuum pump having an exhaust capacity of 25 L/min or over.
- 2) **Intake tube for sample** In accordance with 6.2.2 a).

6.5.3 **Reagents**

Reagents to be used shall be as follows.

- a) **Oxygen** As specified in JIS K 1101.
- b) **Hydrogen** Grade 3 or Grade 4 specified in JIS K 0512.
- c) **Acetone** As specified in JIS K 8034.
- d) **Water** A2 or A3 in classification and quality specified in clause 4 of JIS K 0557 or that equivalent to or higher grade.
- e) **Hydrogen peroxide (3 %)** That obtained by adding 9-volume of water to 1-volume of hydrogen peroxide specified in JIS K 8230. In the case of preservation, it shall be stored in a brown bottle with a ground stopper.
- f) **Potassium hydrogen phthalate buffer solution (20 g/L)** Dissolve 20 g of potassium hydrogen phthalate specified in JIS K 8809 in water and dilute to 1 L.

- g) **Dimethyl-sulfonazo III indicator** The indicator shall be prepared by dissolving 0.1 g of dimethyl-sulfonazo III (2,7-bis [(2-sulfo-4-methylphenyl) azo]-1,8-dihydroxynaphthalene-3,6-disulfonic acid sodium salt) in 100 ml of water. In the case of preservation, it shall be stored in a brown bottle and, when the colour change at the end point of the titration becomes unclear, it shall be newly prepared.
- h) **Standard potassium sulfate solution (0.002 5 mol/L)** Weigh out about 0.44 g of potassium sulfate specified in **JIS K 8962** which has been dried at 105 °C to 110 °C for 3 h or over, to the nearest 0.1 mg, transfer it to a volumetric flask (1 000 ml) and add water up to the marked line.

Obtain the mole concentration of the standard potassium sulfate solution to three significant figures by the following formula.

$$C = \frac{K}{174.27}$$

where, C : mole concentration of standard potassium sulfate solution (mol/L)

K : mass of weighed potassium sulfate (g)

174.27 : mass of potassium sulfate molecule

NOTE : One ml of the standard potassium sulfate solution (0.002 5 mol/L) is equivalent to 0.080 15 mg (80.15 µg) of sulfur.

- i) **Standard barium perchlorate solution (0.002 5 mol/L)** Weigh out about 0.98 g of barium perchlorate (trihydrate) specified in **JIS K 9551**, transfer it to a volumetric flask, add a small amount of water and four or five drops of perchloric acid (10 %) specified in **JIS K 8223**, and make 1 L by adding water. The standardization of the solution shall be carried out as follows.

- 1) Take 10 ml of standard potassium sulfate solution (0.002 5 mol/L) with a volumetric pipette, transfer it to a beaker (500 ml), and then add 10 ml of water to the solution.
- 2) Add about 50 ml of acetone and 5 ml of potassium hydrogen phthalate buffer solution (20 g/L), drip four or five drops of dimethyl-sulfonazo III indicator and titrate with barium perchlorate solution while agitating with a magnetic stirrer. When the colour of the solution turns from purple to greenish blue and remains without changing colour for 1 min or over, take it as the end point¹⁾.

Note ¹⁾ The end point is easily observed when the titration is carried out using a white plate as background.

- 3) In the case of the blank test, the end point shall be obtained by taking 20 ml of water and titrating according to the procedure of 2).
- 4) Obtain the mole concentration of the standard barium perchlorate solution to three significant figures by the following formula.

$$M = \frac{10 \times C}{A - B}$$

where, M : mole concentration of the standard barium perchlorate solution (mol/L)

A : titre of barium perchlorate solution (ml)

B : titre of barium perchlorate solution in blank test (ml)

C : mole concentration of the standard potassium sulfate solution (mol/L)

- j) **Standard barium perchlorate solution (0.005 mol/L)** Weigh out about 1.95 g of barium perchlorate (trihydrate), and prepare the solution in the same manner as h) and standardize it.

6.5.4 Procedure of test

The procedure of the test shall be as follows.

- a) **Preparation of vessel for sulfur content test** The vessel for sulfur content test shall be washed and dried as follows.

Detach the valves, wash the inner parts of the vessel and valves with volatile hydrocarbon such as toluene containing no sulfur at first, then with acetone, and dry with clean compressed air.

Leave it upside-down for about 10 min, and then dry by blowing in clean compressed air.

- b) **Weighing out of sample** Introduce the sample from the sample vessel taken in 6.2 into the vessel for sulfur content test by means of the intake tube for sample, and weigh to the nearest 0.1 g.

NOTE : The mass of sample to be weighed should conform to table 4.

Table 4 Mass of sample to be weighed

Sulfur content mass fraction %	Mass of sample to be weighed g
0.000 1 to under 0.001 0	50 to 30
0.001 0 to under 0.003 0	Under 30 to 20
0.003 0 to under 0.030 0	Under 20 to 5

- c) **Preparation of combustion apparatus** Continuous caution shall be exercised to maintain safety, and protective articles such as a face shelter, protecting glasses, and leather gloves are indispensable, since this test is carried out by means of high temperature combustion in oxyhydrogen flame. Especially it is necessary to previously establish the measures and procedure, such as stoppage of the combustion of samples, pulling out the burner and shutting off the hydrogen reducing valve in such emergencies as cuts in water supply, power failure and stoppage of vacuum pump. Since the operational methods differ to some extent depending on the type of apparatus, the procedure of operation, the adjustment of the flow rate and pressure of oxygen and hydrogen and pressure reduction in the system shall be in accordance with the operation manuals designated for each device.

- 1) Confirm that there is no leakage in the connecting parts of gas pipings and that cleanness pervades the internal surfaces of glass parts in the combustion apparatus.
- 2) Confirm that the amounts of oxygen and hydrogen in cylinders are sufficient for the combustion operation, and make adjustment by means of each pressure regulator so that gases to be sent to the apparatus have the required pressures described in the operation manuals.
- 3) As shown in figure 9, connect one end of the connecting tube of stainless steel fitted with auxiliary control valve (if necessary, immerse it in a water bath kept at 60 °C to 80 °C) with the sample outlet valve of the vessel for sulfur content test, and the other end with the sample port of the burner by the use of a small tube of nylon or silicone rubber.
- 4) Start the vacuum pump, insert the burner in the combustion tube, holding it by hand, open the primary and secondary oxygen reducing valves, and adjust them to the respective required flow rate while watching flowmeters. Then open the pressure reducing valve, and regulate the system to the required pressure.
- 5) Let the cooling water flow throughout the cooling system.
- 6) Detach the trap for absorbing tube, pour 50 ml of hydrogen peroxide solution (3 %) into the absorbing tube, and return the trap for absorbing tube to the original position.
- 7) Take out the burner from the combustion tube, and keep it on the burner holder.

d) **Combustion** The combustion of the sample shall be as follows.

- 1) Open the hydrogen reducing valve, regulate it to the required flow rate by observation of the flowmeter, and expel the air from the piping to the burner.
- 2) Keeping the burner on the holder, ignite and by watching the flame, confirm that no backfire is generated.

Then insert the burner into the combustion tube rapidly taking care not to let the flame of the burner contact with the ground part of the combustion tube.

NOTE : While a backfire phenomenon can be observed visually, it can also be identified by touching with fingers as the grounding part of the burner that has become hot. In the case of a backfire phenomenon, a slight raise of the hydrogen pressure is recommended.

- 3) After confirming that the system is kept under the required pressure, open the sample outlet valve of the vessel for sulfur content test, and then burn the sample by opening gradually the auxiliary control valve. In this case, by adjusting properly the flame length with the auxiliary control valve or the pressure reducing valve, ward off incomplete combustion.

When the cooling water boils in the combustion tube, a combustion tube may burst due to vibrations. Therefore, it is necessary to increase the amount of cooling water or decrease the velocity of combustion.

- 4) Continue the combustion until the flame of the sample goes out after sucking all the sample in the vessel for sulfur content test.

- e) **Stoppage** The stoppage of apparatus shall be as follows.
- 1) When the combustion of the sample terminates, regulate the pressure in the system to the predetermined pressure by closing the auxiliary control valve, close the hydrogen reducing valve, detach the burner from the combustion tube, and then close reducing valves in the order of primary oxygen and secondary oxygen.
 - 2) Adjust the pressure in the system to the atmospheric pressure by closing the pressure reducing valves, and stop the vacuum pump and cooling water.
- f) **Titration of absorbent solution** The titration of absorbent solution shall be as follows.
- 1) Wash the trap for absorbing tube, the combustion tube and the absorbing tube with water, and wash out the trapped substance into the drain trap together with the absorbent solution.
 - 2) Transfer the content of the drain trap and its washings to a beaker (500 ml), heat to concentrate to 20 ml to 50 ml exercising caution in contamination from outside, and then cool to room temperature.
 - 3) Add 5 ml of potassium hydrogen phthalate buffer solution (20 g/L) and acetone of about double amount of the whole solution in the beaker, and further drip four to five drops of dimethyl-sulfonazo III indicator.
 - 4) Depending on the sulfur content in the sample, select the standard barium perchlorate solution whose concentration is shown in table 5, and titrate while agitating with a magnetic stirrer. When the colour of the solution turns from purple to greenish blue and remains without changing colour for 1 min or over, take it as the end point.

Table 5 Standard barium perchlorate solution for use in titration

Sulfur content in sample mass fraction %	Concentration of standard barium perchlorate solution mol/L
0.000 1 to under 0.003 0	0.002 5
0.003 0 to under 0.030 0	0.005 0

- g) **Blank test** The blank test shall be carried out by the same operations and for the same combustion time as the sample using only oxygen and hydrogen without the sample.

6.5.5 Calculation and result

The sulfur content shall be calculated to five places of decimals by the following formula, and rounded off to four places of decimals in accordance with **JIS Z 8401**.

$$S = \frac{32.06 \times M \times (A - B)}{W} \times 10^{-1}$$

where, S: sulfur content in sample (mass fraction %)
W: mass of weighed sample (g)

M : mole concentration of standard barium perchlorate solution (mol/L)

A : titre of standard barium perchlorate solution for absorbent solution (ml)

B : titre of standard barium perchlorate solution for blank test (ml)

32.06 : atomic weight of sulfur

6.5.6 Precision

The precision on 0.000 5 mass fraction % to 0.030 0 mass fraction % sulfur content shall be specified as follows. The tolerance (probability 0.95) on the test results obtained by this test method shall be as follows.

When the test results exceed the tolerance, they shall be treated according to the specification of **JIS Z 8402-6**.

- a) **Repeatability** When a person has tested twice continuously within a short time the same sample with the same tester in the same laboratory, the tolerances on difference in test results shall be as shown in figure 10.
- b) **Reproducibility** When two persons have tested once the same sample respectively with the different testers in different laboratories, the tolerances on difference in test results shall be as shown in figure 10.

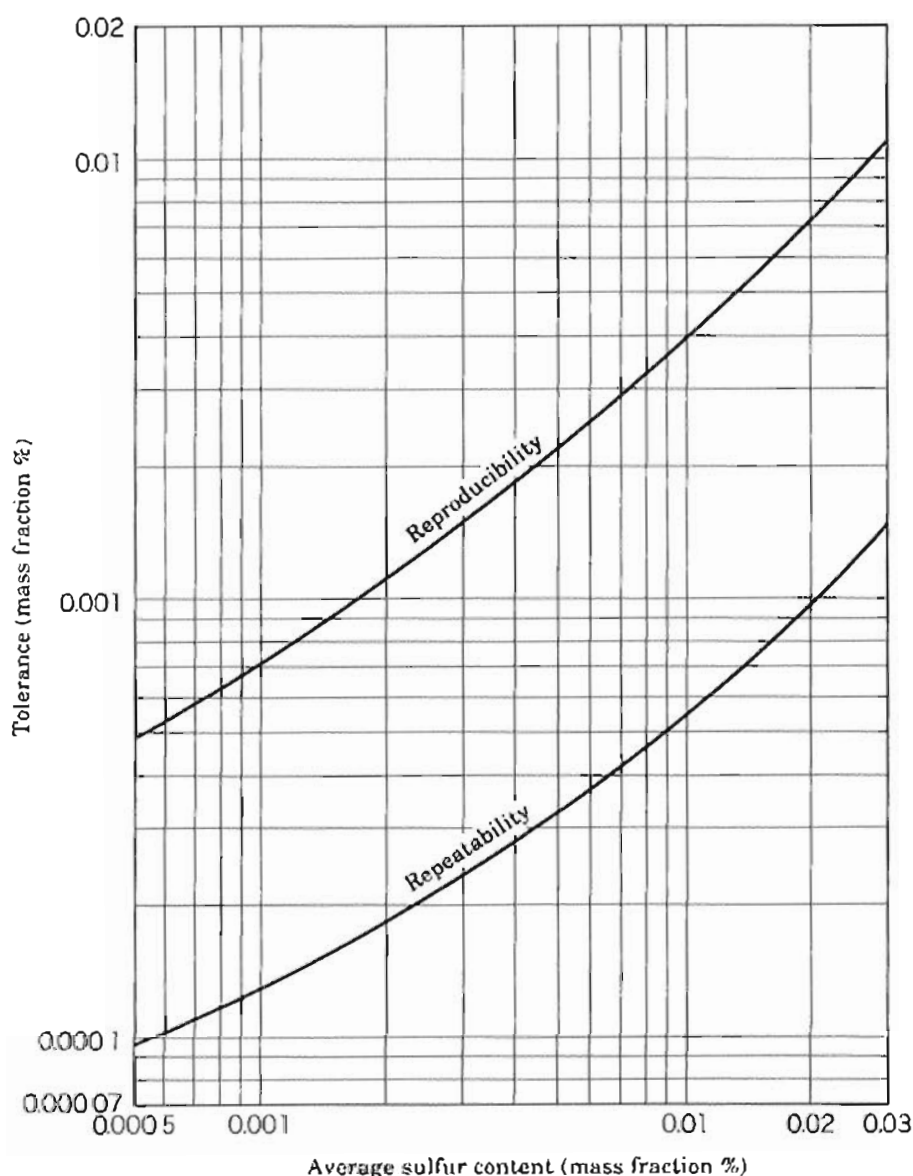


Figure 10 Precision in sulfur content test

6.6 Test method for sulfur content (oxidative microcoulometry method)

6.6.1 Principle

Introduce a sample into a heated combustion tube, and burn in an oxygen and inert gas current. Absorb sulfur dioxide generated by combustion in electrolyte, perform coulometric titration, and obtain sulfur content from the quantity of electricity consumed at that time. This method shall apply to the sample of 0.000 1 mass fraction % or over in sulfur content.

Correct the sulfur content in the sample by the corrective coefficient which has been preliminarily obtained by using standard sulfur solution.

6.6.2 Sulfur content tester

The sulfur content tester shall be composed of a) to f) (refer to figure 11).

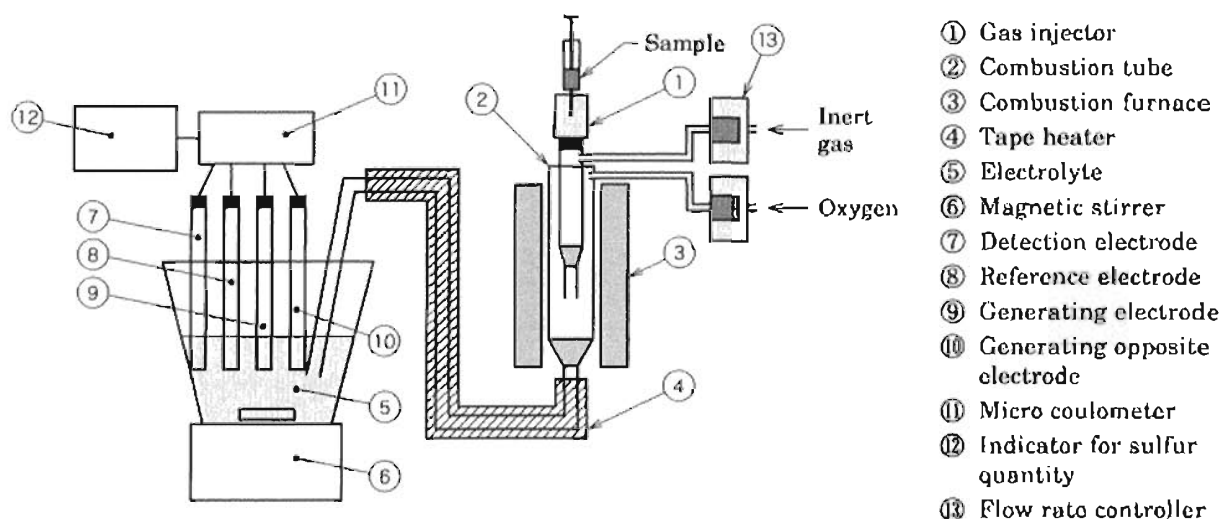


Figure 11 Tester for oxidative microcoulometry method (example of gas injector)

- a) **Combustion furnace** The inlet part and outlet part of the combustion tube of which can be independently heated by regulation.
- b) **Combustion tube** That can burn a sample in an oxygen and inert gas current, and it shall be made of quartz.
- c) **Titration cell** An electrolyte bath made of glass with a magnetic stirrer having a detection electrode, a reference electrode and a pair of generating electrodes built-in, the detection electrode and the reference electrode of which can detect variation in the concentration of triiodide ion generated by absorption of sulfur dioxide and the generating electrodes of which can generate triiodide ion equivalent to this variation quantity in concentration.
- d) **Micro coulometer** That compares continuously the potential difference preliminarily provided between the detection electrode and the reference electrode with the potential difference between both electrodes during titration and when there is some difference, can supply a current necessary for compensating to the generating electrodes.
- e) **Indicator for sulfur quantity** That can indicate or record the quantity of electricity supplied to the generating electrodes by converting that to the quantity of sulfur. The indicator which can record the quantity as the integrated peak area value will suffice.
- f) **Flow rate controller** That can flow defined amount of oxygen and inert gas.

6.6.3 Apparatus

- a) **Microsyringe** Of 10 μ l to 100 μ l capacity and that can inject liquid sample.
- b) **Gastight syringe** Of 5 ml to 10 ml capacity that can inject gas sample.

6.6.4 Reagents

The reagents for the oxidative microcoulometry method shall be as follows.

- a) **Oxygen** As specified in **JIS K 1101**.
- b) **Inert gas** Argon or helium of 99.99 vol % or over in purity.
- c) **Water** A2 or A3 in classification and quality specified in clause 4 of **JIS K 0557** or that equivalent to or higher grade.
- d) **Electrolyte** Electrolyte prepared by dissolving a specified amount of potassium iodide specified in **JIS K 8913**, sodium azide specified in **JIS K 9501**, and acetic acid specified in **JIS K 8355** in water and diluting total quantity with water to 1 000 ml.

The quantity of each reagent varies according to the tester to be used. An example thereof is given in table 6.

Table 6 Quantity of each reagent in electrolyte (example)

Reagent name		Vertical tester	Horizontal tester
Potassium iodide	g	5.0	0.5
Sodium azide	g	0.5	0.6
Acetic acid	ml	6	5

- e) **Solvent** Solvents specified in the following 1) or 2) shall be used.
 - 1) **Hexane** As specified in **JIS K 8848**.
 - 2) **Toluene** As specified in **JIS K 8680**.
- f) **Sulfur compound** The stable material with sulfur contained in which the value of sulfur content is not changed for long term shall be used. Concretely, that which is not less than 99 % in purity and contains the isomer of compound indicated in the following 1) to 2) may be used.
 - 1) **Dibutyl sulfide** $[(CH_3(CH_2)_3)_2S]$ Molecular weight: 146.29, sulfur content: mass fraction 21.915 %
 - 2) **Dibutyl disulfide** $[(CH_3(CH_2)_3)_2S_2]$ Molecular weight: 178.36, sulfur content: mass fraction 35.950 %
- g) **Preparation of standard sulfur solution**
 - 1) **Standard sulfur solution (2 500 µg/ml)**
 - 1.1) **Preparation of standard sulfur solution using dibutyl sulfide**

Weigh out 1.14 g of dibutyl sulfide into a volumetric flask (100 ml) to the nearest 0.1 mg, dissolve by adding solvent, and prepare by further adding solvent to the mark.

Obtain the sulfur concentration of this solution from the following formula.

$$A = \frac{32.06 \times W}{146.29} \times 10^4$$

where, A : concentration of sulfur (µg/ml) of standard sulfur solution (2 500 µg/ml)

W : mass of weighed dibutyl sulfide (g)

32.06 : atomic weight of sulfur

146.29 : mass of dibutyl sulfide molecule

1.2) Preparation of standard sulfur solution using dibutyl disulfide

Weigh out 0.70 g of dibutyl disulfide into a volumetric flask (100 ml) to the nearest 0.1 mg, dissolve by adding solvent, and prepare by further adding solvent to the mark.

Obtain the sulfur concentration of this solution from the following formula.

$$A = \frac{64.12 \times W}{178.36} \times 10^4$$

where, A : concentration of sulfur ($\mu\text{g/ml}$) of standard sulfur solution (2 500 $\mu\text{g/ml}$)

W : mass of weighed dibutyl sulfide (g)

64.12 : atomic weight of sulfur (32.06) $\times$ 2

178.36 : mass of dibutyl disulfide molecule

2) **Preparation of standard sulfur solution (250 $\mu\text{g/ml}$)** Weigh out 25 ml of standard sulfur solution (2 500 $\mu\text{g/ml}$) into a volumetric flask (250 ml) by using a volumetric pipette, and prepare by adding solvent to the mark.

3) **Preparation of standard sulfur solution (20 $\mu\text{g/ml}$)** Weigh out 2 ml of standard sulfur solution (250 $\mu\text{g/ml}$) into a volumetric flask (25 ml) by using a volumetric pipette, and prepare by adding solvent to the mark.

Moreover, the sulfur standard solution shall be preserved hermetically in refrigerator and the period of use should be up to about three months for the sulfur standard solution (2 500 $\mu\text{g/ml}$) and up to about one month for the sulfur standard solution (250 $\mu\text{g/ml}$) and (20 $\mu\text{g/ml}$).

6.6.5 Preparation of tester

The preparation of the tester for the oxidative microcoulometry method shall be as follows.

- After washing the inside of a titration cell with electrolyte, put electrolyte again to an extent that each electrode is sufficiently immersed.
- Connect respective terminals of a detection electrode, a reference electrode, and generating electrodes to the circuit of a micro coulometer.

In the case of a vertical tester, set end point electric potential at this stage.

- Connect the intake tube for gas of the titration cell to the end of a combustion tube outlet part, electrify a tape heater, and keep the intake tube for gas of the titration cell at 100 °C or higher.
- Set the flow rate of oxygen and inert gas, the temperature of a combustion furnace, the micro coulometer, etc. as measuring conditions. An example of measuring conditions is given in table 7.

Table 7 Measuring conditions (example)

Items			Condition for tester
Oxygen flow rate	ml/min		200 to 350
Inert gas flow rate	ml/min		80 to 200
Combustion furnace temperature	Inlet part	°C	800 to 1 050
	Outlet part	°C	800 to 1 050
End point electric potential	mV		250 to 270

6.6.6 Measurement of corrective coefficient

The corrective coefficient of oxidative microcoulometry method shall be measured in accordance with a) to d).

- a) Select a standard sulfur solution corresponding to the sulfur content approximate value of a sample from table 8, and measure the quantity given in table 8 into a microsyringe.

Table 8 Classification and measuring off quantity of standard sulfur solution to be measured

Sulfur content approximate value of sample mass fraction %	Classification	Quantity of solution to be measured μl
0.000 1 or over to and excl. 0.010 0	Standard sulfur solution (20 μg/ml)	10 to 100
0.010 0 or over up to and incl. 0.030 0	Standard sulfur solution (250 μg/ml)	10

- b) Introduce the standard sulfur solution taken in the microsyringe into a combustion tube by the following method.
- Gas injector injection method** Insert the needle point of microsyringe to the injection opening of gas injector, inject the sulfur standard solution quickly and read the injection quantity²⁾.
 - Direct injection method** Insert the needle point of the microsyringe into a combustion tube inlet part through a sample injection opening, inject standard sulfur solution at 1.0 μl/s to 1.2 μl/s, and read the injection quantity.

In order to inject the sample at constant speed, a dispenser or an automatic injector should preferably be used.

Note ²⁾ Suck the air into the microsyringe before and after injection of the standard solution for corrective coefficient, obtain the liquid quantity in microsyringe, and obtain the injection quantity from said difference.

- c) Read the value indicated on a sulfur quantity indicator, calculate the corrective coefficient from the following formula, and round off to two places of decimals in accordance with **JIS Z 8401**.

$$f = \frac{b}{V \times N}$$

where, f : corrective coefficient
 b : sulfur quantity (ng)
 V : injection quantity of standard sulfur solution (μl)
 N : concentration of standard sulfur solution ($\mu\text{g/ml}$)

- d) Repetitively measure the corrective coefficients, average the successive three time values when the corrective coefficients are stabilized at a certain value within a range of 0.65 to 0.95, take it as the average corrective coefficient (F), and use that for calculation of the sulfur content of the sample. Where the corrective coefficient does not fall within this range, prepare the standard sulfur solution again, and measure again. Where the result of a remeasurement does not fall within this range, check the apparatus and the operation method.

For confirmation of the average corrective coefficient in measurement of the sample, perform confirmation once each series of tests and perform once each 4 h where a series of tests exceed 4 h. Where a difference from the average corrective coefficient exceeds 0.05, obtain the average corrective coefficient again.

6.6.7 Procedure for weighing out of sample

The procedure for weighing out of the sample shall be as follows.

- a) **Preparation** For the sample vessel which is prepared in 6.2, connect the needle valve to the sample vessel outlet (liquid side) as given in figure 12, and connect the sample vessel to the intake opening of a gas collecting bag made of plastic film of fluorine resin or the like via the three-way cock made of tetrafluoroethylene resin by using a stainless steel tube, an tetrafluoroethylene resin tube, etc.

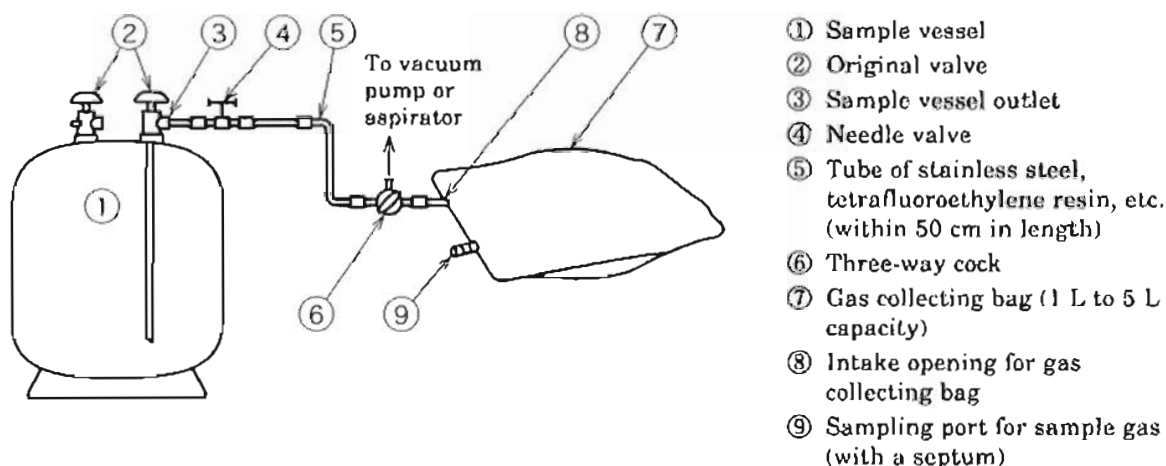


Figure 12 Connection of sample vessel to gas collecting bag (example)

- b) **Washing with sample** Gradually open the original valve of a sample vessel, and regulate appropriately so that the bag does not swell quickly. When the swelling of the gas collecting bag becomes approximately 50 % to 70 % of the tolerable volume, close the original valve, and exhaust the sample gas 3 min later. Perform this operation twice.

- c) **Weighing out of sample** Take a gasified sample into a gas collecting bag in the same way as in b). Wash a gastight syringe two or three times with the sample gas through the septum of a sampling opening 3 min later after closing the original valve. Thereafter, weigh out the sample gas as shown in table 9.

Moreover, for preventing the loss of sulfur content and preventing the contamination due to exposure or the contact with the sample container, take the sample in liquid state and measure it quickly.

Table 9 Mass of sample to be weighed

Sulfur content approximate value of sample mass fraction %		Mass of sample to be weighed ^{a)} ml
0.000 1 min.	Under 0.010 0	10
0.010 0 min.	0.030 0 max.	5
Note ^{a)} For the sample of over 0.030 0 mass fraction % in sulfur content, regulate the mass of sample to be weighed according to the mass fraction % of sulfur content of the sample.		

6.6.8 Measurement of sample

Inject the sample weighed out in a gastight syringe from the gas injector of apparatus or sample injection opening and read the value indicated in a data processor. However, injection speed shall be 0.1 ml/s to 0.5 ml/s.

Moreover, for injecting the sample at a constant speed, an automatic injector should be used.

Observe the combustion tube during combustion, and confirm the complete combustion of the sample. When soot is observed, carry out the retest according to the following procedures.

- After the whole parts are cleaned according to the instruction manual of manufacturer, carry out the retest according to 6.6.6.
- Measure by reducing the sample injection quantity.

6.6.9 Calculation and result

The sulfur content shall be calculate to five places of decimals according to the following formula and rounded to four places of decimals according to JIS Z 8401.

$$S = \frac{22.4 \times B}{V \times \frac{273.15}{273.15 + t} M \times F \times 10^4}$$

where, S : sulfur content (mass fraction %)
 B : sulfur quantity (ng)
 V : sample injection quantity (ml)
 t : sample temperature (°C)
 M : mass per mole of a sample (g/mol)
 F : average corrective coefficient

The temperature of the sample shall be room temperature.

Calculate the mass per mole of the sample from the following formula by using the content (mol %) of each analytical component of the sample obtained by the method of analyzing composition of 6.10 and the mass per mole of each component as given in table 10.

$$M = \sum \left(M_i \times \frac{C_i}{100} \right)$$

where, M : mass per mole of a sample (g/mol)

M_i : mass per mole of a component i as given in table 10 (g/mol)

C_i : content of a component i in accordance with 6.10 (mol %)

Table 10 Mass per mole of each hydrocarbon

Unit: g/mol

Component i	Mass per mole
Ethane + ethylene ^{a)}	30.069
Propane	44.096
<i>n</i> -Butane	58.123
iso-Butane	58.123
<i>n</i> -Pentane	72.150
iso-Pentane	72.150
Propylene	42.080
1-Butylene	56.107
cis-2-Butylene	56.107
trans-2-Butylene	56.107
iso-Butylene	56.107
1,3-Butadiene	54.091
Note ^{a)} Since ethane and ethylene cannot be separated by the method of analyzing composition in 6.10, the mass per mole of ethane is adopted as the component of ethane + ethylene.	

6.6.10 Precision

The precision for 0.000 1 mass fraction % to 0.030 0 mass fraction % sulfur content shall be specified as follows. The tolerance (probability 0.95) for the test results obtained by this test method shall be as follows.

When the test results exceed the tolerance, they shall be treated according to the specification of JIS Z 8402-6.

- a) **Repeatability** When a person has tested twice continuously within a short time the same sample with the same tester in the same laboratory, the tolerances on difference in test results shall be shown in table 11 and figure 13.

- b) **Reproducibility** When two persons have tested once the same sample respectively with the different testers in different laboratories, the tolerances on difference in test results shall be shown in table 11 and figure 13.

Table 11 Precision

Unit: Mass fraction %	
Precision	Calculating formula
Repeatability	$y = 0.059\ 8x - (0.393\ 9 \times 10^{-4})$
Reproducibility	$y = 0.179\ 4x + (4.568\ 5 \times 10^{-4})$

where, x : average value of sulfur content (mass fraction %)
 y : tolerance (mass fraction %)

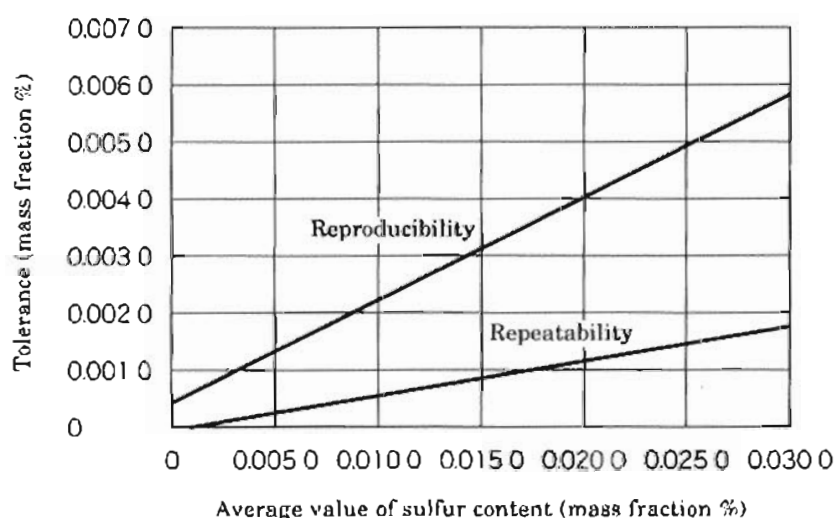


Figure 13 Precision in sulfur content test

6.7 Test method for sulfur content (ultraviolet fluorescence method)

6.7.1 Principle

The sample is introduced in a heated combustion tube (750 °C to 1 100 °C) and decomposed to oxidize in the atmosphere of oxygen, and the sulfur compound in sample is oxidized to sulfur dioxide. Next, after the moisture in the combustion generation gas containing this sulfur dioxide is removed, ultraviolet light is irradiated. The sulfur dioxide absorbs energy from the ultraviolet light and is converted to the sulfur dioxide under excitation state. The fluorescence emitted, when the excited sulfur dioxide returns to the sulfur dioxide under ground state, is detected by a phototube and the sulfur content is obtained from the quantity of fluorescence. For sulfur content, this method is applied to the sample of 0.000 1 % to 0.030 0 % in mass fraction.

6.7.2 Sulfur content tester

The tester shall be composed of a) to e) (refer to figure 14).

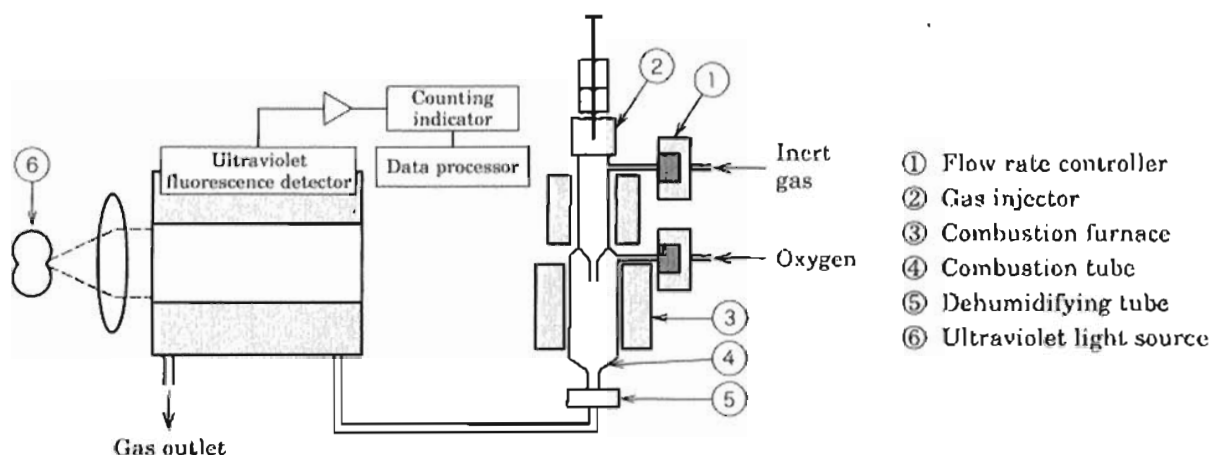


Figure 14 Tester for ultraviolet fluorescence method (An example of gas injector)

- a) **Combustion furnace** That which is capable of being installed horizontally (horizontal type) or vertically (vertical type) and of being maintained at the sufficient high temperature to oxidize all of the sulfur and sulfur compound to sulfur dioxide (SO_2) by decomposing the sample thermally.
- b) **Combustion tube** That can burn a sample completely in an oxygen and inert gas current, made of quartz.
- c) **Flow rate controller** That can flow defined amount of oxygen and inert gas.
- d) **Dehumidifying tube** That which is capable of removing the moisture content in combustion generation gas and of preventing the SO_2 dissolution loss³⁾.

Note ³⁾ An electric dehumidifier may be used.

- e) **Ultraviolet fluorescence detector** That is composed of an ultraviolet light source and a fluorescence detector and capable of measuring the quantity of fluorescence emitted by irradiating ultraviolet light to sulfur dioxide quantitatively by using a photomultiplier.

6.7.3 Apparatus

The apparatus shall be as follows.

- a) **Microsyringe** Of 5 μl to 25 μl capacity and that can inject liquid sample.
- b) **Gastight syringe** Of 5 ml to 10 ml capacity that can inject gas sample.
- c) **Balance** Capable of weighing to an accuracy of 0.1 mg.
- d) **Volumetric flask** As specified in JIS R 3505.

6.7.4 Reagents

The reagents for the ultraviolet fluorescence method shall be as follows.

- a) **Oxygen** As specified in JIS K 1101.
- b) **Inert gas** Argon or helium of 99.99 vol % or over in purity.
- c) **Solvent** Solvents specified in the following 1) or 2) shall be used.