

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing Methods for
Aviation Fuels**

JIS K 2276—1989

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

JAPANESE INDUSTRIAL STANDARD

J I S

Testing Methods for Aviation Fuels

K 2276-1989

1. Scope

This Japanese Industrial Standard specifies testing methods for aviation gasolines and aviation turbine fuels.

Remark: The units and numerical values given in { } in this Standard are based on the traditional units and are currently the criteria in force.

2. General Matters

General matters shall be as follows:

- (1) Common matters to chemical analysis shall be in accordance with JIS K 0050.
- (2) Common matters to potentiometric titration shall be in accordance with JIS K 0113.
- (3) Common matters to absorptiometric analysis shall be in accordance with JIS K 0115.
- (4) Rounding off of numerical values shall be in accordance with JIS Z 8401.
- (5) Method of sampling shall be in accordance with JIS K 2251.

3. Definitions

For the purposes of this Standard following definitions apply:

- (1) oxidation stability (latent residual substance method) The oxidation stability is used as the aim for gum generation tendency during storage of aviation fuels and, expressed as follows:
 - (a) precipitate amount The amount of residual substances to be obtained by filtering washings when the sample container is washed with the sample oxidized by the specified method and gum solvent. It is expressed by number of milligrammes (mg) per 100 ml of sample.
 - (b) insoluble gum amount An amount of adhering matters in a sample container which is insoluble to gum solvent. It is expressed by number of milligrammes (mg) per 100 ml of sample.

Applicable Standards and Corresponding International Standards: See pages 161 to 163.

- (c) soluble gum amount An amount of nonvolatile residual substances in filtrate after measuring the precipitate amount. It is expressed by number of milligrammes (mg) per 100 ml of sample.
- (d) latent gum amount The total amount of soluble gum amount and insoluble gum amount. It is expressed by number of milligrammes (mg) per 100 ml of sample.
- (e) total latent residual substance amount The total amount of precipitate amount, insoluble gum amount and soluble gum amount. It is expressed by number of milligrammes (mg) per 100 ml of sample.
- (2) deposition point A temperature at which crystals of hydrogen carbide generated when the sample is cooled are extinguished when the temperature of sample is raised.
- (3) total acid number A number of milligrammes (mg) of potassium hydroxide required for neutralizing the total acidic components contained in 1 g of sample.
- (4) luminometer number A scale to indicate a temperature of flame to a specific brilliancy at green-yellow zone of visible spectral when the sample is burned under the specified conditions.

Generally, in aviation turbine fuels, as the saturated hydrogen carbide content is more much, and as the aromatic hydrogen carbide content is more little, the luminometer number becomes more large and good in combustibility.

- (5) naphthalene content The total amount of naphthalene, acenaphthalene and their alkyl derivatives.
- (6) thermal stability A condition of generated accumulated matters under the resembled conditions as in fuel system to be used for evaluation of thermal stability of aviation fuels.

Informative Reference: As the test methods, there are A method and B method. Testers used in A method and B method are called respectively Fuel Coker (ASTM CRC Fuel Coker), JFTOT (Jet Fuel Thermal Oxidation Tester). However, the values between the both methods are not always coincide with each other.

- (7) water separation index A numerical value to indicate water separation performance of aviation fuel. As this value is larger, it indicates that the water separation performance is more good. As the test methods, there are water separometer method and micro separometer method, and respective their test results shall be appended with WSIM, MSEP.

Informative Reference: WSIM and MSEP are abbreviations respectively of Water Separation Index Modified and Micro Separometer Surfactants.

- (8) explosibility A volume percentage relative to the explosion lower limit of concentration of combustible gas generated under specified conditions to be used as the aim to generate inflammable mixed gas from aviation fuels.
- (9) electrical conductivity A numerical value to be used for evaluation of difficulty for charging of static electricity of aviation fuel added with static electricity preventing agent. It is expressed by ps/m.
- (10) supercharge method octane value A scale to express antinock property of fuel of spark ignition type engine with a supercharger. It shall be expressed by the value as follows:
 - (a) in the case of supercharge method octane value not more than 100
Take the supercharge method octane value of 2, 2,4-trimethyl pentane, (hereafter referred to as the "isooctane") of high antinock property as 100, and the supercharge method octane value of heptane of low antinock property, as 0, compare antinock properties of positive standard fuel by mixing of isooctane and heptane and of sample, and express by an integer nearest to the capacity % of isooctane in the positive standard fuel indicating the same antinock property as in sample or by a performance number.
 - (b) in the case of supercharge method octane value exceeding 100
It is expressed by tetraethyl-lead amount added to isooctane or by performance number.

4. Classification of Testing Methods

Testing methods shall be classified as given in Table 1.

Table 1. Classification of Testing Methods

Testing method	Applicable oil	Clause number
Oxidation stability (latent residual substance method)	Aviation gasoline	5.
Deposition point	Aviation gasoline and aviation turbine fuel	6.
Water solubility	Aviation gasoline and aviation turbine fuel	7.
Total acid number	Aviation turbine fuel	8.
Mercaptan sulfur content (potentiometric titration)	Aviation turbine fuel	9.
Doctor test	Aviation turbine fuel	10.
Luminometer number	Aviation turbine fuel	11.
Naphthalene content (ultraviolet spectroscopy)	Aviation turbine fuel	12.
Silver-plate corrosion	Aviation turbine fuel	13.
Thermal stability (A method)	Aviation turbine fuel	14.1
Thermal stability (B method)	Aviation turbine fuel	14.2
Water separation index (water separometer method)	Aviation turbine fuel	15.
Particulate contaminants (test room filtration method)	Aviation turbine fuel	16.
Explosibility	Aviation turbine fuel	17.
Electrical conductivity	Aviation turbine fuel	18.
Supercharge method octane value	Aviation gasoline	Appendix 6

Remark: The method to test water separation index according to micro separometer is shown in Reference 1 [water separation index testing method (micro separometer method)].

5. Oxidation Stability Testing Method (Latent Residual Substance Method)

5.1 Summary of Testing Method Put a sample 100 ml into a bomb, press-in oxygen at 0.7 MPa {7 kgf/cm²} and immerse into a constant temperature tank at 100°C for 16 h. Relating to the oxidized sample, measure the precipitate amount, insoluble gum amount and soluble gum amount, and obtain the latent gum amount, separate amount and total latent residual substance amount.

5.2 Oxidation Stability Tester (Latent Residual Substance Method)
The oxidation stability tester shall be of structure as shown in Fig. 1 and, consist of (1) to (7).

- (1) Bomb A bomb of such structure as shown in Fig. 2, endurable to water pressure of 1.5 MPa {15 kgf/cm²} for 1 h, at 15 to 25°C fill with oxygen of 0.7 MPa {7 kgf/cm²}, immerse the bomb into a water bath kept at 100 ± 1°C, and the lowering of pressure after 24 h shall be within 0.014 MPa {0.14 kgf/cm²} from the highest pressure.
- (a) Bomb Body and Bomb Cap The bomb body and bomb cap shall be those shown in Fig. 3 and Fig. 4, made of stainless steel (SUS 304), and the inner surface of which is so polished that it is easily washed and prevented against corrosion.

- (b) Fastening Metal Fittings Fastening metal fittings shall be made of copper alloy as shown in Fig. 5.
- (c) O Ring The O ring specified in JIS B 2401.
- (d) Conduit A conduit of shape and dimensions shown in Fig. 4, made of material same as that of bomb cap and shall be polished thoroughly so as the inner surface to be easy for washing and to prevent corrosion. The conduit shall be provided with a metallic disc, connecting tube attaching opening and needle valve.
- (e) Core Bar The core bar shall be of shape and dimensions shown in Fig. 6 and made of material same as that of bomb cap.
- (2) Sample Container and Cover The sample container and cover shall be those specified in Fig. 94 and Fig. 95 of JIS K 2839.
- (3) Pressure Gauge The pressure gauge shall be a Bourdon tube or recording type pressure gauge capable of measuring up to 1.5 MPa {15 kgf/cm²} (gauge pressure). The length of graduation interval corresponding to 0.34 MPa {3.5 kgf/cm²} at the scale between 0.7 to 1.5 MPa {7 to 15 kgf/cm²} shall be not less than 25 mm at arc state part and subdivision graduation, not more than 0.025 MPa {0.25 kgf/cm²}.
- Further, the error shall be not more than 0.015 MPa {0.15 kgf/cm²}. The pressure gauge shall be connected with bomb directly or by means of a connecting tube.
- (4) Connecting Tube The connecting tube shall be a copper tube of shape as shown in Fig. 1 or flexible tube of a suitable material to connect the conduit with the pressure gauge. The capacity of connecting tube including the part of conduit shall be not more than 30 ml.
- (5) Thermometer The thermometer shall be a thermometer No. 6 (GOS) specified in JIS B 7410.
- (6) Constant Temperature Tank The constant temperature tank shall be of structure and dimensions as shown in Fig. 1, the volume, be not less than 18 l in the case of one piece bomb and in the case of two pieces or more bomb, be of such size that for increasement of each one piece bomb, the volume increases not less than 8 l. The cap of constant temperature tank shall be provided with such a hole and thermometer inserting opening that when the bomb is entered the metallic disc of conduit becomes just a cap, be of such structure that when the bomb is entered in the constant temperature tank, the position of cap of bomb is able to be maintained at 50 mm at least under the liquid surface, and separately, provided with an auxiliary cap to be used when the bomb is not entered. In the case of using a bath liquid of which boiling point is between 99.5 and 100.5°C, a heating device and a reflux condenser capable of boiling vigorously the bath liquid shall be provided. In the case of using a bath liquid of which boiling point is not less than 100.5°C, a temperature regulator capable of regulating the bath temperature uniformly at 100 ± 0.1°C shall be provided.

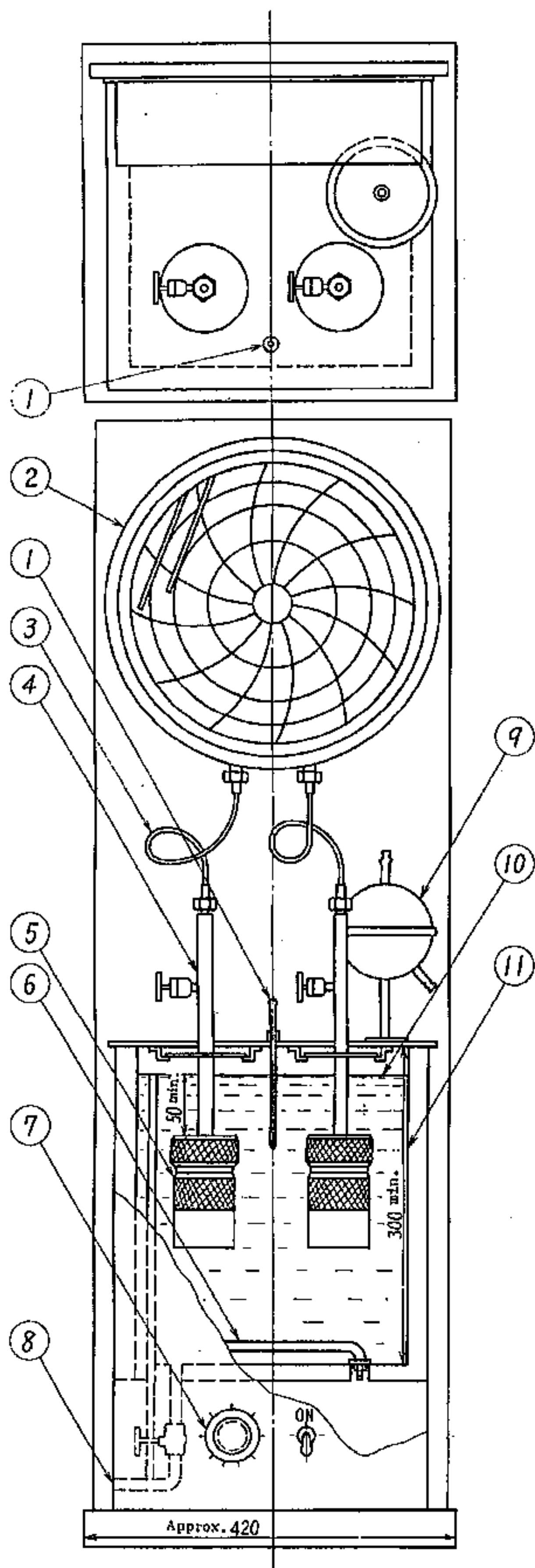
The insulation resistance of heater, when measured between the charged part and the frame stand by using a insulation resistance tester of 500 V, shall be not less than 5 M Ω . However, in the case of including the thermoelectric circuit, it may be not less than 0.5 M Ω .

- (7) Sintered-Glass Filter A crucible type sintered glass filter 1 G 4 specified in JIS R 3503 shall be used.

Fig. 1. Oxidation Stability Tester (An Example)

Unit: mm

Two Piece Unit



- ① Thermometer
- ② Recording type pressure gauge
- ③ Connecting tube
- ④ Conduit
- ⑤ Bomb
- ⑥ Electric heater
- ⑦ Temperature regulator
- ⑧ Water exhaust opening
- ⑨ Reflux condenser
- ⑩ Bath liquid
- ⑪ Constant temperature tank

Fig. 2. Bomb

Unit: mm

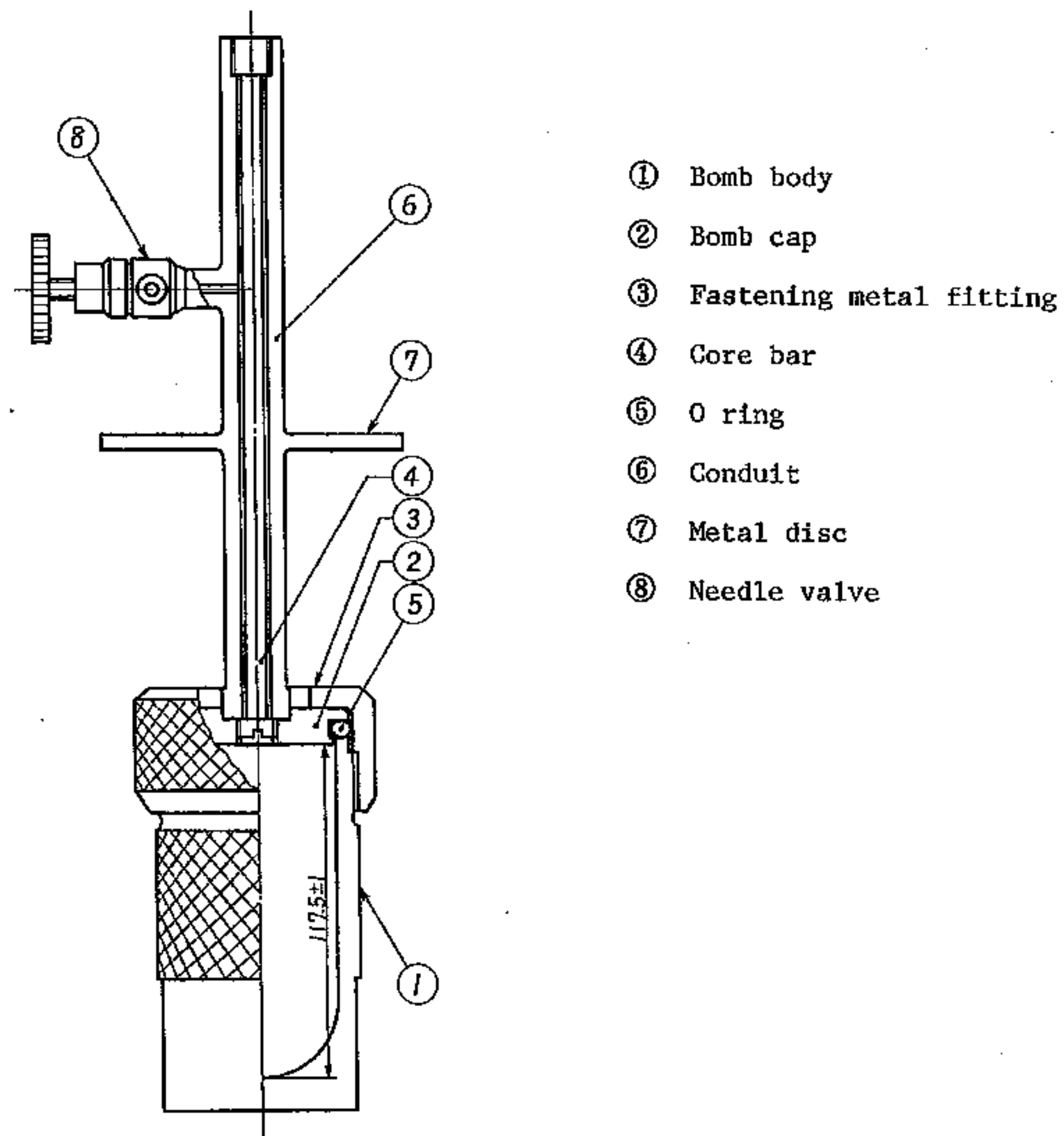
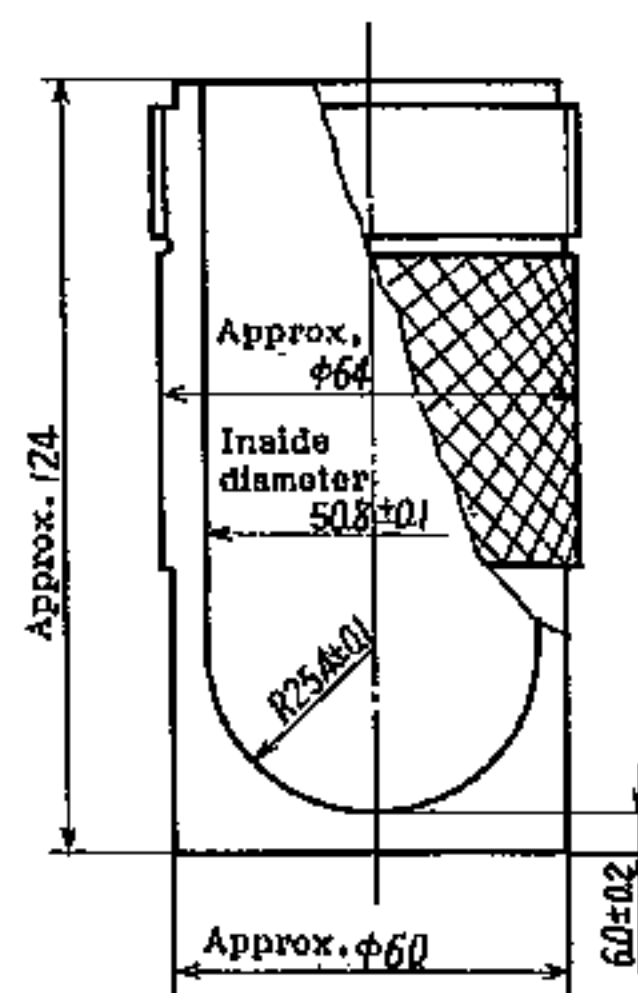


Fig. 3. Bomb Body

Unit: mm



8
K 2276-1989

Fig. 4. Cap of Bomb and Conduit

Unit: mm

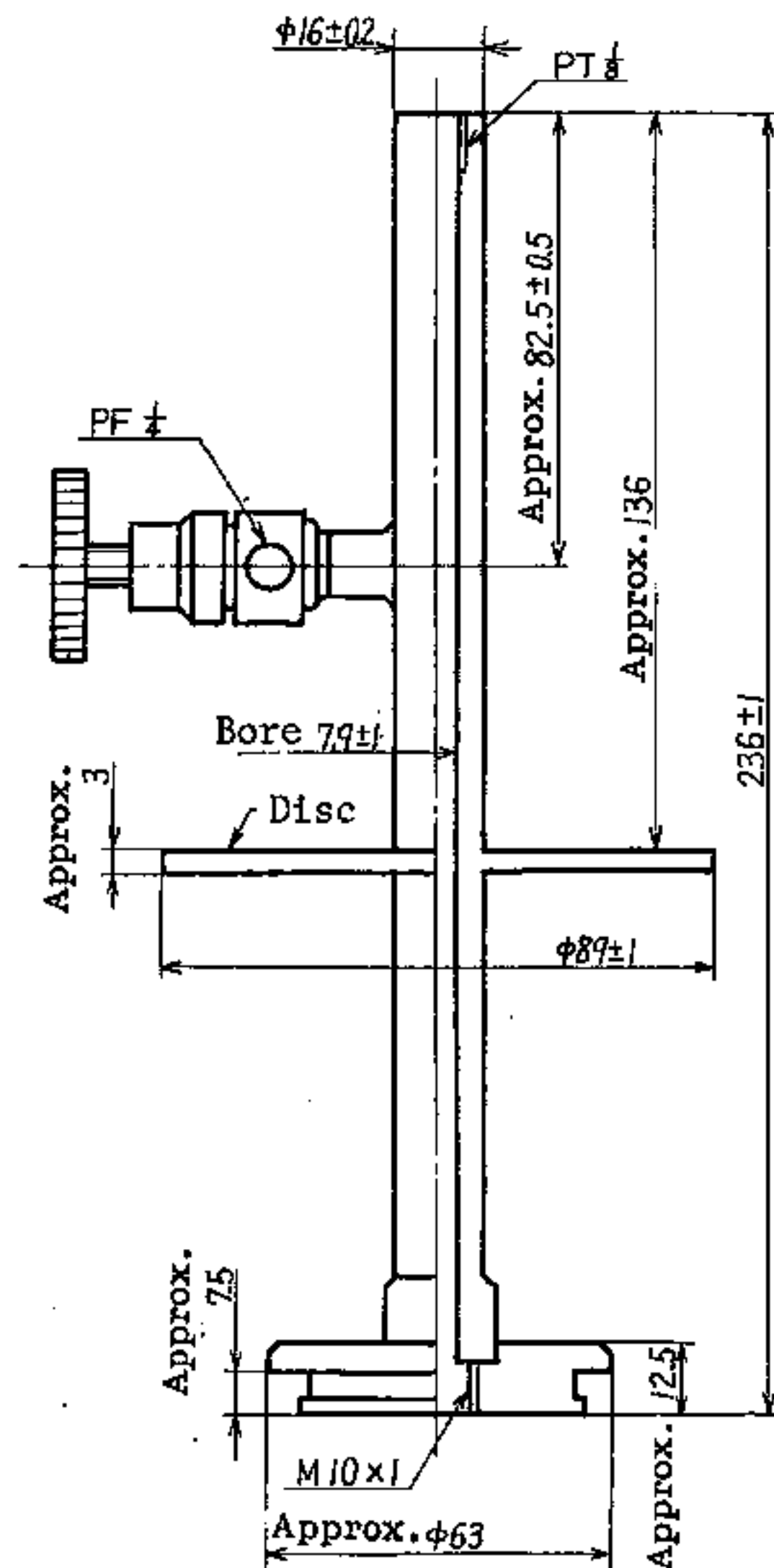


Fig. 5. Fastening Metal Fitting

Unit: mm

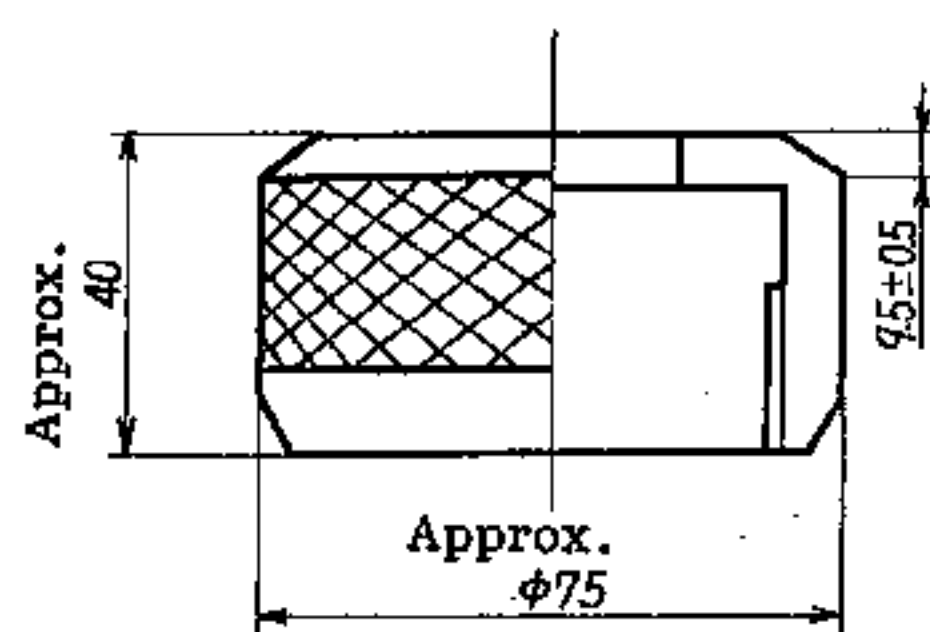
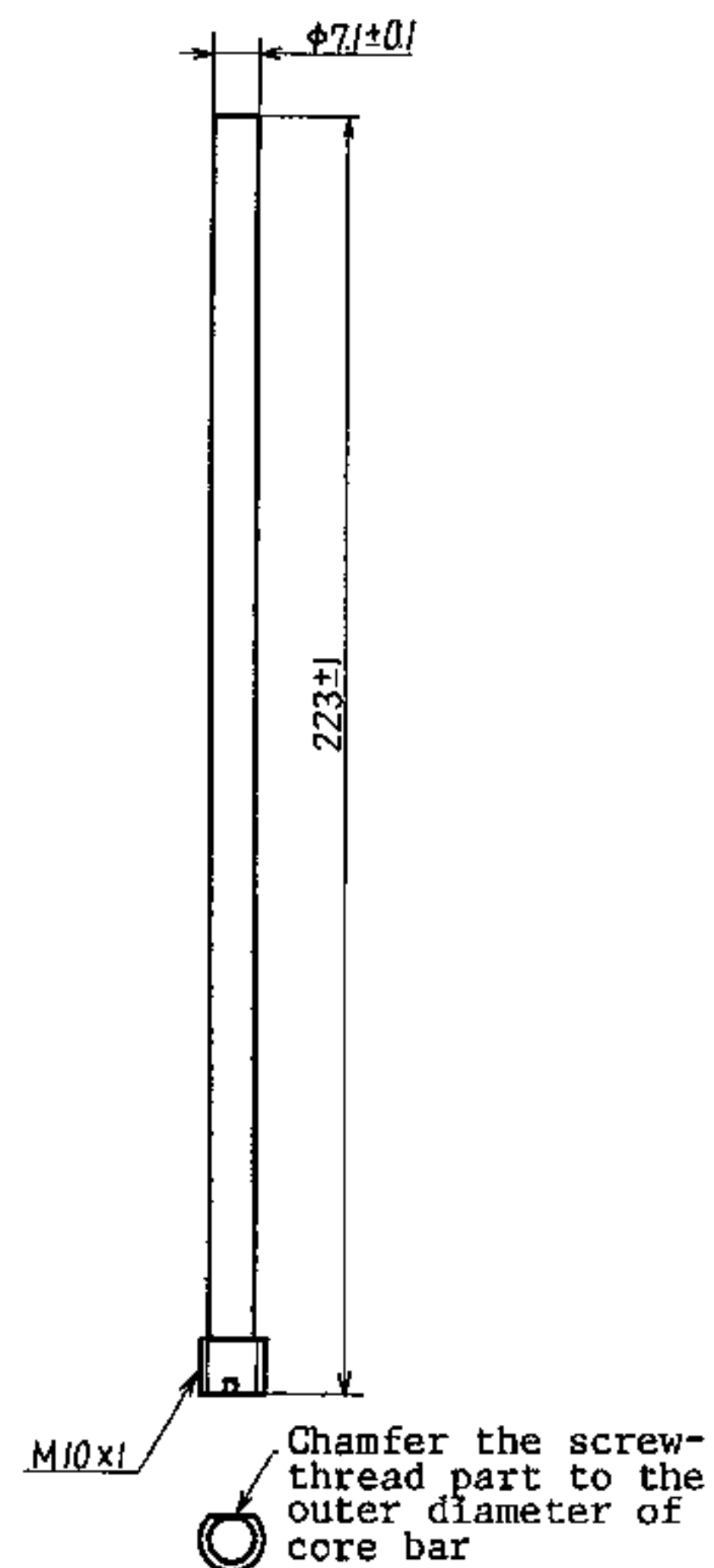


Fig. 6. Core Bar

Unit: mm



5.3 Reagents Reagents for oxidation stability testing method (latent residual substance method) shall be as follows:

- (1) Gum Solvent An equal amount mixture of toluene specified in JIS K 8680 and acetone specified in JIS K 8034.
- (2) Detergent for Glass Devices The sulfuric acid specified in JIS K 8951 saturated with potassium dichromate specified in JIS K 8517 or sodium dichromate dihydrate specified in JIS K 8518 (hereafter referred to as the "chromate mixture (¹)") or the detergent equivalent thereto.

Note (¹) The chromate mixture completed with using shall be treated to make harmless.

5.4 Sampling Method The sampling method shall be in accordance with the sampling method for oxidation stability test of aviation fuels specified in JIS K 2251.

5.5 Preparation of Test The preparation of oxidation stability testing method (latent residual substance method) shall be as follows:

- (1) After removing the adhering matters of sample container, immerse it together with its cover into the glass device detergent for at least 6 h. Then take out from the glass device detergent by means of sample-container scissors. Hereinafter, use the sample-container scissors for handling the sample container. Then, wash with city water, further wash sufficiently with water, after drying in a dryer at 100 to 150°C for at least 1 h, allow to stand to cool in a desiccator without containing desiccating agent for not less than 2 h, and weigh correctly to the nearest 0.1 mg.
- (2) Wipe the internal parts of bomb and cap by means of a clean cloth soaked-in with gum solvent at first, and then wipe by means of a clean dry cloth. When removing the gum content and residual sample from the ring state clearance between the conduit internal wall and the core bar, detach the core bar from the conduit and clean the both carefully.

Further, clean the tube connecting with the bomb at all internal and external parts. At each test, dry the bomb and connecting tubes completely before its starting.

- (3) In the case of using a constant temperature tank to regulate the bath temperature by a temperature regulator, regulate the bath temperature at $100 \pm 0.1^\circ\text{C}$, and maintain this temperature until completion of the test.
- (4) In the case of using a constant temperature tank to use by boiling the bath liquid, regulate the bath temperature at 99.5 to 100.5°C by using water or by adding a high boiling matter such as ethylene glycol to water.

5.6 Procedures of Test The test procedures of oxidation stability testing method (latent residual substance method) shall be as follows:

- (1) Make the bomb and sample at a temperature of 15 to 25°C, enter a sample container of known mass in a bomb and pour the sample of 100 ± 1 ml into it. Cover the sample container, close the cap of bomb, and enter oxygen until it becomes 0.69 to 0.71 MPa {7.0 to 7.2 kgf/cm²}. Then, open slightly the needle valve to discharge the gas gently so as not to exceed 0.34 MPa {3.5 kgf/cm²} in 1 min. Repeat this operation to expel the air entered at first. Further, enter oxygen until 0.69 to 0.71 MPa {7.0 to 7.2 kgf/cm²} and examine the leakage by viewing the pressure gauge. At this time neglect the primary pressure lowering (generally, not exceeding 0.05 MPa {0.5 kgf/cm²}) to be generated by dissolution of oxygen in the sample. Thereafter, when the pressure lowering rate does not exceed 0.015 MPa {0.15 kgf/cm²} in 10 min, consider that there is no leakage, and proceed the test as it is.
- (2) Enter the bomb containing sample and oxygen in the constant temperature tank with taking so care as not to move with shaking, and record this time as the time of test starting. Allow to stand bomb as it is in a constant temperature tank for 16 h. In case where the bath temperature at test starting is at other than 100.0°C, adjust the time of immersing into the bomb by using the correction factor shown in Table 2.

Table 2. Correction Factor of Time of Immersing Bomb

Bath temperature °C	Correction factor
99.5	1.06
99.6	1.04
99.7	1.03
99.8	1.02
99.9	1.01
100.0	1.00
100.1	0.99
100.2	0.98
100.3	0.97
100.4	0.96
100.5	0.95

Remark: For obtaining the corrected immersing time at a bath temperature, multiply 16 h by correction factor.

- (3) After oxidizing at 100°C for equivalent time of 16 h, take out the bomb from constant temperature tank, and with the needle valve closed as it is cool quickly with water. After cooling, draw out the pressure gradually so as not to exceed 0.34 MPa {3.5 kgf/cm²} in 1 min and detach the cap of bomb to take out the sample container.

- (4) In the case where even the precipitate amount is specified but precipitate is not confirmed in the oxidized sample, or where even the precipitate is confirmed but the precipitate amount is not specified in Standard, transfer the oxidized sample from the sample container to a flask, wash the internal part of sample container by gum solvent of each 10 ml twice, add this washing to the flask, mix sufficiently and measure the soluble gum amount according to (6).

In the case where the precipitate is confirmed in the oxidized sample and the precipitate amount is specified in Standard, filter the oxidized sample by a sintered-glass filter of known mass and preserve the washing. Further, wash the internal part of sample container by gum solvent of 10 ml twice, and remove the residual gum and precipitate. At this time, filter the washing at each time by means of a sintered-glass filter used at the first, add to the filtrate previously preserved, and mix sufficiently by stirring. Preserve this filtrate as for determination of soluble gum amount. After drying the sintered-glass filter in a drier at 100 to 150°C for 1 h, allow to stand to cool in a desiccator containing no desiccating agent, and measure to the nearest 0.1 mg. Record the increasement of mass as the precipitate amount (A).

- (5) Dry the sample container at 100 to 150°C for 1 h, allow to stand to cool in a desiccator containing no desiccating agent and weigh to the nearest 0.1 mg. Record the increasement of mass as the insoluble gum amount (B).
- (6) Separate the filtrate obtained in (4) into two parts (difference between the two parts is within 2 ml), relating to respective whole amount measure the not-washed existent gum according to JIS K 2261, and record the total amount as the soluble gum amount (C).

Remark: The sample shall not be exposed to strong sunlight before test and during the test.

5.7 Results Results of oxidation stability testing method (latent residual substance method) shall be as follows:

- (1) Latent Gum Amount Round off the total amount of insoluble gum amount (B) and soluble gum amount (C) to an integer in milligramme unit and take the amount per 100 ml of sample (mg/100 ml).
- (2) Precipitate Amount Round off the precipitate amount (A) to an integer in mg unit and take it as the amount per 100 ml of sample (mg/100 ml).
- (3) Total Latent Residual Substance Amount Round off the total amount of precipitate amount (A), insoluble gum amount (B) and soluble gum amount (C) to an integer in milligramme unit and take it as the amount per 100 ml of sample (mg/100 ml).

5.8 Accuracy The accuracy of oxidation stability testing method (latent residual substance method) shall be as follows:

- (1) Repeatability When the same sample is tested twice in the same testing room, by the same person by using the same tester with changing the day or time, the difference of test results shall not exceed the tolerance of Table 3.

12
K 2276-1989

- (2) Reproducibility The difference of two test results obtained by testing respectively once the same sample in different two testing rooms by different person by using other tester shall not exceed the tolerance of Table 3.

Table 3. Accuracy

Unit: mg/100 ml

Measuring item	Range	Repeating tolerance	Reproducing tolerance
Latent gum amount	Less than 5	2	3
	5 or over to and excluding 10	3	4
	10 or over to and excluding 20	4	6
Precipitate amount	Less than 2	1	1
Total latent residual substance amount	Less than 5	2	4
	5 or over to and excluding 10	3	5
	10 or over to and excluding 20	5	7

6. Deposition-Point Testing Method

6.1 Summary of Testing Method Take approximately 25 ml of sample into a sample container, and in a Dewar vessel containing refrigerant while mixing by stirring allow to cool until the crystal of hydrogen carbide deposits. When the crystal has deposited, take out the sample container from the Dewar vessel, while mixing by stirring gradually warm, and measure the temperature when the crystal of hydrogen carbide has extinguished completely.

6.2 Deposition-Point Tester The deposition tester shall be of structure shown in Fig. 7 and, consist of (1) to (5).

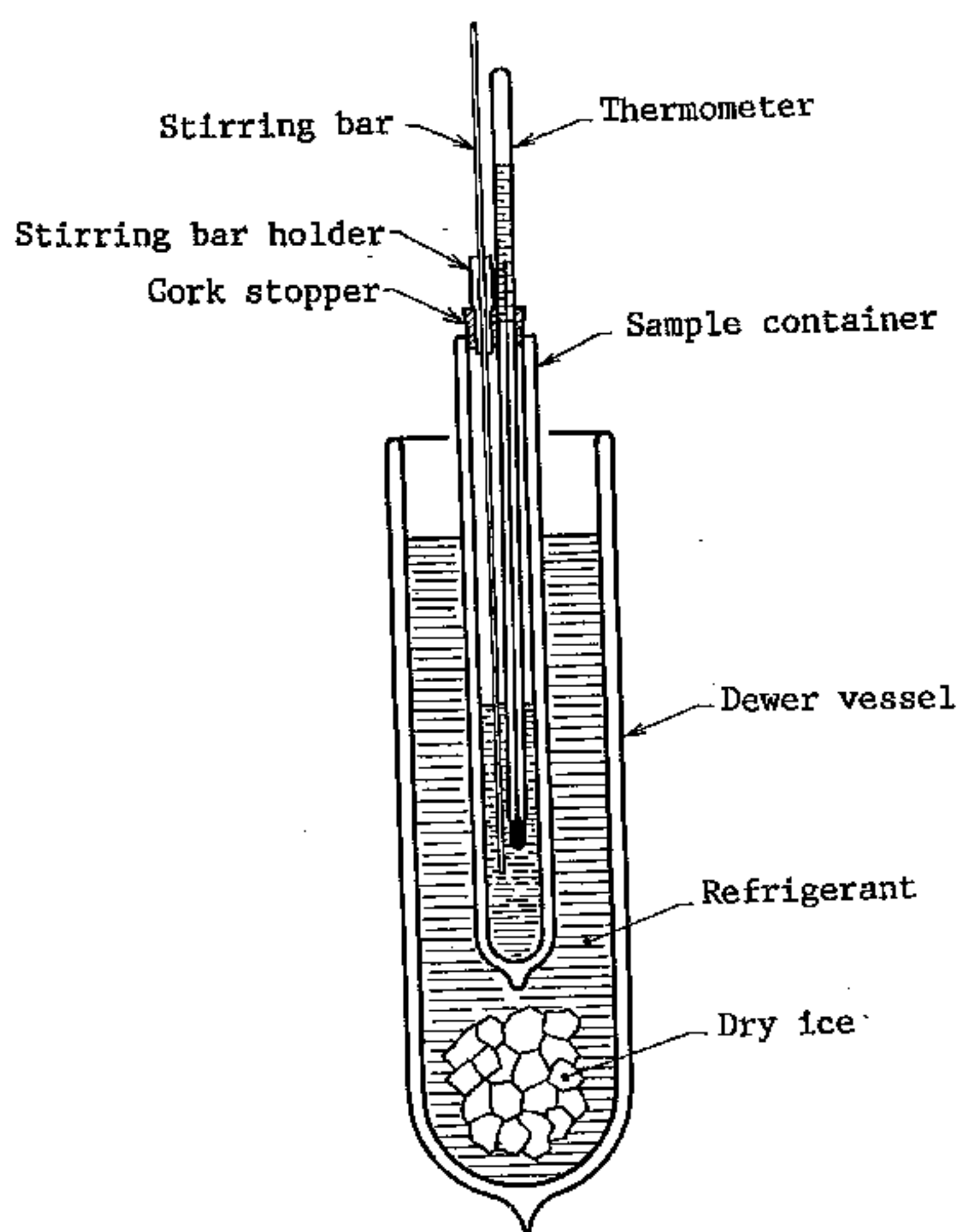
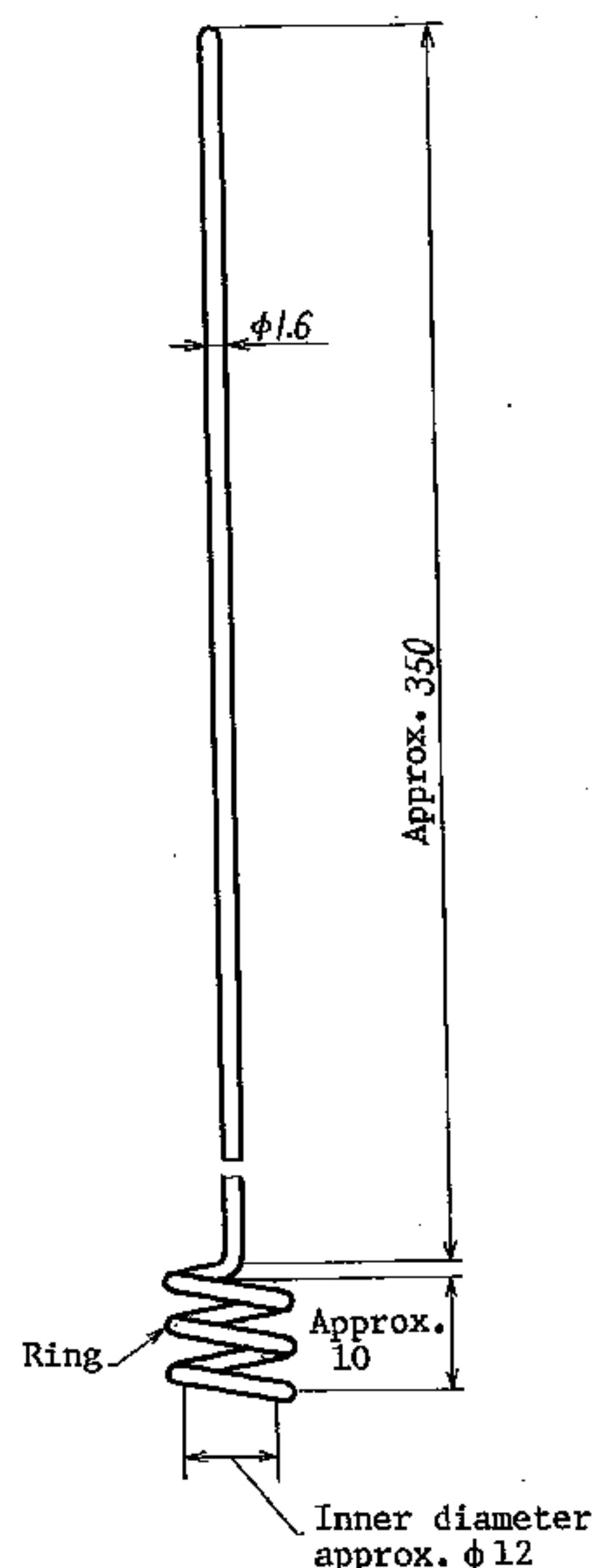
Fig. 7. Deposition-Point Tester
(An Example)

Fig. 8. Stirring Bar

Unit: mm



(1) Sample Container The sample container specified in Fig. 91 of JIS K 2839.

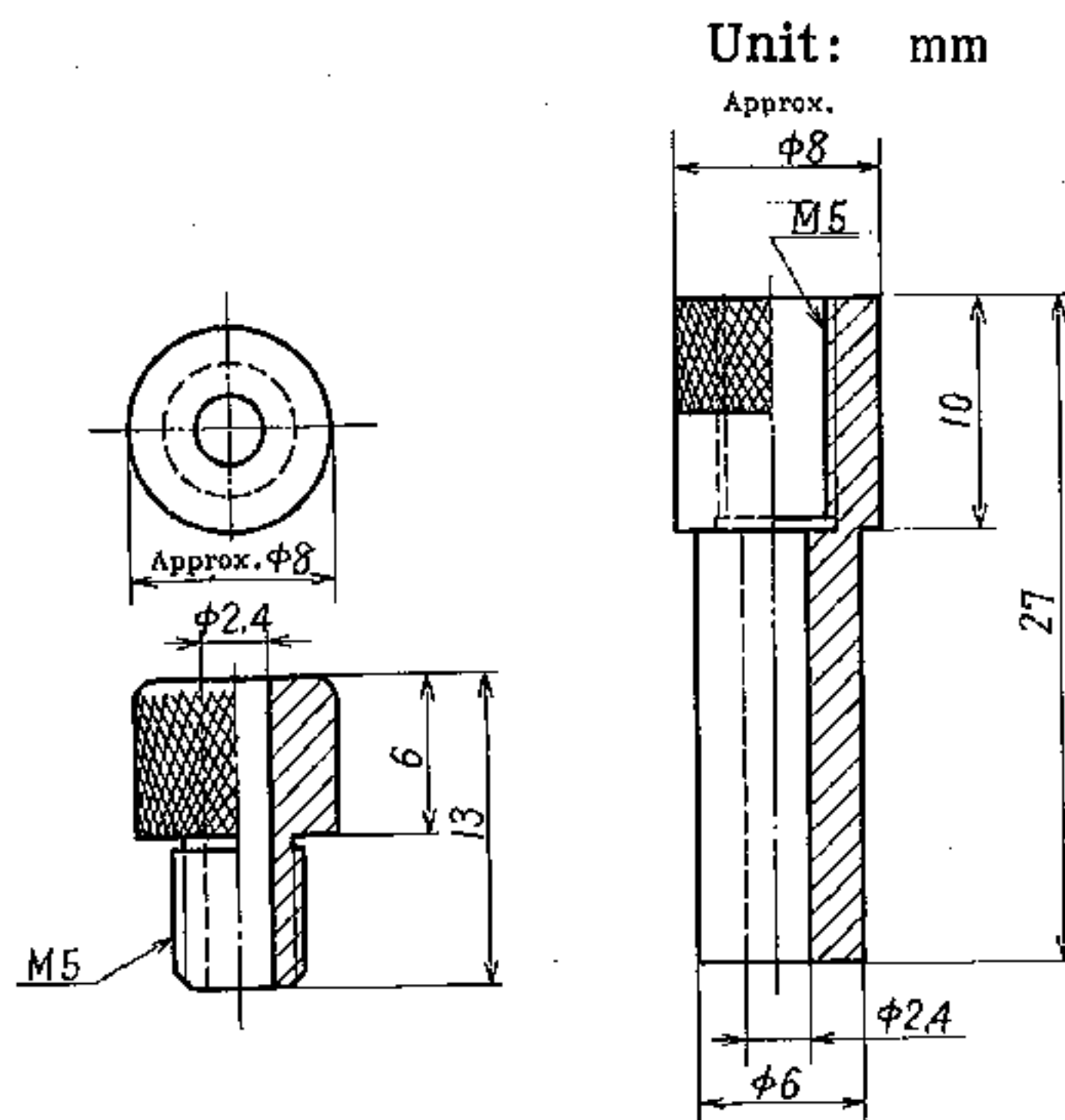
(2) Dewer Vessel The Dewer vessel specified in Fig. 22 of JIS K 2839.

Remark: Instead of Dewer vessel by using a refrigerator, a cooling tank capable of cooling the sample container may be used.

(3) Stirring Bar The stirring bar shall be of shape and dimensions shown in Fig. 8, made of brass wire or stainless steel wire of 1.6 mm in diameter and its tip end is wound in three spirals.

(4) Stirring Bar Holder The stirring bar of shape and dimensions shown in Fig. 9, made of brass.

Fig. 9. Stirring Bar Holder (An Example)



Remark: Instead of stirring bar holder, a humidity preventer [the one specified in Fig. 92 (Type I) or Fig. 93 (Type II) of JIS K 2839)] may be used.

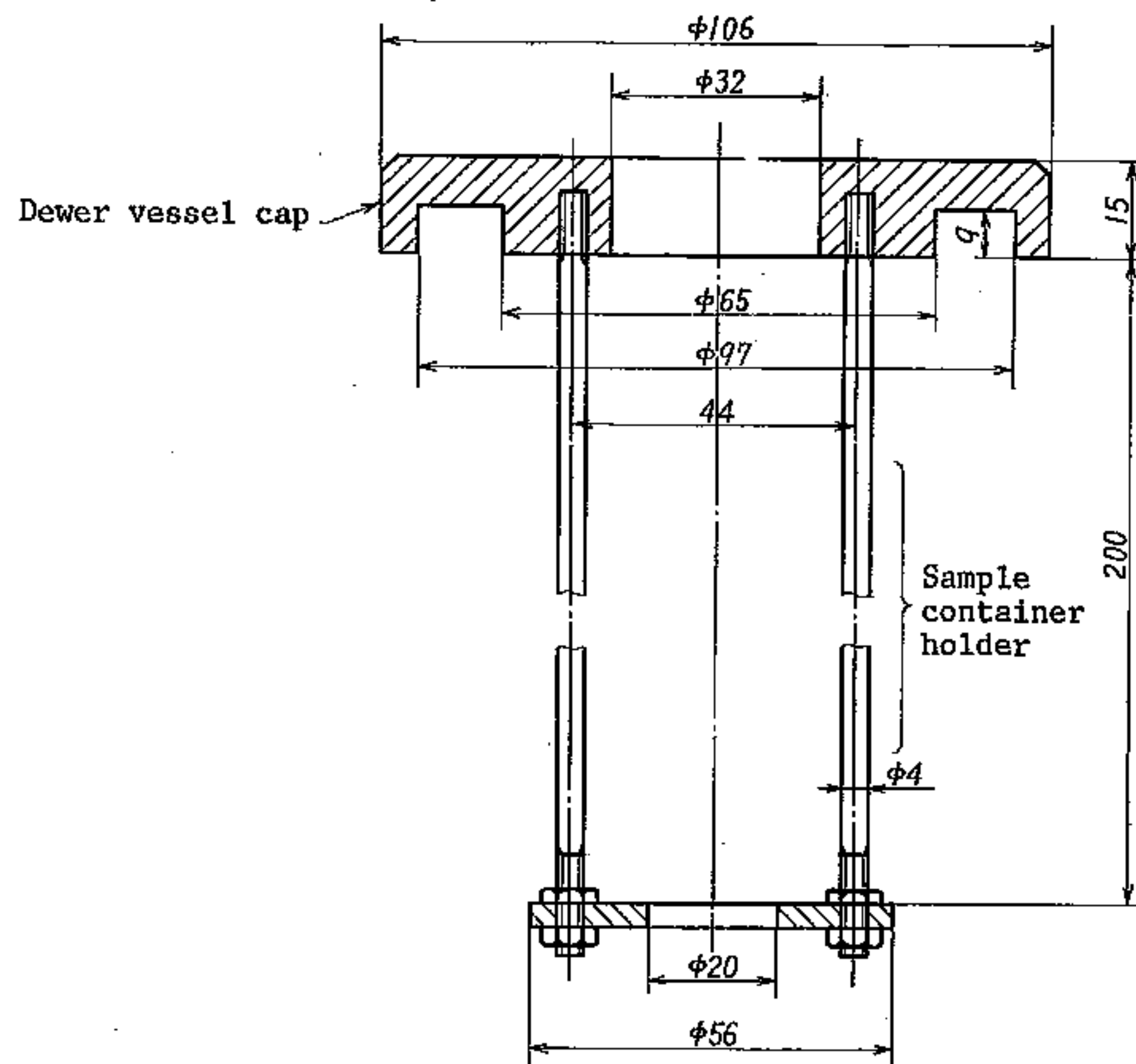
- (5) Thermometer A thermometer of thermometer number 71 (FP) specified in JIS B 7410, and preliminarily the errors at 0, - 40, - 50 and - 60°C shall be obtained according to Annex (correction testing method) of JIS B 7410 shall be obtained.

Informative Reference: In the case where the Dewar vessel cap, outer cylinder, etc. are used, the shape and dimensions are shown in the following;

- (a) Dewar Vessel Cap The Dewar vessel cap is of shape and dimensions shown in Informative Reference Fig. 1, made of synthetic resin or metal and at its middle part, the inserting hole of sample container is provided and at its lower part, the sample container holder is provided.

Informative Reference Fig. 1. Dewer vessel Cap and Sample Container Holder (An Example)

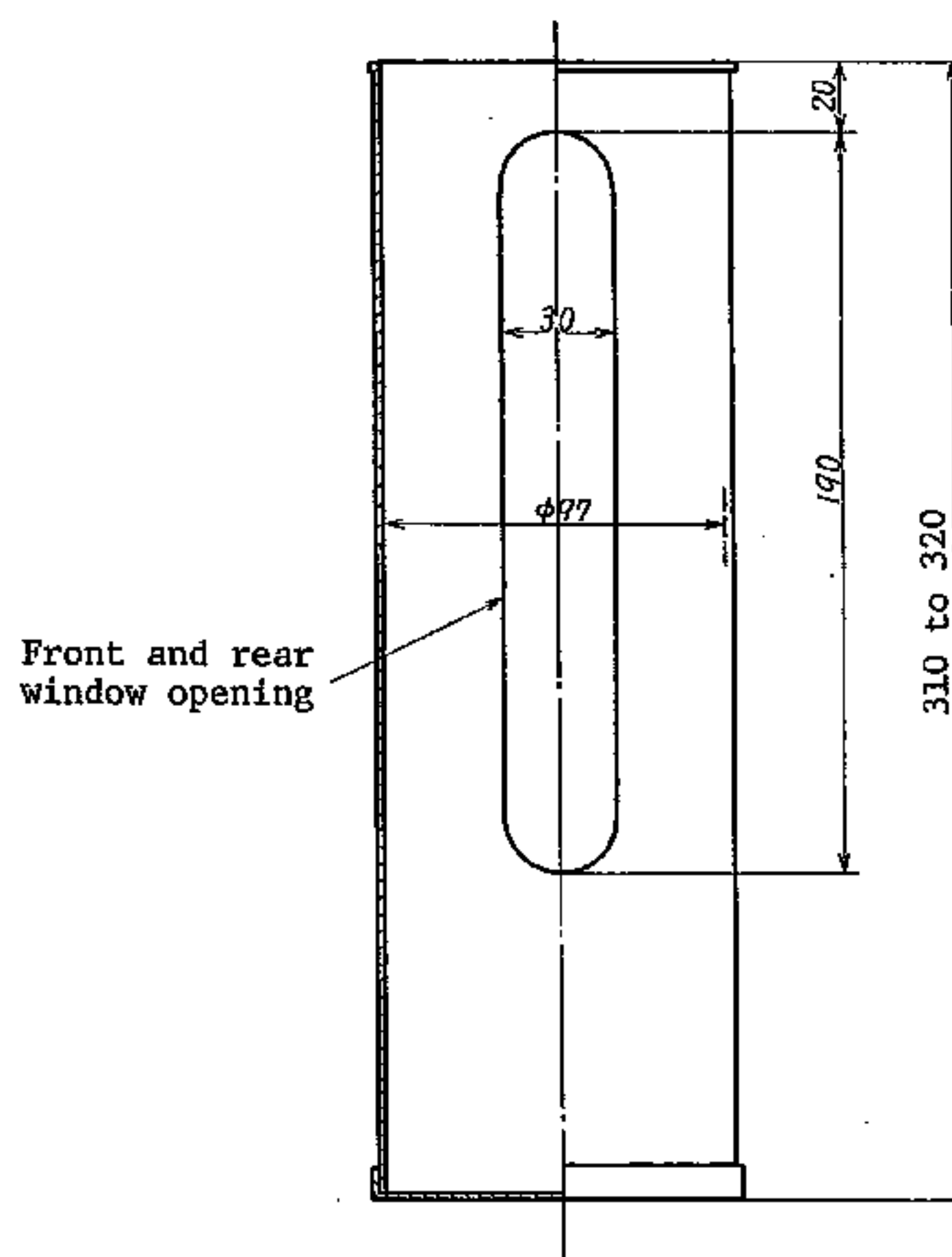
Unit: mm



- (b) Outer Cylinder The cylinder of shape and dimensions shown in Informative Reference Fig. 2, made of metal, which is capable of viewing directly the sample container from the front and rear windows when a Dewer vessel is entered in its inside.

Informative Reference Fig. 2. Outer Cylinder (An Example)

Unit: mm



6.3 Procedures of Test The procedures of test for deposition point testing method shall be as follows:

- (1) Take approximately 25 ml of sample into a clean and dried sample container. Stopper rigidly the sample container with a cork stopper provided with a stirring bar, thermometer, etc. Regulate the position of thermometer so that the bulb of thermometer comes at the center of sample. Wet ⁽²⁾ the absorbent cotton filled in a stirring bar holder with dropping down one drop of ethanol, and adjust the fastening of screw threads of stirring bar holder until such degree that the stirring bar moves smoothly without applying a large force.

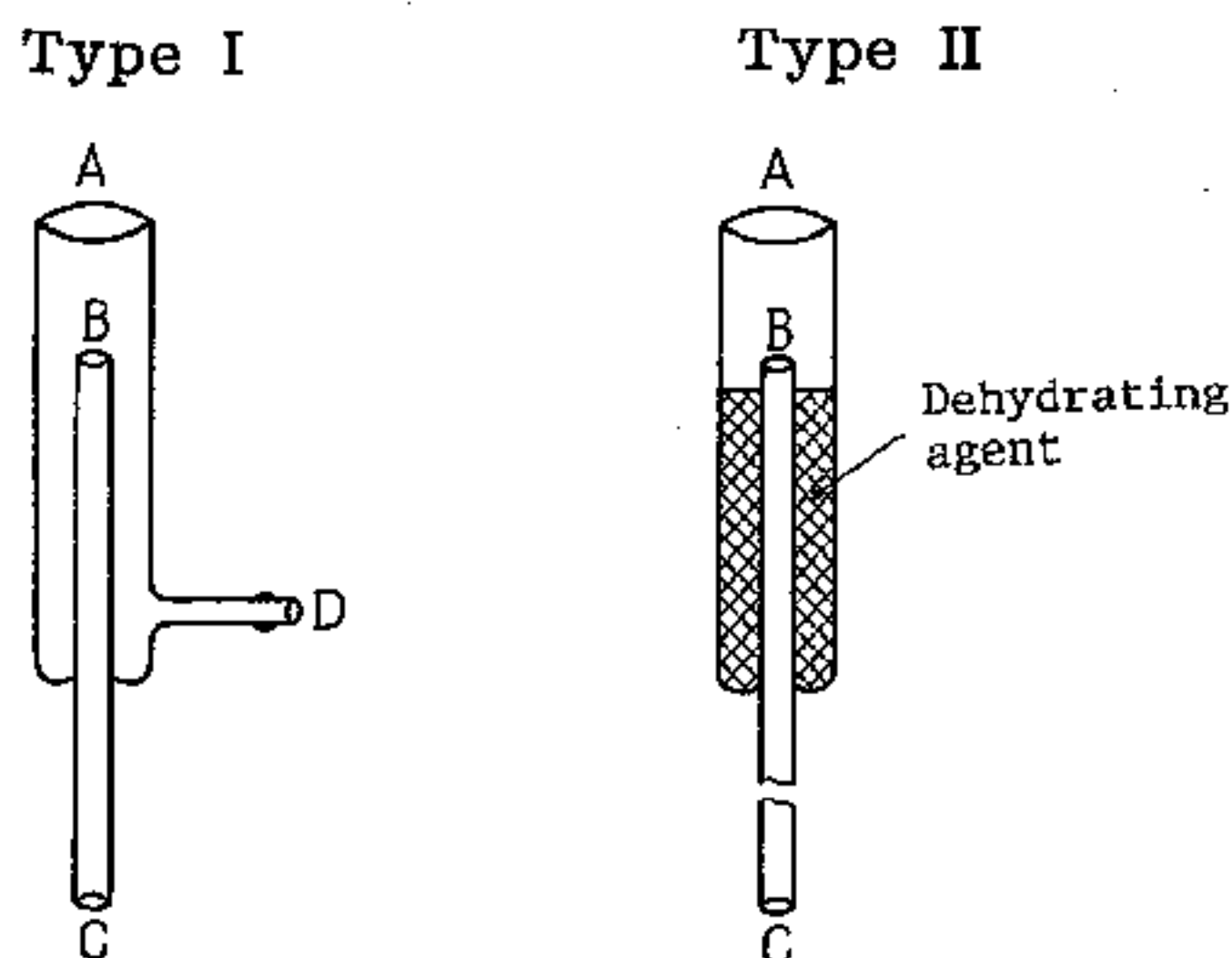
Note ⁽²⁾ Because it is a test at a low temperature, it is required for prevention of condensation of moisture content in air in the sample container.

Remark: In the case where instead of stirring bar holder a moisture proof device is used, the procedures shall be carried out as follows:

In the Type I moisture proof device (Fig. 10), after inserting the part of C instead of stirring bar holder of Fig. 9, pass the stirring bar through C, B and A. Connect the conduit of dry air with D, while feeding continuously the dry air enter the sample container into the Dewar vessel, and continue this until completion of test. Pass the dry air through U-tube filled with calcium sulfate (anhydride) or silica gel (hereafter referred to as the "dehydrating agent"), then, pass the U-tube packed with glass beads of which surface is covered with phosphorous pentaoxide so as to be used.

In the case of Type II moisture proof device (Fig. 10) pack the dehydrating agent of approximately 1.7 mm in grain size from B until the 5 mm lower part. After inserting the part C instead of stirring bar holder of Fig. 9, pass the stirring bar through C, B and A. Then, pack the glass wool containing dehydrating agent of the same grain size up to the part of A. The glass wool shall be exchanged at each use of 3 to 4 times.

Fig. 10. Moisture Proof Device (An Example)



- (2) Enter the sample container into the Dewar vessel containing refrigerant (³). Make the liquid surface of refrigerant upper than the liquid surface of sample always during the test.

Note (³) Use a suitable liquid (for example acetone or ethanol) cooled by dry ice. Instead of liquid cooled by dry ice, liquid nitrogen may be used. Because the liquid oxygen and liquid air accompanies risk when the sample container is broken, those shall not be used.

- (3) Taking care so that the ring of stirring bar does not go out upper the liquid surface of sample, mix by stirring. Except the time of observing, mix the sample by violently and continuously stirring (⁴).

When the crystal (⁵) of hydrogen carbide has appeared, stop the mixing by stirring and record the temperature. Then, take out the sample container from Dewar vessel, and while mixing continuously by violently stirring, gradually warm at room temperature. When the crystal of hydrogen carbide has disappeared, record the temperature. In the case where the difference between the temperatures when the crystal appeared and when disappeared exceeds 3°C, repeat the cooling and warming until the difference becomes within 3°C.

Notes (⁴) When the deposition point is known preliminarily, it may be mixed by stirring at times until the temperature of sample becomes within 10°C of anticipated deposition point. However, thereafter, it shall be mixed by stirring violently. An electrically driven stirrer may be used.

(⁵) When cloudiness is generated in the sample at nearly - 10°C, and ever further lowered the temperature the degree of cloudiness does not become larger, it is due to moisture content and therefore it is neglected. Relating to such sample that this cloudiness interferes the observation of crystal of hydrogen carbide, after removing the moisture content by dehydrating with sodium sulfate (anhydride), retest it.

6.4 Result As to the deposition point, the temperature when the crystal of hydrogen carbide has disappeared completely shall be corrected by error of thermometer and rounded off to the nearest 0.5°C.

6.5 Accuracy The accuracy of deposition-point testing method shall be as follows:

- (1) Repeatability When the same sample is tested twice in the same testing room by the same person by using the same tester with changing the day or time, the difference of test results shall not exceed 0.7°C.
- (2) Reproducibility The difference of two test results obtained by testing the same sample respectively once in different two testing rooms by different persons by using other tester shall not exceed 2.6°C.

18
K 2276-1989

7. Water Solubility Testing Method

7.1 Summary of Testing Method At room temperature mix 80 ml of sample and 20 ml of phosphate buffer solution by shaking violently for 2 min, and obtain the amount of component to be mixed with water in sample from the volume variation of water layer.

Further, evaluate the surface states and separation states of the sample layer immediately after reading out of volume variation and the water layer.

7.2 Water Solubility Tester The water solubility tester shall be as follows:

Measuring Cylinder A measuring cylinder specified in JIS R 3505 of 100 ml in nominal capacity (with stopper type), having scale interval of 1 ml.

7.3 Reagents Reagents for water solubility testing method shall be as follows:

- (1) Phosphate Buffer Solution Dry potassium dihydrogen phosphate and dipotassium hydrogen phosphate specified in JIS K 9007 and JIS K 9017 at 110°C for 3 h to remove the water content. After leaving cool, dissolve potassium dihydrogen phosphate 4.7 g and dipotassium hydrogen phosphate 11.5 g in water 1 l. This pH is approximately 7.
- (2) Detergent for Glass Devices The chromic acid mixed solution ⁽⁶⁾ specified in 5.3 (2) or the detergent equivalent thereto.
- (3) Hexane The hexane specified in JIS K 8848.
- (4) Heptane The heptane specified in JIS K 2280.

Note ⁽⁶⁾ The chromic acid mixed solution completed with using shall be treated to make harmless.

7.4 Washing of Measuring Cylinder and Stopper Wash the measuring cylinder and stopper before testing according to the following method until water drip becomes capable completely.

Wash the measuring cylinder and stopper by hot water to remove ⁽⁷⁾ the oil film. As required, carry out brushing. Then, after immersing into glass-device detergent, rinse in the sequence of city water, water, phosphate buffer solution to drip water.

In case where the water can not be dripped off cleanly from the internal wall of measuring cylinder, after immersing the measuring cylinder into the glass-device detergent heated at approximately 65°C for approximately 30 min, rinse in the sequence of city water, water, phosphate buffer solution to drip water.

Note ⁽⁷⁾ Instead of washing by hot water, by washing with hexane or heptane and by rinsing in the sequence of acetone, city water, the oil film may be removed.

7.5 Procedures of Test Procedures of test for water solubility testing method shall be as follows:

- (1) At room temperature, take phosphate buffer solution 20 ml into a measuring cylinder, read its amount to the nearest 0.5 ml unit and record. Then, add sample 80 ml at room temperature and stopper the measuring cylinder.
- (2) Shake the measuring cylinder at an amplitude of 13 to 25 cm at 2 to 4 reciprocation per 1 s strongly up and down correctly for 2 min ⁽⁸⁾.

Then, after standing still the measuring cylinder at a place without vibration for 5 min, read out the volume of water layer to the nearest 0.5 ml unit, and record the variation of its volume.

Note ⁽⁸⁾ Do not shake the measuring cylinder in spiral state, because this has a fear that the generated emulsified layer is broken.

- (3) Observe the surface states and separation states of sample layer and water layer immediately after reading-out of volume variation and evaluate according to Table 4 and Table 5, respectively.

Table 4. Evaluation of Surface State

Evaluation number	Surface state
1	Transparent and clean state
1 b	The area occupied by small transparent bubbles is approximately not more than 50 % of surface and the state without small pieces, lace state or film state.
2	The state where small pieces, lace state or film state is generated.
3	The state where lace state matters are spread sparsely or a small amount of foreign matters are generated.
4	The state where lace state matters are collected tightly and thick or a large amount of foreign matters is generated.

Table 5. Evaluation of Separation State

Evaluation number	Separation state
(1)	The state where the emulsified matters or precipitates are free completely on the both layers or upper part of sample layer.
(2)	Except that small air bubbles and water drops exist on the sample layer, the state same as (1).
(3)	The state where emulsified matters or precipitates exist on both layers or upper part of sample layer, or where small drops exist on water layer or internal wall of measuring cylinder.

7.6 Results Results of water solubility testing method shall be as follows:

- (1) Volume Variation Express the volume variation as the water-layer volume variation by unit of 0.5 ml.
- (2) Surface State Relating to surface states of sample layer and water layer, express by evaluation number of Table 4.
- (3) Separation State Relating to separation state of sample layer and water layer, express by evaluation number of Table 5.

7.7 Accuracy The accuracy is not specified.

8. Total Acid Number Testing Method

8.1 Summary of Testing Method Dissolve the sample in mixed solvent of toluene, 2-propanol and a little amount of water, taking α -naphtholbenzein as indicator, while blowing-in nitrogen at room temperature, titrate by using potassium hydroxide standard 2-propanol solution to measure the total acid number.

Remark: This method applies to measurement of total acid number of aviation turbine fuels in the range of 0.001 to 0.100 mg KOH/g.

8.2 Total Acid Number Tester The total acid number tester consists of following (1) to (3).

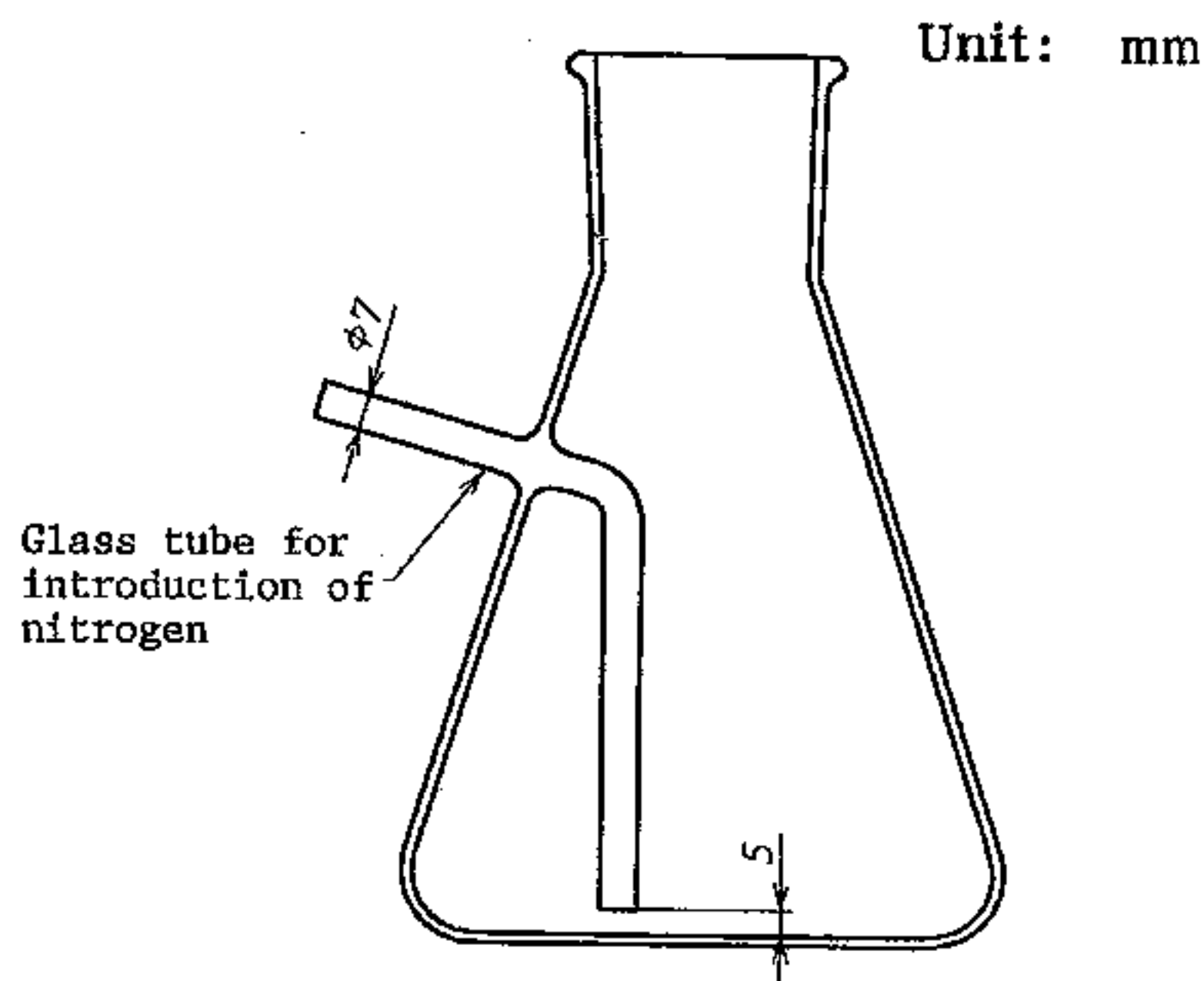
- (1) Burette A burette specified in Fig. 99 of JIS K 2839.

Remark: A burette of not less than 2 ml in capacity, having scale interval of 0.01 ml may be used.

- (2) One Mark Volumetric Pipet One mark volumetric pipets of 0.1 ml and 100 ml in capacity.

- (3) Flask for Titration An Erlenmeyer flask of 500 ml in capacity provided with a glass tube for introduction of nitrogen, and its one example is shown in Fig. 11.

Fig. 11. Flask for Titration (An Example)



8.3 Reagents Reagents for total acid number testing method shall be as follows:

- (1) 2-Propanol 2-Propanol specified in JIS K 8839.
- (2) Water The water removed of carbon dioxide by boiling.
- (3) Phenolphthalein Indicator Dissolve phenolphthalein 0.10 g specified in JIS K 8799 in mixed solution of ethanol 50 ml specified in JIS K 8101 and water 50 ml.
- (4) N/100 Potassium Hydroxide Standard 2-Propanol Solution (hereafter referred to as the "N/100 KOH solution") Take 2-propanol approximately 1 l into an Erlenmeyer flask of 2 l, add to this potassium hydroxide 0.6 g specified in JIS K 8574, and while so mixing by stirring on steam bath that lumps are not generated on the bottom, boil gently for 10 to 15 min. Then, draw out from steam bath, add barium hydroxide 0.2 g specified in JIS K 8577, and after boiling gently again approximately for 10 min, cool to room temperature.

Shield this solution from atmosphere, after leaving as it is for a night (approximately 16 h), filter the supernatant by a membrane filter made of ethylene tetrafluoride resin of pore diameter of 10 μm so as not to expose to atmosphere to the degree not required, and taking so cares that this filtrate does not contact the cork, rubber, or grease for stopper cock having a fear to be sponified or the like, preserve with attaching a protective tube filled with soda lime or the like.

The standardization of this solution shall be performed according to the following operation ⁽⁹⁾, and restandardize at times at such intervals that the variation of normality does not become 0.0002 N or more.

- (a) Dry potassium biphthalate specified in JIS K 8809 at 120°C for not less than 2 h, weigh out its approximately 0.1 g to the nearest 0.1 mg, dissolve in water, transfer into a one mark volumetric flask 1 l, and add water up to the marked line.
- (b) Take its 100 ml into a flask for titration by using a one mark volumetric pipette, add 6 drops of phenolphthalein indicator solution, and titrate by using N/100 KOH solution until the colour of solution turns to pink from colorless.

Repeat this operation not less than twice.

- (c) Relating to water 100 ml, carry out the blank test according to the operation of (b).
- (d) Obtain the normality of N/100 KOH solution according to the following formula to four places of decimals.

$$N = \frac{100W}{204.23 \times (V - b)}$$

where, N : normality of N/100 KOH solution

W : sampling amount of potassium biphthalate (g)

V : average value of amount of N/100 KOH solution required for titration until end point (ml)

b : amount equivalent to V in blank test (ml)

Note ⁽⁹⁾ The body expansion coefficients of organic solvents are comparatively large and therefore the standardization by N/100 KOH solution is carried out at a temperature near the temperature at using time.

- (5) Mixed Solvent 2-Propanol 495 ml added with toluene specified in JIS K 8680 ⁽¹⁰⁾ 500 ml and water 5 ml.

Note ⁽¹⁰⁾ Because the vapour of toluene is poisonous, take care so as not to absorb. Specially it is dangerous when entering in eyes.

- (6) α -Naphtholbenzein Indicator Solution α -Naphtholbenzein 1.0 g specified in JIS K 8693 dissolved in mixed solvent 100 ml.

- (7) Nitrogen The nitrogen not inferior to Grade 2 specified in JIS K 1107.

8.4 Washing of Flask for Titration In order to remove the contamination of oil and the like so as to make the flask for titration, after washing in the sequence of suitable solvent (precipitate naphtha, isooctane, etc.), acetone, city water, further wash in the sequence of detergent or chromic acid mixed solution ⁽¹¹⁾, city water, water to make clean.

Note ⁽¹¹⁾ The chromic acid mixed solution completed with using shall be treated to make harmless.

8.5 Procedures of Testing The procedures of test for total acid number testing method shall be as follows:

- (1) Weigh out sample 100 ± 5 g at the accuracy of 0.5 g into a flask for titration.
- (2) Add mixed solvent 100 ml, and α -naphtholbenzein indicator solution 0.1 ml to the flask for titration in which the sample is weighed out, and blow-in nitrogen at a flow rate of 600 to 800 ml per minute for 3 min.
- (3) While blowing-in nitrogen, titrate the sample solution at a temperature not higher than 30°C by using N/100 KOH solution (¹²).

As approaching to the end point, the orange colour turns to green brown colour. Take the first point when having turned to the green or green-brown colour remains as it is for 15 s not to return to the original as the end point.

- (4) As to the blank test, add α -naphtholbenzein indicator 0.1 ml to mixed solvent 100 ml in a flask for titration and titrate by N/100 KOH (¹²).

Note (¹²) Carry out the titration at a place where local exhausting of gas is applied.

8.6 Calculation and Result Calculate the total acid number of sample according to the following formula and round off to three places of decimals.

$$A = \frac{(B - C) N \times 56.1}{W}$$

where, A: total acid number (mgKOH/g)

B: amount of N/100 KOH solution required for titration of sample (ml)

C: amount of N/100 KOH solution required for titration of blank test (ml)

N: normality of N/100 KOH solution

W: amount of taken sample (g)

8.7 Accuracy The accuracy of total acid number testing method shall be as follows:

- (1) **Repeatability** When the same sample is tested twice in the same test room by the same person by means of same tester with changing day or time, the difference of test results shall not exceed the tolerance of Table 6.
- (2) **Reproducibility** The difference of two test results obtained by testing respectively once the same sample in different two test rooms by different person by means of different tester shall not exceed the tolerance of Table 6.

Table 6. Accuracy

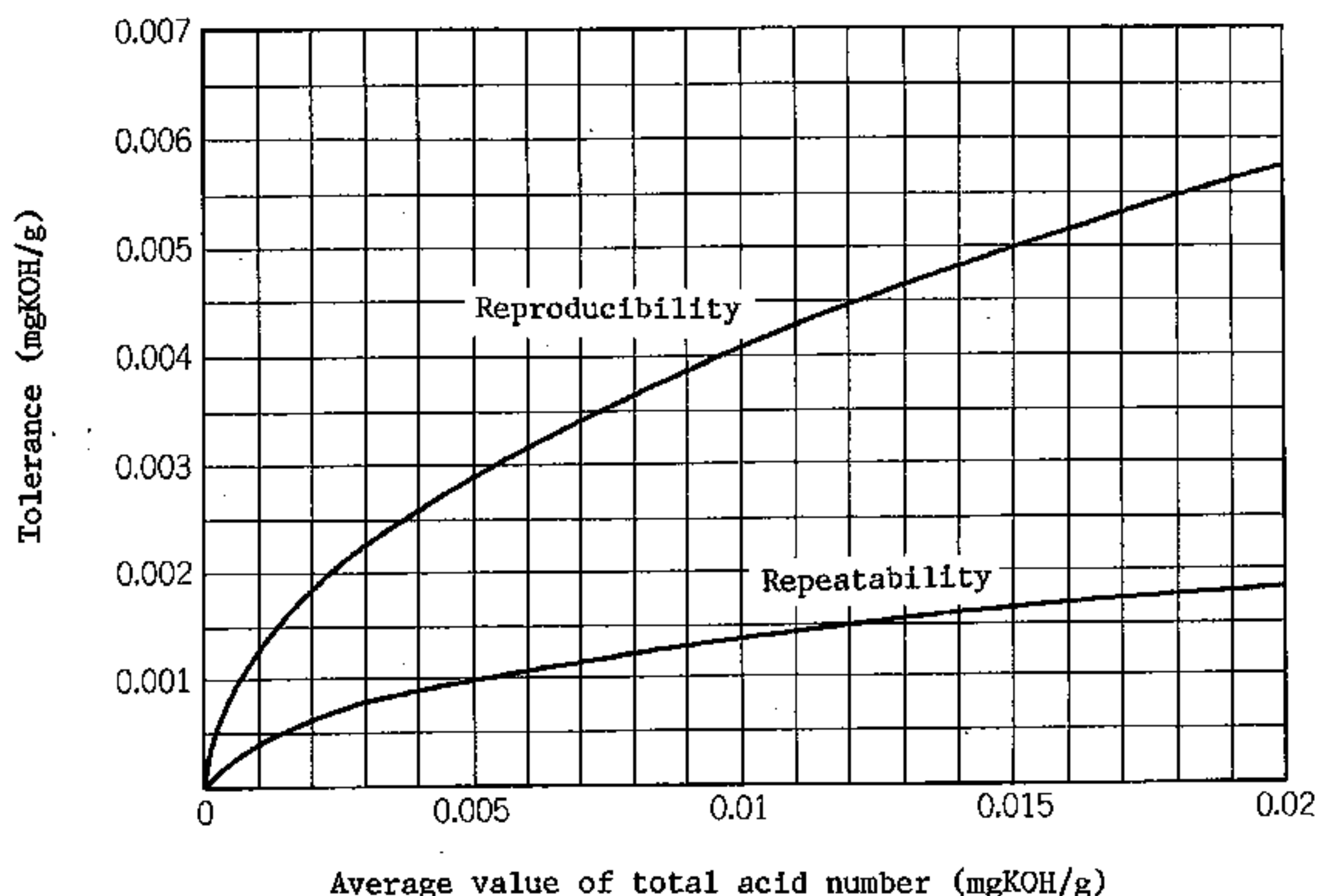
Unit: mgKOH/g

Total acid number	Repeatability tolerance	Reproducibility tolerance
Over 0.001 up to and incl. 0.100	$0.013\ 2\sqrt{A}$	$0.040\ 6\sqrt{A}$

Remark: A in the Table is the average value of test results (mgKOH/g).

Informative Reference: Calculated tolerances of Table 6 are shown in Informative Reference Fig. 3.

Informative Reference Fig. 3. Accuracy



9. Mercaptan Sulfur Content Testing Method (Potentiometric Titration)

9.1 Summary of Testing Method After removing hydrogen sulfide by washing the sample with sodium hydrogencarbonate, determine the mercaptan sulfur content by potentiometric titration. Namely, add titration solvent to the sample removed of hydrogen sulfide, and by using silver-silver sulfide electrode, glass electrode carry out the potentiometric titration with silver nitrate standard 2-propanol solution. At this time, the mercaptan precipitates as mercaptid and therefore obtain the titration end point from the potentiometric titration curve to determine mercaptan sulfur content.

- Remarks 1. This method is the method to measure the mercaptan sulfur content in aviation turbine fuels containing mercaptan sulfur content of from 0.0003 mass % up to 0.01 mass %, and even organic sulfur compound such as sulfide, disulfide, thiophane, etc. and element sulfur less than 0.0005 mass % are contained in the sample, the measurement is not interfered.
2. Hydrogen sulfide interferes the measurement and therefore shall be removed according to 9.5 (3).

9.2 Mercaptan Sulfur Content Tester (Potentiometric Titration) The tester shall consist of following (1) to (6).

- (1) Potentiometer (hereafter referred to as the "meter") The meter which actuates at input less than 9×10^{-12} A, and has sensitivity of ± 2 mV at least in the range of ± 1 V or the meter having performance equivalent or superior thereto.

Further, at the time of use, it shall be shielded electrostatically and be grounded.

- (2) Electrodes The electrodes consist of contrast electrode and indicating electrode, the contrast electrode is the one specified in JIS Z 8805 and the indicating electrode is the silver wire of approximately 2 mm in diameter attached to the insulated holder.
- (3) Burette The burette specified in Fig. 101 of JIS K 2839.
- (4) Tall Beaker A tall beaker of 300 ml in capacity specified in JIS R 3503.
- (5) Stirrer A stirrer made of glass of propeller type or electromagnetic type.
- (6) Abrasive Paper A abrasive paper of AA (alumina property) No. 1000 specified in JIS R 6253.

9.3 Reagents Reagents shall be as follows:

- (1) Sodium Hydrogen Carbonate Dissolve sodium hydrogen carbonate 10 g specified in JIS K 8622 in water 200 ml.
- (2) N/10 Potassium Iodide Standard Solution Weigh out potassium iodide approximately 16.6 g specified in JIS K 8913 correctly to the nearest 0.01 g, put into a one mark volumetric flask 1 l, and after dissolving in water of a small amount, put-in water up to the marked line.

Calculate the normality according to the following formula and round off to four places of decimals.

$$N = \frac{M}{166.01 V}$$

where, N : normality of potassium iodide standard solution

M : sampling amount of potassium iodide (g)

V : prepared amount of reagent (l)

- (3) N/10 Silver Nitrate Standard 2-Propanol Solution (Hereafter Referred to as the N/10 AgNO₃ Solution) Dissolve silver nitrate 17 g specified in JIS K 8550 in water 100 ml, and dilute by 2-propanol (¹³) specified in JIS K 8839 to 1 l. Store this solution in a brown bottle, and at each one week standardize by N/10 potassium iodide standard solution (¹⁴).

Notes (13) If peroxide exists, the measuring value appears lower and therefore pass through a column of active alumina to refine.

(14) Enter water 100 ml in tall beaker 300 ml, add 6 drops of nitric acid and boil for 5 min to remove the nitrogen oxide. After standing to cool, take N/10 potassium iodide standard solution 5 ml by using a pipette and enter into a tall beaker. Relating to this, by using N/10 AgNO_3 solution, obtain the normality of N/10 AgNO_3 solution from the bending point of potentiometric titration curve.

(4) N/100 Silver Nitrate Standard 2-Propanol Solution (Hereafter Referred to as the "N/100 AgNO_3 Solution") At each test day dilute N/10 AgNO_3 solution with 2-propanol (13) to 10 times to prepare.

(5) Sodium Sulfide Solution Dissolve sodium sulfide 15 g specified in JIS K 8949 in water to dilute to 500 ml.

Further, prepare this solution at each time of use.

(6) Titration Solvent Dissolve sodium acetate (trihydrate) 2.7 g specified in JIS K 8371 or sodium acetate (anhydride) 1.6 g specified in JIS K 8372 in water containing no oxygen, pour into 2-propanol (13) 975 ml, add acetic acid 4.6 ml specified in JIS K 8355 and so store as not to contact atmosphere. Before using, pass nitrogen violently through this solvent for 10 to 15 min to remove oxygen, and then use this solvent.

Remark: Because the low molecular weight mercaptans are difficult to be scavenged by this titration solvent, in the case of measuring mercaptan in gasoline, the following alkaline titration solvent should preferably be used. Dissolve sodium acetate (trihydrate) 2.7 g or sodium acetate (anhydride) 1.6 g in water 25 ml containing no oxygen, pour into 2-propanol (13) 975 ml and store so as not to contact atmospheric air. Before using this solvent pass nitrogen violently through this solvent for about 10 min to remove oxygen and then use it.

(7) N/10 Sodium Acetate Alcohol Solution Dissolve sodium acetate (trihydrate) 13.6 g into alcohol 1 l which is prepared by mixing 1 part of methanol specified in JIS K 8891 and 10 parts of ethanol specified in JIS K 8101 (99.5), and pass violently nitrogen gas to mix.

(8) Lead Acetate Paper Dissolve lead acetate (trihydrate) 5 g specified in JIS K 8374 in water 100 ml and after adsorbing in filter paper, dry it.

9.4 Preparation of Test The preparation of test for mercaptan sulfur content testing method (potentiometric titration) shall be as follows:

- (1) Glass Electrode Before and after each titration, wipe the electrode with a clean cloth and rinse with water. At times (when using continuously, at least at a rate of once per week) clean the electrode with mixing by stirring in cool chromic acid mixed solution ⁽¹⁵⁾ for several seconds (10 s max.). When not using, immerse the lower half of electrode into water.

Note ⁽¹⁵⁾ The chromic acid mixed solution completed with use shall be treated to make harmless.

- (2) Silver-Silver Sulfide Electrodes At each test day, generate new silver sulfide on electrode according to the following method to be used. Polish by abrasive paper until clean silver surface appears. Place the electrode at the measuring position, immerse it into the mixed solution of sodium sulfide solution 8 ml and N/10 sodium acetate alcohol solution 97 ml, and while mixing by stirring, gradually add from a burette N/10 AgNO₃ solution 10 ml requiring 10 to 15 min ⁽¹⁶⁾. Take out the electrode from the solution, wash with water and wipe with a soft and clean cloth. Between the measurements immerse the electrode in the titration solvent 100 ml containing N/10 AgNO₃ solution 0.5 ml.

Note ⁽¹⁶⁾ In the case where even according to this operation, a uniform silver sulfide coating film is not generated on the silver wire, further drop gradually N/10 AgNO₃ solution 10 ml with requiring 10 to 15 min.

9.5 Procedures of Test The procedures of test for mercaptan sulfur content testing method (potentiometric titration) shall be as follows:

- (1) In the case of such sample that existence of no hydrogen sulfide is known, conduct according to (4).
- (2) As to such sample that the existence of hydrogen sulfide is unknown, immerse approximate 1/2 of small piece of lead acetate paper in sample, and observe the condition of colour change of lead acetate paper. When the vapor layer contacting part of lead acetate paper or the part immersed in sample has not changed in colour, proceed the test according to (4). When changed in colour to black or brown colour, hydrogen sulfide is contained, and therefore remove the hydrogen sulfide according to the following method.
- (3) Put the sample of amount 3 to 4 times the required amount for test and sodium hydrogen carbonate of volume of 1/2 of sample into a separatory funnel to shake violently. Flow out the aqueous solution layer, examine existence of hydrogen sulfide according to the operation of (2), if hydrogen sulfide is confirmed, add new sodium hydrogen carbonate solution, and repeat the similar operation until the existence of hydrogen sulfide is not confirmed. Add to the sample in which existence of hydrogen sulfide has become not to be confirmed the water of 30 to 50 ml, shake violently, and after flowing out the aqueous layer, proceed the test according to (4).
- (4) Weigh out the sample 20 to 50 ml containing no hydrogen sulfide into a tall beaker containing titration solvent 100 ml by means of a pipette ⁽¹⁷⁾ or weigh out the equivalent amount by mass, immediately place the tall beaker on a holder stand for titration, regulate the position of tall beaker so as approximately half of both electrodes to be immersed and fix.

Fill a burette with N/100 AgNO_3 solution and place a burette at such position that its tip becomes at a position approximately 25 mm under the liquid surface in the tall beaker. Then regulate the speed of stirrer so as to mix by stirring violently in the degree that the liquid is not spattered.

Note (¹⁷) Preliminarily measure the density (g/cm^3) at the temperature at taking time.

- (5) Record the first reading of the burette and meter, add suitably little by little the N/100 AgNO_3 solution, after waiting until a definite potential difference is indicated, record the reading of the burette and meter. At this time, if the variation of potential difference does not exceed 6 mV per minute, the potential difference shall be considered to be constant.

In the case where when N/100 AgNO_3 solution is dropped little by little, the variation of potential difference is small, add 0.5 ml by 0.5 ml. When the variation of potential difference has exceeded 6 mV per 0.1 ml, add each 0.05 ml of N/100 AgNO_3 solution. At nearly titration end point, 5 to 10 min is required until the potential difference becomes constant. It is important to wait until a constant potential difference is attained, but in order to avoid oxidation of sulfur compound by oxygen in atmospheric air, the time required for whole titration shall be made as shorter as possible.

Remark: In a newly prepared electrode, the readings of potential difference may disperse, and it is possible to be caused when the electrode system is not under the normal condition. However, this phenomenon disappears usually when it is titrated continuously.

- (6) Continue the titration until the reading of meter exceeds + 350 mV (¹⁸) and the variation of potential difference per 0.1 ml of N/100 AgNO_3 solution becomes approximately constant. Remove the solution after titration, rinse thoroughly the both electrodes at first with ethanol, then with water and wipe with a dry soft cloth.

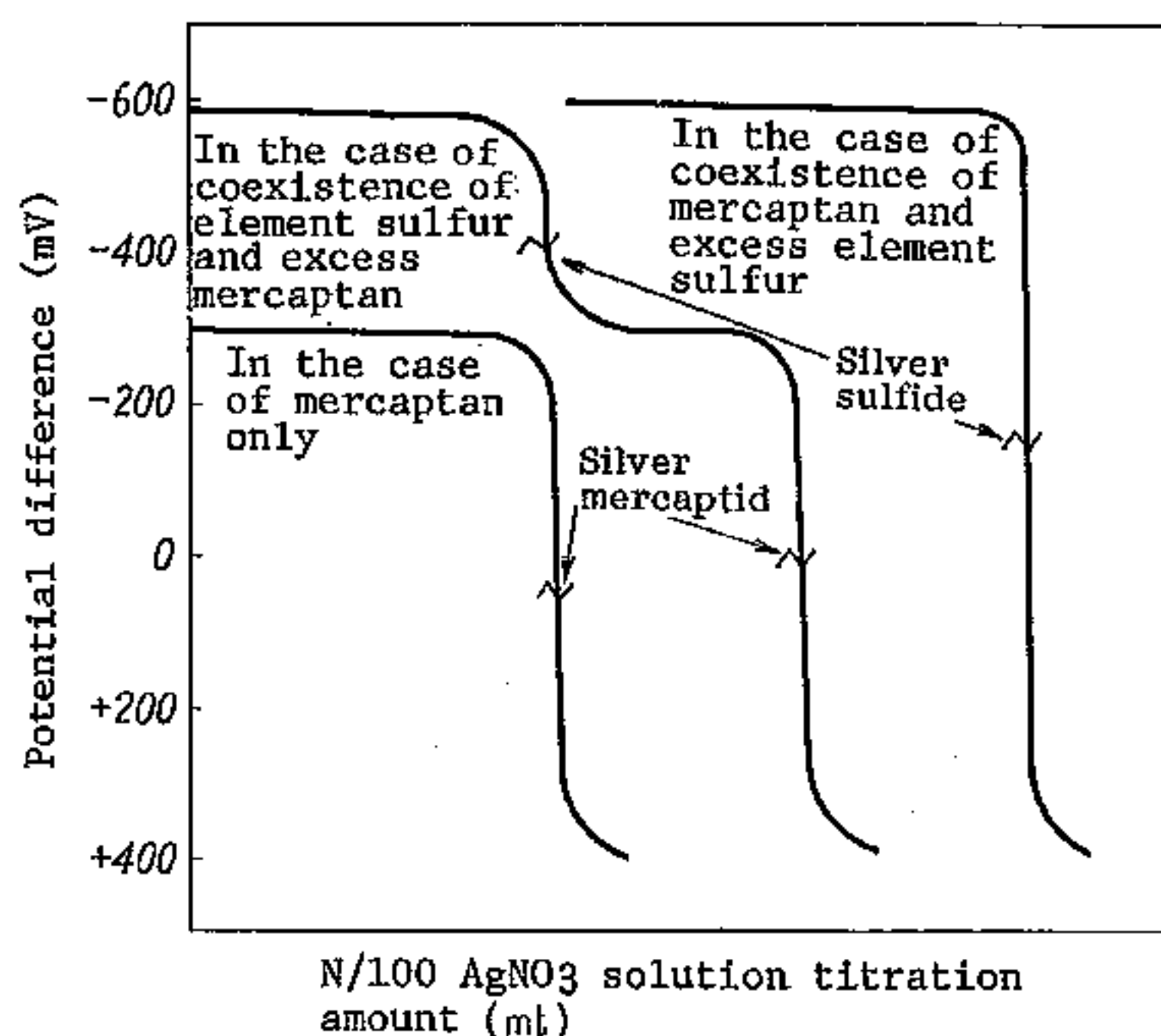
In the case where in the same day the test is carried out continuously, until transferring to the next measurement, immerse the both electrodes in the titration solvent 100 ml put-in with N/10 AgNO_3 solution approximately 0.5 ml at least for 5 min.

Note (¹⁸) Depending upon the meter, the symbols of positive and negative of potential difference may become reverse.

- (7) Plot the potential differences corresponding to titration amounts of N/100 AgNO_3 solution, prepare the titration curve as shown in Fig. 12 and obtain the titration end point (¹⁹) from its bending part.

Note (¹⁹) Obtain the titration end point according to JIS K 0113. Namely, relating to titration curve, draw two tangents of 45 degrees in inclination including bending part, and take the point where the bisectors parallel to this intersects the titration curve as the end point.

Fig. 12. Titration Curve (An Example)



- (8) In the case where mercaptan only exists in sample, the titration curve has a flat part at nearly - 300 to - 350 mV, and after rapid variation, since passing approximately + 300 mV, again it becomes loose as shown by the leftmost curve in Fig. 12. From this bending point, obtain the titration end point.
- (9) In the case where the element sulfur and mercaptan coexist in sample, in the titration solvent the element sulfur and mercaptan react in equivalent, and during titration the silver sulfide precipitates.

In the case where mercaptan exists more excessive than element sulfur in sample, at approximately - 550 to - 350 mV precipitate of silver sulfide is generated and then until reaching approximately + 300 mV precipitate of silver mercaptid is generated. This is shown in Fig. 12. At this time, because the generation of silver sulfide is based on the mercaptan equivalent to element sulfur, the titration end point is obtained from the bending part due to completion of precipitation of silver mercaptid, and the total titration amount until reaching this end point is used for calculation.

Further, in the case of sample in which element sulfur exists in excess relative to mercaptan, precipitate of silver sulfide is generated until reaching approximately + 300 mV as shown by the rightmost curve in Fig. 12. From the bending part due to completion of precipitation the titration end point is obtained.

9.6 Calculation and Results Calculate the mercaptan sulfur content according to the following formula and round off to four places of decimals.

$$S = \frac{3.206 AN}{M}$$

where, S : mercaptan sulfur content (mass %)

A : amount of N/100 AgNO_3 solution required until titration end point (ml)

N : normality of $N/100$ AgNO_3 solution used for titration

M : taking amount of sample (²⁰) (g)

Note (²⁰) In the case where the sample is weighed out by means of a pipette, it is obtained by the number of ml of sample multiplied by density (g/cm^3) at the temperature when weighing out.

9.7 Accuracy The accuracy of mercaptan sulfur content testing method (potentiometric titration) shall be as follows:

- (1) Repeatability The difference of test results, when the same sample is tested twice in the same testing room by the same person with the same tester with changing day or time, shall not exceed the tolerance of Table 7.
- (2) Reproducibility The difference of two test results obtained by testing the same sample respectively once in different two testing room by different person by using different tester shall not exceed the tolerance of Table 7.

Table 7. Accuracy

Unit: mass %	
Repeatability tolerance	Reproducibility tolerance
$0.00007 + 0.027 S$	$0.00031 + 0.042 S$

Remark: Herein S is the average value of test results.

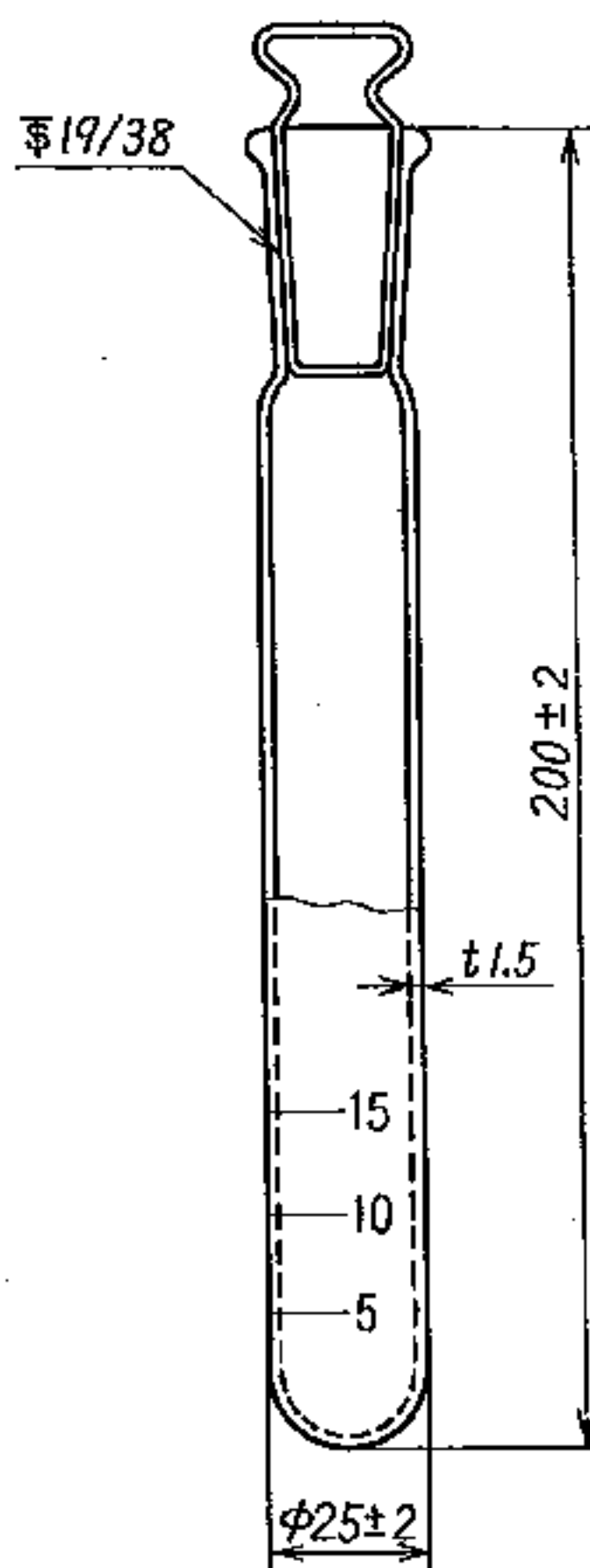
10. Doctor Testing Method

10.1 Summary of Testing Method Add doctor solution to the sample to mix by shaking and thereafter observe the change in colour of doctor solution layer. In case where there is no change in colour, further add powder sulfur, observe the degree of change in colour of powder sulfur and judge whether hydrogen sulfide, and mercaptan exist in sample or not.

10.2 Doctor Tester

Test Tube A test tube made of glass with ground stopper of approximately 50 ml in capacity, and attached with graduation lines at each 5 ml from the bottom part up to 15 ml. Its one example is shown in Fig. 13.

Fig. 13. Test Tube (An Example)



10.3 Reagents Reagents for doctor testing method shall be as follows:

- (1) Doctor Solution Dissolve sodium hydroxide 125 g specified in JIS K 8576 in water to make 1 l, after adding lead monoxide 60 g specified in JIS K 8090, mix this by shaking violently for 15 min, or leave as it is for 24 h (during it at times mix by shaking). After standing still incline the supernatant solution or after transferring to a bottle by means of a siphon, stopper tightly to store. When even standing still the supernatant solution does not become transparent, filter with filter paper.

Further, in the case where when using, this solution is not transparent completely, again it shall be filtered.

- (2) Powder Sulfur Pulverize the sulfur specified in JIS K 8088, put into a tightly closed container and store.

10.4 Procedures of Test Procedures of test for doctor testing method shall be as follows:

- (1) Take a sample 10 ml and doctor solution 5 ml into a test tube, and after mixing by shaking violently for 15 s, observe the change in colour ⁽²¹⁾ of doctor solution layer. At this time, in case where the doctor solution layer is changed in colour, don't carry out the operation of (2).

Note ⁽²¹⁾ In the case of changing to black it indicates the existence of hydrogen sulfide and in the case of yellow to brown colour, the existence of mercaptan.

32
K 2276-1989

- (2) In case where change in colour of doctor solution layer is not confirmed, add a so small amount of powder sulfur as to cover the surface of sample and doctor solution layer ⁽²²⁾, after again mixing by violently shaking for 15 s, stand still, and observe the change in colour of doctor solution layer and powder sulfur within 2 min ⁽²³⁾.

Notes ⁽²²⁾ Because if the powder sulfur is added in excess, the coloured precipitate is diluted by sulfur and the judgement may be mistaken, add the powder sulfur to the degree not to precipitate.

⁽²³⁾ In the case where the powder sulfur is changed in colour, it indicates existence of mercaptan.

10.5 Results Results shall be in accordance with Table 8.

Table 8. Results

Change in colour	Result
(1) The case where the doctor solution layer changed in colour.	Cationic (or positive or sour)
(2) The case where the doctor solution layer is not changed in colour and where the powder sulfur changed in colour over the whole surface.	
The case where the doctor solution layer is not changed in colour and the powder sulfur kept bright yellow colour (including the case where the powder sulfur is slightly changed in colour to grey and accompanied with black spots)	Cathodic (or negative or sweet)

10.6 Accuracy The accuracy is not specified.

11. Luminometer Number Testing Method

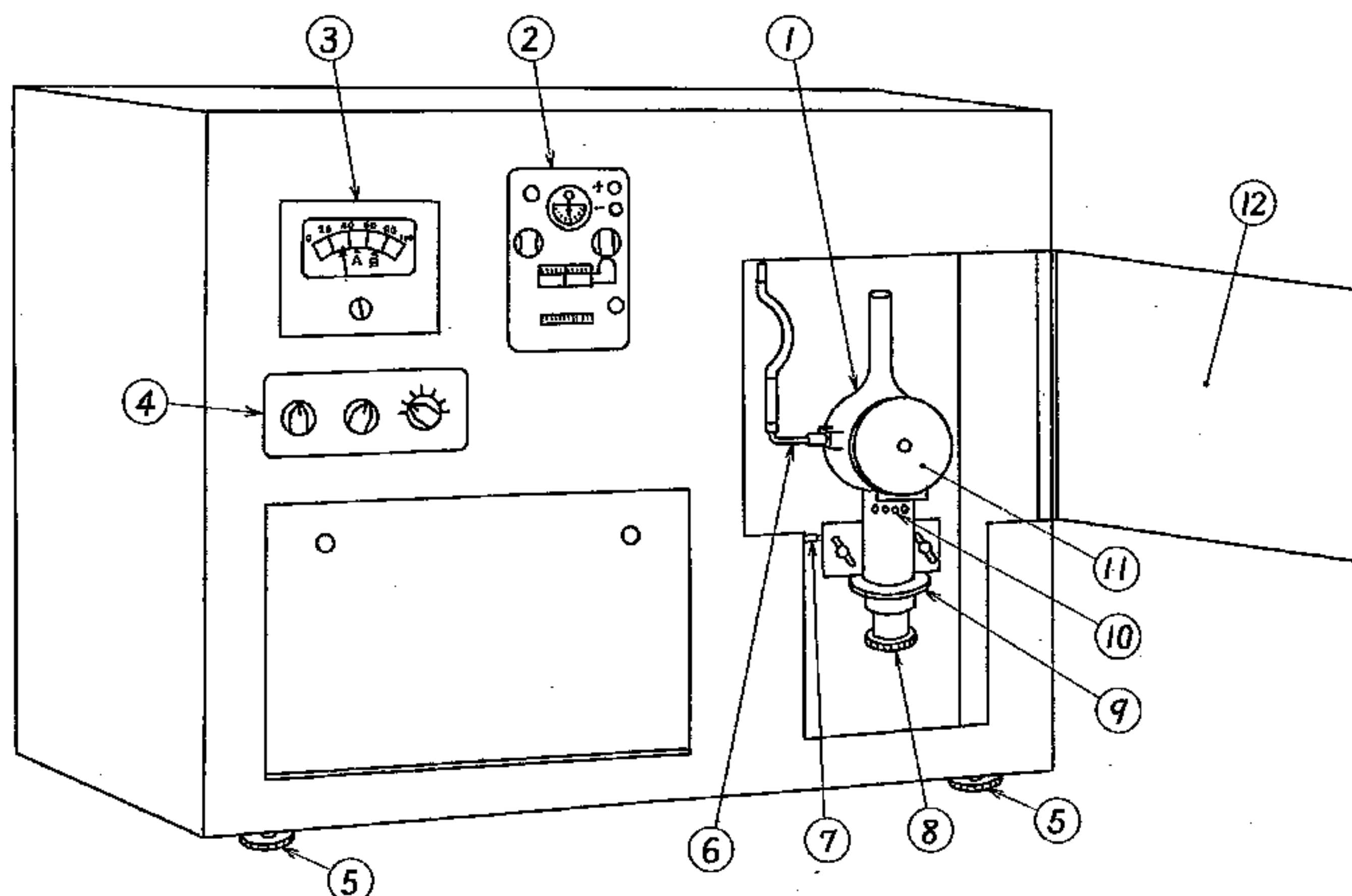
11.1 Summary of Testing Method The luminometer number is obtained by burning the sample in a luminometer lamp and by obtaining the relation between the temperature to be measured with thermocouple set just above the flame and the brilliance of flame to be measured through optical filter by means of a photoelectric tube or that equivalent thereto.

Namely, at first burn the tetralin, record the reading of luminance meter and thermometer at its smoke point and take these as the reference points. Next, burn in the order of isooctane and sample, plot the relation between the respective readings of luminance meter and the thermometer and obtain the reading of thermometer relative to the reading of luminance meter at reference point. Obtain the difference between the sample temperature and tetralin temperature corresponding to reference point by dividing by the difference between the isooctane temperature and tetralin temperature corresponding to reference point.

11.2 Luminometer Number Tester The luminometer number tester shall be as follows:

- (1) Luminometer Number Tester The luminometer number tester is as shown in Fig. 14, consists of a lamp, luminance meter, and thermometer, the system figure of tester is shown in Fig. 15 and detail drawing of luminance meter and thermometer is shown in Fig. 16.

Fig. 14. Luminometer Number Tester (An Example)

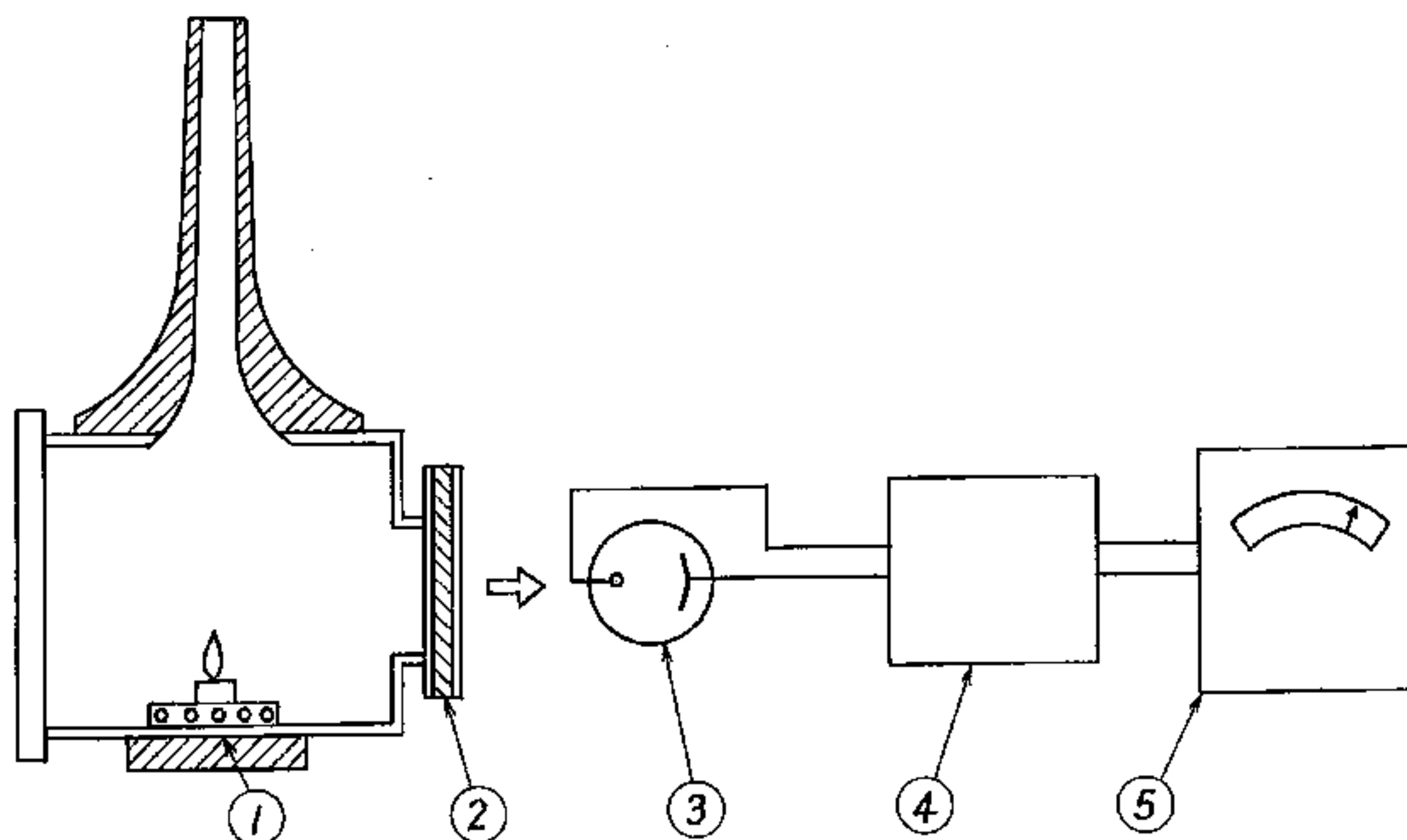


- | | |
|--|--|
| ① Lamp | ⑦ Thermocouple for measurement of temperature of tester inside |
| ② Thermometer | ⑧ Sample container |
| ③ Luminance meter | ⑨ Ring for regulation of height of flame |
| ④ Luminance meter switch board | ⑩ Vent hole |
| ⑤ Horizontal regulating screw | ⑪ Peep hole of lamp |
| ⑥ Thermocouple for measurement of lamp temperature | ⑫ Door |

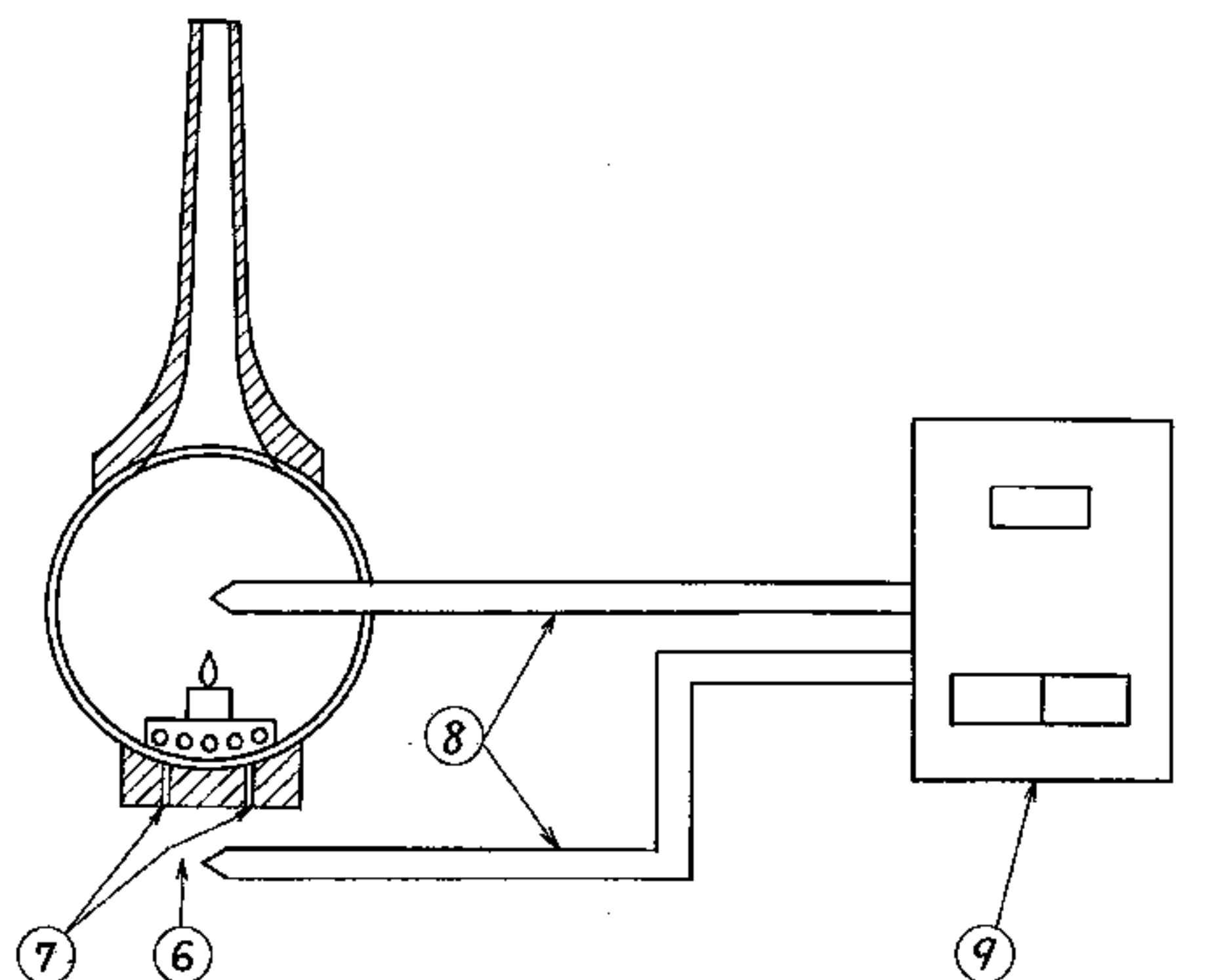
Informative Reference: This tester is made of Erdco Engineering Co. in U.S.A. recognized by ASTM and CRC.

Fig. 15. System Figure of Luminometer Number Tester
(An Example)

Luminance Measurement



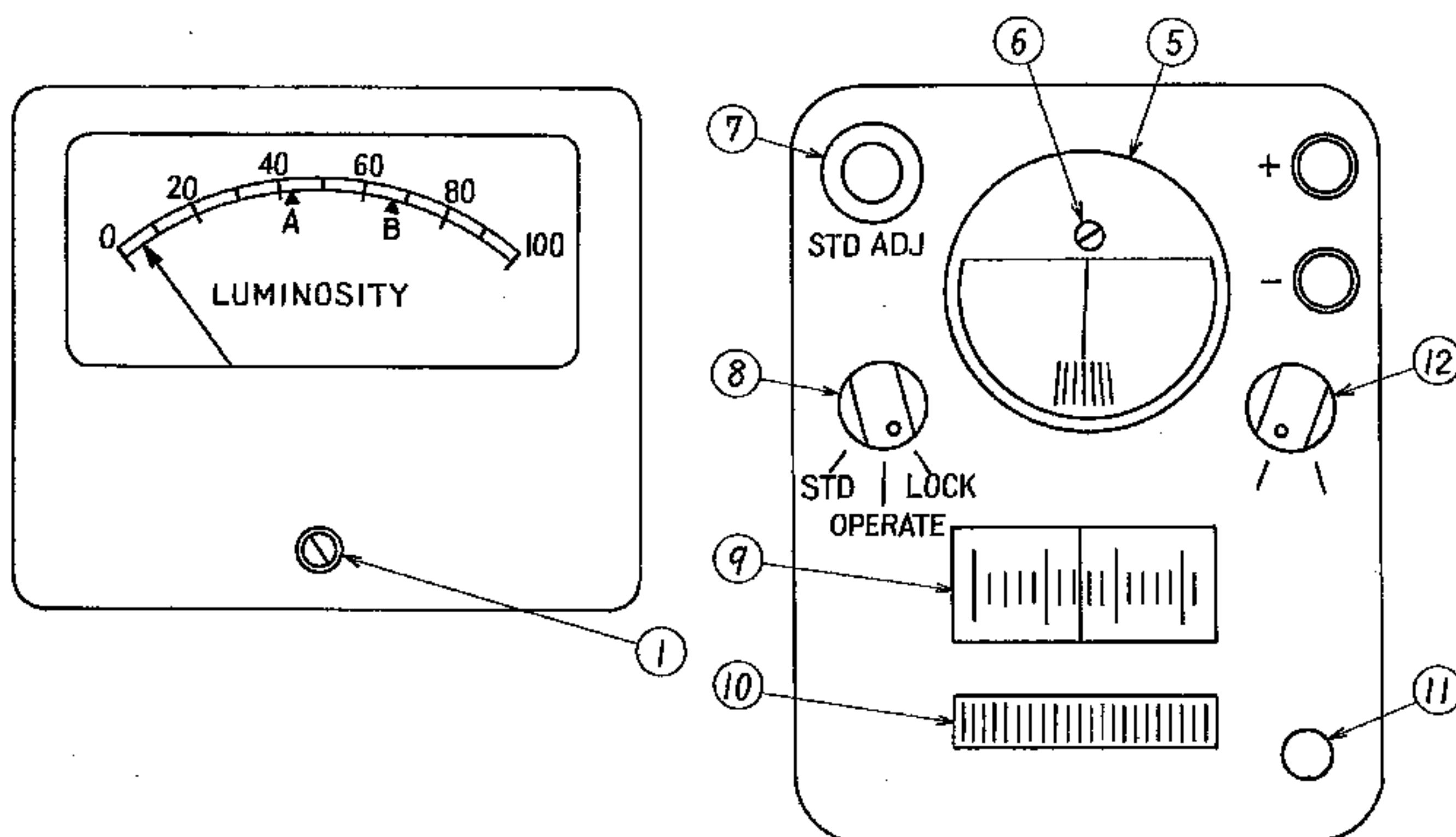
Temperature Measurement



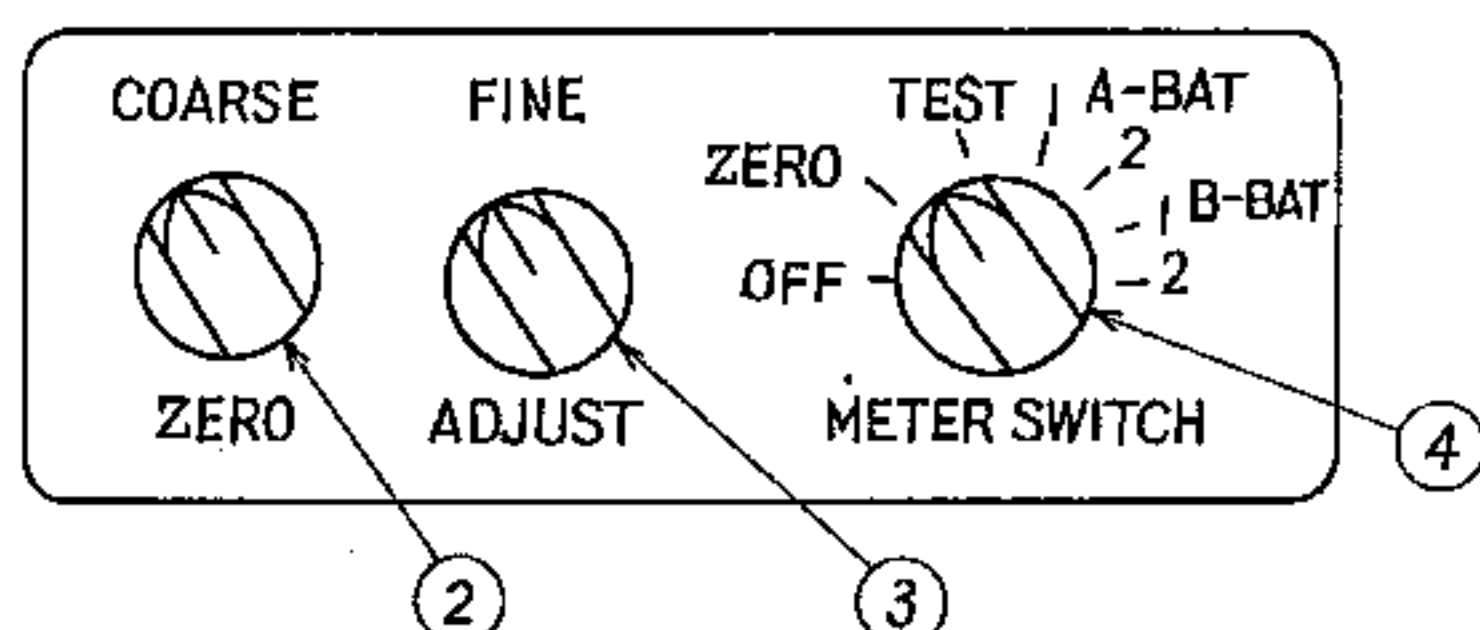
- ① Lamp (side face)
- ② Orange filter (480 to 700 nm)
- ③ Photoelectric tube or that equivalent
- ④ Amplifier
- ⑤ Luminance meter

- ⑥ Lamp (front)
- ⑦ Vent hole
- ⑧ Thermocouple
- ⑨ Thermometer

Fig. 16. Luminance Meter and Thermometer (An Example)
(Luminance Meter) (Thermometer)



(Luminance Meter Switch Board)



- ① Screw for regulating luminance meter pointer
- ② Knob for rough regulating zero point of luminance meter
- ③ Knob for fine regulating zero point of luminance meter
- ④ Luminance meter switch
- ⑤ Galvanometer

- ⑥ Screw for regulating galvanometer zero point
- ⑦ STD regulating knob
- ⑧ Thermometer switch
- ⑨ Temperature scale plate
- ⑩ Dial for temperature measurement
- ⑪ Test button
- ⑫ Change-over switch

(2) Sample Container and Wick The sample container and wick specified in JIS K 2537.

36
K 2276-1989

11.3 Reagents Reagents for luminometer number testing method shall be as follows:

- (1) Isooctane Isooctane specified in JIS K 2280.
- (2) Tetralin Tetralin of property shown in Table 9.

Table 9. Properties of Tetralin

Item		Specified value
Density g/cm ³ (25°C)		0.961 to 0.966
Refractive index n_D^{20}		1.5409 to 1.5417
Dis- tillation property	Initial boiling point °C	200 min.
	Dry point °C	210 max.

- (3) Acetone Acetone specified in JIS K 8034.
- (4) Methanol Methanol specified in JIS K 8891.
- (5) Toluene Toluene specified in JIS K 8680.

11.4 Preparation of Tester The preparation of tester for luminometer number testing method shall be as follows:

- (1) Washing of Lamp For each test, wash the upper most part and inside of wick tube guide with acetone by using a small brush with taking care ⁽²⁴⁾ so as not to attach stains to optical filter. At that time, preliminarily protect the optical filter by a thin cloth or wash with detaching the optical filter.

Note ⁽²⁴⁾ In case where blots are attached to the optical filter, clean by using lens-polishing cloth or the like.

- (2) Preparation of Wick and Sample Container

- (a) At each test wash the wick by using mixed solution of equal amount of toluene and methanol by performing reflux and extraction not less than 25 times with Soxhlet extractor, thereafter leave as it is in air for not less than 5 min to dry, further dry in a drier at 100 to 110°C for 30 min ⁽²⁵⁾, and store in a desiccator.

Note ⁽²⁵⁾ If drying in air is not sufficient, there is a fear to explosion in the drier. Therefore carry out drying in air sufficiently or use ventilation drier of explosion-preventive type to prevent explosion.

- (b) Wash the sample container sufficiently with acetone to dry ⁽²⁶⁾.

Note ⁽²⁶⁾ If drying is insufficient and sample or acetone remains, the test result is influenced.

- (c) Insert a washed and dried wick into the wick tube of cleaned sample container, raise the wick so as to extrude out by 6.5 mm from its upper end, and thereafter revolve its top end to correct the twist of wick.
- (d) Cut the wick to arrange so that the wick goes out horizontally by 6.5 mm from the upper end of wick tube by using a clean, sharp blade ⁽²⁷⁾. At this time, if the wick is soaked with isooctane, tetralin or sample, it becomes easy to arrange by cutting.

Note ⁽²⁷⁾ Depending upon the blade, it is coated with rust-preventive agent and therefore it shall be used with washing off by using detergent.

(3) Regulation of Lamp

- (a) In order to make axial line of flame vertical, place a level on a ring for regulating flame height, and carry out level regulation by means of a horizontal regulating screw.
- (b) Confirm that the protective tube including the temperature measuring contact point of thermocouple for lamp-temperature measurement is clean.
- (c) Set the temperature measuring contact point of thermocouple for lamp temperature measurement correctly on the central line of lamp. At this time, the bottom part of thermocouple protective tube becomes 25.4 mm above from wick tube guide ⁽²⁸⁾.

Note ⁽²⁸⁾ In cases where the position of thermocouple is changed or exchanged, these positions shall be confirmed without fail.

(4) Regulation of Thermometer

- (a) Confirm that the thermocouple for temperature measuring of inside of tester and that for lamp temperature are at the respective definite positions and are connected correctly with the thermometer.
- (b) Regulation of mechanical zero point

Make the thermometer switch "OPERATE", turn the screw for galvanometer zero point regulation to align the pointer of galvanometer with 0 graduation ⁽²⁹⁾.

Note ⁽²⁹⁾ In case where regulating of zero point is impossible, exchange the cell (1.5 V) in the thermometer.

- (c) Regulation of electrical zero point

Make the thermometer switch "STD", turn the knob for STD regulation and align the pointer of galvanometer with 0 graduation ⁽²⁹⁾.

(5) Regulation of Luminance Meter

- (a) Make the luminance meter switch "OFF" and by means of screw for luminance meter pointer regulation align its pointer with 0 graduation.
- (b) Change over the luminance meter switch to "ZERO", and by means of the knob for coarse regulation of luminance meter zero point and the knob for micro regulation of luminance meter zero point again align the pointer with 0 graduation.
- (c) Then, confirm that when made the luminance meter switch "A-BAT, 1" and "A-BAT, 2", its pointer exceeds "A" point, and when made "B-BAT, 1" and "B-BAT, 2", its pointer exceed "B" point (³⁰).

Note (³⁰) If it is not exceeded, the voltage is lowered, and therefore the cell (A cell 1.5 V, B cell 22.5 V) shall be exchanged. In case where also B cell is exchanged, the cell (135 V) in the photoelectric cell case also shall be exchanged.

11.5 Procedures of Test The procedures of test for luminometer number testing method shall be as follows:

(1) Stability of Tester

- (a) Take tetralin 20 ml into a clean, dried sample container at room temperature, attach to this the wick tube by screwing-in.
- (b) After tetralin has soaked in up to upper end of wick, attach the sample container to a lamp, and confirm that there no blocking in vent tube of sample container and ignite the wick (³¹).

Note (³¹) Take care so that the flame does not approach within 3 mm from the thermocouple protective tube and the reading of thermometer does not exceed 538°C.

Further, in case where soots adhered on the thermocouple protective tube, extinguish the fire, clean the protective tube and after lowering in little the sample container, ignite again.

- (c) Confirm that it is burning without the flame discharges soots from the peep hole of lamp (³²), and burn as it is at least for 15 min to warm the tester.

Note (³²) Usually, the tester is so regulated that the reading of luminance meter at the position just before a extremely small amount of soots begin to appear from the top end of flame with burning tetralin becomes 45 to 55. If it runs out from this range, regulate the variable resistor of amplifier so as the reading to become 45.

(2) Measuring of Reference Point

- (a) When the tester has been warmed according to performing of operation of (1), make the luminance meter switch "TEST" (³³), after lowering the sample container until the reading of luminance meter becomes approximately 30, again make the luminance meter switch "ZERO", and thermometer switch "STD", and carry out zero regulation respectively.

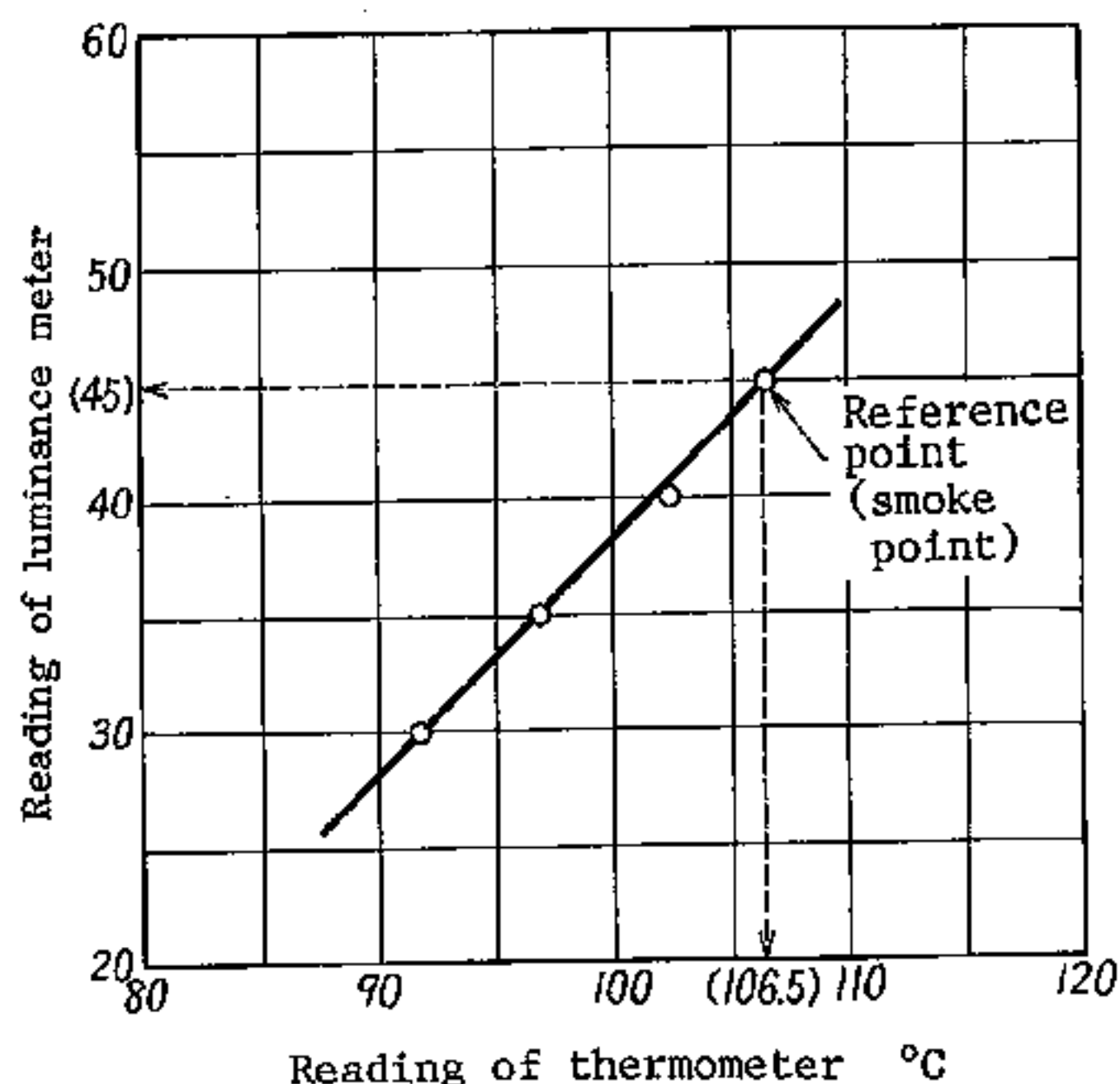
Note (³³) In order to stabilize the luminance meter, leave as it is at least for 30 s after switching-in.

- (b) Again make the luminance meter switch "TEST", and thermometer switch "OPERATE", turn the dial for temperature measurement to align the pointer of galvanometer with 0 graduation.
- (c) Close the door, after maintaining at least for 5 min, turn the dial for temperature measurement to align the pointer of galvanometer with 0 graduation and then respectively record readings of luminance meter and thermometer.
- (d) Then, raise the wick so as the reading of luminance meter rises approximately 5 graduations, after leaving as it is for 5 min, similarly to the before clause record respectively readings of luminance meter and thermometer.

In such a way, operate so as the reading of luminance meter to rise by each approximately 5 graduations and record readings at four points in total. At the last point, raise the wick until the position (smoke point) just before the extremely little soot begins to appear from the top end of flame and measure. At this time, in the case where it is capable of judging clearly that soots adhered on the thermocouple protective tube, lower the wick to stop the measurement, and after cleaning the protective tube, remeasure.

Remark: As to the measured results, as shown in Fig. 17 take the reading of thermometer on abscissa and the reading of luminance meter on ordinate to enter in graph paper, draw a straight line passing through a highest point, and confirm that each point is approximately on this straight line. In case where these are not in linear relation, the warming of tester is insufficient or there is problem in tester or operation, and therefore remeasure.

Fig. 17. Measuring Results of Reference Point by Tetralin (An Example)

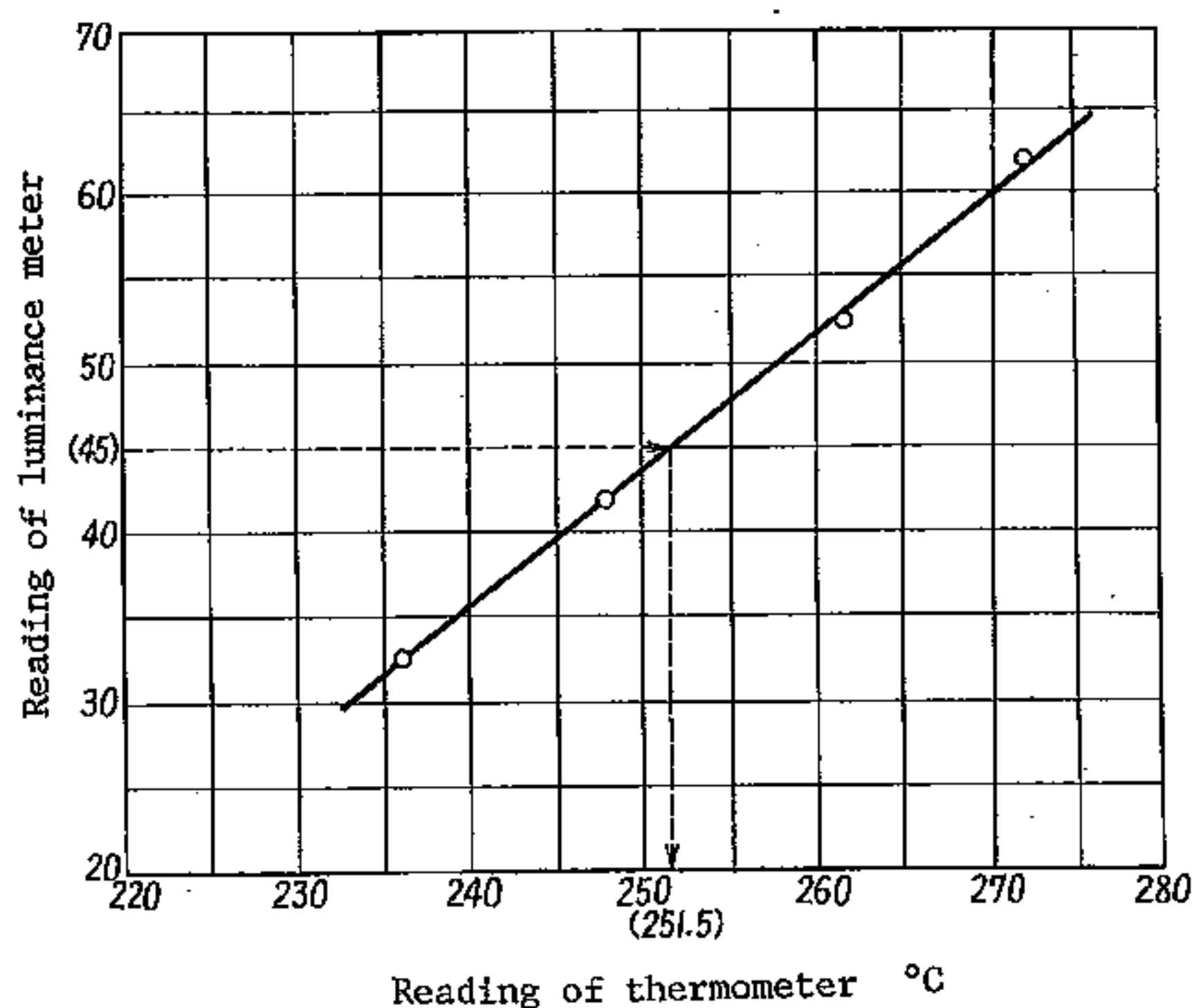


- (e) Repeat the operation of (a) to (d) four times in total. Average the reading of luminance meter and reading of thermometer at smoke point of each time and take it as the reference point.
- (3) Measurement of Isooctane Take isooctane 20 ml into a sample container, carry out the measurement each once before and after measurement of sample according to the operation similar to (1) to (2) ⁽³⁴⁾, and record the measured value. At this time measure at four points in total of each two upper and lower points with taking the reading of luminance meter at reference point obtained by tetralin at the center.

Further, carry out the measurement at each point so as the interval of readings of luminance meter to be apart approximately each 10 graduations (refer to Fig. 18).

Note ⁽³⁴⁾ In the case of sample not less than two, carry out the first measurement before the first sample and the second measurement, after completion of all measurements of the day on the sample.

Fig. 18. Measuring Results of Isooctane
(An Example)



- (4) Measurement of Sample Take sample 20 ml into a sample container, carry out the measurement on total 4 points of upper and lower each two points with placing the reading of luminance meter at reference point obtained by tetralin similarly to (3) at the center, and record the measured value.

Remarks 1. As to the sample high in luminometer number, the generation of condensate water, air leak of lamp, and setting error of reference point give a specially large influence the results.

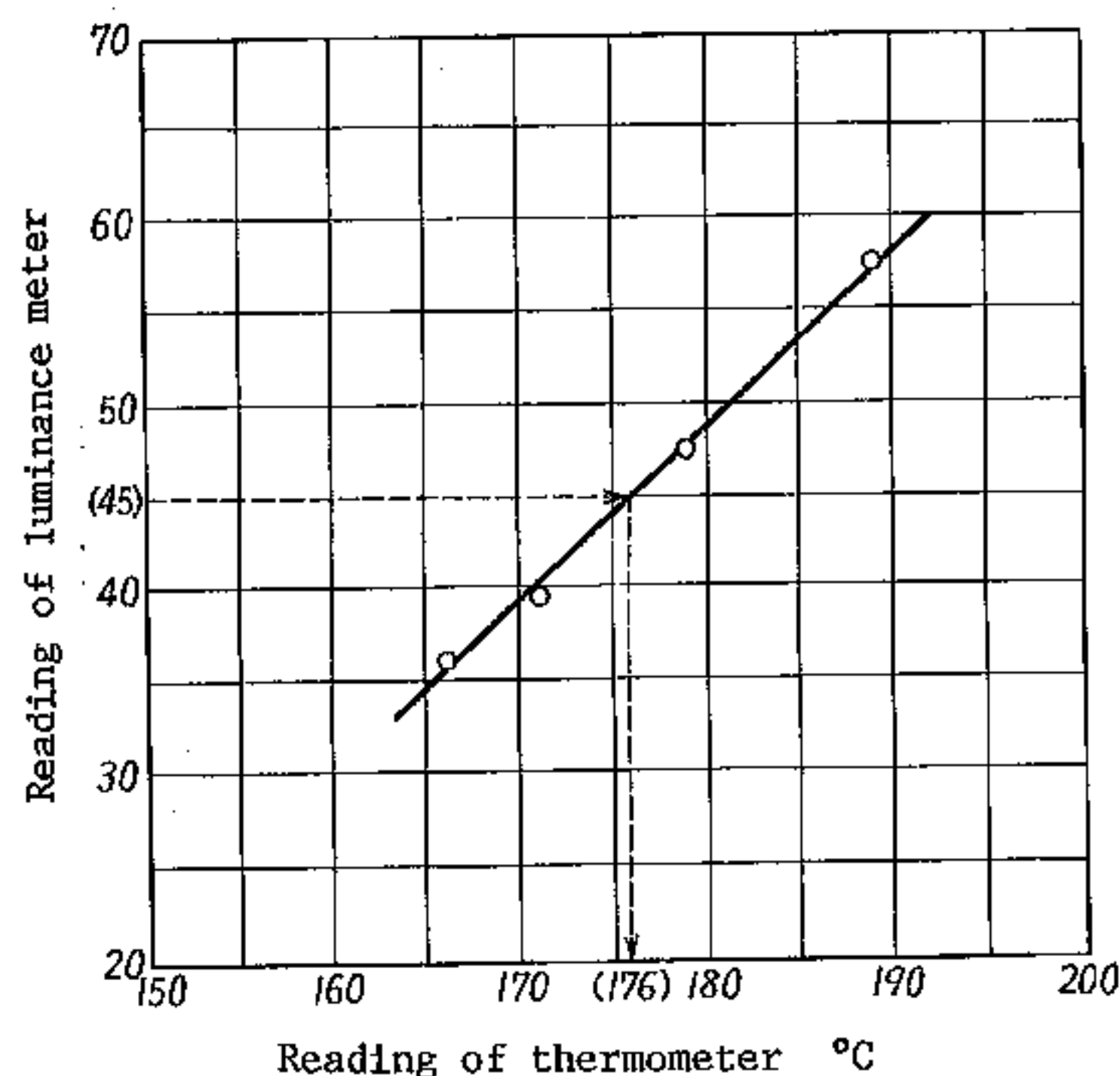
Further, in order to prevent generation of condensate water, the stabilization of tester should preferably be carried out preliminarily. In case if the condensate water is generated, moisture content adheres on the glass of peep hole, adheres on the optical filter and the reading of luminance meter becomes unstable, and therefore open the door of lamp room, and burn the sample to remove.

2. Because the sample low in luminometer number generates soots at comparatively low reading of luminance meter, while confirming the state of flame from the peep hole of lamp, carry out the measurement. In case where soot is generated and burned, stop the measurement and clean the optical filter and thermocouple protective tube.

11.6 Calculation and Results The calculation and results of luminometer number testing method shall be as follows:

- (1) Take readings of thermometer on abscissa, take readings of luminance meter on ordinate, enter the twice measuring values of isooctane obtained in 11.5 (3) on graph paper (refer to Fig. 18), and obtain respective most suitable linear relation. From this Figure read out respectively readings of thermometer corresponding to readings of luminance meter at reference point, obtain their average value, and take this as the isooctane temperature corresponding to the reading of luminance meter at reference point.
- (2) From the relation between the readings of thermometer and the readings of luminance meter obtained in 11.5 (4), similarly to the before clause, obtain the temperature of sample corresponding to the reading of luminance meter at reference point (refer to Fig. 19).

Fig. 19. Measuring Results of Sample
(An Example)



- (3) Calculate the luminometer number according to the following formula and round off to one place of decimal.

$$L.N. = \frac{T_3 - T_1}{T_2 - T_1} \times 100$$

where, $L.N.$: luminometer number

T_1 : temperature at reference point (°C)

T_2 : temperature of isooctane corresponding to reading of luminance meter at reference point (°C)

T_3 : temperature of sample corresponding to reading of luminance meter at reference point (°C)

11.7 Accuracy The accuracy of luminometer number testing method shall be as follows:

- (1) **Repeatability** When the same sample is tested twice in the same testing room by the same person by means of the same tester with changing day or time, the difference of test results shall not exceed the tolerance of Table 10.
- (2) **Reproducibility** The difference between two test results obtained by testing respectively once the same sample in different two testing room by different person by means of different tester shall not exceed the tolerance of Table 10.

Table 10. Accuracy

	Repeatability tolerance	Reproducibility tolerance
Luminometer number	6.1	8.8

12. Naphthalene Content Testing Method (Ultraviolet Spectrophotometry)

12.1 Summary of Testing Method After diluting the sample by diluent for absorption spectral, the absorbance at the wavelength of 285 nm of this solution is measured and the naphthalene content is calculated.

- Remarks 1. This method is a measuring method of naphthalene content of aviation turbine fuels of which end point according to JIS K 2254 is not higher than 315°C and of which naphthalene content is not more than 5 volume %.
2. Though the saturated hydrocarbons, olefins, thiophenes, and alkyl of benzein or cycloalkyl derivative do not interfere the measurement, but phenanthrolines, dibenzothiophenes, biphenyls, benzothiophenes and anthracenes interfere and give errors the measuring value.

12.2 Naphthalene Content Tester (Ultraviolet Spectrophotometry) The naphthalene content tester (ultraviolet spectrophotometry) consists of following (1) to (4):

- (1) **Photoelectric Spectrophotometer or Photoelectric Photometer** The meter of structure specified in JIS K 0115, capable of measuring absorbance at 285 nm.
- (2) **Absorption Cell** The absorption cell made of quartz and of 10.00 ± 0.05 mm in length of liquid layer.
- (3) **Pipette** One mark volumetric pipettes specified in JIS R 3505 of 5 ml and 10 ml.
- (4) **One Mark Volumetric Flask** One mark volumetric flasks specified in JIS R 3505, of 25 ml and 50 ml.

12.3 Reagents Reagents shall be as follows:

Diluent for Absorption Spectra Isooctane or cyclohexane for absorption spectra.

Remark: Instead of isooctane for absorption spectra, the isooctane specified in 3. (1) of Annex 6 may be used, by refining as follows:

Pass isooctane 4 to 5 l through column filled with silica gel of 76 μ m (bore 50.8 to 76.2 mm, length 600 to 900 mm) and in wavelength zone of 240 to 300 nm when contrasted with water if its transmittance is not less than 90 %, it may be used. Put refined isooctane into a clean glass container with ground stopper to store.

For refining of diluent generally new silica gel is used, but the activated silica gel according to the following treatment may be used.

Pour acetone 500 ml to silica gel used for refining of isooctane, flow it out, then reduce pressure to dry, thereafter spread thinly in a drier at 400°C and heat until it becomes white color. Put the activated silica gel into a tightly closed container to store.

12.4 Procedures of Test Procedures of test shall be as follows, but the handling of absorption cell, washing, regulation of photometer, measurement of absorbance shall be carried out carefully according to JIS K 0115.

(1) Preparation of Sample

- (a) Primary Dilution Liquid Add diluent for absorption spectra 10 to 15 ml into a clean, dried one mark volumetric flask 25 ml, stopper and weigh the mass to the nearest 1 mg. Then, add sample approximately 1 g into the one mark volumetric flask, stopper and weigh again the mass to the nearest 1 mg. Take the difference between the both masses as the sample taking amount. Then fill diluent for absorption spectra up to the marked line and mix by shaking thoroughly.
- (b) Secondary Dilution Liquid After taking the primary dilution liquid 5.0 ml into one mark volumetric flask 50 ml by means of a pipette, fill the diluent for absorption spectra up to the marked line and mix by shaking thoroughly.
- (c) Third Dilution Liquid After taking the secondary dilution liquid 5.0 ml by means of a pipette into one mark volumetric flask 50 ml, fill the diluent for absorption spectra up to the marked line and mix by shaking thoroughly.

Remark: Prepare the sample by diluting so as the absorbance at 285 nm to enter the range of 0.2 to 0.8. As required, use secondary dilution liquid 10 ml by diluting with diluent for absorption spectra to 25 ml.

(2) Measurement of Absorbance

- (a) Measurement of Cell Correction Absorbance Fill the diluent for absorption spectra into absorption cell for sample and absorption cell for contrast, measure the cell correction absorbance at 285 nm and record.
- (b) Measurement of Sample Absorbance Take third dilution liquid into the thoroughly washed, dried absorption cell for sample, immediately apply the cover, thereafter measure ⁽³⁵⁾ the sample absorbance at 285 nm and record.

Note ⁽³⁵⁾ When the absorbance of third dilution liquid is less than 0.2, by using suitably the dilution liquid entering in 0.2 to 0.8 carry out the similar operation.

12.5 Calculation and Result Calculate the naphthalene content according to the following formula, round off to two places of decimals and express by volume % or mass %.

$$N_r = \frac{AK}{33.7M} \times \frac{B}{1.00} \times 100$$

$$N_M = \frac{AK}{33.7M} \times 100$$

where, N_r : naphthalene content (volume %)

N_M : naphthalene content (mass %)

A : correction absorbance (value of sample absorbance subtracted by cell correction absorbance)

K : dilution coefficient (amount of diluent for absorption spectra used for diluting sample 1 g, l)

For example, in the case of primary dilution liquid

$$K=0.025$$

in the case of secondary dilution liquid

$$K=0.25$$

in the case of third dilution liquid

$$K=2.5$$

in the case of dilution liquid of Remark of 12.4 (1) (c)

$$K=0.625$$

M : taken amount of sample (g)

B : density of sample at 15°C (g/cm³)

33.7: average extinction coefficient of C_{10} to C_{11} naphthalene derivatives at wavelength 285 nm ($l/g \cdot cm$)

1.00: average density of C_{10} to C_{11} naphthalene derivatives at 15°C (g/cm^3)

12.6 Accuracy The accuracy of naphthalene content testing method (ultraviolet spectrophotometry) shall be as follows:

- (1) Repeatability When tested twice the same sample in the same testing room by the same person with the same tester with changing the day or time, the difference of test results shall not exceed the tolerance of Table 11.

Table 11. Repeatability

Unit: volume % or mass %	
Naphthalene content	Repeatability tolerance
0.67 or under	0.05
Over 0.67 up to and incl. 3.0	$0.09 x - 0.01$

Remark: Herein, x : average volume of test results

- (2) Reproducibility The difference of two test results obtained by testing respectively once the same sample in different two testing room by different persons with different tester shall not exceed the tolerance of Table 12.

Table 12. Reproducibility

Unit: volume % or mass %	
Naphthalene content	Reproducibility tolerance
0.60 or under	0.11
Over 0.60 up to and incl. 3.0	$0.20 x - 0.01$

Remark: Herein, x : average volume of test results

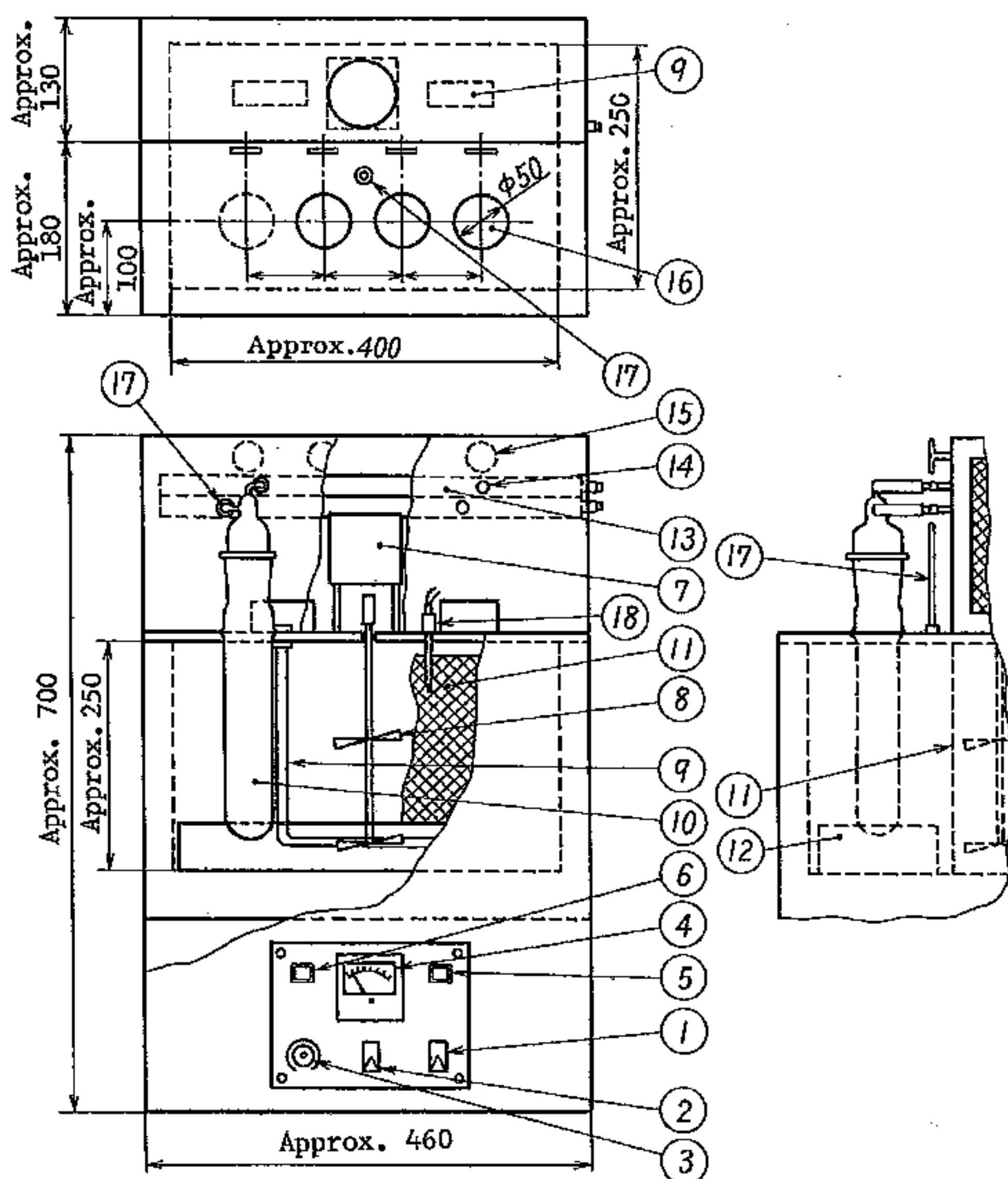
13. Silver Plate Corrosion Testing Method

13.1 Summary of Testing Method Immerse a thoroughly polished silver plate into sample 250 ml completely, after keeping in constant temperature water tank at $50 \pm 1^\circ\text{C}$ for 4 h, take out the silver plate, wash it and then judge the corrosion property of sample according to silver plate corrosion classification table.

13.2 Silver Plate Corrosion Tester The silver plate corrosion tester shall be of structure and dimensions shown in Fig. 20 and, consist of each part of (1) to (5).

Fig. 20. Silver Plate Corrosion Tester (An Example)

Unit: mm



- | | | |
|-------------------------|-------------------------------|--------------------------------------|
| ① Power source switch | ⑦ Electrically-driven stirrer | ⑬ Supply-discharge water branch tube |
| ② Electric motor switch | ⑧ Stirrer vane | ⑭ Supply-water connecting opening |
| ③ Voltage regulator | ⑨ Heater | ⑮ Supply-water adjusting valve |
| ④ Ampere meter | ⑩ Test tube | ⑯ Test-tube inserting opening |
| ⑤ Display lamp | ⑪ Perforated plate | ⑰ Thermometer |
| ⑥ Signal lamp | ⑫ Test-tube receiving stand | ⑱ Sensor for temperature adjusting |

(1) Test Tube The test tube as shown in Fig. 21, which consists of following each part.

- (a) Sample Container A container made of brown hard glass ⁽³⁶⁾ of approximately 350 ml in capacity, attached with a bulge for fixing the inserting depth at a portion of approximately 25 mm from lower part of ground stopper.

Note ⁽³⁶⁾ If it is shielded from light, it may be made of transparent glass.

- (b) Cooler A cooler made of hard glass, attached with a hook to suspend silver plate holder, and provided with supply exhaust opening of cooling water at upper part.

Fig. 21. Test Tube Unit: mm

