

N E S	Brake Fluid, Non-Petroleum Base – Automotive
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Applicable and Reference Standards: See page 45.

Table 2 (Continued)

Designation			NR 3	NR 4
Item				
Metal corrosion (at 100±2°C after 120 hrs.)	Metal test pieces	Appearance	No visible pitting or roughness excluding stains and slight discoloration	
		Weight change, mg/cm ²	Tin plate	± 0.2 max.
			Steel	± 0.2 max.
			Cast aluminium	± 0.1 max.
			Cast iron	± 0.2 max.
			Brass	± 0.2 max.
			Copper	± 0.2 max.
			Zinc	± 0.2 max.
	Fluid properties after test	Appearance	(1) No crystalline deposit (¹) should form or adhere to either the wall of glass jar or the surface of metal strips. (2) No jelling at 23±5°C	
		pH	7.0 to 11.5	
		Sedimentation by centrifuge, % (volume)	0.1 max.	
	Rubber cup condition	Appearance	(1) Tack free (2) No rubber degradation	
		Hardness decrease, Hs or IRHD	0 to -15	
		Base diameter increase (mm)	0 to 1.4	
		Base diameter increase rate, %	0 to 4.4	
		Volume increase rate, %	0 to 16	
Low temperature fluidity	(at -40±2°C after 144 hrs.)	Identification of hiding chart	Boundary line identifiable	
		Appearance	No stratification and precipitate	
		Bubble flow time, s	10 max.	
	(at -50±2°C after 6 hrs.)	Identification of hiding chart	Boundary line identifiable	
		Appearance	No stratification and precipitate	
		Bubble flow time, s	35 max.	
Evaporation (at 100±2°C)	Evaporation loss, %		80 max.	
	Residue properties	Appearance (at 23±5°C)	No precipitate remains that is gritty or abrasive when rubbed with fingertips	
		Low temperature fluidity (at -5±1°C for 60±10 min)	Residue shall pour at a rate of 5 mm or more/5 seconds.	

Table 2 (Continued)

Item		Designation	NR 3	NR 4
Swelling of rubber	SBR cup (at 120±2°C after 70 hrs.)	Appearance	(1) Tack free (2) No rubber degradation	
		Base diameter increase, mm	0.15 to 1.40	
		Base diameter increase rate, %	0.6 to 4.4	
		Volume increase, %	0 to -15	
		Hardness decrease, Hs or IRHD	1 to 16	
		Fluid properties after test	(1) Excluding crystalline structures ⁽¹⁾ , sedimentation shall be 0.3 % (volume) max.	
	EPDM cup (at 120±2°C after 70 hrs.)	Appearance	(1) Tack free (2) No rubber degradation	
		Base diameter increase, mm	To be reported	
		Base diameter increase rate, %	To be reported	
		Volume increase, %	To be reported	
		Hardness decrease, Hs or IRHD	To be reported	
		Fluid properties after test	(1) Excluding crystalline structures ⁽¹⁾ , sedimentation shall be 0.3 % (volume) max.	
	EPDM rubber strip (at 120±2°C after 70 hrs.)	Appearance	(1) Tack free (2) No rubber degradation	
		Hardness decrease, Hs or IRHD	0 to -10	
		Volume increase, %	0 to -10	
		Fluid properties after test	(1) Excluding crystalline structures ⁽¹⁾ , sedimentation shall be 0.3 % (volume) max.	

Figure 1 Boiling point test apparatus

Unit: mm

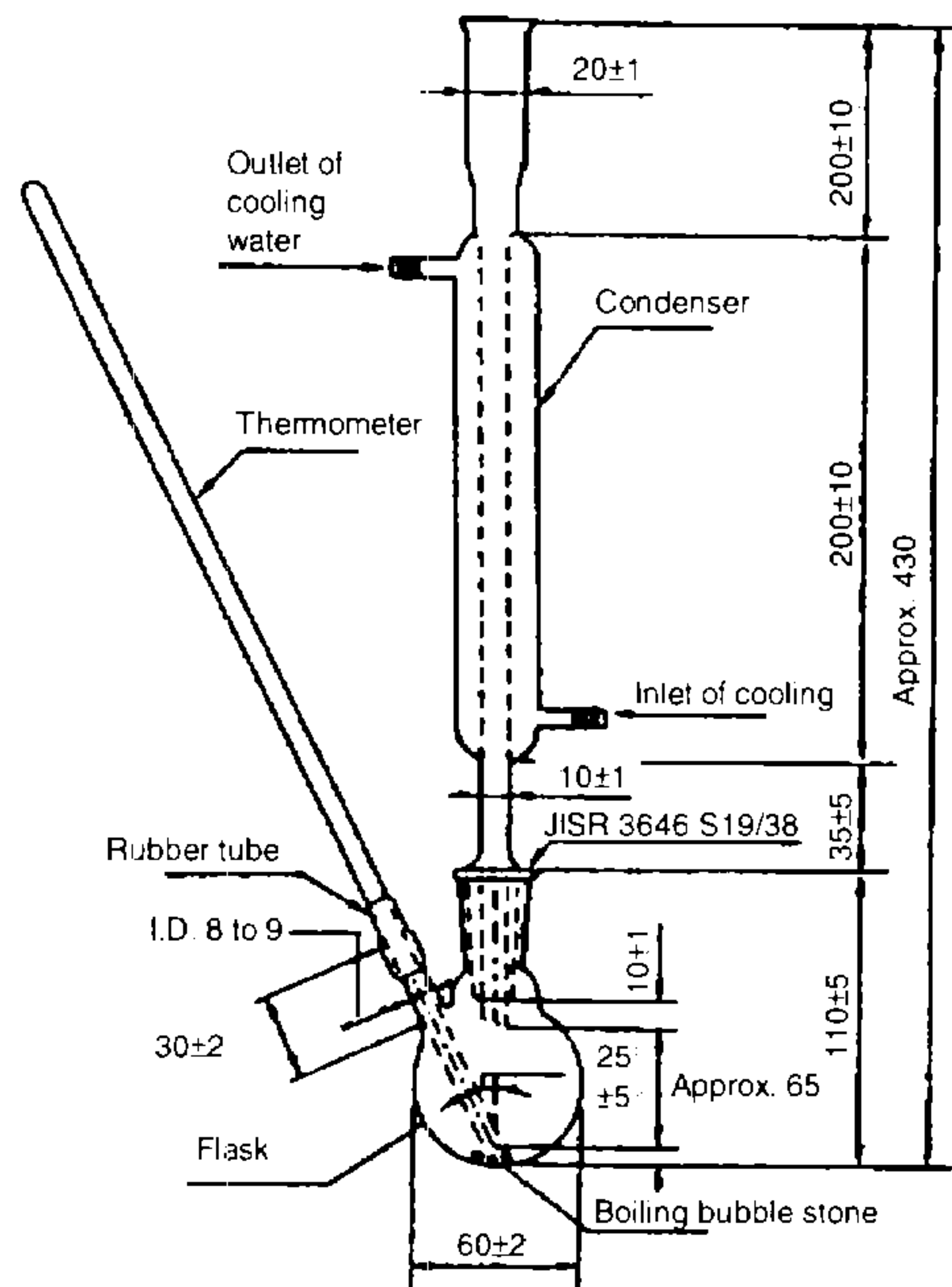
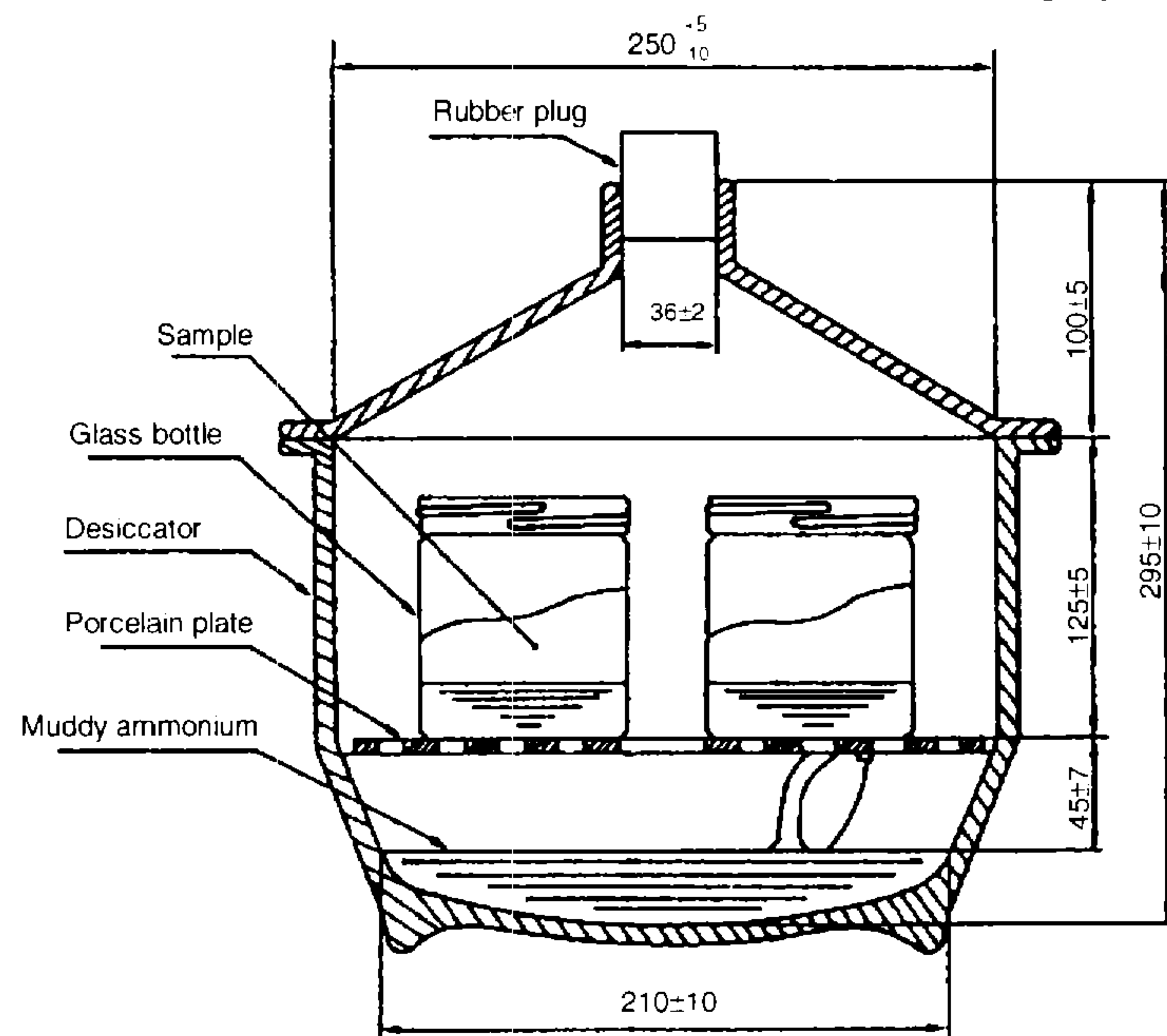


Table 3 Thermometer

Class	Name	For grease dropping point
	Symbol of thermometer	DP
	Thermometer No.	38
Graduation range (°C)		−5 to +300
Test temperature (°C)		–
Immersion (mm) (A)		76
Graduation	Graduation (°C)	1
	Long graduation line (°C)	Every 5
	Graduation figure (°C)	Every 10
	Error of graduation (°C)	1 max.
Expansion chamber	Allowable heating temperature (°C)	400
Shape of top		Circular
Total length (mm) (B)		390 ± 5
Diameter (mm) (C)		6.0 to 7.0
Bulb	Length (mm) (D)	10 to 15
	Diameter (mm) (E)	5.0 to 6.0
Position of graduation	Distance between lower end of bulb and designated graduation line (mm) (F)	100 to 110 (to 0°C)
	Distance between lower end of bulb and designated graduation line (mm) (G)	329 to 358 (to 300°C)

Figure 2 Desiccator

Unit: mm



(2) Reagent

- 1) Triethelene glycol monomethyl ether (hereinafter referred to as "TEGME") - Commercial products with its moisture adjusted to 0.50 ± 0.05 %.

Reference: TEGME commercially available is of the quality shown in Table

Table 4 Quality of TEGME

Item	Specified value
Appearance	Transparent liquid free from suspended matters
Color (APHA)	100 max.
Purity, %	94 min.
Water, %	0.3 max.
Boiling point, °C	240 min.
Acidity (as acetate), %	0.02 max.

- 2) Grease - Heat resistant grease shall be used.

(3) Preparation for measurement

The preparation for measurement shall be as follows:

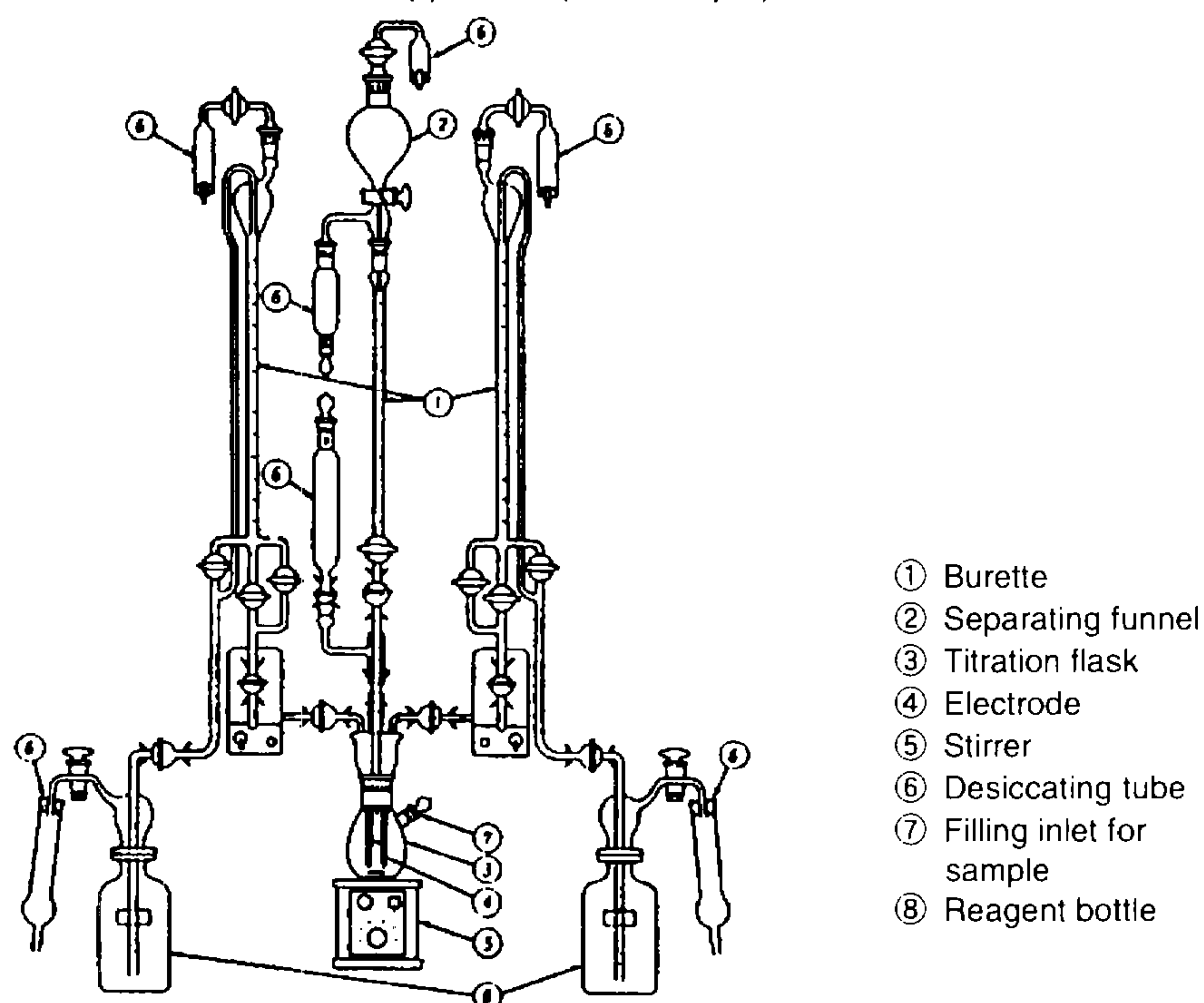
- 1) Heat resistant grease is thinly applied to the contact surfaces of desiccator.
- 2) 450 ± 10 mL of water is placed in each desiccator and the cover placed on it.

(4) Operation

The operation shall be as follows:

- 1) Place 350 ± 5 mL each of the sample in two glass bottles and each uncovered glass bottle will then be placed in each desiccator.
- 2) Place 350 ± 5 mL each of TEGME in two glass bottles and each uncovered glass bottle will then be placed in each desiccator in which the samples are put.
The two glass bottles in the desiccator shall be maintained so that they do not contact each other nor contact the wall of the desiccator.

Figure 3 Volumetric titration apparatus (An example)



- a. Burette - Made of glass tube with the graduation in the unit of 0.02 mL.
- b. Reagent bottle - The reagent bottle shall be made of dark brown colored glass (for Karl Fischer's reagent).
- c. Separating funnel - The separating funnel shall be designed so that a desiccating tube can be connected with the upper part, and a burette and desiccating tube with the lower part (for water- methanol standard solution).
- d. Titration flask - Made of glass with a flat bottom and a filling inlet for sample removal and having a capacity of 250 mL.
- e. Electrode - A platinum electrode.
- f. Stirrer - Capable of stirring at a proper speed such as the "magic stirrer".
- g. Titration end point detector - Magic eye or micrometer.
- h. Desiccating tube

Remark: When an automatic tester is used, its instruction manual shall be followed.

(b) Sampler

The sampler shall be a syringe or a dedicated sampler as shown in Figure 4.

Figure 4 Sampler (an example)

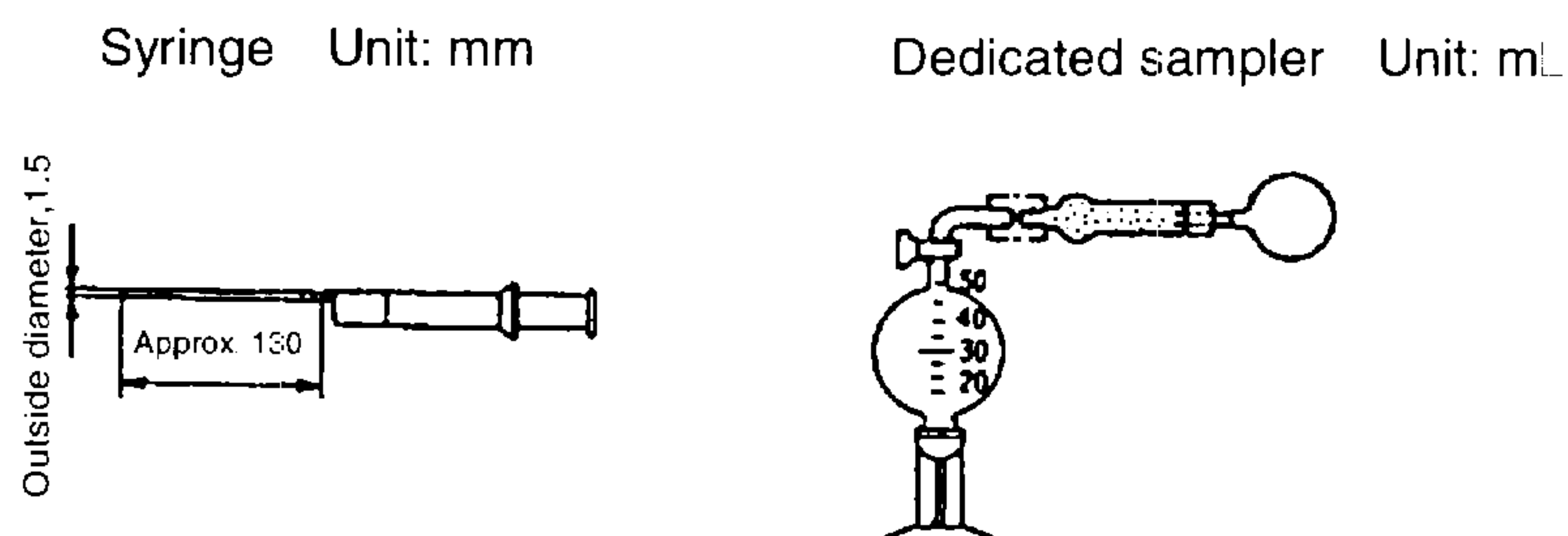
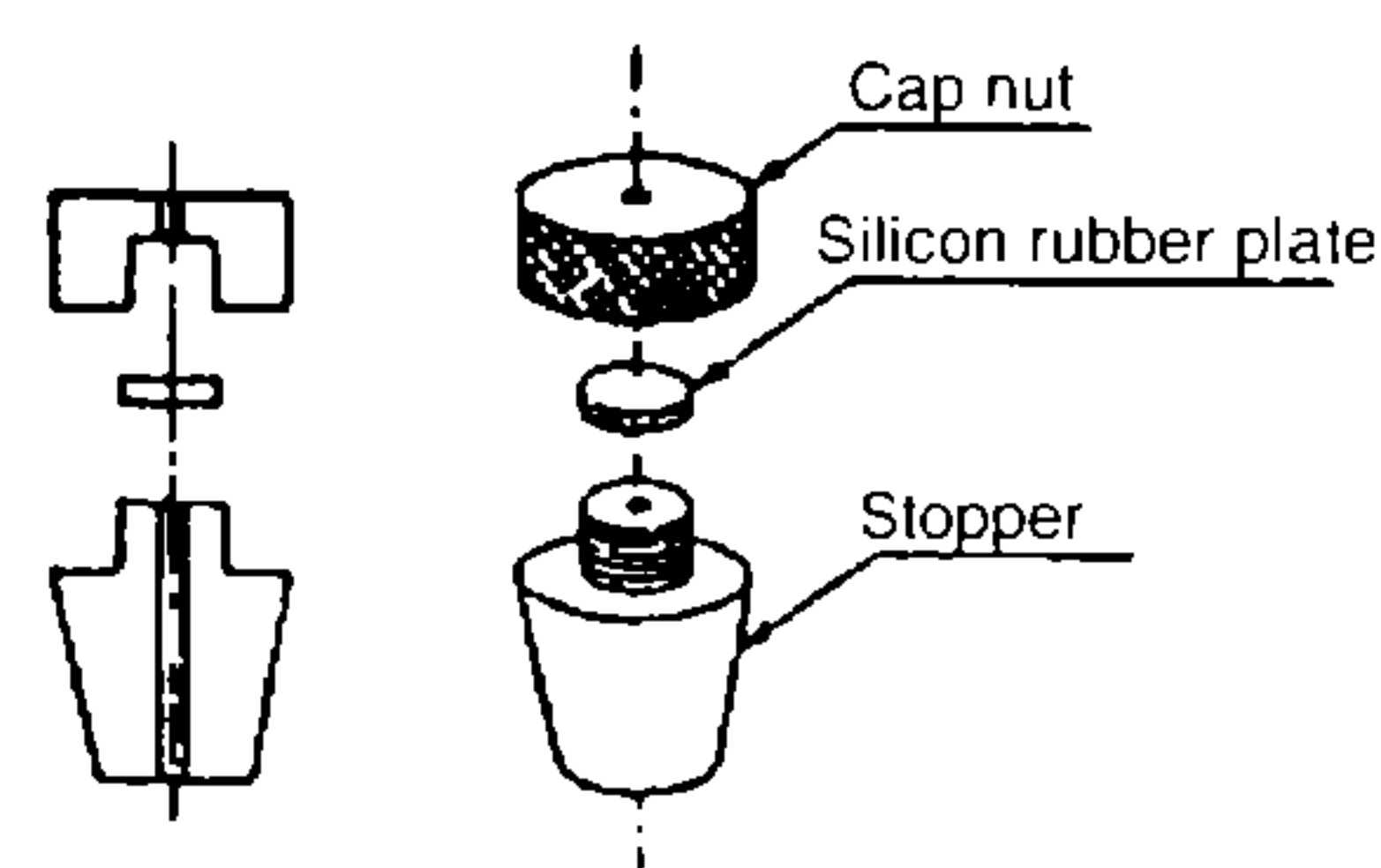


Figure 5 Stopper



5) Method B - For standardization using water-methanol standard solution

- (a) The inside of titration flask shall be made anhydrous in accordance with the method specified in (a) of Method A.
- (b) Drop water-methanol standard solution in the quantity shown in Table 7 from the burette into the titration flask, and read the quantity in units of 0.01 mL.

Table 7 Sampling volume of water-methanol standard solution

Factor, mgH ₂ O/mL	Sampling volume, mL
0.5	2
1.0	4.5

- (c) Next, repeat titration by Karl Fischer's reagent to completion ⁽⁷⁾. Read the quantity of Karl Fischer's reagent required for this titration in units of 0.01 mL.
- (d) The factor of Karl Fischer's reagent shall be calculated down to three digits of the significant figure by the following formula:

$$F = \frac{PA_{12}}{V_2}$$

where F: Factor of Karl Fischer's reagent (mgH₂O/mL)

P: Factor of water-methanol standard solution (mgH₂O/mL)⁽⁹⁾

A₂: Quantity of water-methanol standard solution (mL)

V₂: Quantity of Karl Fischer's reagent required for titration (mL)

Note ⁽⁹⁾: The factor of water-methanol standard solution may change due to the absorption of moisture from the atmosphere while the solution is stored. Therefore, the factor of the solution shall be periodically checked using Karl Fischer's reagent which was standardized by Method A.

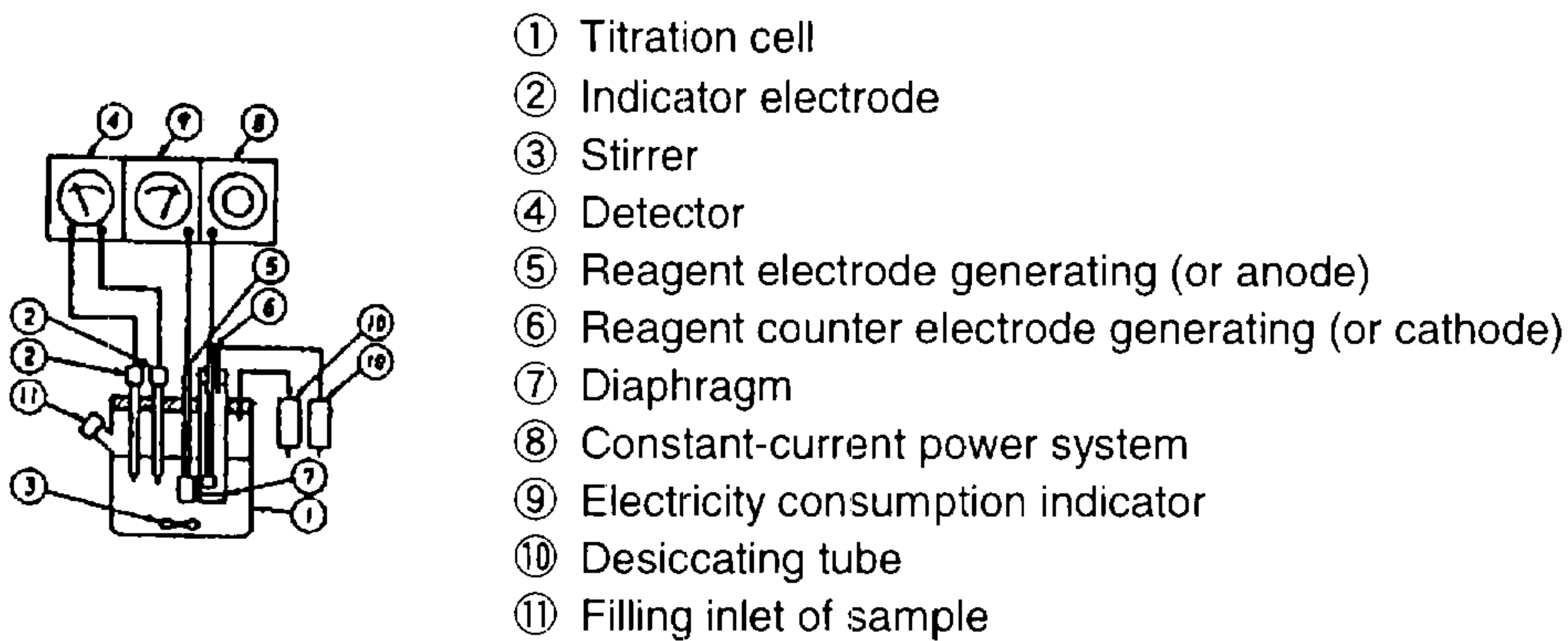
6) Test procedure

- (a) Take 100 mL of the mixed solvent in the titration flask and then cover the flask with the stopper, and operate the stirrer. Then, complete the titration ⁽⁷⁾ with the Karl Fischer's reagent. The inside of the titration flask shall be made anhydrous.
- (b) Take the sample ⁽¹¹⁾ using the sampler ⁽¹⁰⁾ previously dried at approximately 150°C for one hour or more and allow to cool in the desiccator in which a desiccant such as silica gel, etc. is stuffed, in accordance with Table 8. Quickly add to the titration flask.
With respect to the sampling volume, obtain by measuring the mass difference of the sampler before and after the sample is taken in units of 10 mg.

Table 8 Sampling volume of the sample

Factor of Karl Fischer's reagent, mgH ₂ O/mL	0.5	1.0
Water content, mass ppm		
100 max.	Approx. 50 g	—
More than 100, and 250 max.	—	Approx. 40 g
More than 250, and 500 max.	—	Approx. 20 g

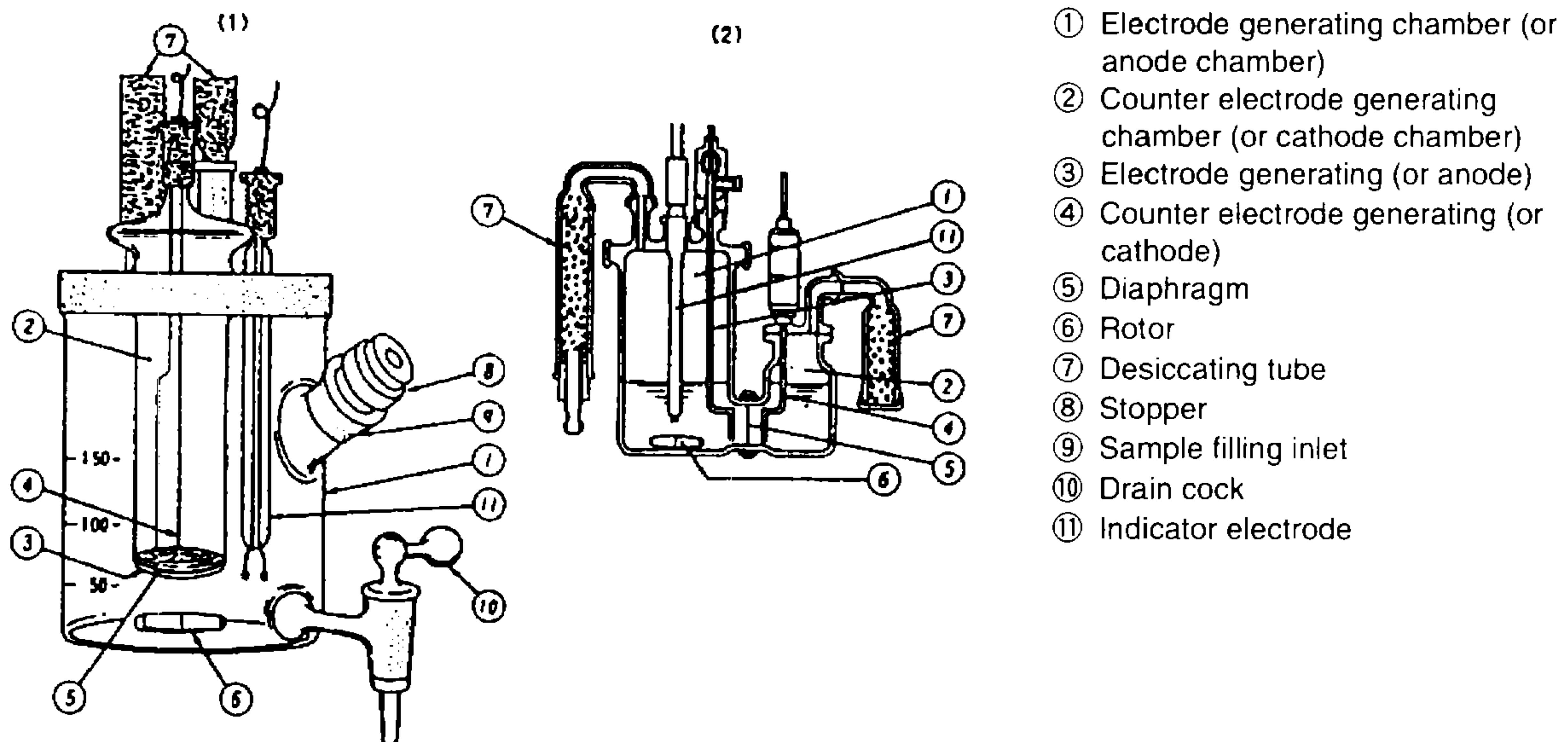
Figure 6 Coulometric titration apparatus (an example)



- a. Titration cell - The titration cell shall be glass in construction and have a capacity of approximately 200 ml, and two chambers, i.e., the reagent electrode generating chamber (or anode) and counter electrode generating chamber (or cathode) which are separated by a diaphragm (ceramic, ion exchange resin membrane, etc.). The cell will have a sample filling inlet with a stopper made of ethylene resin tetra-fluoride or silicon rubber.

Figure 7 Details of titration section (examples)

Remark: The titration cell is equipped with a desiccating tube in which desiccants such as silica gel, activated alumina, aluminum sodium silicate (anhydride), etc., are packed. Sliding couplers will be used for connection of glass ware to prevent moisture absorption from the atmosphere.



- b. Titration completion detector - Able to detect any change of polarization current or voltage between indicator electrodes, when direct/alternate constant voltage or constant current is applied between the indicator electrodes which are immersed in the generator electrode liquid.
- c. Electricity consumption indicator - Capable of indicating the quantity of electricity (C) or water (Pg) consumed.
- d. Constant-current power unit and electrolysis current control unit - For the constant-current power unit, utilize an electronic unit that uses a commercial power source; however, when several mA of electric current is required, use a layer-built cell with which a resistor having a large resistance value is connected. The electrolysis electrical current control unit must be capable of varying electrolytic current according to water content, and include a proportional control circuit.
- e. Reagent electrode generating (or anode) - Platinum electrode
- f. Reagent counter electrode generating (or cathode) - Platinum electrode
- g. Indicator electrode - Platinum electrode
- h. Desiccating tube

Remark: The units shall be handled in accordance with their instruction manual.

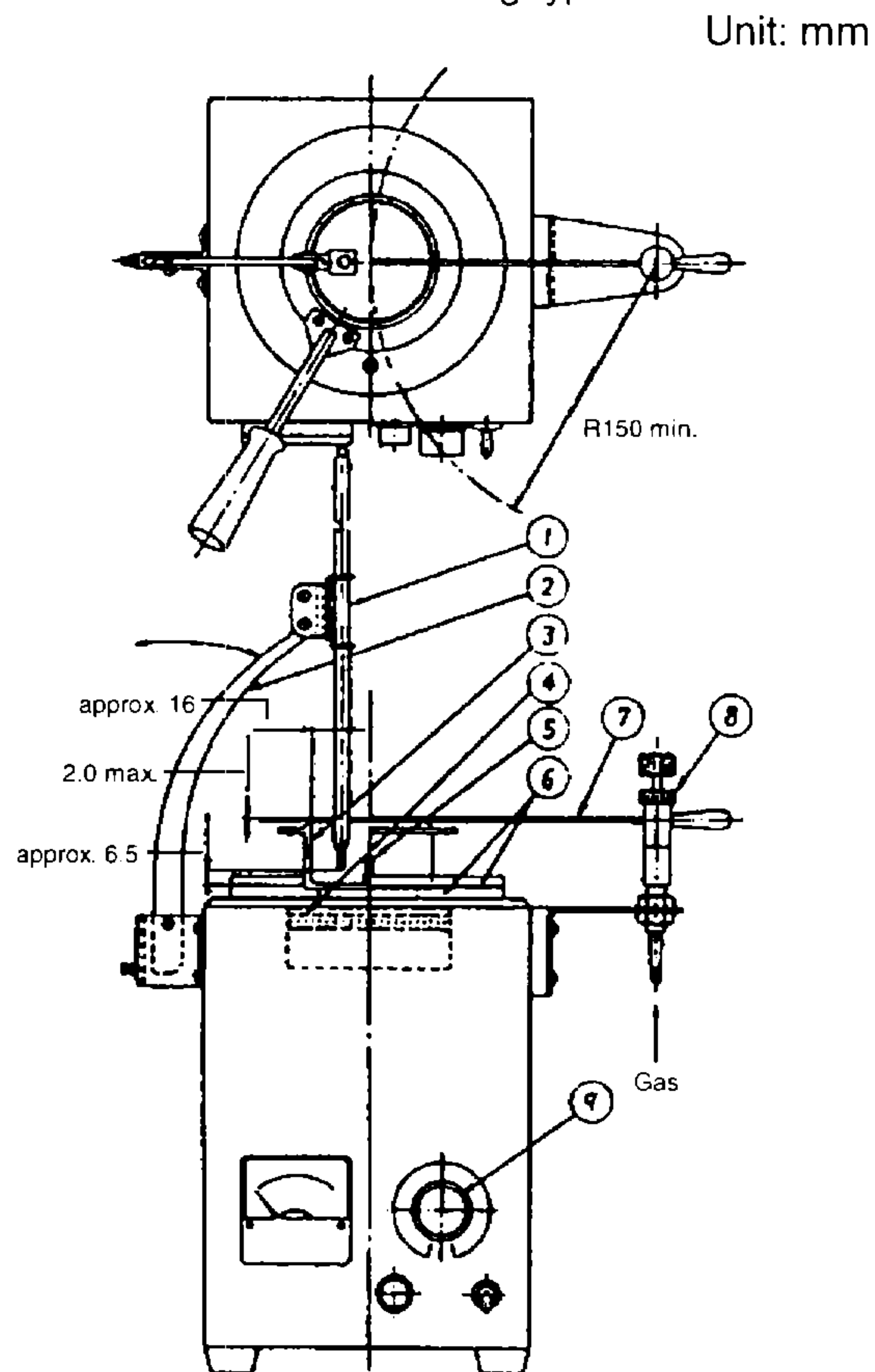
5.3 Flash point

(1) Instrument and apparatus

1) Cleveland Open-cup Flash Tester

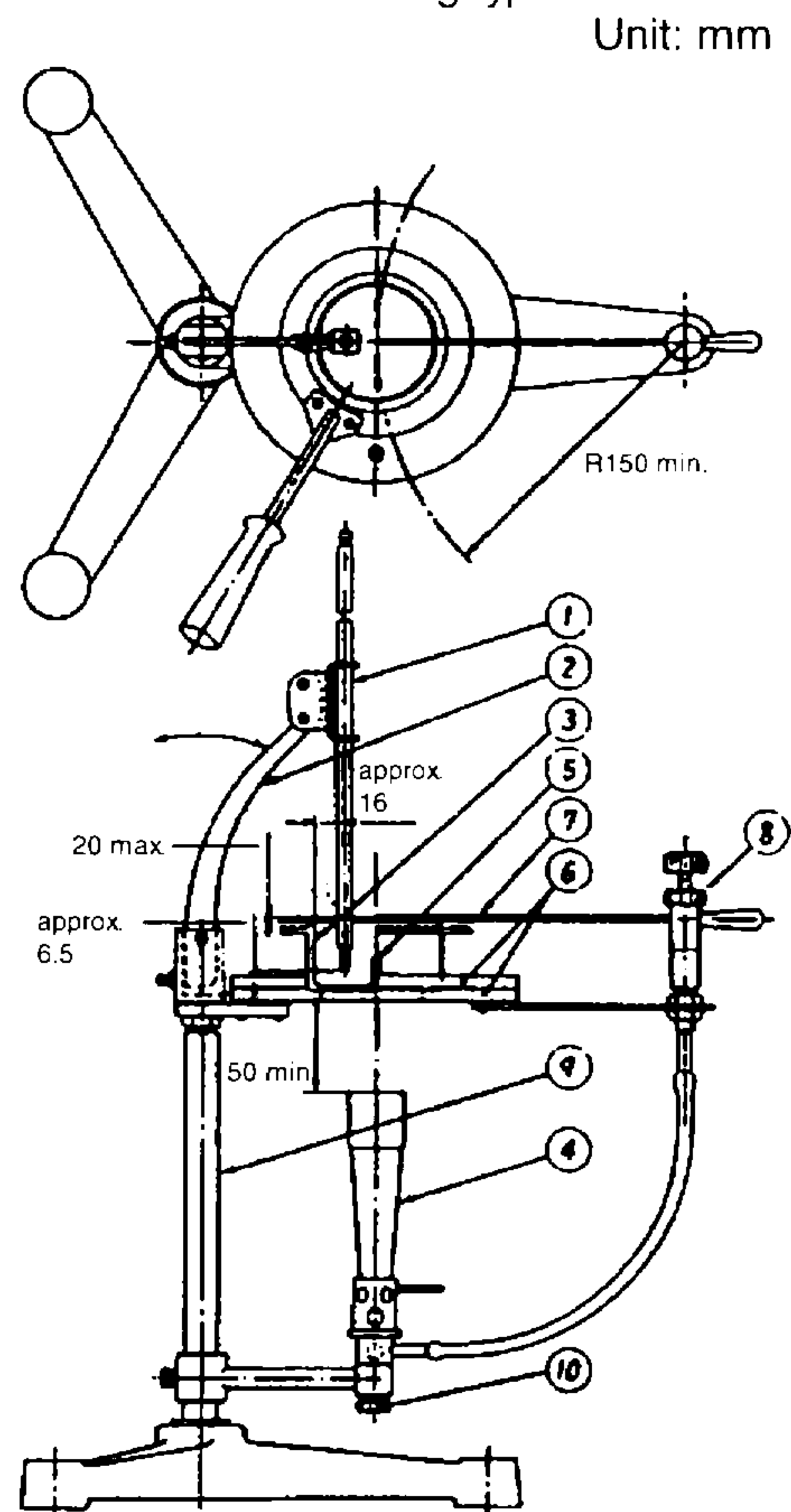
The tester shall have the shape and dimensions shown in Figure 8 and Figure 9 and be composed of the following, a) through g):

Figure 8 Cleveland Open-cup Flash Tester
(an example)
Electric heating type



- | | |
|----------------------|----------------------|
| ① Thermometer | ⑥ Heating plate |
| ② Thermometer holder | ⑦ Test flame nozzle |
| ③ Sample cup | ⑧ Gas control valve |
| ④ Electric heater | ⑨ Heating controller |
| ⑤ Standard tube | |

Figure 9 Cleveland Open-cup Flash Tester
(an example)
Gas heating type



- | | |
|----------------------|-------------------------------|
| ① Thermometer | ⑥ Heating plate |
| ② Thermometer holder | ⑦ Test flame nozzle |
| ③ Sample cup | ⑧ Gas regulating valve |
| ④ Gas burner | ⑨ Heating plate holder |
| ⑤ Standard tube | ⑩ Gas burner regulating valve |

a) Sample cup

The sample cup shall have the shape and dimensions shown in Figure 10 and be made of brass or copper alloy having the coefficient of thermal conductivity equivalent thereto. A proper handle shall be fitted to the flange of the sample cup.

b) Heating plate

The heating plate will be a round metallic plate of 145 to 160 mm in diameter or a square plate having sides measuring 145 to 160 mm as shown in Figure 11 α and η , and a hard asbestos plate covering the whole surface of the metallic plate.

- e) Thermometer
Conform to No. 32 of thermometer (COC) specified in JIS B 7410.
- f) Thermometer holder
During the test, the holder shall be capable of holding the thermometer vertically in the position shown in Figure 8 and Figure 9, and will be easily removed before and after the test.
- g) Heating plate holder
The holder shall be a suitable unit, capable of holding the heating plate horizontally.

(2) Preparation

- 1) Place the test equipment on a stable, horizontal stand in a room free of wind and strong sunlight.
- 2) Wash the sample cup with a suitable solvent. When gummy deposits or oxidized films are observed on the inside of the sample cup, thoroughly clean the sample cup with steel wool until the metal surface is exposed.
- 3) The thermometer shall be held near its middle and the inside wall of the sample cup and the lower end of the mercury bulb shall be positioned 6.5 mm above the inner bottom surface.

(3) Operation

- 1) Fill the sample cup with the sample ⁽¹⁵⁾ up to the marked line and remove air bubbles on the sample's surface. When excess sample is taken in the sample cup, it shall be removed by a pipette or other suitable method to maintain it at the level of the marked line. If the sample sticks on the outside of the sample cup, clean up the sample cup and take the sample in the sample cup.

Note ⁽¹⁵⁾: Before the sample is taken in the sample cup, a sample having high viscosity may be heated until it becomes more fluid. However, do not exceed the temperature which is 55°C below the anticipated flash point of the sample.

- 2) Ignite the test flame and match the flame's size with the standard tube or adjust it so that its diameter is 4 ± 0.8 mm.
- 3) Heat the sample at a rate of 14 to 17°C per minute, and when the temperature reaches 55°C below the anticipated flash point, adjust the heat so that the sample's temperature is raised at the rate of 5.5 ± 0.5 °C per minute until 28°C below the anticipated flash point.
- 4) When the mercury is at 28°C below the anticipated flash point, pass the test flame for every 2°C increase in temperature ⁽¹⁶⁾ across the center of the sample cup, for a second or so, at a right angle to the diameter passing through the thermometer or drawing an arc with the radius of 150 mm or more. The center of the test flame must be moved horizontally 2 mm or less above the upper brim of the sample cup.

Note ⁽¹⁶⁾: After the mercury rises to the point of 17°C below the anticipated flash point, do not breathe into the sample cup.

- 5) When conspicuous ignition ⁽¹⁷⁾ is observed on the surface of the sample, the thermometer reading shall be recorded.

Note ⁽¹⁷⁾: Do not mistake a waxy flame, which may appear around the test flame, as ignition.

Remark: For measuring the ignition point, after the measurement of the flash point, continue to heat at the rate of 5.5 ± 0.5 °C per minute, and move the test flame every 2°C increase. When the sample continues to burn for at least 5 seconds, record the first reading of the thermometer.

(4) Correction for the atmosphere

When the atmosphere at the time of test is 95.32 kPa {715 mmHg} or less, add the correction value shown in Table 10 to the measured value.

Table 10 Atmosphere correction

Atmosphere kPa {mmHg}	Correction value, °C
95.32 to 84.66 {715 to 635}	2.8
84.53 to 73.33 {634 to 550}	5.5

- 2) Thermometer - Specified in JIS B 7410 (Liquid-in-Glass Thermometer for Testing of Petroleum Products), and the number and temperature range of each thermometer shall be as shown in Table 12.

Table 12 Thermometer for Kinematic viscosimeter

Thermometer's number	Temperature range, °C	Fractionalized graduation, °C	Immersion
14	-51.5 to -33.5	0.1	Total immersion
15	-19.2 to -16.4	0.05	Total immersion
16	18.5 to 21.5	0.05	Total immersion
17	23.5 to 27.5	0.05	Total immersion
18	28.5 to 31.5	0.05	Total immersion
19	36.5 to 39.5	0.05	Total immersion
20	48.5 to 51.5	0.05	Total immersion
21	53.5 to 55.5	0.05	Total immersion
22	73.5 to 76.5	0.05	Total immersion
23	78.5 to 81.5	0.05	Total immersion
24	97.5 to 101.5	0.05	Total immersion
88	38.5 to 41.5	0.05	Total immersion

3) Thermostatic chamber

The thermostatic chamber shall have a structure as shown in Figure 13. With respect to constant temperature performance, for the test temperature of 15 to 100°C, fill the bath with water and maintain at a constant temperature of 70°C, then insert a Bechmann's thermometer (using the thermometer's supporting hole) and monitor to maintain a constant temperature within the range of $\pm 0.01^\circ\text{C}$ at 50 mm or deeper below the liquid's surface. For test temperatures outside the range of 15 to 100°C, a suitable bath liquid shall be used, and a constant temperature shall be maintained in the range of $\pm 0.03^\circ\text{C}$. When the measurement is made with a 500V insulation resistance tester, the insulation resistance between the charged part and chamber frame must be 5 M-ohms or more. However, if an electric heater circuit is included, the resistance may be 0.5 M-ohms or more.

- Bath - The bath container shall be of glass or metal construction and be large enough that the viscosimeter can be sufficiently immersed, and at the time of measurement all parts of the sample are covered by the bath liquid at the height of 20 mm or greater. The container shall also be constructed so that the viscosimeter and thermometer can be seen from outside.
- Bath cover - The bath cover will be of metal or synthetic resin construction which can accommodate the viscosimeter holder. It will be equipped with an insertion hole for the thermometer and temperature controller.
- Viscosimeter holder - The viscosimeter holder will be constructed as shown in Figure 14 which can hold the viscosimeter vertically, and be constructed of metal or synthetic resin laminated plate.
- Stirrer - The stirrer shall be a motor driven type of 100 W or more, have minimal vibration, and be capable of uniformly distributing the bath temperature.
- Electric heater and temperature controller - The electric heater shall be capable of raising the bath temperature from the room temperature to approximately 100°C within 100 minutes. The temperature controller will maintain the bath temperature within the specified accuracy.

4) Sand glass

Stop watch or electrical timer having the graduation of 0.2 seconds or less. Instrument error of the sand glass shall be within $\pm 0.05\%$ in a test for fifteen minute test.

5) Moisture proofing unit

The moisture proofing unit is of glass construction and used to test at dewpoint temperature or lower. Figure 15 shows a shape and an example of usage.

(2) Preparation

1) Viscosimeter calibration

The viscosimeter calibration shall be in accordance with (a) or (b) below.

(a) Method for viscosimeter correction by standard liquid

The standard liquid for use in viscosimeter calibration (hereinafter referred to as "standard liquid") specified in JIS Z 8809 (Standard Liquids for Calibrating Viscosimeters) will be used. For this, use the standard liquid having an outflow time of 200 seconds or more, at calibration temperature (40°C is desirable). However, for viscosimeters listed in Table 10, use the standard liquid with the minimum outflow time or as specified shall be selected.

Record the outflow time to at least the nearest 0.1 seconds in accordance with 5.4 (3), and calculate the viscosimeter constant in four digits of the significant figure using the following formula:

$$C = \frac{v}{t}$$

where C: Viscosimeter constant (mm^2/s^2 {cSt/s})

v: Kinematic viscosity of the standard liquid (mm^2/s {cSt})

t: Outflow time (s)

(b) Method by calibrated viscosimeter

(i) Use either a viscosimeter which was calibrated in (a) above or any calibrated viscosimeter, select a hydrocarbon with the outflow time of 200 sec or more. However, for any viscosimeter listed in Table 13, a hydrocarbon with the outflow time of the minimum outflow time specified or more must be selected.

(ii) Place both a previously calibrated viscosimeter and a viscosimeter in which need of calibrating simultaneously in the thermostatic chamber. For hydrocarbon selected in (i) above, measure the outflow time at least to the nearest 0.1 seconds in accordance with 5.4 (3). Calculate the viscosimeter constant to four digits of the significant figure by the following formula:

$$C_1 = \frac{t_2 \times C_2}{t_1}$$

where C₁: Viscosimeter constant (mm^2/s^2 {cSt/s})

C₂: Constant of previously calibrated viscosimeter (mm^2/s {cSt/s})

t₁: Outflow time on previously calibrated viscosimeter (s)

t₂: Outflow time of hydrocarbon on viscosimeter to be calibrated (s)

Remark: With respect to the viscosimeter constant C, when the gravitational acceleration g at the place where a viscosimeter was calibrated and the place where the same viscosimeter is used differs 0.1 % or more, correct the viscosimeter constant using the following formula:

$$C_3 = \frac{g_2}{g_1} \times C$$

where C₃: Viscosimeter constant corrected at the place of use (mm^2/s^2 {cSt/s})

g₂: Gravitational acceleration at the place of use (cm/s^2)

C: Viscosimeter constant at the place of calibration (mm^2/s^2 {cSt/s})

g₁: Gravitational acceleration at the place of calibration (cm/s^2)

Remarks: As samples with high wax content such as lubricating and fuel oils are susceptible to heat background, when the kinematic viscosity is measured at 95°C or less, a preheating treatment must be conducted.

To obtain a representative sample, heat the sample contained in the bottle to approximately 50°C and stir. To confirm if all wax is dissolved, insert a bar into the bottom of the bottle; transfer approximately 100 ml of sample to a 150 ml of conical flask which will be loosely covered with a cork or rubber stopper. Immerse the flask into the boiling water bath for thirty minutes. After well stirring the sample, remove it from the boiling water bath and filter with a wire net with 0.074 mm mesh. Place the sample in the viscosimeter which was previously placed in the thermostatic chamber. Complete the test after one hour of preheating.

Table 14 Viscosimeter for which the minimum outflow time is specified

Viscosimeter	Viscosimeter constant, mm^2/s^2 {cst/s} ⁽²⁰⁾	Minimum outflow time, s
Cannon - Fensuke viscosimeter	0.002	470
	0.004	460
	0.008	330
	0.015	240

Note ⁽²⁰⁾: $\text{mm}^2/\text{s}^2 = \text{cSt/s}$

- (f) Leave the viscosimeter in the thermostatic chamber until it reaches the test temperature. As the time required to reach test temperature varies according to the type of viscosimeter and test temperature. Confirm in advance the time requiring to reach equilibrium temperature.
When the quantity of sample needs to be adjusted depending upon the type of viscosimeter, adjust it after it reaches the test temperature.
- (g) When the test temperature is at dew point or less, to prevent condensation of water in the viscosimeter, the test shall be carried out by attaching a moisture-proofing device to the viscosimeter. When the test temperature is 0°C or below, put the sample in the viscosimeter at room temperature, and after the sample fills the capillary tube, place the viscosimeter in the thermostatic chamber.
- (h) By suction (in the case that volatile component is not contained) or compression, raise the sample's level approximately 5mm beyond the time measuring start mark of the viscosimeter.
- (i) Allowing the sample to flow out naturally, the time at which the sample's meniscus passes the viscosimeter's mark for measuring time shall be read to the nearest 0.1 seconds. When the outflow time does not reach the time specified in (d) above, the test shall be repeated using a viscosimeter with a smaller capillary.
- (j) When the improved Oswald type or suspended level type viscosimeter is used, measure the outflow time by repeating the operation mentioned in (h) and (i). When the outflow times measured twice are within 0.2 % of each other, the mean value is used for the calculation of the kinematic viscosimeter. When a counterflow viscosimeter is used, conduct the test twice ensuring the Kinematic viscosity value is within 0.35 % of each other.

If the test results are not within these limits, disregard them.

(4) Calculation and result

Calculate the Kinematic viscosity by the following formula and round off to four digits of the significant figure. The test temperature (°C) shall be appended.

$$v = C \times t$$

where v: Kinematic viscosity (mm^2/s {cSt})
 C: Viscosimeter constant (mm^2/s {cSt/s})
 t: Outflow time (s)

- Remarks: 1. With respect to Cannon-Fensuke, Cannon-Manning semi-micro viscosimeter and Cannon-Fensuke viscosimeter for opaque liquid, the quantity of the sample is determined at room temperature. Therefore, there is an error due to thermal expansion or contraction (See JIS Z 8803).
2. When the surface tension of the standard solution and the sample is different, an error will occur as the true effective height of liquid column will differ (See JIS Z 8803). However, the surface tension of a normal sample is almost equal to that of the standard liquid (See JIS Z 8809), therefore the difference may be ignored.

5.6 Liquid stability

5.6.1 High temperature stability

- (1) Instrument
Use the instrument specified in (1) (Apparatus and instrument) of 5.2.1 (Dry Equilibrium Reflux Boiling Point).
- (2) Operation
 - 1) Place 60 ± 1 ml of a new sample in the flask when measuring the boiling point. Assemble the apparatus in accordance with (2) (Operation) of 5.2.1 (Dry Equilibrium Reflux Boiling Point).
 - 2) Heat the sample to $185 \pm 2^\circ\text{C}$, then maintain the temperature for 120 ± 2 min. Heat it so that one to five drops per second are condensed within five minutes. Immediately adjust the heat to a point where one to two drops per second are condensed at this equilibrium reflux rate for next 5 ± 2 min. Maintain the reflux rate for another two minutes during which the temperature will be read four times every thirty seconds. Record the mean value as the measured equilibrium reflux boiling point.
- (3) Calculation and report
 - 1) Correct the measured equilibrium reflux boiling point by calibrating the thermometer in accordance with (3) (Calculation and report) of 5.2.1 (Dry equilibrium reflux boiling point).
 - 2) Report the difference between the mean value of these two corrected equilibrium reflux boiling points and the dry equilibrium reflux boiling point obtained by the method of 5.2.1 (Dry Equilibrium Reflux Boiling Point) as the change of boiling point.
 - 3) The initial measuring temperature is the highest temperature achieved in the following minute while maintained in the state of 2) above.

5.6.2 Chemical stability

- (1) Instrument
Use the instrument specified in (1) (Apparatus and instrument) of 5.2.1 (Dry Equilibrium Reflux Boiling Point).
- (2) Reagent
Comparative brake fluid [specified in SAE J 1703].
- (3) Operation
 - 1) Place 30 ml of the sample and 30 ml of the comparative brake fluid in the flask, then mix. Heat the mixture in accordance with (2) (Operation) of 5.2.1 (Dry Equilibrium Reflux Boiling Point). Control the temperature for 10 ± 2 min so that the liquid is refluxed at the rate of one to five drops per second.
 - 2) After the reflux rate reaches one to five drops per second, control the temperature for 5 ± 2 min. so that an equilibrium rate of one to two drops per second is maintained.

5.7 Corrosiveness to metal

- (1) Apparatus and instrument
The apparatus and instrument are as follows:
 - 1) Waterproof abrasive paper - No. 320 of AA or CC specified in JIS R 6253 (Waterproof Abrasive Papers)
 - 2) Steel wool - Commercial product # 00
 - 3) Test tube - Specified in Figure 20.
 - 4) Centrifugal separator - Specified in 4.3 (2) of JIS K 2503.
 - 5) Thermostatic chamber - Capable of maintaining the temperature at $100 \pm 2^\circ\text{C}$.
 - 6) Glass bottle - Two bottles specified in Figure 19, having a hole of 0.8 ± 0.1 mm in diameter at the center of lid.
 - 7) Ethylene tetra-fluoride resin tape or ethylene tetra-fluoride resin plate - The ethylene tetra-fluoride resin tape shall be a commercial product for piping, and the ethylene tetra-fluoride resin plate shall be suitable to the lid of 6) above and have a slightly larger hole than the opening of 6).
 - 8) Desiccator - Must have a proper capacity, and silica gel or calcium chloride as the desiccant shall be used.
 - 9) Micrometer - Able to read down to 0.01 mm.