

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing methods for
crude petroleum**

JIS K 2601—1990

Translated and Published

by

Japanese Standards Association

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

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JAPANESE INDUSTRIAL STANDARD
Testing Methods for Crude Petroleum

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1. Scope

This Japanese Industrial Standard specifies the testing methods for crude petroleum.

- Remarks 1. Applicable Standards to this Standard are shown in Attached Table 1.
2. The corresponding International Standard to this Standard is shown in the following.
- ISO 3680-1983 Paints, varnishes, petroleum and related products-flash/no flash test-rapid equilibrium method
3. In this Standard the units and numerical values shown in { } are in accordance with traditional units and are Standard values.

2. General Matters

The general matters shall be as follows:

- (1) Matters common to chemical analysis are in accordance with JIS K 0050.
- (2) Matters common to potentiometric titration are in accordance with JIS K 0113.
- (3) Matters common to absorptiometric analysis are in accordance with JIS K 0115.
- (4) Rounding-off method for numerical values are in accordance with JIS Z 8401.
- (5) Sampling methods are in accordance with the sampling method of secondary sample specified in JIS K 2251 or an equivalent method.

3. Classification of Testing Methods

The classification of testing methods shall be as given in Table 1.

4. Testing Method for Density

The testing method for density shall be in accordance with JIS K 2249. However, the class of testing method shall be described clearly.

5. Testing Method for Vapor Pressure

The testing method for vapor pressure shall be in accordance with JIS K 2258. The specifications of accuracy are not to be applied. Further, in the case of sample difficulty in flow, the sample shall be taken at the lowest temperature capable of flowing.

Reference Standards and Attached Table 1 Applicable Standards: See pages 62, 63, 64 and 65.

Table 1. Classification of Testing Methods

Testing method	Number of clause
Testing method for density	4.
Testing method for vapor pressure	5.
Testing method for kinematic viscosity	6.
Testing method for pour point	7.
Testing method for carbon residue	8.
Testing method for sulfur	9.
Testing method for nitrogen	10.
Testing method for ash	11.
Testing method for heat of combustion	12.
Testing method for water content	13.
Testing method for flash point	14.
Testing method for water and sediment	15.
Testing method for salt (titrimetric method)	16.
Testing method for salt (conductometric method)	17.
Testing method for distillation of crude petroleum by ordinary pressure method	18.
Testing method for wax	19.
Testing method for hydrogen sulfide	20.
Preparation method of water saturated toluene for water and sediment test	Annex

Remarks: The homogenizing method of sample for water and sediment test by using a stirrer is shown in Informative Reference (Homogenizing method of sample for water and sediment test).

6. Testing Method for Kinematic Viscosity

Measure the kinematic viscosity at 30°C, 50°C or 75°C according to the testing method for kinematic viscosity specified in JIS K 2283 (1). However, do not apply the specifications of accuracy.

Note (1) Make the testing temperature for kinematic viscosity not lower than the temperature at which the sample flows completely.

Further, at the time of measuring the kinematic viscosity, according to the operation "when a volatile component is contained", make the time required for the test operation as short as possible.

7. Testing Method for Pour Point

The testing method for pour point shall be in accordance with that specified in JIS K 2269. However, the specifications of accuracy are not to be applied.

8. Testing Method for Carbon Residue

The testing method for carbon residue shall be in accordance with JIS K 2270. However, the specifications of accuracy are not to be applied.

Further, relating to the crude petroleum having a large quantity of water and sediment or of light substance, recover the petroleum residue according to 18.5, measure the carbon residue in the obtained petroleum residue and it may be calculated according to the following formula.

$$A = \frac{B \cdot m}{M}$$

where A: carbon residue in crude petroleum (mass %)

B: carbon residue in petroleum residue obtained in the ordinary-pressure distillation test (mass %)

m: mass of petroleum residue obtained in the ordinary-pressure distillation test (g)

M: mass of sample weighed out into a distillation flask in the ordinary-pressure distillation test (g)

9. Testing Method for Sulfur

The testing method for sulfur shall be in accordance with the combustion tube type (air method) or radiation type (excitation method) specified in JIS K 2541. However, relating to the sample which is impossible to measure to 0.1 mg unit due to volatility, the specifications of accuracy are not to be applied.

10. Testing Method for Nitrogen

The testing method for nitrogen shall be in accordance with JIS K 2609. However, specifications of accuracy are not applied.

11. Testing Method for Ash

The testing method for ash shall be in accordance with the testing method for ash specified in JIS K 2272. However, the specifications of accuracy are not applied.

Further, relating to the crude petroleum having a large quantity of water and sediment or light substance, recover the petroleum residue according to 18.5, measure the ash in the obtained petroleum residue and may be calculated according to the following formula.

$$C = \frac{D \cdot m}{M}$$

where C: ash in the crude petroleum (mass %)

D: ash in the petroleum residue obtained in ordinary pressure distillation test (mass %)

m: mass of petroleum residue obtained in ordinary pressure distillation test (g)

M: mass of sample weighed out into the distillation flask in ordinary pressure distillation test (g)

12. Testing Method for Heat of Combustion

The testing method for heat of combustion shall be in accordance with JIS K 2279. However, specifications of accuracy are not applied.

13. Testing Method for Water Content

The testing method for water content shall be in accordance with JIS K 2275.

14. Testing Method for Flash Point

14.1 In the Case of Sample Flowing at Room Temperature The testing method for flash point in this case shall be in accordance with Tag closed type or Penskey-Martens closed type specified in JIS K 2265. However, the specifications of accuracy are not applied.

14.2 In the Case of Sample not Flowing at Room Temperature

14.2.1 Testing Method for Flash Point of Site Closed Type

- (1) Summary of Testing Method Determine the temperature for confirming whether it flashes or not hereafter referred to as the "judgement temperature" in the range of 0 to 110°C.

Put the sample 2 to 3 g into the sample cup kept at the judgement temperature, and leave as it is for 60 (2). Apply the test flame to observe whether it flashed or not, and express whether it flashes or not at the judgement temperature.

Note (2) In the case where the judgement temperature is not higher than room temperature, leave as it is for 5 to 10 min.

Reference: Make the judgement temperature the temperature dividing the first petroleum, second petroleum or third petroleum according to the Fire Service Law, that is 21°C or 70°C, or the temperature estimated from the quantity of light fraction.

- (2) Seta Closed Type Flash-Point Tester The Seta closed type flash-point tester consists of (2.1) to (2.4) and its one example is shown in Fig. 1.

Further, the insulating performance of the tester, in the condition of closed electric circuit when the insulation resistance between the power source terminal and the case is measured shall be 5 MΩ or more. However, in the case of containing the electric heating circuit, it may be 0.5 MΩ or more.

- (2.1) Heating Block The block made of aluminium alloy or corrosion resistant metal having good heat conduction and provided with a cylindrical dent of approximately 10 mm in depth, approximately 50 mm in diameter, hereinafter referred to as the "sample cup", for holding the sample, and with the O ring for keeping air tightness adhering tightly with the lid and the thermometer-inserting hole. The details are shown in Fig. 1.
- (2.2) Lid The lid made of brass or suitable metal of such structure that it is attached on the upper surface of the sample cup and opened and closed with a hinge and lid retainer, and in the closed condition it adheres tightly with the O ring and closes tightly the space above the sample. The lid is provided with three windows, sample injection tube, flame exposure mechanism for test flame and movable plate, etc.

The details are shown in Fig. 2.

- (a) Movable Plate The movable plate is made of brass or suitable metal, which slides along the movable-plate guide adhering tightly with the lid, opens and closes three windows provided at the lid and of such mechanism to allow to apply the test flame. When in the open condition, the two windows of the movable plate must coincide correctly with two windows of a corresponding lid.

Further, the sliding surface between the upper surface of the lid and the movable plate must be ground-in and be so made as the sample vapor does not leak. The details are shown in Fig. 3.

- (b) Flame Exposure Mechanism for Test Frame The flame exposure mechanism is made of brass or stainless steel, and of such structure that it is capable of adjusting the size of the test flame at 3.5 ± 0.5 mm and when the movable plate is open, it allows to apply the test flame in the middle window.

Further, when allowed to apply the test flame, the center of tip of test flame nozzle must coincide with the plane of the lower surface of the lid in the range of ± 0.1 mm. The details are shown in Fig. 1.

- (2.3) Thermometer The thermometer specified in Table 2.

Further, in the clearance between the mercury bulb of thermometer and the thermometer inserting hole of the heating block must be filled with a paste good in heat conductivity.

Reference: As to the paste good in heat conductivity, the grease of silicone series mixed with metal oxide is suitable.

As to goods on the market, there is Dow Corning 340 Heat Sink Compound, KS 609 and the like.

Table 2. Glass Thermometer for Seta Closed Type Flash Point Test

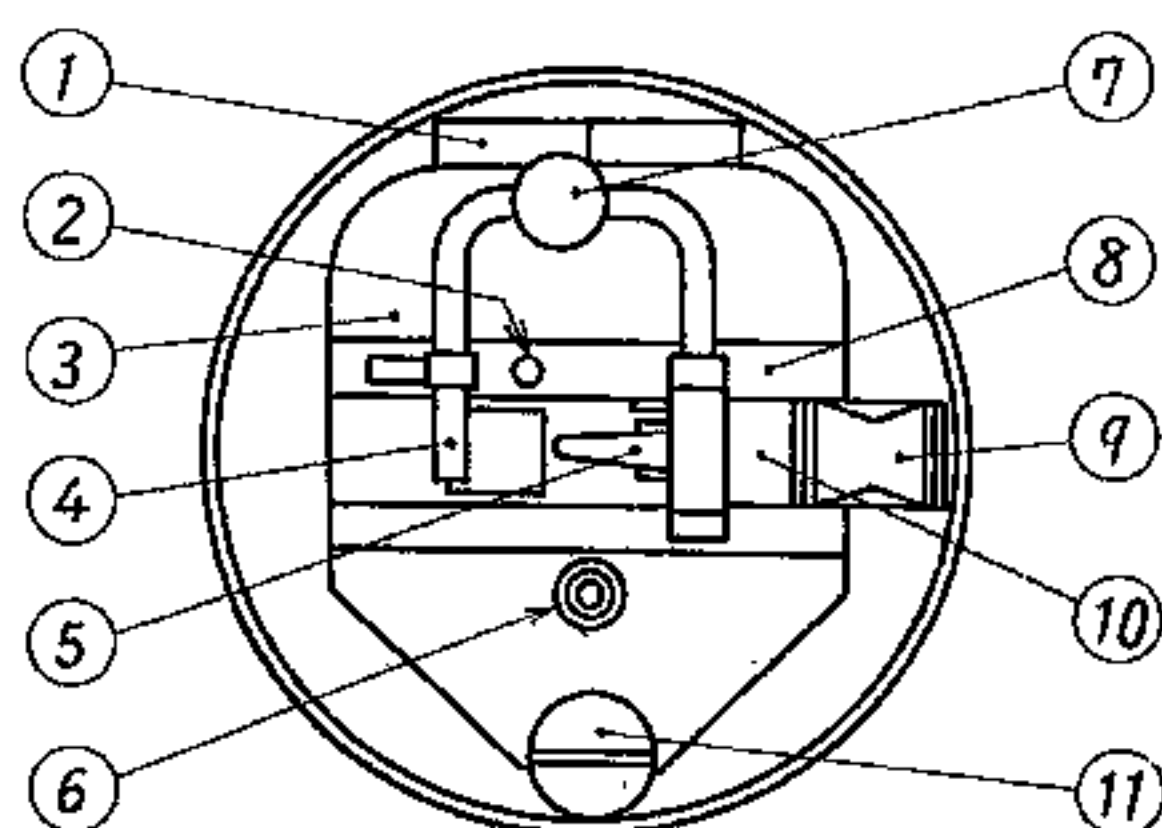
Scale range °C		0 to 110
Immersing mm		44.5 ± 1
Scale °C	Scale interval	1
	Long scale line	5
	Scale numeral	10
	Scale error	0.5
Expansion chamber	Thermometer permissible heating temperature °C	120
Total length mm		200^{+4}_{-2}
Diameter mm		6 to 7
Bulb mm	Length	10 to 14
	Diameter	4.50 to 4.65
Position of scale	Distance from the lower end of bulb up to 0°C scale line	50 ± 2
	Distance from the lower end of bulb up to 110°C scale line	165 to 185
Blister		Without

- (2.4) Heater Electric heating type, having the temperature adjusting mechanism capable of keeping the sample cup at set temperature $\pm 0.2^{\circ}\text{C}$ in case the setting temperature is ordinary temperature or higher and capable of indicating that the heating block has reached the set temperature.
- (2.5) Sample Tube The sample tube made of stainless steel or suitable metal, having a bore of approximately 10 mm and suitable length.
- (2.6) Cooling Bag In the case where the temperature setting is not higher than room temperature, the cooling bag to be used for cooling the heating block, which is a plastic bag sealed with liquid low in freezing temperature and the cooled insulator on the market is cooled by a refrigerator or the like, or corresponding to the test temperature the plastic bag or the like filled with crushed ice or a mixture of crushed ice and sodium chloride.
- Reference: It is convenient to use an electronic cooling element attached with temperature regulator instead of cooling bag.
- (2.7) Sample Finish Presser The sample finish presser to be used for spreading the sample on the bottom part of sample cup so as to be a uniform thickness by pressing the sample taken in the sample cup, which is a suitable metal disk of rather smaller diameter than the internal diameter of sample cup attached with a handle or a glass or corresponding metal beaker of suitable size may be used.

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Fig. 1. Seta Closed Type Flash Point Tester

Unit: mm



- | | |
|----------------------------|------------------------------|
| ① Hinge | ⑨ Knob |
| ② Standard bulb | ⑩ Movable plate |
| ③ Lid | ⑪ Lid stopper |
| ④ Guide flame nozzle | ⑫ Thermometer |
| ⑤ Sample flame nozzle | ⑬ O ring for seal |
| ⑥ Sample injection opening | ⑭ Heating block |
| ⑦ Gas adjusting valve | ⑮ Thermometer inserting hole |
| ⑧ Movable-plate guide | |

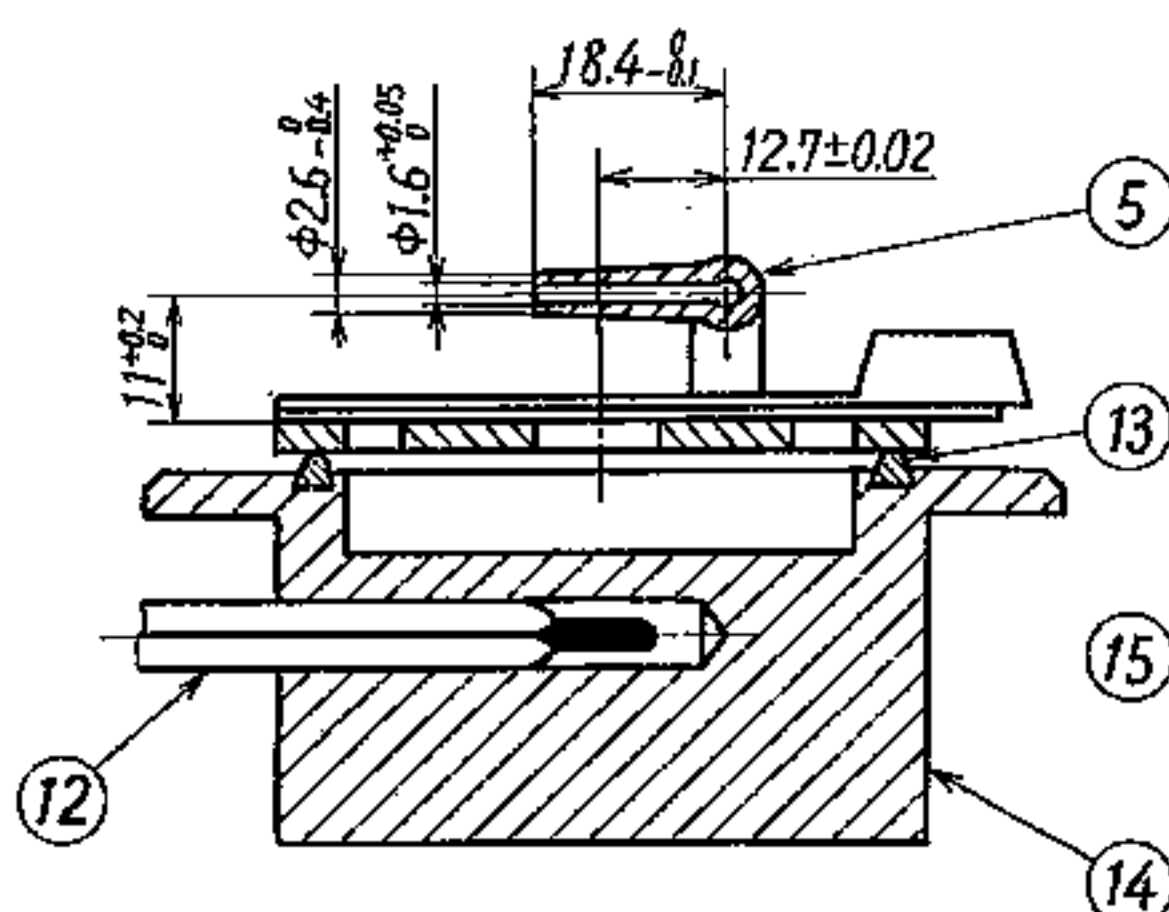


Fig. 2. Lid

Unit: mm

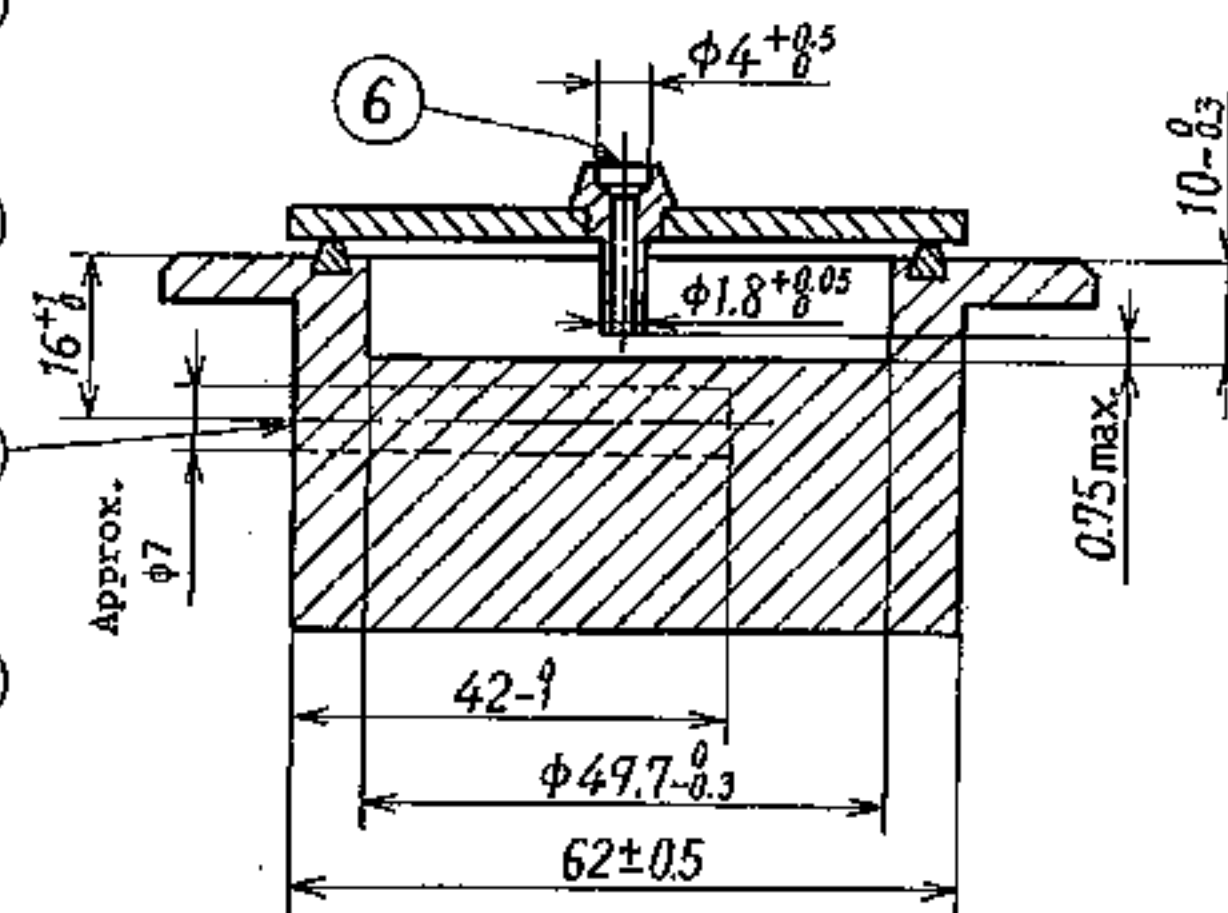
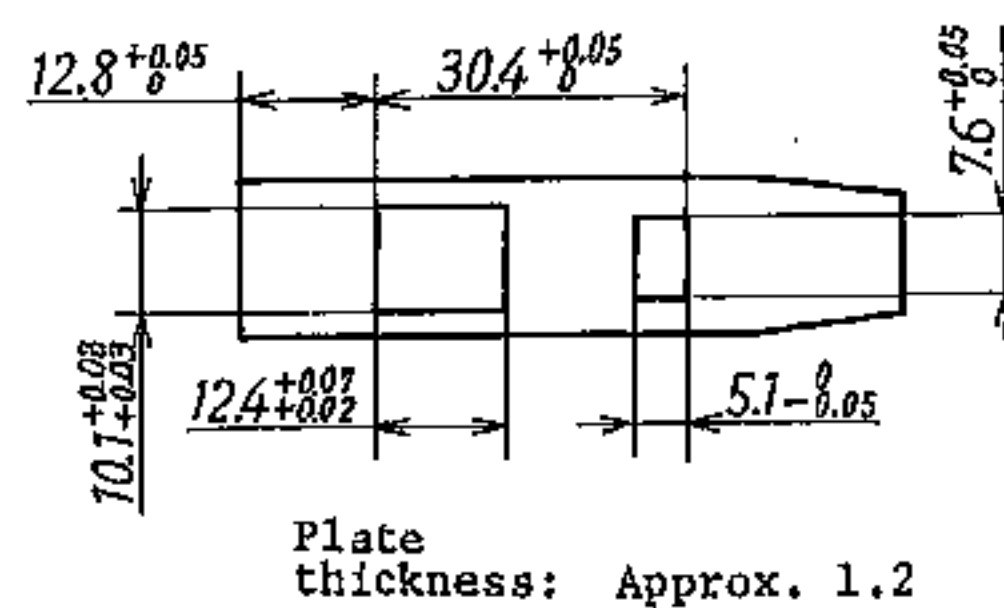
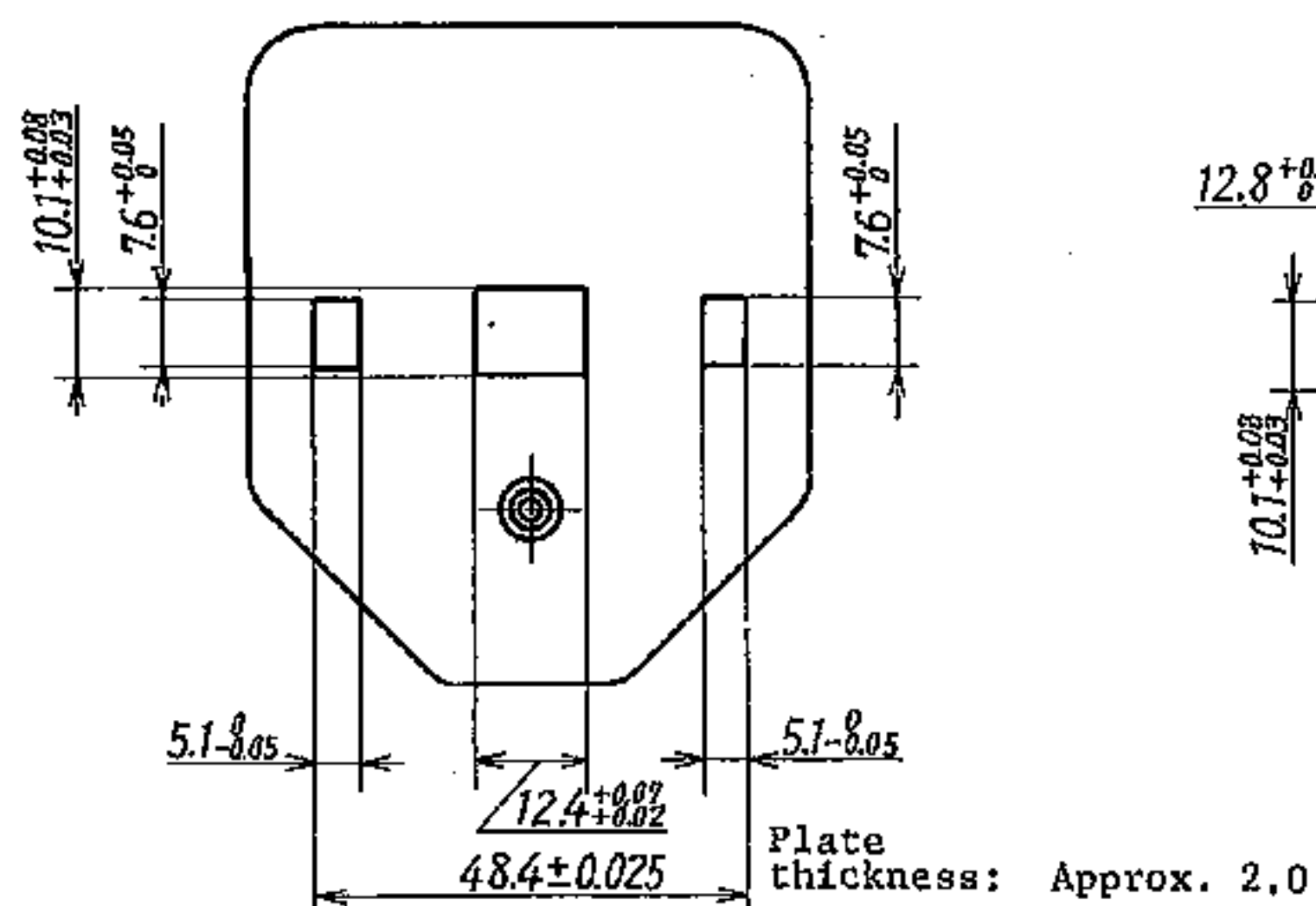


Fig. 3. Movable Plate

Unit: mm



- (3) Sample Until executing the test, preserve the sample in a tightly closed container. Further, take care that the space above the contents in the container does not to exceed 10% of the total capacity of the container.

Remarks: Because plastic containers (made of polypropylene, polyethylene, etc.) have the potential to diffuse the evaporating substance in the sample through the wall surface, do not use them.

- (4) Preparation of Tester Place the tester in a place of as little draft as possible. Avoid direct sunlight and strong illumination.
- (5) Setting of Temperature Set the judgement temperature by integer unit, and take it as the setting temperature.

Remarks: In case of atmospheric pressure exceeding the range of 99.4 to 103.2 kPa {746 to 774 mmHg}, calculate the setting temperature from the judgement temperature according to the following formula and round off to an integer.

$$T_s = T - 0.25 (101.3 - P_1)$$

$$\{T_s = T - 0.033 (760 - P_2)\}$$

where T_s : setting temperature (°C)

T : judgement temperature (°C)

P_1 : atmospheric pressure (kPa)

P_2 : atmospheric pressure (mmHg)

- (6) Procedures of Test Corresponding to the setting temperature obtained in (5), carry out the test according to (6.1) case of setting temperature exceeding room temperature or (6.2) case of setting temperature not higher than room temperature.

(6.1) Case of Setting Temperature Exceeding Room Temperature

- (a) Maintain the sample, sample finish presser and clean sample tube at a temperature lower by approximately 10°C than the setting temperature. However, in the case where the setting temperature is higher by 10°C than room temperature, maintain the sample, sample finish presser and sample tube at room temperature.
- (b) Wipe the inside of sample cup, lid, and movable plate with tissue paper or the like to make clean. After closing the sample injection opening with a heat-resistant rubber plug or the like, close the lid.

- (c) Turn on the switch of heater, regulate the temperature adjuster to coincide with the reading of thermometer correctly with the setting temperature and stabilize the temperature.
- (d) After inserting the sample tube into the sample, block the upper end of the sample tube with your finger to draw out and take out the sample. In this case, the position of sampling should preferably be positioned at a point approximately at an equal distance from the side wall of sample container and the center of sample container and in the depth, be positioned at a point of approximately equal distance from the upper surface of sample and bottom face of sample container.

Remarks: Depending upon the caking of the sample, in some cases the sampling by sample tube is difficult. In this case, carry out the sampling by using a spatula or the like. Do not take the sample from the surface.

- (e) Open the lid of the tester, apply pressure to the upper end of sample tube by using rubber ball or the like, and push out the sample 2 to 3 g ⁽³⁾ into the sample cup. Immediately place a thin plastic film ⁽⁴⁾ on the sample cup, press from above it with the sample finish presser, and broaden the sample so the sample attains as uniform a thickness as possible on the whole surface of the sample cup bottom. Remove the sample finish presser and plastic film, close the lid, fix the lid stopper and start the second watch. Carry out the operation of (e) as quickly as possible.

Notes ⁽³⁾ From the bore of the sample tube and density of sample, preliminarily calculate the pressing out length.

⁽⁴⁾ Use the plastic film for packaging at the time when in Radiation type excitation method of JIS K 2541, enter the polyester film to be used as cell window material or in household, placing foods into electric refrigerators or electronic ranges.

- (f) Open the gas adjusting valve, ignite the guide flame and test flame, and so adjust the size of test flame to coincide with the standard bulb.
- (g) After the elapsed time of 60 s since placing the sample in the sample cup, open the movable plate for 2.5 ± 0.5 s to allow to apply the test flame, and observe the existence of ignition ⁽⁵⁾.

Note ⁽⁵⁾ At a temperature near the flash point, a blue-white ring may appear on the periphery of the test flame, but do not regard this as the flash and when the flame is broadened on the surface of the sample, regard it as the flash.

- (h) When having flashed, express as "flash" according to (7) and end the test. In case of no flash, repeat the operation of (a) to (g), and when having flashed, express as "flash" according to (7). In case of non-flash in either test results, express as "non-flash" according to (7).
- (6.2) Case of Setting Temperature not Higher than Room Temperature
- (a) Maintain the sample, sample finish presser, and clean sample tube at a temperature lower by 3 to 5°C than the setting temperature.
- (b) Wipe the inside surface of the sample cup, lid and movable plate with tissue paper or the like to make clean. Close the sample injection tube with a heat resistant rubber plug or the like.
- (c) Open the lid, place the cooling module ⁽⁶⁾ into the sample cup or close the lid to cover the cooling bag on the lid, and cool the heating block until the thermometer indicates a temperature lower by 3 to 5°C than the setting temperature.

Note ⁽⁶⁾ The cooling module is a cylindrical container made of stainless steel or suitable metal, having the outer diameter of size to adhere tightly in the sample cup and the height of 200 mm or more,

Further, cover the outer wall of part other than the part adhering tightly to the sample cup with a suitable heat-insulating material. Put crushed ice or a mixture of crushed ice and sodium chloride into this, place into the sample cup and cool the heating block.

- (d) After inserting the sample tube into sample, by drawing out with closing the upper end of sample tube with your finger and take the sample. At this time, the sampling position should preferably be the position where the top end of the sample tube is at approximately equal distance from the side wall of sample container and the center of the sample container and in depth at the position of approximately equal distances from the upper surface of sample and the bottom face of sample container.

Remarks: Depending upon the calking condition of sample, in some cases the sampling by sample tube may be difficult. In this case, by using a spatula or the like carry out the sampling. Do not take the sample from the surface.

- (e) Open the lid of the tester, and in the case where the condensate water is confirmed, wipe off with tissue paper or the like. Apply pressure to the upper end of sample tube by rubber ball or the like, and press out the sample 2 to 3 g ⁽³⁾ into the sample cup. Immediately place a thin plastic film ⁽⁴⁾ on the sample cup, press the sample finish presser from above it, and broaden the sample so the sample attains a uniform thickness on the whole surface of the bottom part of the sample cup as much as possible. Remove the sample finish presser and plastic film, close the lid and fix by lid and fix by lid stopper, and start the second watch.

Carry out the operation of (e) as quickly as possible.

- (f) Cover the lid with a cooling bag, keep the heating block cool, and keep the temperature of the heating block in the range from the setting temperature until the temperature is lower by 3°C than that temperature for 5 min or more.
- (g) After removing the cooling bag, open the gas adjusting valve, ignite the guide flame and test flame, and adjust the size of test flame to a diameter of 3.5 ± 0.5 mm.
- (h) Leave the tester as it is, when the thermometer has indicated the setting temperature, open the shutter for 2.5 ± 0.5 s to allow to apply the test flame, and observe the existence of flash ⁽⁵⁾. However, in case where the time since starting the moving of the second watch in (e) until test flame is applied exceeds 10 min, change the keeping cool temperature of (f) and retest.
- (i) In the case of having flashed, express as "flash" according to (7) and end the test. In case of no-flash, repeat the operation of (a) to (h) and in the case of having flashed express as "flash" according to (7). In case where both test results are no-flash, express as "no flash" according to (7).
- (7) Results Express the results of Seta closed type flash point test by the judgement temperature determined in (5) to the integer unit and in the judgement temperature express that it is flash or no-flash. At that time, describe that it is the result of Seta closed type flash point test for example by mark (SCC) and the like (Refer to Examples 1 and 2.).

Remarks: In the case where the setting temperature is corrected, express by the not-corrected setting temperature, not by the temperature when the test is carried out actually that is the corrected setting temperature.

Example 1. Flash point (SCC) ... 21°C, flash

Example 2. Flash point (SCC) ... 5°C, no flash

(8) Accuracy The accuracy is not specified.

15. Testing Method for Water and Sediment

15.1 Summary of Testing Method Respectively take the sample 50 ml and water saturated toluene 50 ml into two scaled test tubes for centrifugal separation, after mixing completely repeat the centrifugal-separation operation at 60°C (7) by using a centrifugal separator for 10 min twice, read out the volume of settled water and sediment layer on the bottom part of scaled test tube, and calculate the water and sediment.

Note (7) In the case where the pour point of sample is not higher than 10°C, the centrifugal separation at 49°C may be performed.

Remarks: The result capable of being obtained according to this testing method is the total quantity of water and sediment. Therefore, for measuring correctly only the water content, the method shall be in accordance with 13.

15.2 Water-and-Sediment Tester The water and sediment tester shall be as follows.

Further, the insulating performance of the tester in the condition of electric circuit "closed", when the insulation resistance between the power source terminal and the case is measured, shall be 5 MΩ or more. However, in the case of containing the electric heating circuit, it may be not less than 0.5 MΩ.

(1) Scaled Test Tube The scaled test tube specified in Fig. 105 or Fig. 183 of JIS K 2839.

Remarks: As to the test, usually the type I scaled test tube is used, but in the case of using a small type centrifugal separator, the type II scaled test tube is used.

(2) Centrifugal Separator The centrifugal separator capable of keeping specified scaled test tubes of even number of not less than two pieces containing the sample at a temperature of 60±3°C (8), capable of rotating at the specified speed, capable of being operated safely even in the flashable atmosphere and of rigid structure. The scaled-test-tube supporting tube is made of metal, and into this as the buffer felt, rubber and other suitable materials are inserted. The rotary part is covered with an enclosure made of rigid metal so as to be able to prevent risk when ruptured. Adjust the rotary speed so as the relative centrifugal force at the bottom part of scaled test tube is 600. Calculate the required number of revolutions per minute according to the following formula.

$$R=1\,335\sqrt{\frac{f}{d}}$$

where R : number of revolutions per min (rpm)

f : relative centrifugal force

d : rotary diameter (the distance between the two bottoms of opposing scaled test tubes in the revolving condition) (mm)

Note (8) In the case of carrying out the test at 49°C, the temperature of solution in the scaled test tube after centrifugal separation for 10 minutes may not be lower than 46°C.

Further, the relation between the rotary diameter and the number of revolutions is shown in Reference Table 1.

Reference Table 1. Required Number of Revolutions of Centrifugal Separator

Rotary diameter of centrifugal separator mm	Number of revolutions per minute so as the relative centrifugal force at 600 rpm
340	1770
360	1720
380	1680
400	1640
420	1600
440	1560
460	1520
480	1490
500	1460

- (3) Heating Bath The heating bath is to be a metal bath or liquid bath of depth capable of immersing the scaled the test tube vertically to the scale line of 100 ml and capable of keeping the bath temperature at 49±3°C and 60±3°C.

15.3 Reagents

- (1) Toluene The toluene specified in JIS K 8680, or pure toluene No. 1 or pure toluene No. 2 specified in JIS K 2435.
- (2) Water-Saturated Toluene The toluene of (1) saturated with water at 60 or 49°C according to Annex (Preparation method of toluene saturated with water for testing of water and sediment).

- (3) Demulsifier Solution Accelerate the separation of water in the sample to prevent the adhering of separated water to the inside wall of scaled test tube, use the demulsifier (phenol series, basic amine series or naphthenic acid series) to be used usually for desalting of crude petroleum in petroleum manufacturing factories and the like. The concentration and using quantity of demulsifier is to be such a degree that positive error on the result of water and sediment is not caused.

Further, put the demulsifier solution into a dark-dense colored bottle, and stopper tightly to store. In case where if stored for a long period, the demulsifier is settled and separated into two layers, newly prepare the demulsifier solution.

Reference: As to the concentration of demulsifier solution, the demulsifier 25% and toluene 75% are suitable but according to the crude petroleum in same cases different concentrations are required.

- 15.4 Sample Immediately before the test, the sample shall be shaken sufficiently to disperse sufficiently the water and sediment in the sample.

Further, the sample which does not flow at ordinary temperature or has high viscosity shall be heated to such viscosity as to be stirred easily. However, because there is a fear of a volatile substance to be volatilized to disperse, it shall not be heated at high temperature more than required, nor heated for a long time.

Reference: In the case where because the viscosity of sample is high it is considered difficult to make uniform by shaking by hand, the sample should preferably be made uniform by using a stirrer. The uniforming method by stirrer is shown in Reference method (uniforming method of sample for water and sediment test).

- 15.5 Procedures of Test The procedures of test shall be as follows:

- (1) Put the sample 50 ml respectively into two scaled test tubes up to the marked line, add the water saturated toluene 50 ml with a pipette and further add the demulsifier solution 0.2 ml by using a measuring pipette. Then, stopper tightly, reverse it approximately 10 times to mix the sample with the water-saturated toluene completely.
- (2) In case where because of the samples extremely high viscosity, the mixing with the water-saturated toluene is difficult, in order to make the mixing easy, at first the water-saturated toluene may be added to the scaled test tube. In this case, take care not to exceed the scale of 100 ml at the time of adding the sample.
- (3) Immerse the scaled test tube with the plug slightly loosened into the heating bath at $60 \pm 3^{\circ}\text{C}$ (°) up to the scale mark of 100 ml, and hold for 15 min or more. Again apply the plug, reverse it to mix completely the sample with the water saturated toluene.

Note (9) In the case where the pour point of sample is not higher than 10°C , it may be heated to $49\pm 3^{\circ}\text{C}$.

- (4) Preserve two scaled test tube into symmetrical scaled test tube holding tube of the preliminarily warmed centrifugal separator (10), revolve at the number of revolutions so as the relative centrifugal force becomes 600 for 10 min.

Note (10) In case where the mass difference between the two scaled test tubes exists, put water in the scaled test tube holding tube to achieve balance.

- (5) When the revolution of the centrifugal separator is stopped, immediately read out the combined volume of water and sediment on the bottom part of scaled test tube". Make the reading, in the case of from 0.1 ml to 1 ml, to the nearest 0.05 ml, in the case of not less than 1 ml, to the nearest 0.1 ml and in the case of not more than 0.1 ml, to the nearest 0.025 ml in the estimation value.

Without mixing by shaking the content liquid, again carry out the centrifugal separation for 10 min.

Note (11) In the case where the boundary between the supernatant and the precipitate, with taking so care if the water and sediment do not flow, gently tilt the scaled test tube and the combined quantity of volumes of water and sediment should preferably be read out.

Further, in the case where, at the time of carrying out the test at $49\pm 3^{\circ}\text{C}$, the matter of wax state is confirmed in the precipitate, make the test temperature at $60\pm 3^{\circ}\text{C}$ and repeat the operation of (3) to (6).

- (6) Repeat the operation of (4) and (5) until the volume of combined quantity of water and sediment is the same twice continuously.

Maintain throughout the centrifugal separation operation the temperature of sample at $60\pm 3^{\circ}\text{C}$ (12) without fail.

Note (12) In the case where the test is carried out at $49\pm 3^{\circ}\text{C}$, maintain the temperature at not lower than 46°C without fail.

15.6 Calculation and Results

- (1) Record the volumes of final water and sediment of respective scaled test tubes. If the difference of two readings exceeds the minimum scale of scaled test tube or when not more than 0.10 ml exceeds 0.025 ml, discard the test results and again carry out the test.
- (2) Take the sum of final reading quantities of two scaled test tubes as the water and sediment (volume %).

15.7 Accuracy The accuracy shall be as follows:

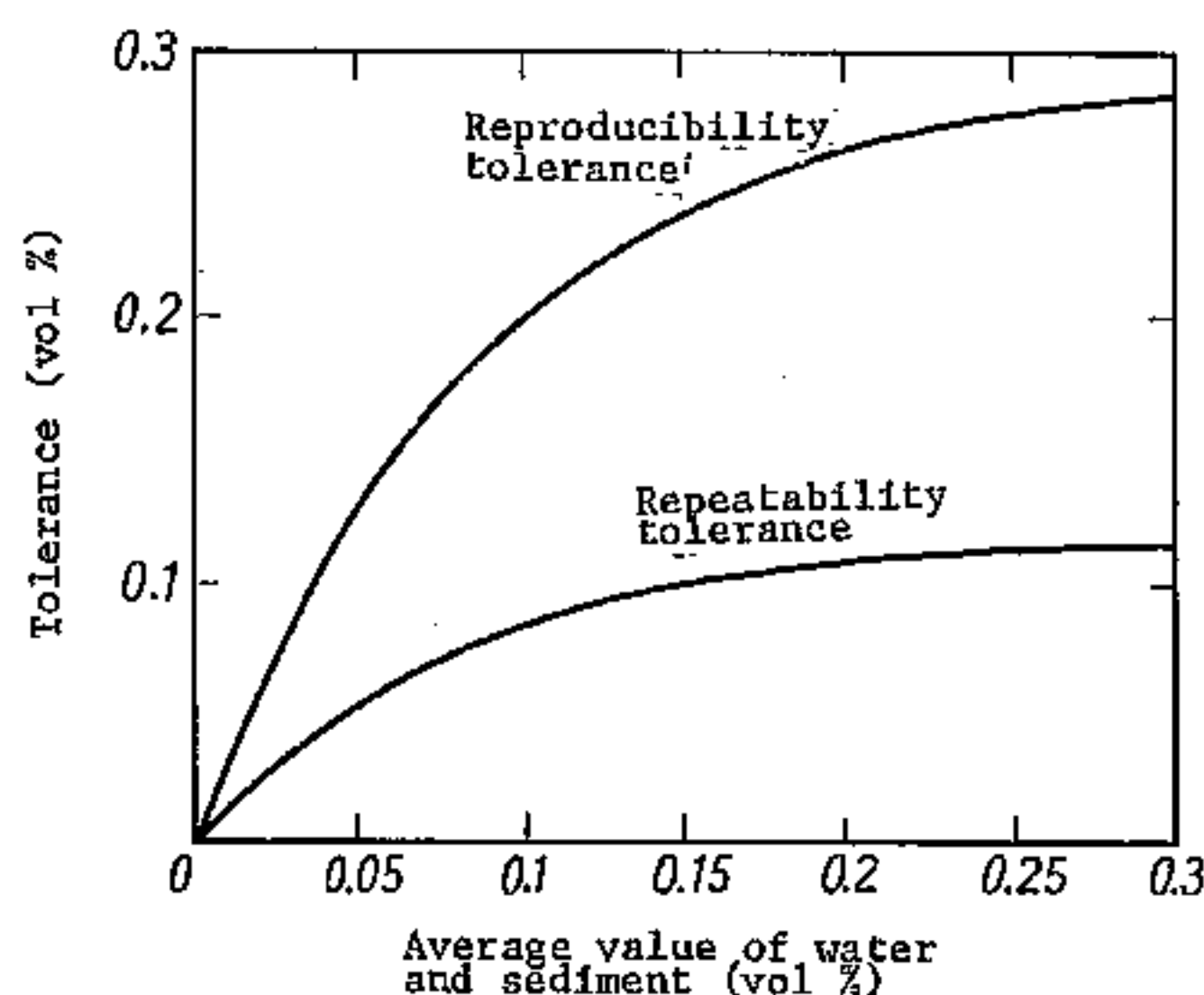
- (1) Repeatability When the same sample is tested twice in the same test room, by the same person with the same tester with changing the day or hour, the difference of test results, in the case where the test results are not more than 0.3 vol %, shall not exceed the tolerance of Fig. 4.

Further, in the case where the range of test results is 0.35 to 1.0 vol %, it shall not exceed 0.12 vol %.

- (2) Reproducibility The difference of two test results obtained by testing respectively once the same sample in different test rooms, by different persons with other testers, in the case where the test results are not more than 0.3 vol %, shall not exceed the tolerance of Fig. 4.

Further, in the case where the range of test results is 0.35 to 1.0 vol %, 0.28 vol % shall not be exceeded.

Fig. 4. Accuracy



16. Testing Method for Salt (Titrimetric method)

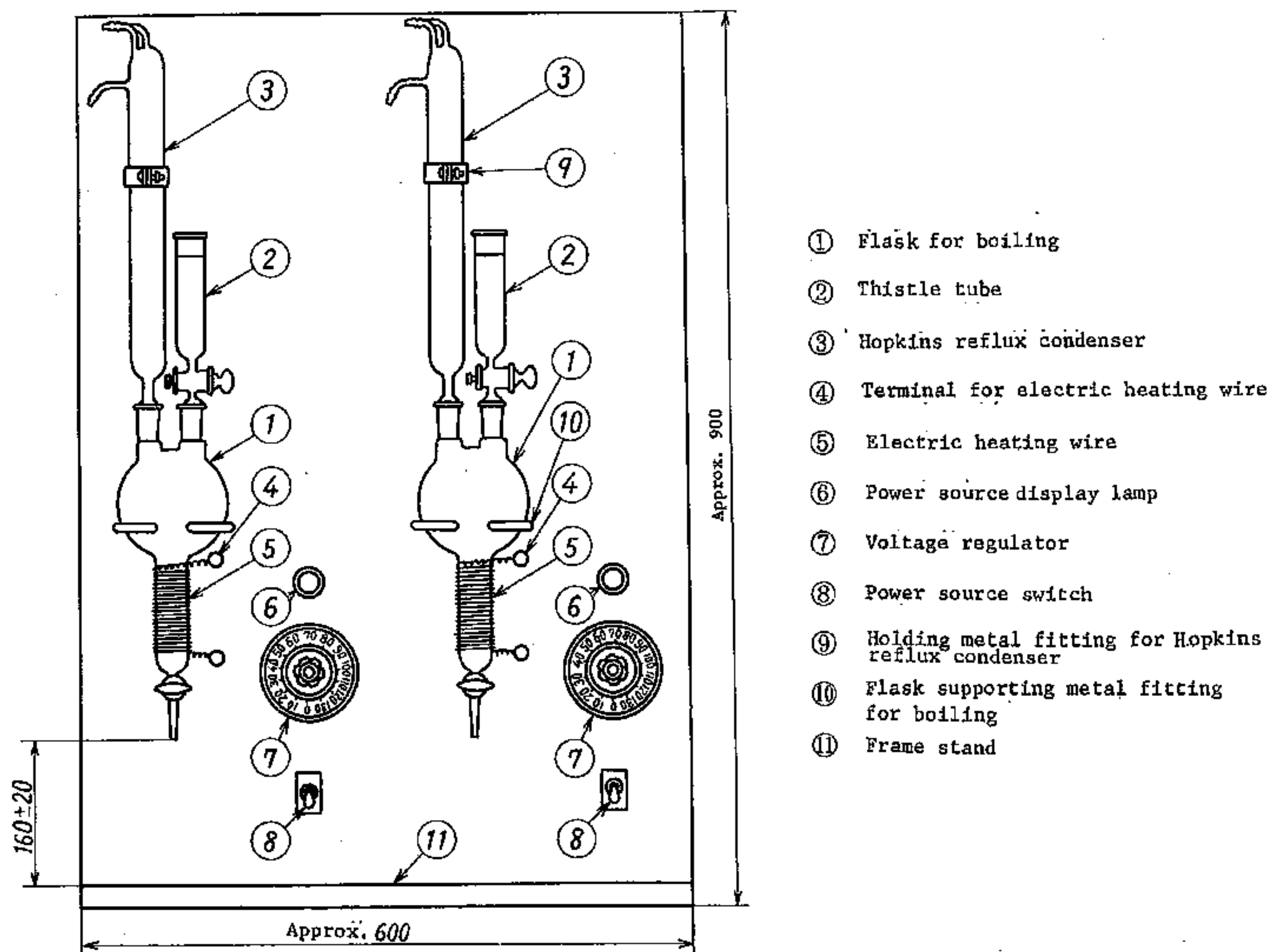
16.1 Summary of Test Method Boil the sample approximately 80 g together with the solvent and water, separate by extraction of the chloride in the sample to the water layer. Titrate this extract by indicator or potential difference to determine the salt as sodium chloride.

16.2 Salt Tester for Titrimetric Method The tester shall be that of structure shown in Fig. 5 and, consist of (1) to (2). Further, the insulating performance of tester, when measuring the insulating resistance between the power source terminal and the case, shall be 5 M Ω or more. However, in the case of including the electric heating circuit, it may be 0.5 M Ω or more.

Fig. 5. Salt Tester for Titrimetric Method (An example)

Two piece hanging

Unit: mm



(1) Extractor The extractor is constituted of (a) to (c), and assembled as shown in Fig. 5.

(a) Flask for Boiling The flask for boiling shown in Fig. 54 specified in JIS K 2839, Provided that the heating tube is wound with nickel-chromium heating wire class 1 specified in JIS C 2520 of 0.26 mm in wire diameter and approximately 2700 mm in length.

(b) Hopkins Reflux Condenser The Hopkins reflux condenser of Fig. 55 specified in JIS K 2839.

(c) Thistle Tube The thistle tube of Fig. 56 specified in JIS K 2839.

(2) Frame Stand The frame stand capable of holding the extractor as shown in Fig. 5 and provided with a voltage regulator for heating (0 to 100 V, 3 A or more).