

JIS

JAPANESE INDUSTRIAL STANDARD

**Petroleum products —
Determination of
distillation characteristics**

JIS K 2254—1990

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Petroleum products - Determination of
distillation characteristics

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1. Scope

This Japanese Industrial Standard specifies the determination of distillation characteristics for petroleum products.

Remarks 1. The following standards are cited in this Standard.

JIS B 7410-Liquid-in-Glass Thermometers for Testing of
Petroleum Products

JIS C 1602-Thermocouples

JIS K 2249-Testing Methods for Density of Crude Oil and
Petroleum Products, and Petroleum Measurement
Tables Based on a Reference Temperature 15°C

JIS K 2251-Method of Sampling Crude Oil and Petroleum
Products

JIS K 2258-Testing Method for Vapor Pressure of Crude Oil
and Petroleum Products (Reid Method)

JIS K 2280-Testing Methods for Octane Number and Cetane
Number

JIS K 2839-Glassware for Testing Apparatus of Petroleum
Products

JIS Z 8401-Rules for Rounding off of Numerical Values

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

ISO 3405-1988 Petroleum products - Determination of
distillation characteristics

3. In this Standard the units and numerical values shown in
{ } are in accordance with traditional units and are the
Standard value.

2. General matters

General matters common to this Standard are as follows:

- (1) Rounding-off method of numerical values shall be in accordance with
JIS Z 8401.
- (2) The sample for test shall be taken according to the sampling method
of secondary sample specified in JIS K 2251 or the method equivalent
thereto.

3. Definitions

Main definitions used in this Standard are as shown in Table 1.

Table 1. Definitions

Term	Definition
(1) initial boiling point	Reading of thermometer when the first drop of distillate oil falls from the lower end of condenser tube (°C)
(2) end point	Maximum thermometer reading obtained at the final step of determination of distillation characteristics (°C). Remarks: Usually it means the reading under the condition when the sample on the bottom of distillation flask is vaporized completely. Informative Reference: As the synonym of end point, there is the "maximum temperature".
(3) dry point	Reading of thermometer when the final drop of sample on the bottom of distillation flask is vaporized (°C). In this case, the inner wall surface of distillation flask and thermometer surface may be wet. Remarks: The end point, rather than the dry point, is intended for general use, but the dry point should preferably be substituted for the end point, when ever the sample can not be obtained at the accuracy specified in 5.8 in end point.
(4) decomposition point	Thermometer reading when the sample in the distillation in the distillation flask begins thermal decomposition (°C). Informative Reference: The thermal decomposition can be understood by characteristic indications of lowering of thermometer reading even generation of white fume and enhancing the heating.

Table 1. (Continued)

Term	Definition
(5) Percent recovered	The percent of recovered oil corresponding to thermometer reading (volume %).
(6) recovered temperature	Thermometer reading corresponding to percent recovered (°C).
(7) percent recovery	Total percent of percent recovered measured according to 5.6 (7) (volume %).
(8) percent total recovery	Sum of percent recovery and percent residue (volume %).
(9) percent loss	100 minus the percent total recovery (volume %).
(10) percent residue	The percent total recovery minus the percent recovery or the volume of residue in distilling flask measured according to Remark of 5.6 (8) (volume %).
(11) percent evaporated	The sum of the percent recovered and percent loss corresponding to thermometer reading (volume %).
(12) percent evaporated temperature	Thermometer reading corresponding to percent evaporated (°C).

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4. Classification of determinations

Determination of distillation characteristics shall be classified into the two types of Table 2.

Table 2. Classification of determinations

Classification	Application division	Example of applicable type oil
Ordinary pressure method	This method applies to fuels which are in a liquid state at ordinary temperature, and the end point under ordinary pressure of 400°C or less.	Automobile gasoline, industrial gasoline, aviation gasoline, aviation turbine fuel, keorsine, gas oil.
Reduced pressure method	This method applies to liquid petroleum products which evaporate completely or partially under the conditions of absolute pressure 0.13 to 6.66 kPa{1 to 50 mmHg} and the maximum liquid temperature of 400°C. Informative Reference: The reduced pressure method is suitable for a sample which according to ordinary pressure method causes thermal decomposition and is difficult to obtain the distilling state.	Reduced pressure gas oil, fule oil, light lubricating oil

Remarks 1. Relating to the sample, when the ordinary pressure method and reduced pressure method are applied, the results of both methods do not coincide necessarily.

2. The method to test distillation property of petroleum fraction (except gasoline) of 538°C or less in end point by using gas chromatograph is shown in Informative Reference (Determination of distillation characteristics of petroleum fractions by gas chromatography).

5. Ordinary pressure method determination of distillation characteristics

5.1 Summary of determination Distill the sample 100 ml under the test conditions specified in Table 3 according to its property, and measure the initial boiling point, recovered temperature, percent recovered, end point, etc.

Remarks: Automatic tester may be used. However, in case where doubt is generated in test results obtained by automatic tester, the results obtained by this determination are applied.

Table 3. Classes of sample and test conditions

Test condition		Class of applied sample			
		Class 1	Class 2	Class 3	Class 4
Characteristics of sample	Reid vapor pressure ⁽¹⁾ kPa { kgf/cm ² }	65.5 min. {0.65 min.}	less than 65.5 {less than 0.65 }	less than 65.5 {less than 0.65 }	less than 65.5 {less than 0.65 }
	Distillation characteristics Initial boiling point °C End point °C	- 250 max.	- 250 max.	100 max. Exceeding 250	Exceeding 100 Exceeding 250
Preparation of tester	Thermometer (thermometer number and thermometer symbol of JIS B 7410)	7 (DIST)	7 (DIST)	7 (DIST)	8 (DIST)
	Diameter of hole of flask support board mm	37.5	37.5	50	50
	Temperature of distillation flask and thermometer at beginning of test °C	13 to 18	13 to 18	13 to 18	Don't exceed room temperature.
	Temperature of flask support board and shield at beginning of test °C	Room temperature max.	Room temperature max.	Room temperature max.	-
	Temperature of receiver and sample at beginning of test °C	13 to 18	13 to 18	13 to 18	13 to room temperature
Conditions during test	Temperature of condensation bath tank °C	0 to 1	0 to 4	0 to 4	0 to 60 ⁽²⁾
	Temperature of receiver cooling bath tank °C	13 to 18	13 to 18	13 to 18	Sample temperature ± 3

Table 3. (Continued)

Test condition		Class of applied sample			
		Class 1	Class 2	Class 3	Class 4
Condi- tions during test	Time from beginning of heating until reaching initial boiling point min	5 to 10	5 to 10	5 to 10	5 to 15
	Time from initial boiling point until reaching percent recovered 5 volume % s	60 to 75	60 to 75	-	-
	Mean percent recovered per minute from 5 volume % recovered temperature until volume of residue in distillation flask becomes approximately 5 ml ml/min	4 to 5	4 to 5	4 to 5	4 to 5
	Time from approximately 5 ml in volume of residue in distillation flask until reaching the end point min	3 to 5	3 to 5	5 max.	5 max.

Notes (¹) This is in accordance with JIS K 2258.

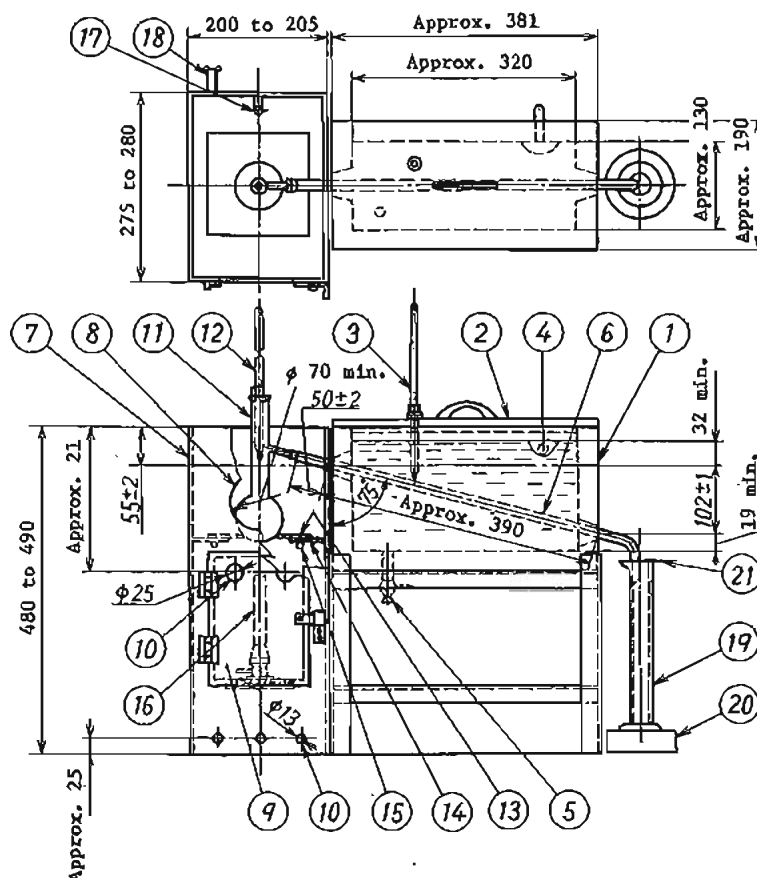
(²) As to temperature of condensation bath tank, corresponding to percent recovery of wax of sample and distillate oil, select the minimum temperature not to condensate the wax in condenser tube between 0°C to 60°C.

Remarks: In the case of carrying out determination of distillation characteristics of sample belonging to class 3 of Table 3, when thermometer 7 (DIST) is not adequate, thermometer 8 (DIST) may be used.
Further, relating to this, Note (¹³) of 5.6 (3) should preferably be referred.

5.2 Ordinary pressure method distillation tester The tester consists of each part of (I) to (8). An example of tester is shown in Fig. 1 (type I) and Fig. 2 (type II).

Further, the insulating performance of type II ordinary pressure method distillation tester, when the insulating resistance between the power source terminal and the case under the condition of closed electric circuit is measured, shall be not less than 5 MΩ. However, in the case of containing the heating circuit, it may be not less than 0.5 MΩ.

Fig. 1. Ordinary pressure method type I distillation tester
(An example) Unit: mm

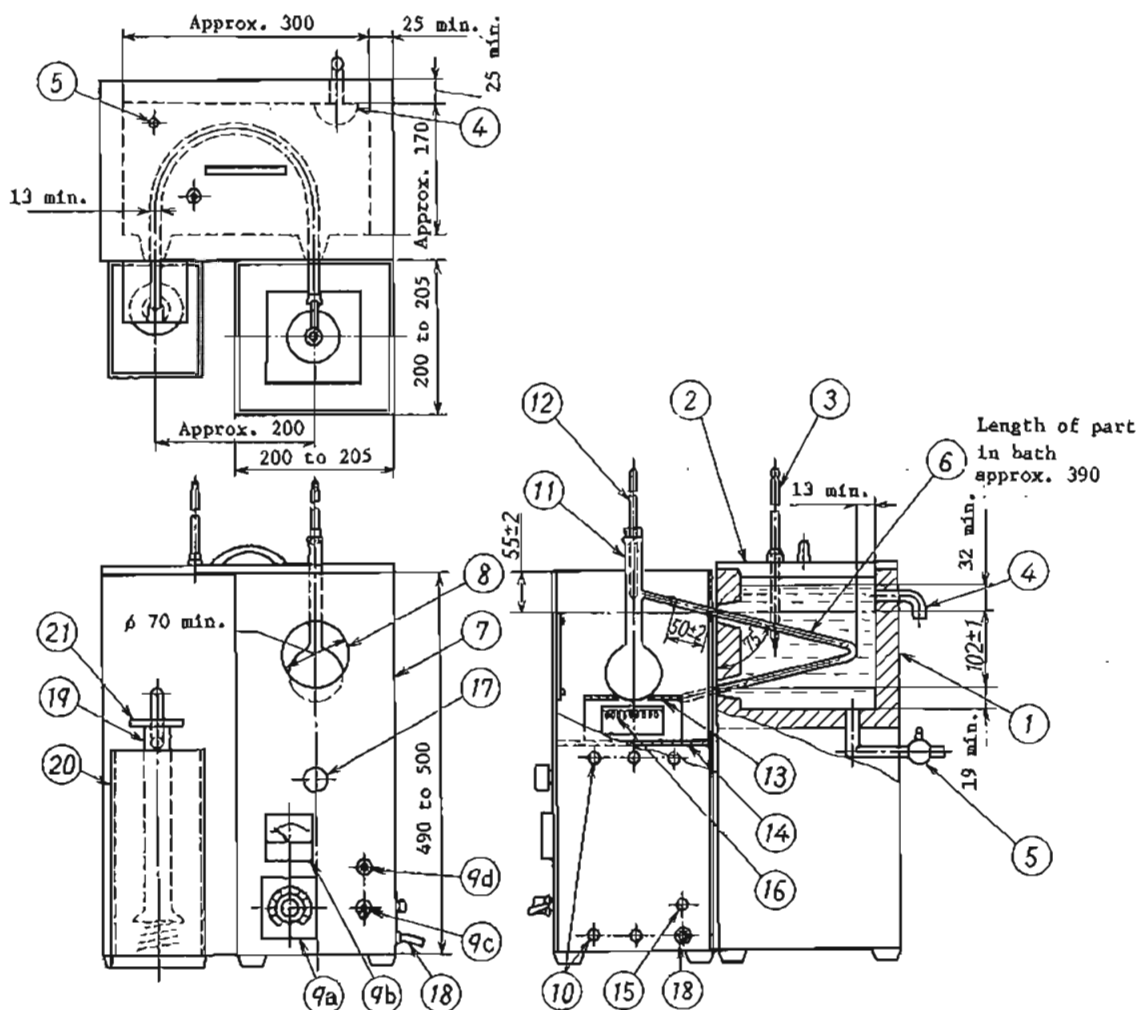


- | | |
|-------------------------------------|--------------------------|
| ① Condensation bath tank | ⑪ Distillation flask |
| ② Cover | ⑫ Thermometer |
| ③ Thermometer for condensation bath | ⑬ Flask support board |
| ④ Spill opening | ⑭ Flask support platform |
| ⑤ Exhaust water cock | ⑮ Ring support |
| ⑥ Condenser tube | ⑯ Gas burner |
| ⑦ Shield | ⑰ Stand |
| ⑧ Peep window | ⑱ Gas hose |
| ⑨ Door | ⑲ Receiver |
| ⑩ Vent hole | ⑳ Receiver stand |
| | ㉑ Filter paper |

Upper part: each two pieces in front and rear

Lower part: each three pieces in four side faces

Fig. 2. Ordinary pressure method type II distillation tester
(An example) Unit: mm



- | | |
|-------------------------------------|---|
| ① Condensation bath tank | ⑪ Distillation flask |
| ② Cover | ⑫ Thermometer |
| ③ Thermometer for condensation bath | ⑬ Flask support board |
| ④ Spill opening | ⑭ Flask support platform |
| ⑤ Exhaust water cock | ⑮ Earth terminal |
| ⑥ Condenser tube | ⑯ Electric heater |
| ⑦ Shield | ⑰ Knob for regulation of flask support platform |
| ⑧ Peep window | ⑱ Power source cord |
| ⑨a Heating regulator | ⑲ Receiver |
| ⑨b Voltmeter (ammeter) | ⑳ Receiver cooling bath tank |
| ⑨c Power source switch | ㉑ Filter paper |
| ⑨d Display lamp | |
| ⑩ Vent hole | |

- (1) Distillation flask The distillation flask of Fig. 42 specified in JIS K 2839.

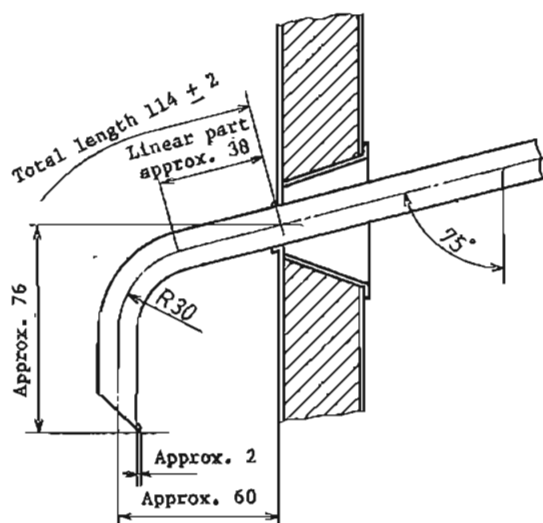
Remarks: The distillation flask is uniform in wall thickness of bulb part and its tube axis of branch tube passes through head tube axis. Particularly, in the case of measuring the dry point, it is required to select that uniform in wall thickness of bulb part.

- (2) Condenser tube and condensation bath tank Those of shape and dimensions shown in Fig. 1 and Fig. 2. Even those of shapes other than these, if the test results can be obtained within the accuracy specified in 5.8, those may be used.

- (a) Condenser tube The condenser is to be seamless brass tubing of 560 ± 2 mm in length, 14 ± 0.5 mm in outer diameter and 0.8 mm to 0.9 mm in wall thickness, and is so attached to the condensation bath tank that its approximately 390 mm is immersed in condensation bath tank, upper end 50 mm and lower end 114 mm go outside the bath tank. The upper end part outside the bath tank is to be of a linear state and of inclination of 75 degrees to vertical line. The lower end part outside the bath tank is cut down in inclination at a sharp angle and bent downward as shown in Fig. 3. The tube end is bent slightly to the inside so that when the receiver is attached the tube end can contact the inside wall of receiver at a position of 25 mm to 32 mm. The part at inside of condensation bath tank is made at a linear state (Fig. 1) or a smooth curved state (Fig. 2), in either case, the mean inclination angle is 75 ± 1 degrees to the vertical line and the difference in height between the inlet and outlet is made 102 ± 1 mm.

Fig. 3. Lower end of condenser tube

Unit: mm



- (b) Condensation bath tank The condensation bath tank is the liquid bath tank made of corrosion resistant metal having insulated walls, provided with the cover. At the attaching part of condenser tube, make the upper part 32 mm or more from the tube axis of condenser tube to bath liquid surface, and make the lower part 19 mm or more to bottom of bath tank, and at other part make the distance between the condenser tube outer periphery and the bath tank wall 13 mm or more. Under the using condition, make the quantity of bath liquid 5.5 l (for plural type, 5.5 l per one piece of condenser tube) or more. To the cover, provide a thermometer insertion opening for condensation bath tank and the like.

Remarks 1. In the case of condensation bath tank of cool water circulating system, the capacity may not satisfy this condition, but take sufficiently into consideration the position and sizes of the cool water inlet and outlet (spill opening) so as to be capable of keeping approximately uniform the bath temperature in the specified temperature range.

2. Out of Class 4, the tester for testing a sample much in wax content is to be provided with the condensate heater and regulator of approximately 1.5 kW in consumption power.

- (3) Shield The shield is to be for type I (gas heating) or type II (electric heating).

- (a) Shield for type I (Refer to Fig. 1) The shield shall be 480 mm to 490 mm in height, 275 mm to 280 mm in depth, 200 mm to 205 mm in width, and approximately 0.8 mm in thickness made of metal, adhered with a heat resistant adiabatic board⁽³⁾ of 1.5 mm to 3.0 mm in thickness on the inside surface.

Provide a door at the front face and drill two air vents of 25 mm in diameter at a position of approximately 215 mm from the upper end to the door and back face⁽⁴⁾.

To the middle part of the side face (condensation bath tank side), drill an elliptical hole so as to be capable of inserting the upper end (protruding part) of condenser tube, at a position of 55 ± 2 mm from the upper end.

Further, drill three air vents of 13 mm in diameter respectively at positions above by approximately 25 mm from the lower end of each surface⁽⁴⁾.

Provide such a stand that it is capable of attaching the gas burner and flask support platform specified in (4) at the inside center of the shield. Make the heights of flask support platform and gas burner capable of suitable adjustment.

The gas burner is to be capable of supplying a heat quantity of not less than 63 kJ {15 kcal} per min and capable of regulating heating freely, and when the cock of the burner is closed and the air pressure of 10 kPa {0.1 kgf/cm²} is added, leakage from either parts must not exist.

Notes (³) Ceramic sheets and the like are used.

(⁴) The venting is to be carried out only from these holes.

Remarks: At the front of shield, a peep window (round shape or square shape of 70 mm or more in size) made of heat resistant glass should preferably be provided at a position where the distillation flask is easily visible.

- (b) Shield for type II (Refer to Fig. 2) The shield shall be of 490 mm to 500 mm in height, 200 mm to 205 mm in depth, 200 mm to 205 mm in width, made of metal approximately 0.8 mm in thickness, adhered with a heat resistant adiabatic board(³) of 1.5 mm to 3.0 mm in thickness.

Provide a peep window (round shape or square shape of 70 mm or more in size is suitable) made of heat resistant glass at a position where the distillation flask is easily visible. At the middle part of the back face, drill an elliptical hole to be capable of inserting the upper end (extruding part) of condenser tube at a position of 55 ± 2 mm from the upper end.

At the inside of the shield attach the electric heater, flask support platform specified in (4), etc., and provide with an apparatus capable of adjusting their height from the outside.

Further, at the front, provide a knob for adjusting of flask support platform, heat regulator knob, voltmeter (or ammeter), switch, etc.

The electric heater is to be of such structure that it is capable of collecting the heating at the central point as far as possible, less in heating delay, and capable of regulating the consuming power at 0 W to 1000 W by the heating regulator.

Remarks 1. As to shield for type II, the vent hole specified for type I is not required, but for protecting the heat regulator or the like from over heat the vent hole may be provided.

Further for attaching a heat regulator or the like if there is no interference, the shield excluding approximately 50 mm of the lower part of shield may be used.

2. In case of an ignition accident due to rupture of distillation flask or the like, for the purpose of preventing the flowing out of spilled sample, the bottom of the shield may be closed or the shield may be placed on a shallow angular dish made of metal.

However, for type I, due to this the vent must not be interfered with.

- (4) Flask support platform The flask support platform is for the purpose of supporting the distillation flask at a suitable position, at the inside of the shield and to shut off the rising gas flow from the lower side of shield to prevent influence upon the distillation flask and make for type I (gas heating) or for type II (electric heating).

- (a) Flask support platform for type I (Refer to Fig. 1) The flask support platform consists of a metallic ring support of 100 mm or more in internal diameter and a heat resistant adiabatic board⁽⁵⁾ of such size as it adheres tightly to the inside surface of shield for type I, drilled with a hole of 76 mm to 100 mm in diameter at its center, having a thickness of 3 mm to 6 mm.

Attach the metallic ring support to the stand at inside of shield to be made adjustable up and down. Attach the heat resistant adiabatic board⁽⁵⁾ horizontally on the metallic ring support, and when the flask support board specified in (5) is placed on this adiabatic board, there will be no clearance between the two sheets.

Notes ⁽⁵⁾ Ceramic fiber formed member, gypsum board, heat resistant porcelain ware, etc. are used. Especially, those which when the part near the central hole is ignited, are difficult in generating rupture due to thermal expansion, chemical variation, etc. are preferable.

- (b) Flask support platform for type II (Refer to Fig. 2) The flask support platform is such suitable mechanism that a heat resistant adiabatic board⁽⁵⁾ of size and of a degree such that it adheres tightly to the inside of shield for type II, of 5 mm to 8 mm in thickness is attached horizontally and the height can be adjusted from outside of the shield.

On the flask support platform, the electric heater specified in (3)(b) is attached.

Remarks: Cover the upper surface of the electric heater with a flask support board specified in (5).

- (5) Flask support board The flask support board of heat resistant adiabatic board⁽⁵⁾ of 5 mm to 6 mm in thickness, of square of 120 mm to 150 mm in length of one side, and at its center the hole shown in Table 4 is drilled.

Table 4. Flask support board

Unit: mm

Applicable sample	Diameter of hole
Those belonging to class 1 and class 2	37.5
Those belonging to class 3 and class 4	50

Remarks 1. At the middle of upper surface of heat resistant adiabatic board⁽⁵⁾ of 20 mm to 25 mm in thickness, a hole of 3 mm to 6 mm in depth and of diameter of dimension of Table 4 is provided and the lower part of this hole may be broadened to 76 mm to 100 mm in diameter.

2. Heating of distillation flask shall be carried out through the hole of this flask support board.

- (6) Thermometer The thermometer of No. 7 (DIST) or thermometer No. 8 (DIST) specified in JIS B 7410.
- (7) Receiver The receiver shown in Fig. 43 specified in JIS K 2839.
- (8) Receiver cooling bath tank The receiver cooling bath tank is such a water bath tank that at a position of 25 mm to 32 mm from the upper end of receiver, when placing the receiver so as the lower end of condenser tube to contact the inside wall of receiver, and immersing the receiver into the bath liquid up to the graduation line of 100 ml at least, can see it transparently, and provided with the mechanism capable of maintaining the receiver at the specified position.

5.3 Check of tester The check of ordinary pressure method distillation tester shall be as follows:

- (1) Prepare two pieces of clean, dried receiver, weigh out each component of quantity given in Table 5 by using burette or pipette to mix and prepare each 100 ml of standard samples A and B.

Informative Reference: The standard sample A corresponds to the samples belonging to class 1 and class 2 of Table 3, and the standard sample B, to the samples belonging to class 4 respectively.

- (2) Distill the standard samples A and B according to the specifications of 5.5 to 5.7, and if the obtained results coincide with the standard distilling temperature in the permissible range given in Table 6, consider the tester and test operation to be normal.

Table 5. Components of standard sample and the mixing ratio

Unit: ml

Component(⁶)	Standard sample A	Standard sample B
Hexane	20	-
Heptane	20	-
Toluene	20	-
Xylene	20	20
2-Ethyl hexanol	10	20
Tetralin	10	20
α -methyl naphthalene	-	20
Hexadecane	-	20

Note (⁶) Guaranteed grade of JIS reagent or that equivalent thereto is used.

Table 6. Standard recovered temperature(⁶) of standard sample A and standard sample B and the permissible range

Unit: °C

	Standard sample A	Standard sample B
Initial boiling point(⁸)	76.0 $\pm$ 2.0	148.5 $\pm$ 2.0
5 volume % recovered temperature(⁸)	88.5 $\pm$ 1.5	160.5 $\pm$ 1.5
10 volume % recovered temperature	92.5 $\pm$ 1.5	166.0 $\pm$ 1.5
20 volume % recovered temperature	97.5 $\pm$ 1.0	175.0 $\pm$ 1.5
30 volume % recovered temperature	102.5 $\pm$ 1.0	184.5 $\pm$ 1.5
40 volume % recovered temperature	109.5 $\pm$ 1.5	196.5 $\pm$ 1.5
50 volume % recovered temperature	116.5 $\pm$ 1.5	208.0 $\pm$ 1.5
60 volume % recovered temperature	126.0 $\pm$ 1.5	221.5 $\pm$ 1.5