

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing Methods for
Water Content of Crude Oil
and Petroleum Products**

JIS K 2275—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.

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Testing Methods for
Water Content of Crude Oil
and Petroleum Products

K 2275-1989

1. Scope

This Japanese Industrial Standard specifies the methods of quantitative determination for water content of crude oil and petroleum products.

2. General Matters

The general matters for this Standard shall be in accordance with the following.

- (1) The general matters in common to Karl Fischer type volumetric titrimetric method and Karl Fischer type coulometric titrimetric method shall be in accordance with JIS K 0113.
- (2) The rounding off of numerical values shall be in accordance with JIS Z 8401.
- (3) Sampling shall be in accordance with the sampling method of the second sample specified in JIS K 2251 or the method equivalent to it.

3. Classification of Test Methods

The test methods shall be classified into three methods as shown in Table 1.

Applicable Standards, Corresponding International Standards and
Reference Standards: See page 27.

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Table 1. Classification of Methods to Test Water Content

Test method	Oil to be applied	Range to be measured (volume or mass)	Remark
Distillation method	Crude oil and petroleum products	0.05 % or more	1. These are inapplicable to the sample (for example, asphalt emulsion) containing additives usable to emulsify or to stabilize water and oil. 2. Volatile and water-soluble substance (for example, alcohols) is determined as water content.
Karl Fischer type volumetric titrimetric method	Crude oil and petroleum products	Crude oil: 0.02 % or more Petroleum products: 20 ppm or more ⁽²⁾	3. The crude oil containing 150 mass ppm or more of total of mercaptan sulfur and hydrogen sulfide can not be applied.
Karl Fischer type coulometric titrimetric method			4. These are inapplicable to the petroleum products containing additives with the substance ⁽¹⁾ (hereafter referred to as an interfering substance), except water, which easily reacts with Karl Fischer reagent. In case of containing interfering substance, when using an evaporating apparatus of water, the specimen, of which the evaporated gas does not contain an interfering substance, may be tested through a water-content evaporating apparatus.

Notes ⁽¹⁾ Interfering substances are as follows.

(1) Free alkali, oxidizing substance, reducing substance, substance easily reacting with iodine such as mercaptan.

(2) Sulfur dioxide, substance reacting with pyridine or methanol to produce water.

⁽²⁾ When petroleum products containing water less than 20 ppm, the more quantity of the sample may be applied.

Remark: When water content in the sample is tested, it is preferable to choose the method according to the kind of oil, anticipated water content concentration, and the presence of interfering substance.

The testing method using hydride reaction for determining water content of crude oil is appended in Informative Reference [Testing Method for Water Content in Crude Oil (Reaction Method with Hydride)]

Informative

Reference: One mass ppm of mercaptan sulfur and 1 mass ppm of hydrogen sulfide in the sample cause a positive error corresponding to 0.3 mass ppm and 0.6 mass ppm of water respectively.

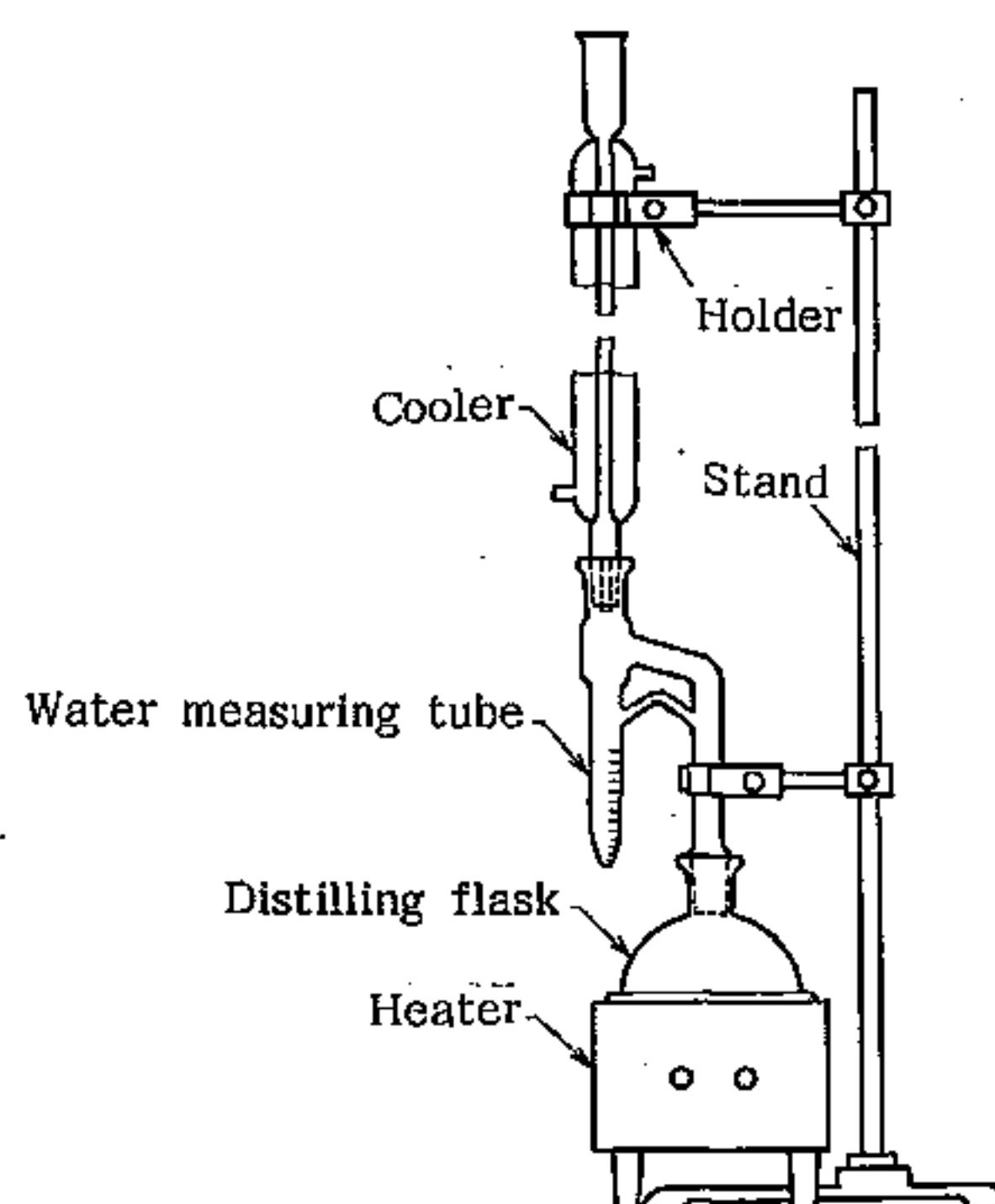
4. Distillation Method

4.1 Outline of Testing Method Put the sample in a distillation flask, and after adding solvent insoluble in water, reflux it while heating. Condensed water and solvent will separate into two layers after successively being trapped in a water-measuring tube, and the solvent returns back in the distilling flask and the water stays in the graduated part of the tube. The water content in the sample shall be obtained from both the water quantity trapped in the tube and the mass of the weighed-out sample.

4.2 Water Content Tester for Distillation Method The tester is composed of (1) to (3), and Fig. 1 shows an example of its assembly.

The insulating performance of the tester should be 5 M Ω or more when the insulating resistance between the power-source terminal and the housing is measured under closed circuit. In the case of being equipped with a heating circuit, 0.5 M Ω is enough.

Fig. 1. Water Content Tester Used for Distillation Method
(Shown for an Example)



- (1) Distilling Flask The distilling flask as specified in Fig. 83 of JIS K 2839.

The distilling flask with 24/40 taper ground joint is available too.

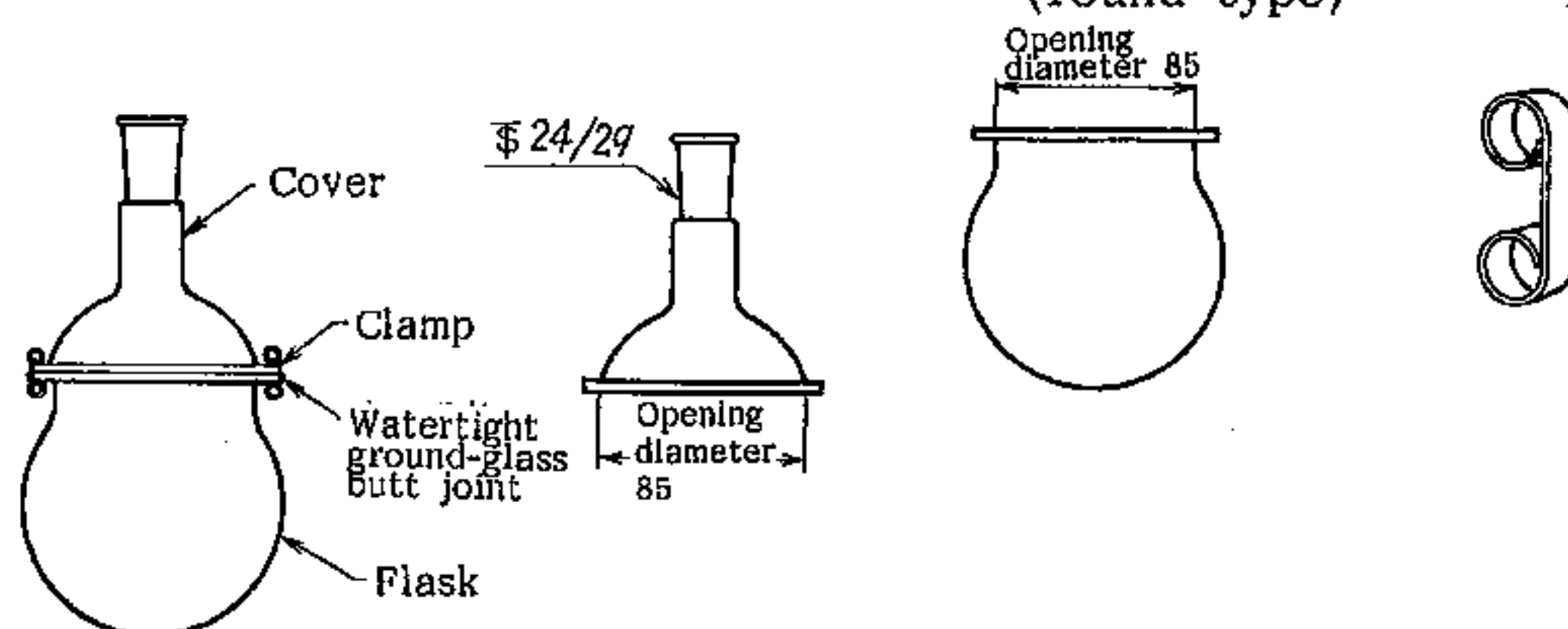
Remark: When a solid or semisolid specimen such as grease is tested, it is allowable to use a separate type distilling flask, as shown in Fig. 2, which is convenient for the operation both of sampling and of washing after the test.

The ground-glass butt joint between the cover and flask shall be watertight.

Fig. 2. Separate Type Distilling Flask (Shown for an Example)

Unit: mm

- (a) Assembly diagram (b) Cover (c) Flask (round type) (d) Clamp



- (2)
- Cooler
- The cooler as specified in Fig. 84 of JIS K 2839.

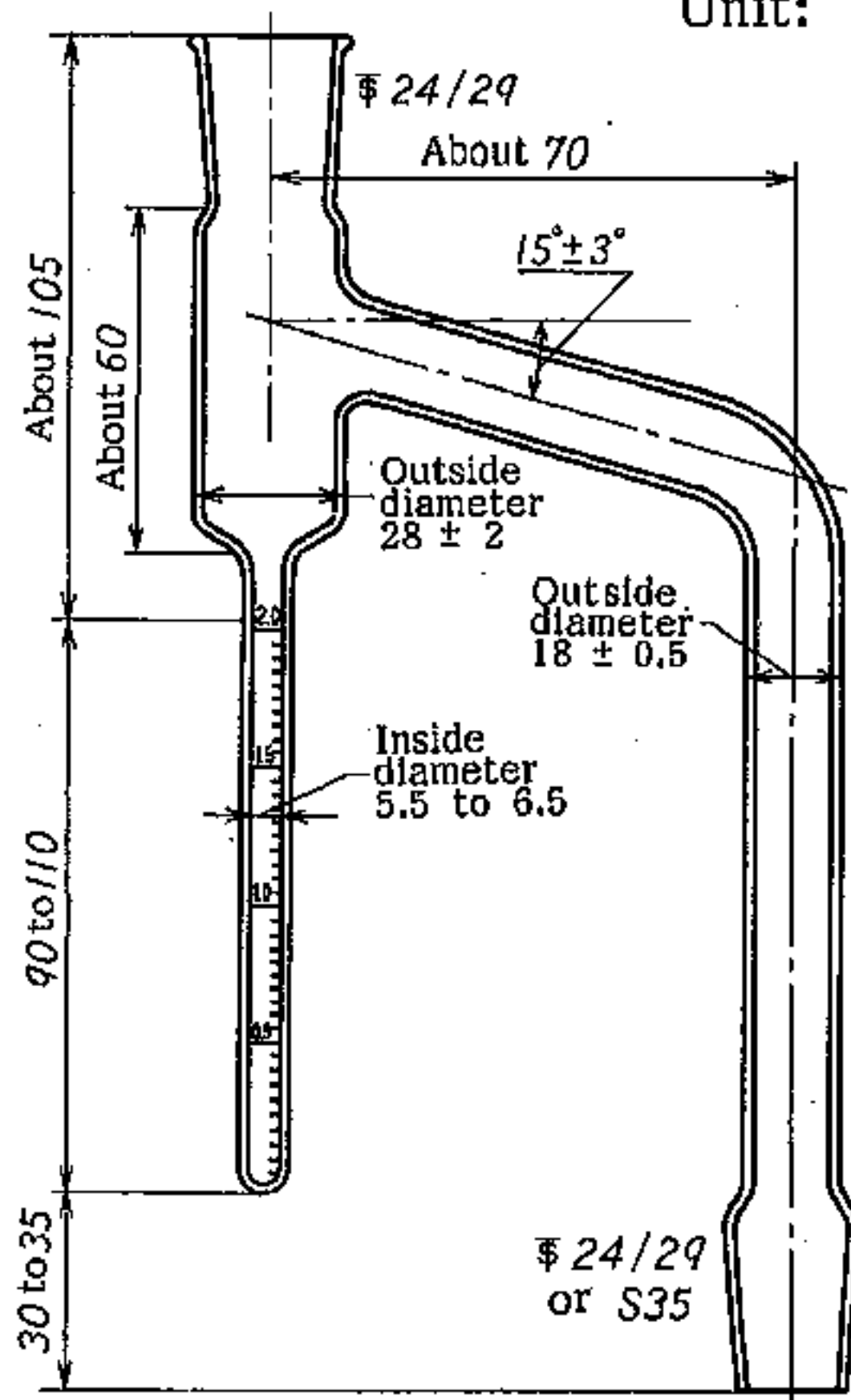
The cooler with lower taper of 24/40 ground joint is available too.

- (3)
- Water Measuring Tube
- The water measuring tube as specified in the following (a) to (c).

- (a)
- 2-ml Water Measuring Tube
- The tube is made of Grade 1 hard glass, equipped with the graduation at every 0.05 ml in scale interval on the graduated place, and its shape and dimension are shown in Fig. 3.

Fig. 3. 2-ml Water Measuring Tube

Unit: mm



Graduation at 20°C

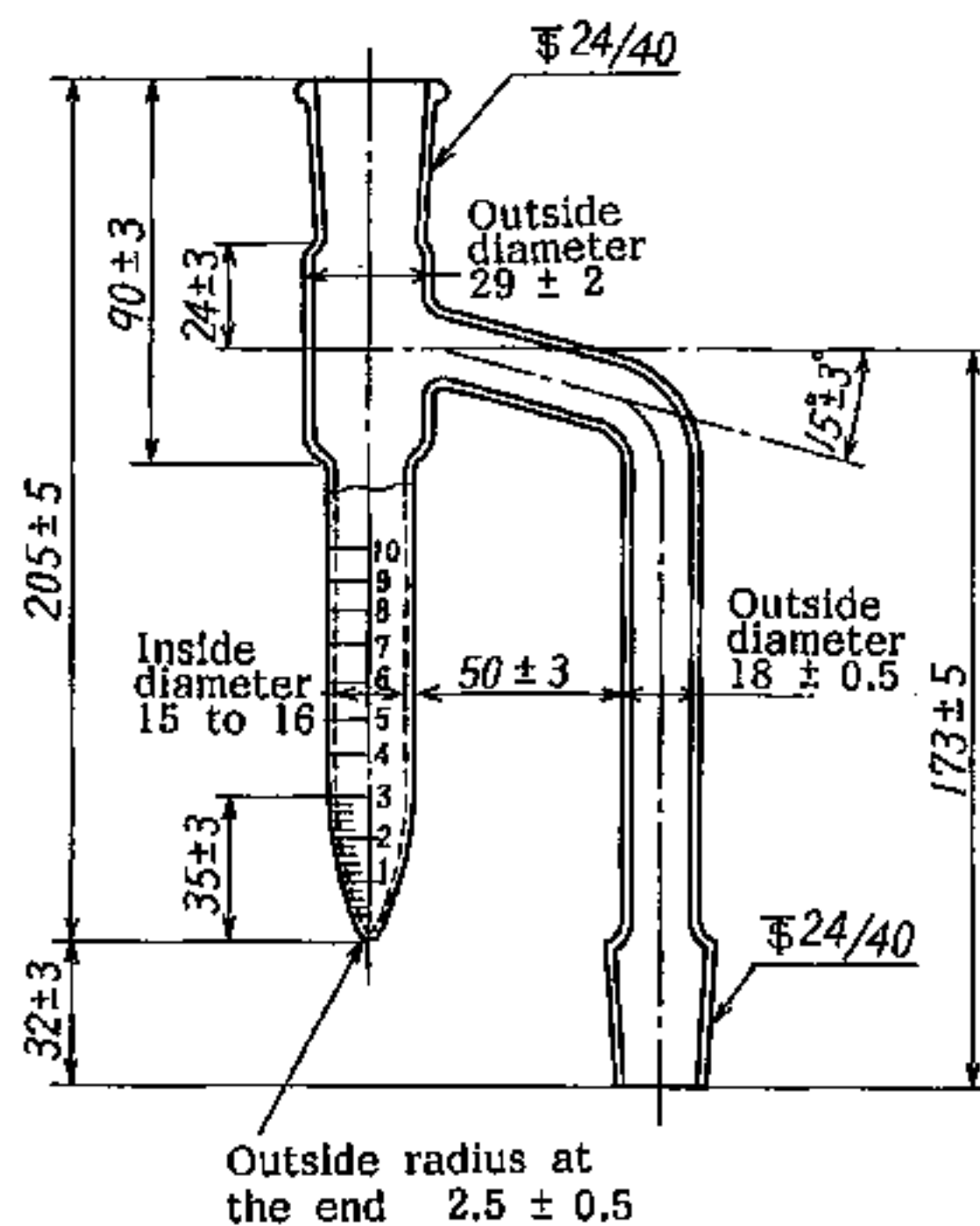
Unit: ml

Range of graduation	Long graduated line	Scale interval	Tolerance on scale	Scale figures
0.00 to 2.00	0.50	0.05	0.05 max.	0.50

- (b) 10-ml Water Measuring Tube The 10-ml water measuring tube shall be as specified in Fig. 85 and Fig. 87 of JIS K 2839.
The tube shown in Fig. 4 is available too.

Fig. 4. 10-ml Water Measuring Tube

Unit: mm



Graduation at 20°C

Unit: ml

Graduation range	Large graduation line	Scale interval	Tolerance on scale	Scale figures
Region of 1.0 or less	1	0.1	0.05 or under	1
Region exceeding 1.0	At every unit	0.2	0.1 or under	At every unit

- (c) 25-ml Water Measuring Tube 25-ml water measuring tube as specified in Fig. 86 and Fig. 88 of JIS K 2839.
The tube specified in Fig. 5 is available.

Remark: The tube with a cock at the lowest end of the graduated part to drain collected water may be available. Fig. 6 shows the schematic diagram of that tube.

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Fig. 5. 25-ml Water Measuring Tube

Unit: mm

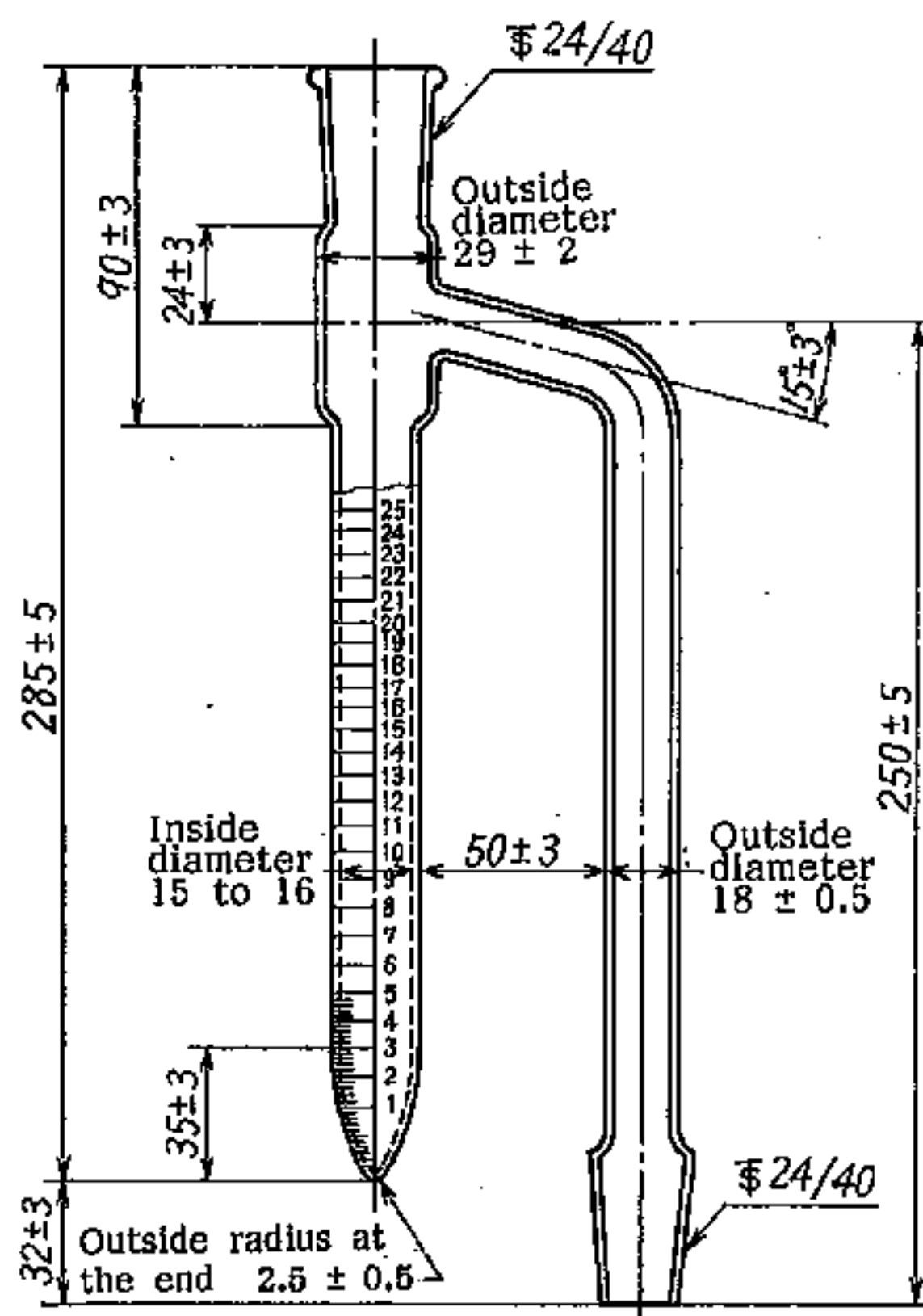
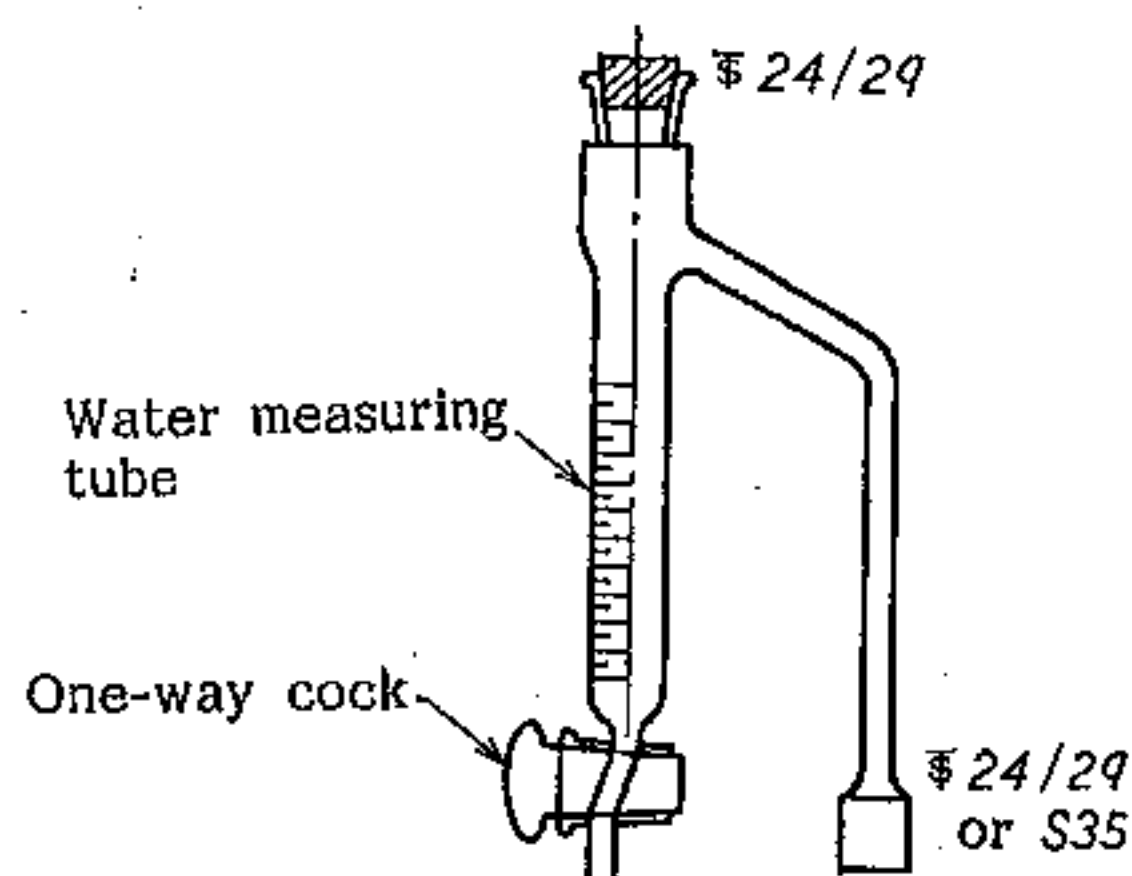


Fig. 6. Water Measuring Tube with a Cock (Schematic Diagram)



Graduation at 20°C

Unit: ml

Graduation range	Large graduation line	Scale interval	Tolerance on scale	Scale figures
Region of 1.0 or less	1	0.1	0.05 max.	1
Region exceeding 1.0	At every unit	0.2	0.1 max.	At every unit

4.3 Solvent The solvent containing no water is suitable.

The examples of solvents used according to the type of oil are shown in Table 2.

4.3.1 Aromatic Solvent

- (1) Xylene The 3 degree xylene specified in JIS K 2435 or the grade 1 specified in JIS K 8271.
- (2) Mixed Solvent of Toluene-xylene The toluene specified in JIS K 8680 or the mixture of 20 volume % No. 1 pure toluene specified in JIS K 2435 and 80 vol % of xylene.
- (3) Solvent Naphth Water-free petroleum naphth or coaltar naphtha with 5 volume % or less distillate at 125°C and 20 volume % or more distillate at 160°C, and having a density (15°C) of 0.8546 g/cm³ or more.

4.3.2 Distilled Oil from Petroleum System With 5 volume % distillate at 90 to 100°C boiling point range and 90 % distillate at 210°C or lower.

4.3.3 Volatile Spirit Solvent

- (1) Petroleum-system Distillate with Narrow Boiling Point Range
Petroleum-system distillate having a boiling point within the range from 100 to 120°C.
- (2) Isooctane Isooctane of 95 volume % in purity or higher.

Remarks 1. When it is difficult to obtain petroleum-system distillate and the petroleum-system distillate of narrow boiling point range, the distillate, which has above-mentioned boiling point range and can be obtained by the distillation of the industrial gasoline specified in JIS K 2201, can be used instead.

2. Isooctane specified in JIS K 2280 can be serviceable.

Table 2. Solvent Usable According to Type of Oil (Example)

Type of oil	Solvent
Crude oil	Xylene
Fuel oil, lubricating oil, petroleum sulfonate	Petroleum system distillate, Xylene
Bituminous matter (such as asphalt)	Aromatic solvent
Grease	Volatile spirit solvent

4.4 Preparation of Test The testing procedures for distillation method shall be as follows.

- (1) Calibration of Tester Whether the apparatuses are correctly assembled or not shall be confirmed by the following operation: place clear hydrocarbon oil and solvent in a distilling flask according to 4.5 (1); add water accurately by the amount shown in Table 3 using a burette or pipette and mix them well; carry out the operation given from 4.5 (2) to (5); and find the quantity of water collected. If the quantity of collected water falls in the tolerance range specified in Table 3, the assembling of the tester and other conditions are regarded as correct. If it falls out of the tolerance range, because such causes are thought as the leakage of steam, too fast rate of heating, inaccurate graduation of the water measuring tube, penetration of water from outside, and so on, the retrial of the calibration shall be carried out after the removal of these causal factors.

Table 3. Tolerance on Calibration of the Tester

Type of water-measuring tube	Amount of water added in a distilling flask (20°C) ml	Tolerance range of of collected water (20°C) ml
2-ml water measuring tube	1.00	1 ± 0.05
10-ml and 25-ml water measuring tubes	1.0	1 ± 0.1
	5.0	5 ± 0.2
	12.0	12 ± 0.2

- (2) Preparation of Sample After homogenizing the sample as shown in the following process, prepare the sample for test. If homogenizing the sample is difficult, carry out two times or more measuring, and average measured values to determine the test result.

- (a) Mix by shaking satisfactorily the sample that is liquid. If necessary, mix by shaking after heating.
- (b) In the case of fragile solid sample (for example, asphalt), smash and mix it satisfactorily.

4.5 Procedure of Test The procedures for the test of distillation method shall be as follows:

- (1) Weigh the sample with ± 1 % accuracy of the sample quantity ⁽³⁾ specified according to anticipated water content into a distilling flask as shown in the following, and put glass beads or zeolite to prevent bumping in the flask.

(a) Case of Fluid Sample Take the sample using a measuring cylinder in terms of volume. Rinse the cylinder first with 50 ml solvent once, then with 25 ml two times to remove the sticking sample from the wall of the cylinder, and transfer completely these washings into the distilling flask.

(b) Case of Solid or Sticky Sample Weigh the sample directly into a distilling flask, and add 100 ml solvent.

Note (³) The quantity of sample should preferably be 200 ml or 200 g for the sample containing the anticipated water content less than 0.1 %, and 100 ml or 100 g for the sample of 0.1 % or more.

In the case of the sample containing much water, less amount of the sample may be taken.

Remark: Since the vapor of toluene is harmful, be careful not to inhale it. Especially, toluene is risky when it gets into the eyes, and be careful in handling toluene as well as other solvents.

(2) Choose the water measuring tube (⁴) which is suitable for the anticipated water content in the sample, assemble it firmly as shown in Fig. 1 with special observation not to let stream or liquid escape from joints.

Note (⁴) Choose a water measuring tube paying attention to the following items.

(a) Case of Sample Containing Distillate Not Higher Than 100°C Such as Crude Oil The tube, whose uppermost graduation line is not exceeded by the distillate of not higher than 100°C, should be choosed.

(b) Case of the Sample Abounding in Water Content The tube, whose uppermost graduation line is not exceeded by the water collected, should be chosen. The employment of the water measuring tube with a cock, however, shall be free of this limitation.

Remark: The inside wall of a cooling tube and a water measuring tube shall be clean enough for condensed water to flow down freely into a graduated part.

(3) Stuff cotton slightly in the top of a cooler, and after passing water in the jacket of the cooler, start heating the distilling flask.

(4) Adjust the heating of the distilling flask so that the condensed liquid falls at a rate of 2 to 5 drops per second from the lower end of the cooler. Continue the distillation until no water is observed in any part other than the water measuring tube and the collected water quantity remains unchanged for 5 minutes or longer.

If rings of water sticking to the cooler tube are observed, either increase the distilling rate carefully or stop the passing of cooling water for a few minutes.