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JAPANESE INDUSTRIAL STANDARD

**Testing Methods for Oxidation
Stability of Lubricating Oils**

JIS K 2514—1982

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JAPANESE INDUSTRIAL STANDARD

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Testing Methods for Oxidation Stability of
Lubricating Oils

K 2514-1982

1. Scope

This Japanese Industrial Standard specifies the testing methods for oxidation stability of lubricating oils for internal combustion engines, turbine oils, and the like.

Remark: The units and numerical values given in { } in this standard are based on the International System of Units (SI) and are appended for reference.

2. Classification of Testing Methods

The testing methods for oxidation stability of lubricating oils shall be classified into three types given in Table 1.

Table 1. Types of Testing Methods

Test method	Applicable type of oil (for example)
Test method for oxidation stability of lubricating oils for internal combustion engines	Lubricating oils for internal combustion engines
Test method for oxidation stability of turbine oils	Turbine oils (containing additives)
Test method for oxidation stability using rotating bomb	Turbine oils

3. Test Methods3.1 Test Method for Oxidation Stability of Lubricating Oils for Internal Combustion Engines

3.1.1 Summary of Test Method A catalyzer and a varnish stick shall be immersed in a test specimen, and after oxidizing the specimen by stirring it with a stirring rod at a prescribed temperature for a prescribed time, the kinematic viscosity and total acid value of the oxidized oil shall be measured and the values obtained shall be compared with those of an unoxidized oil.

In addition, the condition of attachment of lacquer-like matter or sludge adhering onto the varnish stick shall be examined.

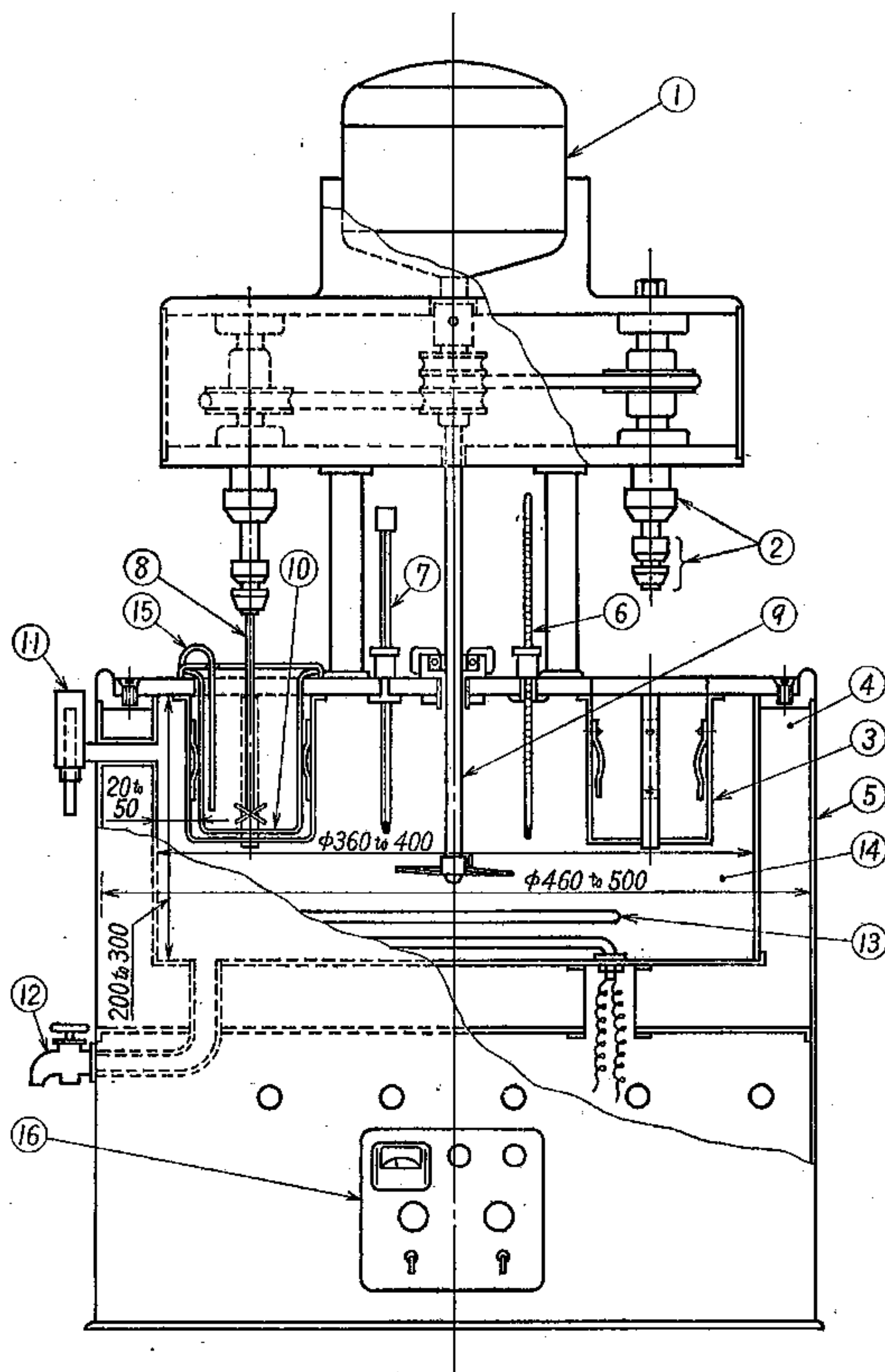
3.1.2 Testing Apparatus for Oxidation Stability of Internal Combustion Engine Lubricating Oils The apparatus shall have the construction shown in Fig. 1 and shall consist of the components of (1) to (6).

Applicable Standards: See page 39.

Fig. 1. Testing Apparatus for Oxidation Stability of Internal Combustion Engine Lubricating Oils (Shown for Example)

With 6 Specimens on

Unit: mm



- | | |
|-----------------------------|----------------------|
| ① Electric motor | ⑨ Stirrer |
| ② Rotor | ⑩ Specimen container |
| ③ Specimen container holder | ⑪ Oil level adjuster |
| ④ Heat insulating material | ⑫ Discharge cock |
| ⑤ Heat insulating wall | ⑬ Electric heater |
| ⑥ Thermometer | ⑭ Bath |
| ⑦ Temperature regulator | ⑮ Varnish stick |
| ⑧ Specimen stirring rod | ⑯ Control panel |

(1) Constant Temperature Bath

- (a) Bath The bath shall be made of metal and have heat insulating walls capable of maintaining a maximum temperature of 200 °C.
- (b) Cover The cover shall have 4, 6 or 8 specimen container inserting holes, specimen container holders, and a thermometer holder.
- (c) Specimen Container Holders The specimen container holders shall be made of metal, have the shape shown in Fig. 1, and be capable of suspending specimen containers at a position about 120 mm below the upper surface of the bath cover and of being immersed in the bath liquid to a depth of 90 mm or more.
- (d) Stirrer The stirrer shall be of the electric motor driven type.
- (e) Electric Heater and Temperature Regulator The electric heater shall be capable of raising the temperature of the constant temperature bath to 200 °C within 60 minutes, and the temperature regulator shall be capable of maintaining the temperature within ± 0.5 °C of the prescribed temperature ranging from 60 to 200 °C.

Remark: The insulation resistance of the heater shall be 5 M Ω or higher when it is measured between the electrically charged portions and the stand by using a 500 V insulation resistance meter. It is permitted that the insulation resistance is 0.5 M Ω or higher when the heating circuit is included.

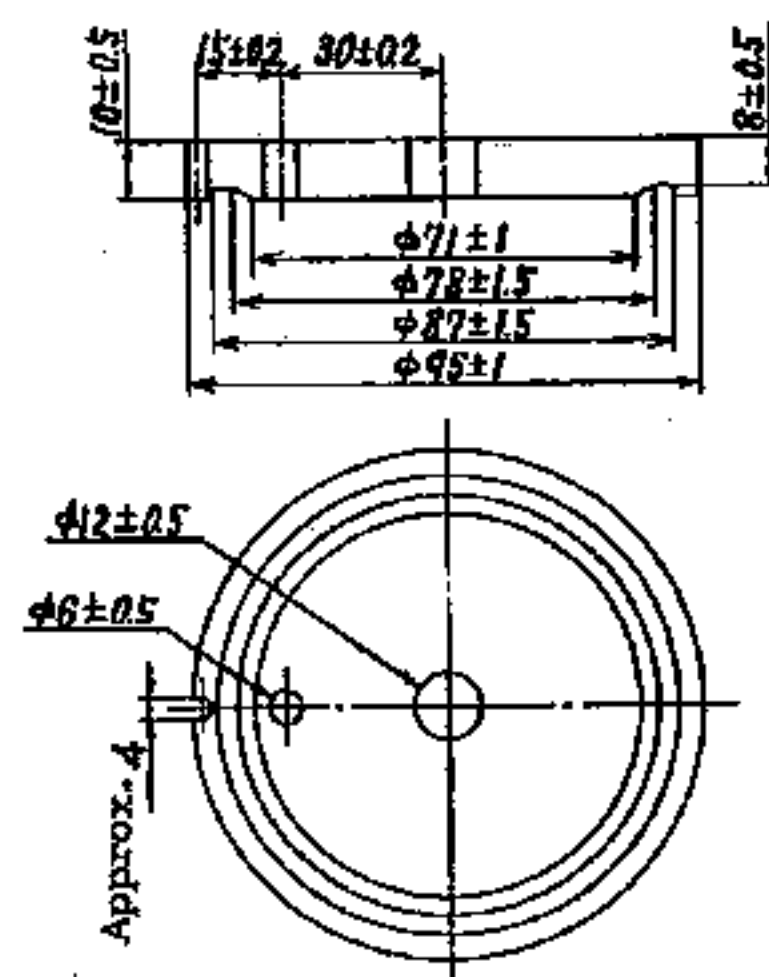
- (2) Thermometer The thermometer shall be No. 73 (ISO) thermometer specified in JIS B 7410.

(3) Specimen Container and Specimen Container Cover

- (a) Specimen Container The specimen containers shall be as specified in Fig. 108 of JIS K 2839.
- (b) Specimen Container Cover The specimen container cover shall be made of phenolic resin or the like having the shape and dimensions given in Fig. 2, each provided with holes for inserting a specimen stirring rod and varnish stick.

Fig. 2. Specimen Container Cover (Shown for Example)

Unit: mm



- (4) Specimen Stirring Rod The stirring rod shall be as specified in Fig. 113 of JIS K 2839, or one made of stainless steel (SUS 304) having the same shape and dimensions as that.
- (5) Rotor The rotor shall have the shape shown in Fig. 1 and such a construction that a specimen stirring rod can be inserted from the bottom and fixed with a chuck, and shall be capable of rotating at a rate of 1300 ± 15 r.p.m. and of producing no deflection at the lower end of the specimen stirring rod.
- (6) Varnish Stick The varnish stick shall be as specified in Fig. 114 of JIS K 2839.

3.1.3 Catalyzer and Other Materials

(1) Catalyzer

- (a) Steel Catalyzer A piece of steel conforming to SPCC specified in JIS G 3141, 0.5 mm in thickness, 26 mm in width, and 121.4 mm in length.
- (b) Copper Catalyzer A piece of copper conforming to C 1100 P specified in JIS H 3100, 0.5 mm in thickness, 26 mm in width, and 60.4 mm in length.

Remark: As shown in Fig. 3, holes (ϕ 1.0 mm) for fixing shall be made at both shoulders of the steel and copper catalyzers.

- (2) Abrasive Cloth Abrasive cloth of fused alumina or No. 400 garnet specified in JIS R 6251.

- (3) Abrasive Paper Abrasive paper of fused alumina or No. 400 garnet specified in JIS R 6252.
- (4) Sanitary Cotton Pharmacopoeial sanitary cotton.
- (5) Heptane Heptane specified in JIS K 2280.
- (6) Naphtha for Precipitation Naphtha specified in JIS K 2503.
- (7) Colour Scale The colour scale shall be as specified in Attached Fig. 2 of Japan Petroleum Institute Standard JPI-5 S-15-80 (Method for Evaluating Land Diesel Engine Pistons).

3.1.4 Preparation for Test The preparation below shall be followed.

- (1) Immerse the specimen container, specimen stirring rod⁽¹⁾, and varnish stick in a detergent or chromic acid mixed solution⁽²⁾ for 2 h or longer, and after rinsing thoroughly them with tap water, wash with water several times, and then dry.

Notes ⁽¹⁾ Specimen stirring rods made of stainless steel, if used, shall be rinsed with another suitable solvent.

⁽²⁾ Where a chromic acid mixed solution is used, an antipollution treatment shall be made after using those articles.

- (2) After wiping thoroughly both catalyzers with sanitary cotton wet with a suitable solvent (heptane, naphtha for precipitation, or the like), polish the catalyzers with abrasive cloth or abrasive paper until fresh surfaces are exposed, and then after removing completely the abrasive powder with dry sanitary cotton, assemble as shown in Fig. 3.

Use the polished catalyzers for the test within as short a time as possible.

At all times handle these polished catalyzer by using cleaned and dried sanitary cotton or cotton gloves to avoid direct contact with the fingers.

- (3) Adjust⁽⁴⁾ the temperature of the constant-temperature bath to $165.5 \pm 0.5^{\circ}\text{C}$ ⁽³⁾.

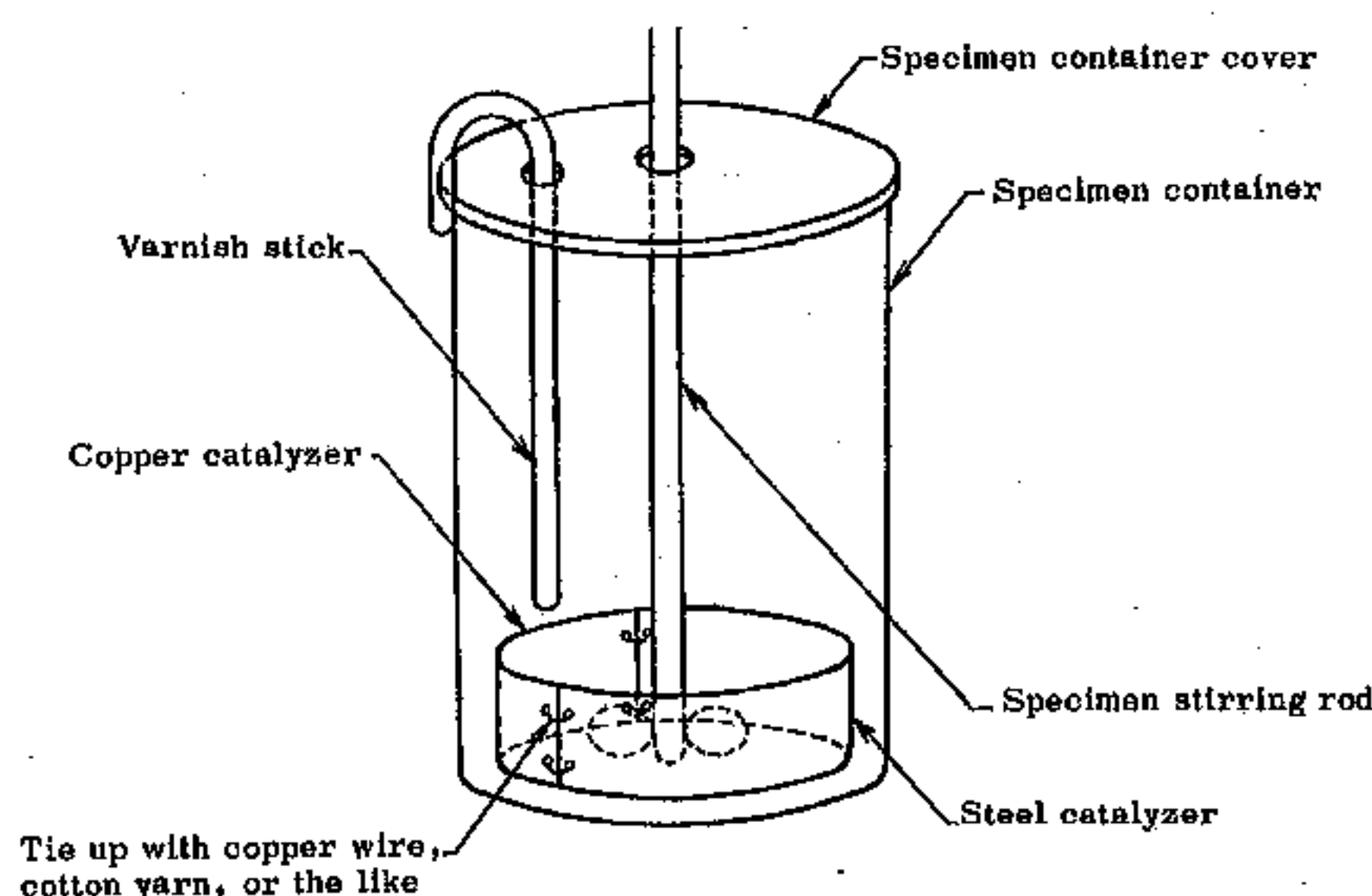
Notes ⁽³⁾ Where an appropriate standard for an individual product stipulates a different temperature, such a temperature shall be used.

⁽⁴⁾ The thermometer shall be inserted as deeply as possible within the range in which the temperature can be read.

3.1.5 Test Procedure The procedure described below shall be followed.

- (1) As shown in Fig. 3, place 250 ml of specimen at room temperature in each of two specimen containers containing catalyzers, and fix those containers to the specimen container holders of the constant temperature bath.

Fig. 3. Method of Assembling Specimen Container and Catalyzers (Shown for Example)



Then insert the specimen stirring rod in the specimen container cover and put the cover on, and after attaching the specimen stirring rod to the rotor so that the lower end of the blade of the stirring rod positions 10 mm above the bottom of the specimen container, attach the varnish stick. In this operation, never allow the fingers to touch directly those portions of the specimen stirring rod and varnish stick which are to be immersed in the specimen.

- (2) Stir the specimen by rotating the specimen stirring rod toward the direction to push down the specimen at 1300 ± 15 r.p.m. for 24 h ⁽⁵⁾.

Note ⁽⁵⁾ Where an appropriate standard for an individual product stipulates a different time, this time shall be used.

- (3) After the lapse of the prescribed time, take the specimen container out of the constant temperature bath, detach the varnish stick, and after taking out the catalyst by using clean tweezers, let the specimen in the specimen container, hereinafter referred to as the "oxidized oil", cool down to room temperature. Promptly carry out the following tests for a specimen before undergoing the oxidation test, hereinafter referred to as the "unoxidized oil", the oxidized oil cooled to room temperature, and the varnish stick.
 - (a) Determine the kinematic viscosity values of the unoxidized and oxidized oils at 40 °C in accordance with JIS K 2283.
 - (b) Determine the total acid values of the unoxidized and oxidized oils by the potential difference titration method specified in JIS K 2501.

- (c) Rinse the varnish stick with naphtha for precipitation, and after removing the oxidized oil, let it cool naturally. Examine the presence of adhering lacquer-like matter or sludge, and if there is any adhering matter, determine and record the colour number corresponding to the degree of colouring of the varnish stick by comparing it with the colour scale.

3.1.6 Calculation and Expression of Results The calculation and the expression of the results shall be made as described below.

- (1) Viscosity Ratio The viscosity ratio shall be obtained from the kinematic viscosity values of the specimen before and after oxidation by the use of the following formula and rounded to the second decimal place.

$$\text{Viscosity ratio} = \frac{D_2}{D_1}$$

where D_1 : kinematic viscosity of unoxidized oil at 40°C (cSt)
{ mm²/s }

D_2 : kinematic viscosity of oxidized oil at 40°C (cSt)
{ mm²/s }

If the difference in test result between two tests does not exceed 14 % of the mean value, the mean value shall be taken as the viscosity ratio.

- (2) Increase of Total Acid Value The difference in total acid value between the specimens before and after oxidation shall be calculated and rounded to the 2nd decimal place. If the difference between two test results does not exceed the tolerance given in Table 2, the mean value of these results shall be taken as the increase of total acid value.

Table 2. Tolerance

Increase of total acid value (mg KOH/g)	Tolerance (mg KOH/g)
0.05 to 1.0 incl.	0.3
Over 1.0 to 5.0 incl.	1
Over 5.0 to 20 incl.	4

- (3) Lacquer Degree The degree of adherence of lacquer-like matter or sludge onto the varnish stick shall be classified by the aid of Table 3 and the result shall be expressed as the lacquer degree.

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Table 3. Criterion on Classification of Lacquer Degree

Lacquer degree	Description
No adherence	No adhering matter is observed on the varnish stick, or the degree of coloring of the varnish stick is lower than colour No. 1
Thinly adhering	The degree of coloring of the varnish stick corresponds to colour Nos. 1 to 2.
Intermediately adhering	The degree of coloring of the varnish stick corresponds to colour Nos. 3 to 5.
Thickly adhering	The degree of coloring of the varnish stick corresponds to colour Nos. 6 to 9.

If the two test results do not agree with each other, the result corresponding to the greater colour number shall be adopted.

3.1.7 Precision No precision is specified.

3.2 Test Method for Oxidation Stability of Turbine Oils

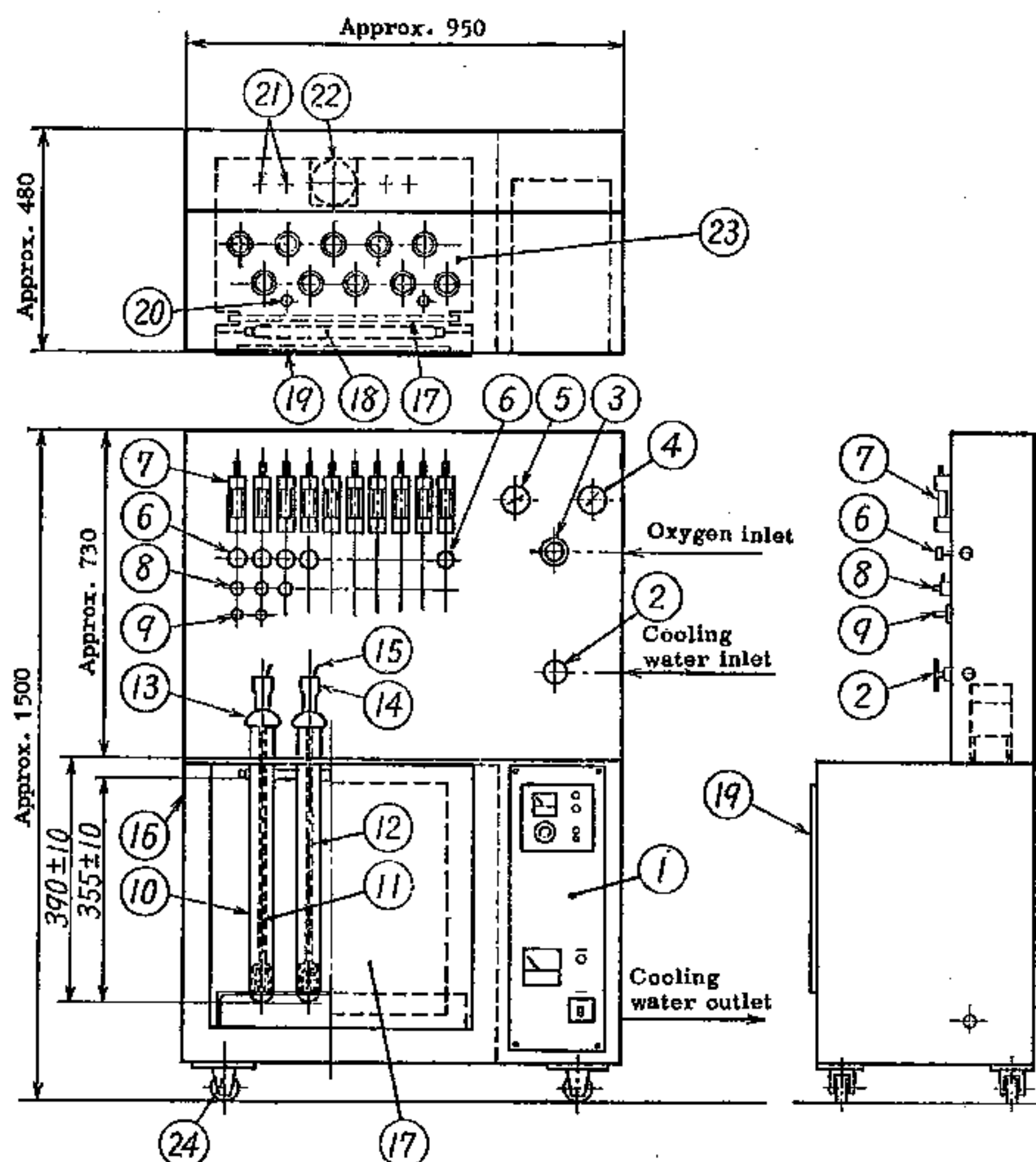
3.2.1 Summary of Test Method The specimen shall be oxidized at 95°C by passing oxygen through it in the presence of catalyzer and water, and the degree of degradation of the oil shall be evaluated on the basis of the total acid value after the prescribed testing time or on the basis of the testing time required to reach the prescribed total acid value.

3.2.2 Testing Apparatus for Oxidation Stability of Turbine Oils The testing apparatus shall have the construction shown in Fig. 4 and consist of the components (1) to (9) below.

Fig. 4. Testing Apparatus for Oxidation Stability of Turbine Oils (Shown for Example)

With 10 Test Tubes on

Unit: mm



- | | |
|-------------------------------------|---|
| ① Control panel | ⑬ Condenser |
| ② Cooling water adjusting valve | ⑭ Sampling tube holder |
| ③ Oxygen adjusting valve | ⑮ Sampling tube |
| ④ Oxygen pressure gauge (primary) | ⑯ Liquid level adjuster |
| ⑤ Oxygen pressure gauge (secondary) | ⑰ Glass pane (heat-resistant glass) |
| ⑥ Oxygen flow adjusting valve | ⑱ Lighting lamp |
| ⑦ Flow meter | ⑲ Window (made of metal) |
| ⑧ Cooling water inlet cock | ⑳ Thermometer for constant temperature bath |
| ⑨ Cooling water outlet | ㉑ Heater |
| ⑩ Test tube | ㉒ Stirrer |
| ⑪ Catalyst | ㉓ Constant temperature bath |
| ⑫ Oxygen blowing tube | ㉔ Caster |

- (1) Constant Temperature Bath As shown in Fig. 4, the bath shall provide a stirrer, electric heater, and a temperature regulator, be capable of maintaining the temperature of the specimen contained in the test tube at 95 ± 0.2 °C, have such a construction that the test tube can be inserted vertically in the constant temperature bath to a depth of 390 ± 10 mm, and provide a liquid level adjuster which is capable of adjusting the level of the bath liquid so as to allow the test tube to be immersed in the bath liquid to a depth of 355 ± 10 mm.

The cover of the constant temperature bath shall provide holes for inserting test tubes, a thermometer holder, and the like.

Remarks 1. Where oil is used as the bath liquid, a safety device shall be provided to prevent overheating.

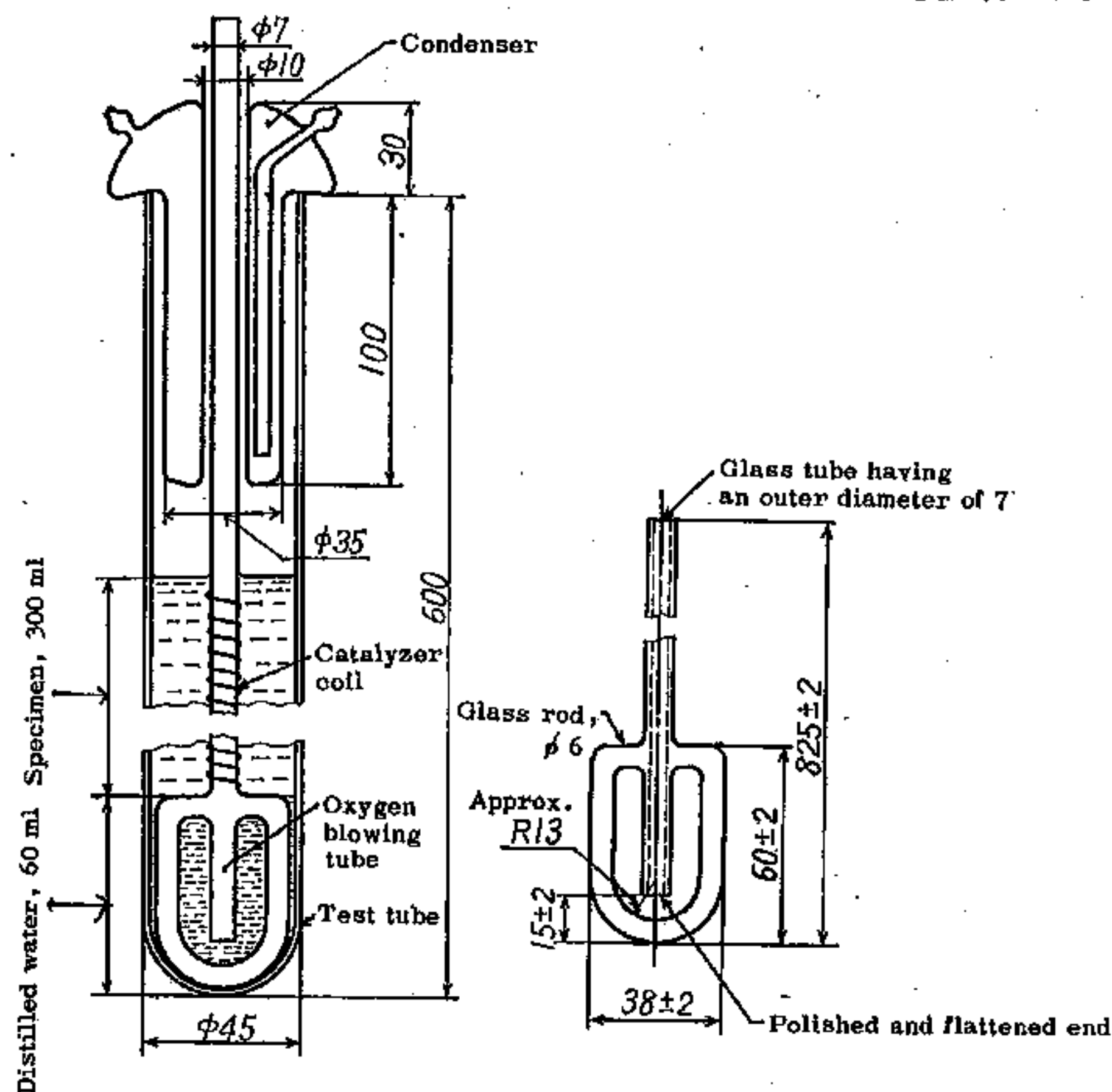
2. The insulation resistance of the heater shall be as specified in Remark of 3.1.2 (1).

- (2) Oxidizer The oxidizer shall be as shown in Fig. 5 and consist of the following components (a) to (c).

- (a) Test Tube The test tubes used shall be as specified in Fig. 116 of JIS K 2839.
- (b) Oxygen Blowing Tube The tube shall have the shape and dimensions shown in Fig. 5.
- (c) Condenser The condenser shall be as specified in Fig. 118 of JIS K 2839.

Fig. 5. Oxidizer

Unit: mm



- (3) Flow Meter A flow meter capable of adjusting the flow rate of oxygen to 3 l/h and having a precision of ± 0.1 l/h.
- (4) Thermometers
- (a) For Constant Temperature Bath (TOS) A thermometer conforming to No. 46 specified in JIS B 7410.
- (b) For Specimen A thermometer as specified in Table 4.

Table 4. Thermometer for Specimen

Scale range	°C	80 to 100
Test temperature	°C	95.0
Immersion depth	mm	76
Scale interval	°C	0.1
Total length	mm	250
Diameter	mm	6.0 to 7.0

- (5) Holder of Thermometer for Specimen A holder made of stainless steel (SUS 304), having the construction and dimensions shown in Fig. 6, and capable of being attached with a thermometer by means of fluoro-rubber O rings of about 5 mm inner diameter.