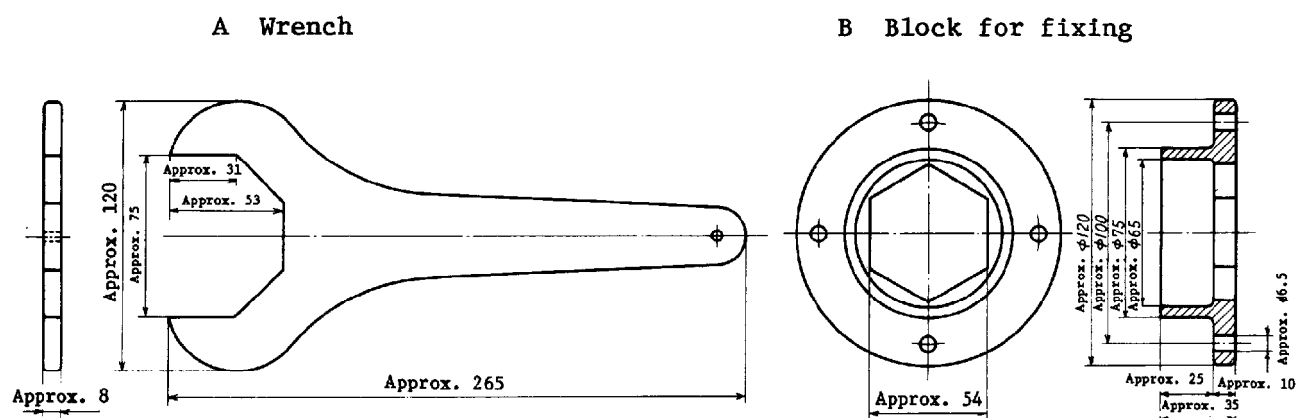


Fig. 22. Fastening tools for bomb (An example)

Unit: mm



- (6) Oxygen introducing tube Oxygen introducing tube shall be a flexible tube of metal or other appropriate material to connect the oxygen vessel and the bomb for introducing oxygen to the bomb, and shall have metal fittings at both ends to connect the bomb and the oxygen vessel.
- (7) Thermostatic oil bath An example of the thermostatic oil bath shall be given in Fig. 19. It shall be equipped with a motor driven agitator, an electric heater and a temperature regulator, and shall be capable of keeping the bath temperature in the range of 99°C to 150°C within $\pm 0.5^\circ\text{C}$.

The lid of the bath shall be provided with bomb inserting holes and a thermometer holder. Bomb inserting holes shall be attached with a guide of the supporting plate, and when the bomb is inserted, the distance between the upper surface of the bomb and the level of the bath liquid shall be about 50 mm or over.

Remarks 1. For the liquid of thermostatic bath, it is desirable to use heat resistant silicone oil or the like of having good thermal stability.

2. A safety device for overheating should preferably be attached.

- (8) Thermometer The thermometer shall be of No. 6 specified in JIS B 7410.

Informative reference: In the case of testing at over 99°C, other appropriate thermometer shall be used.

5.8.3 Solvents Petroleum benzine specified in JIS K 8594 shall be used.

5.8.4 Preparation of test Preparation of test shall be done as follows:

- (1) Wash and clean the sample container with a suitable solvent and after washing with warm soap water, rinse with tap water and then with distilled water thoroughly, and dry in a dryer. Thereafter, the sample container shall not be touched with hand directly.

- (2) Wash sufficiently the inner surface of the bomb, the sample container holder, the lid, and the attaching pipe for pressure gauge with petroleum, benzine, and then dry thoroughly.

5.8.5 Test procedures Test procedures shall be as follows:

- (1) Take samples each weighing 4.00 ± 0.01 g in five sample containers so as not to enter air bubbles, make the surface of each sample smooth, and place on the shelves of the sample container holder. When assembling the bomb, stuff loosely rounded glass wool in the bottom of the attaching pipe for pressure gauge.
- (2) Put the sample container holder in the bomb, close with the lid and the fastening nuts. Introduce slowly oxygen specified in JIS K 1101 into the bomb up to the oxygen pressure of $0.69 \text{ MPa}\{7.0 \text{ kgf/cm}^2\}$ and then release slowly. Repeat the above procedure 4 times. When the pressure reaches $0.69 \text{ MPa}\{7.0 \text{ kgf/cm}^2\}$ at the fifth charging of oxygen, close the needle valve tightly, and check the presence of the gas leakage either by leaving the bomb quietly for several hours or by immersing the bomb in water.
- (3) After ascertaining that no leakage of the gas exists, put the bomb in the thermostatic oil bath kept at $99 \pm 0.5^\circ\text{C}$. Since the pressure of the bomb rises at the beginning of the immersion in the bath, continue the operation of releasing oxygen from time to time for about 2 h so that the pressure may hold stability at $0.76 \pm 0.005 \text{ MPa}\{7.7 \pm 0.05 \text{ kgf/cm}^2\}$.
- (4) Read out the decrease of oxygen pressure after 100 h elapse from immersing the bomb in the thermostatic oil bath. Record the pressure every 24 h during the testing period.

5.8.6 Result The average value of two measured results [pressure drop $\text{MPa}\{\text{kgf/cm}^2\}$] obtained in 5.8.5, for the same sample is taken as oxidation stability, which is rounded off to the nearest $0.005 \text{ MPa}\{0.05 \text{ kgf/cm}^2\}$ in accordance with JIS Z 8401 to be taken as the test result.

5.8.7 Precision⁽¹⁷⁾ The precision shall be as follows.

Note ⁽¹⁷⁾ The precision shall be applicable only to the sample wherein oxygen is absorbed approximately in proportion to time. It should not be applied to the sample wherein the oxygen absorption accelerates rapidly in the midway.

- (1) Repeatability The tolerance on the difference between two successive test results, obtained by the same person with the same apparatus in the same laboratory on identical test sample shall be as given in Table 19.

Table 19. Repeatability

Pressure drop (MPa){kgf/cm ² }	Tolerance (MPa){kgf/cm ² }
Under 0.034 {0.35}	0.015 {0.15}
0.034 {0.35} to 0.069 {0.70} excl.	0.029 {0.30}
0.069 {0.70} to 0.137 {1.40} excl.	0.039 {0.40}
0.137 {1.40} to 0.378 {3.85} excl.	0.069 {0.70}

- (2) Reproducibility The tolerance on the difference between two single and independent test results, obtained by different persons with different apparatuses in different two laboratories on identical test sample shall be as given in Table 20.

Table 20. Reproducibility

Pressure drop (MPa){kgf/cm ² }	Tolerance (MPa){kgf/cm ² }
Under 0.034 {0.35}	0.039 {0.40}
0.034 {0.35} to 0.069 {0.70} excl.	0.054 {0.55}
0.069 {0.70} to 0.137 {1.40} excl.	0.083 {0.85}
0.137 {1.40} to 0.378 {3.85} excl.	0.137 {1.40}

Remarks: The test result exceeds the tolerance shall be treated in accordance with JIS Z 8402.

5.9 Test method for deleterious particles

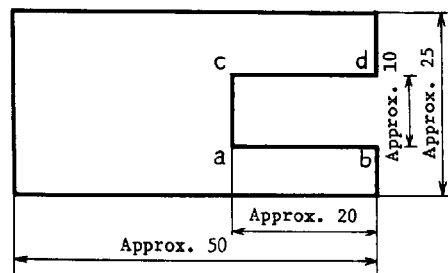
5.9.1 Summary of test method Fill the notch of the specified templet with sample in a clean environment, and then measure the number of the deleterious particles contained in the sample according to the size by using a microscope.

5.9.2 Apparatus and device The apparatus and device shall be composed of the following items (1) to (4).

- (1) Microscope The microscope of about 100 magnifications provided with an ocular micrometer and a mechanical stage.
- (2) Templet The templet shall be a metallic plate with 0.1 ± 0.01 mm thickness of the shape and dimensions as shown in Fig. 23, which shall have a notch of about 10 mm in width and about 20 mm in length.

Fig. 23. Templet

Unit: mm



- (3) Desiccator The desiccator shall have a size enough to contain slide glasses for microscope, and shall be capable of obtaining reduced pressure by the aid of a vacuum pump.
- (4) Vacuum pump The vacuum pump shall be able to reduce rapidly the pressure of air in the desiccator to 1.33 kPa{10 mmHg} or under.

5.9.3 Test procedures Test procedures shall be as follows:

- (1) Remove the surface of the sample with a spatula, place the templet on the slide glass, and fill the notch with the sample⁽¹⁸⁾ slightly over the upper surface of the templet while pressing the end without notch by hand.
- Note ⁽¹⁸⁾ If necessary, air shall be removed by pretreatment.
- (2) When air bubbles are contained in the grease existing in the notch of the templet, put the sample together with slide glass in the desiccator to subject to the pressure reduction treatment, which is made by keeping pressure 1.33 kPa{10 mmHg} or under for 10 min to 15 min. Slide the cover glass while pressing it to the templet so that the excess sample be scraped off from the edge of the notched side of the templet.
- (3) Place the slide glass on the stage of the microscope, and adjust the ocular lens and stage so that the graduation of the micrometer comes to line a-b of the notch.
- (4) Move the stage along the line a-c of the notch while focussing on the deleterious particles, record the numbers of deleterious particles crossing the graduation of the ocular micrometer while classifying into four classes of, 10 μm or over to and excluding 25 μm , 25 μm or over to and excluding 75 μm , 75 μm or over to and excluding 125 μm , and 125 μm or over, continue the measurement until the graduation arrives at the line c-d of the notch of the templet, and take this classification as first section. In this case, for a fibrous matter measurement shall be made for the width, not for the length.

Remarks: Particles 10 μm or smaller shall not be counted.

- (5) Shift the stage along the line c-d of the notch adjacent to the first section by equal distance of the graduation length of micrometer, and count and record the numbers of particles by the method specified in (4) until the stage reaches the line a-c of the notch. Repeat the procedure to about 10 mm on the line a-b or c-d of the notch.

5.9.4 Calculation and result Calculate the number of deleterious particles in the sample per 1 cm³ by the formula below, and express the number of particles in an average for every size obtained to digit of integer from three test results by rounding off the value according to JIS Z 8401.

$$A' = \frac{1000(A+B+C+D)}{T \cdot S \cdot N}$$

$$B' = \frac{1000(B+C+D)}{T \cdot S \cdot N}$$

$$C' = \frac{1000(C+D)}{T \cdot S \cdot N}$$

$$D' = \frac{1000D}{T \cdot S \cdot N}$$

where, A' : the number of particles of 10 μm or over in the maximum size per 1 cm³ sample (particles/cm³)

B' : the number of particles of 25 μm or over in the maximum size per 1 cm³ sample (particles/cm³)

C' : the number of particles of 75 μm or over in the maximum size per 1 cm³ sample (particles/cm³)

D' : the number of particles of 125 μm or over in the maximum size per 1 cm³ sample (particles/cm³)

A : total number of particles of 10 μm or over to and excl. 25 μm (particles)

B : total number of particles of 25 μm or over to and excl. 75 μm (particles)

C : total number of particles of 75 μm or over to and excl. 125 μm (particles)

D : total number of particles of 125 μm or over (particles)

T : sectional area of notch of templet (mm²)(¹⁹)

S : length of graduation of ocular micrometer (mm)

N : the number of measuring sections

Note (¹⁹) Measure the thickness of templet and the width of the notch to calculate the sectional area (mm²).

5.10 Test method for ash content

5.10.1 Summary of test method After burning the sample until carbon disappears, weigh the residue to obtain the ash content.

5.10.2 Apparatus and device The apparatus and device shall be composed of the following (1) and (2).

- (1) Crucible The crucible shall be of 15 ml capacity, and made of porcelain, quartz or platinum⁽²⁰⁾.

Note ⁽²⁰⁾ When a sample contains lead, zinc and other substances which react with platinum at high temperature, platinum crucible shall not be used.

- (2) Muffle furnace A muffle furnace of appropriate size and capable of maintaining the temperature up to 600°C.

5.10.3 Test procedures Test procedures shall be as follows:

Heat a crucible to red, and weigh after cooling in a desiccator. Take 2 g to 5 g of sample into this crucible, and weigh to the nearest 0.01 g. Heat the crucible, and burn it slowly⁽²¹⁾. After burning, further ignite strongly in the muffle furnace until the carbon disappears. Leave the crucible and the content still in a desiccator for cooling, and weigh.

Note ⁽²¹⁾ When a sample scatters by foaming, add 1 ml to 2 ml of ethyl alcohol before heating.

5.10.4 Calculation and result Calculate ash content according to the following formula. Round off the average of two measured results of the same sample obtained in 5.10.3 to one place of decimal in accordance with JIS Z 8401 to be taken as the test result.

$$A = \frac{W_r}{W_s} \times 100$$

where, A : ash content (mass %)

W_r : mass of ash (g)

W_s : mass of sample (g)

5.11 Test method for worked stability

5.11.1 Summary of test method After working the sample one hundred thousand times in the specified worker, keep it at 25°C, further work 60 times, and measure the penetration.

5.11.2 Tester for worked stability The tester for worked stability shall consist of (1) to (6) below.

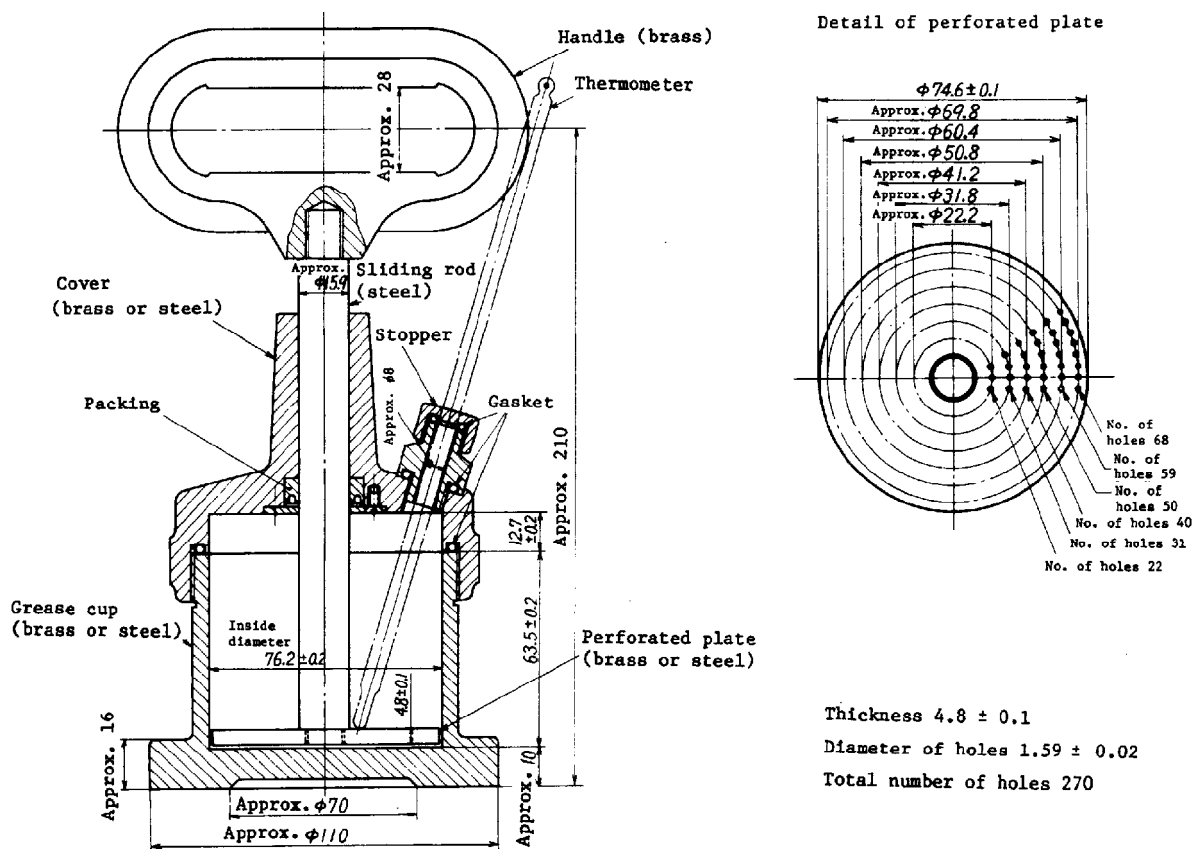
- (1) Worker for worked stability The worker is exemplified in Fig. 24, and capable of moving up and down the perforated plate attached to the top of the sliding rod.

The gland of the sliding rod and the jointing part of the cup and the cover or the like shall be so constructed that the sample in the cup should leak extremely little for one hundred thousand up and down double strokes.

- (2) Motor driven working apparatus The apparatus shall be similar to the motor driven working apparatus specified in 5.3.2 (6) in construction and shall be capable of moving up and down perforated plate of the worker for the worked stability at a rate of 60 ± 10 double strokes per 1 min for 67 mm to 69 mm travel. The mechanism of up and down movement shall be capable of withstanding the test for worked stability, and it is preferable that the motor to be used is of 0.75 kW.
- (3) Penetrometer As specified in 5.3.2 (1).
- (4) Cone As specified in 5.3.2 (2) and (3).
- (5) Spatula As specified in 5.3.2 (7).
- (6) Thermostatic water bath As specified in 5.3.2 (9).

Fig. 24. Worker for worked stability (An example)

Unit: mm



5.11.3 Test procedures Test procedures shall be as follows:

- (1) Prepare about 500 g of sample which is enough to fill the cup of the worker, and leave it still until it coincides with room temperature of the testing place.

Remarks: The temperature of the testing room should preferably be kept at 15°C to 30°C .

- (2) After leaving the sample for enough time in the testing place, fill the cup of clean grease worker with sample with a spatula with care not to allow air to enter the sample, and mound it to a mountain shape so that the central part will be about 10 mm or higher than the rim of the cup, open the cock on the cover of the grease worker, and assemble the grease worker. Then depress the perforated plate to the bottom of the cup, close the cock, fit the worker to the motor driven working apparatus, and work the sample until it reaches one hundred thousand times continuously (about 28 h).
- (3) After working, immediately remove the grease worker from the motor driven working apparatus, leave it in the thermostatic water bath kept at $25 \pm 0.5^{\circ}\text{C}$ for 2 h, and prepare the sample by the method specified in 5.3.3 (1)(b) and (c). In this case, the perforated plate as specified in 5.11.2 shall be used.
- (4) Measurement of the penetration of the sample shall be made according to the method specified in 5.3.4 (2).

5.11.4 Result The average of three test results obtained in 5.11.3 for the same sample shall be taken as worked stability, and rounded off to an integer in accordance with JIS Z 8401 to be taken as the test result.

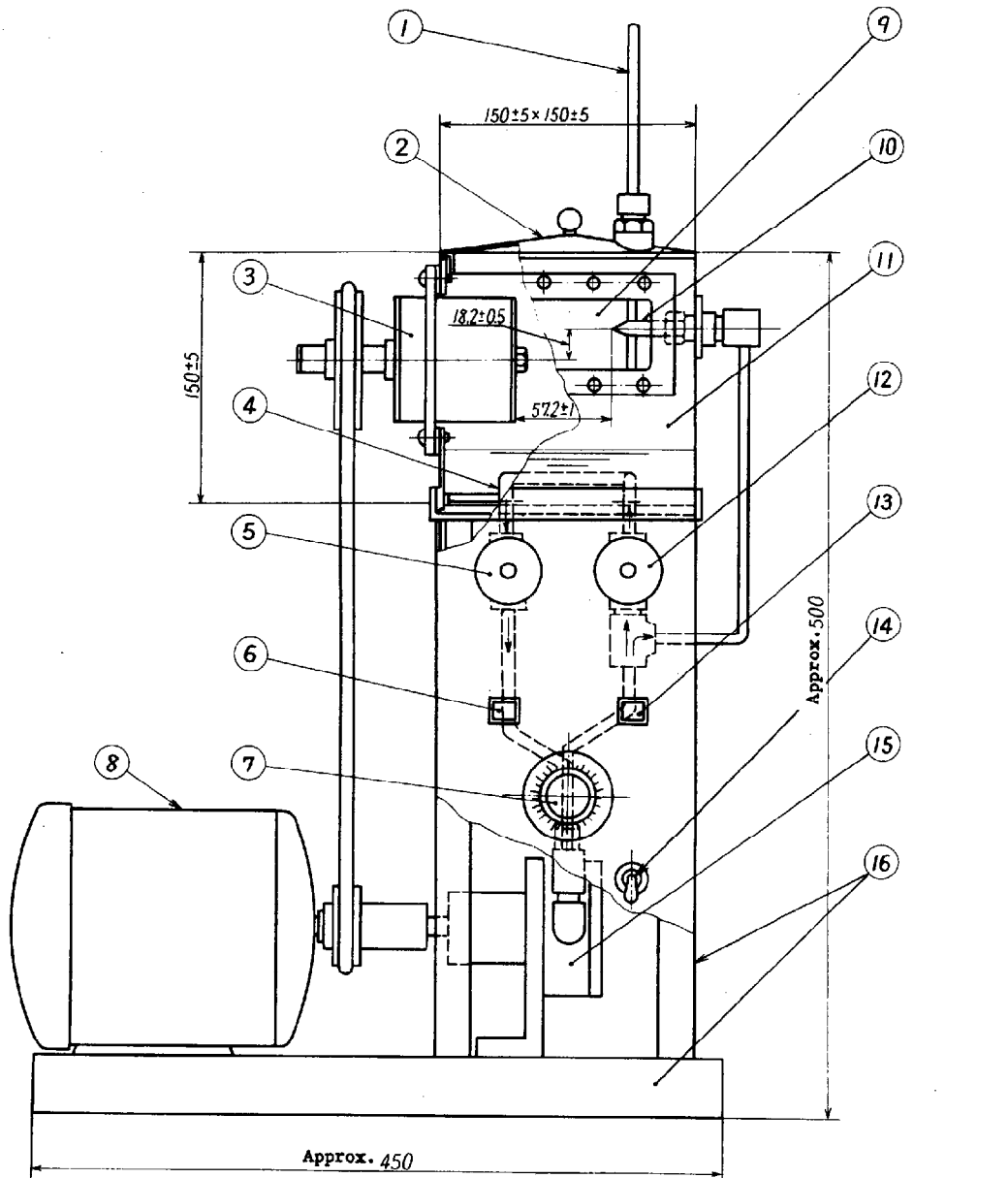
5.12 Test method for water washout resistance

5.12.1 Summary of test method Fit the ball bearing having been packed with the sample in a housing, and rotate at a rate of 600 ± 30 revolutions per min, and jet distilled water kept at $38 \pm 2^{\circ}\text{C}$ or $79 \pm 2^{\circ}\text{C}$ to the ball bearing housing at the rate of 5 ± 0.5 ml per s. Obtain the loss of the sample (mass %) after 1 h lapse.

5.12.2 Testing apparatus for water washout resistance The testing apparatus for water washout resistance shall be composed of the following items (1) to (4). An example of the construction of testing apparatus for water washout resistance is given in Fig. 25.

Fig. 25. Construction of testing apparatus for water washout resistance (An example)

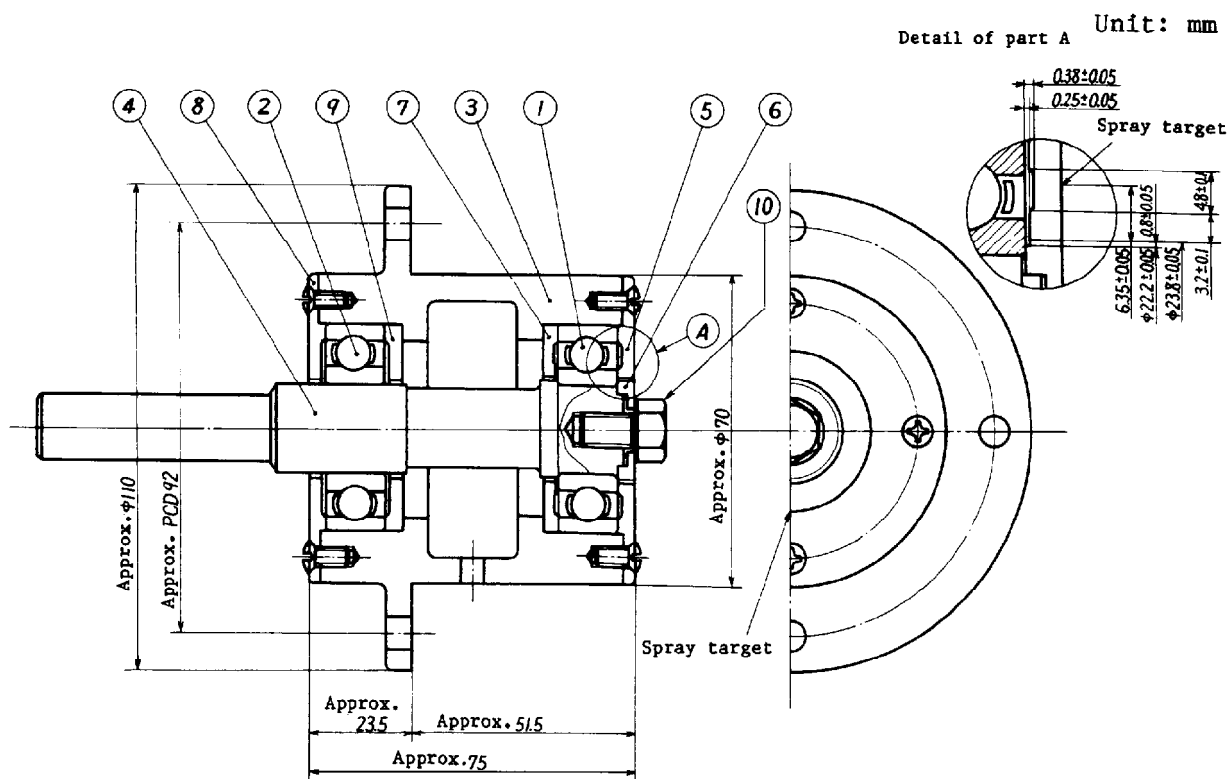
Unit: mm



- | | |
|---|--|
| ① Thermometer | ⑨ Peep window |
| ② Lid | ⑩ Spray nozzle |
| ③ Housing | ⑪ Thermostatic water bath |
| ④ Electric heater | ⑫ By-pass valve (for regulating the amount of spray) |
| ⑤ Flow regulating valve | ⑬ Indicator lamp of power source |
| ⑥ Signal lamp of temperature regulation | ⑭ Switch of power source |
| ⑦ Temperature regulator | ⑮ Pump |
| ⑧ Motor | ⑯ Frame |

- (1) Rotating mechanism of ball bearing for test The mechanism is to rotate the ball bearing contained in the housing shown in Fig. 26 at a rate of 600 ± 30 revolutions per min, and shall consist of the housing, the ball bearing for test and a suitable driving mechanism.

Fig. 26. Construction of housing and shaft (An example)



- | | |
|--|--|
| ① Ball bearing for test | ⑦ Outer ring holder of ball bearing for test |
| ② Ball bearing for supporting | ⑧ Outer ring retainer of ball bearing for supporting |
| ③ Housing | ⑨ Outer ring holder of ball bearing for supporting |
| ④ Shaft | ⑩ Fastening bolt |
| ⑤ Outer ring retainer of ball bearing for test | |
| ⑥ Inner ring retainer of ball bearing for test | |

(a) Ball bearing for test Tolerance class 0, 6204 open type specified in JIS B 1521.

(b) Housing and shaft The housing and the shaft shall be as shown in Fig. 26 in shape and dimensions, made of brass or stainless steel (SUS304), and capable of being attached or detached easily to the thermostatic water bath.

- (2) Thermostatic water bath The water bath shall be as shown in Fig. 25 in shape and dimensions and equipped with the electric heater, temperature regulator, suitable lid and frame, capable of keeping water temperature at $38 \pm 2^\circ\text{C}$ or $79 \pm 2^\circ\text{C}$. The bath shall be easily attached with the housing and spray nozzle at the position shown in Fig. 25.
- (3) Water spray mechanism The mechanism shall consist of the spray nozzle, by-pass valve, flow-rate regulating valve, pump, motor and others, and shall be capable of circulating and spray the warm water in the bath to the housing at a rate of 5 ± 0.5 ml per s.

The test for the spray flow rate shall be made by measuring the flow quantity for 60 s or 30 s after jointing a rubber tube to the top end of the spray nozzle and putting the other end of the rubber tube to a receiving measuring cylinder. The spray flow rate of the spray nozzle shall be regulated by the operation of flow-rate regulating valve and the by-pass valve.

- (a) Spray nozzle The nozzle shall be 1.0 ± 0.1 mm in inner diameter, and when attached to the thermostatic water bath, the spray shall be able to impinge on the spray target specified in Fig. 26 without spreading.
- (b) Pump The pump shall be capable of spray the warm water from the spray nozzle without pulsations at the specified flow rate.
- (4) Thermometer The thermometer shall be a glass thermometer which is readable at 38°C and 79°C with scale interval of 1°C or under, or a thermometer at least equal in quality thereto.

5.12.3 Thermostatic air bath That shall be capable of keeping the specified temperature within $\pm 1^\circ\text{C}$ by electric heater.

5.12.4 Reagent Solvent shall be petroleum benzine specified in JIS K 8594.

5.12.5 Preparation of test Preparation of test shall be carried out as follows:

- (1) Clean the water bath and the water circulating passages with distilled water. Wipe off any oil scum adhering on to the inner wall of the bath.
- (2) Clean the ball bearing for test with petroleum benzine, and dry.

5.12.6 Test procedures Test procedures shall be as follows:

- (1) Pack the ball bearing for test of known mass with 4.00 ± 0.05 g of sample. Attach this bearing and outer ring retainer (Fig. 26 ⑤), outer ring holder (Fig. 26 ⑦) and inner ring retainer (Fig. 26 ⑥) of ball bearing for test of known mass into the housing, as in Fig. 26.
- (2) Add minimum 750 ml of distilled water in the bath⁽²²⁾, start heating and driving the motor. At this time, care shall be taken so that diverting water spray is introduced into water with a rubber tube connected to the tip of the spray nozzle to prevent the housing from being wetted, and adjustment is made so as to keep the water temperature at $38 \pm 2^\circ\text{C}$ or $79 \pm 2^\circ\text{C}$.