- b) 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 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.
- c) 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.
- d) Move the stage along the line a-c of the notch while focussing on the foreign matter, record the numbers of foreign matter crossing the graduation of the ocular micrometer while classifying into four classes of, $10 \ \mu m$ or over to and excluding $25 \ \mu m$, $25 \ \mu m$ or over to and excluding $75 \ \mu m$, $75 \ \mu m$ or over to and excluding $125 \ \mu m$, and $125 \ \mu m$ 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 cas, for a fibrous matter measurement shall be made for the width, no for the length.

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

e) 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 \mathbf{d}) until the stage reaches the line a-b of the notch. Repeat the procedure to about 10 mm on the line a-b or c-d of the notch.

13.5 Calculation method and precision

a) **Calculation method** Calculate the number of particles per 1 cm³ by every size according to the following formula, round off the mean value of three measured results obtained on the same sample in **13.4** to the nearest 1 according to the specifications of **JIS Z 8401**, and take it as the test result.

$$A' = \frac{1\ 000(A+B+C+D)}{T\times S\times N}$$
$$B' = \frac{1\ 000(B+C+D)}{T\times S\times N}$$
$$C' = \frac{1\ 000(C+D)}{T\times S\times N}$$
$$D' = \frac{1\ 000D}{T\times S\times N}$$

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 μ m or over to and excl. 25 μ m (particles)
- B: total number of particles of 25 μ m or over to and excl. 75 μ m (particles)
- C: total number of particles of 75 μ m or over to and excl. 125 μ m (particles)
- D: total number of particles of 125 µm or over (particles)
- T: sectional area of notch of templet $(mm^2)(^{14})$
- S: length of graduation of ocular micrometer (mm)
- N: the number of measuring sections
 - Note (14) Measure the thickness of templet and the width of the notch to calculate the sectional area (mm²).
- b) **Precision** The precision is not specified.

13.6 Report of test results The test report shall contain at least the following information.

- a) Name of sample, place of sampling and date of sampling
- b) Designation of test method and the result obtained according to 13.5
- c) Date of test
- d) Specially mentioned matters

Unit: mm

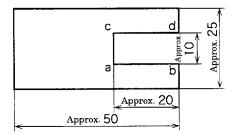


Figure 25 Templet (an example)

14 Test method for ash content

14.1 Principle of test The sample is weighed out in a crucible, burnt until the sample becomes ash and carbide matter, then put in an electric furnace and heated at 600 °C. After the carbonaceous matter has completely become ash, it is left to cool in a desiccator as it is and the mass is weighed to obtain ash content.

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14.2 Reagents The reagents shall be as follows:

a) Ethanol, specified in JIS K 8102.

14.3 Test apparatus The test apparatus for ash content shall be in accordance with the following items a) to d).

- a) **Crucible** The crucible shall be of 15 ml⁽¹⁶⁾ in capacity, and made of porcelain, quartz or platinum⁽¹⁵⁾.
 - Notes (15) When a sample contains lead, zinc and other substances which react with platinum at high temperature, platinum crucible shall not be used.
 - (16) A crucible with different capacity may be used as the result of considering the expected amount of ash content of the sample and the thermal expansion of the sample when burnt.
- b) **Electric furnace** The furnace capable of regulating the temperature of furnace inside at 600 °C \pm 25 °C.
- c) **Desiccator** The desiccator shall have an appropriate size and be used without desiccant.
- d) **Balance** The balance capable of weighing the total mass of crucible and sample to the nearest 0.01 g.

14.4 Sampling method and preparation method of sample The sample shall be taken and prepared according to the sampling method of primary sample and preparation method of secondary sample specified in JIS K 2251 or the methods applied correspondingly to these.

14.5 Test procedures The test procedures shall be as follows:

- a) Place the crucible in the electric furnace maintained at $600 \text{ }^{\circ}\text{C} \pm 25 \text{ }^{\circ}\text{C}$, heat it, leave it cool as it is in a desiccator, and then weigh the mass to the nearest 0.01 g.
- b) Weigh out 2 g to 5 g of the sample in this crucible and read out the mass to the nearest 0.01 g.
- c) Heat the crucible containing weighed out sample with gas burner, and burn the sample gradually⁽¹⁷⁾.

Note (17) When the sample foams and scatters during burning, add 1 ml to 2 ml of ethanol before heating.

d) When the sample starts to burn, regulate the heating so as to continue to burn at a constant state thereafter.

Information : An electric heater such as hot plate may be used.

e) After the burning of the sample has finished and the contents in the crucible has become the carbonaceous substance, place the crucible in the electric furnace maintained at 600 °C \pm 25 °C and heat it until the carbonaceous substance can not be completely observed.

f) Take out the crucible from the electric furnace, leave it cool as it is in a desiccator to the room temperature, and then weigh the mass to the nearest 0.01 g.

14.6 Calculation method and precision

a) **Calculation method** Calculate the ash content according to the following formula, round off the mean value of two measured results on the same sample obtained according to 14.5 to the nearest 0.1 according to the specifications of JIS Z 8401, and take it as the test result.

$$A = \frac{W_{\rm r}}{W_{\rm s}} \times 100$$

where, A : ash content (%)
 $W_{\rm r}$: mass of ash (g)

 W_{s} : mass of sample (g)

b) **Precision** The precision is not specified.

14.7 **Report of test results** The test report shall contain at least the following information.

- a) Name of sample, place of sampling and date of sampling
- b) Designation of test method and the result obtained according to 14.6
- c) Date of test
- d) Specially mentioned matters

15 Test method for worked stability

15.1 **Principle of test** After the sample is worked one hundred thousand times in the specified worker, it is kept at $25 \,^{\circ}$ C for the specified time, further worked 60 times, and the penetration is measured.

15.2 Test apparatus The test apparatus shall be composed of the following items **a**) to **f**), and an example is as shown in figure 26.

- a) Worker for worked stability The worker shall be 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.
- b) Motor driven working apparatus The apparatus shall be similar to the motor driven working apparatus specified in 7.2 c) 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 71 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.
- c) **Penetrometer** As specified in 7.2 a).

- d) Cone As specified in 7.2 b) 1).
- e) Spatula As specified in 7.2 h)
- f) Thermostatic water bath As specified in 7.2 f).

15.3 Sampling method and preparation method of sample The sample shall be taken and prepared according to the sampling method of primary sample and preparation method of secondary sample according to JIS K 2251 or the methods applied corresponding to these.

15.4 Test procedures The test procedures shall be as follows:

a) Prepare the sample of amount (approximately 500 g) enough to overfill the cup of the worker, and leave it as it is until the sample reaches the room temperature in the testing place.

Remarks : The temperature of the testing place should preferably be kept within the range 15 °C and 30 °C.

- b) After leaving the sample as it is 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).
- c) After working, immediately remove the grease worker from the motor driven working apparatus, leave it as it is in the thermostatic water bath kept at 25 °C \pm 0.5 °C for 2 h, and prepare the sample by the method specified in **7.5 a**) **3**). In this case, the perforated plate as specified in **15.2** shall be used.
- d) Measurement of the penetration of the sample shall be made according to the method specified in 7.5 b) and 7.5 c).

15.5 Calculation method and precision

- a) **Calculation method** Round off the mean value of three measured results on the same sample obtained in 15.4 to the nearest 1 according to the specifications of **JIS Z 8401**, and take it as the worked stability.
- b) **Precision** The precision is not specified.

15.6 Report of test results The test report shall contain the following information.

- a) Name of sample, place of sampling and date of sampling
- b) Designation of test method and the result obtained according to 15.5
- c) Date of test
- d) Specially mentioned matters

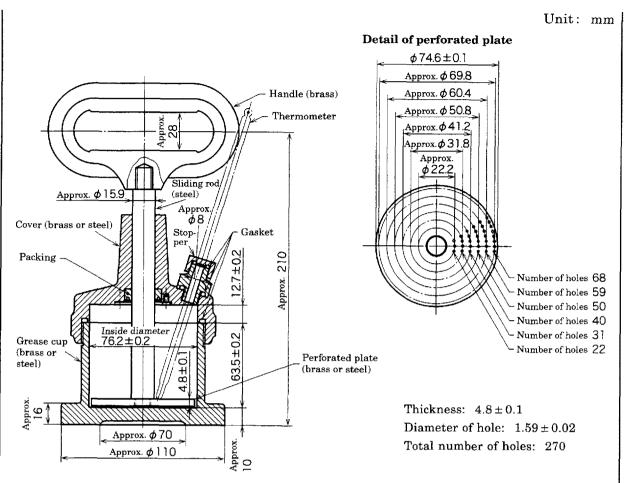


Figure 26 Worker for worked stability (an example)

16 Test method for water washout resistance

16.1 Principle of test The grease is packed in a ball bearing. The bearing is then inserted in a housing with specified clearances, and rotated at 63 rad/s \pm 3 rad/s. Water, controlled at 38 °C or 79 °C, impinges on the bearing housing at a rate of 5 ml/s \pm 0.5 ml/s for 60 min. The amount of sample washed out in 60 min is taken as a measure of the resistance of the sample (grease) to water washout.

16.2 Reagents The reagents shall be as follows:

a) Water, of grade A3 of water specified in JIS K 0557.

b) Solvent, of petroleum benzine specified in JIS K 8594.

16.3 Test apparatus The test apparatus for water washout resistance shall be composed of the following items a) to i). An example of the construction of test apparatus for water washout resistance is shown in figure 27.

a) Rotation mechanism of ball bearing for test The rotation mechanism is to rotate the ball bearing for test in the housing as shown in figure 28 at 63 rad/s \pm 3 rad/s. That shall be composed of a housing, a ball bearing for test and an appropriate driving mechanism.

- 1) **Ball bearing for test**, conforming to the requirements of open type 6204, grade 0, clearance C3 of **JIS B 1521**.
- 2) Housing and shaft, of shape and dimensions as shown in figure 28, made of brass or stainless steel (SUS304), and being attachable or detachable easily to the thermostatic water tank.
- b) **Thermostatic water tank**, of shape and dimensions as shown in figure 27, equipped with electric heater, temperature regulator, appropriate cover and base, capable of maintaining water temperature at $38 \degree C \pm 1.7 \degree C$ or $79 \degree C \pm 1.7 \degree C$. The water tank shall be easily attached with the housing and jet nozzle at the position shown in figure 27.
- c) Water jet mechanism, consisting of the jet nozzle, by-pass valve, flow-rate regulating valve, pump, motor and others and capable of circulating and jetting the warm water in the water tank to the housing at a rate of $5 \text{ ml/s} \pm 0.5 \text{ ml/s}$. The flow rate of jet shall be obtained so that a rubber tube is jointed to the top end of jet nozzle, the other end of rubber tube is received in a measuring cylinder and the flow rate for 10 s is measured.
 - 1) Jet nozzle, of $1.0 \text{ mm} \pm 0.1 \text{ mm}$ in inside diameter. When it is attached to the thermostatic water tank, the water stream shall be capable of impinging on the jet target specified in figure 28 without spreading.
 - 2) **Pump**, capable of jetting the warm water from the jet nozzle without pulsations at the specified flow rate.
- d) **Thermometer**, of a glass thermometer capable of reading 38 °C and 79 °C with scale interval of 1 °C or under, or a thermocouple equivalent to this in quality.
- e) **Thermostatic air bath**, of <u>electric heating type</u> and capable of maintaining the specified temperature at ± 3 °C by natural convection.
- f) **Timer**, capable of indicating tenth of a second.
- g) **Watch glasses**, of sufficient size to accommodate a ball bearing for test, outer ring holder, outer ring retainer and inner ring retainer.
- h) Measuring cylinder, of 100 ml in capacity.
- i) **Balance**, capable of weighing to 1 mg.

16.4 Sampling method and preparation method of sample The sample shall be taken and prepared according to the sampling method of primary sample and preparation method of secondary sample specified in **JIS K 2251** or the methods applied correspondingly to these.

16.5 Preparation of test The preparation of test shall be as follows:

a) Each test will require a quantity of grease sufficient to fill two test ball bearings for test (approximately 4 g each). A minimum of 15 g of sample shall be supplied. Examine the sample for any indication of non-homogeneity such as oil separation, phase changes or gross contamination. If any abnormal conditions are found, prepare a new sample.

- b) Clean the thermostatic water tank and water circulating passages with water, and wipe off any oil scum which has been deposited on the inner surface of thermostatic water tank.
- c) Clean the ball bearing for test with petroleum benzine and dry it.
- d) Ensure that the water flow rate can be maintained within the specified limits for a 1-h test run.

16.6 Test procedures The test procedures shall be as follows:

- a) Carry out the test in duplicate. Pack the tared ball bearing for test with 4.00 g ± 0.05 g of sample. Insert this bearing, outer ring retainer (figure 28 ⑤), outer ring holder (figure 28 ④) and inner ring retainer (figure 28 ⑥) into the housing as shown in figure 28. Record the mass of bearing, outer ring retainer (figure 28 ⑥), outer ring holder (figure 28 ④) and inner ring retainer (figure 28 ⑥) to the nearest 0.01 g.
- b) Add a minimum of 750 ml of preheated water in the thermostatic water tank (¹⁸), and start the motor. At this time, introduce water through a rubber tube connected to the end of jet pipe so that the housing is not splashed with water directly, and regulate the water temperature at 38 °C \pm 1.7 °C or 79 °C \pm 1.7 °C.

Note (18) The water level shall be below the lower end of the housing.

- c) When the water reaches the specified temperature, adjust the by-pass value to give a flow rate, by putting the end of rubber tube into the measuring cylinder, of 5 ml/s \pm 0.5 ml/s. Determine the flow rate from the volume of liquid flowing into the cylinder for a period of 10 s, as measured with the timer.
- d) Detach the rubber tube from the jet pipe, and adjust the water jet so that it impinges on the jet target $6.4 \text{ mm} \pm 0.05 \text{ mm}$ above the upper end of the clearance between outer ring retainer and inner ring retainer, and start the test and continue for $60 \text{ min} \pm 5 \text{ min}$ from the moment when the rotation speed of the bearing reaches $63 \text{ rad/s} \pm 3 \text{ rad/s}$.
- e) Shut out the motor and electric heater, detach the ball bearing for test, outer ring holder, outer ring retainer and inner ring retainer, place them on a tared watch glass and dry them in the thermostatic air bath maintained at 77 °C \pm 6 °C(¹⁹) for 15 h. In such a case, separate the outer ring holder, outer ring retainer and inner ring retainer from the bearing and place them on the watch glass with inner side upward.
 - Note (19) Some mass loss can be experienced for greases continuing low-viscosity oils because of oil evaporation during drying. The drying temperature should be increased to 93 °C \pm 3 °C for greases containing high-viscosity oils, to facilitate removal of water during the time period specified.
- f) After drying, leave them cool as they are in a desiccator to room temperature, weigh the mass of the ball bearing for test, outer ring holder, outer ring retainer, inner ring retainer and watch glass to the nearest 0.01 g, and obtain the loss of the sample grease⁽²⁰⁾.

Note (20) The grease remaining on the outer ring holder, outer ring retainer, inner ring retainer and watch glass and any leakage occurring during drying period, should not be considered as grease loss.

16.7 Calculation method and precision

a) Calculation method For the water washout characteristics, calculate each test result by mass rate according to the following formula, and round off the mean value of two test results to the nearest 1 according to the specifications of JIS Z 8401. Mention clearly, also, the temperature of drying the ball bearing for test, outer ring holder, outer ring retainer, and inner ring retainer and the sample grease.

$$\Delta m_{\rm e} = m_2 - m1$$
$$\Delta m_{\rm a} = m_3 - m1$$
$$w = \frac{\Delta m_{\rm e} - \Delta m_{\rm a}}{\Delta m_{\rm e}} \times 100$$

where, w: water washout characteristics (mass %)

- m_1 : mass of ball bearing for test, outer ring holder, outer ring retainers and inner ring retainer (g)
- m_2 : mass of sample grease before test, ball bearing for test, outer ring holder, outer ring retainer and inner ring retainer (g)
- m_3 : mass of sample grease after test, ball bearing for test, outer ring holder, outer ring retainer and inner ring retainer (g)
- b) **Precision** The tolerance (probability: 0.95) of test results obtained according to this test method shall be as follows:
 - 1) **Repeatability** When the same sample is tested twice in the same laboratory by the same person by using the same tester within a short time successively, the tolerance of test results shall be as follows.

At 38 °C
$$r = 0.8 (X + 2)$$

At 79 °C $r = 0.6 (X + 4.6)$
 r : repeatability X: mean value of two test results

2) **Reproducibility** When the same sample is tested once in two different laboratories by different persons by using different testers respectively, the tolerance of difference of two test results shall be as follows.

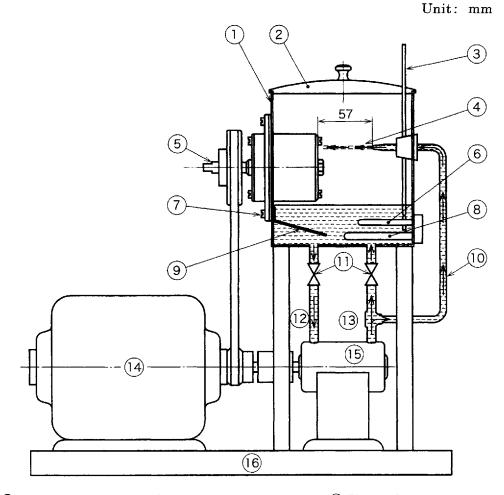
At 38 °C
$$R = 1.4 (X + 2)$$

At 79 °C $R = 1.1 (X + 4.6)$
 R : reproducibility X: mean value of two test results

16.8 Report of test results The test report shall contain the following information.

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- a) Name of sample, place of sampling and date of sampling
- b) Designation of test method and the result obtained according to 16.7
- c) Date of test
- d) Specially mentioned matter

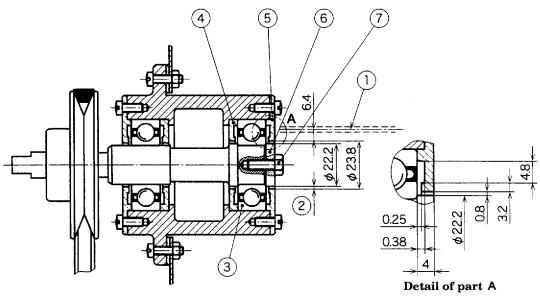


- (1) Thermostatic water tank (size: approx. 150 mm × approx. 150 mm × approx. 150 mm)
- 2 Cover
- 3 Thermometer
- (4) Jet nozzle (capillary of 1 mm diameter) (Flow rate: 5 ml/s \pm 0.5 ml/s)
- (5) Ball bearing (rotation speed: 63 rad/s \pm 3 rad/s)
- (6) Temperature regulator (thermostat, etc.)
- ⑦ Water in thermostatic water tank (minimum amount: 750 ml)

- 8 Electric heater
- 9 Baffle
- 1 Water supply pipe
- Flow-rate regulating valve and by-pass valve
- 🕲 Return pipe
- 13 Bypass
- (1) Motor
- (5) Fluid pump
- 🚯 Base

Figure 27 Construction of test apparatus for water washout characteristics (an example)





- ① Centreline of capillary, 1 mm in diameter
- 2 Annular opening, 0.8 mm wide
- 3 Ball bearing for test of type 6204 C3
- ④ Outer ring holder of ball bearing for test
- (5) Outer ring retainer of ball bearing for test
- 6 Inner ring retainer of ball bearing for test
- ⑦ Fastening bolt for inner ring retainer of ball bearing for test

Figure 28 Construction of housing (an example)

17 Test method for leakage tendency

17.1 Principle of test The wheel-hub and bearing is filled with the specified quantity of sample grease respectively and rotated under the specified conditions. Then the total mass of leaked grease and oil is obtained.

17.2 Reagent The reagent shall be as follows:

a) Solvent, of petroleum benzine specified in JIS K 8594.

17.3 Test apparatus The test apparatus for leakage tendency shall be composed of the following items a) to g). An example of the construction of the test apparatus for leakage tendency is shown in figure 29.

- a) **Thermostatic air bath** The air bath shall be as shown in figure 29 in shape and dimensions, equipped with a heater capable of maintaining the bath temperature at 113 °C \pm 2 °C, a temperature regulator and a fan. The heater shall be capable of raising the bath temperature to 113 °C within 15 min \pm 5 min. The bath contains the rotating mechanism of wheel bearing specified in **b**) below.
- b) **Rotating mechanism of wheel bearing** The mechanism is to rotate the wheel hub at a rate of 660 r.p.m. \pm 30 r.p.m., and shall consist of the spindle, wheel hub, bearing, hub pulley, transmission pulley, V-belt, electric motor and the like as shown in figure 29.