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Petroleum asphalts

1 Scope This Japanese Industrial Standard specifies petroleum asphalts (hereafter referred to as "asphalt") used for road pavements, constructions for water use, waterproofings, electric insulations and general industries.

Remarks 1 Since this Standard does not specify over the whole of safe use, when hazardous reagents, operations and apparatuses are used, suitable forbidden items on safety and health should preliminarily be determined.

2 Standards cited in this Standard are given in Attached Table 1.

3 The units and numerical values given in { } in this Standard are based on the traditional units and are appended for informative reference.

2 General matters Rounding off of numerical values shall be in accordance with JIS Z 8401, and application method of inspection shall be in accordance with JIS Z 8402.

3 Definitions The definitions of principal terms used in this Standard shall be as follows:

(1) **straight asphalts** The bituminous substances obtained from the crude oil by treating with an ordinary or a reducing pressure distillation plant or others.

Remarks : Depending on the grade of crude oil, blowing a small quantity of air or blending an asphalt having different penetration may be carried out.

Straight asphalts with the penetration 40 or less are used principally for industries and others, and those exceeding 40, mainly for road pavements and constructions for water use.

(2) **blown asphalts** The oxidative polymerized substance obtained by heating straight asphalt with a sufficient amount of blowing air.

Remarks : Because blown asphalts have a high softening point and small susceptibility, they are used for waterproofings, electric insulations and others.

(3) **asphalts for waterproofing constructions** Asphalts with the improved properties for a waterproofing layer.

Remarks : Mainly used for waterproofing constructions for reinforced concrete or steel frame buildings, and the equivalent.

(4) **penetration** The penetration indicates the measure for the hardness of an asphalt, and is expressed by the penetrating length of the specified needle into the sample perpendicularly under the test conditions. 0.1 mm is defined as one unit.

Further, since the penetration varies according to temperature, the penetration shall be expressed by its test temperature.

The penetration in this Standard is measured at 25 °C, which is expressed by penetration (25 °C).

(5) **softening point** The softening point is defined as the temperature at which asphalts begin to soften and at which the sample sags downward the specified distance, when the

sample is heated under the test conditions.

- (6) **ductility** The ductility expresses the ductile property of asphalts, and is measured by the distance to which it will elongate before breaking when both ends of the specimen of the form specified are stretched at a test temperature (15 °C or 25 °C) and at a test speed, and the distance is expressed in cm.

Further, since the ductility varies according to temperature, the ductility shall be expressed by its test temperature.

- (7) **soluble content in toluene** The content is the measure expressing the purity of the asphalt. The sample is dissolved in toluene, and the insoluble content is removed through filtering. The content is expressed in percentage.
- (8) **flash point** The lowest temperature of a sample at which a mixture of air and oil vapor catches fire, when the sample is heated in test conditions and a small flame approaches the oil surface.
- (9) **mass variation percentage by thin-film heating and penetration residual percentage after thin-film heating** These percentages are the measure for the estimation of the deteriorating tendencies of asphalts in the state of thin film by heating; by heating the samples under the test conditions, variations of the masses and the penetrations before and after heating are obtained and expressed in percentage to the value before heating.
- (10) **evaporation mass variation percentage** An evaporation mass variation percentage is the measure for evaluation of the stability of asphalts during storage by heating; by heating the samples under the test conditions, the variation of mass before and after heating is expressed in percentage.
- (11) **penetration ratio after evaporation** A penetration ratio after evaporation is the measure for evaluation of the tendency of the separation of light quality content from heavy quality content during storage by heating; by heating the samples under the test conditions, the ratio of the penetration of the not stirred sample to that of the stirred sample is expressed in percentage.
- (12) **penetration index** The index expresses the susceptibility of asphalts, and is obtained from the relation between the penetration and the softening point of the sample (refer to 6.11).
- (13) **susceptibility** The susceptibility expresses the variable properties of asphalts according to a high or low temperature such as the hardness, the viscosity or the like.
- (14) **density** Density means mass per unit volume of asphalts, which is expressed in g/cm³.

Since the density varies according to temperature, the density shall be indicated by its test temperature.

The density in this Standard is measured at 15 °C, which is expressed by density (15 °C).

- (15) **Saybolt Furol second** The second is the measure for the relative viscosities of asphalts at each specified temperature; it means the time required for flowing down of the sample of the specified volume through the small hole of the testing apparatus, and is expressed in second.
- (16) **kinematic viscosity** The viscosity is the quotient of the viscosity divided by the

density of the liquid in the same states (temperature and pressure), and expressed in cm^2/s {St} or mm^2/s {cSt}. In this Standard, the kinematic viscosity is obtained by multiplying the time for which a specific quantity of the sample heated to test temperature flows through capillaries by kinematic viscosity constant.

- (17) **Fraass brittle point** The Fraass brittle point is the measure for the flexibility of asphalts at a low temperature, and is a temperature at which the thin film of asphalts becomes brittle and cracks appear at first, when the thin film of asphalts coated on a steel plate is cooled under specified conditions and bent.
- (18) **sagging length** The length is the measure for a fluid resistance of asphalts at a high temperature; and is defined as the sagging length of the sample expressed in mm when the sample is poured in a templet of the specified form and hung perpendicularly under the test conditions.
- (19) **heating stability** The heating stability is the measure for a thermal stability of asphalts at the time of heating to melt, and is expressed as the difference of the Fraass brittle points before and after heating, when the sample is heated under specified conditions.

4. Classification The asphalts shall be classified into three types : straight asphalts, blown asphalts, and asphalts for waterproofing construction.

Straight asphalts and blown asphalts shall be classified according to the penetration at 25 °C as shown in Table 1.

Asphalts for waterproofing constructions shall be classified according to the use as shown in Table 2.

Table 1 Classification of straight asphalt and blown asphalt

Classification		Penetration (25 °C)
Straight asphalt	0 to 10	0 to 10 incl.
	10 to 20	Over 10 to 20 incl.
	20 to 40	Over 20 to 40 incl.
	40 to 60	Over 40 to 60 incl.
	60 to 80	Over 60 to 80 incl.
	80 to 100	Over 80 to 100 incl.
	100 to 120	Over 100 to 120 incl.
	120 to 150	Over 120 to 150 incl.
	150 to 200	Over 150 to 200 incl.
Blown asphalt	200 to 300	Over 200 to 300 incl.
	0 to 5	0 to 5 incl.
	5 to 10	Over 5 to 10 incl.
	10 to 20	Over 10 to 20 incl.
	20 to 30	Over 20 to 30 incl.
	30 to 40	Over 30 to 40 incl.

Table 2 Classification of asphalt for waterproofing construction

Classification		Use
Asphalt for waterproofing construction	Grade 1	Used for the parts of indoor and underground constructions under suitable temperature conditions during and after constructions, having ordinary susceptibility, and relatively soft property.
	Grade 2	Used for roofings for walk of a slow inclination in general districts, having relatively small susceptibility.
	Grade 3	Used for exposed roofings in general districts or roofings in relatively high temperature districts, having small susceptibility.
	Grade 4	Used for roofings and other parts in general districts as well as in cold districts having especially small susceptibility, and relatively soft property.

5. Quality and performance The asphalt shall be uniform and contain almost no water. When heated up to 180 °C, it shall not foam significantly. When tested according to 6, straight asphalts and blown asphalts shall comply with the requirements in Table 3, and asphalts for waterproofing constructions, with the requirements in Table 4, respectively.

Table 3 Quality of straight asphalt and blown asphalt

Classification	Penetration (25 °C)	Softening point °C	Ductility		Soluble content in toluene mass %	Flash point °C	Thin – film heating			Evaporation		Pene- tration index	Density (15 °C) g/cm³
			(15 °C) cm	(25 °C) cm			Mass variation percentage mass %	Penetration residual percentage %	Mass variation percentage mass %	Penetration ratio after evaporation %			
Straight asphalts	0 to 10	55.0 min.	–	–	99.0min.	260 min.	–	–	0.3 max.	–	–	–	1.000 min.
	10 to 20	Over 10 to 20 incl.	–	5 min.	–	–	–	–	–	–	–	–	–
	20 to 40	Over 20 to 40 incl.	–	50 min.			–	–		–	–		
	40 to 60	Over 40 to 60 incl.	10 min.	–			–	58 min.		–	110 max.	–	
	60 to 80	Over 60 to 80 incl.	100 min.	–			–	55 min.		–	–	–	
	80 to 100	Over 80 to 100 incl.	–	–	–	–	50 min.	–	–	–	–	–	
	100 to 120	Over 100 to 120 incl.	–	–			–	–	–	–	–		
	120 to 150	Over 120 to 150 incl.	38.0 to 48.0	–	–	240 min.	–	–	0.5 max.	–	–	–	–
150 to 200	Over 150 to 200 incl.	30.0 to 45.0	–	–			1.0 max.	–	–	–	–		
200 to 300	Over 200 to 300 incl.	–	–	–	210 min.	–	–	–	–	–	–	–	
Blown asphalts	0 to 5	130.0 min.	–	0 min.	98.5 min.	210 min.	–	–	0.5 max.	–	–	3.0 min.	–
	5 to 10	110.0 min.	–	–		–	–	–	–	–	–	3.5 min.	–
	10 to 20	90.0 min.	–	1 min.		–	–	–	–	–	–	2.5 min.	–
	20 to 30	80.0 min.	–	2 min.		–	–	–	–	–	–	–	–
	30 to 40	Over 30 to 40 incl.	65.0 min.	–		3 min.	–	–	–	–	–	1.0 min.	–
		–	–	–		–	–	–	–	–	–	–	–

Remarks: For straight asphalt of grades 40 to 60, 60 to 80, 80 to 100 and 100 to 120, each kinematic viscosity at 120 °C, 150 °C and 180 °C respectively shall be added to the test report.

Table 4 Quality of asphalts for waterproofing constructions

Classification		Softening point °C	Penetration (25°C)	Penetration index	Mass variation percentage after evaporation mass %	Flash point °C	Soluble content of toluene	Fraass brittle point °C	Sagging length mm	Heating stability (difference of Fraass brittle points) °C
Asphalts for waterproofing constructions	Grade 1	85 min.	25 to 45 incl.	3.5 min.	1 max.	250 min.	98 min.	– 5 max.	–	5 max.
	Grade 2	90 min.	20 to 40 incl.	4.0 min.	1 max.	270 min.	98 min.	– 10 max.	–	
	Grade 3	100 min.	20 to 40 incl.	5.0 min.	1 max.	280 min.	95 min.	– 15 max.	8 max.	
	Grade 4	95 min.	30 to 50 incl.	6.0 min.	1 max.	280 min.	92 min.	– 20 max.	8 max.	

6. Test method

6.1 Sampling method Samples of asphalts shall be taken according to the sampling method for asphalts specified in JIS K 2251.

6.2 Test apparatus in general The insulation performance of test apparatus, when insulation resistance between the terminal of a power source and the outer case is measured under a state wherein the electric circuit is closed, shall be 5 MΩ or over. However, in the case of including an electric heating circuit, 0.5 MΩ or over will suffice for the purpose.

6.3 Test method of penetration

6.3.1 Summary of test method Measure the penetrating length of the specified needle into the sample kept at a fixed temperature in a thermostatic water bath in a fixed time.

6.3.2 Penetration test apparatus The penetration test apparatus shall be composed of the following (1) to (6), and an example of the assembly of (1) to (3) is shown in Fig. 1.

(1) Penetrometer

- (a) **Needle** The needle shall be of the shape and dimensions as shown in Fig. 2, made of stainless steel (SUS440C specified in JIS G 4303) or one equivalent or superior thereto in hardness, attached to the center of the stem made of brass, and its mass shall be 2.5 ± 0.02 g.

The roughness of the ground surface of the taper part shall be 0.2 μm to 0.3 μm.

- (b) **Dropping apparatus** The apparatus shall have such construction that by pushing the stopper, allow the needle with the needle holder and the weight to penetrate into the sample perpendicularly; furthermore, the frictional resistance against the penetration shall be extremely small.
- (c) **Needle holder** The holder shall be the one for holding the needle as shown in Fig. 1, and its mass shall be 47.5 ± 0.02 g.
- (d) **Weight** The weight shall be of ring shape to be attached to the needle holder as

shown in Fig. 1, made of brass, and its mass shall be 50 ± 0.05 g.

- (e) **Dial gauge** The dial gauge shall be as shown in Fig. 1 and shall be equipped with the graduation by scaling 0.1 mm of penetration depth of needle as 1 in penetration, and the rack shall be movable up and down 40 mm and over.
- (f) **Test table and mounting** The mounting shall be provided with a test table which can be moved up and down, a pillar made of metal, a level and an adjusting screw for the horizontal, and to the pillar, an arm for the dial gauge shall be attached, and a mechanism for minute adjustment to contact the tip of the needle with the surface of the sample, necessary in testing, shall also be provided.
- (2) **Test container** The container shall be a metal or heat resistant glass flat bottomed cylinder with dimensions as shown in Fig. 3 (a) shall be used for the sample of penetration under 200. The container having dimensions as shown in Fig. 3 (b) shall be used for the sample of penetration 200 or over.
- (3) **Glass container and tripod-type metallic stand** The glass container shall be made of borosilicate glass-1 specified in JIS R 3503 and an example thereof is given in Fig. 4, and the tripod-type metallic stand shall have the shape and dimensions as shown in Fig. 4, made of brass (or nickel plated or chromium plated brass made).

Remarks : The glass container corresponds to Fig. 139 specified in JIS K 2839.

- (4) **Thermostatic water bath** The bath shall be a thermostatic water bath of volumetric capacity at least 10 l capable of placing the test container and the glass container in a row and of keeping at 25 ± 0.1 °C.

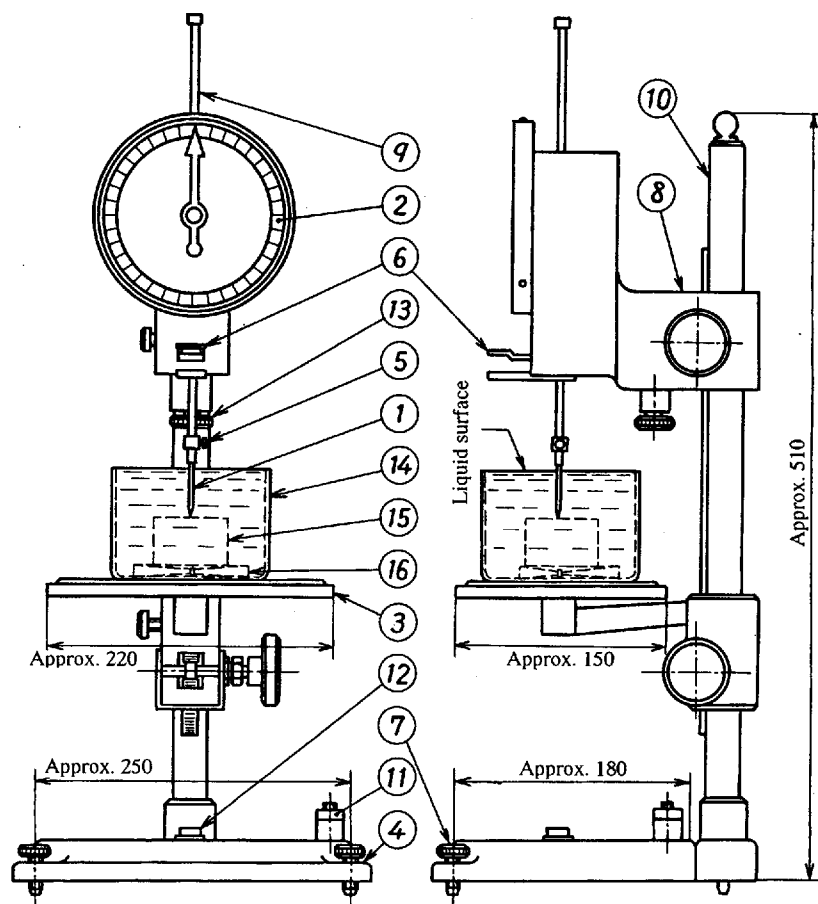
Further, a perforated mounting is installed at a position of at least 100 mm under the water surface and at least 50 mm from the bottom.

- (5) **Second watch** A stopwatch whose accuracy shall be within ± 0.05 % per 15 min, and the minimum graduation shall be 0.1 s, or an electric timer and others shall be used.
- (6) **Thermometer** The thermometer shall be of thermometer number 17 (VIS) specified in JIS B 7410. The thermometer shall be corrected by preliminarily obtaining errors in graduation at each test temperature in accordance with Annex to JIS B 7410.

Remarks : An automatic tester may be used. However, it shall be used after confirming no significant difference from the results obtained by this method in accordance with JIS Z 8402.

In case where a doubt is arisen concerning the test results obtained with the automatic tester, the result obtained by this Standard shall be adopted.

Unit : mm



- | | |
|--------------------------------------|---|
| ① Needle | ⑨ Rack for the measurement of penetration |
| ② Dial gauge | ⑩ Pillar |
| ③ Test table | ⑪ Weight |
| ④ Mounting | ⑫ Level |
| ⑤ Needle holder | ⑬ Mechanism for minute adjustment |
| ⑥ Stopper | ⑭ Glass container |
| ⑦ Adjusting screw for the horizontal | ⑮ Test container |
| ⑧ Arm for dial gauge | ⑯ Tripod-type metallic stand |

Fig. 1 Penetrometer (an example)

Unit : mm

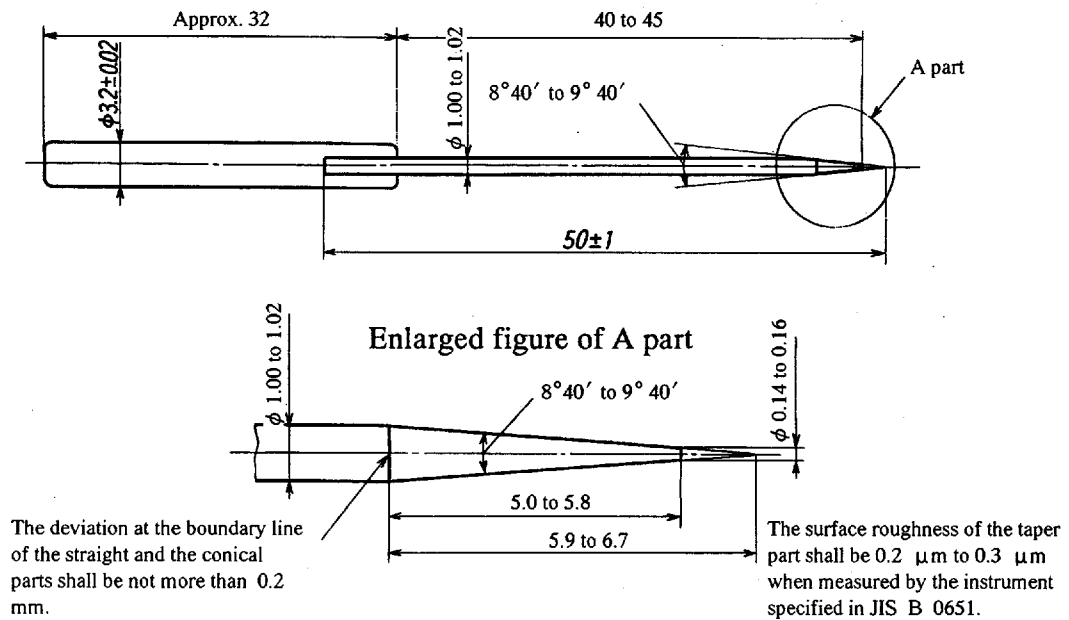


Fig. 2 Needle for penetration test

Unit : mm

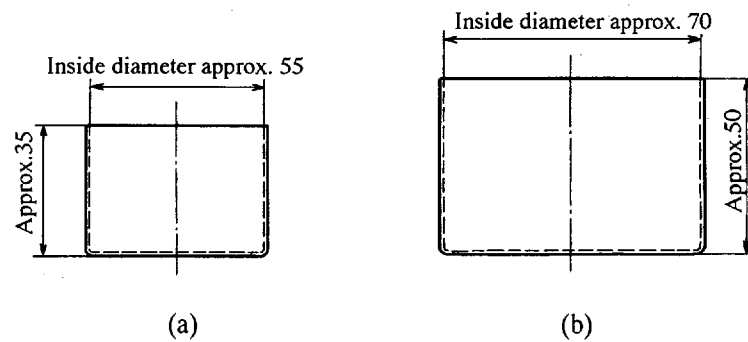


Fig. 3 Test container

Unit : mm

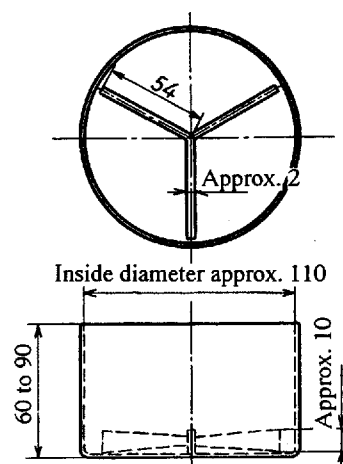


Fig. 4 Glass container and tripod-type metallic stand

6.3.3 Preparation of sample The preparation of the sample for penetration test shall be as follows.

- (1) The sample shall be melted by avoiding the local overheating and by not raising the temperature to 90 °C or higher than the expected softening point, and at a temperature as low as possible with slow stirring avoiding foams in the sample. The sample shall not be heated for 30 min or longer.
- (2) When the sample has sufficient fluidity and uniformity, take the sample in a test container. The quantity of the sample shall be such that the depth of the sample shall be 10 mm or deeper than the expected penetration length of the needle.
- (3) Cover the test container with a filter paper, an aluminium foil or the like for protection from dust, and leave it for 1 h to 1.5 h at room temperature of 15 °C to 30 °C. In the case of the use of the test container of Fig. 3 (b), leave it for 1.5 h to 2 h.

Then place it on the perforated mounting in the thermostatic water bath kept at 25 ± 0.1 °C in a row with the glass container placed the tripod stand in it, and leave for 1 h to 1.5 h. When the container of Fig. 3 (b) is used, it shall be left for 1.5 h to 2 h.

6.3.4 Procedure of test The procedure of a penetration test shall be as follows.

- (1) Confirm that waterdrops or foreign substances do not adhere on the needle holder, the weight, the stopper and others. Every time of the test, clean the needle with a gauze wetted with toluene or other suitable solvent, then wipe the needle with a dry gauze to the direction of the needle tip and attach it to the needle holder.
- (2) Transfer the test container on the tripod stand of the glass container in thermostatic water bath, place the glass container filled with water as it is on the test table of the penetrometer. By adjusting the light source properly, project the shadow of the tip of the needle on the surface of the sample, and allow the tip of the needle to contact the surface of the sample by adjusting the height of the test table so as to contact the tip of the needle with its shadow.
- (3) Gently push the rack which is engaged in the pinion of the dial gauge to the upper end of the needle holder, adjust the indicator of the dial gauge at 0 point, then press the stopper and allow the needle to penetrate into the sample by self weight for 5 s ⁽¹⁾. Press the rack again to the upper end of the holder gently, read the indication of the dial gauge to the nearest 0.5. If the test container moves during the measurement, repeat the measurement.

Note ⁽¹⁾ For the accuracy of the penetration period, the following procedures are desirable. Start the second watch before start of the test. When the second watch indicates an arbitrary graduation, press the stopper and allow the needle to penetrate, then release the stopper after the specified period of time has elapsed precisely.

- (4) The measurements shall be conducted three times on the sample in the same container, and every measuring point shall be always 10 mm or over apart from the peripheral wall of the test container, and in the second test and thereafter, the position shall be selected at a point 10 mm or over apart from the previous penetration point ⁽²⁾.

After each measurement, return the sample and the glass container into the

thermostatic water bath and renew the water in the glass container.

Note (2) For the test of the sample having the penetration 200 or over, prepare three needles, don't remove the needles from the sample until the measurements have been completed.

Further, the following procedure may be allowed : Prepare respective sample for three test containers. Perform each one measurement per one container.

- (5) Obtain the difference of maximum and minimum values among measured values and the average value of the measured values. If the difference of the maximum and minimum values is within the range specified in Table 5, round the average value to the integer in accordance with JIS Z 8401 and make it the penetration (25 °C).

Table 5 Range of measured penetration

Average value of measured penetration	Range of measured penetration
0 or over to and excl. 50.0	2.0
50.0 or over to and excl. 150.0	4.0
150.0 or over to and excl. 250.0	6.0
250 or over	8.0

6.3.5 Precision The precision of a penetration test shall be as follows .

- (1) **Repeatability** When duplicate tests are carried out on the same sample in the same test room by the same person and the apparatus at different time or days, the difference of test results shall not exceed the tolerance given in Table 6.

Table 6 Repeatability

Penetration (25 °C)	Tolerance
Under 50	1
50 or over up to and incl. 300	3 % of average value

- (2) **Reproducibility** When two tests are carried out in different test rooms and by different persons and apparatuses on the same sample for one time respectively, the difference of two test results shall not exceed the tolerance given in Table 7.

Table 7 Reproducibility

Penetration (25 °C)	Tolerance
Under 50	4
50 or over up to and incl. 300	8 % of average value

6.4 Test method for softening point (ring-and-ball method)

6.4.1 Summary of test method Fill the specified rings with samples, support them horizontally in a water or glycerin bath and place a specified ball on the center of each sample. Read the temperatures at which the sample encapsulating the ball touches the bottom plate of the ring holder, when the bath temperature is raised at a rate of 5 °C per minute.

6.4.2 Softening point test apparatus The softening point test apparatus shall be composed of the following (1) to (7), and an example thereof is given in Fig. 5.

- (1) **Ring** The ring shall be a shouldered ring having the shape and dimensions as shown in Fig. 6 (a), made of brass or brass with nickel or chromium plating.
- (2) **Ball** The ball shall be a 3/8 (dia. 9.525 mm) grade 60 or superior ball specified in JIS B 1501 with the mass of 3.5 ± 0.05 g.
- (3) **Ball-centering guide** The guide shall have the shape and dimensions as shown in Fig. 6 (b), made of brass or brass with nickel or chromium plating.
- (4) **Ring holder** The ring holder shall be made of brass or brass with nickel or chromium plating as shown in Fig. 7 and capable of supporting rings and a thermometer as described below:

Rings can be supported horizontally under such a position that the distance between the upper surface of the ring and the upper edge of the heating bath is 75 mm or over, and the distance between the former and the bath liquid surface is 50 mm or over downwards, respectively.

The distance between the lower surface of the ring and the upper surface of the bottom plate of ring holder shall be 25 mm, and the bottom plate shall be 13 mm to 19 mm above the bottom of the heating bath.

The thermometer shall be supported at the position where bottom end of the mercury bulb is the same horizontal plane as the lower surface of the ring, and within 10 mm from the ring, without touching the sample shelf.

- (5) **Heating bath** The material of a heating bath shall be made of borosilicate glass-1 specified in JIS R 3503, and an example of the dimensions is as given in Fig. 5.

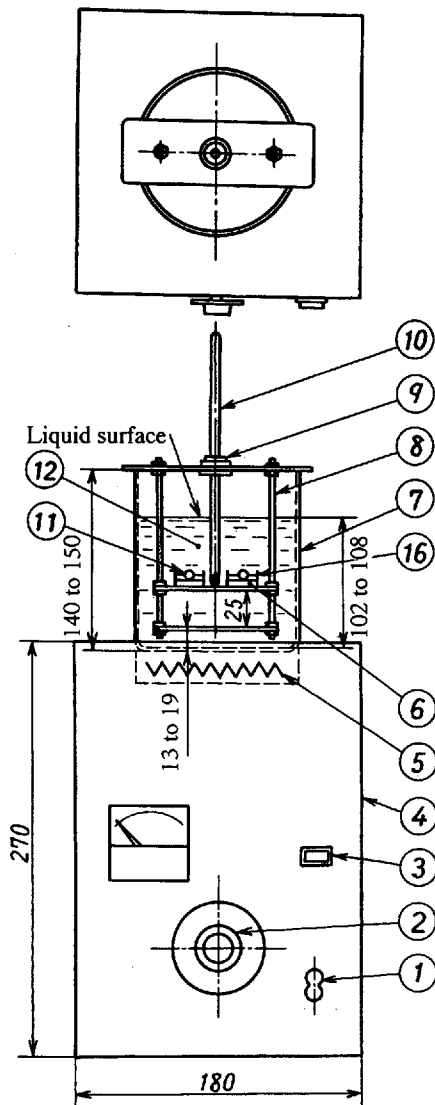
Remarks : Fig. 140 specified in JIS K 2839 corresponds to the heating bath.

- (6) **Thermometer** The thermometer shall be that of thermometer No.33 (SP) and 34 (SP) specified in JIS B 7410, which shall be preliminarily corrected by obtaining the error of graduation in accordance with Annex to JIS B 7410.
- (7) **Heater** The heater shall be a gas burner or an electric heater capable of raising the bath-liquid temperature at a heating rate of 5 °C per minute or over, and the insulation resistance shall conform to 6.2.

Remarks : An automatic tester may be used. However, after confirming no significant difference from the result obtained by this method in accordance with JIS Z 8402, the automatic tester shall be used.

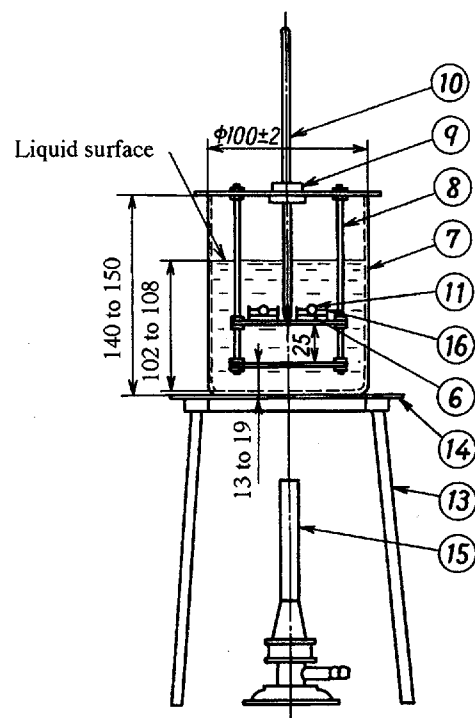
In the case where any doubt is arisen concerning the test result obtained with the automatic tester, the result obtained by this method shall apply.

Unit:mm



(a) Electric heating system

- ① Power switch
- ② Voltage regulator
- ③ Indication lamp
- ④ Mounting
- ⑤ Electric heater
- ⑥ Ring
- ⑦ Heating bath
- ⑧ Ring holder
- ⑨ Cork stopper
- ⑩ Thermometer
- ⑪ Ball
- ⑫ Bath liquid
- ⑬ Tripod stand
- ⑭ Wire net with ceramics
- ⑮ Gas burner
- ⑯ Ball-centering guide



(b) Gas heating system

Fig. 5 Softening point test apparatus (an example)

Unit : mm

Unit : mm

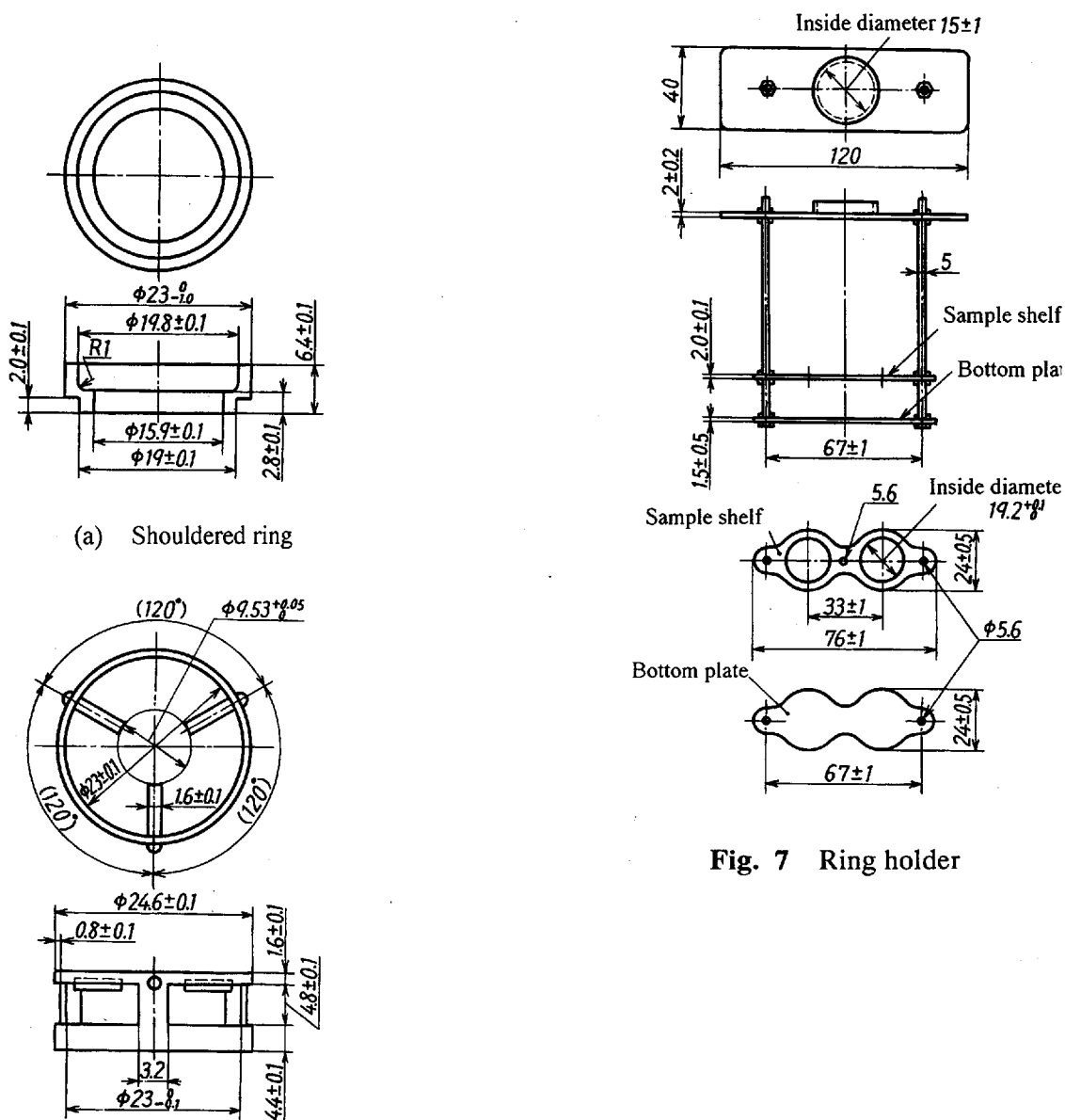


Fig. 7 Ring holder

(b) Ball-centering guide

Fig. 6 Ring and ball-centering guide

6.4.3 Preparation of samples The preparation of samples for a softening point test shall be as follows.

- (1) Avoid partial overheating of a sample, and fuse the sample at a temperature as possible with slowly stirring so as not to reach the temperature 90 °C or higher than low as the expected softening point so that no foaming occurs in the sample. Do not heat the sample for 30 min or longer.
- (2) Heat two rings at the same temperature as the sample, place them on the metallic plate coated with release agents such as silicone grease, an equal weight mixture of glycerin-dextrin or others.
- (3) Pour a slight excess of the sample into two rings, and allow to stand to cool at room

temperature for 30 min or longer. Cool the sample which is soft at room temperature at a temperature at least 8 °C lower than an expected softening point for 30 min. After cooling, scrape off the excess sample to the same height as the upper edge of the ring by using a warmed knife, spatula, etc.

- (4) The tests shall be completed within 4 h after pouring the sample into the rings.

6.4.4 Procedure of test The procedure of a softening point test shall be as follows.

- (1) Place two rings poured with the sample, the thermometer and the ball – centering guides on the ring holder, and assemble in the heating bath.
- (2) When the softening point is 80 °C or less, fill the heating bath to the height of 102 mm to 108 mm with the distilled water newly boiled (³) and cooled to 5 °C, and when it is over 80 °C, fill in the same way with glycerin at about 32 °C.

Note (³) If foam adheres on the surface of the sample, it may influence the test result, therefore, newly boiled distilled water shall be used for the bath water.

- (3) Maintain the bath temperature for 15 min at 5 °C for the softening point 80 °C or less and at 32 °C for the softening point over 80 °C.
- (4) Place the balls whose temperature has been preheated to the same temperature of the bath liquid with a pincette in the center of ball centering guides, and start heating.
- (5) From 3 min after the start of heating to the softening point, heat the bath whose temperature increases uniformly at a rate of 5 ± 0.5 °C per minute.

For precautions of not exposing to wind, an enclosure should be installed if necessary. If the temperature increasing rate can not be observed, retest with a new sample.

- (6) Read and record the indication of the thermometer when the sample becomes soft and elongates gradually, and finally touches the bottom plate. No correction on the exposing part of the thermometer shall be carried out. When the difference between the two test results exceeds 1 °C, retest.
- (7) The average value of two measured values shall be rounded to 0.5 °C unit and made the softening point.

6.4.5 Precision The precision of a softening point test shall be as follows:

- (1) **Repeatability** When duplicate tests are carried out in the same test room and by the same person and apparatus on the same sample in different days or time, the difference of test results shall not exceed the tolerance shown in Table 8.

Table 8 Repeatability

Softening point	Tolerance °C
80 °C or lower	1.0
Over 80 °C	2.0

- (2) **Reproducibility** When tests are carried out in two different test rooms, by different persons and apparatuses on the same sample each one time, respectively, the difference of obtained test results shall not exceed the tolerance shown in Table 9.

Table 9 Reproducibility

Softening point	Tolerance °C
80 °C or lower	4.0
Over 80 °C	8.0

6.5 Test method for ductility

6.5.1 Summary of test method After the preparation of the sample of the specified shape by pouring the sample in the mould, keep it in a thermostatic bath at a fixed temperature, and then elongate the sample horizontally with a specified speed and measure the distance to which it elongates before breaking.

6.5.2 Ductility test apparatus The ductility test apparatus shall be composed of the following (1) to (4), and an example thereof is given in Fig. 8.

- (1) **Test device for ductility** The device shall be a water bath having the shape and dimensions as shown in Fig. 8, the surface of its inside bottom shall be white, and the device shall consist of the following (a) to (d).
- (a) **Travelling mounting** The mounting shall be of the shape and dimensions as shown in Fig. 9, made of metal, with least vibrations in travelling by means of rotation of a threaded main shaft, provided with pins for fitting the holes of clips of moulds, a holder for a thermometer, and an attaching device for the pointer of the ductility.

An engaging section of threads of the main shaft shall be split threads.

- (b) **Fixed mounting** The mounting shall be of the metallic one having the form as shown in Fig. 8 and having pins for fitting the holes of the clips of the moulds. It shall be fixed to the internal wall of the bath.

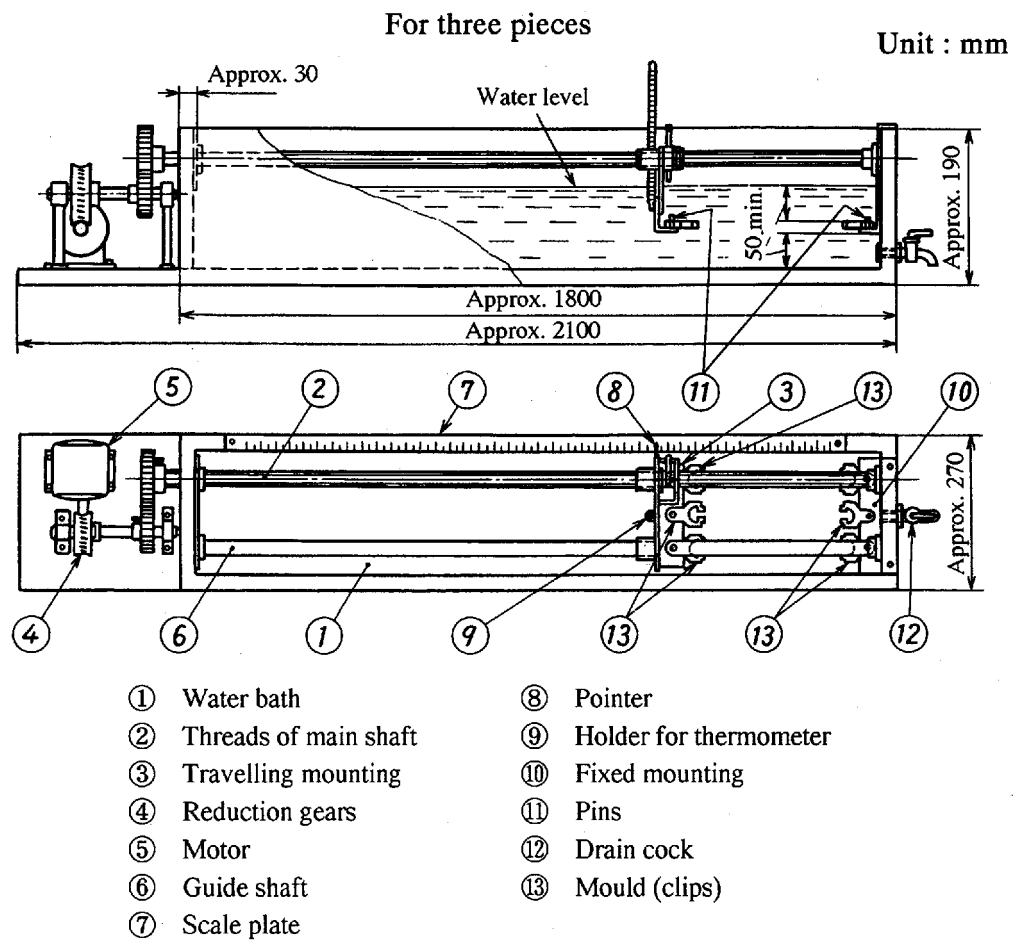


Fig. 8 Construction of test device for ductility (an example)

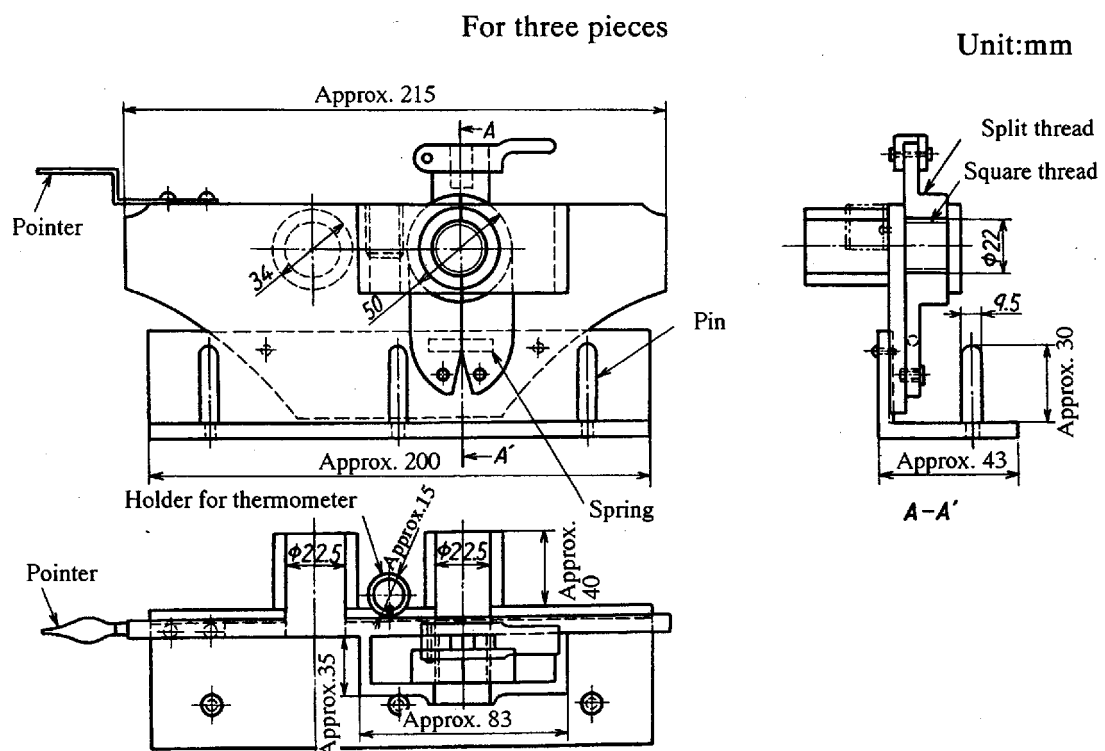


Fig. 9 Travelling mounting (an example)

Unit : mm

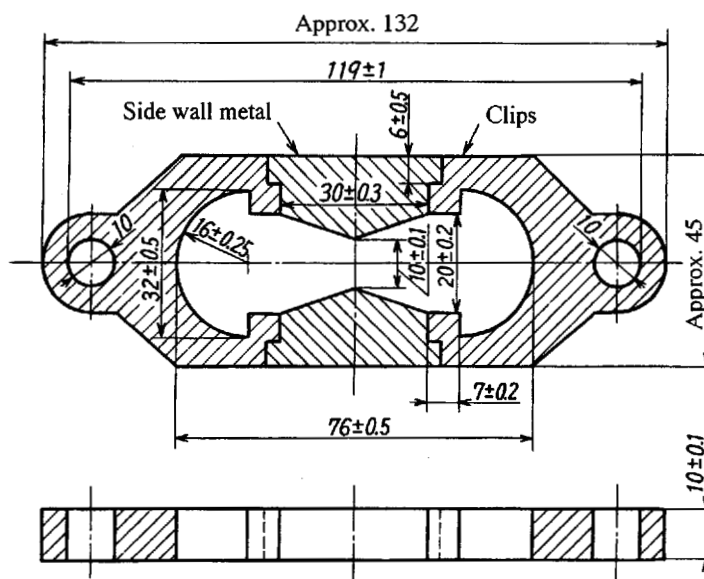


Fig. 10 Mould

- (c) **Travelling apparatus** The apparatus shall be a motor driven apparatus capable of elongating the samples at a speed of 5 ± 0.25 cm per minute, and shall have a construction capable of rotating the main shaft threads by a motor installed outside of the water bath.
- (d) **Scale plate** The plate shall be attached to the upper side wall of the water bath, having 150 cm in length for reading the travelling length of the travelling mounting, and the minimum scale value shall be 0.5 cm.

Remarks : For regulation of water temperature, an apparatus for circulating water of which the temperature is regulated may be used.

- (2) **Mould** The mould shall have the shape and dimensions as shown in Fig. 10, consist of two clips and two side wall metals, made of brass, and the inside surface shall have precise finishing.
- (3) **Metallic plate** The plate shall be made of brass or stainless steel, so smooth as to have a close contact throughout the plane of the mould, 2 mm or over in thickness, and about 150 mm $\times$ 170 mm in size.
- (4) **Thermostatic water bath** The bath shall have 10 l or over in volumetric capacity, and be provided with a temperature regulator capable of maintaining the water temperature within ± 0.1 °C of the test temperature and perforated mounting at a position of 100 mm or over in the depth from the water surface and 50 mm or over from the bottom.

6.5.3 Preparation of samples The preparation of samples for ductility test shall be as follows.

- (1) The sample shall be melted by avoiding local overheating and by not raising the temperature to 90 °C or higher than the expected softening point, and at a temperature as low as possible with slow stirring to avoid generation of foam in the sample. The sample shall not be heated for 30 min or longer.

- (2) Pass the sample through a wire sieve of 300 μm in sieve opening specified in JIS Z 8801, and pour into the mould which has been assembled on the metallic plate by a little excess ⁽⁴⁾.

Note ⁽⁴⁾ In filling the mould by the sample, take care neither to destroy nor to deform the mould. Cautions shall be taken not to produce bubbles.

- (3) When the mould is assembled on the metallic plate, coat the metallic plate surface and inside wall of side wall metal of the mould with a release agent such as silicone grease or a mixture of equal weight of glycerin-dextrin or others in advance to prevent the sample from sticking.
- (4) After pouring the sample into the mould, allow it to cool by standing at room temperature for 30 min to 40 min, as placed on the metallic plate, transfer it to the perforated mounting in the thermostatic water bath kept at $\pm 0.1^\circ\text{C}$ of the test temperature, and immerse for 30 min.
- (5) Then, take out the sample together with the metallic plate from the thermostatic water bath, and cut off the excess of the sample above the mould along the upper surface of the mould by using a warmed knife, spatula, etc. Place it again with the metallic plate in the thermostatic water bath, and leave gently for 85 min to 95 min.

6.5.4 Procedure of test The procedure of a ductility test shall be as follows.

- (1) Fill the water in the test device for ductility ⁽⁵⁾, and maintain the water temperature at the test temperature $\pm 0.5^\circ\text{C}$ during the measurement of the ductility.

Note ⁽⁵⁾ When the samples are attached to the test device for ductility in testing, there shall be water layers of 25 mm or over above and under the samples.

- (2) Take out the sample together with the mould from the thermostatic water bath, detach the side wall of the mould, fit the holes of the clips to the pins of the test device for ductility; adjust the pointer to 0, elongate ⁽⁶⁾ the sample at a speed of 5 ± 0.25 cm per minute by a motor. When the sample breaks, read the indication of the pointer in 0.5 cm unit, and record it.

Note ⁽⁶⁾ When the sample appears on the water surface or touches the bottom of the test device, regulate the density of the water by adding methanol or sodium chloride to the water of the test device.

Do not disturb water in measurement of ductility.

- (3) Round off the average value of three measured values to the nearest 1 cm in accordance with JIS Z 8401, and make it the ductility. Append the test temperature.

6.5.5 Precision The precision shall not be specified.

6.6 Test method for soluble content in toluene

6.6.1 Summary of test method The sample is dissolved in toluene and filtered through a glass fibre filter. The insoluble material is washed with toluene, dried, and the mass is weighed.

6.6.2 Test apparatus for soluble content in toluene and reagent The test apparatus for