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**Test methods of creosote oil,
prepared tar and tar pitch**

ICS 71.100.50; 75.140

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Aromatic Industry Association, Inc. (JAIA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 2425**:1983 is replaced with this Standard.

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Test methods of creosote oil, prepared tar and tar pitch

1 Scope This Japanese Industrial Standard specifies test methods of creosote oil, prepared tar and tar pitch produced from coal tar.

2 Normative references The standards listed in attached table 1 contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) listed in the table shall be applied.

3 Terms and definitions For the purpose of this Standard, the following terms and definitions apply.

- a) **density** mass per unit volume of sample and shall be expressed as follows by attaching its temperature condition

In this Standard the units of mass, volume and temperature shall be g, cm³ and °C respectively.

1) **density (15 °C)** density of sample at 15 °C, expressed in g/cm³

2) **density (40 °C)** density of sample at 40 °C, expressed in g/cm³

- b) **specific gravity** a ratio of the mass of a certain volume of sample to the mass of water of equal volume to it

Namely, it is the ratio of the density of the sample to the density of water and also called a relative density. The specific gravity shall be expressed as follow by attaching the temperature conditions of the sample and water.

Moreover, specific gravity is an absolute number.

1) **specific gravity (15/4 °C)**, ratio of the sample of a certain volume at 15 °C to the mass of water of equal volume to it at 4 °C.

- c) **Engler degree** a ratio of the time required when the specified volume of sample flows down from the pore of tester at the specified temperature to the time required when the same temperature and volume of distilled water flows down from the pore of tester

1) **Engler degree (40/20 °C)** Engler degree at 40 °C in temperature of sample and 20 °C in temperature of distilled water

4 General matters The general matters common to the tests shall be as specified below in addition to the matters specified in **JIS K 0050**.

- a) In the measurements or tests, when the scale values are to be read at the liquid level, reading either at the upper edge or at the lower edge of the liquid surface which has been raised by surface tension shall follow the descriptions as divided below. For creosote oil, reading at the upper edge shall be adopted in all cases.

- 1) Densimeter (hydrometer) At upper edge
 - 2) Measuring cylinder and others At lower edge
- b) All hydrometer and thermometers used shall be corrected by using a standard hydrometer and standard thermometer before use.

5 Sampling and preparation of specimen

5.1 Extraction of representative sample The representative sample shall be taken in accordance with Annex.

5.2 Preparation of specimen

5.2.1 For creosote oil The specimen shall be prepared by taking about 1 L of the representative sample and by mixing it.

5.2.2 For prepared tar The specimen shall be prepared by taking about 1 kg of the representative sample and by mixing it.

5.2.3 For tar pitch The specimen shall be prepared as follows.

- a) **Appliances** The following appliances shall be used.
- 1) **Sample reducer** As specified in **3.1.2** of Annex.
 - 2) **Iron mortar** A suitable one.
 - 3) **Closed type mixer** A suitable one.
 - 4) **Screens** Screens of 840 μm and 250 μm mesh.
 - 5) **Drier** A suitable hot air fan drier or vacuum drier.
- b) **Procedure** After grinding about 500 g of sample on an iron mortar or iron plate or powdering it to about 5 mm particle size by using a closed type mixer, reduce it to about 100 g by using a sample reducer or by the increment reduction method.

Then powder it to 840 μm or smaller or 250 μm or smaller in particle size according to the division of test items given in table 1, and then dry it in a drier (at 35 °C or lower)⁽¹⁾ for 4 h.

Table 1 Particle size of test specimen

Test item	Particle size
Measurement of density, measurement of softening point, determination of fixed carbon content, and determination of ash content	840 μm max.
Determination of toluene-insoluble content, and determination of quinoline-insoluble content	250 μm max.

Note ⁽¹⁾ Put 30 g to 50 g of powdered sample on a stainless steel pan (156 mm in length, 25 mm in height, and 125 mm in width) or the like, and level it to a uniform thickness of about 2 mm, and then dry it in a drier.

5.3 Preparation of dehydrated specimen

5.3.1 For creosote oil The dehydrated specimen shall be prepared by heating about 200 ml of the sample to about 50 °C, and after adding about 60 g of calcium chloride specified in **JIS K 8125** or about 100 g of dried salt⁽²⁾ as drying agent and mixing well, maintaining the mixture at 50 °C for about 10 min, and after natural cooling, filtering it to obtain the dehydrated specimen.

Note (2) This salt shall be prepared by drying salt at about 120 °C for 1 h or longer.

5.3.2 For prepared tar The dehydrated specimen shall be prepared either by the method using a distilling flask or by the method using a hydroextractor mentioned below.

a) **Method using distilling flask** The method using distilling flask shall be as follows.

1) **Appliances** The following appliances shall be used.

1.1) **Distilling flask** A flask of about 500 ml capacity, as shown in figure 1.

1.2) **Thermometer** A bar mercury thermometer graduated by 1 °C in the range of 0 °C to 360 °C and scaled with numeral at every 10 °C, having dimensions given in table 2.

Table 2 Thermometer

Unit: mm	
Item	Dimension
Total length	330 ± 10
Outer diameter of stem	6 ± 1
Distance from lower end of mercury bulb to 0 °C graduation mark	60 ± 2

1.3) **Cooler** As shown in figure 2.

1.4) **Receptacle** A receptacle of about 50 ml, as shown in figure 3.

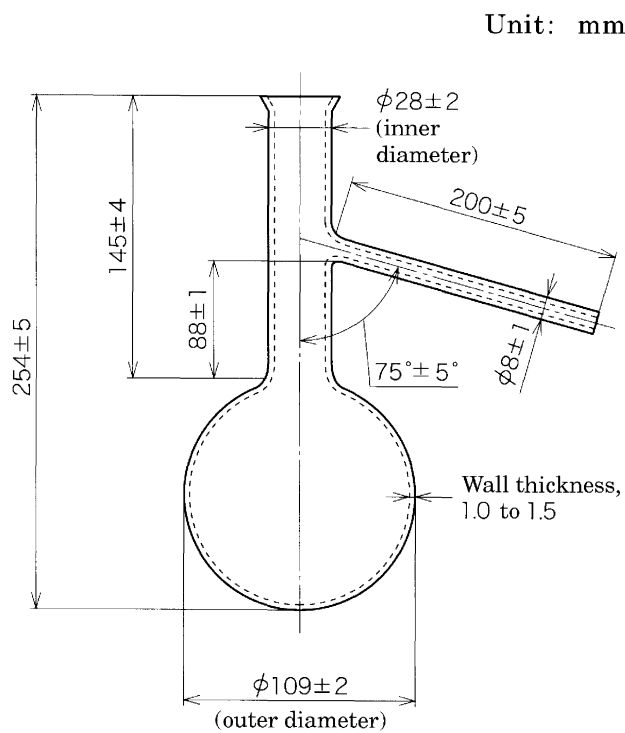


Figure 1 Distilling flask

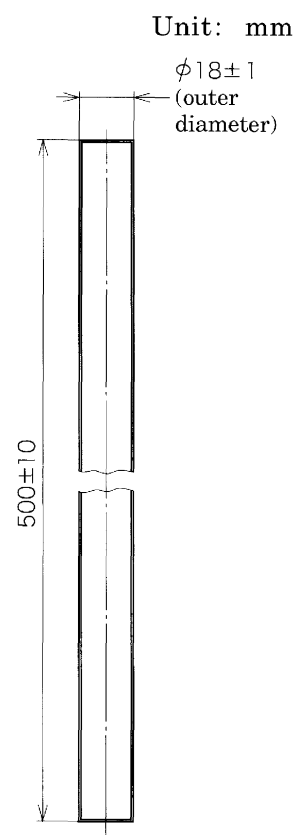


Figure 2 Cooler

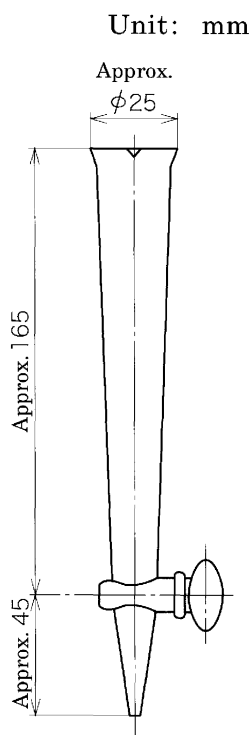


Figure 3 Receptacle

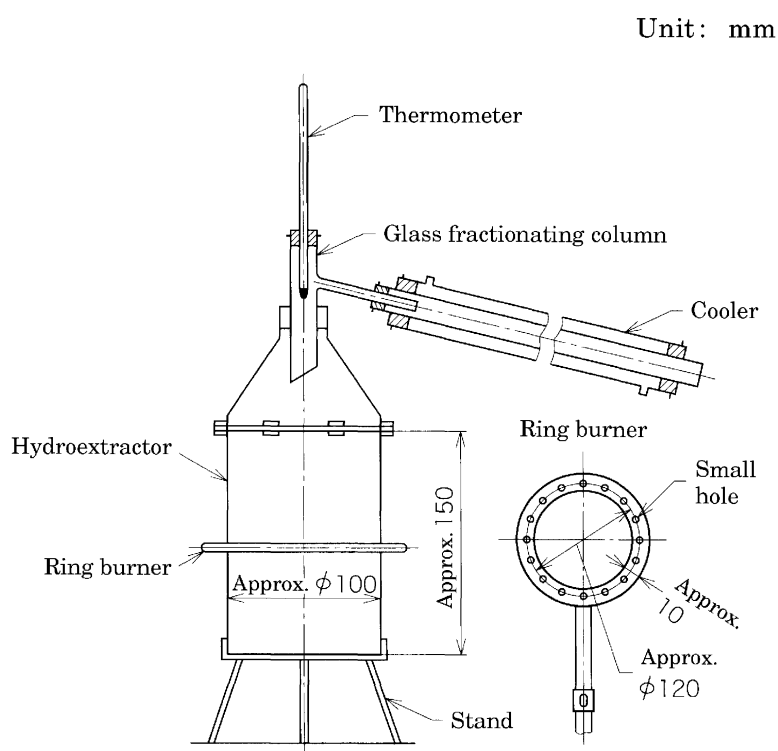


Figure 4 Example of dehydrating apparatus

- 2) **Procedure** Take about 300 g of specimen into a distilling flask, put pumices or glass pieces in the flask, attach a thermometer with a cork plug, and after placing the thermometer so that the upper and of the mercury bulb of the thermometer positions at the lower root of the branch tube of the distilling flask, put the distilling flask on a suitable heating device, with the branch tube inserted in the cooler by about 35 mm, and put a receptacle at the end of the cooler.

When crystals occurs in the cooler during distillation, let them flow down by a suitable degree of heating. Thus, when 180 °C is reached, stop heating, and let the distillate remaining in the cooler flow down into the receptacle, return the distillate in the receptacle to the tar in the flask after removing the water layer, and mix it well with the tar to obtain a dehydrated specimen.

When crystals deposit in the distillate in the receptacle, melt the crystals by heating the receptacle, and remove the water layer after its boundary has become distinct.

- b) **Method using hydroextractor** Take about 500 g of specimen into a metal hydroextractor, assemble the whole apparatus as shown in figure 4, and heat the test specimen from its upper layer to evaporate water.

As the water evaporates, move the position of the ring burner successively downward, and lastly heat the bottom of the hydroextractor to make all the water distilled out. Return the oil distilled out during this process to the tar in the hydroextractor after the hydroextractor has cooled, and mix the tar well with the oil to obtain a dehydrated specimen.

6 Measuring method for density

6.1 Measuring method for density of creosote oil

6.1.1 Measuring method for density by means of hydrometer

6.1.1.1 Measuring method for density by means of I-type hydrometer The measuring method for density by means of I-type hydrometer shall be as follows.

- a) **Appliances** The appliances shall be as follows.

- 1) **Hydrometer** An I-type-A hydrometer made of soda silicate glass of No. 9 or No. 10, graduated with the density (15 °C) as specified in **JIS K 2249**.
- 2) **Cylinder** A glass cylinder with out-flow mouth, about 40 mm in inner diameter and about 350 mm in length.
- 3) **Thermometer** A bar mercury thermometer graduated with 1 °C in the range of 0 °C to 100 °C and scaled with numeral at every 10 °C, having a suitable shape.
- 4) **Water bath** A thermostatic bath or a suitable bath having an inner capacity of 5 L or larger.

- b) **Procedure** Put the specimen heated to about 40 °C in a cleaned and dried cylinder, and put them into a water bath containing water maintained at about 40 °C, quietly float a hydrometer (densitometer) on the water, and leave it to stand still for 10 min. In the above procedure, take care not to allow the difference in temperature between the water in the bath and the specimen to exceed 5 °C. After leaving it standing still, take the reading of the hydrometer. Take enough care not to allow the specimen to adhere unduly onto the outside of neck of the hydrometer positioning above the water.

Take out the hydrometer and promptly put a thermometer and stir, then take the reading of the thermometer by positioning it so that the upper end of the mercury column appears a little above the liquid level, and take this temperature as the temperature of the specimen at the measurement. Take such a quantity of specimen into the cylinder that when the hydrometer is put in the water, the distance between its lower end and the cylinder bottom is not smaller than 15 mm, and make the measurement at 35 °C to 45 °C.

Calculate the density (40 °C) by the following formula:

$$D_{40} = D_t + 0.000\ 70(t - 40) - 0.000\ 5$$

where,

D_{40} : density (40 °C) (g/cm³)

D_t : measured density (g/cm³)

t : measured temperature (°C)

0.000 70 : temperature conversion factor of density
(g/cm³ °C⁻¹)

0.000 5 : temperature correction value of I-type hydrometer itself (g/cm³)

NOTE : The temperature correction value, for which generally the value of 0.000 5 is sufficient, shall be calculated by the following formula when extra precision is required.

$$\text{Temperature correction value} = 0.000\ 023 D_t \times (20 - t)$$

where, 0.000 023 : coefficient of thermal expansion of I-type hydrometer itself (°C⁻¹)

6.1.1.2 Measuring method for density by means of a specific gravity hydrometer The measuring method for density by means of a specific gravity hydrometer shall be as follows.

- a) **Appliances** The appliances shall be as follows.

- 1) **Specific gravity hydrometer** One of the following shall be used.

- 1.1) Large-size 19-piece set hydrometer of No. 6 or No. 7 specified in Annex 4 (normative) of **JIS B 7525**.
- 1.2) A hydrometer made of soft glass, graduated to 0.001 specific gravity values (15/4 °C), in the scale range of 1.000 to 1.100, and scaled by numeral at every 0.01; 300 mm ± 20 mm in length.

- 2) **Cylinder** As specified in **6.1.1.1 a) 2)**.

- 3) **Thermometer** As specified in **6.1.1.1 a) 3)**.
- 4) **Water bath** As specified in **6.1.1.1 a) 4)**.
- b) **Procedure** The procedure shall be in accordance with **6.1.1.1 b)**.
The following equation shall be used to calculate the density (40 °C):

$$D_{40} = D_t + 0.000\ 70(t - 40) - 0.000\ 6$$

where,

D_{40} : density (40 °C) (g/cm³)
 D_t : measured density (g/cm³)
 t : measured temperature (°C)
0.000 70 : temperature conversion factor of density (g/cm³ °C⁻¹)
0.000 6 : temperature correction value of the specific gravity hydrometer itself (g/cm³)

NOTE : The temperature correction value, for which generally the value of 0.000 6 is sufficient, shall be calculated by the following formula when extra precision is required.

$$\text{Temperature correction value} = 0.000\ 025 D_t \times (15 - t)$$

where, 0.000 025 : coefficient of thermal expansion of specific gravity hydrometer (°C⁻¹)

6.1.2 Measuring method for density by oscillation-type density meter The measurement of density for creosote oil by oscillation-type density meter shall be in accordance with **7.3** of **JIS K 0061**.

6.2 Measuring method for density of 235 °C to 315 °C fraction of creosote oil

6.2.1 Pycnometer method The pycnometer method shall be as follows.

- a) **Appliance** The following appliance shall be used:
Pycnometer As specified in figure 5.
- b) **Procedure** The following procedure shall be carried out.
- 1) Weigh a dried clean pycnometer with its lid on. Then fill this pycnometer with freshly distilled water at 40 °C to the degree of overflowing, and after putting the pycnometer in a water bath maintained at 40 °C for 30 min or longer, take it out and promptly remove the water in the capillary tube in the lid by absorbing it with a piece of paper or the like to make the liquid level to agree with the marked line of the capillary tube. After wiping off the moisture adhering to the outside of the pycnometer with a dry cloth, weigh the pycnometer.
 - 2) Then after removing the water in the pycnometer and drying it, carry out the same procedure as above by putting the specimen at 40 °C in the pycnometer, and lastly, after wiping off completely the water, oil, and the like

adhering to the outside of pycnometer, weigh the pycnometer and calculate the density of the specimen (40 °C) by the following formula:

$$D_{40} = \frac{M_3 - M_1}{M_2 - M_1} \times 0.992\ 2$$

where, D_{40} : density (40 °C) (g/cm³)
 M_1 : mass of dried pycnometer (g)
 M_2 : mass of pycnometer (g)
 M_3 : mass of (pycnometer + specimen) (g)
 0.992 2 : density of water (40 °C) (g/cm³)

Unit: mm

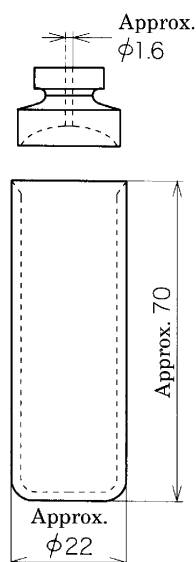


Figure 5 Pycnometer

6.2.2 Measuring method for density by oscillation-type density meter The measurement of density of 235 °C to 315 °C fraction of creosote oil shall be conducted in accordance with **7.3** of **JIS K 0061**.

6.3 Measuring method for density of prepared tar

6.3.1 Measuring method for density by means of a specific gravity hydrometer The measurement of density by means of a specific gravity hydrometer shall be as follows.

- a) **Appliances** The following appliances shall be used.
 - 1) **Specific gravity hydrometer** Either of the following.
 - 1.1) No. 6, 7, 8, 9, 10 or 11 large-size 19-piece set hydrometers specified in Annex 4 of **JIS B 7525**.
 - 1.2) A hydrometer made of soft glass, graduated to 0.005 specific gravity values (15/4 °C), in the scale range of 1.000 to 1.250, and scaled by numeral at every 0.05; 300 mm ± 20 mm in length.

- 2) **Cylinder** As specified in **6.1.1.1 a) 2)**.
 - 3) **Thermometer** As specified in **6.1.1.1 a) 3)**.
 - 4) **Water bath** As specified in **6.1.1.1 a) 4)**.
- b) **Procedure** Take about 500 g of specimen into a beaker, heat gradually on a water bath to about 55 °C while stirring well, and pour the specimen into a dried clean cylinder with care not to allow the bubbles to produce. Put the cylinder into a water bath heated to about 50 °C, and after ascertaining the absence of bubbles in the specimen, float a specific gravity hydrometer quietly at the centre of the cylinder, and leave it standing still for 10 min. Then take the reading of the hydrometer. Take sufficient care not to allow the specimen to adhere unduly to the outside of the neck of the specific gravity hydrometer positioning above the liquid.

Take out the specific gravity hydrometer, and after promptly putting a thermometer into the specimen and stirring, take the reading of the thermometer with its upper end of mercury column appearing a little above the liquid level, and take this reading as the temperature of the specimen at the measurement. Take such a quantity of specimen that, when the specific gravity hydrometer is put in the cylinder, the distance between its lower end and the cylinder bottom is 15 mm, and the measurement shall be made at 45 °C to 55 °C.

Calculate the density (15 °C) by the following formula:

$$D_{15} = D_t + 0.000\ 6(t - 15) - 0.001$$

where, D_{15} : density (15 °C) (g/cm³)

D_t : measured density (g/cm³)

t : measured temperature (°C)

0.000 6 : temperature conversion factor of density
(g/cm³ · °C⁻¹)

0.001 : temperature correction value of specific gravity hydrometer itself (g/cm³)

NOTE : The temperature correction value, for which generally the value of 0.001 is sufficient, shall be calculated by the following formula when extra precision is required.

$$\text{Temperature correction value} = 0.000\ 025 \times D_t \times (15 - t)$$

where, 0.000 025 : coefficient of thermal expansion of specific gravity hydrometer (°C⁻¹)

6.3.2 Measuring method for density by oscillation-type density meter The measurement of density of prepared tar shall be as specified in **7.3 of JIS K 0061**.

6.4 Measuring method for density of tar pitch The measurement of density of tar pitch shall be as follows.

- a) **Appliances** The following appliances shall be used.

- 1) **Pycnometer** A pycnometer with thermometer (0 °C to 40 °C) (having a marked line and perfect ground-glass fit), as shown in figure 6.

Alternatively, a normal pycnometer with thin tube may be used.

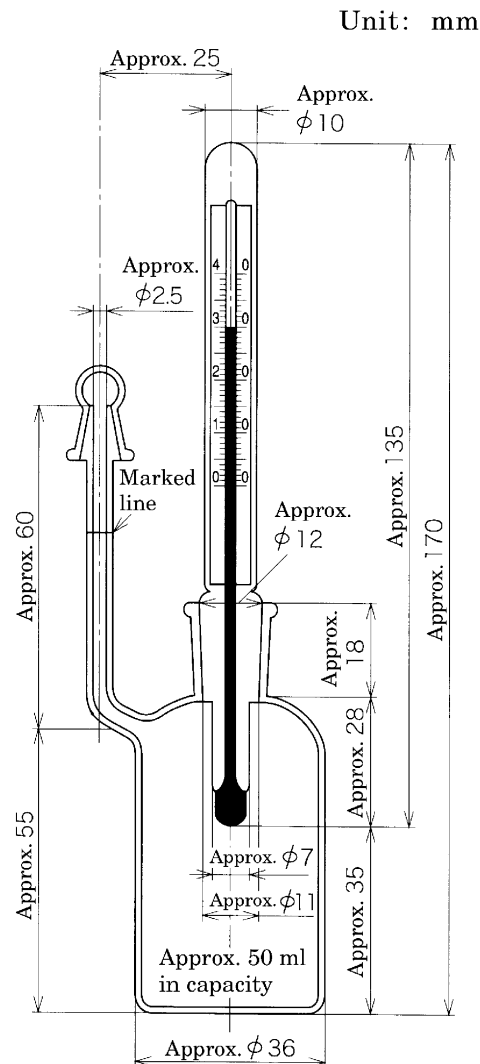


Figure 6 Pycnometer

- 2) **Syringes** Syringes of 20 ml and 2 ml in capacity, with syringe needle.
 - 3) **Thermostatic bath** As appropriate.
 - 4) **Thermometer** A double-wall mercury thermometer having a temperature range of 0 °C to 50 °C.
- b) **Reagent** 2,2,4-Trimethylpentane specified in **JIS K 9703** shall be used.
- c) **Procedure** Put about 10 g of specimen (840 μm or smaller in particle size) in a pycnometer of known mass (M_2), and measure the mass of the pycnometer (M_3), and then introduce 2,2,4-trimethylpentane into the pycnometer to fill about half of its inner volume.

Shake the pycnometer well to make the specimen and 2,2,4-trimethylpentane in the pycnometer to have affinity for each other, and after expelling bubbles

thoroughly from the mixture, leave it standing still. After the specimen in the pycnometer has completely settled, add 2,2,4-trimethylpentane to the vicinity of the marked line of the pycnometer by using a syringe, and put the pycnometer in a thermostatic bath maintained at 30.5 °C.

When the temperature in the pycnometer has reached 30.0 °C, add 2,2,4-trimethylpentane by using a syringe to make its liquid level agree with the marked line of the pycnometer. Then take the pycnometer out of the thermostatic bath, wipe off all the water adhering to the outside of the pycnometer, and measure its mass (M_4) after it reaches the normal temperature. Then carry out the same procedure as above by using 2,2,4-trimethylpentane alone, and measure the mass of the pycnometer (M_1), and calculate the density (30 °C) of the specimen by the following formula:

$$D_{30} = \frac{M_3 - M_2}{(M_1 - M_2) - (M_4 - M_3)} \times 0.683 \ 2$$

where, D_{30} : specific gravity (30 °C) (g/cm³)
 M_1 : mass of (pycnometer + 2,2,4-trimethylpentane) (g)
 M_2 : mass of pycnometer (g)
 M_3 : mass of (pycnometer + specimen) (g)
 M_4 : mass of (pycnometer + specimen + 2,2,4-trimethylpentane) (g)
 0.683 2 : density of 2,2,4-trimethylpentane at 30 °C (g/cm³)

7 Distillation test method

7.1 Distillation test method for creosote oil The distillation test method for creosote oil shall be as follows.

a) **Appliances** The following appliances shall be used.

- 1) **Distilling flask** A distilling flask of 250 ml capacity, around whose neck thermal insulation materials may be wound. Figure 7 shows an example of flask.
- 2) **Thermometer** A bar mercury thermometer with 1 °C graduations in the range of 100 °C to 360 °C and scaled with figures at every 10 °C; having dimensions given in table 3.

Table 3 Thermometer

Unit: mm	
Item	Dimension
Outer diameter of stem	5 to 7
Length of mercury bulb	10 to 20
Distance from lower end of mercury bulb to 360 °C graduation mark	280 to 350

- 3) **Cooler** As shown in figure 2.
- 4) **Receptacle (A)** A measuring cylinder of 100 ml capacity and about 260 mm total length, having 0.2 ml graduations in the range of 0 ml to 7 ml and 1 ml graduations in the range of 7 ml to 100 ml, and having 1 ml scale figures in the range of 0 ml to 10 ml and 10 ml scale figures in the range of 10 ml to 100 ml; having a shape and dimensions shown in figure 8.

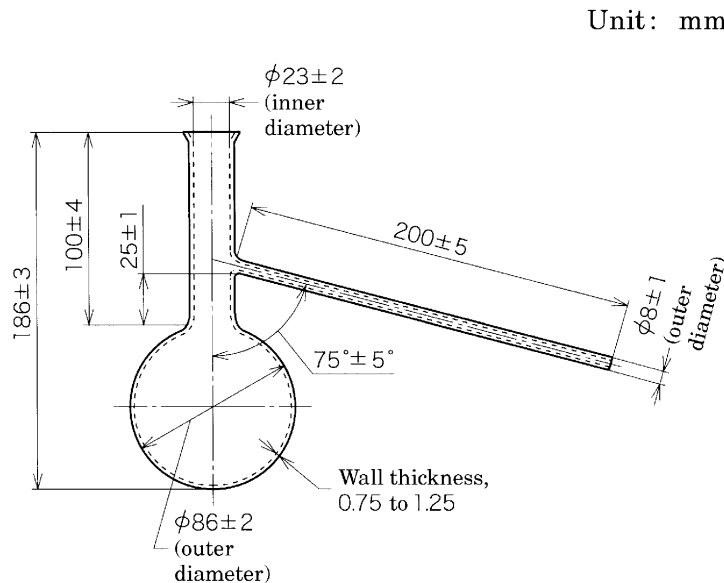


Figure 7 Example of distilling flask

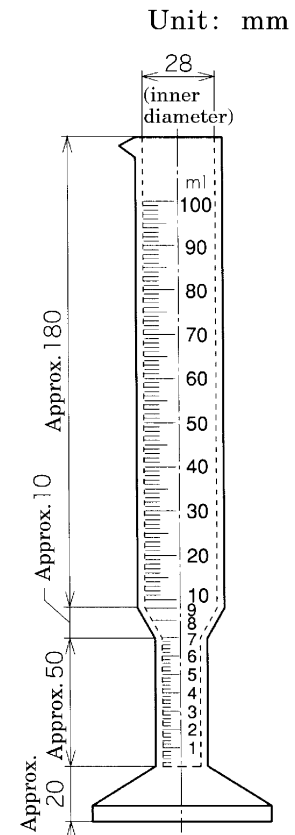


Figure 8 Receptacle (A)

- 5) **Receptacle (B)** A measuring cylinder of about 28 mm in inner diameter and about 220 mm in total length, with a capacity of 100 ml, having 1 ml graduations.
- 6) **Wind shield and cover** As shown in figure 9.

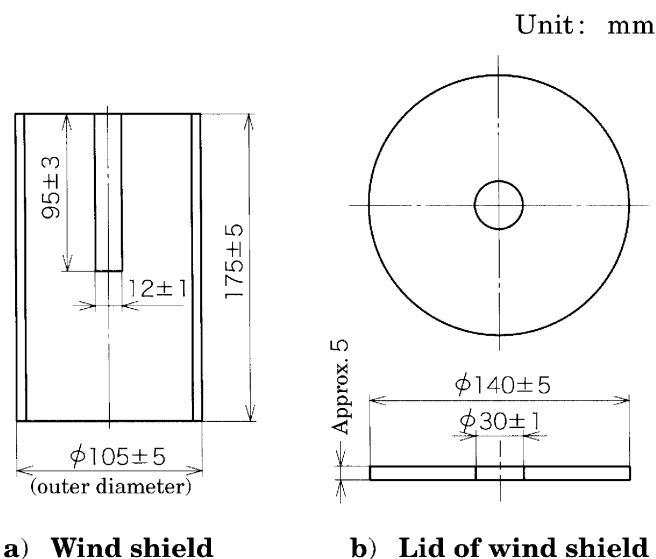


Figure 9 Example of wind shield and cover

- b) **Procedure** Measure 100 ml of dehydrated specimen⁽³⁾ into a dried clean distilling flask, and fix a thermometer to it with a cork plug. After fixing the thermometer in such a manner that the upper end of the mercury bulb positions at the lower root of the branch tube of the distilling flask, mount the distilling flask on a suitable heating apparatus. Connect the branch tube to the cooler by inserting it by about 35 mm into the cooler, place the receptacle (A) at the end of the cooler, and assemble the whole apparatus as shown in figure 10.

Note ⁽³⁾ In the case where the specimen contains 0.5 % (volume fraction) or less water, it may be furnished to the test as it stands.

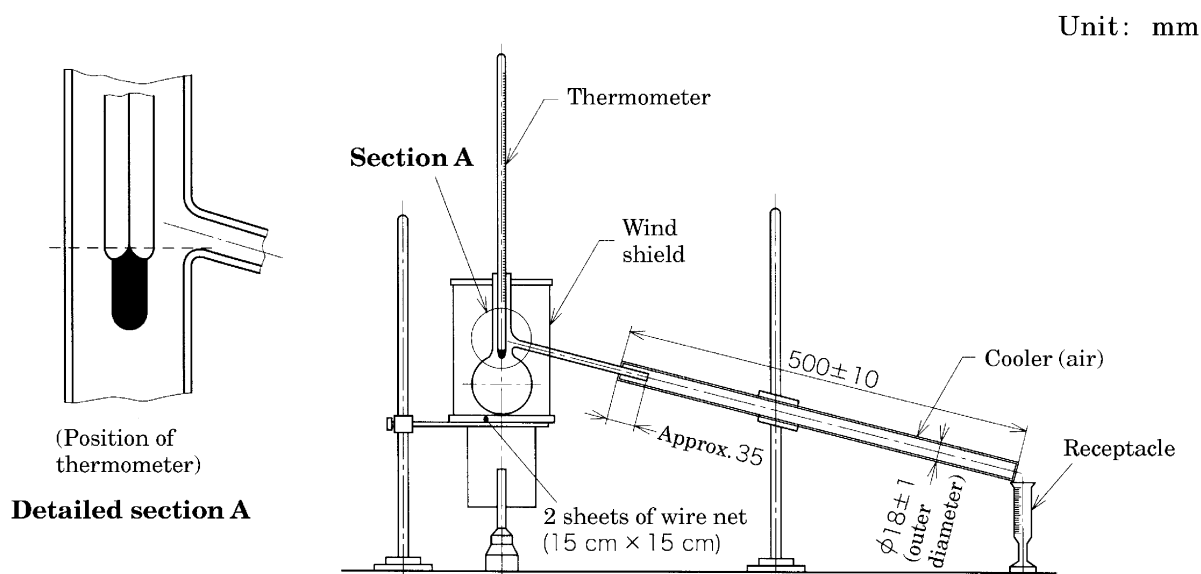


Figure 10 Example of distilling apparatus

After distillation has begun, adjust heating⁽⁴⁾ so that the distillate falls from the cooler to the receptacle at a rate of 60 drops per minute. During distillation, if crystals are formed in the cooler, let them flow down by suitable heating.