Doc. PCD 06 (27387) WC
January 2025

BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

तार और बिटुमिनस सामग्री के परीक्षण की पद्धतियाँ — आसवन परीक्षण

(IS 1213 *का तीसरा पुनरीक्षण*)

Draft Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS — DISTILLATION TEST

(*Third Revision* of IS 1213)

(ICS 75.140)

Bitumen, Tar and Related Products Sectional Committee, PCD 06	Last date for comment: **18 March 2025**

FOREWORD

(*Formal clauses to be added later*)

This standard was originally published in 1958 as 'Methods for testing tar and bituminous materials — Distillation test' and subsequently revised in 1978. 'Methods for testing tar and bituminous materials' was originally published as series of 22 standards in the form of a booklet, as listed below:

IS No.	*Title*
1201 : 2004	Sampling
1202 : 1978	Determination of penetration
1204 : 1978	Determination of residue of specified penetration
1205 : 1978	Determination of softening point
1206 (Part 1) : 1978	Determination of viscosity: Part 1 Industrial viscosity
1206 (Part 2) : 1978	Determination of viscosity: Part 2 Absolute viscosity
1206 (Part 3) : 1978	Determination of viscosity: Part 3 Kinematic viscosity
1207 : 1978	Determination of equiviscous temperature (EVT)
1208 : 1978	Determination of ductility
1209 : 1978	Determination of flash point and fire point

1210 : 1978	Float test
1211 : 1978	Determination of water content dean and stark method
1212 : 1978	Determination of loss on heating
1213 : 1978	Distillation test
1214 : 1978	Determination of matter insoluble in benzene (WITHDRAWN due to toxic nature of benzene)
1215 : 1978	Determination of matter insoluble in toluene
1216 : 1978	Determination of solubility in carbon disulphide trichloroethylene
1217 : 1978	Determination of mineral matter ash
1218 : 1978	Determination of phenols
1219 : 1978	Determination of naphthalene
1220 : 1978	Determination of volatile matter content

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian standards separately for each test so as to make it user friendly. Accordingly, second revision of the standard was taken up in 2020 to formulate an individual standard on distillation test and to replace mercury thermometers with suitable temperature measurement device, calibrated with appropriate accuracy, precision and sensitivity.

The third revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

a) Method for distillation of road tar and digboi type cutback bitumen have been deleted from the scope and procedures.
b) Method for distillation of bitumen emulsion has been incorporated in the scope and procedure.
c) Terminology of cutback bitumen, bitumen emulsion, creosote, and anthracene oil have been incorporated.
d) Fig. No. 5 illustrating apparatus for method of distillation of crude coal tar has been incorporated.
e) Reference clauses have been updated.

In reporting the results of a test analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:2022 ‘Rules for rounding off numerical values (*second revision*).’

1 SCOPE

This standard prescribes the methods for the distillation test for crude coal tar, cutback bitumen, bitumen emulsion, creosote and anthracene oil.

2 REFERENCES

The following standards contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
IS 334: 2023	Glossary of terms relating to bitumen and tar (*fourth revision*)
IS 1203: 2022	Methods for testing tar and bituminous materials — Determination of penetration (*second revision*)
IS 1205: 2022	Methods for testing tar and bituminous materials — Determination of softening point — Ring and ball apparatus (*second revision*)
IS 1211: 2022	Methods for testing tar and bituminous materials — Determination of water content — Dean and stark method (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the following definition and those given in IS 334 shall apply.

3.1 Distillation — The process by which the more volatile constituents are separated from the less volatile ones.

3.2 Cutback Bitumen — A type of bitumen that has been diluted with a volatile solvent to reduce its viscosity, facilitating application at lower temperatures.

3.3 Bitumen Emulsion — A mixture of bitumen and water, stabilized with an emulsifying agent, allowing the bitumen to be dispersed in fine droplets for mixing and laying at ambient temperatures.

3.3 Creosote — Creosote oil is obtained by distillation of the wood and contains a mixture of aromatic compounds, including phenols, hydrocarbons, and terpenes.

3.3 Anthracene oil — Anthracene oil is a complex combination of polycyclic aromatic hydrocarbons obtained from coal tar having an approximate distillation range of 300 °C to 400 °C.

4 SIGNIFICANCE AND USE

4.1 This test method is used for quantitative determination of residue, volatile constituent's/oil distillates in bitumen emulsion, coal tar, cutback bitumen, creosote and anthracene oil. This test method shall also be used to obtain residue and oil distillate for further testing as applicable in the product specification.

5 METHOD A (FOR CUTBACK BITUMEN AND BITUMEN EMULSION)

5.1 Apparatus

5.1.1 *Distillation Flask*

500 ml, side arm having dimensions as shown in Fig. 1.

5.1.1.1 The distance from the center of the side tube at the junction of the neck to the top of the neck should be (12 ± 1) mm.

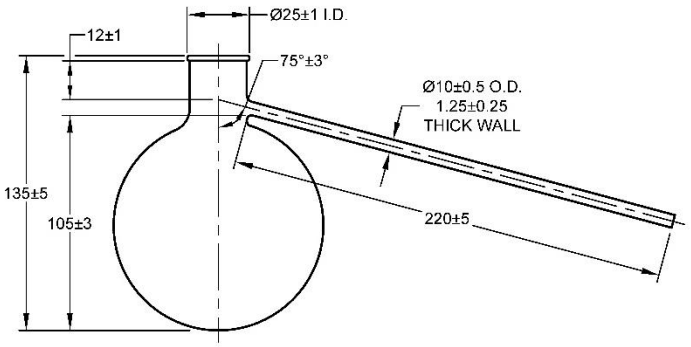


FIG. 1 DISTILLATION FLASK

5.1.1.2 The side tube shall slope downwards from the junction with neck so that the acute angle between the side tube and the neck is (75 ± 3) °.

5.1.2 *Thermometer*

Temperature measuring device as specified in either **5.1.2.1** or **5.1.2.2**

5.1.2.1 *Liquid–in–glass thermometers*

Of high distillation, total immersion type, graduated in centigrade degrees as specified, having a range of 2 °C to 400 °C and conforming to the following requirements:

Liquid	Any low hazard precision liquid
Filling above liquid	Nitrogen gas
Temperature range	2 °C to 400 °C
Subdivision	1 °C
Total length	378 mm to 384 mm
Stem diameter	6.0 mm to 7.0 mm
Bulb diameter	Not larger than stem diameter
Bulb length	10 mm to 15 mm
Distance of bottom of bulb to graduation line at 0 °C	25 mm to 45 mm

Top finish	Glass ring
Longer graduation lines at each	5 °C
Graduations numbered at each multiple	10 °C
Immersion	Total
Scale error at any point up to 370 °C shall not exceed	1 °C

5.1.2.2 *Digital electronic thermometer/ device*

Confirming to the following requirements:

Temperature range	2 °C to 400 °C
Accuracy	1 °C
Thermal response time	(4 ± 2) s
Immersion depth	> 375 mm
Display resolution	0.5 °C

5.1.3 *Condenser (Water)*

A 250 mm glass-jacketed condenser (*see* Fig. 2) with the dimension's land tolerances given below:

Length of jacket excluding the necks	(250 ± 5) mm
Outside diameter of adapter of condenser tube	(23 ± 1) mm
Length of adapter	(75 ± 5) mm
Outside diameter of condenser tube proper	(12.5 ± 0.5) mm
Overall length of condenser tube including adapter	(475 ± 25) mm

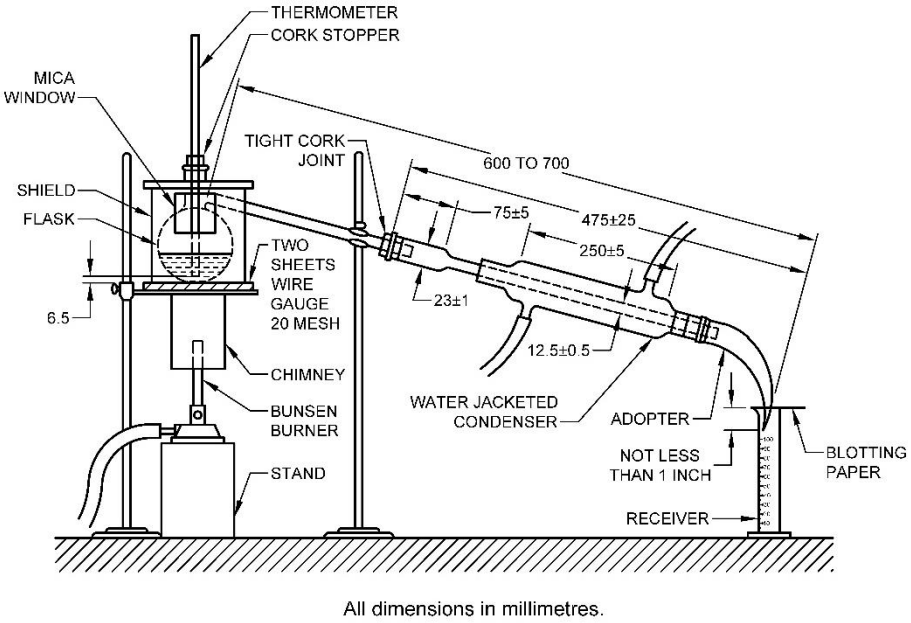


FIG. 2 DISTILLATION APPARATUS ASSEMBLY

5.1.4 *Adapter*

Curved design (*see* Fig. 2) having a heavy wall and reinforced top glass, with an angle of approximately 105 ° and with a diameter at the large end of approximately 18 mm. The outlet end shall be ground to an angle of (45 ± 5) °C with inside vertical. The small end shall have a diameter of not less than 5 mm.

5.1.5 *Shield*

Galvanized iron, lined with 3 mm asbestos, fitted with transparent covered windows, of the form and dimensions shown in Fig. 3, used to protect the flask from air currents and to prevent radiation. The cover (top) may be of transit board made in two parts, or it may be of galvanized iron lined with 3 mm asbestos.

5.1.6 *Crow Receivers*

25 ml, 50 ml or 100 ml size and of dimensions and tolerances shown in Fig. 4 may be used.

5.1.7 *Residue Container*

The container for the distillation residue shall be a 250 g metal container approximately 76 mm in diameter and 54 mm deep, provided with a lid.

5.2 Procedure

5.2.1 *Preparation of the Sample*

Distil a known volume of the thoroughly mixed sample from the distillation flask with the condenser, until water ceases to come over. Separate the water from the oil and return the oil to the distillation residue when the residue has cooled to 40 °C. This procedure shall be adopted only if the water content of the material, as determined according to IS 1211 is in excess of 0.5 percent.

NOTE — Clause **5.2.1** is not applicable for bitumen emulsion.

5.2.1.1 Thoroughly stir and agitate the sample if necessary to ensure a complete mixture.

5.2.2 *Assembly of Apparatus*

5.2.2.1 Support the flask on a tripod or ring over two sheets of IS 20 mesh wire gauze, 150 mm square (*see* Fig. 2). Connect to the condenser tube by a light cork joint. Insert the thermometer through a cork in the neck of the flask with the bottom of the bulb 6 mm from the bottom of the flask, the axis of the bulb of the flask through the center of the neck being vertical and the thermometer aligned on this axis.

5.2.2.2 The distance from the neck of the flask to the outlet end of the adapter shall be not more than 700 mm and not less than 600 mm. The burner/ heating mantle should be protected from draughts by a suitable shield or chimney (*see* Fig. 3).

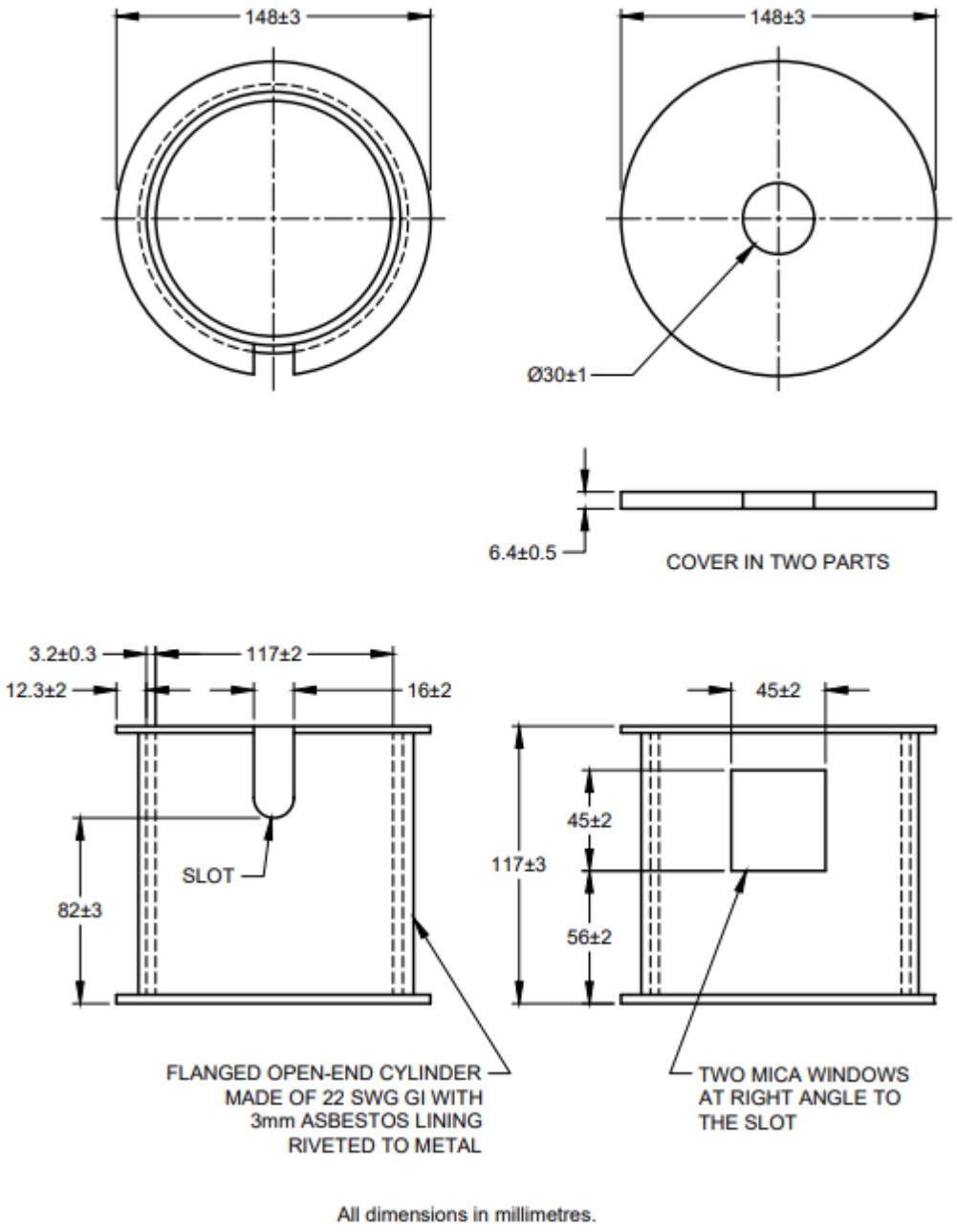

148±3
148±3
Ø30±1
6.4±0.5
COVER IN TWO PARTS
3.2±0.3
117±2
12.3±2
16±2
SLOT
82±3
45±2
117±3
45±2
56±2
FLANGED OPEN-END CYLINDER MADE OF 22 SWG GI WITH 3mm ASBESTOS LINING RIVETED TO METAL
TWO MICA WINDOWS AT RIGHT ANGLE TO THE SLOT
All dimensions in millimetres.

FIG. 3 SHIELD

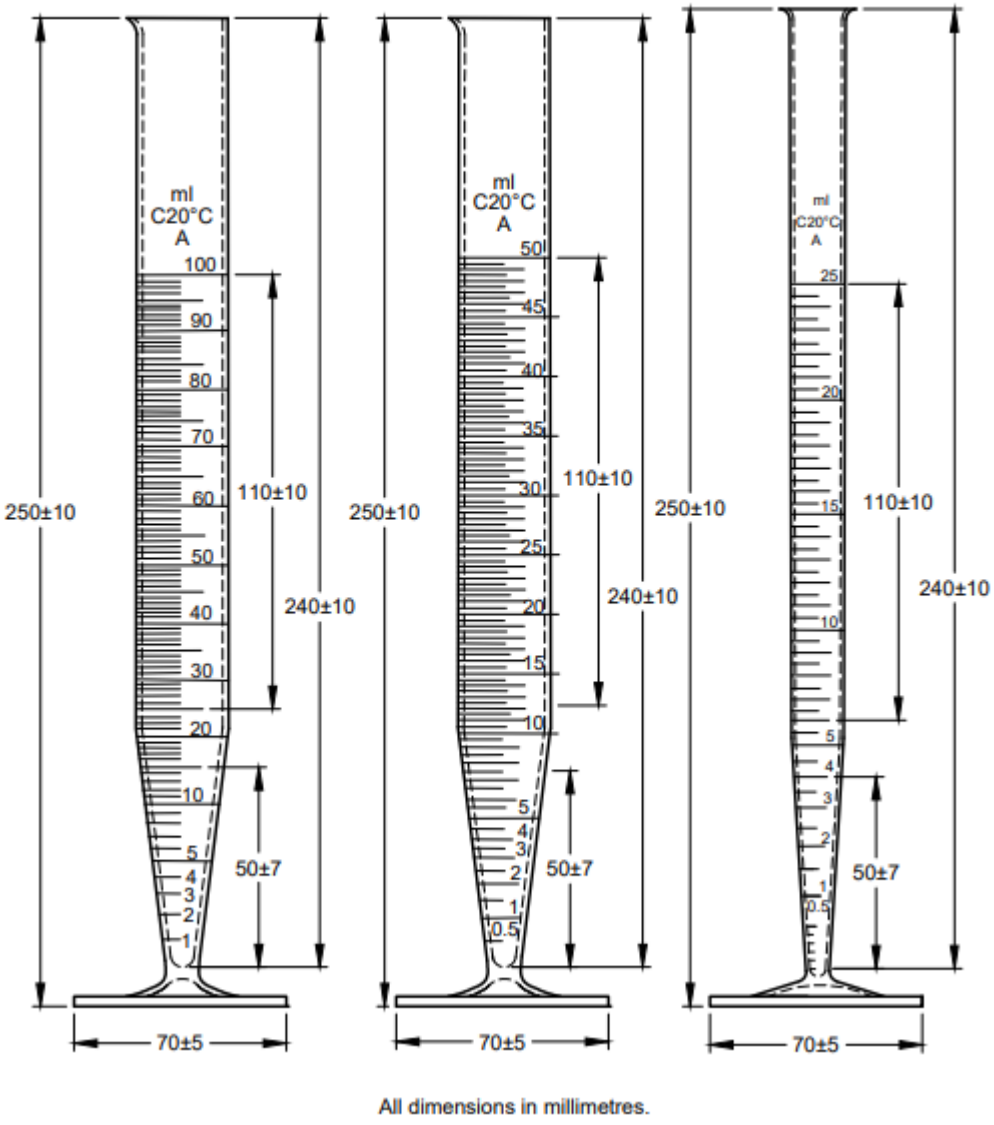


FIG. 4 CROW RECEIVERS 100 ML, 50 ML AND 25 ML

5.2.2.3 Adjust the adapter over the end of the condenser tube so as to conduct the distillate into the receiver. During the distillation, cover closely the top of the receiver with a piece of blotting paper or its equivalent, which shall be cut so as to fit the adapter tightly. The adapter shall extend into the receiver at least 25 mm but not below the 100 ml mark.

5.2.3 *Distillation*

5.2.3.1 Measure 200 ml of the material into the flask, assemble the apparatus and heat so that the first drop comes over in 5 min to 15 min. Adjust the rate of distillation between 50 drops to 70 drops per

min except that near the end of the distillation the heat input shall not be so rapid as to result in a temperature in excess of 360 °C.

5.2.3.2 Should the sample foam, the distillation rate will have to be reduced, but the normal rate shall be resumed as soon as possible. If excess foaming persists, the distillation may be more easily controlled by reducing the rate of heating.

5.2.3.3 Collect the distillate in the crow receivers and record the volume of distillate at all specified temperatures. Record also the volume of any separate water. When the maximum specified temperature of the test is indicated by the thermometer, discontinue the heating and drain into the receiver any oil which may remain in the condenser tube. Measure by volume the residue and keep it for further tests.

5.2.4 *Procedure for Handling Residue*

5.2.4.1 When the temperature reaches 360 °C, extinguish the flame and immediately (within 36 s) pour the residue into the residue container, placing this on its lid to prevent too rapid cooling at the bottom.

5.2.4.2 Allow the residue to cool in a position free from draughts to a temperature that is below its fuming point and at the same time suitable for pouring.

5.2.4.3 Stir the residue and pour into the receptacles specified for testing for properties, such as, penetration and softening point.

5.3 Report

5.3.1 *Asphaltic Residue*

Calculate the percent residue to the nearest 0.1 as follows:

$$R = \left[\frac{200 - TD}{200}\right] \times 100$$

where,

R = residue content, in volume percent; and
TD = total distillate recovered to 360 °C, in ml.

5.3.1.1 Report as the residue from distillation to 360 °C, percent volume by difference.

5.3.2 *Total Distillate*

Calculate the percent total distillate to the nearest 0.1 as follows:

$$\text{TD percent} = \left[\frac{TD}{200}\right] \times 100$$

5.3.2.1 Report as the total distillate to 360 °C, volume percent.

5.3.3 *Distillate Fractions*

5.3.3.1 Determine the percentage by volume of the original sample by dividing the observed volume (in milliliters) of the fraction by 2. Report to the nearest 0.1 as volume percent as follows:

Up to 190 °C
Up to 225 °C
Up to 260 °C
Up to 316 °C

5.3.3.2 Determine the percentages by volume of total distillate by dividing the observed volume in milliliters of the fraction by the milliliters recovered to 360 °C and multiply by 100. Report to the nearest 0.1 as the distillate, volume percent of total distillate to 360 °C as follows:

Up to 190 °C
Up to 225 °C
Up to 260 °C
Up to 316 °C

5.3.4 Where penetration, viscosity, or other tests have been carried out, report with reference to this method as well as to any other method used. (Example 'Penetration according to IS 1203 of residue as in IS 1213').

5.3.5 If the first fraction obtained contains water, note the volume of water. Deduct this from the volume of oil taken and correct all the fractions to a percentage based on the volume of the water free sample.

5.3.6 A convenient method for determining the volume of water is to transfer this fraction, after noting the volume, to a tube or cylinder graduated in 0.1 ml and to add about 15 ml to 20 ml of benzol. This almost always causes a clear separation between the oil and water.

5.3.7 Barometric pressure correction as stipulated as under section 9 shall be applied where necessary.

5.4 Precision

Duplicate results shall not differ by more than the following:

Repeatability	*Reproducibility*
2 ml distillate	3 ml distillate

5.5 Precaution

During the distillation process, the thermometer should remain in its original position and submerged in the solution continuously.

6 METHOD B (FOR CRUDE COAL TAR)

6.1 Apparatus

6.1.1 *Distillation Flask*

A flask with dimensions and tolerances (*see* Fig. 5) as given below shall be used:

Distillation capacity, ml	750
Capacity of bulb, ml	975 ± 20
Internal diameter of neck between side taper and bulb, mm	28 ± 1
Internal diameter of side tube, mm	8.5 ± 0.5
External diameter of side tube, mm	11 ± 0.5
Length of side tube, mm	160 ± 5
Radius of curvature at the base of the neck, mm	12
Thickness of walls of bulb neck of side tube, mm	1 to 1.5

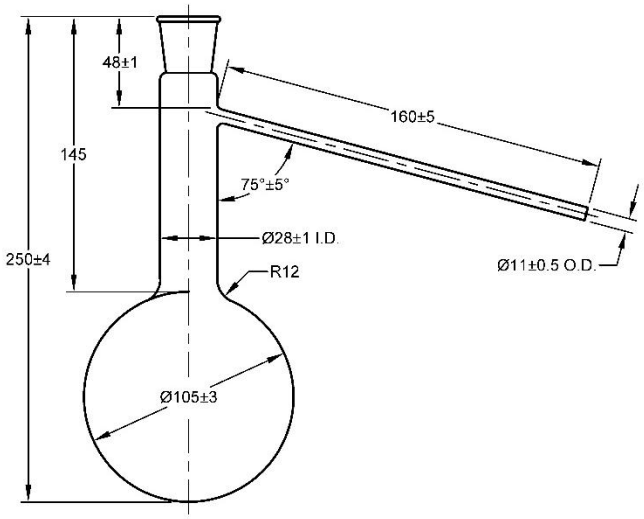


FIG.5 DISTILLATION FLASK

6.1.1.1 The distance from the centre of the side tube of each flask at the junction of the neck to the top of the neck shall be (75 ± 3) mm. The distance from the centre of the side tube of each flask at the junction with the neck to the liquid surface, when the flask is vertical and contains a quantity of liquid equal in volume to the distillation capacity of the flask, shall be (90 ± 3) mm.

6.1.2 *Thermometer* — Same as specified in Method A (*see* **5.1.2**).

6.1.3 *Air Condenser*

Made from a straight tube of good quality resistance glass, with one end finished square with the axis and the other end ground at an angle of 45° with the axis and conforming to the following dimensions:

Internal diameter	(20 ± 1) mm
Overall length	(600 ± 10) mm
Wall thickness	(1.0 to 1.5) mm

6.1.3.1 The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

6.1.4 *Metal Heating Bath* (*See* Fig. 6)

The bath shall contain fusible alloy melting below 70 °C in such quantity that when the bottom of the flask is 5 mm from the bottom of the bath, the level of the molten metal is about 15 mm below the rim of the bath.

6.1.5 *Crow Receivers* — Same as specified in Method A (*see* **5.1.6**).

6.2 Procedure

6.2.1 *Preparation of Sample* — Same as specified in Method A (*see* **5.2.1**).

6.2.2 *Assembly of Apparatus* — Set up the distillation flask as in Method A (*see* **5.2.2**).

6.2.2.1 Smoke the bottom of the flask over a luminous flame. Heat the fusible metal to a temperature slightly higher than its melting point and immerse the flask centrally in the bath, the bottom of the flask being at least 5 mm above the bottom of the bath.

6.2.3 *Distillation*

Take 250 ml of the material and carry out the distillation steadily at the rate of 5 ml /min and, without interrupting the distillation, collect oil fractions separately in the graduated receiver as follows:

a) Up to 210 °C

b) 210 °C to 230 °C
c) 230 °C to 270 °C
d) 270 °C to 300 °C
e) 300 °C to the stage of pitch

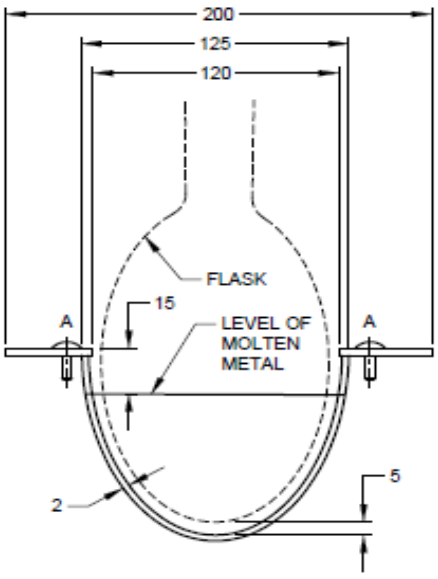
6.2.3.1 Should solids tend to deposit during the distillation, warm the condenser so that such solids are collected in the fraction with which they come over.

6.3 Report

6.3.1 Record the final temperature of distillation reached in the test.

6.3.2 Note and report the volume and appearance of each fraction. Determine and report the specific gravity of each fraction at 27 °C. Nucleate fractions (a) to (d) with a small crystal of naphthalene and fraction (e) with a small crystal of anthracene and cool the fractions to (15.5 ± 0.5) °C. Keep at this temperature, with occasional stirring, till solids separate out or for 4 h, whichever is less, and report the result of this observation and the percentage of oil draining out of each fraction in which solids make an appearance.

6.3.3 Barometric pressure correction as stipulated as under section 9 shall be applied where necessary.



All dimensions in millimetres.

A - A : THREE OR FOUR PINS OR SIMILAR DEVICE TO RETAIN FLANGE CENTRAL, IF FLANGE AND BOWL ARE NOT IN ONE PIECE.

FIG. 6 METAL HEATING BATH

7 METHOD C (FOR CREOSOTE AND ANTHRACENE OIL)

7.1 Apparatus

7.1.1 *Distillation Flask*

A flask with dimensions and tolerances as given below shall be used:

Distillation capacity	150 ml
Capacity of bulb	(195 ± 7) ml
Internal diameter of neck between side, taper and bulb	(18 ± 1) mm
Internal diameter of side tube	(5.0 ± 0.5) mm
External diameter of side tube	(7.0 ± 0.5) mm
Length of side tube	(120 ± 4) mm
Radius of curvature at the base of neck	10 mm
Thickness of walls of bulb, neck, side tube	0.8 mm to 1.2 mm

7.1.2 *Thermometer* — Same as specified in Method A (*see* **5.1.2**).

7.1.3 *Air Condenser*

Made from a straight tube of good quality resistance glass, with one end finished square with the axis and the other end ground at an angle of 45° with the axis and conforming to the following dimensions:

Internal diameter	(20 ± 1) mm
Overall length	(600 ± 10) mm
Wall thickness	1.0 mm to 1.5 mm

7.1.3.1 The side arm of the flask shall extend at least 25 mm beyond the cork in the upper end of the condenser.

7.1.4 *Draught Screen*

Rectangular, made of 0.711 mm standard wire gauge sheet metal, with the dimensions shown in Fig. 5 and open at top and bottom and complying with the following requirements:

a) In each of the two narrow sides of the draught screen there shall be two circular holes, 25 mm in diameter each, and situated as illustrated in Fig. 6. In each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the positions as shown in Fig. 6. The diameter of each of the holes centrally situated in the longer sides shall be 25 mm and of the remaining ten holes 12 mm. At the middle

of each of the wider sides a vertical slot with the dimensions as shown in Fig. 6 shall be cut downwards from the top of the screen.

b) A sheet of hard asbestos of silica board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides of neck of the flask. The flask shall be placed in position and pressed down so as to close completely the hole in the board. The supports for this board may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

c) In one of the narrow side of the screen a door shall be provided, having the dimensions and position as shown in Fig. 6. In each of the narrow sides of the screen a mica window shall be placed centrally, with bottom of the window level with the top of the asbestos shelf. The dimensions and position of the windows shall be as shown in Fig. 7.

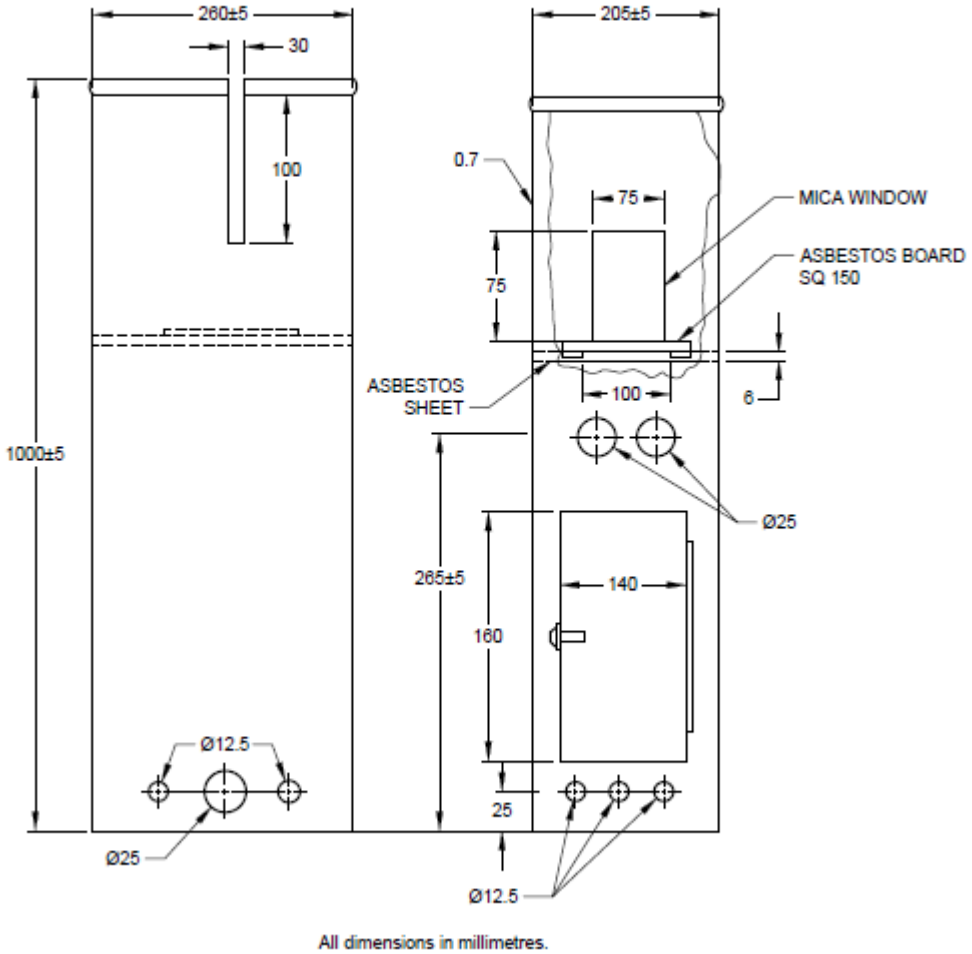


FIG. 7 DRAUGHT SCREEN

7.1.5 *Crow Receivers* — Same as specified in Method A (*see* **5.1.6**).

7.2 Procedure

7.2.1 Preparation of Sample Same as specified in Method A (*see* **5.2.1**).

7.2.2 *Assembly of Apparatus*

Set up the distillation flask complete with thermometer, air condenser and receiver. The thermometer shall be so fitted in the flask that the bottom of the capillary is level with the lower edge of the side tube joint and the immersion mark is level with the top of the cork.

7.2.3 *Distillation*

7.2.3.1 Thoroughly mix the sample as prepared under **7.2.1** in a 250 ml flask. Weigh (100 ± 0.5) g of the mixture directly into the weighed distillation flask and start the distillation, using the naked flame of the burner. Continue the distillation at the rate of (5.0 ± 0.5) ml per min. If for any reason the distillation rate falls outside the specified limits at any time after the first 5 ml of distillate have collected and before the final specified temperature is reached, discard the test and start another test on a further portion of the material.

7.2.3.2 The specified distillation rate corresponds approximately to 90 drops per min or three drops in each 2 s, but this should only be taken as a guide. Graduated receivers should preferably be used for the collection of the distillate in order that the rate in milliliters per minute may be kept under close observation.

7.2.3.3 Change the receiver at each specified temperature (*see* **7.3.1**) without stopping the distillation. Extinguish the flame when the thermometer reaches the highest specified temperature, and include in the final fraction the oil which drains from the condenser within 5 min after the flame has been extinguished. Should solids tend to deposit during the distillation, warm the condenser so that such solids are collected in the fraction with which they come over.

7.2.3.4 During the progress of distillation, the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

7.3 Report

7.3.1 Report the cumulative weights of fractions obtained at the following ranges of temperatures:

0 °C to 210 °C
0 °C to 235 °C
0 °C to 315 °C
0 °C to 355 °C

7.3.2 Also, report the weight of the residue.

7.3.3 If the first fraction contains water, determine the amount of water. Deduct this amount from the weight of oil taken for the test, and report all the fractions as percentage by weight based on the weight of the water-free material.

7.3.4 A convenient method for determining the amount of water is to transfer this fraction, after weighing, to a tube of cylinder graduated in 0.1 ml and to add 13 ml to 20 ml of benzol. This almost always causes a clear separation between the oil and water, and assuming a density of 1.00 g/ml, the weight of water can be calculated.

7.3.5 Barometric pressure correction as stipulated as under section **9** shall be applied where necessary.

8 PRECISION

The duplicate results shall not differ by more than the following:

	Repeatability	*Reproducibility*
Up to 230 °C	1.6 g	3.5 g
Above 230 °C to 315 °C	1.5 g	3.4 g
Total distillate	1.7 g	1.7 g

9 BAROMETRIC PRESSURE CORRECTION

If the barometric pressure during the period of test remains within the range of (760 ± 5) mm, no correction need be applied to the specified distillation temperatures. If the pressure is beyond that range, apply the following correction to the specified temperatures:

Correction for temperature in degrees centigrade = 0.000 12 (760 – P) (t + 273)

where,

P = corrected barometric pressure during test, and

t = specified temperature in degrees centigrade.