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An American National Standard

Standard Test Method for Distillation of Pitch[1]

This standard is issued under the fixed designation D 2569; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Replaced the word "asbestos" in 8.1.1 editorially in August 2005.

1. Scope

1.1 This test method covers a distillation test for pitch applicable whenever the amount of distillate below 270°C is less than 2.0 % by this test method.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 140 Practice for Sampling Bituminous Materials[2]

E 1 Specification for ASTM Thermometers[3]

3. Summary of Test Method

3.1 A 100-g sample is distilled from an electrically heated flask in a shield at a designated rate. The weight of distillate fractions at specified temperatures and the time required to collect the main fraction are recorded.

4. Significance and Use

4.1 In usual practice, most of the oils distilling below 360°C are removed in the manufacture of high-softening-point pitches. Excessive distillation in this range may indicate an abnormal product which could cause some problems of fuming and instability in use.

5. Apparatus

5.1 *Flask*—A side-arm distillation flask, as shown in Fig. 1, having the following dimensions:

Diameter of bulb, outside, mm	86.0 ± 1.5
Diameter of neck, inside, mm	22.0 ± 1.0
Diameter of tubulature, inside, mm	10.0± 0.5
Height of flask, outside, mm	131.0 ± 1.5
Vertical distance, bottom of bulb, outside, to horizontal tangent at tubulature, inside, mm	93.0 ± 1.5
Length of tubulature, mm	220 ± 5
Angle of tubulature, °	1.0 to 1.5
Thickness of tubulature wall, mm	75 ± 2

5.2 *Condenser Tube*— A tapered glass condenser, as shown in Fig. 2, having the following dimensions:

Outside diameter of small end, mm	12.5 ± 1.5
Outside diameter of large end, mm	28.5 ± 3.0
Length, mm	360 ± 4
Length of tapered part, mm	100 ± 5

5.3 *Source of Heat*—Electric heater,[4] 750-W, with variable transformer control as shown in Fig. 2, with a direct-reading voltmeter (120 V) or ammeter (5 A). Transformer settings shall be reproducible within 2 % on the percent input dial. The heater shall be fitted with an upper refractory top, with the dimensions shown in Fig. 3, and a 110-mm square plate of 6-mm thick non-asbestos containing cement board, having an 80-mm center hole as shown in Fig. 4.

5.4 *Shield*—A stainless steel shield with mica windows[5] lined with 3-mm thick high temperature cloth, provided with a two-piece cover of 6-mm thick transite board (non-asbestos) of the form and dimensions shown in Fig. 4.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05.OF on Industrial Pitches.

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[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] The Precision "Ful-Kontrol" heater meets these requirements; it can be purchased through supply houses.

[5] The sole source of supply of the mica windows known to the committee at this time is Humboldt Mfg Co., Inc., 7302 W. Agatite Ave., Chicago, IL 60656. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

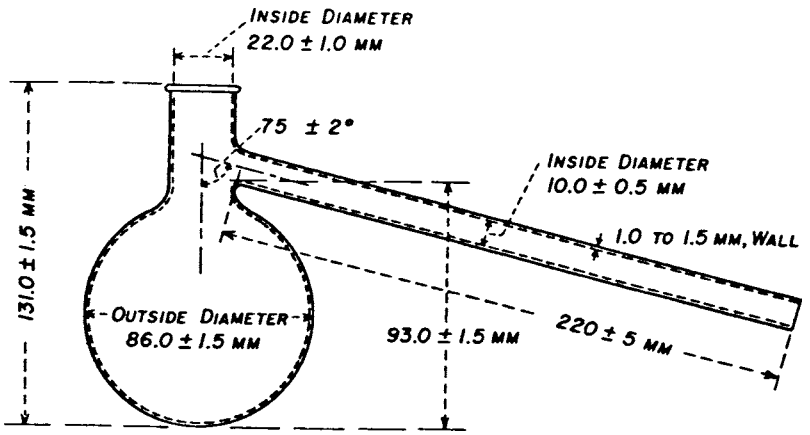

FIG. 1 Distillation Flask

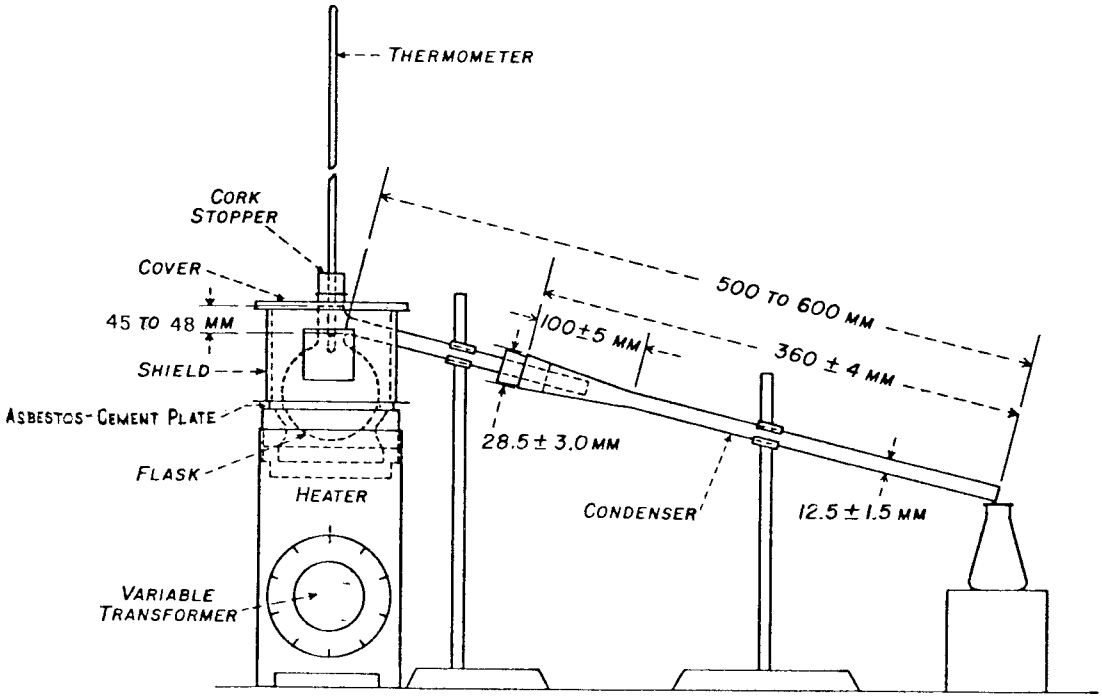

FIG. 2 Apparatus Assembly for Pitch Distillation

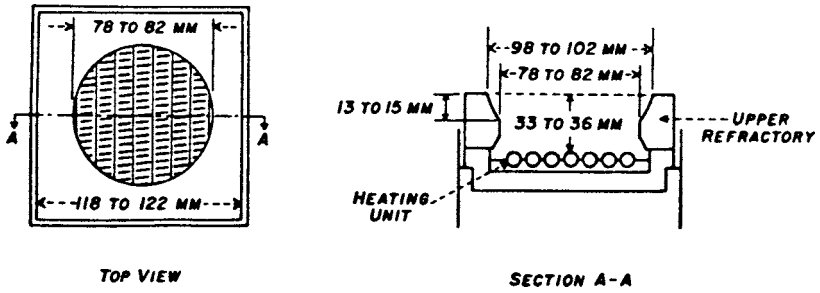

FIG. 3 Upper Part of Electric Heater

5.5 *Receivers*—Erlenmeyer flasks or beakers, having a nominal capacity of 50 to 125 mL and tared to the nearest 0.05 g.

5.6 *Thermometer*—An ASTM High Distillation Thermometer, having a range from – 2 to 400°C and conforming to the requirements for Thermometer 8C as prescribed in Specification E 1 or a suitable digital thermometer with the same precision as 8C.

5.7 *Timer*—Stop clock or stop watch, calibrated in seconds or tenths.

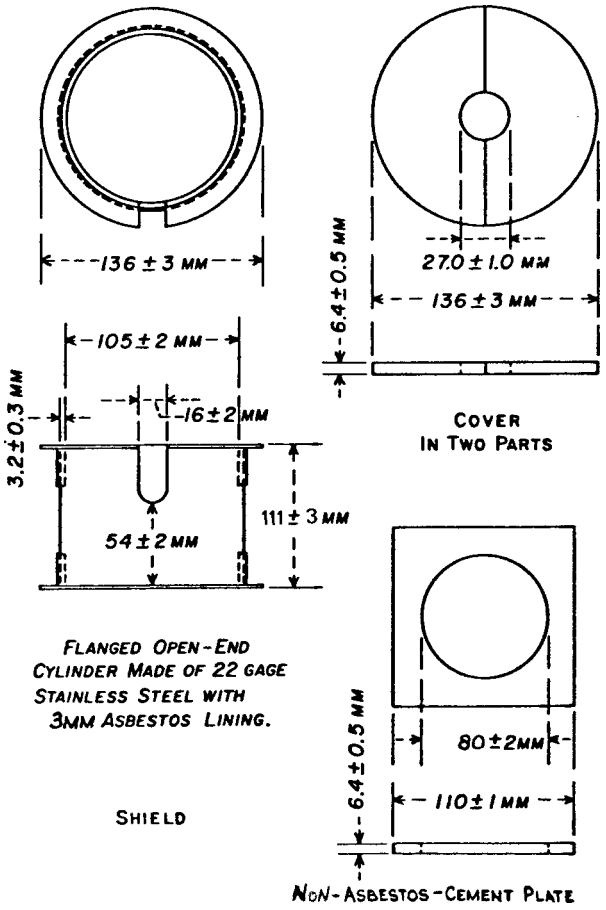

FIG. 4 Shield, Cover, and Non-Asbestos-Cement Plate

6. Sampling

6.1 Samples from bulk shipments shall be taken in accordance with Practice D 140, and shall be free of foreign substances. Thoroughly mix the sample before withdrawing a representative portion for the determination or for dehydration.

6.2 *Hard Pitch*—If the solid sample contains free water, air-dry a representative portion.

6.3 *Soft Pitch*—If the presence of water is indicated by surface foam on heating, maintain a representative portion at a temperature between 125 and 150°C in an open container until foaming ceases. Do not overheat, and immediately remove from the heat source when the foam subsides.

7. Sample Preparation

7.1 *Hard Pitch*—If the pitch can be crushed at room temperature, prepare a working sample of approximately 150 g by crushing, mixing, and quartering a representative portion of the dry sample. Crushing may be done with a small jaw crusher and a mullite mortar and pestle. No particle of the crushed sample shall be larger than 10 mm in any dimension.

7.2 *Soft Pitch*—Heat a portion of the dry sample to the lowest temperature at which it can be mixed and poured.

7.3 *Preservation of Samples*—Store samples as large lumps or solidified melts in closed containers. Do not save crushed samples for future testing since changes in composition may occur in pulverized pitch.

8. Preparation of Apparatus

8.1 Assemble the apparatus shown in Fig. 2 as follows:

8.1.1 Place the refractory on the heater with the larger opening facing upwards. Center the heat–resistant refractory board plate with the hole on the refractory and place the distillation shield on the plate.

8.1.2 Insert the thermometer through a new, rolled, tightly fitting cork in the neck of the flask, with the bottom of the cork 24 to 27 mm above the lowest point of the juncture between the side-arm tubulature and the neck of the flask. The top of the thermometer bulb must be level with this point. Align the stem of the thermometer on the axis of the bulb of the flask.

8.1.3 Place the flask within the shield so that it is seated in the hole in the non-asbestos containing cement board, and connect the condenser tube to the side arm of the flask with a tight cork so that the side arm projects 30 to 50 mm beyond the cork. The distance from the neck of the flask to the outlet of the condenser tube shall be not more than 600 nor less than 500 mm. Support the flask and condenser tube so that the latter is aligned with the side arm and the thermometer is vertical. Place the cover on top of the shield around the neck of the flask.

9. Procedure

9.1 Weigh 100 ± 1 g of the sample into a tared distillation flask. Assemble the apparatus as described in Section 8.

9.2 Set the control on the electric heater so that the sample of pitch completely melts, but does not boil, in not less than 10 min nor more than 30 min. Reset the control to provide a voltage or amperage reading which, in past experience on a similar type of pitch, has given a distillation rate of between 0.03 and 0.05 g/s for the total distillate between 300 and 360°C. Do not change this setting during the course of the distillation.

9.3 When the distillate begins to condense in the side arm, start heating the condenser with a small gas flame to lower the viscosity of the first distillate and to prevent the deposition of solids. Continue heating as required throughout the distillation, but keep the temperature of the condenser low enough to ensure complete condensation of the vapors.

9.4 Collect the fractions in tared receivers, changing them as the thermometer indicates the maximum temperature for each. Unless otherwise specified collect the following fractions:

to 270°C
270 to 300°C
300 to 360°C

9.5 Do not change the position of the thermometer during the distillation. Make no correction for the emergent stem of the thermometer, but if the barometric pressure at the time of the distillation lies outside the range of 756 to 765 torr (101 to 102 kPa), change receivers at the adjusted temperatures shown in Table 1.

9.6 If the first drop of distillate falls from condenser below 300°C, start the timer as the receiver for the 300°C fraction is put into place. When the maximum temperature for the test (see Note 1), corrected for barometric pressure if required, is reached, replace the receiver by another flask, stop the timer, shut off the heater, and remove the cover from the shield as

TABLE 1 Adjustment of Fractionation Temperatures for Varying Barometric Pressures

Barometric Pressure, torr (kPa)	Distillation Temperature, °C		
786 to 795 (104.8 to 106.0)	272	302	362
776 to 785 (103.5 to 104.7)	271	301	361
766 to 775 (102.1 to 103.3)	271	301	361
756 to 765 (100.8 to 102.0)	270	300	360
746 to 755 (99.5 to 100.7)	269	299	359
736 to 745 (98.1 to 99.3)	269	299	359
726 to 735 (96.8 to 98.0)	268	298	358
716 to 725 (95.5 to 96.7)	267	297	357
706 to 715 (94.1 to 95.3)	267	297	356
696 to 705 (92.8 to 94.0)	266	296	356
686 to 695 (91.5 to 92.7)	265	295	355
676 to 685 (90.1 to 91.3)	265	295	354
666 to 675 (88.8 to 90.0)	264	294	353
656 to 665 (87.5 to 88.7)	264	293	353
646 to 655 (86.1 to 87.3)	263	292	352
636 to 645 (84.8 to 86.0)	262	292	351
626 to 635 (83.5 to 84.7)	262	291	350
616 to 625 (82.1 to 83.3)	261	290	350
606 to 615 (80.8 to 82.0)	260	290	349
596 to 605 (79.5 to 80.7)	260	289	348

quickly as possible. If the first drop falls from the condenser at a temperature above 300°C, start the timer as it falls into the receiver.

NOTE 1—The distillation should not be carried to a temperature above 360°C. At this point the temperature of the residue in the flask is near 450°C, the temperature at which pitch begins to decompose. Decomposition will make the results meaningless.

9.7 Calculate the distillation rate by dividing the weight, in grams, of the distillate betweeen 300 and 360°C by the time, in seconds, required to collect it. If the rate falls outside the limits of 0.03 to 0.05 g/s, discard the results and repeat the test with an appropriate adjustment of the heater control setting.

10. Report

10.1 Report the results of the distillation test as percentages, to the nearest 0.1 %, based on the dry sample weight.

11. Precision and Bias

11.1 The following criteria shall be used for judging the acceptability of the results (95 % confidence level):

11.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless they differ by more than 1.5 percentage points for the total distillate to 360°C.

11.1.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless they differ by more than 3.0 percentage points for the total distillate to 360°C.

11.1.3 *Bias*—This test method has no bias because the value of distillation of pitch is determined in terms of this test method.

12. Keywords

12.1 distill; distillate; distillation; fractionation; pitch