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Highway and Transportation Officials Standard
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Standard Test Method for Distillation of Cut-Back Asphaltic (Bituminous) Products[1]

This standard is issued under the fixed designation D 402; 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.

This standard has been approved for use by agencies of the Department of Defense. This method was adopted as a joint ASTM-IP standard in 1961.

1. Scope

1.1 This test method covers a distillation test for cut-back asphaltic (bituminous) products.

1.2 The values given in SI units are to be regarded as the standard. The inch-pound units 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 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure[2]

D 370 Test Method for Dehydration of Oil-Type Preservatives[3]

E 1 Specification for ASTM Thermometers[4]

E 133 Specification for Distillation Equipment[5]

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[4]

2.2 *IP Standards:*

IP 123/ASTM D 86, Distillation of Petroleum Products

Thermometers as specified in IP Standards

Crow Receiver as specified in British Standards 658:1989

C.O.3—Standard Methods for Testing Tar and its Products (Published by the U.K. Standardization of Tar Products Tests Committee)

3. Summary of Method

3.1 Two hundred millilitres of the sample are distilled in a 500-mL flask, at a controlled rate, to a temperature in the liquid of 360°C (680°F), and the volumes of distillate obtained at specified temperatures are measured. The residue from the distillation, and also the distillate, may be tested as required.

4. Significance and Use

4.1 This procedure measures the amount of the more volatile constituents in cut-back asphaltic products. The properties of the residue after distillation are not necessarily characteristic of the bitumen used in the original mixture, nor of the residue which may be left at any particular time after field application of the cut-back asphaltic product. The presence of silicone in the cut-back may affect the distillation residue by retarding the loss of volatile material after the residue has been poured into the residue container.

5. Apparatus

5.1 *Distillation Flask*, 500-mL side-arm, having the dimensions shown in Fig. 1.

5.2 *Condenser*, standard glass-jacketed, of nominal jacket length from 200 to 300 mm and overall tube length of 450 ± 10 mm (see Fig. 3).

5.3 *Adapter*, heavy-wall (1-mm) glass, with reinforced top, having an angle of approximately 105°. The inside diameter at the large end shall be approximately 18 mm, and at the small end, not less than 5 mm. The lower surface of the adapter shall be on a smooth descending curve from the larger end to the smaller. The inside line of the outlet end shall be vertical, and the outlet shall be cut or ground (not fire-polished) at an angle of 45 ± 5° to the inside line.

5.4 *Shield*, steel, lined with 3-mm fire proof insulation and fitted with transparent mica windows, of the form and dimensions shown in Fig. 2, used to protect the flask from air currents and to reduce radiation. The cover (top) shall be made in two parts of 6.4-mm fire proof insulation.

5.5 *Shield and Flask Support*—Two 15-cm^2 sheets of 16-mesh Chromel wire gauze on a tripod or ring.

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[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability and Distillation Tests.

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In the IP, this method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

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

[5] *Annual Book of ASTM Standards*, Vol 14.02.

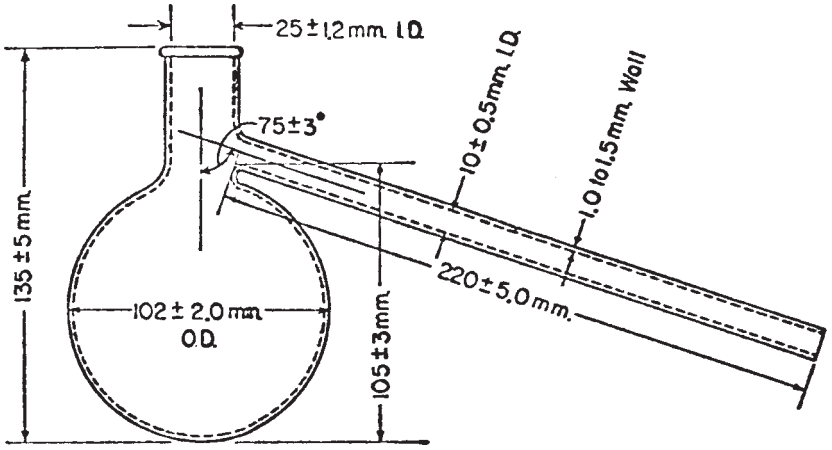


FIG. 1 Distillation Flask

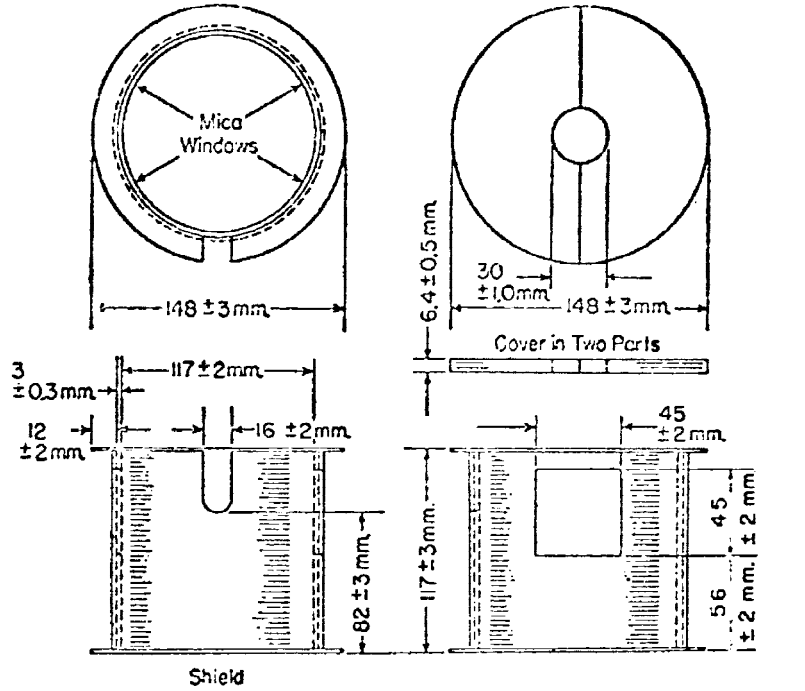


FIG. 2 Shield

5.6 *Heat Source*—

5.6.1 Adjustable Tirrill-type gas burner or equivalent.

5.6.2 An electric heater equipped with a transformer capable of controlling from 0 to 750 W. The shield and support shall be a refractory with an opening of 79 mm, with the upper surface beveled to 86 mm to accommodate the specified 500-mL flask. When the flask is placed on the refractory, there should be a distance of approximately 3 mm between the bottom of the flask and the heating elements.

5.7 *Receiver*—A standard 100-mL graduated cylinder conforming to dimensions of Specification E 133, or a 100-mL Crow receiver as shown in Fig. 4 of this test method.

NOTE 1—Receivers of smaller capacity having 0.1-mL divisions may be used when low volumes of total distillate are expected and the added accuracy required.

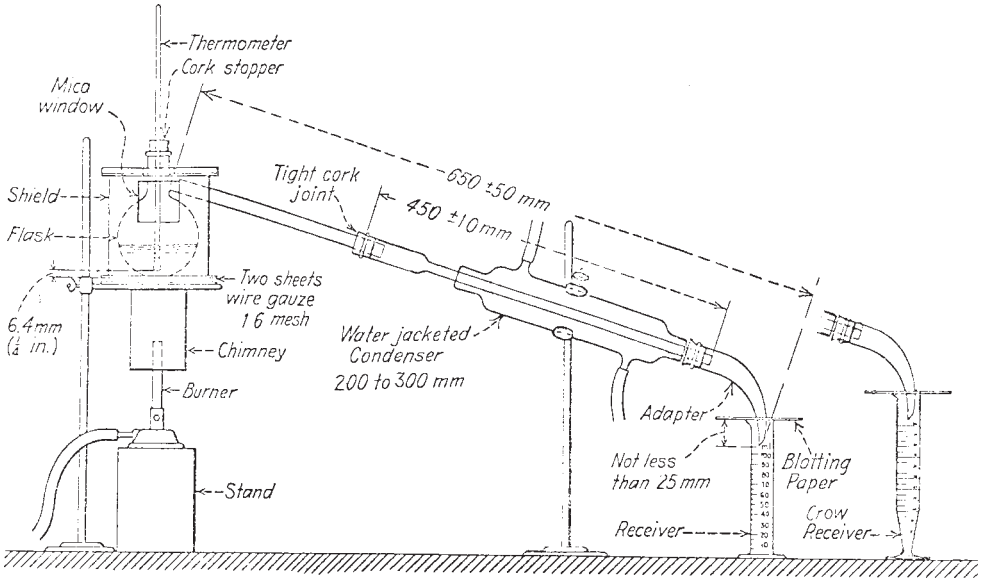
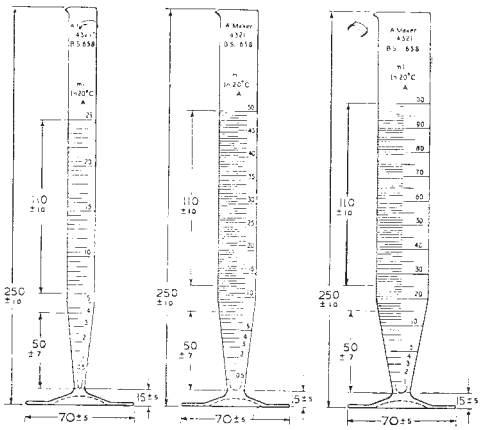


FIG. 3 Distillation Apparatus



All dimensions are in millimetres

FIG. 4 Crow Receivers of Capacity 25, 50, and 100 mL

5.8 *Residue Container*—A seamless metal container with slip on cover of 75 ± 5 mm in diameter, and 55 ± 5 mm in height.

5.9 *Thermometer*— ASTM High Distillation Thermometers having a range from −6 to 400°C (20 to 760°F) and conforming to the requirements for Thermometers 8C (8F) as prescribed in Specification E 1, or IP Thermometer 6C conforming to IP Specifications for Standard Thermometers, or an equivalent thermometric device that has been calibrated in accordance with Test Method E 220. ASTM 8C Thermometers shall be used for referee testing.

6. Sampling

6.1 Stir the sample thoroughly, warming if necessary, to ensure homogeneity before removal of a portion for analysis.

6.2 If sufficient water is present to cause foaming or bumping, dehydrate a sample of not less than 250 mL by heating in a distillation flask sufficiently large to prevent foaming over into the side arm. When foaming has ceased, stop the distillation. If any light oil has distilled over, separate and pour this back into the flask when the contents have cooled just sufficiently to prevent loss of volatile oil. Mix the contents of the flask thoroughly before removal for analysis. An alternative procedure is described in Test Method D 370.

7. Preparation of Apparatus

7.1 Calculate the weight of 200 mL of the sample from the specific gravity of the material at 15.6/15.6°C. Weigh this amount ± 0.5 g into the 500-mL flask.

7.2 Place the flask in the shield supported by two sheets of gauze on a tripod or ring. Connect the condenser tube to the tubulature of the flask with a tight cork joint. Clamp the condenser so that the axis of the bulb of the flask through the center of its neck is vertical. Adjust the adapter over the end of the condenser tube so that the distance from the neck of the flask to the outlet of the adapter is 650 ± 50 mm (see Fig. 3).

7.3 Insert the thermometer through a tightly fitting cork in the neck of the flask so that the bulb of the thermometer rests on the bottom of the flask. Raise the thermometer 6 mm from the bottom of the flask using the scale divisions on the thermometer to estimate the 6 mm distance above the top of the cork.

7.4 Protect the burner by a suitable shield or chimney. Place the receiver so that the adapter extends at least 25 mm but not below the 100-mL mark. Cover the graduate closely with a piece of blotting paper, or similar material, suitably weighted, which has been cut to fit the adapter snugly.

7.5 The flask, condenser tube, adapter, and receiver shall be clean and dry before starting the distillation. Place the seamless residue container on its cover in an area free from drafts.

7.6 Pass cold water through the condenser jacket. Use warm water if necessary to prevent formation of solid condensate in the condenser tube.

8. Procedure

8.1 Correct the temperatures to be observed in the distillation if the elevation of the laboratory at which the distillation is made deviates 150 m or more from sea level. Corrected

temperatures for the effect of altitude are shown in Table 1 and Table 2. If the prevailing barometric pressure in millimetres of mercury is known, correct the temperature to be observed with the corrections shown in Table 3. *Do not correct for the emergent stem of the thermometer.*

NOTE 2—Table 3 covers a wide range of temperatures from 160 to 360°C (320 to 680°F) and is to be preferred for world-wide specifications other than ASTM/IP specifications.

8.2 Apply heat so that the first drop of distillate falls from the end of the flask side-arm in 5 to 15 min. Conduct the distillation so as to maintain the following drop rates, the drop count to be made at the tip of the adapter:

50 to 70 drops per minute to 260°C (500°F)
20 to 70 drops per minute between 260 and 316°C (500 and 600°F)
Not over 10 min to complete distillation from 316 to 360°C (600 to 680°F)

8.2.1 Record the volumes of distillate to the nearest 0.5 mL in the receiver at the corrected temperatures. If the volume of distillate recovered is critical, use receivers graduated in 0.1-mL divisions and immersed in a transparent bath maintained at 15.6 ± 3°C.

NOTE 3—Some cut-back asphaltic products yield either no distillate or very little distillate over portions of the temperature range to 316°C (600°F). In this case it becomes impractical to maintain the above distillation rates. For such cases the intent of the method shall be met if the rate of rise of temperature exceeds 5°C (9°F)/min.

8.3 When the temperature reaches the corrected temperature of 360°C (680°F), cut off the heat and remove the flask and thermometer. With the flask in a pouring position, remove the thermometer and immediately pour the contents into the residue container. The total time from cutting off the heat to starting the pour shall not exceed 30 s. When pouring, the side-arm should be substantially horizontal to prevent condensate in the side-arm from being returned to the residue.

NOTE 4—The formation of skin on the surface of a residue during cooling entraps vapors which will condense and cause higher penetration results when they are stirred back into the sample. If skin begins to form during cooling, it should be gently pushed aside. This can be done with a spatula with a minimum of disturbance to the sample.

TABLE 1 Corrected Distillation Temperatures for Various Altitudes, °C

Elevation above Sea Level, m	Distillation Temperatures for Various Altitudes, °C				
−300	192	227	262	318	362
−150	191	226	261	317	361
0	190	225	260	316	360
150	189	224	259	315	359
300	189	223	258	314	358
450	188	223	257	313	357
600	187	222	257	312	356
750	186	221	256	311	355
900	186	220	255	311	354
1050	185	220	254	310	353
1200	184	219	254	309	352
1350	184	218	253	308	351
1500	183	218	252	307	351
1650	182	217	251	306	350
1800	182	216	250	306	349
1950	181	216	250	305	348
2100	180	215	249	304	347
2250	180	214	248	303	346
2400	179	214	248	303	346

TABLE 2 Corrected Distillation Temperatures for Various Altitudes, °F

Elevation above sea level, m (ft)	Distillation Temperatures for Various Altitudes, °F				
−300	377	440	503	604	684
−150	375	438	502	602	682
0	374	437	500	600	680
150	373	436	499	598	678
300	371	434	497	597	676
450	370	433	495	595	675
600	369	431	494	593	673
750	368	430	493	592	671
900	366	429	491	590	669
1050	365	427	490	589	668
1200	364	426	488	587	666
1350	363	425	487	586	665
1500	362	424	486	584	663
1650	360	422	484	583	661
1800	359	421	483	581	660
1950	358	420	482	580	658
2100	357	419	481	579	657
2250	356	418	479	577	655
2400	355	416	478	576	654

TABLE 3 Factors for Calculating Temperature Corrections

Nominal Temperatures, °C (°F)	Correction[A] per 10 mm Hg Difference in Pressure, °C (°F)
160 (320)	0.514 (0.925)
175 (347)	0.531 (0.957)
190 (374)	0.549 (0.989)
225 (437)	0.591 (1.063)
250 (482)	0.620 (1.116)
260 (500)	0.632 (1.138)
275 (527)	0.650 (1.170)
300 (572)	0.680 (1.223)
315.6 (600)	0.698 (1.257)
325 (617)	0.709 (1.277)
360 (680)	0.751 (1.351)

[A] To be subtracted in case the barometric pressure is below 760 mm Hg; to be added in case barometric pressure is above 760 mm Hg.

8.4 Allow the condenser and any distillates trapped in the condenser neck to drain into the receiver and record the total volume of distillate collected as total distillate to 360°C (680°F).

8.5 When the residue has cooled until fuming just ceases, stir thoroughly and then, when the material reaches 135 ± 5°C (275 ± 9°F), pour into the receptacles for testing for properties such as penetration, viscosity, or softening point. Proceed as required by the appropriate ASTM or IP method from the point that follows the pouring stage.

8.6 If desired, the distillate, or the combined distillates from several tests, may be submitted to a further distillation, in accordance with Test Method D 86 – IP 123, or, when the distillate is of coal-tar origin, Method C.O.3.

9. Calculation and Report

9.1 *Asphaltic Residue*—Calculate the percent residue to the nearest 0.1 as follows:

$$R = [(200 - TD)/200] \times 100 \quad (1)$$

where:
R = residue content, in volume percent, and
TD = total distillate recovered to 360°C (680°F), mL.

9.1.1 Report as the residue from distillation to 360 (680°F), percent volume by difference.

9.2 *Total Distillate*— Calculate the percent total distillate to the nearest 0.1 as follows:

$$TD\ \% = (TD/200) \times 100 \quad (2)$$

9.2.1 Report as the total distillate to 360°C (680°F), volume percent.

9.3 *Distillate Fractions*:

9.3.1 Determine the percentages by volume of the original sample by dividing the observed volume (in millilitres) of the fraction by 2. Report to the nearest 0.1 as volume percent as follows:

Up to 190°C (374°F)
Up to 225°C (437°F)
Up to 260°C (500°F)
Up to 316°C (600°F)

9.3.2 Determine the percentages by volume of total distillate by dividing the observed volume in millilitres of the fraction by the millilitres recovered to 360°C (680°F) and multiplying by 100. Report to the nearest 0.1 as the distillate, volume percent of total distillate to 360°C (680°F) as follows:

Up to 190°C (374°F)
Up to 225°C (437°F)
Up to 260°C (500°F)
Up to 316°C (600°F)

9.4 Where penetration, viscosity, or other tests have been carried out, report with reference to this test method as well as to any other method used. *Example*—Penetration (ASTM D5 or IP 49) of residue from ASTM D402/IP 27.

10. Precision and Bias

10.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

10.1.1 *Repeatability*— Duplicate values by the same operator shall not be considered suspect unless the determined percentages differ by more than 1.0 volume % of the original sample.

10.1.2 *Reproducibility*— The values reported by each of two laboratories, shall not be considered suspect unless the reported percentages differ by more than the following:

Distillation Fractions, volume percent of the original sample:	
Up to 175°C (347°F)	3.5
Above 175°C (347°F)	2.0
Residue, Volume percentage by difference from the original sample	2.0

10.2 Criteria for judging variability of test results on the distillation residue have not been determined.

11. Keywords

11.1 cut-back asphalt; distillate; residue