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Standard Test Method for Distillation of Cutback Asphalt[1]

This standard is issued under the fixed designation D402/D402M; 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.

1. Scope

1.1 This test method covers a distillation test for cutback asphalts.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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:*[2]

D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure
D370 Practice for Dehydration of Oil-Type Preservatives
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E133 Specification for Distillation Equipment
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E644 Test Methods for Testing Industrial Resistance Thermometers
E1137 Specification for Industrial Platinum Resistance Thermometers

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 cutback asphalt. 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 cutback asphalt. The presence of silicone in the cutback asphalt 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.

[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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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

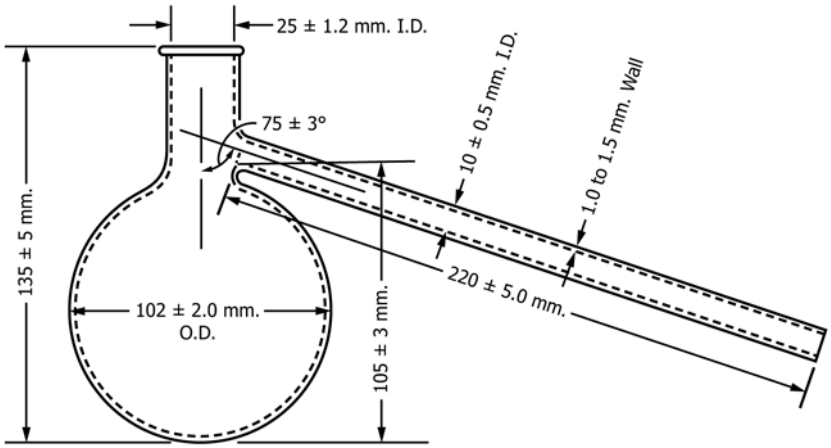


FIG. 1 Distillation Flask

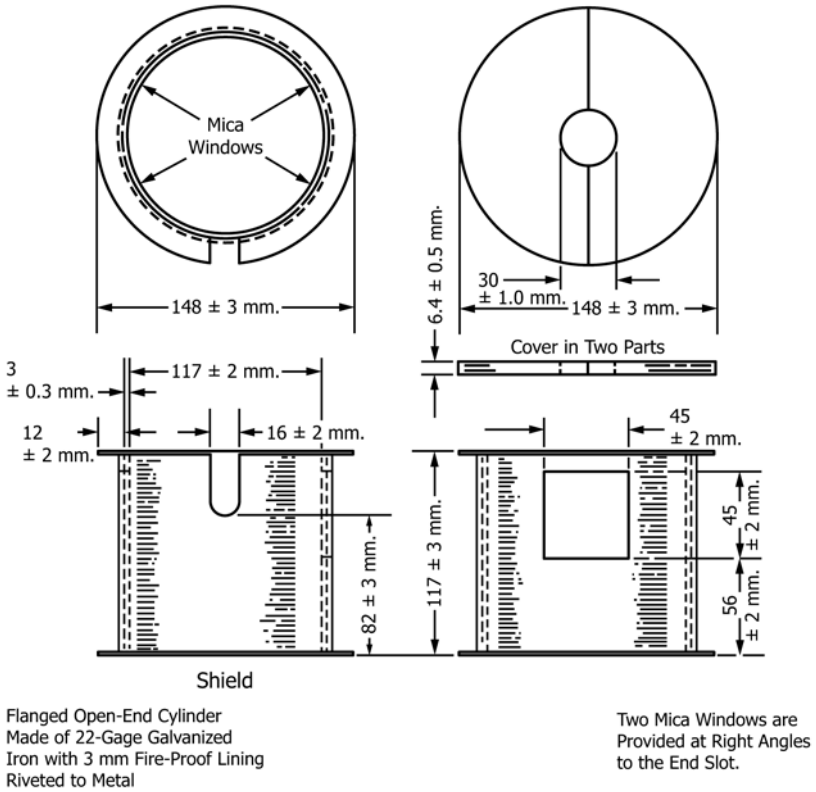


FIG. 2 Shield

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 E133, 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.

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*—The thermometer shall be one of the following:

5.9.1 An 8C (8F) thermometer which conforms to the requirements of Specification E1. Calibrate the thermometer in accordance with one of the methods in Test Method E77.

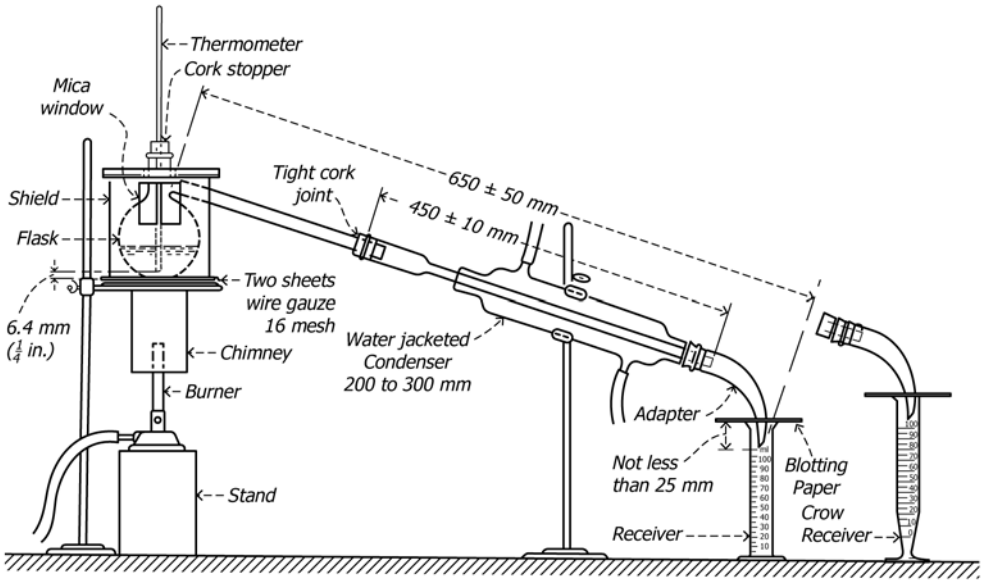
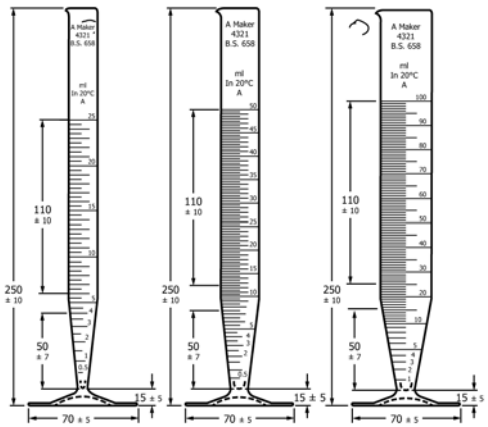


FIG. 3 Distillation Apparatus



All dimensions are in millimetres

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

5.9.2 A platinum resistance thermometer (PRT) with a probe which conforms to the requirements of Specification E1137. The PRT shall have a 3- or 4-wire connection configuration and the overall sheath length shall be at least 50 mm [2 in.] greater than the immersion depth. Calibrate the PRT system (probe and readout) in accordance with Test Methods E644. Corrections shall be applied to ensure accurate measurements within 1°C [2°F].

5.9.3 A metal-sheathed Type T thermocouple with a sensor substantially-similar in construction to the PRT probe described in 5.9.2. Calibrate the thermocouple system (sensor and readout) in accordance with Test Method E220. Corrections shall be applied to ensure accurate measurements within 1°C [2°F].

6. Hazards

6.1 **Warning—**Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury, mercury-containing products, or both, into your state may be prohibited by state law.

7. Sampling

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

7.2 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 Practice D370.

8. Preparation of Apparatus

8.1 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.

8.2 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).

8.3 Insert the thermometric device through a tightly fitting cork in the neck of the flask so that the bulb of the thermometric device rests on the bottom of the flask. Raise the

thermometric device approximately 6 mm from the bottom of the flask using the scale divisions or a reference mark on the thermometric device to estimate the 6 mm distance above the top of the cork.

8.4 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.

8.5 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.

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

9. Procedure

9.1 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 (if used).*

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 specifications.

9.2 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]

9.2.1 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 cutback asphalt 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.

9.3 When the temperature reaches the corrected temperature of 360°C [680°F], turn off the heat and remove the flask containing the thermometric device. With the flask in a pouring position, remove the thermometric device and immediately pour the contents into the residue container. The total time from turning off the heat to starting the pour shall not exceed 60 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.

9.4 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].

9.5 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 method from the point that follows the pouring stage.

9.6 If desired, the distillate, or the combined distillates from several tests, may be submitted to a further distillation, in accordance with Test Method D86.

10. Calculation and Report

10.1 *Asphalt 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.

10.1.1 Report as the residue from distillation to 360 [680°F], percent volume by difference.

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

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

10.2.1 Report as the total distillate to 360°C [680°F], volume percent.

10.3 *Distillate Fractions:*

10.3.1 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]

10.3.2 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]

10.4 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) of residue from ASTM D402.

11. Precision and Bias

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

11.1.1 *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.

11.1.2 *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

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

12. Keywords

12.1 cutback asphalt; distillate; residue