



Standard Test Method for Loss on Heating of Oil and Asphaltic Compounds¹

This standard is issued under the fixed designation D6/D6M; 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.

ϵ^1 NOTE—Units information was editorially revised in February 2011.

1. Scope

1.1 This test method covers the determination of the loss in mass (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed.

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 and regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D5 Test Method for Penetration of Bituminous Materials
E1 Specification for ASTM Liquid-in-Glass Thermometers
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Summary of Test Method

3.1 Fifty grams of material, spread out in a dish 55 mm in diameter, is heated in moving air for 5 h at 163°C [325°F] and the percent loss of mass determined along with a comparison, before and after, of any other desired characteristics. This test

method provides only a relative measurement of the volatility of a material under test conditions.

4. Significance and Use

4.1 This test method is useful in characterizing certain petroleum products by the determination of their loss of mass upon heating under standardized conditions.

5. Apparatus

5.1 *Oven*—The oven shall be electrically heated and shall conform to the performance requirements of Specification E145 Type I, Grade B, for operating temperatures up to 180°C [365°F]. During the tests for compliance to Specification E145 requirements, the oven shelf, properly placed, shall be in place and rotating. In addition, it shall also comply with the following requirements:

5.1.1 *Construction (Note 1)*—The oven shall be rectangular with minimum interior dimensions of 330 mm [13 in.] in each direction. The oven shall have in front a tightly fitting hinged door, which shall provide a clear opening substantially the same as the interior height and width of the oven. The door may contain a window with dimensions of at least 100 by 100 mm [4 by 4 in.], and with two sheets of glass separated by an air space, through which a vertical thermometer located as specified in Section 8, may be read without opening the door; or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily. The oven shall be adequately ventilated by convection currents of air and for this purpose shall be provided with openings for the entrance of air and for the exit of heated air and vapors. Openings may be of any size and arrangement provided the requirements of Specification E145, Type I, Grade B, are met.

5.1.2 *Rotating Shelf*—The oven shall be provided with a circular metal shelf having a minimum diameter of 250 mm [9.8 in.]. (A recommended form of aluminum shelf is shown in Fig. 1.) The shelf shall be suspended by a vertical shaft and centered with respect to the horizontal interior dimensions. The shelf shall be provided with a mechanical means of rotating it

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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.

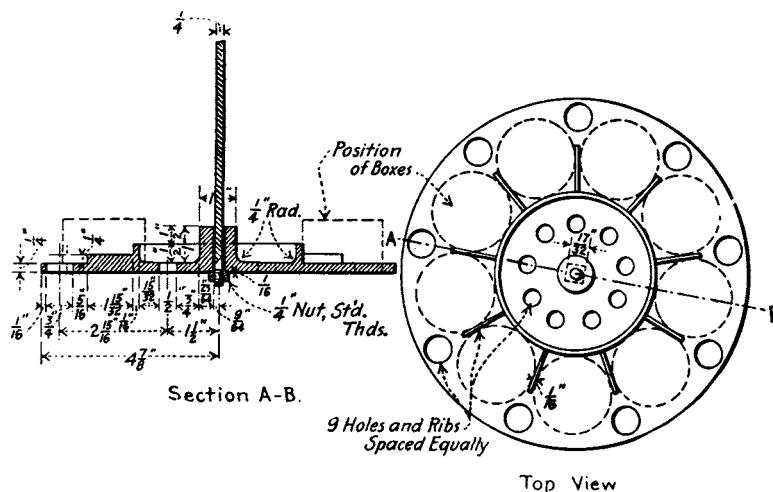


FIG. 1 Aluminum Shelf

at the rate of 5 to 6 rpm. The shelf shall be vertically located as close to the center of the oven as permitted by compliance with the requirements of **7.2** regarding thermometer placement.

NOTE 1—Continued use of existing smaller ovens complying with the requirements of this test shall be permitted for an indefinite period to provide for normal amortization of present equipment. However, all new ovens purchased should comply with the minimum dimensions and requirements of this test method.

5.2 Thermometer—An ASTM Loss on Heat Thermometer graduated in Celsius degrees, having a range from 155 to 170°C and conforming to the requirements for Thermometer 13C as prescribed in the Specification **E1**.

5.3 Container—The container in which the sample is to be tested shall be of metal or glass, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows: diameter, 55 mm [2.17 in.], depth, 35 mm [1.38 in.].

NOTE 2—Containers known in the drug trade as seamless “ointment boxes” may be obtained in dimensions conforming to the above requirements.

6. Technical Hazards

6.1 Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. When extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given in Section **9**. Results obtained on samples showing evidences of foaming during the test shall be rejected.

7. Preparation of Sample

7.1 Thoroughly stir and agitate the sample as received, warming, if necessary, to obtain a complete mixture before the portion for analysis is removed.

7.2 First test the material under examination for water and, if water is present, remove it by suitable methods of dehydration before subjecting the material to the loss on heating test; or, obtain another sample that is free of water.

8. Procedure

8.1 Place 50.0 \pm 0.5 g of the sample of the water-free material to be tested in a tared container conforming to the requirements of **5.3**, and weigh to the nearest 0.01 g. If the material has been heated to facilitate transfer, cool the sample to room temperature before making this initial weighing. Bring the oven to a temperature of 163°C [325°F], and place the container with the weighed sample on and near the circumference of the circular shelf, in one of the recesses if the recommended shelf is used. Close the oven and rotate the shelf during the entire test at a rate of 5 to 6 rpm. Maintain the temperature at 163 \pm 1°C [325 \pm 1.8°F] for 5 h after the sample has been introduced and the oven has again reached that temperature. The 5-h period shall start when the temperature reaches 162°C [324°F], and in no case shall the total time that a sample is in the oven be more than 5 h and 15 min. At the conclusion of the heating period, remove the sample from the oven, cool to room temperature, weigh to the nearest 0.01 g, and calculate the loss due to heating.

8.2 Determine temperatures by means of the specified thermometer, which shall be supported from the shaft of the

TABLE 1 Reproducibility of Results

Volatilization Loss, %	Numerical Correction	True Volatilization Loss, %
5.0	\pm 0.50	4.50 to 5.50
5.5	\pm 0.51	4.99 to 6.01
6.0	\pm 0.52	5.48 to 6.52
10.0	\pm 0.60	9.40 to 10.60
15.0	\pm 0.70	14.30 to 15.70
25.0	\pm 0.90	24.10 to 25.90
40.0	\pm 1.20	38.80 to 41.20

circular shelf in a vertical position approximately 19 mm [0.75 in.] inside the periphery of the shelf, and with the bottom of the thermometer bulb 6 mm [0.25 in.] above the shelf.

NOTE 3—If additional periods of heating are desired, it is recommended that they be made in successive increments of 5 h each.

NOTE 4—When it is required that the penetration or other characteristics of the sample after heating be determined, the residue should be melted in the container at the lowest possible temperature and thoroughly mixed by stirring, taking care to avoid incorporating air bubbles in the material. For the penetration test, the well-mixed residue shall be brought to standard temperature and tested as prescribed in Test Method D5. For other tests, the well-mixed residue should be tested according to standard conditions as required by the test procedure involved.

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9. Precision and Bias

9.1 Up to 5 % loss in mass, the results obtained may be considered as correct within 0.5. Above 5 % loss in mass, the numerical limit of error increases 0.01 for every 0.5 % increase in loss by volatilization as shown in Table 1.

9.2 *Bias*—The procedure in Test Method D6 has no bias because the value of the loss on heating of oil and asphaltic compounds test, is defined in terms of the test method.

10. Keywords

10.1 asphalt; heating; oil; oven