

AMERICAN SOCIETY FOR TESTING MATERIALS
1315 SPRUCE STREET, PHILADELPHIA, PA

A.S.T.M.

STANDARD METHOD OF TEST
FOR
LOSS ON HEATING OF OIL AND ASPHALTIC
COMPOUNDS¹

A.S.T.M. DESIGNATION: D 6 - 30

This method is issued under the fixed designation D 6, the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Adopted, 1911; Revised, 1916, 1920, 1927, 1930.

1. This test covers the determination of the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination shall, therefore, first be tested for water and if water is found to be present, it shall be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample shall be obtained which is free from water.

APPARATUS

2. The oven shall be rectangular in form with double walls and heated by electricity. Its interior dimensions shall be as follows: height, exclusive of space occupied by the heating element, not less than 29.26 cm. (11.5 in.), width and depth, each, at least 5.08 cm. (2 in.) greater than the diameter of the revolving shelf.

The oven shall be provided with a perforated metal circular shelf approximately 24.8 cm. (9.75 in.) in diameter. A recommended form of aluminum shelf is shown in Fig. 1. This shelf shall be placed in the center of the oven, with respect to all dimensions of the interior of the same, suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of 5 to 6 revolutions per minute.

One side of the oven shall be hinged and equipped to serve as a tight fitting door which shall contain a window at least 4 inch square, with double glass, through which a thermometer, located in front of and level with the revolving shelf, may be read without opening the door.

¹Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving material.

DSW 330741

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One round vent hole each shall either be placed in the top and bottom of the oven or one only near the top and one only near the bottom on each side of the oven. Each hole shall be not less than 1.27 cm. (0.5 in.) nor more than 1.60 cm. (0.63 in.) in diameter and they shall both be left open during the operation of the oven.

3. The thermometer shall conform to the following requirements. The specifications cover a special thermometer graduated in Centigrade degrees, the range being 155 to 170 °C. (Requirements not copied as this thermometer does not cover the 200°F. point).

4. 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: The American Can company's 3-oz. Gill style flat-bottom seamless ointment box, deep pattern, fulfills these requirements.

PREPARATION OF SAMPLE

5. The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

PROCEDURE

6. Fifty grams of the water-free material to be tested shall be weighed into a tared container conforming to the requirements of Section 4. The oven shall be brought to a temperature of 163°C. (350°F.), and the box containing the sample placed in one of the recesses of the revolving shelf. The oven shall then be closed and the shelf rotated 5 to 6 revolutions per minute during the entire test. The temperature shall be maintained at 163°C. (325°F.) for 5 hours after the sample has been introduced and the oven has again reached that temperature. The sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated.

7. During the 5 hour period the temperature shall not vary more than 1°C. All tests showing a greater variation in temperature shall be rejected.

8. 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. Where 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.

DSW 330742

STLCOPCB4077386

ACCURACY

9. Up to 5 per cent loss in weight the results obtained may be considered as correct within 0.5. Above 5 per cent loss in weight the numerical limit of error increases 0.01 for every 0.5 per cent increase in loss by volatilization as follows:

<u>Volatilization Loss, per cent</u>	<u>Numerical Correction</u>	<u>True Volatilization Loss, per cent</u>
5.0	±0.50	4.50 to 5.50
5.5	±0.51	4.99 to 6.01
6.0	±0.52	5.48 to 6.52
10.0	±0.60	9.40 to 10.60
15.0	±0.70	14.30 to 15.70
25.0	±0.90	24.10 to 25.90
40.0	±1.20	38.80 to 41.20

NOTE: For the determination of loss on heating at 200° F. on Aroclors 1248 and 1250 the above procedure was modified in that temperature of oven was maintained at 200° F. instead of 165° C.

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