Designation: D1203 – 16

Standard Test Methods for Volatile Loss From Plastics Using Activated Carbon Methods[1]

This standard is issued under the fixed designation D1203; 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 U.S. Department of Defense.

1. Scope*

1.1 These test methods cover the determination of volatile loss from a plastic material under defined conditions of time and temperature, using activated carbon as the immersion medium.

1.2 Two test methods are covered as follows:

1.2.1 *Test Method A, Direct Contact with Activated Carbon*—In this test method the plastic material is in direct contact with the carbon. This test method is particularly useful in the rapid comparison of a large number of plastic specimens.

1.2.2 *Test Method B, Wire Cage*—This test method prescribes the use of a wire cage, which prevents direct contact between the plastic material and the carbon. By eliminating the direct contact, the migration of the volatile components to the surrounding carbon is minimized and loss by volatilization is more specifically measured.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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.*

NOTE 1—This standard and ISO 176 address the same subject matter, but differ in technical content.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Documents:*

ISO 176 Determination of the Loss of Plasticizers from Plastics by the Activated Carbon Method[3]

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminologies D883 and D1600 unless otherwise indicated.

4. Significance and Use

4.1 The test methods are intended to be rapid empirical tests which have been found to be useful in the relative comparison of materials having the same nominal thickness.

NOTE 2—When the plastic material contains plasticizer, loss from the plastic is assumed to be primarily plasticizer. The effect of moisture is considered to be negligible.

4.2 Correlation with ultimate application for various plastic materials shall be determined by the user.

5. Apparatus

5.1 *Balance*—An accurate analytical balance, equipped with Class S weights or better.

5.2 *Oven or Bath*—A thermostatically controlled oven or bath capable of maintaining the temperature to within ±1°C of the test temperature, which normally will be in the range from 50 to 150°C.

5.3 *Containers*—Metal cans or wide-mouth screw-top jars, of cylindrical form, approximately 100 mm in diameter and approximately ½ L in capacity.

NOTE 3—Pint paint cans work well.

5.4 *Micrometer*—A micrometer capable of measuring to the nearest 0.0025 mm for measuring the thickness of the test specimens.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.11 on Plasticizers).

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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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

***A Summary of Changes section appears at the end of this standard**

5.5 *Metal Cages (for Test Method B)*—Wire cages constructed from approximately 30-mesh bronze gauze, in cylindrical form, having a diameter of 60 mm and a height of 6 mm, formed by soldering a strip of gauze at right angles to the periphery of a disk of bronze gauze. One of the bases acts as a lid.

5.6 *150 mL Beaker* graduated in 10 mL intervals.

6. Material

6.1 *Activated Carbon, 5/14 Mesh*—It has been found that different types and grades of activated carbon give differing results, thus making it necessary for the purchaser and the seller to agree on the same type and grade in order to obtain concordant results. Care shall be taken that an airtight storage container is used for the activated carbon and that fresh material is used for each test. The activated carbon shall be screened through a 14-mesh screen immediately prior to use to eliminate fines.

7. Test Specimens

7.1 The test specimens shall be 50 ± 1 mm diameter disks made of the plastic material to be tested. Three specimens of each formulation shall be tested.

7.2 Thickness of the test specimens shall be 0.25 ± 0.025 mm.

NOTE 4—If another thickness is desired to be tested due to purchase specifications or other considerations, it shall be specified in the report.

7.3 Direct comparison of values between materials shall not be made unless all specimens so compared do not vary by more than ±10 % from a given nominal thickness.

NOTE 5—This precaution is necessary because of discrepancies that may arise due to edge effects, depletion of volatiles, and the fact that the percent weight loss is an inverse function of thickness.

8. Conditioning

8.1 *Conditioning*—Condition the test specimens at 23 ± 2°C and 50 ± 10 % relative humidity for not less than 20 h prior to test in accordance with Procedure A of Practice D618. Preferably, specimens shall be suspended to assure free air circulation among the specimens. In cases of disagreement, the tolerances shall be ±1°C and ±5 % relative humidity.

9. Procedure—Test Method A, Direct Contact with Activated Carbon

9.1 Weigh the conditioned specimens individually on the analytical balance and designate this weight as W_1. Weight of individual specimens shall be within ±10 % of each other.

9.2 Spread 120 cm^3 of activated carbon evenly on the bottom of a container. Place one specimen on top of the activated carbon and cover it with 120 cm^3 of activated carbon. Place a second specimen (Note 4) on top of the first and cover it with 120 cm^3 of the carbon, followed by a third specimen and then 120 cm^3 more of activated carbon. Place a cover on the container in such a manner that the container will be vented. This is necessary to assure that any possible pressure build-up in the container during heating is relieved. Take care that in no case shall the carbon be packed by pressure other than the weight of the composite sandwich in the container.

NOTE 6—Only specimens of the same composition or formulation shall be tested in a single container, because of the possibility of cross-migration between varying compositions.

9.3 Place the container upright in the oven or bath. Unless otherwise specified, the temperature of the oven or bath shall be 70 ± 1°C and the duration of the test 24 h.

9.4 At the end of the 24-h period, remove the container from the oven or bath. Then, within 1 h, remove the specimens from the container, brush free of carbon, and recondition in accordance with Section 8.

9.5 After reconditioning, reweigh the specimens and designate this weight as W_2. Weight of individual specimens shall be within ±10 % of each other.

10. Procedure—Test Method B, Wire Cage

10.1 Proceed as in Section 9 (Test Method A), except place every individual specimen in a small metal wire-mesh cage constructed as indicated in 5.5, and maintain the temperature at 100 ± 1°C.

NOTE 7—If other conditions of test are desired, they may be employed, but shall be specified in the report.

11. Calculation

11.1 Calculate the volatile loss, expressed as percent weight loss based on the original specimen weight, as follows:

$$\text{weight loss, \%} = \left[\left(W_1 - W_2 \right) / W_1 \right] \times 100 \quad (1)$$

where:

W_1 = initial weight of test specimen, and
W_2 = final weight of test specimen.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, and previous history,

12.1.2 Actual thickness to the nearest 0.025 mm for each of the three specimens tested, and the average of the three,

12.1.3 Percent weight loss recorded to two significant figures of each of the three specimens, and the average of the three,

12.1.4 Any observations as to distortion or change in appearance of the specimens,

12.1.5 Type of activated carbon used, and

12.1.6 Test temperature and duration of test.

13. Precision and Bias

13.1 *Precision:*[4]

13.1.1 Table 1 is based on a round robin conducted in 1988 per Practice E691, involving three materials tested by five laboratories. All tests were run by Test Method A for 24 h at 90°C. For each material, all the samples were prepared at one

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1129.

TABLE 1 Plasticizer Volatility–Round Robin Test

Material	Volatility	Average Loss,%	S_r [A]	S_R [B]	r [C]	R [D]
Sample 1	High	19.46	0.70	2.43	1.98	6.88
Sample 2	Medium	3.83	0.35	0.87	0.98	2.48
Sample 3	Low	0.81	0.12	0.42	0.35	1.20

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $r = 2.8\ S_r$.
[D] $R = 2.8\ S_R$.

source, but the individual specimens were prepared by the laboratory which tested them. Each test result was the average of 23 individual determinations. Three laboratories obtained two test results for each material, whereas two test laboratories obtained one test result for each material.

13.1.1.1 The properties used in the analysis are volatility of plasticizer from the plastic. The three materials were selected to represent low, medium, and high volatility plasticizers. (**Warning—**The following explanations of *r* and *R* (13.1.2 – 13.1.2.3) are only intended to present a means of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to acceptance or rejection of material, as those data are specific to this round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of these test methods should apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.1.2 – 13.1.2.3 would then be valid for such data.)

13.1.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing 23 specimens:

13.1.2.1 *Repeatability, r* (comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the *r* value of the material.

13.1.2.2 *Reproducibility, R* (comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results should be judged not equivalent if they differ by more than the *R* value for that material.

13.1.2.3 Any judgment in accordance with 13.1.2.1 or 13.1.2.2 would have approximate 95 % (0.95) probability of being correct.

13.2 *Bias*—There are no recognized standards by which to estimate bias of these test methods.

14. Keywords

14.1 plasticizer loss; vinyl; volatile loss; volatility; weight loss

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1203 - 10) that may impact the use of this standard. (April 1, 2016)

(1) Added ± to 7.1 specimen diameter.
(2) Changed "Weight of individual specimens shall be within a tolerance of ±10 %." to "Weight of individual specimens shall be within ±10 % of each other." in 9.1 and 9.5.
(3) Moved Table 1 to Precision and Bias section.