



Standard Test Method for Weight Loss of Plasticizers on Heating¹

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1. Scope *

1.1 This test method covers the determination of the relative volatility of plasticizers, or volatile contaminants, or both, at elevated temperatures.

1.2 The text of this test method references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of this test method.

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—There are no ISO standards covering the primary subject of this test method.

2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing²

D 883 Terminology Relating to Plastics²

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³

3. Terminology

3.1 *General*—Definitions are in accordance with Terminology D 883 and abbreviations with Terminology D 1600, unless otherwise indicated.

4. Summary of Test Method

4.1 Plasticizers are heated in crystallizing dishes in a circulating air oven on a rotating turntable, at either 105°C (221°F) or 155°C (310°F). The specimens are removed from the oven,

cooled and weighed, after heating periods of 2, 4, and 24 h. The weight lost in these times is determined and reported as percent plasticizer loss.

5. Significance and Use

5.1 The quantity of volatile components in a plasticizer has influence on fuming during processing, and the retention of flexibility and other properties in the end product (see 5.2). This test method may be used to measure the volatile components under closely controlled conditions.

5.2 Results obtained by this test method are not strictly equivalent to those obtained during processing where conditions of temperature and air flow are quite different.

5.3 Volatility is dependent upon air flow and temperature. Due to the difficulty of controlling air flow, results may vary widely between ovens. Therefore, control plasticizers shall be run simultaneously when making close comparisons.

6. Apparatus

6.1 *Oven*—Forced-ventilation laboratory oven, Type II, Grade A, with rotating turntable driven at a rate of 2 to 6 rpm as specified in Specification E 145.

6.2 *Crystallizing Dishes*, 50 mm (2 in.) in diameter and 35 mm (1 3/8 in.) in height.

7. Temperatures of Test

7.1 The test temperatures shall be 105 ± 1°C (221 ± 2°F) or 155 ± 2°C (310 ± 5°F).

NOTE 2—Preferably volatile plasticizers are tested at 105°C and permanent-type plasticizers are tested at 155°C. Volatile plasticizers and permanent-type plasticizers should not be tested together, as the permanent-type plasticizer may gain weight by absorption of volatiles.

8. Conditioning

8.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ± 1°C (± 1.8°F) and ± 2 % relative humidity.

8.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 10.01 and 14.02.

tolerances shall be $\pm 1^{\circ}\text{C}$ ($\pm 1.8^{\circ}\text{F}$) and $\pm 2\%$ relative humidity.

9. Procedure

9.1 Adjust the oven to the desired temperature with the air ducts open.

9.2 Dry the cleaned dishes in the oven at the test temperature for 30 min, desiccate, cool, and tare on the analytical balance. This weight is W_D .

NOTE 3—Under the conditions of this test some plasticizers may gain weight due to oxidation. To eliminate this possibility, add 0.1 % bisphenol A to each plasticizer in such a manner as to ensure complete dissolution.

9.3 Using a pan balance, add 10.0 ± 0.1 g of plasticizer to each dish.

NOTE 4—To maintain a constant surface area for volatilization, avoid allowing plasticizer to run down sides of dish.

9.4 Reweigh the dishes and contents on the analytical balance. This weight is W_0 .

9.5 Place the dishes on the oven turntable equally spaced from the center (avoid the periphery).

9.6 After 2 h remove the dishes, desiccate, cool, and reweigh analytically. This weight is W_2 .

9.7 Return the dishes to the oven, heat for an additional 2 h, cool, and weigh as in 9.6. This is weight W_4 . Return the dishes to the oven, heat an additional 20 h, cool, and weigh as in 9.6. This is weight W_{24} .

10. Calculation

10.1 Calculate the percent plasticizer loss between the specified time intervals as follows:

$$\text{Loss } (T_A - T_B), \% = [(W_A - W_B)/(W_A - W_D)] \times 100$$

where T_A and W_A are the time and weight at the beginning of a particular period and T_B and W_B are the time and weight at the end of that heating period. The appropriate numerical subscripts are used to denote the heating interval.

NOTE 5—Generally, percent loss is calculated between 0 and 2 h, 0 and 4 h, and 0 and 24 h. This allows the integral loss curve to be drawn directly.

10.2 Calculate the plasticizer loss relative to unit area of exposed surface as follows:

$$\text{Loss, g/mm}^2 = (W_2 - W_{24})/(\pi d^2/4) = 1.273 (W_2 - W_{24})/d^2$$

10.3 Calculate the plasticizer average volatility rate as follows:

$$\begin{aligned} \text{Average volatility rate, g/mm}^2 \cdot \text{h} &= (W_2 - W_{24})/(22\pi d^2/4) \\ &= 0.0579 (W_2 - W_{24})/d_2 \end{aligned}$$

where:

- W_2 = plasticizer + dish weight after 2 h in oven, g,
- W_{24} = plasticizer + dish weight after 24 h in oven, g,
- d = internal diameter of dish, mm, and
- 22 = time interval of test, h.

11. Report

11.1 Report the following information:

- 11.1.1 Plasticizer identification,
- 11.1.2 Temperature of the test, and
- 11.1.3 Any deviations from the test and reasons for these deviations.

11.2 The report may include one or all of the following as required:

- 11.2.1 Plasticizer loss between time intervals 0 and 2 h, 0 and 4 h, and 0 and 24 h (see Note 5), or
- 11.2.2 Plasticizer loss for each specified time interval.
- 11.2.3 Linear graphical presentation of percent plasticizer loss versus time.
- 11.2.4 Average volatility rate for the period between 2 and 24 h as specified in 10.3.

12. Precision

12.1 The percent confidence limits of the average of duplicate determinations has been found to be $\pm 18\%$ of the percentage volatility reported.

13. Keywords

13.1 plasticizer; plasticizer volatility

SUMMARY OF CHANGES

Committee D-20 has identified the location of selected changes to this edition of this test method since the last issue that may impact the use of this test method.

D 2288 – 97:
Section 1.2 added.

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