Designation: D6150 – 16

Standard Test Method for Estimating Processing Losses of Plastisols and Organosols Due to Volatility[1]

This standard is issued under the fixed designation D6150; 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 describes a procedure for the determination of the relative volatility of polyvinyl chloride plastisols and organosols at elevated temperatures.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes, excluding those in tables and figures, shall not be considered as requirements of this 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 is no known ISO equivalent to this standard.

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

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

3. Terminology

3.1 *Definitions*—The terms used in this test method are in accordance with Terminology D883 and abbreviations are in accordance with Terminology D1600, unless otherwise indicated.

4. Summary of Test Method

4.1 Plastisols or organosols are weighed in aluminum dishes and heated in a circulating air oven at 177°C (350°F) for 10 min. The specimens are removed from the oven, cooled, and reweighed. The weight loss is determined and reported as either percent weight loss or weight loss per unit area of exposed surface.

5. Significance and Use

5.1 The volatile components of a plastisol or organosol influence the weight loss during processing. It is possible that this information will be useful to the producer and user and to environmental interests for estimating the volatiles emitted by the plastisol or organosol during processing.

5.2 Results obtained by this test method are not strictly equivalent to those experienced during product processing wherein conditions of temperature, air flow, coating mass, and configuration are potentially quite different.

5.3 This test method is not necessarily applicable to all types of plastisol and organosol applications. Any change in the specified testing time or temperature to accommodate unique applications shall be included in the report (see 7.3).

6. Apparatus

6.1 *Oven,* forced–ventilation laboratory oven, Type II, Grade A, with 100 to 200 air exchanges/h as specified in Specification E145.

6.1.1 It is acceptable to use a rotating turntable drive at a rate of 1 to 6.

6.1.2 It is acceptable to use a tray to fit the turntable to minimize the temperature drop in the oven.

6.2 *Aluminum Foil Dishes,* 57 mm in diameter by 18 mm high with a smooth (planar) bottom surface.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials, Section D20.15.07 on Vinyl Chloride Polymers.

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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.

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

7. Procedure

7.1 Ensure the plastisol or organosol is homogeneous. If necessary, mix the sample by hand or mechanical stirrer until homogeneous and deaerate.

7.2 Tare three aluminum dishes to the nearest 0.1 mg.

7.3 Add the plastisol or organosol specimen in such a manner as to entirely cover the bottom of the dish. The weight added to each dish shall vary by only ±0.1 g of the selected weight. If the thickness of the final product cannot be accurately determined, add 6.0 ± 0.1 g to each dish.

NOTE 2—A more representative measurement of weight loss is possible if the thickness of the sample in the aluminum dish approaches the thickness of the material during processing. The weight of the specimens added to the dish is allowed to vary according to the application.

7.4 Reweigh the dishes to the nearest 0.1 mg.

7.5 Place only three dishes from a single specimen in the oven on a shelf or turntable, or both, perpendicular to the airflow.

7.5.1 If a rotating turntable is used, place the dishes on the turntable equally spaced from the center.

7.5.2 Due to the short heating time, place the specimens into the oven as quickly as possible to minimize the temperature drop in the oven.

7.6 Heat the aluminum foil dishes containing the specimens in the forced draft oven (6.1) for 10 min ± 20 s at 177°C (350°F).

7.7 Remove the dishes from the oven, place immediately in a desiccator, cool for 15 min to ambient temperature, and weigh to 0.1 mg.

8. Calculation

8.1 Calculate the percent volatile matter, V, in the plastisol or organosol as follows:

$$V = [((W_2 - W_1) - (W_3 - W_1))/(W_2 - W_1)] \times 100 \quad (1)$$

where:

W_1 = weight of aluminum foil dish,
W_2 = weight of dish plus specimen, and
W_3 = weight of dish plus specimen after heating.

8.2 Calculate the weight loss relative to unit area of exposed surface as follows:

$$\text{weight loss, g/mm}^2 = [(W_2 - W_1) - (W_3 - W_1)]/(\pi d^2/4)$$
$$= 1.273[(W_3 - W_1) - (W_2 - W_1)]/d^2 \quad (2)$$

where:

W_1 = weight of aluminum foil dish,
W_2 = weight of dish plus specimen,
W_3 = weight of dish plus specimen after heating, and
d = external diameter of specimen after heating in mm.

9. Report

9.1 Report the following information:

9.1.1 Sample identification.

9.1.2 Average of percent volatile loss of the sample run in triplicate or the average of the volatile weight loss of the sample relative to unit area, or both, g/mm^2.

9.1.3 Any deviations from the test and the reasons for these deviations.

10. Precision and Bias

10.1 The precision of this test method is based on an intralaboratory study of ASTM D6150, Standard Test Method for Estimating Processing Losses of Plastisols and Organosols Due to Volatility, conducted in 1996. Six laboratories participated in this study, testing three specific masses (3 gm, 6 gm, and 9 gm) of thirteen different formulations. Every "test result" represents an individual determination. The laboratories reported a single test results for each material. Except for the lack of reported replicates, Practice E691 was followed for the design and analysis of the data; the details are given in an ASTM Research Report.[3]

10.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

10.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

10.1.1.2 Repeatability limits cannot be determined without replicate test results from the participating laboratories.

10.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

10.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

10.1.2.2 The average Reproducibility limit determined from all reported data is 0.019 grams.

10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.4 Any judgment in accordance with 10.1.1 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The lack of reported replicate results essentially guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the reproducibility limit as a general guide, and the associated probability of 95 % as only a rough indicator of what can be expected.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1265. Contact ASTM Customer Service at service@astm.org.

10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

10.3 The precision statement was determined through statistical examination of 234 results, from 6 laboratories, on the 13 materials described as follows:

Sample 1: 50 PHR Butyl Benzyl Phthalate formulation
Sample 2: 75 PHR Butyl Benzyl Phthalate formulation
Sample 3: 100 PHR Butyl Benzyl Phthalate formulation
Sample 4: 50 PHR Di 2-Ethyl Hexyl Phthalate formulation
Sample 5: 75 PHR Di 2-Ethyl Hexyl Phthalate formulation
Sample 6: 100 PHR Di 2-Ethyl Hexyl Phthalate formulation
Sample 7: 50 PHR Di Iso Decyl Phthalate formulation
Sample 8: 75 PHR Di Iso Decyl Phthalate formulation
Sample 9: 100 PHR Di Iso Decyl Phthalate formulation
Sample 10: 75 PHR Di 2-Ethyl Hexyl Phthalate formulation with 25 PHR CaCO3
Sample 11: 75 PHR Di 2-Ethyl Hexyl Phthalate formulation with 50 PHR CaCO3
Sample 12: 75 PHR Di 2-Ethyl Hexyl Phthalate formulation with 3 PHR mineral spirits
Sample 13: 75 PHR Di 2-Ethyl Hexyl Phthalate formulation with 6 PHR mineral spirits

11. Keywords

11.1 organosol; plastisol; poly(vinyl chloride) (PVC); volatiles

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D6150 - 15) that may impact the use of this standard. (May 1, 2016)

(1) Revised Section 10 to include reproducibility.

Committee D20 has identified the location of selected changes to this standard since the last issue (D6150 - 09) that may impact the use of this standard. (September 1, 2015)

(1) Updated subcommittee information to include "Section D20.15.07 on Vinyl Chloride Polymers."
(2) Subsection 5.1—Changed "influences" to "influence" to match plural subject.
(3) Subsection 7.1—Clarified to indicate that the plastisol/organosol is already made but needs to be checked for homogeneity for use in the method.
(4) Subsection 7.3—Moved first two sentences to a note and changed "accurate" to "representative."
(5) Subsection 7.4—Removed "should."