

Designation: D 2115 - 92 (Reapproved 1997)

# Standard Practice for Oven Heat Stability of Poly(Vinyl Chloride) Compositions<sup>1</sup>

This standard is issued under the fixed designation D 2115; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This practice lists procedures for determining the relative thermal stability of sheet or molded poly(vinyl chloride) compounds as indicated by discoloration due to exposure to an elevated temperature at controlled oven conditions.
- 1.2 A specimen preparation technique using a two-roll mill is provided for compositions that are not in molded or sheeted form
- 1.3 This practice is not intended for use in purchasing specifications because the conditions of processing plastic compounds vary widely, and the degree of correlation of data obtained by this practice to process exposure has not been determined. However, despite this limitation, this practice does yield data of value in judging the comparative service quality of poly(vinyl chloride) compositions.
- 1.4 The values stated in SI units are to be regarded as the standard. The values in parentheses are given for information only.
- 1.5 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 is similar in content, but not technically equivalent to ISO 305-1990.

#### 2. Referenced Documents

- 2.1 ASTM Standards:
- D 883 Terminology Relating to Plastics<sup>2</sup>
- D 1600 Terminology for Abbreviated Terms Relating to Plastics<sup>2</sup>
- 2.2 ISO Standard:
- ISO305–1990 Plastics—Determination of Thermal Stability of Poly(Vinyl Chloride), Related Chlorine-Containing Polymers and Copolymers and Their Compounds—

Discoloration Method<sup>3</sup>

### 3. Terminology

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

#### 4. Significance and Use

- 4.1 Poly(vinyl chloride) compositions degrade by discoloration on prolonged exposure to heat. The degree of discoloration is related to the condtion of exposure, such as length of period and temperature. When the conditions of exposure are fixed and controlled, then the relative resistance to discoloration due to heat of two or more compositions may be determined. The precision of heat stability testing is also dependent on the thickness of the specimens and the history of heat exposure prior to testing. This practice allows for control or the reporting of these variables.
- 4.2 This practice is particularly applicable for determining gross differences in the heat stabilities of poly(vinyl chloride) compositions that are detectable as a color change. It is not intended to measure absolute thermal stability. Although the observed color changes may be evidence of degradation, molecular degradation phenomena such as chain-scission or cross-linking may not be identifiable.

Note 2—While discoloration caused by exposure to elevated temperature is commonly regarded as evidence of degradation in poly(vinyl chloride) compositions, it must be borne in mind that some vinyl compositions may undergo substantial change in color without appreciable loss of mechanical strength, chemical resistance, etc., whereas others may resist change in color yet undergo substantial change in other properties. Therefore, resistance to discoloration should not be taken as sole evidence of thermal stability. It is recommended that this test be supplemented by others such as tensile strength and elongation, impact strength, and deflection temperature under load (for rigid compositions), Durometer hardness (for nonrigid compositions), and dilute solution viscosity, and that the test results before and after oven exposure be compared to determine the degree of degradation which has been produced in each of the properties measured.

4.3 This practice may be used to predict the relative discoloration in processing, provided that the compositions in

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>&</sup>lt;sup>3</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.



question are tested at the relative maximum temperatures developed in processing.

4.4 This practice is not applicable to materials that will cross-contaminate during oven exposure.

## 5. Apparatus

- 5.1 *Mill*—A two-roll laboratory plastic or rubber mill (for use for preparing specimens from the basic ingredients or from molding or other compounds). Provision shall be made for heating the rolls to the desired temperature and for maintaining this temperature constant over the surfaces of both rolls within  $\pm 3^{\circ}$ C.
- 5.2 *Oven*—The oven shall be of the forced-air type and shall have the following additional requirements:
- 5.2.1 The oven temperature shall be controlled by an accurate, reliable thermoregulator maintaining the set point within  $\pm 0.5$ °C or better.
- 5.2.2 The oven shall be equipped with a calibrated ASTM thermometer, and the proper stem correction shall be applied to the temperature measurement.
- 5.2.3 The oven shall be maintained at the temperature of test for at least 1 h prior to insertion of the specimens.
- 5.2.4 The air distribution in the oven shall be adjusted to provide a temperature uniform throughout the test area of the oven to an extent sufficient to produce specimens of uniform color when checked in accordance with 5.2.5. This will usually require an air flow of not less than 0.283 m<sup>3</sup>/min (10 ft<sup>3</sup>/min).
- 5.2.5 Prior to test operation, the uniformity of temperature within the oven shall be verified. This may be done by inserting, at the test temperature, a rack of eight to ten specimens of the same stock distributed over the area to be used in the test and exposed until an early stage of discoloration is reached. The formulation (Note 3) should be such as to cause a sharp change in color within 45 to 60 min under test conditions. Lack of uniform temperature distribution, as indicated by differences in the color of the exposed specimens, shall be corrected before proceeding with the tests.
- Note 3—A suitable formulation for this purpose comprises poly(vinyl chloride) homopolymer resin 100, dioctyl phthalate 50, barium stearate 1.2, cadmium stearate 0.8 (parts by weight).
- 5.2.6 The inside of the oven shall be free of contamination or surface deposits. Stainless steel oven liners should be used to reduce corrosion due to continued exposure to decomposition gases.
- 5.3 *Specimen Supports*—The specimens shall be supported by new, clean aluminum foil placed on removable oven racks.
- Note 4—If an oven equipped with rotating specimen-holding devices is employed, the specimen must be supported so that there is no appreciable elongating or necking down during the test period to alter the specimen dimensions, especially the thickness. The uniformity of oven condition should be checked in a manner similar to 5.2.5, using the specimen mounting facility provided on the oven-rotating device.

## 6. Test Specimens

- 6.1 Test specimens shall be prepared by compounding on a laboratory two-roll mill as follows:
- Note 5—If the sample has been precompounded, omit 6.1.2 and 6.1.3. Specimens in sheeted form shall be cut to size according to 6.1.5.
  - 6.1.1 The batch size shall be such that a rolling bank 6.35 to

12.7 mm ( $\frac{1}{4}$  to  $\frac{1}{2}$  in.) in diameter is obtained across the face of the mill with the rolls set to give a sheet  $0.816 \pm 0.08$  mm ( $0.040 \pm 0.004$  in.) thick.

Note 6—Other specimen thicknesses may be used. However, heat stability is affected by thickness, and all comparative tests shall be made on specimens of the same nominal thickness.

- 6.1.2 Weigh all components to an accuracy of  $\pm$  1 % of their nominal weight.
- 6.1.3 Premix the solid and liquid components separately in suitable containers until homogeneous in appearance. Add dry components to the container holding the liquid components, then blend together until homogeneous.
- 6.1.4 Charge the batch or composition to the mill rolls which have been preheated to a temperature suitable for complete fluxing of the compositions. A temperature of 149  $\pm$  3°C (300  $\pm$  5.4°F) is suitable for most poly(vinyl chloride) compounds.
- 6.1.5 Adjust the rolls to give a  $0.816 \pm 0.08$ -mm (0.040  $\pm$  0.004-in.) thick sheet after banding. Cut and mix the compound continuously for 5 min after banding; then cut from the rolls.

Note 7—When this practice is used to discern differences in stability effects of compounding ingredients, that is, resin, stabilizers, etc., it is important to maintain consistent conditions of temperature and time in the preparation of the specimens.

6.1.6 Cut the sheet into specimens not less than 25.4 by 25.4 mm (1 by 1 in.).

#### 7. Procedure

7.1 Prepare a specimen support of sufficient size to hold one specimen of each of the different compositions under test, for each time interval of test.

Note 8—As an example, suitable exposure times at  $177 \pm 0.5^{\circ}$ C ( $350\pm 0.9^{\circ}$ F) for the composition of Note 3 might be 30, 45, 60, 90, 120 min, in which case five pieces of aluminum foil would be cut such that each is large enough to hold one specimen of this and each other composition to be tested but not too large to prevent placing all the foils on the oven rack at one time.

- 7.2 Place one specimen of each composition on each of the foil supports.
  - 7.3 Place all of the foils on a removable oven rack.
- 7.4 Place the rack in the oven at the test temperature, keeping the oven door open for the minimum time and extent necessary. The oven air-circulating fan shall be off while the door is open. Use an oven temperature of 177  $\pm$  1.0°C (350  $\pm$  1.8°F) if the temperature of test is not specified.
- Note 9—The temperature of the oven must be carefully controlled to the requirements of this specification if test reproducibility is to be attained. The rate of decomposition for most poly(vinyl chloride) compounds will double for each  $10^{\circ}\text{C}$  rise in temperature. Temperature variations of  $1^{\circ}\text{C}$  from test to test may alter decomposition values by 10~%.
- 7.5 Remove one foil of specimens at selected periodic intervals over an exposure range to discoloration, preferably blackening (Note 8). These specimens may be mounted on a chart for examination to estimate relative discoloration or stability. Do not trim the specimens after exposure.

#### 8. Report

8.1 Report the following information:



- 8.1.1 Composition or compound identification,
- 8.1.2 Mill roll temperature and time to banding or the conditions of temperature and time employed in sample preparation if the specimen was heat fused, molded, extruded or laminated.
  - 8.1.3 Test temperature,
  - 8.1.4 Exposure times,
  - 8.1.5 Relative heat stabilities based on time of exposure and

degree of discoloration as compared to standard sample or control, and

8.1.6 Observed changes in the test specimens that may be due to heat exposure, that is, bubbles, blisters, embrittlement, spotting, etc.

## 9. Keywords

9.1 heat stability; poly(vinyl chloride)

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