

Designation: D 5814 - 9502

# Standard Practice for Determination of Contamination in Recycled Poly(Ethylene Terephthalate) (PET) Flakes and Chips Using a Plaque Test<sup>1</sup>

This standard is issued under the fixed designation D 5814; 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 covers an indication of the quality of recycled transparent poly(ethylene terephthalate) by examination of a wafer or plaque formed by melting a representative sample and quenching it to prevent crystallization.
- 1.2 Specific contaminants and impurities such as aluminum particles, dirt particles, paper, and fibers are identified in the transparent wafer.
- 1.3 The overall color of the plaque is indicative of oxidizable contaminants such as ethylene-vinyl acetate (EVA) glue residue and the number of bubbles present in the plaque gives an indication of the moisture content of the sample.
- 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. Specific precautionary statements are given in Section 8.

Note 1-There is no equivalent ISO standard.

### 2. Referenced Documents

2.1 ASTM Standards:

<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee D=20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods. Current edition approved—Oct. March 10, 1995. 2002. Published—December 1995. May 2002. Originally published as D 5814 – 95. Last previous edition D 5814 – 95.



D 1600 Terminology for Abbreviated Terms Relating to Plastics<sup>2</sup>

D 5033 Guide for the Development of <u>ASTM</u> Standards Relating to the <u>Proper Recycling and Use of Recycled Plastics<sup>2</sup> E 380 Practice</u>

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System<sup>3</sup>

# 3. Terminology

- 3.1 *Definitions:* The terminology used in this practice is in accordance with Terminology D 1600 and Guide D 5033. Units and symbols are in accordance with Practice E 380. IEEE/ASTM SI 10: Standard for Use of the International System of Units (SI).
  - 3.2 Definitions of Terms Specific to This Standard:
  - 3.2.1 plaque—a transparent wafer resulting from melting and rapid quenching of the polymer.

### 4. Summary of Test Method

4.1 Transparent poly(ethylene terephthalate) flakes are melted in an aluminum pan, then the molten sample is rapidly quenched in ice water to prevent crystallization. The resulting clear plaque is examined for color indicative of oxidizable contaminants, bubbles indicative of moisture, and solid contaminant particles.

# 5. Significance and Use

5.1 Presence of paper, metal, or incompatible polymer contamination in poly(ethylene terephthalate) renders the recycled polymer unfit for use in secondary product manufacturing operations. This procedure is useful for identifying different types of contamination in recycled PET flakes.

### 6. Apparatus

- 6.1 Oven, forced convection type capable of heating samples to 300°C.
- 6.2 Tongs, long arm.
- 6.3 Stopwatch, 0.1-s accuracy.
- 6.4 Bucket, 1 L, stainless steel.
- 6.5 Aluminum Weighing Dishes. 4
- 6.6 Thermally Insulated Gloves.

# 7. Materials

- 7.1 Virgin Poly(ethylene terephthalate).
- 7.2 *Ice*.
- 7.3 Paper Towels.

# 8. Hazards

8.1 Always wear thermally insulated gloves when introducing or removing the polymer sample from the oven.

### 9. Procedure

9.1 Preheat the oven to  $275 \pm 5^{\circ}$ C and equilibrate for 30 min.

Note 2—Accuracy of both time and temperature is critical to valid sample-to-sample comparisons using this test.

- 9.2 Obtain three aluminum pans and label them "A," "B," and "C." Weigh  $5.5 \pm 0.1$  g of sample into each of the pans. Tap each sample pan gently on a flat surface to evenly spread the chips.
  - 9.3 Weigh 5.5  $\pm$  0.1 g of virgin PET into another pan.
- 9.4 Quickly open the oven door, place the pans in the center of the oven, and close the oven door to minimize heat loss. It is important that this step take place as quickly, efficiently, and reproducibly as possible.
- 9.5 Start the stopwatch as soon as the oven temperature recovers and stabilizes at 275°C. After 2 min, record the oven temperature (that should be 275°C).
- 9.6 Prepare an ice water bath by filling the 1-L bucket with cold tap water. Add ice and decant off some of the water to maintain a 5-cm floating ice level on the surface of the water. The bucket should be about 90 % full with the ice and water slurry. Place the bucket with the ice slurry as close to the oven as possible.
- 9.7 At 10.00 min on the stopwatch, quickly remove the samples from the oven with the tongs and immediately submerge them into the ice bath. This rapid quenching prevents crystallization of the PET.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.03.

<sup>&</sup>lt;sup>3</sup> Available from ASTM-<del>Standards, Vol 14.02.</del> International, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.

<sup>&</sup>lt;sup>4</sup> Aluminum weighing dishes, available from Fisher Scientific, or exact equivalents, have been found suitable for this purpose.



- 9.8 Remove the pans from the ice bath and blot dry with paper towels. Remove the hardened plastic disks formed in the oven (plaques) from the aluminum pans by peeling the pans from the plaques. Label each plaque with the "A," "B," or "C" designation earlier assigned to each sample.
- 9.9 Place the plaques on a white background. Compare the sample and virgin PET plaques with respect to color, number and description of particles, and the number of bubbles.

# 10. Report

- 10.1 Report the following information:
- 10.1.1 The sample plaque color,
- 10.1.2 The number and description of particles in the sample plaques, and
- 10.1.3 The number of bubbles in the sample plaques.

## 11. Keywords

11.1 contamination; plaque test; poly(ethylene terephthalate); recycled plastic

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