The Association of Postconsumer Plastic Recyclers

# Flake to Plaque Bleeding Label Test

## PET-B-01

APR recognizes that packaging innovation drives the growth of bottles available for recycling and growth of supply of bottles is essential to the well being of the plastic bottle recycling industry. APR also recognizes that some innovations may create bottles that present technical challenges for recycling.

This document is a preliminary screening procedure to help innovators determine potential difficulties in plastic and/or paper labels, closures, and attachments to be used for PET bottles. In particular, sleeve labels proposed for PET bottles should be considered for the Bleeding Label. Experience has shown some sleeve labels can be technically problematic.

The intent is to confirm that a decorated plastic and/or plastics label does not 'bleed' ink such as to discolor the PET flake or subsequent moldings. Bleeding labels have been recognized as a recycling problem for many years as water-dissolving inks can degrade the quality of recycled PET flake. Non-floating labels that are not removed elsewhere in the recycling process will mean the pigments on the label flakes will discolor the PET product.

**This test does not replace the more thorough Critical Guidance testing,** but should be considered a preliminary screening for color effects. The Bleeding Label applies to decorated labels.

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## Note to the Reader

THIS GUIDANCE HAS BEEN PREPARED AS A SERVICE TO THE PLASTICS PACKAGING INDUSTRY TO PROMOTE THE MOST EFFICIENT USE OF THE NATION'S PLASTICS RECYCLING INFRASTRUCTURE AND TO ENHANCE THE QUALITY AND QUANTITY OF RECYCLED POSTCONSUMER PLASTICS. THE INFORMATON CONTAINED HEREIN REFLECTS THE INPUT OF APR MEMBERS FROM A DIVERSE CROSS-SECTION OF THE PLASTICS RECYCLING INDUSTRY, INCLUDING PROFESSIONALS EXPERIENCED IN THE RECYCLING OF THE POSTCONUMER PLASTIC BOTTLES DISCUSSED IN THIS GUIDANCE. IT OFFERS VALUABLE INSIGHT ON HOW LABEL DESIGN IMPACTS CONVENTIONAL PLASTICS RECYCLING SYSTEMS AND PROVIDES USEFUL RECOMMENDATIONS FOR UNDERSTANDING HOW TO MAKE THEM COMPATABLE WITH CURRENT RECYCLING SYSTEMS.

BECAUSE NEW TECHNOLOGY DEVELOPMENTS ARE ALWAYS BEING MADE, THIS GUIDANCE CANNOT ANTICIPATE HOW THESE NEW DEVELOPMENTS MIGHT IMPACT PLASTIC BOTTLE RECYCLING. WHILE THE INFORMAITON IN THIS GUIDANCE IS OFFERED IN GOOD FAITH BY APR AS AN ACCURATE AND RELIABLE DISCUSSION OF THE CURRENT CHALLENGES FACED BY THE PLASTICS RECYCLING INDUSTRY, IT IS OFFERED WITHOUT WARANTY OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING **WARRANTIES OF MERCHANTABILITYOR FITNESS FOR A PARTICULAR PURPOSE, WHICH ARE EXPRESSLY DISCLAIMED.**

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**Bleeding Labels Flake to Plaque**

The following is from APR's ***Protocol for Producing PET Flake and Plaques for Evaluation and Evaluating for Discoloration from "Bleeding Labels"***. Labels are divided into 3 groups for evaluation: wrap-around, adhered and sleeve labels. Labels should be fully decorated with all intended colors and colorant quantity.

*The following protocol is designed to provide a generic wash process for evaluation of the effects of "bleeding labels" on recycled PET material. A "bleeding label" is one with water dispersible or soluble inks that result in discolored wash water and stained PET flake*

> ***This protocol does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.***

***MATERIALS***

**Sample A**

**Unlabeled PET Bottle** (hereinafter referred to as the "Intended Test Bottle" for the specific sleeve label that is the subject of the testing)

- Bottles should be made by using an APR PET control resin on the list below if possible.

| *Low IV, Water Bottle Innovation Controls* | *CSD and Non-Water Bottle Innovation Controls* |
|---|---|
| Auriga Polyclear® Splash 3301 | Auriga Polyclear® Refresh 1101 |
| M&G Cleartuf® Turbo II | M&G Cleartuf® MAX |
| DAK Laser+® W L40A | DAK Laser+® B90A |

- If an APR control resin is not used, the PET resin used to make these bottles should meet the PET QUICK TEST FOR COLOR:
  - After two heat histories, namely preform into bottle and forming into 3 mm plaques, the resin used should have a transmission CIELAB L* greater than 82

and should exhibit a b* less than 3 units greater than a plaque made from a named Control Resin also with two melt histories.

- If the resin used to make the bottle is not an APR Control Resin and does not meet the conditions listed above, please discuss the situation with APR.
- Because this testing is designed to evaluate the labels rather than the PET resin, accommodation is possible for non-conforming PET resins.

- The same resin must be used for all bottles used in this evaluation.

**Sample B**

**Note:** The following describes how "generic" labels should be evaluated to obtain insight on how they may affect the recyclability of PET bottles. Actual labels that are decorated and sized to fit a particular bottle and are intended to be sold in the market on that specific bottle, should be evaluated with the exact label weight or surface area for the bottle they will be attached to. These label/bottle combinations are referred in the APR Critical Guidance Documents as "Intended" packages. Otherwise the "generic" studies outlined below should be followed for all label evaluations.

**Wrap-Around Labels** are defined as labels where the leading edge is adhered minimally to the bottle then wrapped with the tailing edge adhered to the leading edge. These will normally constitute about 3% or less of the package weight. After processing at a whole bottle wash facility including steps such as dry grinding and air elutriation, it has been determined that there will be a 60% reduction of label in the processed PET flake. This reduces the label content in the ground flake from 3% to 1.2%. The APR studies all Innovations (including labels) at a maximum level in the recycle stream at 50%. This maximum study level at 50% is achieved by blending 50% of the unlabeled control bottle flake with 50% of the wrap-labeled test bottle flake, thus reducing the concentration from 1.2% to 0.6%. Therefore wrap-around labels will be evaluated at a study level of 0.6 wgt-%.

**Sleeve Labels** are defined as labels that cover a bottle from neck to base and adhere to the bottle without the use of adhesives, although adhesives may be used to adher the leading edge to the trailing edge of the label. They are then tightly heat-shrunk onto the contoured shape of a bottle. These sleeve labels on average make up approximately 6% by weight of the bottle. Whole bottle washing, dry grinding, and air elutriation are not effective in removing significant levels of shrink-wrapped labels. As noted for wrap-around labels, shrink labels will also be evaluated at a maximum level of 50%. This can be achieved in two ways, diluting the ground bottle shrink wrap flake at 50% with unlabeled control flake, or by using a shrink label on a bottle that makes up 3% by weight of the bottle. In either case,

sleeve labels will be evaluated at a study level of 3% by weight.

**Adhered Labels** are defined as labels that are attached over the entire contact surface of the label to the bottle using adhesives. These labels typically cover 40% of the surface area of the bottle. Again using the 50% maximum study level, adhered labels will be evaluated at 20% surface area coverage.

The following terms will be used below to describe the test samples:

- Sample A is the control material consisting of PET flake without label material
- Sample B is the test material of PET flake with label material present.

## Equipment/Supplies List

- Test Labels
  - Wrap-around, 0.6% by weight per test
  - Sleeve/Stretch, 3% by weight per test
  - Adhered, 20% surface area per test
- PET Flake
  - Ground flake about 1 cm (3/8 inch) nominal in size
  - Quantity determined by the number of tests to be run and batch sizes required for each test
- Glass Beakers sized appropriately for the batch size
  - Example: 2000 ml beaker to prepare a 1000 ml caustic wash solution.
- Stainless Steel baffled beaker preferred for washing sized appropriately for batch size
  - Example: 6 inch diameter by nine inches in height with four 3/4 inch baffles for a 250g flake wash.
- Hot plate capable of heating to $100^0$C
- Scale or balance capable of measuring 500 (+\- 0.5*)* grams
- Overhead stirrer capable of 1000 rpm
- Stirring impeller - 3 blades, pitched, and 3.0 inch diameter/or sized appropriate for batch vessel
- Colorimeter/spectrophotometer
- Thermometer
- Strainer - non-aluminum, fine mesh
- Graduated cylinder, 1000 ml
- Triton X- 100 nonionic surfactant

- Caustic ( NaOH)
- Desiccant Dryer
- Injection Molding Unit
- 3mm Thick plaque mold

*** Be sure to read all material safety data sheets.**

**PET Sample Wash Procedure**

***Be sure to use appropriate laboratory safety procedures / Gloves, safety glasses, etc.***

1. Prepare Sample A – Unlabeled Control PET Flake
2. Prepare Sample B – Test Flake (500 grams). Cut up labels into 1.0 cm (3/8 inch) squares and Sample with PET as required.
   i. 0.6% Wrap (500 gram weight %)
   ii. 3% Sleeve (500 gram weight %)
   iii. 20% adhered area

3. Prepare a wash solution of 0.3% by weight Triton X-l00 (6.0 gms or 5.7 m1 per 2,000 ml water) and 1.0% by weight caustic (20 gms NaOH per 2,000 ml water).

   **Note:** Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of caustic.

4. Wash each Bottle Flake Sample separately at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated (1000rpm) water at 88 ± 2 °C (190°F) for 15 minutes. After 15 minutes of washing, stop agitation and remove agitator. Stop heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables.

5. Separate sinking solids from wash solution by pouring mixture through a strainer. Add sinking solids to room temperature rinse water at an approximate ratio of 500 grams sinking solids to 2 liters of water. Let stand for five minutes to allow remaining lights to float to the surface.

6. Repeat sink/float once again until all visual floatable labels have been removed.

7. Transfer PET flakes to strainer, rinse flakes in cold running tap water while vigorously stirring the flakes for 10 minutes using the manual stirring bar. Drain the material.

8. Air dry flake.

9. Examine without magnification from a distance of 12 inches using illumination typical for reading and remove all visual labels from test sample.

**Evaluation Flake**

***The protocol does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.***

1. Utilities the samples of clear test PET flake washed without the presence of labels (Sample A) and sample of test PET flake washed with labels present according to this procedure (Sample B). Remove all label flakes from the PET flake sample containing label material before making color measurements.
2. Perform color analysis, according to the machine manufacturer's instructions, on each sample produced (test of "bleeding label" test PET flake and control PET flake) using the following format:
   a. Take five measurements, in reflectance or transmission, on each sample. Record data as X, Y, Z tristimulus values, CIE XYZ, CIE L*a*b*or Hunter **L a b** color coordinates, or equivalent. Adjust the position of the sample holder prior to each measurement to expose different sample areas to measurement.
   b. Report all of the axis readings (such as **L, a, b**) for all five samples and the average for each sample.
3. Evaluation guidance. All three color measurements, **L**, **a**, and **b**, are important to the possible use of the recyclate. The human eye can discern about one **b** unit change. The **L** measures brightness vs. dinginess of the flake. **a** measures red/green color components. **b** measures yellow/blue color components.

   Depending on the end use, different amounts of variation from the control can be accepted. Generally, changes of more than two **a** or **b** units or five **L** units are excessive. Absolute **b** values over 3 for flakes measured in reflection may be unacceptable for reuse in bottles.

Comparisons should be made between control and test samples for the same treatment.

4. Discolored water should be evaluated to see if the discoloration is acceptable for release to municipal sewer systems without further treatment. Strongly discolored water is likely to be unacceptable to many municipal wastewater treatment systems and labels which cause such discoloration should be avoided.

**Injection Molding**

1. From the washed Sample A and B prepare of injection molding.
2. Sample B needs to be carefully examined and any visible pieces of label flake removed. This test is for only the ink and adhered residue, if any, on the flakes this is **Sample B**.
3. Desiccant dry Samples A and B separately at least 4 hours at 320 ± 20°F (160 ± 12°C) to achieve moisture below 50 ppm.
4. Injection mold a minimum of 25 3 mm thick plaques from **Sample A**, control, first. Then mold a minimum of 25 3 mm thick plaques from **Sample B** under identical conditions if possible. If the processing conditions need to be changed, document and report the changes.
5. Randomly select 5 plaques from each sample for color and haze measurement

**Evaluation Guidance**

1. Color and Haze Measurements. Measure CIELAB in transmission on 5 randomly selected plaques for each Sample. Average results.

| |
|---|
| L* >82 for all Samples |
| Δ b* & Δ a* of Test Sample vs. Control Sample guidance: < 1.5 |
| Haze of Control Sample guidance: < 9.5% Haze |
| Haze of Test Sample guidance: < 20% Haze |

Note: Color Measurement

1. Measure color in transmission for color and haze using 3 mm amorphous plaques.
2. Calibrate spectrophotometer to the manufacturer's recommendations.

3. Measurements should be made with Hunter Miniscan XE or equivalent using d65 light in transmission. The reported number should be the average of at least five color measurements of CIELAB on at least five plaques.

**Black speck and/or Inclusions**

1. Black Specks and Inclusions. Black specks will be formed predominantly from non-PET materials that will thermally decompose during extrusion and injection molding under PET's high melt-temperature processing conditions. Inclusions are non-meltable components of the PET melt stream and can by definition include black specks. Both black specks and inclusions negatively affect the visual appearance of a bottle and can contribute to burst failures during blow molding and later in pressurized bottles. This benchmark test does not include melt filtration to remove such non-meltable impurities, therefore the presence of black specks and inclusions in molded plaques should be considered a warning that the label and or adhesive may be the cause and as such would require additional study.

   Note: Prior to injection molding, visually examine each sample for evidence of remaining label material. Remaining label and adhesives can contribute to black specks and inclusions. While there is no definitive level of label or adhering adhesive that can be considered acceptable, amounts greater than 200ppm may be very problematic in achieving acceptable molded plaques.

2. Twenty-five (25) plaques are to be molded from both of the Samples and viewed without magnification from 12 inches away. Count any plaque that contains a black speck or inclusion greater than 0.015 inches.

3. Record visual observations and summarize in the event black specs and/or inclusions are present.