The Association of Postconsumer Plastic Recyclers

Flake to Plaque Thermoform Label Test
PET-B-02

The following protocol is designed to provide guidance on the compatibility of PET thermoform construction (including label and adhesive) with PET recycling. Further testing will be needed to confirm recycling of PET thermoforms with PET bottles.

The PET resins used for thermoforms are taken to be chemically similar to the PET resins used for bottle making and needs limited examination. It is recognized the intrinsic viscosity of thermoform PET is generally lower than the intrinsic viscosity of carbonated soft drink bottle PET, but can be nearly the same as for water bottle PET.

Labeled PET commercial thermoforms are compared to unlabeled PET commercial thermoforms without postconsumer recycled content.

A generic wash process is described for evaluation of the effects of thermoform components (adhesives, labels, etc.) on recycled PET material.

The Appendix A describes testing for the effect of label ink decoration and printing.

Note: Unless a thermoform is pre-labeled, the label is expected to typically cover about 6% of the surface of the thermoform. Test evaluation criteria are based on using thermoforms with approximately 140 square inches of external surface and a minimum of 8 square inches of affixed label. If a subject label exceeds 8 square inches, it should be examined at its complete size. At this time we do not see normalizing results as useful.

Note: The base PET resin used for thermoforms should when formed into 3 thick mm plaques from flakes without any additives, surface coatings, or lubricants exhibit a b* less than 7 and % haze less than 18% when viewed in transmission.

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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Recommended Test Protocol

1. Materials:

a. Approximately 14 pounds of each labeled thermoform sample(s) will be needed for this study.

b. Approximately 2 to 4 pounds of each sample is to be removed, and the control PET taken from these thermoforms by physically cutting away and discarding the labeled area. Only the unlabeled area of these thermoforms will be used. Grind the label-free material, approximately 2 to 4 pounds, of the unlabelled thermoform material to nominal ¼" to ½" size flake. This is the **CONTROL MATERIAL.**

c. Estimate the approximate weight ratio of label material to PET thermoform material. Record as "Initial weight % label on thermoforms".

d. Grind approximately10-12 lb of the labeled thermoforms to nominal ¼" to ½" size flake. This is the **TEST MATERIAL**. Initially, it will have label material included with PET flakes.

e. Preferred - Baffled beaker - stainless steel, six inch diameter by nine inches in height with four ¾ inch baffles.

   a. Alternate - similar size beaker or stainless steel container without baffles.

f. Hot plate capable of heating to 190^0C.

g. Scale or balance capable of measuring 500 (+\- 0.5*)* grams.

h. Oven capable of drying flake at 40^0C.

i. Cookie sheet (or other suitable tray with a minimum area of 120 sq.in.).

j. Cover for beaker which can accommodate thermometer and stirring impeller rod.

k. Overhead stirrer capable of 1,000 RPM.

l. Stirring impeller - pitched, 3 blades, and 3.0 inch diameter.

m. Manual stirring rod.

n. Thermometer.

o. Strainer - non-aluminum.

p. Distilled or deionized water.

q. Graduated cylinder.

r. Triton X- 100 nonionic surfactant (available from Union Carbide at 1-800-969-2707).
   a. Be sure to read all material safety data sheets.

s. Caustic (granular NaOH).
   a. Be sure to read all material safety data sheets.

t. Desiccant dryer, capable of achieving air with -40°C dew point.

u. Injection molding machine with molds 3 mm thick and of size for color measurement.

v. Color measurement device.

2. PET Sample Wash Procedure -- CONTROL MATERIAL WASH

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

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

2. Mix **CONTROL MATERIAL** flake at a ratio of 500 grams solids per 2500 ml wash solution. Wash in highly agitated (1,000 rpm with impeller 0.6 cm from bottom of wash container) water at 88+/-2°C for 15 minutes. Record composition of the wash solution.

3. After 15 minutes of washing, stop agitation and remove agitator. Remove heating source. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables. 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 2.5 liters of water. Let stand for five minutes to allow remaining lights to float to the surface. Repeat sink/float step once again.

4. 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. Air dry the flake or dry at 40°C.

5. Repeat until the nominal 4 pounds (~1.8 kg) of **CONTROL MATERIAL** flake are washed. Blend batches for a uniform **CONTROL MATERIAL** flake sample.

3. PET Sample Wash Procedure -- TEST MATERIAL WASH

1. Set up and adjust elutriation facility using about 2-3 lb of **CONTROL MATERIAL** blend flake to achieve a 2% or less loss rate of PET flakes. Note settings. Recombine and save the **CONTROL MATERIAL** flake sample**.**

2. Elutriate the remaining 10 lb. of ground, unwashed **TEST MATERIAL** flake using the settings from Step 1 for **CONTROL MATERIAL WASH** flake. This is **TEST MATERIAL FIRST ELUTRIATION** sample.

3. Prepare a wash solution of 0.3% by weight Triton X-l00 (6.0 grams or 5.7 m1 per 2,000 ml water) and 1.0% by weight caustic (20 grams 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. Mix elutriated **TEST MATERIAL** flake at a ratio of 500 grams solids per 2,500 ml wash solution. Wash in highly agitated (1,000 rpm with impeller 0.6 cm from bottom of wash container) water at 88+/-2°C for 15 minutes. Record composition of the wash solution.

5. After 15 minutes of washing, stop agitation and remove agitator. Remove heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables. 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 2.5 liters of water. Let stand for five minutes to allow remaining lights to float to the surface. Repeat sink/float step once again.

6. 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. Air dry flake or dry at 40°C.

7. Repeat until the nominal 10 pounds (4.5 kg) of **TEST MATERIAL** flake are washed. Blend batches for a uniform **TEST MATERIAL** flake sample.

4. PET Evaluation

A. Color Measurement for Washed CONTROL MATERIAL Flake and Washed TEST MATERIAL Flake

1. Perform color analysis, according to the machine manufacturers instructions, on each sample produced (washed CONTROL MATERIAL flake and washed TEST MATERIAL flake) using the following format:

a. Take five measurements, in reflectance, 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. Record separately for **Control Material** and **Test Material.**

c. For either undecorated or printed/decorated label being evaluated, measure both the Control and the Test Material wash water for discoloration.

B. APR Fluorescence Measurement

For flake samples of Test Material and Control Material no substantial difference of emission fluorescence intensity for excitation or absorption wavelength between 320 and 390 nm. Fluorescence intensity depends on many variables. Substantial difference is more than a 50% difference in area under the curve for fluorescence intensity vs. wave length for tests conducted under identical conditions. Photographic evidence showing little visible difference for black light illumination is sufficient.

C. CONTROL MATERIAL Plaque Evaluation

Note: At this stage of the evaluation, it is permissible to re-grind both the Control and Test Material flake to a smaller flake size that allows for uniform extrusion operations. Too large a flake for the extruder feed throat can result in inconsistent feed and ultimately to overheating and yellowing of material. Be sure to include all ground material.

1. Air dry or 40°C dry and blend **CONTROL MATERIAL** washed flakes.

2. Set up elutriation using about 3 lb of washed and air dried **CONTROL**

MATERIAL flake to achieve a 2% or less loss rate of PET flakes. Note settings. This is **CONTROL MATERIAL SECOND ELUTRIATION** sample.

3. Desiccant hot air dry **CONTROL MATERIAL** flakes at 320+/- 40°F for a minimum of 4 hr to less than 50 ppm moisture. Overheating some PET resins in drying can cause unwanted yellowing.

4. Injection mold 25 3mm plaques from nominal 4 lb of washed **CONTROL MATERIAL** flake.
    i. Select 6 representative plaques.
        1. Measure L*a*b* and Haze by transmission
        2. Perform APR fluorescence measurement.
    ii. Report any unusual effects seen during injection molding, such as fuming.

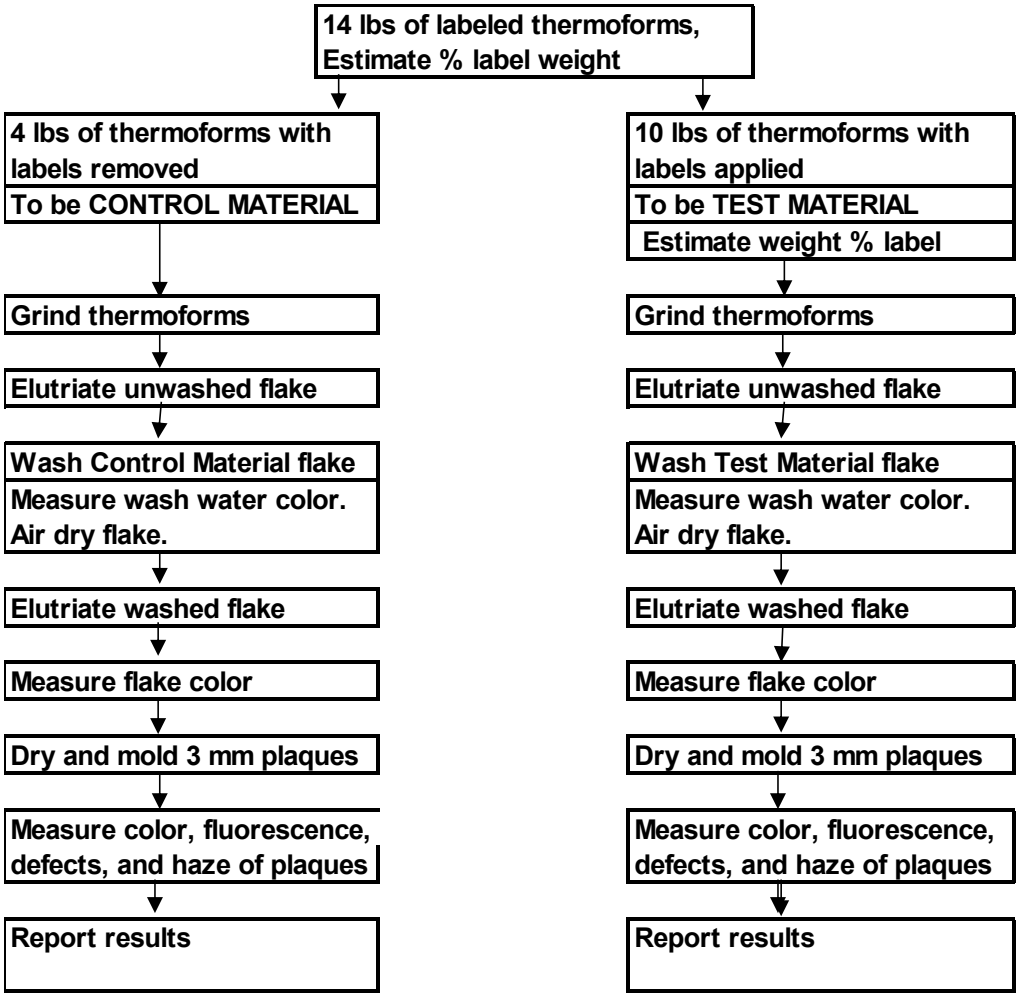
D. TEST MATERIAL Plaque Evaluation

1. Air dry or 40°C and blend **TEST MATERIAL** washed flakes.

2. Elutriate 10 lb of the washed **TEST MATERIAL** flake under same conditions identified from Step 2, **CONTROL MATERIAL Plaque Evaluation**. This is **TEST MATERIAL SECOND ELUTRIATION** sample**.**

3. Desiccant hot air dry the washed **TEST MATERIAL** flake at 320+/-40°F for a minimum of 4 hr to less than 50 ppm moisture. Overheating some PET resins in drying can cause unwanted yellowing.

4. Injection mold 3mm plaques from about 8 lb of washed **TEST MATERIAL** flake
    a. Label plaques in order of molding
        i. Number 1 through 25
    b. Select 6 representative plaques
        i. Measure L*a*b* and Haze by transmission
        ii. Perform APR fluorescence test
    b. Report any unusual effects seen during injection molding such as fuming or evidence of particles or black specks from any residual label present or evidence of haze variation seen from any residual label present.



5. Data Reporting

A report spreadsheet or tabular report format will be developed. The data reported will consist of:

- Initial weight % label on thermoforms, without treatment.
- L*a*b* of washed **CONTROL MATERIAL** flake after air drying and blending.
- L*a*b* of washed **TEST MATERIAL** flake after air drying and blending.
- Discoloration of wash water for **CONTROL** and **TEST MATERIAL** flake wash
- Average of 5 measurements of L*a*b* and haze on 6 representative plaques from both the **CONTROL MATERIAL** and the **TEST MATERIAL** samples
- Fluorescence observations

- Report on unusual coloration or haze in entire set of molded plaques including occurrence of black specks over 15 mils. For 25 plaques each of **CONTROL MATERIAL** and **TEST MATERIAL**, note number of plaques containing defects exceeding 15 mils.

6. Evaluation of Results

The results for the color and haze measurements need to be comparable to what is acceptable for recycled PET bottles. Until such time as different guidance is developed, the measure of acceptability is taken to be what is appropriate per existing APR testing documents.

Flake b* - Absolute b* values must be under 3 for air dried flakes measured in reflection for CONTROL MATERIAL and TEST MATERIAL

Plaque L* - Must be greater than 82 for both CONTROL MATERIAL and TEST MATERIAL

Plaque b* - The CONTROL MATERIAL plaque b* must be below 7.0. A Δb* over 3.0 measured in transmission is unsuitable, comparing CONTROL MATERIAL and TEST MATERIAL

Plaque haze - The CONTROL MATERIAL plaque must exhibit a % haze less than 18%. Δ% haze values of TEST MATERIAL over CONTROL MATERIAL greater than 10% are unacceptable

Fluorescence - No increase in fluorescence from TEST MATERIAL plaque compared to CONTROL MATERIAL plaque. No substantial fluorescence in CONTROL MATERIAL (no optical brighteners added to PET resin)

Defects - An allowable number of TEST MATERIAL defects based on the number of defects seen for CONTROL MATERIAL

RESULTS REPORT

Thermoform Sample	**Adhesive used** _____ **Label used** _____ **Thermoform used** _____
Investigator	
Date	

Data Subject	**Control Material**	**Test Material**	**Evaluation Target**
Initial % label on thermoforms	XXXXXXXXX		XXXXXXXXXXXXX
L* color, Washed Flake			
a* color, Washed Flake			
B* color, Washed Flake			b* less than 3.0
Wash water discoloration			Minimal discoloration
L* color, 3 mm Plaques			L* greater than 82
a* color, 3 mm Plaques			
B* color, 3 mm Plaques			Control Material b* less than 7.0. Δb* less than 3.0
Haze, Plaques			Control material % haze less than 18%. Δ haze ≤10%
Fluorescence, Plaques			Negligible, both samples
Specks and other unusual observations			For 25 plaques each of Control and Test Materials, with failure defined as any plaque with specks exceeding 15 mils, the allowable is defined below

ACCEPTABLE LEVEL OF DEFECTS IN TEST MATERIAL PLAQUES 25 plaques tested												
For CONTROL MATERIAL with defects over 15 mils	0	1	2	3	4	5	6	7	8	9	10	11
Number of TEST MATERIAL defects over 15 mils	3	5	7	8	10	11	12	13	14	15	16	17
Unpaired t test, 0.05 statistical significance												

APPENDIX A

Suitability of Label Decoration and Printing

It is harmful to recycling for a label and adhesive to be the source of technical interference with the recycling process. The inks or colorants used should also be compatible with the recycling process or the recycled PET product. Typically, technical problems with inks are referred to as 'bleeding" because of the visual staining of wash water and subsequent discoloring of clear PET flakes. Thermal printing inks create an additional challenge as the pigment may not stain the water yet be a technical barrier in achieving the desired quality for the PET recyclate. The examination of inks used for label decoration and printing is appropriate for labels used on thermoformed packaging. Further --

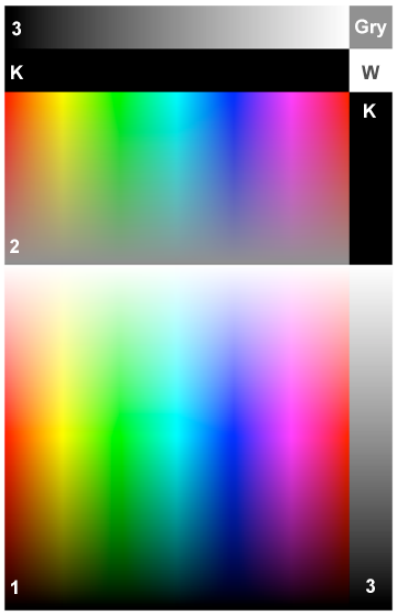
1. A label that has passed the protocol in the undecorated form is preferable to an unexamined one, although a decorated and printed system can be tested all at once.

2. The label shall be fully background colored as is the commercial intent. This means the background color should be as intense as used commercially. Label may be laminated or coated.

3. Representative imprinting is encouraged if the label is to be subsequently imprinted with package data. The amount of imprinting, such as would be done at a point-of-sale printing, should be representative of what is expected for the label being examined. The imprinting must be of the nature used at the point-of-sale printing.

4. For a given inking system, the recommendation is to test the label with the largest surface area and greatest amount of ink and number of colors. Then labels with smaller area and less ink used are expected to perform as well or better.

5. The printing on the label should approximate or match the test image below for color range and intensity unless matching a commercial application, in which case the commercial image should be used.



Default print test image.
http://www.imatest.com/guides/modules/print-test/

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6. The decorated and printed label, a minimum of 8 square inches per nominal 140 square inches of external thermoform surface, shall be processed as above into flake and flake processed into 3 mm plaques. If the intended label exceeds about 6% of the external thermoform area, the label shall be tested at its intended coverage area relative to the thermoform. The label need not be applied to the thermoform. This is TEST MATERIAL.

7. The following testing will examine the substrate materials and construction, the adhesive system, the decoration and printing, and any overcoating.

8. CONTROL MATERIAL is as defined above.

9. Testing is as listed above.

10. The guidance criteria for inking systems on labels, utilizing plaques of control and test material, includes --

    Plaque L* - Greater than 82 for both CONTROL MATERIAL and TEST MATERIAL.

    Plaque b* - CONTROL MATERIAL plaque b* must be below 7.0. A Δb* over 3.0 measured in transmission is unsuitable for recycling, comparing CONTROL MATERIAL and TEST MATERIAL.

Plaque haze - Desirable for the CONTROL MATERIAL plaque to exhibit a % haze less than 18%. Δ% haze values of TEST MATERIAL over CONTROL MATERIAL greater than 10% are unsuitable for recycling.

Fluorescence - No increase in fluorescence from TEST MATERIAL plaque compared to CONTROL MATERIAL plaque is desirable. No substantial fluorescence in CONTROL MATERIAL (no optical brighteners added to PET resin) is desirable.

Defects - The desired number of TEST MATERIAL defects should be comparable to and based on the number of defects seen for CONTROL MATERIAL.

Results Report

Thermoform Decoration Sample **Thermoform used** _____ **Label and Adhesive used** _____		**Background inks** _____ **Prink inks** _____	
Investigator		**Date**	
Data Subject	**Control Material**	**Test Material**	**Evaluation Target**
Initial % label on thermoforms, by surface area	XXXXXXXXX		XXXXXXXXXXX
Wash water discoloration			Minimal discoloration
L* color, 3 mm Plaques			L* greater than 82
a* color, 3 mm Plaques			
b* color, 3 mm Plaques			Control Material b* less than 7.0 Δb* less than 3.0
Haze, 3 mm Plaques			Control Material % haze less than 18%. Δ haze ≤10%
Fluorescence, Plaques			Negligible, both samples
Specks and other unusual observations			For 25 plaques each of Control and Test Materials, with failure defined as any plaque with specks exceeding 15 mils, the allowable is defined below

ACCEPTABLE LEVEL OF DEFECTS IN TEST MATERIAL PLAQUES 25 plaques tested												
For CONTROL MATERIAL with defects over 15 mils	0	1	2	3	4	5	6	7	8	9	10	11
Number of TEST MATERIAL defects over 15 mils	3	5	7	8	10	11	12	13	14	15	16	17
Unpaired t test, 0.05 statistical significance												