



Association of Postconsumer Plastic Recyclers
1001 G Street, NW, Suite 500
Washington D.C. 20001

www.plasticsrecycling.org

Protocol for Evaluating PET Thermoform Labels and Adhesives for Compatibility with PET Recycling

- *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 need no 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 virgin PET thermoforms are compared to unlabeled virgin PET thermoforms*
- *A generic wash process is described for evaluation of the effects of thermoform components (adhesives, labels, etc.) on recycled PET material.*

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 determine the applicability of regulatory limitations prior to use.

Equipment/Supplies List

- Approximately 20 pounds of each labeled thermoform sample(s) will be needed for this study.
- Approximately 5 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 pounds, of the unlabelled thermoform material to about 1 cm (3/8 inch) flake. This is the **CONTROL MATERIAL**.
- Estimate the approximate weight ratio of label material to PET thermoform material. Record as "Initial weight % label on thermoforms"

- Grind approximately 15 lb of the labeled thermoforms to about 1 cm (3/8 inch) flake. This is the **TEST MATERIAL**. Initially, it will have label material included with PET flakes.
- Preferred - Baffled beaker - stainless steel, six inch diameter by nine inches in height with four 3/4 inch baffles.
 - Alternate - similar size beaker without baffles
- Hot plate capable of heating to 190°C
- Scale or balance capable of measuring 500 (+/- 0.5) grams
- Oven capable of drying flake at 40°C
- Cookie sheet (or other suitable tray with a minimum area of 120 sq.in.)
- Cover for beaker which can accommodate thermometer and stirring impeller rod
- Overhead stirrer capable of 1,000 rpm
- Stirring impeller - pitched, 3 blades, and 3.0 inch diameter.
- Manual stirring rod
- Thermometer
- Strainer - non-aluminum
- Distilled or deionized Water
- Graduated cylinder
- Triton X- 100 nonionic surfactant (available from Union Carbide at 1-800-969-2707)
- Caustic (granular NaOH)
 - Be sure to read all material safety data sheet
- Desiccant dryer, capable of achieving air with -40°C dew point
- Injection molding machine with molds 3 mm thick and of size for color measurement
- Color measurement device.

PET Sample Wash Procedure

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

CONTROL MATERIAL Wash

1. Prepare a wash solution of 0.3% by weight Triton X-100 (6.0 grams or 5.7 ml 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 2,000 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. 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 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 2 pounds (1 kg) of **CONTROL MATERIAL** flake are washed. Blend batches for a uniform **CONTROL MATERIAL** sample

TEST MATERIAL Wash

1. Set up and adjust elutriation facility using about 2.5lb of **TEST MATERIAL** flake to achieve a 2% or less loss rate of PET flakes. Note settings.
2. Elutriate the remaining 12.5 lb. of **TEST MATERIAL** flake using the settings from Step 1. Record amount of label material removed before washing.
 - a. Perform a hand sortation on a representative sample to estimate percentage of flake remaining with labels attached.
 - b. Report percent of label remaining as “% label after 1st elutriation”
3. Prepare a wash solution of 0.3% by weight Triton X-100 (6.0 grams or 5.7 ml 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 2,000 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 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 12.5 pounds (5.7 kg) of **TEST MATERIAL** flake are washed. Blend batches for a uniform **TEST MATERIAL** sample.

PET Evaluation

This procedure 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 determine the applicability of regulatory limitations prior to use.

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. Measure the wash water for bleeding inks for the **Test Material** sample

APR Fluorescence Measurement

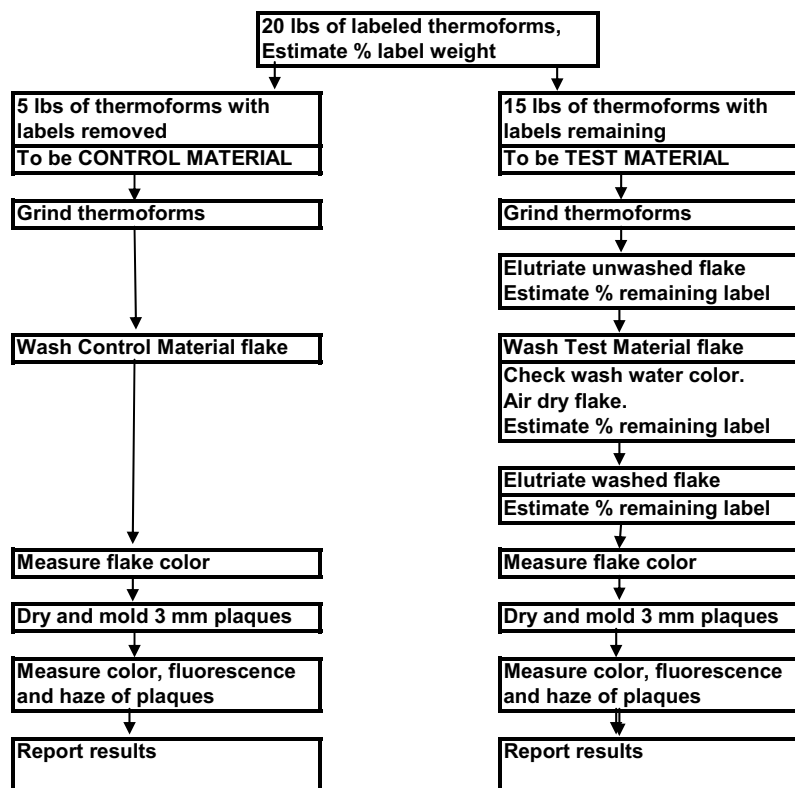
For flake samples of Test Material and Control Material no substantial difference of emission fluorescence intensity for excitation or absorption wavelength between 320 and 390 nm. Fluorescence intensity depends on many variables. Substantial difference is more than a 50% difference in area under the 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.

CONTROL MATERIAL Evaluation

1. Air dry or 40°C dry and blend **CONTROL MATERIAL** washed flakes.
2. Desiccant hot air dry **CONTROL MATERIAL** flakes at 320°F for a minimum of 4 hr to less than 50 ppm moisture.
3. Injection mold 3mm plaques from nominal 2 lb of washed **CONTROL MATERIAL** flake
 - a. Select 6 representative plaques
 - i. Measure L*a*b* and Haze by transmission
 - ii. Perform APR fluorescence measurement.
 - b. Report any unusual effects seen during injection molding, such as fuming.

TEST MATERIAL Evaluation

1. Air dry or 40°C and blend **TEST MATERIAL** washed flakes.
 - a. Perform a hand sortation on a representative sample to estimate percentage of flake remaining with label
 - b. Report "% label remaining after wash", before 2nd elutriation.
2. Set up elutriation using about 2.5lb of washed and air dried **TEST MATERIAL** flake to achieve a 2% or less loss rate of PET flakes. Note settings.
3. Elutriate 10 lb of the washed **TEST MATERIAL** flake under conditions identified from Step 2
 - a. Perform a hand sortation on a small representative sample of flake to estimate percent label remaining with flake
 - b. Report percent of labels remaining as "% label after 2nd elutriation".
4. Desiccant hot air dry the washed **TEST MATERIAL** flake at 320°F for a minimum of 4 hr to less than 50 ppm moisture
5. Injection mold 3mm plaques from about 8 lb of washed **TEST MATERIAL** flake
 - a. Label plaques in order of molding
 - i. Number 1 through 50
 - b. Select 6 representative plaques
 - i. Measure L*a*b* and Haze by transmission
 - ii. Perform APR fluorescence test
 - c. 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.



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.
- Weight % label after 1st elutriation, and before wash for **TEST MATERIAL**
- 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 from **TEST MATERIAL** flake wash
- Weight % label remaining after wash, before 2nd elutriation, for **TEST MATERIAL**
- Weight % label remaining after 2nd elutriation of **TEST MATERIAL**
- 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 10 mils. For 50 plaques of

TEST MATERIAL, note if 6 or more plaques contain black specs exceeding 10 mils.

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* - Δb^* over 3.0 measured in transmission is unsuitable, comparing CONTROL MATERIAL and TEST MATERIAL
- Plaque haze - % haze values over 14% are unacceptable for CONTROL MATERIAL and TEST MATERIAL
- 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)

This document does not guarantee acceptance or rejection of tested materials. APR encourages users of this protocol to discuss results with various knowledgeable parties using and specifying post consumer PET material to determine usefulness of subject

RESULTS REPORT

Thermoform Sample			
Investigator			
Date			
Data Subject	Control Material	Test Material	Evaluation Target
Initial % label on thermoforms	XXXXXXXXXX		XXXXXXXXXXXXXXXXXX
Weight % label after 1 st elutriation	XXXXXXXXXX		XXXXXXXXXXXXXXXXXX
L* color, Washed Flake			
a* color, Washed Flake			
b* color, Washed Flake			b* less than 3.0
Wash water discoloration	XXXXXXXXXX		No discoloration
Weight % label after wash	XXXXXXXXXX		XXXXXXXXXXXXXXXXXX
Weight % label after 2 nd elutriation	XXXXXXXXXX		XXXXXXXXXXXXXXXXXX
L* color, Plaques			L* greater than 82
a* color, Plaques			
b* color, Plaques			Δb^* less than 3.0
Haze, Plaques			Less than 14%
Fluorescence, Plaques			Negligible, both samples
Specks and other unusual observations			For 50 plaques made of Test Material, 5 or fewer plaques with specks exceeding 10 mils allowed. From 50 plaques made with Control Material, 2 or fewer plaques with specks exceeding 10 mils allowed