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Champions for Change
Critical Issues Guidance for Innovations
Sleeve Label Substrate for PET Bottles Critical Guidance Document

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 the benefits of sleeve labels for PET bottles in that adhesives are not used and adhesive residue therefore is not encountered in PET bottles with sleeve labels being offered for recycling. This document outlines other considerations relative to evaluating sleeve labels for their impact on PET bottle recycling, with a special focus on the separation of flake particles and is intended for both shrink and stretch sleeve labels.

It should be noted that this document does not address the difficulty of NIR identification of sidewall resin for full sleeve labels or the phenomenon of 'drum-tight' shrinkage in pre-wash systems. It does not include time as a variable. Innovations which include time as a factor will require separate analysis.

In addition, this document does not address the detailed questions about bottle making or other applications making and performance. APR has a separate Applications Guidance Document that provides guidance on testing for applications which may use postconsumer PET, including bottles. It is recommended that those evaluations be conducted only after the innovator is satisfied that the innovation has satisfied the intent of the guidance herein offered.

This document represents a screening tool to help the innovator understand the approximate effect of the innovation on plastic bottle recycling in several concentration scenarios. It offers:

- A limited number of critical, testable properties for PET bottles that represent key technical considerations for recycling. Other issues may also be important.
- Guidance on test sample preparation and test methods.

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- Guidance values for interpreting test results.

Innovators may petition APR for recognition for meeting or exceeding the most stringent guidance for all parts of this document. Innovators may also petition APR for Recycling Guidance Recognition for innovations that meet or exceed the most stringent guidance offered in its Applications Guidance.

The inability of an innovation to meet specified guidance values does not imply recycling failure, but should be a clear message that significant technical challenges might exist under certain circumstances and mitigation of the issue may be needed to avoid degrading the value of the stream of recyclable bottles. While sorting capability may address the effect of technically problematic bottles on the current stream of recyclable bottles, innovators are cautioned not to rely on either automatic sorting or dilution as justification for introducing innovations that have not been evaluated. Through the former, new introductions may contribute to decreased yields and increased costs. The latter does not preclude the possibility of overall degradation of the recyclables stream.

This document recommends testing at 0% and 50% innovation material content. The 0% innovation testing is baseline or control testing. Due to the commercial reality of variable and diverse bale content, it is advisable for innovators to consider the impacts of high levels of their innovations on the bottle reclaiming industry.

The protocol offered in this document is based upon APR's **PET Bottle Critical Guidance Document** and learnings from APR-sponsored testing on sleeve labels. This protocol is modified from the **PET Bottle Critical Guidance Document** in the following aspects:

1. The samples include a control (a PET bottle without any label), a label control (a combination of PET bottle and polypropylene wrap-around label), and the test sample (the PET bottle with sleeve label applied).

    a. The label control is to confirm the test results reflect the test sample and not difficulties with the test procedure.

2. The elutriation normally performed immediately after grinding is omitted.

3. A clumping test is added.

4. Testing for solid stating effects and DSC measurements on PET are omitted.

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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THE INFORMATION AND PROTOCOLS PROVIDED HEREIN DO NOT PURPORT TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH APPROPRIATE SAFETY AND HEALTH PRACTICES PRIOR TO USE. APR AND ITS MEMBERS ACCEPT NO RESPONSIBILITY FOR ANY HARM OR DAMAGES ARISING FROM THE USE OF OR RELIANCE UPON THIS INFORMAIOTN BY ANY PARTY.

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Recommended Test Protocol

1. **Materials:**

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

      i. 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 L44A	DAK (Wellman) HP 806

      ii. 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 meltings 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.

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

   b. **Whole bottle sleeve labels** (hereinafter the "Innovation label" that is the subject of the testing)

      i. The label should be designed for the bottle on which it will be commercially used.

      ii. The label will be applied to the Intended Test Bottle.

      iii. The label may be (a) unprinted, in which case any technical difficulties meeting the guidance criteria will not be attributed to printing or decoration;

or (b) printed, in which case certain technical challenges may or many not be attributed to the printing or decoration.

iv. **Note**: To understand the impact of choice of label substrate, the Innovation label should be unprinted if possible so that only the effect of the label material itself can be studied in this program without interferences of inks or over-coating materials. A fully decorated Innovation label can be studied using this protocol to assess the effects of a commercial label. However, there may be complicating influences due to the decoration itself that might not be separable from the label substrate material effects.

v. If the label is printed, the investigator may wish to test the label per Appendix C (the Protocol for Producing PET Flake for Evaluation and Evaluating for Discoloration from "Bleeding Labels").

c. **Wrap-around polypropylene label**

i. An undecorated label is recommended. Alternatively, a decorated wrap-around polypropylene label tested (as per Appendix C) or known in the marketplace to not cause ink bleeding in hot caustic water wash can be used.

ii. This label material is not to be applied to the bottle, so as not to introduce adhesive, but will be ground with control bottles.

2. **Applicable Terms:**

a. **Unlabeled Control Bottle** is defined as 100% by weight of unlabeled Intended Test Bottles.

b. **Labeled Control Bottle** is defined as 97.5% by weight of unlabeled Intended Test Bottles, and 2.5% by weight of wrap-around polypropylene label. Cut wrap-around label is to be added to the control bottles during grinding.

c. **Labeled Test Bottle** is the Intended Test Bottle with the Whole Bottle Sleeve Label applied where the label is defined as a shrink or stretch sleeve label intended to cover the Intended Test Bottle from neck to base.

3. **Preparation of Test Samples**

a. **Sample 1**: Unlabeled Control Bottles

i. Grind the unlabeled Intended Test Bottles to nominal ¼ to ½ inch size flake.

b. Sample 2: Labeled Control Bottles

i. Grind the unlabeled Intended Test Bottles with the 2.5% by weight of wrap-around label to nominal ¼ to ½ inch size flake or create a blend of 2.5% wrap-around label and 97.5% Unlabeled Control Bottle, each ground to nominal ¼ to ½ inch size flake.

Note 1: The wrap-around label material can contain static electricity and easily stick to the walls of the grinder and other vessel. Care should be taken to include all material that sticks to the walls of the grinder with the final ground flake material.

Note 2: It is permissible to grind the wrap-around label separately from the bottles and spike it into the appropriate weight of blended ground Intended Test Bottle flake.

c. Sample 3: Labeled Test Bottles

i. Grind the Intended Test Bottle with sleeve label applied to nominal ¼" to ½ inch size flake insuring that all label material is not lost to static cling in the grinder.

4. **Air Elutriation**

a. A pre-wash elutriation will not be performed after grinding, in order to produce a robust-case scenario approximating a pre-wash situation.

b. Elutriate a portion of Sample 1 to establish an elutriation setting that allows no more than 1.2 % of the PET to be carried over with label. Save sample for subsequent testing.

5. Prepare Bottle Flake Blends

a. Blend A is 100% Sample 1, Unlabeled Control Bottle

b. Blend B is 50% Sample 2, Labeled Control Bottle (with loose wrap-around label) and 50% Sample 1, Unlabeled Control Bottle

c. Blend C is 50% Sample 3, Labeled Test Bottle (with full sleeve label) and 50% Sample 1, Unlabeled Control Bottle

6. **Flake Washing**

a. 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).

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

c. Wash each Bottle Flake Blend separately at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated water at 88 $\pm$ 2 °C for 15 minutes. Record the composition of the wash solution. 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.

i. Note the weight of wrap-around label removed from Bottle Flake Blend B and list as a percent of initial weight and actual weight.

ii. Note the weight of sleeve label removed from Bottle Flake Blend C and list as a percent of initial weight and actual weight.

d. 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.

i. Note the weight of wrap-around and sleeve label removed for each Bottle Flake Blend and list as a percent of initial weight and actual weight.

e. Repeat sink/float once again.

i. Note the weight of label removed and list as a percent of initial weight, ___ %, and actual weight, ____ grams.

f. 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.

g. Air dry flake without losing any residual label film material. Visually examine flake for the presence of label film residue and note approximate weight percentage, ___%. Examine without magnification from a distance of 12 inches using illumination typical for reading.

h. If, in the opinion of the investigator, a dye staining can make more certain the presence of label film material among PET flakes, the investigator use and discuss the staining technique. Return any weighed residue to the PET flake mix.

i. Air elutriate to remove light fractions with one pass. Set up air elutriation system so that it is 1.2% as outlined in step 4b above.

i. Note the weight of wrap-around and sleeve label and PET removed and list as a percent of initial weight and actual weight removed for each Bottle Flake Blend.

ii. Note the weight of wrap-around and sleeve label removed and list as a percent of initial weight and actual weight.

iii. Visually examine the PET flake for the presence of label film residue and note approximate weight percentage, ___%. Examine without magnification from a distance of 12 inches using illumination typical for reading. If, in the opinion of the investigator, a dye staining can make more certain the presence of label film material among PET flakes, the investigator may so do and discuss the staining technique.

i. If necessary for those label materials that are less dense than water, sink/float the air separated label material to find the weight % of label of the total mass removed. Return any weighed residue to the PET flake mix.

j. On the basis of weighed label material removed, calculate the amount of residual label material still with flake of Bottle Flake Blends B and C samples and report weights and calculated residue as ___ parts per million (ppm) in the flake.

k. Retain 2 lb. samples of each variable for clumping evaluation.

7. Clumping/Agglomeration Evaluation

Using the 2 lb samples of washed flake for each Bottle Flake Blend from step 6.k:

i. Adjust the circulating oven temperature to 407 °F, ± 5 °C.

ii. Weigh and record the washed flake samples.

iii. Using a glass or Teflon®-lined baking dish for each washed flake sample, layer the washed flake to a depth of 1.5 +/- 0.25 inches.

iv. After 1.5 hours, remove the samples from the oven and allow to cool to room temperature without disturbing

v. Gently transfer the contents of the pan to a sieve with 0.625 inch opening and gently shake the screen to cause single flakes to fall through. Hand remove single flakes that are oversized and unable to pass through the sieve and place with flakes that passed through. Agglomerated flake that break up during this sieving would not be felt to be a problem.

vi. Weigh all agglomerates that cannot pass through the sieve. Include material fused to the baking dish, if any. Any flake that melts and sticks to the baking pans should be weighed and added to the weight of agglomerated material.

vii. Calculate the % of clumping as (weight of material left on sieve and in baking pan)/ (initial weight).

8. **Extrusion/Pelletization (second melt history)**

i. Desiccant dry Bottle Flake Blends A, B, and C for at least 4 hours at 320 $\pm$ 20°F (160 $\pm$ 12°C) to achieve moisture below 50 ppm. Do not remove clumps of label residue and flakes or label residue.

ii. Extrude and pelletize the Control Blend A and the Test Blends B and C separately.

iii. For Blend A, B, and C measure back pressure after extruding through 40/250/40 mesh, equal to 63 micron, for 30 minutes. Extrusion rate should be at least 375 gm/cm^2 per hour.

- Note any fuming, smoking, or odors during extrusion, sticking between flakes during drying, fouling of process equipment, or creation of unsafe conditions, such as increased fire potential.
- Note any buildup on the screen pack.
- Measure IV of extrudate from each sample.

iv. Calculate the weight-average of flake IV from the IV's measured on Control and Test Bottle.

v. Calculate the delta IV, average flake minus extruded pellet, for Blends A, B, and C.

9. Plaque Molding (third melt history)

a. Dry each of the pelletized Blends with desiccated air at 320 ± 20°F (160 ± 12°C) for 4 to 6 hours to achieve less than 50 ppm moisture content.

b. Injection mold a minimum of 50 3 mm plaques from Blend A first. Then mold a minimum of 50 3 mm plaques from Blends B, and C under identical conditions if possible. If the processing conditions need to be changed, document and report the changes.

c. Randomly select 5 plaques from each sample for color and haze measurement.

10. Data Reporting and Guidance

a. Record the amount in weight and/or ppm of the label remaining after washing for:

i. Blend B, 50% loose wrap-around label.

ii. Blend C, 50% applied sleeve label.

Note: a staining for the label material can be done so long as stained label material is not included in any further testing of color effects on PET.

b. Record the amount in weight and/or ppm of the label remaining after post-wash elutriation for:

i. Blend B, 50% loose wrap-around label.

ii. Blend C, 50% applied sleeve label.

Note: a staining for the label material can be done so long as stained label material is not included in any further testing of color effects on PET.

c. Clumping/Agglomeration of flake

i. Examine the agglomerated weight of Blend A, B, and C.

<5% by weight clumping (guidance value to be met)
5%-10% by weight (further study recommended)

>10% by weight (technically problematic for recycling)

Note: Failure of Blend B to meet the guidance value above is taken to mean the test procedure needs examination, particularly if Blend C also fails.

d. Extrusion/pelletization

i. Report extrusion pressure for Blend A (no guidance value)

Guidance: less than 10% higher extrusion pressure for 30 minutes for Blend B or Blend C vs. Blend A. No build up on screen.

ii. Record a rate of at least 375 gm/cm^2 per hour.

iii. Measure IV on each pelletized material with ASTM D 4603 and solution IV with phenol/tetrachlorethane at 30°C.

The Δ IV for Blend B or Blend C pellets vs. Blend A pellets guidance:
≤0.025 (guidance value to be met)
0.025-0.04 (further study recommended)
>0.04 (technically problematic for recycling)

e. Plaque Molding (3 mm) (nominal 2 inches x 2 inches plaques), at least 50

i. Measure IV on plaques of each Blend with ASTM D 4603 and solution IV with phenol/tetrachlorethane at 30°C.

The Δ IV for Blend B or Blend C plaques vs. Blend A plaques guidance:
≤0.025 (guidance value to be met)
0.025-0.04 (further study recommended)
>0.04 (technically problematic for recycling)

Note: Failure of Blend B is taken to mean the test procedure needs examination, particularly if Blend C also fails.

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

A. L* >82 for all Blends
B. Δb* of Blend B or Blend C vs. Blend A guidance:
<1.5, (guidance value to be met)
1.5 to 5.5, (further study recommended)
>5.5, (technically problematic for recycling)
C. Haze of Blend B or Blend C vs. Blend A guidance:
<9.5%, (guidance value to be met)
9.5-14%, (further study recommended)
>14%, (noticeable, technically problematic for recycling)

Note: Failure of Blend B is taken to mean the test procedure needs examination, particularly if Blend C also fails.

Note: Color Measurement

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

iii. Black Specks – 50 plaques molded each for Blend A, Blend B, and Blend C and viewed without magnification from 12 inches away. Count any plaque with a speck greater than 0.015 inches as failed.

Failures seen for Blend A	0	1	2	3 or more retest
Allowed failures for Blend C	2	4	6	
Retest if Blend C has X failures and Blend B also has X failures	X= 3 or more	X= 5 or more	X= 7 or more	

a. Pass/Fail based on 5% Significance using an unpaired t-Test comparing Blend C vs. Blend A. 50 plaques of each.
b. Note if streaks of haze or color are seen in any examined plaque.

c. Also, note the presence of unmelted materials other than black specks (such as fibers of paper or metallized material) and provide a quantification of such.
d. Failure of Blend B is taken to mean the test procedure needs examination, particularly if Blend C also fails.

iv. Fluorescence (visual, no more for Blends B and C than for Blend A)

f. Other observations and guidance for Blends B and C

i. Fuming during extrusion (no more than for Blend A)

ii. Smoking during extrusion (no more than for Blend A)

iii. Unusual odor during extrusion (no more than for Blend A)

iv. Equipment fouling (no more than for Blend A)

v. Unsafe condition (no more than for Blend A)

Appendix A -- Control Resins

All data shown in this table have been taken from what are believed to be current resin data sheets

APR Control Resin Listing	Type	IV	L*	b*	Test Method
Auriga Polyclear Splash 3301	Water	0.74			
M&G Cleartuf Turbo II	Water	0.74±0.02	70.0 min	-1.5 max	M&G
DAK Laser+ W L44A	Water	0.75±0.02	82.0±2.0	-3.0±2.0	CIE
Auriga Polyclear Refresh 1101	CSD/non water	0.83			
M&G Cleartuf MAX	CSD/non water	0.84±0.02	70.0 min	-0.5 max	M&G
DAK (Wellman) PermaClear HP806	CSD/non water	0.84±0.02	79.0±2.0	-2.5±2.0	CIE

European PET Bottle Platform
Artenius Flow
CEPSA CepsaPET SR08
Equipolymers Lighter C93
Indorama RAMAPET N1, N180, N1(S)
M&G Clearfuf P82

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Appendix B -- IV Adjustment for Water

The presence of inconsistent amounts of water can confound the determination of intrinsic viscosity, ItV or IV. If, as the guidance indicates, a difference is noted between test compositions and controls, it is very important that the moisture in both the test and control samples be the same. The table and equation below allow for corrections when the measurements of moisture of the dried samples shows there to be a difference in water content. A correction example is given.

Moisture Correction Values				
	Dryest sample IV			
	0.70	0.75	0.80	0.85
Delta ppm H2O	Change in IV, dL/g	Change in IV, dL/g	Change in IV, dL/g	Change in IV, dL/g
0	0.000	0.000	0.000	0.000
5	0.003	0.004	0.004	0.005
10	0.006	0.007	0.008	0.010
15	0.009	0.011	0.013	0.015
20	0.012	0.014	0.017	0.019
25	0.015	0.018	0.021	0.024
P.D. Richie, Society of Chemical Industry Monogr., 13, 107 (1961)				
(phenol/tetrachloroethane 60/40 wt/wt solvent at 25°C)				

Delta IV due to water =
(dry IV)-0.00075*(1000000/((2000000/((dry IV)/0.00075)^(1/0.68))/2+(Δppm water)/18))^0.68

Example: Consider a case where the measured IV on the extruded control flake is 0.750 with a measured moisture content of 17 ppm, and the extrudate from the Labeled Test Bottle has a measured IV of 0.724 with a moisture of 42 ppm. Using the above Table, the difference in moisture between the samples is 25 ppm, thus if the Labeled Test Bottle material were to have been dried to the same level of moisture as the control, the predicted IV would increase by 0.018 dL/g. Thus, rather than comparing a control IV of 0.750 to an Innovation control IV of 0.724 where the difference would be 0.026, the comparison would be made between the control at 0.750 and the Labeled Test Bottle material (moisture corrected) at 0.742, now showing a difference of 0.008. This process has the result of greatly reducing the impact of residual moisture on the extruded IV values. Alternatively, each measured IV could also be normalized to 0 ppm yielding the same results.

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Moisture differences may benefit from further study when the differences in IV are >0.025 to ≤0.040 dL/g.

Appendix C – Bleeding Labels

Protocol for Producing PET Flake for Evaluation and Evaluating for Discoloration from "Bleeding Labels"

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.

Equipment/Supplies List

For wrap around and other adhered labels, not shrink sleeve labels

- Label for evaluation, 0.3 grams per test. (not for shrink sleeve labels)
- Clean, PET flakes about 1 cm (3/8 inch) nominal (100 grams/test plus 100 grams for control) (See Appendix B below)
- Beaker – 800 ml. The beaker ID should be about 9 cm (3.5 inches) and have a slurry height to diameter ratio of 0.8
- 500 to 800 ml beaker, for mixing solution
- Hot plate capable of heating to 90^0C
- 125 mm watch glass to cover beaker when heating
- Scale or balance capable of measuring 500 (+\- 0.5*)* grams
- Overhead stirrer capable of 600 rpm
- Stirring impeller - pitched, and 4 cm (1.75 inch) diameter.
- Colorimeter/spectrophotometer
- Thermometer
- Strainer - non-aluminum, fine mesh
- Distilled or deionized Water
- Graduated cylinder, 500 ml
- 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 sheets.

Developed by Rutgers, The State University of New Jersey, Center for Plastics Recycling Research, - Busch Campus, Piscataway, NJ 08855, for NAPCOR, National Association for PET Container Resources

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PET Sample Wash Procedure

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

1. Cut up 0.3 grams of label into 0.7 cm (1/4 inch) squares.
2. Prepare "wash" solution in the mixing beaker consisting of 400 ml water (for a slurry ratio of 4:1), 0.3% by weight (1.2 grams of Triton X-l00) and 1.0% by weight (4 grams) caustic (NaOH).
   Note: Triton X-100 must be dissolved in cold water prior to the addition of caustic!
3. Add "wash solution" to 800 ml beaker. Heat solution on hot plate to 85C (185^0F) while covered with watch glass cover to minimize evaporation.
4. Add the 0.3 grams of label and 100 grams of clean, clear PET flake to the solution
5. Insert overhead stirrer so that the impeller is 1/2 inch above the bottom of the beaker.
6. Turn on overhead stirrer and adjust to 540 rpm.
7. Continue agitation for 15 minutes and maintain solution at 85C (185F)
8. Turn off and remove overhead stirrer. Remove beaker from heat and immediately strain solution, labels, and flake. Save solution for visual observation
9. Immediately rinse the remaining labels and PET flake with distilled or deionized water, approximately with 200 ml of water. Separate and dry the PET flake from the slurry.
10. To prepare the control PET flake for comparison, follow above procedure while omitting the label.

Note: Labels tested should be from commercial production operations. If preliminary testing is conducted on developmental labels from pilot-scale equipment, the testing should be repeated with commercially produced labels.

Evaluation of PET Container Component Color

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.

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1. Produce 100gram clear PET flake samples according to this procedure. Be sure to produce a sample of control flake from recycled PET bottles without the "bleeding label" to be evaluated.
2. Perform color analysis, according to the machine manufacturer's instructions, on each sample produced (test of "bleeding label"-colored PET and control PET) 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.

The guidance contained in 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 test materials. Any guarantees or warranties are expressly disclaimed, including without limitation any implied warranties of MERCHANTABILITY or FITNESS FOR A PARTICULAR PURPOSE.

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APPENDIX A of Bleeding Label Protocol for Sleeve Labels

Shrink sleeve labels

The preceding testing is intended for labels that constitute about 3% of the package weight and for processes that elutriate label scrap before washing. For labels, such as whole bottle shrink sleeve labels, that constitute more than 3% and up to 12% of the package weight and to include reclaiming processes which elutriate label scrap before washing, make the following adjustments to the procedure above:

Equipment and Supplies

Label for evaluation, 6.0 grams (not 0.3 grams) per test.
See Appendix B below

PET Sample Wash Procedure

1. Cut up 6.0 (not 0.3) grams of label into 0.7 cm (1/4 inch) squares.

2.-3. Same as previously listed.

4. Add the 6.0 (not 0.3) grams of label and 100 grams of clean, clear PET flake to the solution

5.-10. Same as previously listed.

Evaluation of PET Container Component

Same as previously listed.
For PET applications, floatable shrink sleeve labels are suggested

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