The Association of Postconsumer Plastic Recyclers

Pressure Sensitive Label Test
PET-CG-04

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 pressure sensitive adhesive (PSA) labels pose special consideration for the substrate choice, area of adhesive coverage, potential for residual adhesive on the PET, and decoration applied to the label. This document outlines considerations relative to evaluating PSA labels for their impact on PET bottle recycling.

This following protocol does not evaluate degradable additives in the label structure. It should be noted that this document 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.
- 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 test protocol is designed to test both an "Intended" and a "Generic" PSA label. An "Intended" PSA label and bottle combination is defined as a decorated label, applied to a specified bottle that will be sold in the market. A "Generic" PSA label is one that can be used on a wide variety of bottles; it may or may not be decorated.

For an "Intended" PSA and bottle combination, this document recommends testing at 50% Intended pressure sensitive (PSA) label applied to the Intended bottle blended with 50% unlabeled control bottle. The Intended label/bottle combination is expected to be decorated, and as such it is up to the label submitter to determine if an unprinted version should be evaluated along with a printed version. Testing both may yield valuable information in the event that the Intended label does not meet CGD guidelines.

For a "Generic" PSA label, this document recommends testing at 100% control bottle with a minimum of 20% surface PSA generic label coverage. The Generic label may be unprinted but should be tested in the form that it will be supplied to the label converters. The submitter may elect to study the label at a higher label coverage area as recognition for a generic label will be given at 2x the maximum % label coverage area studied.

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**. 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) and the test sample (the PET bottle with pressure sensitive adhesive (PSA) label applied).

2. The elutriation normally performed immediately after grinding is omitted and the calibration level is set up on the control at 1.2%.

3. A clumping test is added.

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

5. Testing at 25% labeled bottle is omitted, but can be done to better understand less favorable results for intended 50/50 blend or generic 20% surface area.

6. There is no guarantee when attempting to determine the component that contributes to undesirable results when working with decorated labels unless the unprinted and printed are processed side by side.

7. In the event bleeding label evaluation is also required, the test samples will need to be processed using the bleeding label protocol through to a plaque production.

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 INFORMATION BY ANY PARTY.

Recommended Test Protocol

1. **Materials:**

   a. **Unlabeled PET Bottle** (hereinafter referred to as the "Intended Test Bottle" for the specific pressure sensitive adhesive 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 L40A	DAK Laser+® B90A

      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 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.

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

   b. **Pressure Sensitive Adhesive Labels** (hereinafter the "Innovation label" that is the subject of the testing)

      i. The label should be of such size as to be appropriate for the Intended application or if generic, then cover a minimum of 20% of the bottle surface area.

ii. The label will be applied to the Test Bottle. (Minimum of 72 hours of staging before testing)

iii. The label shall be printed or decorated or coated as it would be for commercial use. Any point-of-purchase printing intended for the label should be present for testing. A Generic label may be tested unprinted to determine the acceptability of the label substrate material and adhesive and any coatings that may be applied prior to decoration.

2. Applicable Terms:

Unlabeled Control Bottle is defined as 100% by weight of unlabeled control bottles. A PET carbonated soft drink bottle is acceptable, after passing the quick color test if not an APR PET approved resin.

Labeled Test Bottle is the Intended Test Bottle with the Pressure Sensitive Adhesive Label applied.

The labeled test bottle blend shall constitute of either (1) 50% intended bottle with intended pressure sensitive adhesive label applied blended with 50% unlabeled approved intended control bottle, or (2) 100% generic control bottle with a minimum of 20% surface coverage

3. Preparation of Test Samples

Sample A: Unlabeled Control Bottles

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

Sample B: Labeled Test Bottles

50% intended bottle with intended pressure sensitive label applied blended with 50% unlabeled approved intended control bottle

OR

100% generic control bottle with a minimum of 20% surface coverage

Sample C: Decorated Labeled Test Bottles

50% intended bottle with intended pressure sensitive label applied blended with 50% unlabeled approved intended control bottle

OR

100% generic control bottle with a minimum of 20% surface coverage
Note: Generic labels may be tested undecorated, in which case there will be no Sample 3 carried through this test protocol.

Grind the Intended Test Bottle or the Generic bottle with pressure sensitive adhesive labels applied to nominal ¼" to ½ inch size flake insuring that no label material is lost to static cling in the grinder. Intended bottle flake is then blended 50:50 with unlabeled control bottle flake. Note that Generic bottle flake is tested at 100% with no dilution with control flake.

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. 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).

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

b. Wash each Bottle Flake Sample separately at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated 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.

i. Note the weight of PSA label removed from Bottle Flake Sample B and/or C and list as a percent of initial weight and actual weight.

c. 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 PSA label removed for Bottle Flake Sample B and/or C and list as a percent of initial weight and actual weight.

d. Repeat sink/float once again.

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

e. 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.

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

g. 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 choose to use and discuss the staining technique.

h. 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 PSA label and PET removed and list as a percent of initial weight and actual weight removed for each Bottle Flake Sample. Note the weight of label removed and list as a percent of initial weight and actual weight.

ii. Visually examine the PET flake for the presence of label 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.

iii. 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.

i. On the basis of weighed label material removed, calculate the amount of residual label material still with flake of Bottle Flake Sample B and/or C

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

6. Clumping/Agglomeration Evaluation

Using the 2 lb samples of washed flake for each Bottle Flake Sample from step 5j. above. Flakes should represent product ready for desiccant drying, and have been processed to remove label residue by washing, sink/float processing, and elutriation.

i. Adjust the circulating oven temperature to 407 ± 5 °F (208 ± 3°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 deemed 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).

7. **Extrusion/Pelletization (second melt history for polymer, first melt history with label material potentially present)**

   i. Desiccant dry Bottle Flake Samples A and B and/or C for at least 4 hours at 320 ± 20°F (160 ± 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, Sample A, and Test, Sample B and/or C separately.

   iii. For Samples A, B and/or 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 delta IV, extruded pellet, for Samples A to B and/or C.

8. **Plaque Molding (third melt history for polymer, second melt history with label material potentially present)**

   a. Dry each of the pelletized Samples 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 Sample A first. Then mold a minimum of 50 3 mm plaques from Samples B, and/or 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.

9. Data Reporting and Guidance

a. Record the amount in weight and/or ppm of any contaminates remaining after washing for a minimum sample size of 500 grams:

i. Sample B and/or C, pressure sensitive adhesive label.

Note:

1. The reported label weight should include any unattached label flakes as well as any label that is still adhered to PET flake and separately weighted for reporting. It is recognized that the reported label weight will probably include some PET flake by default.
2. 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 any contaminates remaining after post-wash elutriation for a minimum sample size of 500 grams:

i. Sample B and/or C, pressure sensitive adhesive label.

Note:

1. The reported label weight should include any unattached label flakes as well as any label that is still adhered to PET flake and separately weighted for reporting. It is recognized that the reported label weight will probably include some PET flake by default.
2. 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 Samples A, B and/or C.

≤1% by weight clumping (guidance value to be met)
>1% by weight (technically problematic for recycling)

d. Extrusion/pelletization

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

Guidance: less than 10% higher extrusion pressure for 30 minutes

for Sample B and/or C vs. Sample 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 Sample B and/or C vs. Sample 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 Sample with ASTM D 4603 and solution IV with phenol/tetrachlorethane at 30°C.

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

ii. 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 Sample B and/or C vs. Sample A guidance: < 1.5 Haze of Sample A guidance: < 9.5% Haze Haze of Sample B and/or C guidance:

< 20% Haze

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 Sample A, B and/or 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 Sample A	0	1	2	3 or more, retest
Allowed failures for Sample B and/or C	2	4	6	

a. Pass/Fail based on 5% Significance using an unpaired t-Test comparing Sample B and/or C vs. Sample 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.

iv. Fluorescence (visual, no more for Sample B and/or C than for Sample A)

f. Other observations and guidance for Sample B and/or C

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

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

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

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

v. Unsafe condition (no more than for Sample 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***	**Color Test Method**
Auriga Polyclear® Splash 3301	Water	0.74±0.02			
M&G Cleartuf® Turbo II	Water	0.74±0.02	70.0 min	-1.5 max	M&G
DAK Laser+® W L40A	Water	0.75±0.02	78 min	-3.0±2.0	CIE
Auriga Polyclear® Refresh 1101	CSD/non water	0.83±0.02			
M&G Cleartuf® MAX	CSD/non water	0.84±0.02	70.0 min	-0.5 max	M&G
DAK Laser+® B90A	CSD/non water	0.84±0.02	78.0 min	-3.0±2.0	CIE

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