The Association of Postconsumer Plastic Recyclers

Direct Print Label Test
PET-CG-05

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

It should be noted that this document does not address the difficulty of NIR identification of sidewall resin for direct print labels. 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" direct print label. An "Intended" direct print label and bottle combination is defined as a decorated label, applied to a specified bottle that will be sold in the market. A "Generic" direct print label is one that can be used on a wide variety of bottles.

For an "Intended" direct print and bottle combination, this document recommends testing at 50% Intended direct print label applied to the Intended bottle blended with 50% unlabeled control bottle.

For a "Generic" direct print label, this document recommends testing a labeled Generic bottle at 100% where the direct print label should represent 20% surface area coverage of the Generic control bottle surface.

Please note that a “Generic” print should include a variation of colors to represent fingerprints with equal % coverage of colors for testing to look at the range and the impact it will have on the recycle stream.

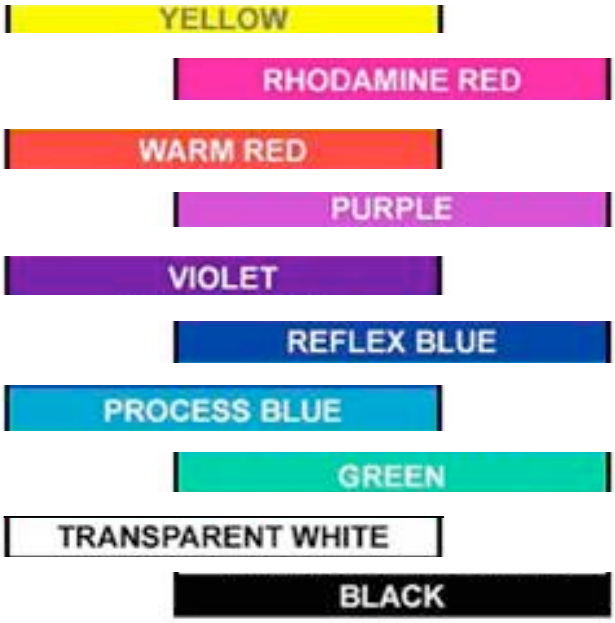
The fingerprinting is a method to evaluate the performance and capability of a printing press. The purpose of the fingerprint is primarily to provide information regarding the process, which can be used for reprographic specifications. There are a number of other areas that the fingerprint covers. By finger-printing, the standards and settings for the machine can be evaluated with a color lay down of various color combinations.

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.

Please refer to images below for APR suggested ideal print testing conditions for "Generic" direct printed bottles.

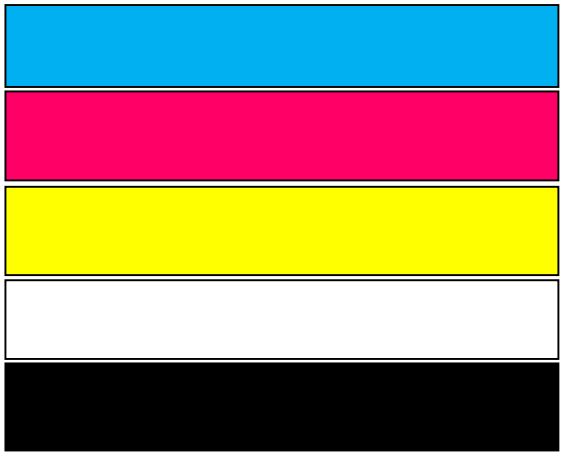
Option 1 Pantone– Clear Bottles
(One Label Multiple Colors)

Option 2 Pantone– Clear Bottles
(Multiple Labels Flood coat on each)

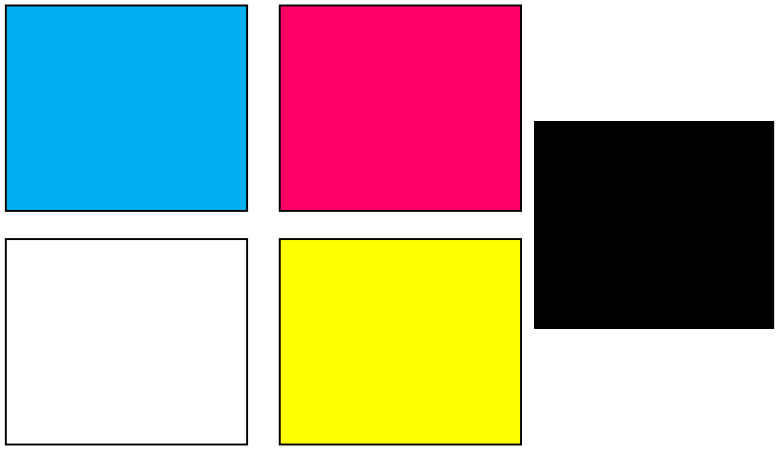


Please refer to images below for APR suggested secondary print testing conditions for "Generic" direct printed bottles.

Option 3 w/4 Color – Clear Bottles
(One Label Multiple Colors)



Option 4 w/4 Color – Clear Bottle
(Multiple Labels Flood coat on each)



In the event option 1-4 cannot be met please contact the APR Technical Director with suggested equivalent option for approval.

When developing the alternate for approval please keep in mind the ideal condition is, to print all potential color options with equal % coverage.

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 direct print label applied).

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.

5. Testing at 25% labeled bottle is omitted, but can be done to better understand less favorable results for intended 50/50 Intended blend or 100% generic.

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 direct print 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. **<u>Direct print Labels</u>** (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 a minimum of 20% surface area coverage

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

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 or Generic Test Bottle with the Direct print label applied.

The labeled test bottle blend shall constitute of either (1) 50% intended bottle with intended direct print label applied blended with 50% unlabeled approved intended control bottle, or (2) 100% generic control bottle with a minimum of 20% surface area coverage of label to PET control bottle.

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: Direct Print Labeled Test Bottles

50% intended bottle with intended direct print label applied blended with 50% unlabeled approved intended control bottle surface.

OR

100% generic control bottle with a minimum of 20% surface area coverage of label to PET control bottle surface.

a. Grind the Intended Test Bottle or the Generic bottle with direct print labels applied to nominal ¼" to ½ inch size flake insuring that minimal 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 A 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 (1,000 RPM) 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.

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.

d. Repeat sink/float once again.

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.

i. Retain all of both the wash and rinse waters for filtration evaluations.

f. Air dry flake without losing any residual label material. Visually examine flake for the presence of direct print label residue and note visual examination observations, without magnification from a distance of 12 inches using illumination typical for reading.

g. 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 direct print 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. (Note the weight will likely include PET flake weight and ppm calculations may not reflect label weight only)

ii. Air dry flake without losing any residual label material. Visually examine flake for the presence of direct print label residue and note visual examination observations, without magnification from a distance of 12 inches using illumination typical for reading.

h. On the basis of weighed label material removed, calculate the amount of residual label material still with flake of Bottle Flake Sample B

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

j. Retain 5 lbs. of flake for each variable for injection molding flake to plaques for flake to product black speck analysis (Sample C).

6. Filtration Evaluation

150 Micron Screen Evaluation

a. Take 150 micron screen and measure the weight using an analytical lab scale.

b. Screen all wash, rinse, and sink float water through a 150 micron screen separately per variable, including the control.

c. Once the water has passed through the screen, carefully remove.

d. Allow the screen to dry and measure the weight of the screen.

e. Record the difference in weight of the screen before and after filtration.

60 Micron Screen Evaluation

f. Agitate and measure 1,000 mL of the previously screened waters from 150 micron samples (wash, rinse and sink float water). Transfer separately to a glass beaker. (Agitation is to be sure unsettled particles resting on the bottom of the bucket or container are included)

g. Take 60 micron screen and measure the weight using an analytical lab scale.

h. Place the screen into the funnel and attach vacuum pump to flask.

i. Turn the vacuum pump switch to on.

j. Slowly pour the 1,000 mL of wash water sample into the funnel. The vacuum pump will begin to draw the water solution into the flask.

k. Once the water has passed through the screen, carefully remove.

l. Allow the screen to dry and measure the weight of the screen.

m. Record the difference in weight of the screen before and after filtration.

7. Clumping/Agglomeration Evaluation

Use 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.

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

b. Weigh and record the washed flake samples.

c. 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.

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

e. 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.

f. 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.

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

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

a. Desiccant dry Bottle Flake Samples A and B 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.

b. Extrude and pelletize Sample A (Control), and Sample B Test separately.

c. For Samples A, B 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.

i. 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.
ii. Note any buildup on the screen pack.
iii. Measure IV of extrudate from each sample.

d. Calculate the delta IV, extruded pellet, for Samples A to B .

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

c. 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, under identical conditions if possible. If the processing conditions need to be changed, document and report the changes. (Repeat steps for injection molding of flake to plaques Sample C)

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

10. Data Reporting and Guidance

a. Record the weight of screened particles from 150 micron screen and 60 micron screen for all samples evaluation.

   i. Subtract the control weight from the 150 micron screens to each sample
      a. Record data, no guidance at this time.

   ii. Subtract the control weight from the 60 micron screens to each sample
      a. Record data, no guidance at this time.

b. Clumping/Agglomeration of flake

   i. Examine the agglomerated weight of Samples A, B .

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

c. 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 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 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)

d. 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 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 vs. Sample A guidance: < 1.5 Haze of Sample A guidance: < 9.5% Haze Haze of Sample B 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.

iii. Black Specks – 50 plaques molded each for Sample A, B, 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	2	4	6	
Allowed failures for Sample C	Record	Record	Record	No Guidance at this time

1. Pass/Fail based on 5% Significance using an unpaired t-Test comparing Sample B vs. Sample A. 50 plaques of each.
2. Note if streaks of haze or color are seen in any examined plaque.
3. 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 than for Sample A)

v. Other observations and guidance for Sample B

1. Fuming during extrusion (no more than for Sample A)
2. Smoking during extrusion (no more than for Sample A)
3. Unusual odor during extrusion (no more than for Sample A)
4. Equipment fouling (no more than for Sample A)
5. 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.