



PET Critical Guidance PET-CG-01

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 some innovations may create bottles that present technical challenges for recycling.

This document is a subset of a larger Applications Guidance Document that provides additional guidance on testing final applications including bottles. As such, this Critical Guidance does not address the detailed questions about bottle making or other applications making and performance. Those evaluations should be conducted after the innovator is satisfied that the innovation has satisfied the intent of the guidance here 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 strives to accomplish the following:

- a. Limited number of critical, testable properties for PET bottles. Other issues may also be important. The properties listed are deliberately few and represent key concerns.
- b. Offer test samples and test methods.
- c. Recommend critical guidance values for interpreting test results
- d. Set the stage for further investigations into the effects on specific end uses after completion of this initial, critical issues examination.

THIS DOCUMENT IS NOT A SPECIFICATION AND DOES NOT IMPLY IN ITS DEFINITIONS, PROCEDURES, OR VALUES FITNESS FOR USE, MARKET ACCEPTABILITY, OR ANY GUARANTEE OR WARRANTY. MEETING THESE GUIDELINES DOES NOT OBLIGATE APR MEMBERS TO BUY BOTTLES CONTAINING THE INNOVATION.

Moreover, the inability of an innovation to meet specified 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 further 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 lists testing at 0%, 25%, and 50% innovation material. 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 guidance contained in this document does not include time as a variable. Innovations which include time as a factor will require additional considerations.

APR would consider a letter of recognition, upon petition, for those innovations that meet or exceed all of the strictest guidance listed. Full Recycling Guidance Recognition would follow meeting or exceeding the strictest guidance for all parts of the Applications Guidance Document.

PET Critical Guidance

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.

Test blends are defined as follows:

- Blend A: 100% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated below. 0% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend B: 75% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated below. 25% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend C: 50% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated below. 50% bottle flake made from Innovation Bottle processed to flake as indicated below.

The virgin PET Control Resin in 3 mm plaques and after three meltings should have a CIELAB b* in transmission less than 3 and an L* greater than 87. The following resins are suitable virgin



PET Control Resins for making Control Bottles to be rendered to flake and blended with flake made from Innovation Bottles:

<i>Low IV, Water Bottle Innovation Controls</i>	<i>CSD and Non-Water Bottle Innovation Controls</i>
Auriga Polyclear® Splash 3301	Auriga Polyclear® Refresh 1101
M&G Cleartuf® Turbo II	M&G Cleartuf® MAX
DAK Laser+® WL40A	DAK Laser+® B90A

Appendix A describes some characteristics of the named control resins.

Reclaim Processing Test Protocol: Label Consideration

If the innovation does not involve labels or adhesives and if it can be correctly argued that labels and adhesives have no impact on the innovation, bottles can be made and processed without the presence of labels or adhesives.

The **Reclaim Processing Test Protocol** is to include but is not limited to the following:

For this Critical Guidance Examination and use for bottles or sheet:

1. Separately, grinding of just whole Innovation Bottles or just whole Control Bottles to nominal ¼ to ½ inch size flake.
2. Air elutriation to remove light fractions with one pass and with set up to accomplish less than 2% PET flake loss from the feed for Control Flake.
3. Prepare a wash solution of 0.3% by weight Triton X-100 (6.0 gms or 5.7 ml 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!
4. Mix bottle flake or pellets at a ratio of 500 gms 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.
7. Air elutriation on air-dried, washed flake for one pass with set up to accomplish less than 2% PET flake loss from the feed for washed Control Flake.



8. Blend flakes from Innovation Bottles and Control Bottles, or acceptable pellets of Innovation Resins and Control Resins (see Note below for “Pre-test for new Innovation Resins”), as needed to make mixtures for Sample A1, B1, and C1 blends.
9. Desiccant drying for at least 4 hours at 320 F +/- 20F (160C +/- 12C) to achieve moisture in pellets below 50 ppm.
10. Melt-filtered pelletization on flake material or special pellet samples
11. Solid stating of pellets to at least 0.80 dl/gm IV.

For use in fiber evaluations

- A. As above, steps 1 to 10, with no solid stating of pellets.

NOTE: INDUSTRY PROCESSING MAY DIFFER FROM THESE STEPS.

Pre-test for new Innovation Resins:

Wash samples of innovation resin and control resin pellets in hot caustic aqueous solution (at least 85°C for 15 minutes. Water must have a pH of 12 to 13). Compare air-dried pellet color of caustic water-exposed pellets to unwashed pellets for innovation resin and for control resin. We are looking for the effect of washing on first the innovation resin and then on the control resin.

If b* difference measured between caustic-washed crystalline innovation resin pellets and unwashed crystalline innovation resin pellets is greater than 1.0 b* units, consider the innovation resin as if it were an additive and make bottles to obtain further test samples per the Additives, Coatings, Labels, Adhesives, & Multilayer Resins Protocol.

If the increase in delta b* for the innovation resin is less than 1.0 b* units, separately repelletize the innovation resin and control resin to add the heat history that making a bottle would have created. Be sure to properly dry the materials. These repelletized samples are A0 (control) and I0 (innovation). Measure the IV drop of the control and of the innovation pellets in making samples A0 and I0. An extrudate IV drop difference for Sample I0 compared to Sample A0 of less than or equal 0.025 dl/gm should not be a problem, from 0.025 to 0.04 needs study, and drop greater than 0.04 probably is a problem.

Then proceed to make blends of pellets instead of flake and to conduct sample preparation and tests as outlined. No further washing of pellets is needed. The blends of samples A0 and I0 are blend Samples A1, B1, and C1. These blends create the blends at step 8 above. Be sure to dry PET pellets with desiccated air at 320 F +/- 20F (160C +/- 12C) for 4 to 6 hours to achieve water



levels below 50 ppm. With properly dried and re-extruded material, create Sample A2, B2, and C2 pellets. The differences of extrudate IV drop for Samples B2 vs. Sample B1 and for Sample C2 vs. Sample C1 compared the extrudate IV drop of Sample A2 vs. Sample A1 of less than or equal to 0.025 dl/gm should not be a problem, from 0.025 to 0.040 needs study, and drop greater than 0.040 probably is a problem. Estimate the IV of Samples A1, B1, and C1 using the weighted arithmetic averages for the Sample A0 and I0 IV's. Samples A3, B3, C3 and Samples A4, B4, C4 and Samples A5, B5, C5 should be prepared and examined as listed below. Appendix B describes moisture effects on IV.

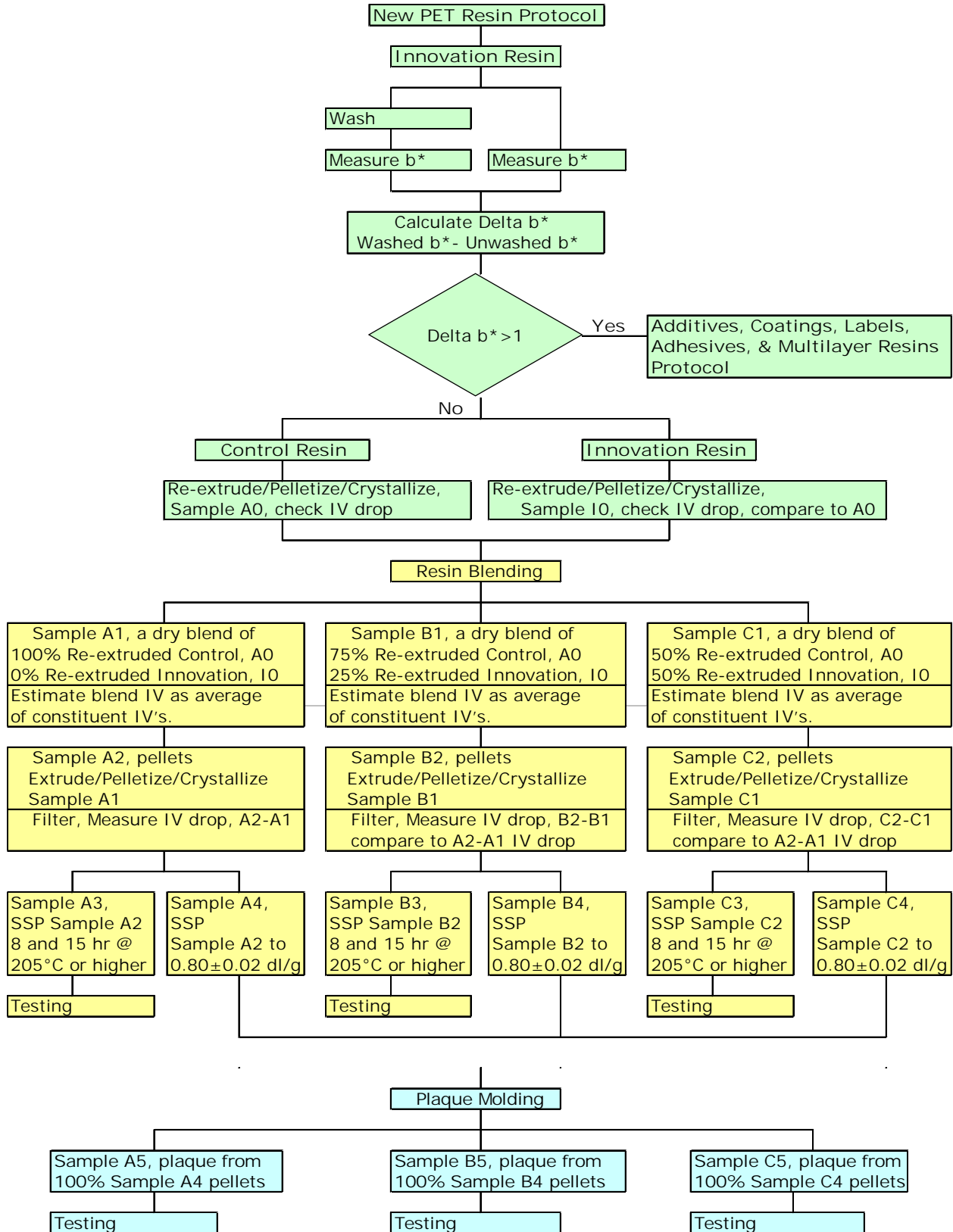
Further testing of new resins will include bottle performance, which the buyers of the new resins will have thoroughly investigated by fit-for-use studies.

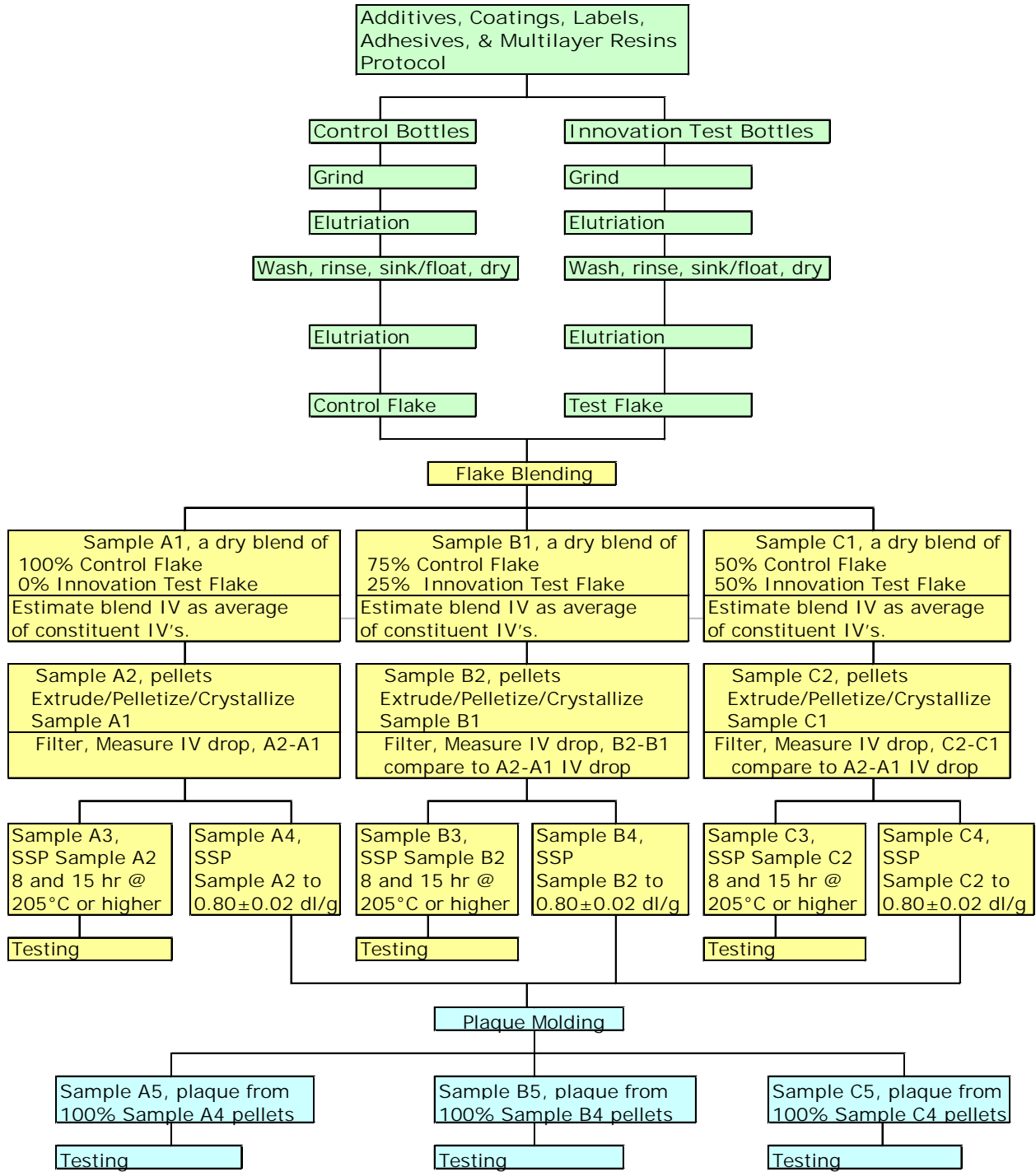
The control resin delta b* should be less than 1.0 b* units change if the test is performing as expected.

NOTE:

Intrinsic viscosity, IV, is a measured surrogate for molecular weight. If the Innovation Resin is known to have a molecular structure such that the measured IV does not represent the melt rheology characteristics of PET at that IV, then the melt rheology should be measured for the control resin, innovation resin, and blends. The innovator then should estimate the impacts for measured melt viscosity differences at near production conditions of temperature and shear rate using the guidance provided in terms of differences in IV drop on melting.

Testing Flow Diagram





Test Sample preparation

Prepare flake from Innovation Bottle and Control Bottle (or from pellets depending on pre-test) per the instructions above, steps 1 through 7.



Dry blend flake from Innovation Bottles and Control Bottles (step 8 above) per the definitions of the blends (samples A1, B1, and C1).

1.0 Sample blend pelletization

Prepare melt blends with flake (or special pellets) dried with desiccated air at 320 F +/- 20F (160C +/- 12C) for 4 to 6 hours to achieve water levels in dried pellets of below 50 ppm. Melt filter each blend. These are Samples A2, B2, and C2.

The difference in extrudate IV drop for Sample B2 vs. Sample B1 and Sample C2 vs. Sample C1 compared to the IV drop of Sample A2 vs. Sample A1 of less than or equal to 0.025 dl/gm should not be a problem, from greater than 0.025 to 0.040 needs study, and drop greater than 0.040 probably is a problem. Use weighted arithmetic averages of components to estimate IV of the mixtures in blends constituting Sample A1, B1, and C1.

1.1 Filterability Test

Property	Critical Guidance
Screen pack life. Feeding flake or pellet blends, Sample A1, B1, and C1	For Sample B1 and C1, less than 10% higher pressure after extruding through 40/250/40 mesh, equal to 63 micron, for 30 minutes compared to Sample A1. No buildup on screen. Rate at least 375 gm/cm ² per hour.

2.0 Solid Stating

2.1 Solid Stating Performance Test

Property	Critical Guidance
Solid stating time for pellets. Treat Sample A2, B2, and C2 pellets under identical conditions of temperature and vacuum or inert gas. Minimum heat transfer fluid temperature is	<ol style="list-style-type: none"> 1. Time is measured from when the heat transfer fluid reaches 190°C. Solid stating Samples A2, B2, and C2 to become Samples A3, B3, and C3. 2. Record IV's measured before and after solid stating for test and control samples. Record solid stating conditions. Failure of Sample A2 pellets to increase in IV may indicate the solid stating conditions need adjustment. The 15 hour cycle time is to investigate target IV's greater than 0.90 dl/gm. 3. Calculate the secant rate as (IV after 15 hours – Initial IV)/15 hours for Samples A3, B3, and C3. Calculate the expected IV



205°C	<p>after 8 and 15 hours for Sample A' using the initial IV for Sample A2 and the Sample A rate, for Sample B' using the initial IV for Sample A2 and the Sample B rate, and For Sample C' using the initial IV for Sample A2 and the Sample C rate. These calculated IV's are normalized to an identical starting point.</p> <p><u>Guidance:</u> No greater than 0.040 IV units difference for Sample A' minus Sample B' or Sample A' minus Sample C' for 8 hours. And No greater than 0.075 IV units difference for Sample A' minus Sample B' or Sample A' minus Sample C' for 15 hours.</p>
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From results of test 2.1, estimate conditions to solid state polymerize pellets to achieve 0.80 ± 0.02 dl/gm IV. Solid state polymerize Sample A2, B2, and C2 pellets to make Samples A4, B4 and C4 solid stated pellets with an IV of 0.80 ± 0.02 dl/gm.

The investigator may withdraw material from the 15 hour test run in order to have the needed Sample A4, B4, and C4 material for additional testing provided the withdrawal does not disrupt the expected smooth progression of IV increase or alter color or other properties.

3.0 Pellet Tests.

3.1 IV drop Test

Property	Test Method	Critical Value
IV change on melting pellets	ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°C	<p>With properly dried Blends A1, B1, and C1 flakes or pellets, the difference of extrudate IV drop for Samples B2 and C2 compared to Sample A2 of less than or equal to 0.025 dl/gm should not be a problem, from greater than 0.025 to 0.040 needs study, and drop greater than 0.040 probably is a problem.</p> <p>Use weighted arithmetic averages of components to estimate IV of the mixtures in blends constituting Sample A1, B1, and C1.</p>



	<p>Appendix B describes moisture effects on IV. This may be important if the moisture levels of the blends differ by more than 10 ppm.</p>	<p>With properly dried Samples A4, B4, and C4 pellets after solid stating, the difference of extrudate IV drop for Samples B5 and C5 compared to Sample A5 of less than or equal 0.025 dl/gm should not be a problem, from 0.025 to 0.040 needs study, and drop greater than 0.040 probably is a problem.</p>
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3.1 Melting Point Test

Property	Test Method	Critical Value
Melting Point	DSC, 10° C/minute. On second melt after 1 st melt rapid quench to test amorphous material.	235 to 255°C for Samples A4, B4, and C4.

4.0 Plaque Tests

<p>Prepare amorphous plaques, 3 mm thick, from Sample A4, Sample B4, and Sample C4 pellets, dried with desiccated air at 320 F +/- 20F (160C +/- 12C) for 4 to 6 hours to achieve water content below 50 ppm. Plaques are Samples A5, B5, and C5.</p>

4.1 L* Test

Property	Test Method	Critical Value
L*	CIELAB 3 mm plaque, test in <u>transmission</u> , <u>absolute</u>	For <u>clear</u> PET, L* greater than 82 for Samples A5, B5, C5.

4.2 b* Test

Property	Test Method	Critical Value
b*	CIELAB 3 mm plaque, test in <u>transmission</u> , Δb^* calculated as b^* for Sample B5 minus b^* for Sample A5 and as b^* for Sample C5 minus b^* for Blend A5. ASTM D 1003-B	Δb^* less than 1.5 should not be a problem. Δb^* between 1.5 and 5.5 needs study and Δb^* over 5.5 is unsuitable for many applications.

4.3 Haze Test

Property	Test Method	Critical Value
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% Haze	3 mm thick sample, test in transmission, measured at 550 nm, ASTM D 1003-B	For Samples A5, B5, and C5 haze less than 9.5% should not be a problem. Haze between 9.5% and 14% needs study and haze over 14% is likely noticeable
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4.4 Specks Test

Property	Test Method	Critical Value
Black Specks	<p>3 mm x 2000 mm² to 2600 mm² plaque, (nominal 2 inches x 2 inches plaques).</p> <p>Prepare 50 plaques each from samples A5, B5, and C5, unfiltered. Plaques viewed without magnification from 12 inches away. Count any plaque with a speck greater than 0.015 inches as failed.</p>	<p>If failed A5 plaques total 0, 2 or fewer failed B5 plaques are acceptable. If 3 or more B5 plaques are failed and 2 or fewer C5 plaques are failed, repeat the complete test.</p> <p>If failed A5 plaques total 1, 4 or fewer failed B5 plaques are acceptable. If 5 or more B5 plaques are failed and 4 or fewer C5 plaques are failed, repeat the test.</p> <p>If failed A5 plaques total 2, 6 or fewer failed B5 plaques are acceptable. If 7 or more B5 plaques are failed and 6 or fewer C5 plaques are failed, repeat the test.</p> <p>If failed A5 plaques total 3, clean the extruder and repeat plaque preparation.</p>

5.0 General Comments

5.1 Other Issues

Recommended Guidelines:



No additional fuming, smoking, or odors during extrusion.
No sticking between flakes during drying.
No fouling of process equipment
No creation of unsafe conditions, such as increased fire potential.

5.2 Further testing

This guidance does not address the detailed questions about bottle making and performance, fiber making and performance, strapping making and performance, or sheet making and

performance. Those evaluations should be conducted after the innovator is satisfied that the innovation has met the intent of the guidance here offered.

5.3 Color Measurement

1. For PET PCR, color measurement must be made on the blend of materials that have undergone three meltings and a solid stating from test samples. This approximates the thermal history seen for bottle-to-bottle recycling.
2. Measure color in transmission for color and haze using 3 mm amorphous plaques.
3. Calibrate spectrophotometer to the manufacturer's recommendations.
4. 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.

The testing called for in this document is intentionally rigorous with regard to test concentrations of the innovation, 25 and 50%. APR's "Criteria to Consider When Evaluating the Recyclability of a PET Variant in the PET Bottle Stream" suggests the variant be evaluated at a multiple of the expected market penetration. The multiples suggested are between 2 to 10. A test at 5 times the expected developed market penetration is frequently used to reflect actual recycling impact.

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+® WL40A	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
European PET Bottle Platform					
Artenius Flow	CSD	0.84±0.02	85.0 min	<1.0	ASTM 6290
CEPSA Cepsa PET SR08					
Equipolymers Lighter C93		0.80±0.02		1.5 max	
Indorama RAMAPET N1		0.80±0.02		-1.5±1.5	CIE
M&G Clearfuf P82		0.80±0.02	80.0 min	+1 max	M&G

Appendix B

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 compared 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 H ₂ O	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 =

$$(\text{dry IV}) - 0.00075 * (1000000 / ((2000000 / ((\text{dry IV}) / 0.00075)^{(1/0.68)})) / 2 + (\text{ppm water} / 18))^{0.68}$$

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 Innovation extrudate 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 Innovation 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

Innovation (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 be subject of more study when the differences in IV are >0.025 to ≤0.040 dL/g.