



## **PET Bottle Applications Test PET-A-01**

The Association of Post Consumer Plastic Recyclers (APR) recognizes that packaging innovation drives the growth of bottles available for recycling and that growth in the supply of these 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 represents a tool to help the innovator understand the approximate effect of the innovation on PET plastic bottle recycling processes.

The APR encourages Innovators to perform comprehensive recycle evaluation studies on new innovation materials intended to be introduced into the PET bottle stream. This Applications Guidance Document describes the protocols to be followed to evaluate the mechanical recyclability of the following Innovation materials that are intended to be made into or incorporated onto PET bottles:

1. New PET Resins
2. Additives
3. Coatings
4. Labels
5. Adhesives
6. Multilayer resins

In particular, a comprehensive recycling evaluation is accomplished by following a step-wise process involving the evaluations of the innovation using Testing Protocols that have been developed by APR. APR recognizes accomplishment and allows upon petition for the Innovator to publicize that they have completed each step. The steps include:

- STEP 1    1.00 CRITICAL GUIDANCE DOCUMENT
  - 1.10 New Resin, Critical Guidance
  - 1.20 Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Protocol, Critical Guidance
  
- STEP 2    2.00 APPLICATIONS GUIDANCE DOCUMENT
  - 2.10 Bottle-To-Bottle Evaluation, (BtB)
  - 2.20 Bottle-To-Sheet Evaluation, (BtSh)
  - 2.30 Bottle-To-Strapping Evaluation, (BtSt)
  - 2.40 Bottle-To-Fiber Evaluation, (BtF)



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Upon the completion of all parts of STEP 2, meeting or exceeding all of the strictest guidance, APR would consider a petition for full Recycling Guidance Recognition.

The screening tool referred to above in STEP 1 as the HDPE Critical Guidance document (CGD) is intended to help identify possible technical challenges that a new Innovation might create for the HDPE recycle stream. An Innovator is requested to first test an innovation material following the CDG before proceeding to STEP 2 and the more extensive Bottle-to-Bottle, Bottle-to-Sheet, Bottle-to-Strapping, and Bottle-to-Fiber evaluations. When coupling the CGD screening protocol with a Bottle-to-End-Use evaluation, it is necessary to increase the quantity of the Control and Innovation materials used in the CGD protocol to produce sufficient material for making the final test bottles. The APR Bottle-to-Bottle recycle evaluation is aligned with the PETCORE Bottle-to-Bottle protocol so that an Innovator (under some conditions) might be able to satisfy the requirements of both protocols by careful selection of processing steps to insure that the requirements of each protocol are met. It should be noted that the PETCORE protocol requires the use of bottle flake made from an innovation material for hot caustic washing and currently does not accept the APR abbreviated New PET Resin protocol. An Innovator will meet the requirements of the APR protocol by starting with hot caustic washed flake as required in the PETCORE protocol.

The Bottle-to-End-Use protocols are sequential. To reduce overall testing costs, the latter protocols build on information gained from the earlier protocols. As such, some issues pertinent to the sheet applications are addressed in the Bottle-to-Bottle Protocol and some issues pertinent to the staple fiber applications are addressed in the Bottle-to-Bottle and Bottle-to-Sheet Protocols.

The guidance contained in this document does not include time as a variable. Innovations which include time as a factor will require additional considerations.

**THIS DOCUMENT IS NOT A SPECIFICATION AND DOES NOT IMPLY IN ITS DEFINITIONS, PROCEDURES, OR VALUES FITNESS FOR USE, MARKET ACCEPTABILITY, SAFETY, 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.



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**THE FOLLOWING PROTOCOLS 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 AND DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE.**

## 1.0 CRITICAL GUIDANCE DOCUMENT (CGD)

### Introduction

The CGD is intended to be a screening tool that can be used by Innovators to gain a quick understanding on the impact of their innovation on the PET recycle stream before proceeding to the more extensive Bottle-to-Bottle Protocol and to the other Applications Guidance protocols. While the CGD protocol is designed as a recycle screening protocol, the material that has been processed in the CGD study can then be used as the starting material to continue the recycle evaluation through a Bottle-to-Bottle study. For new PET resins, the Innovator is given an opportunity by the APR to simplify the recycle evaluation if the new resin can be shown to not discolor during a "pellet" wash as defined in the CGD. This can eliminate the need for the new resin to be made into bottles that are ground into flake and subjected to a hot water caustic wash for the CGD. However, the Innovator may also elect to begin the recycle evaluations by supplying the Innovation in bottle form for use in the CGD and the Bottle-to-Bottle protocols. These options are more fully described below. Be aware that the Bottle-to-Sheet protocol requires the use of bottle flake, not resin pellets.

### Testing Procedures

The procedures needed for performing the tests called for in the recycling protocols are listed below:

- Color and Haze By Transmission (ASTM D-1003-B)
- Intrinsic Viscosity By Solution (ASTM D4603)
- Acetaldehyde (ASTM F2013)
- Black Specks and Gels (see the Bottle-to-Sheet Protocol)
- Impact (ASTM 5420)
- Fluorescence (see the Bottle-to-Fiber Protocol)
- Bottle testing follows International Society of Beverage Technologists' test protocols.

### Testing Protocols

The detailed protocols to be followed are listed below and are described in detail in this document.

- 1.10 New Resin Bottle-to-Bottle Evaluation, Critical Guidance



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- 1.20 Additives, Coatings, Labels, Adhesives, and Multilayer Resins Bottle-to-Bottle Evaluation, Critical Guidance

## Control Resins

The virgin control resins that can be selected for use are listed below. The virgin PET control resins are the same for the Critical Guidance Document and this Applications Guidance Document. When evaluating a New PET Resin, the Innovator is requested to select a Control resin based upon its intended end-use application. These resins are to be used to make both the Control flake and the Innovation bottle flake that will contain the additive, coating, label, adhesive or multilayer resin for the recycle study.

<i>Low IV, Water Bottle Innovation Controls</i>	<i>CSD and Non-Water Bottle Innovation Controls</i>
<b>Auriga Polyclear® Splash 3301</b>	<b>Auriga Polyclear® Refresh 1101</b>
<b>M&amp;G Cleartuf® Turbo II</b>	<b>M&amp;G Cleartuf® MAX</b>
<b>DAK Laser+® W L40A</b>	<b>DAK Laser+® B90A</b>

Appendix A describes some characteristics of the control resins.

## Labels and Adhesives

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, the innovation samples can be made and processed without the presence of labels or adhesives.

### **1.10 New PET Resin Protocol Evaluation, Critical Guidance**

#### **Background**

A market penetration of 5% would be considered normal for a new resin. Assuming that there can be spot market instances where a resin may constitute a higher percentage of the recycle stream, a 25% study level (a 5x increase) will be evaluated. Additionally, a 50% study level is included for unusual circumstances that might be encountered; failure to meet the test requirements for the 50% level will not necessarily be considered as failing the protocol requirements.

A suitable Control resin should be selected from the APR acceptable resin list above as the Control and blend resin to be evaluated along with the new resin blend compositions. In order to determine if the abbreviated resin evaluation is acceptable, there should be a negligible change in surface yellowness induced by a hot caustic wash on the new resin when compared to the new resin before the hot caustic wash. The b\* color of the resin pellets should increase



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no more than 1 (more yellow) after the hot caustic wash. If the increase in yellowness is greater than 1, then the new PET resin should be made into bottles that are then ground into flake for use in the recycle studies. The evaluation protocol is shown on the "New PET Resin Evaluation Flow Schematic". Note that if the Critical Guidance Document is being used as a screening evaluation process only, the molding of plaques from the test materials will be the final evaluation step. However, if a Bottle-to-Bottle evaluation is to be performed, additional evaluation steps will need to be performed beyond the Critical Guidance Document to produce innovation content bottles for performance testing.

### **New PET Resin Test Protocol**

This evaluation protocol allows the use of resin pellets in place of bottle flake when performing the recycling study unless the new resin has a tendency to yellow significantly during the hot caustic wash. If the new resin is found to be acceptable as defined by minimal color increase during the wash, then both the Control and the new resin pellets will be subjected to two extrusion melt heat histories, solid state processing, and finally injection molded into 3mm plaques for testing. If the resin exceeds the degree of yellowing as defined above, then the new resin will be evaluated according to the ***Additives/Coatings/Labels/Adhesives/Multilayer Resin Evaluation Protocol*** which requires the new resin to be first made into bottles, which are then ground into flake and subjected to a hot caustic water wash.

For all extrusion and molding steps, the process should first be established on the Control resin and then used without changes for the new resin. Any required processing changes for the new resin content samples should be documented and reported. It is recognized that minor process changes may be needed and such changes will not be considered significant when judging the new resin's suitability for recycling.

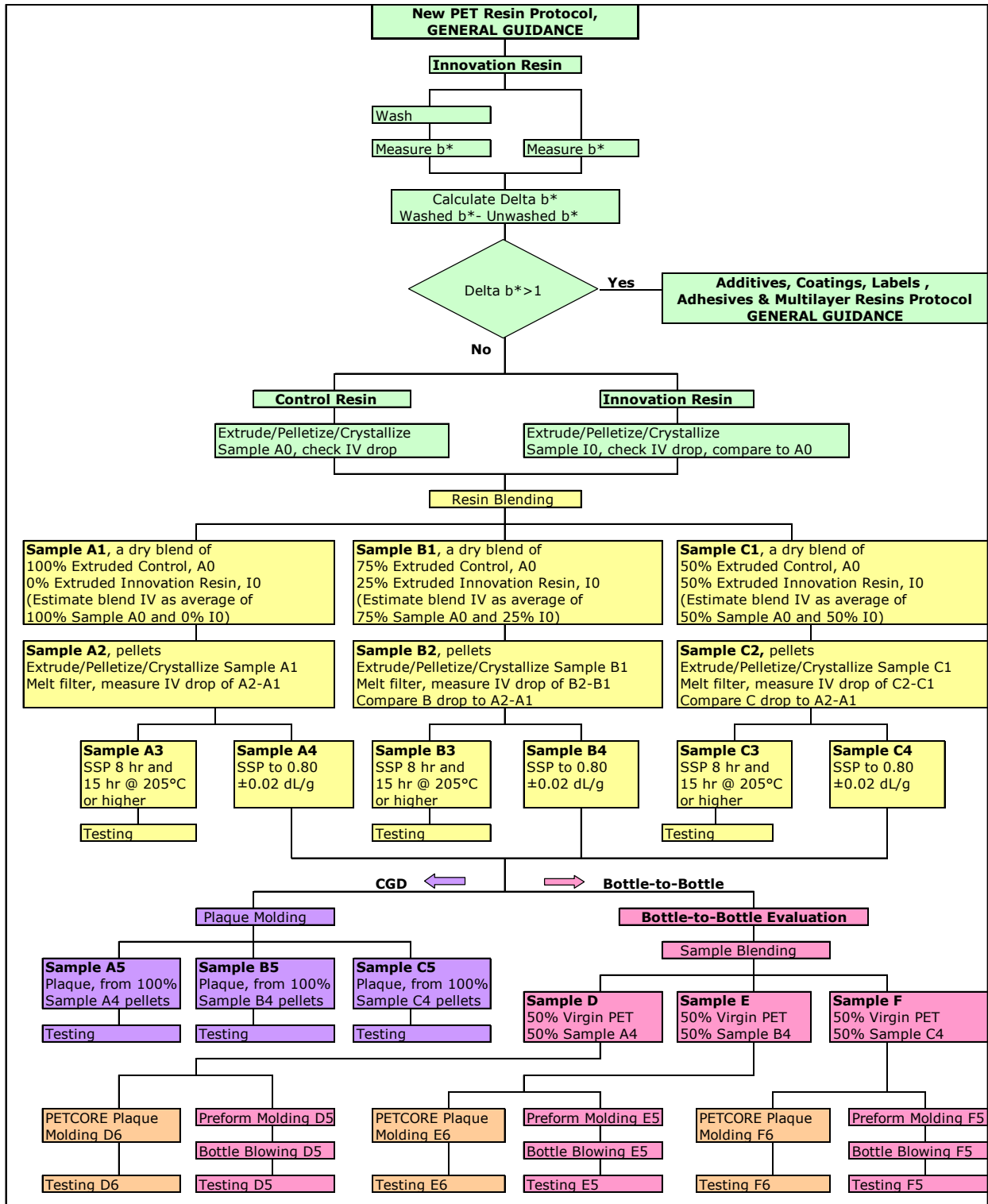
If the innovator is interested in investigating Bottle-to-Sheet applications, be aware that blown bottles are needed for that protocol.

The details of the new PET resin test protocol are intended to be coincident with the separately published PET Critical Guidance named "Critical Issues Guidance for Innovations".



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## New PET Resin Evaluation Flow Schematic





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## Evaluation Protocol Steps

### 1.1.1. New Resin Wash Trial

1. Measure by reflectance the  $b^*$  value of the New Resin pellets.
2. Wash 300g of the New Resin pellets using the APR Wash Protocol found in the “Protocol for Producing PET Flake for Evaluation”.
3. Dry the pellets at  $\leq 100^\circ\text{C}$  to remove surface moisture and measure the  $b^*$  value of the washed resin pellet samples.
4. Calculate the wash induced yellowness change ( $Db^*$ ) by subtracting the  $b^*$  of the washed sample from the unwashed sample for the New Resin.

#### ***1.1.1.1 Guidelines for Impact on Recyclability***

- a. An increase in yellowness in  $b^* > 1$ , requires that the New Resin follow the Additives/Coatings/Labels/Adhesives/Multilayer Resin Protocol.***
- b. If the increase in yellowness in  $b^*$  is  $\leq 1$ , proceed to the First Melt Heat History – Pellet Extrusion step.***

### 1.1.2 Pellet Extrusion- First Melt Heat History

1. Dry each resin at  $302^\circ\text{F} \pm 20^\circ\text{F}$  ( $150^\circ\text{C} \pm 12^\circ\text{C}$ ) or higher for at least 4 hours to  $< 50$  ppm moisture.
2. Extrude each resin.
3. Pelletize and crystallize the Control resin (referred to as “A0”) and New Resin (referred to as “I0”). Note if pellets stick to one another during crystallization.

#### ***1.1.2.1 Guidelines for Impact on Recyclability***

- a. The New Resin may be extruded under conditions that are optimum for this resin; the extrusion conditions do not have to be identical to the Control Resin.***
  - b. Resin pellets should not stick together during drying.***
  - c. Measure and report the IV drop of Samples A0 and I0 after extrusion.***
4. Proceed to "Sample Blending" in Section 1.2: **Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Protocol.**

## ***1.20 Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Protocol, Critical Guidance***

### **Background**

The control bottles required for this test must be made from one of the APR acceptable resins. The bottles can be supplied from commercial sources or blown as a separate test set and do not have to be of any special design or size. The additive, coating, label, adhesive or multilayer resin to be evaluated should be incorporated into or onto bottles made using the same control resin.



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Note: New Resins can also be evaluated following this protocol if this is the preferred path an Innovator wishes to follow. New Resins must proceed by this path if the yellowness change on hot caustic washing induces a  $b^*$  increase  $>1$  as indicated in Section 1.1, New PET Resin Protocol Evaluation.

### **Additives, Coatings, Labels, Adhesives and Multilayer Resins Test Protocol**

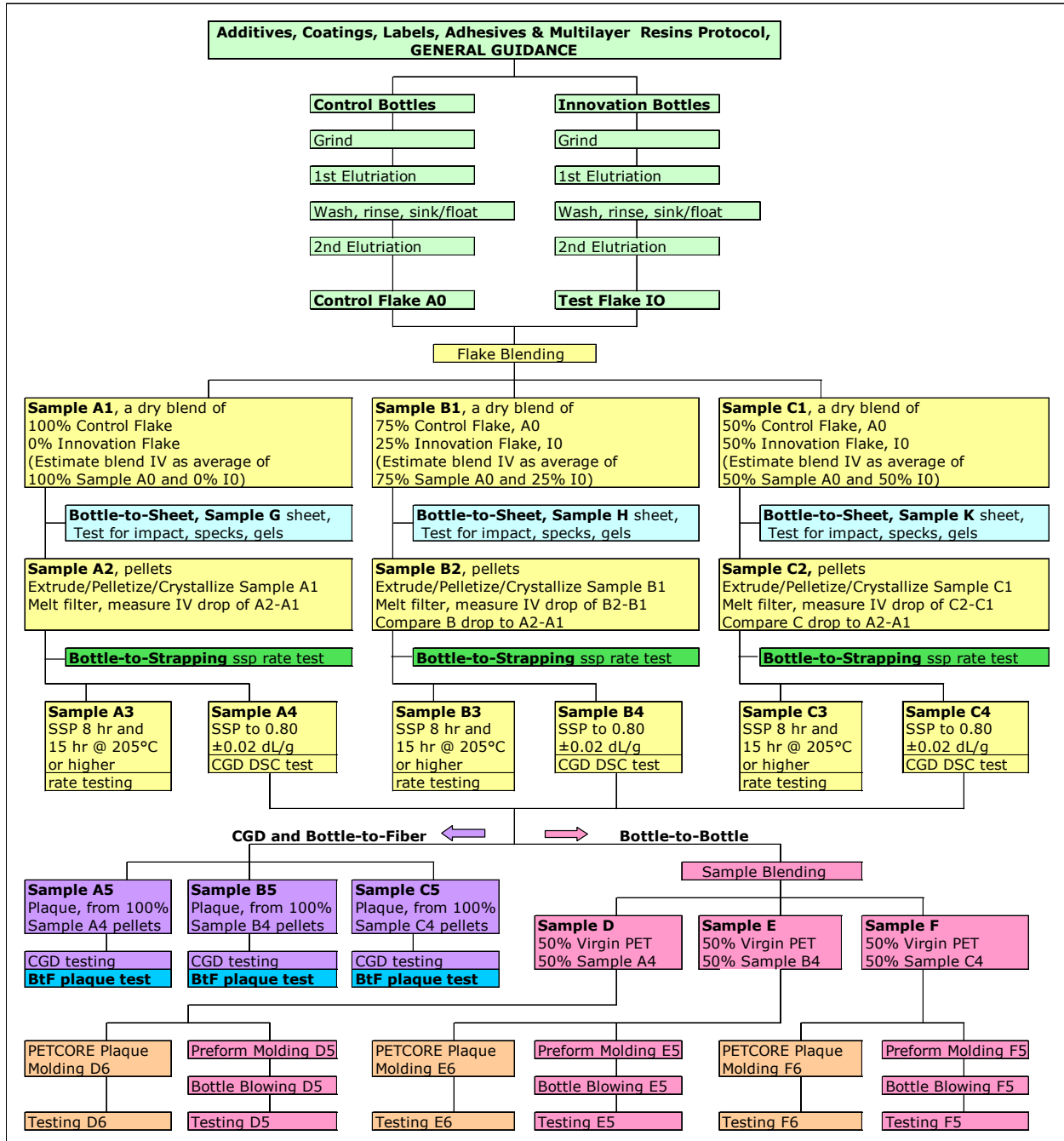
For all extrusion and molding steps, the process should first be established on the Control resin and then used without changes for the innovation materials. Any required processing changes for the innovation material content samples should be documented and reported. It is recognized that minor process changes may be needed and these will not be considered significant when judging the innovation materials suitability for recycling. This evaluation is intended to be coincident with the separately published PET Critical Guidance document.





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## Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Flow Schematic





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## Evaluation Protocol Steps

### 1.2.1 Control and Innovation/Variant Bottle Manufacture

1. Control bottles should be made from one of the acceptable resins listed above under Control Resins. The Innovation bottles should be made with this same resin incorporating the additive, coating or multilayer resin at the intended use level.

### 1.2.2 Flake Preparation

1. The Control bottles and Innovation bottles should be separately dry-ground to nominal ¼" to ½" 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. (Note: This step may be eliminated if these samples are wet ground or are intended to follow both the PETCORE and the APR Protocols. If omitted, more innovation failures may occur.)

### 1.2.3 Flake Wash

1. Prepare a wash solution of 0.3% by weight Triton X-100 (6.0 grams or 5.7 ml per 2,000 ml water) and 1.0% by weight caustic (20 grams NaOH per 2,000 ml water). Note: Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of caustic!
2. Mix bottle flake or pellets at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated (1,000 rpm with impeller 0.6 cm from bottom of wash container) water at 88+/-2°C for 15 minutes. Record composition of the wash solution.
  - a. The APR wash may be substituted by the PETCORE wash if both protocols are being followed with the recycling study. The PETCORE wash protocol is also an aggressive hot caustic wash.
3. After 15 minutes of washing, stop agitation and remove agitator. Remove heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables. Separate sinking solids from wash solution by pouring mixture through a strainer. Add sinking solids to room temperature rinse water at an approximate ratio of 500 grams sinking solids to 2 liters of water. Let stand for five minutes to allow remaining lights to float to the surface. Repeat sink/float step once again.
4. Transfer PET flakes to strainer, rinse flakes in cold running tap water while vigorously stirring the flakes for 10 minutes using the manual stirring bar. Drain the material. Air dry flake.



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5. A second air elutriation to remove light fractions with one pass and with less than 2% loss set for the Control Flake may be carried out on the dried washed flake. (Note: This step may be eliminated if these samples are intended to follow both the PETCORE and the APR Protocol. If omitted, more innovation failures may occur.)

### 1.2.4 Sample Blending

If the New Resin protocol is being followed, pellets will be blended at this step. Otherwise flake will be used to make the required Samples.

1. Create the following three test blends from the washed flake for study:

Sample A1:	100% Control Material	0% Innovation Material
Sample B1:	75% Control Material	25% Innovation Material
Sample C1:	50% Control Material	50% Innovation Material

### 1.2.5 Extrusion/Pelletization

1. Dry Samples A1, B1 and C1 at 320°F ± 20°F (160°C ± 12°C) or higher for at least 4 hours to <50 ppm moisture.
2. Extrude under conditions determined by the control sample using a 40/250/40 mesh screen pack.
3. Pelletize each of the sample blends giving each their second melt heat history.

#### **1.2.5.1 Guidelines comparing Samples B2 and C2 to Control A2**

- a. No more than 10% higher pressure required on extrusion of the Innovation/Variant compared to the Control.**
- b. The extrusion rate should be at least 375 gm/cm<sup>2</sup> per hour for 30 minutes.**
- c. No additional fuming, smoking or odors should be noticed when extruding the Innovation/Variant compared to the Control.**
- d. No additional fluorescence in the pellets as measured by the APR test protocol.**
- e. Resin pellets or flakes should not stick together during drying.**
- f. IV change on extrusion of Samples A1 to A2, B1 to B2, and C1 to C2**
  - i. An IV drop ≤0.025 dL/g should not be a problem**
  - ii. An IV drop of >0.025-0.040 dL/g needs study**
  - iii. An IV drop >0.040 dL/g probably is a problem**

**Note: The starting IV of the blend samples should be estimated as a blended weight average of the individual pellet IV's (the Control pellet or flake and the Innovation pellet or flake). Appendix B describes moisture effects on IV. This may be important if the moisture levels of the blends differ by more than 10 ppm.**

### 1.2.6 Solid State Processing

1. Solid state each Sample (A2, B2 and C2) at a minimum of 205°C for 15 hours. The initial starting IV of each of the samples should be obtained after pelletization. The SSP



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conditions of temperature and vacuum should be identical for Samples B2 and C2 compared to Sample A2. Solid stating times should be measured from  $T_0$ , which is defined as the time when the heat transfer fluid reaches 190°C.

2. At  $T_8$  hours, remove a sample and measure the IV
3. Stop the SSP at  $T_{15}$  hours and measure the IV.

### **1.2.6.1 Guidelines comparing Samples B3 and C3 to Control A3**

- a. **Samples B3 and C3 should be within 0.04 dL/g of Sample A3 after 8 hr.**
- b. **Samples B3 and C3 should be within 0.075 dL/g of Sample A3 after 15 hr.**

4. Solid state another set of Samples (A2, B2 and C2) under conditions suitable to raise the IV of the extruded pellets to  $0.80 \pm 0.02$  dL/g. NOTE: If large enough batch sizes of Samples A2, B2 and C2 are available, it is permissible to remove enough sample from the SSP device from the above experiments (Step 1) when each sample has reached the  $0.80 \pm 0.02$  dL/g target. Enough sample must be removed to allow for molding of 3mm plaques and, if necessary, for blending with virgin resin to make preforms and bottles for additional testing. It is also necessary to maintain a sufficient quantity of material in the SSP unit for continuing the solid stating experiment to 8 and 15 hours. Note: This sampling must be done quickly in order to not affect the IV build rate for the long SSP trial and under conditions to protect the 0.80 dL/g sample being removed.
5. Using DSC with a heating rate of 10°C/minute, measure the melting point of Blend Samples A4, B4 and C4 on the second melt after rapid quenching of the first melt.

### **1.2.6.2 Guidelines comparing Samples B4 and C4 to Control A4**

- a. **The melting point for Blend Samples A4, B4 and C4 should be 235° to 255°C on the second melt after rapid quenching of the first melt.**

### **1.2.7 Plaque Molding**

1. Dry the 0.80 dL/g Samples A4, B4 and C4 with desiccated air at 320°F  $\pm$  20°F (160°C  $\pm$  12°C) for 4 to 6 hours to achieve less than 50 ppm moisture content.
2. Injection mold 3mm plaques from the control Sample A4 first. Then mold 3mm plaques from Samples B4 and C4 under identical conditions if possible. If the processing conditions need to be changed, then these changes must be documented and reported.

### **1.2.7.1 Guidelines comparing Samples B5 and C5 to Control A5**

- a. **IV Change on injection molding of plaques**
  - i. **Plaque IV drop for Sample Blends B5 and C5 compared to Sample Blend A5 should be  $\leq 0.025$  dL/g.**
  - ii. **IV drop from  $>0.025$  to  $\leq 0.040$  dL/g needs further study**
  - iii. **and an IV drop  $>0.040$  dL/g probably is a problem.**
- b. **3mm Plaque Color for Sample Blends A5, B5 and C5**



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- i. *For clear PET, L\* should be >82 for Samples A5, B5, and C5*
- ii. *Db\* of Sample Blends B5 and C5 compared to A5*
  1. *<1.5 should not be a problem*
  2. *>1.5 to 5.5 needs further study*
  3. *>5.5 is unsuitable for many applications*
- c. *Haze for Sample Blends A5, B5 and C5*
  - i. *<9.5% should not be a problem*
  - ii. *9.5% to 14% needs further study*
  - iii. *>14% is likely noticeable*
- d. *Black Specs (plaques viewed without magnification)*
  - i. *Visual inspection on at least 50 plaques from 12 inches away, counting any plaque as failed if one or more specs seen greater than 0.015 inches. 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. 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. 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. If failed A5 plaques total 3, clean the extruder and repeat plaque preparation.*

### 1.2.8 General Issues:

***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.***

***NOTE: This completes the Critical Guidance Document requirements.***

The preceding is not a specification and does not imply in its definitions, procedures, or values fitness for use, market acceptability, or any guarantee or warranty. Inability of an innovative bottle to meet specific critical values does not imply recycling failure, but should be a clear message that a significant issue might exist under certain circumstances and mitigation of the issue may be needed to avoid degrading the value of the stream of recyclable bottles.

**MEETING THESE GUIDELINES DOES NOT OBLIGATE APR MEMBERS TO BUY BOTTLES CONTAINING THE INNOVATION. INNOVATORS ARE REQUESTED TO CONDUCT ADDITIONAL TESTING UNDER THIS AND OTHER GENERAL GUIDANCE DOCUMENTS**



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***These protocols do 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.***

## 2.0 APPLICATIONS GUIDANCE DOCUMENT

### Introduction

The Applications Guidance testing is to be sequential, following testing done per the Critical Guidance Document above. Issues important to sheet applications depend on successful testing on bottles and issues important to fibers applications depend on successful testing for bottle and sheet.

### Testing Protocols

The detailed protocols to be followed are listed below and are described in detail in this document.

- 2.10 Bottle-to-Bottle Protocol
- 2.20 Bottle-to-Sheet Protocol
- 2.30 Bottle-to-Strapping Protocol
- 2.40 Bottle-to-Fiber Protocol

### Control Resins

The control resins to be used are listed above.

**If a Bottle-to-Bottle study is being performed, then continue with the following steps.**

### **2.10 Bottle-to-Bottle Protocol, (BtB)**

The Bottle-to-Bottle evaluation program is designed to show processing and bottle performance differences between a control material and that control material containing recycle-content Innovation material. It is a **comparative** study that does not rely on the final blown bottles meeting absolute performance criteria.

Since the BtB program is designed to make 2L Carbonated Soft Drink (CSD) bottles, the optimal study will be one where the initial control resin selected for use in the CGD screening will be selected from the list of APR approved CSD and non-Water Bottle Innovation Control PET resins listed above. The Control CSD virgin resin called for in Step 1 below ideally would be identical to the CGD control resin.



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***It is recognized that if the Innovation being studied in the CGD was a non-CSD resin or an additive that was incorporated into a non-CSD resin, then the  $0.80 \pm 0.2$  dL/g material that is produced in the CGD study (Solid State Processing, Step 4), may not be ideal for CSD bottle performance.*** However this non-CSD base material can still be blended with a CSD control (selected from the table on page 2) to begin the BtB evaluation. When this is the case, it is important to recognize that the resulting control bottles and Innovation recycle-content bottles may not perform ideally in all of the CSD tests. Since the bottle test performance of the Innovation recycle-content bottles will be compared to the control bottles, it will still be possible to judge the Innovation's acceptability for the recycle stream if the bottle test criteria are met. Because the non-CSD control materials are currently found in the recycle stream, then any new materials similar to these that do not result in significant differences in recycle-content bottle performance are, therefore, also expected to be acceptable.

### 2.1.1 Injection Molding of Preforms and Plaques (needed if submitting data to PETCORE)

1. Create the following test blends of Samples A4, B4 and C4 by blending each at 50% with the Control CSD virgin resin.
  - Sample D: 50% Virgin PET, 50% Sample A4 (0% Innovation Bottle)
  - Sample E: 50% Virgin PET, 50% Sample B4 (12.5% Innovation Bottle)
  - Sample F: 50% Virgin PET, 50% Sample C4 (25% Innovation Bottle)
2. Dry the 0.80 dL/g Samples D, E and F with desiccated air at  $320^{\circ}\text{F} \pm 20^{\circ}\text{F}$  ( $160^{\circ}\text{C} \pm 12^{\circ}\text{C}$ ) for 4 to 6 hours to achieve less than 50 ppm moisture content.
3. Remove a portion of each sample, maintain dryness and use to mold plaques in Step 4.
4. Injection mold preforms from the control Sample D first. Then mold preforms from Samples E and F under identical conditions used for Sample D if possible. If the processing conditions need to be changed, then these changes must be documented and reported.

#### ***2.1.1.1 Guidelines comparing Samples E and F to Control D***

- a. There should be no significant processing changes needed for Samples E and F compared to D. Small differences in process settings are acceptable.***
- b. IV Change on injection molding of preforms***
  - i. IV drop for Sample E and F preforms compared to Sample D preforms should be  $\leq 0.025$  dL/g.***
  - ii. IV drop from  $>0.025$  to  $\leq 0.040$  dL/g needs further study***
  - iii. An IV drop  $>0.040$  dL/g probably is a problem.***
- c. Acetaldehyde concentration***
  - i. The preforms from Samples E and F should not exhibit an acetaldehyde increase of more than 35% compared to Sample D.***
- d. Black Specs, particulates or gels (preforms or plaques viewed without magnification)***





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- i. Visual inspection of 50 preforms from 12 inches away, counting any sample as failed if one or more specs seen greater than 0.015 inches. If failed D samples total 0, 2 or fewer failed E samples are acceptable. If 3 or more E samples are failed and 2 or fewer F samples are failed, repeat the complete test. If failed D samples total 1, 4 or fewer failed E samples are acceptable. If 5 or more E samples are failed and 4 or fewer F samples are failed, repeat the test. If failed D samples total 2, 6 or fewer failed E samples are acceptable. If 7 or more E samples are failed and 6 or fewer F samples are failed, repeat the test. If failed D samples total 3, clean the extruder and repeat sample preparation.*
5. Injection mold 3mm plaques from each of the 50:50 blend samples. (Note: this step is required only to meet PETCORE requirements.)

### 2.1.2 Blow Molding of Bottles

1. The preforms molded from Samples D, E and F should be blow molded into 2L straight-wall petaloid base CSD bottles. Sample D preforms should be blown first and followed by Samples E and F each blown under the identical conditions used for A if possible. If the processing conditions need to be changed, then these changes must be documented and reported.

#### 2.1.2.1 Guidelines comparing Samples E and F to Control D

- a. There should be no significant differences required when processing Samples E and F compared to D. Small differences in blow mold settings are acceptable.*
- b. A reduction in performance of bottles made from Samples E and F should not exceed the specified test limits when compared to Sample Bottles D for the following performance tests:*
  - i. Visual inspection for black specs, particulates or gels*
  - ii. Section weights*
  - iii. Brimful and fillpoint volumes*
  - iv. Burst strength*
  - v. Drop impact*
  - vi. Top Load*
  - vii. Stress Crack resistance*
  - viii. Thermal stability*
  - ix. CO<sub>2</sub> loss by FTIR or by Septum Test*





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## Bottle Test Guideline Criteria

Test	Measurement	Guidance
Appearance	Black specks	No more than indicated over Control D
	Particulates	No more than indicated over Control D
	Gels	No more than indicated over Control D
Color & Haze	L*	Report measurement, no guidance
	a*	Report measurement, no guidance
	b*	Report measurement, no guidance
	Haze	Report measurement, no guidance
Bottle Dimensions	Height	± 5% of Control D
	Upper Label Panel Diameter	± 5% of Control D
	Lower Label Panel Diameter	± 5% of Control D
Section Weights	Base	± 5% of Control D
	Panel	± 5% of Control D
	Shoulder	± 5% of Control D
Material Distribution	Base	Report measurement, no guidance
	Foot	Report measurement, no guidance
	Label	Report measurement, no guidance
	Shoulder	Report measurement, no guidance
Total Bottle Weight	Weight	± 5% of Control D
Capacity	Brimful	± 5% of Control D
	Fillpoint	± 5% of Control D
Burst Strength	Burst Pressure	Less than 10% decrease from Control D
Top Load	Max. Load Empty	Less than a 10% decrease from Control D
Drop Impact	40°F, Bottle axis vertical	No more than 1 additional failure than the Control D
	40°F Bottle axis horizontal	No more than 1 additional failure than the Control D
Stress Crack Resistance	Average Time	<25% reduction compared to Control D: No problem 25%-50% reduction: Needs further study >50% reduction: May be problematic
Shelf-Life	CO2 Loss by FTIR	No more than a 5% decrease compared to the Control D
	Septum Test	No more than a 5% decrease compared to the Control D
Thermal Stability	Height	No more than 5% increase over Control D



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Change in Fill Line	No more than 5% increase over Control D
Upper Panel Increase	No more than 5% increase over Control D
Lower Panel Increase	No more than 5% increase over Control D
Number of Rockers	0

Bottle test methods are available with membership from the International Society of Beverage Technologists, a global, non-commercial, non-profit technical society of beverage professionals, <http://www.bevtech.org/>.

### 2.1.3 Conclusion. PET Bottle-to-Bottle Protocol

A New Resin, Additive, Coating, Label, Adhesive or Multilayer Resin that meets all of the above test criteria for Sample C (50% innovation, as part of the Critical Guidance in Section 1.20) and Sample F (25% innovation, as part of Section 2.10) would be considered acceptable for introduction into the recycle stream for bottle making. The Innovation material may still be considered acceptable for bottle making if the Sample F test criteria are not all met but all of the Sample C and Sample E test criteria are met and the Innovation material under evaluation is not expected to be present in the recycle stream at the 25% innovation concentration of Sample F even in localized recycling environments. If most, but not all, of the above test criteria are met for Samples C and F, further explanation or testing may be required to demonstrate acceptability for recycling.

**If a Bottle-to-Sheet study is being performed, then continue with the following steps.**

### **2.20 Bottle-to-Sheet Protocol, (BtSh)**

Today, considerable amount of recycled bottle flake is currently converted into sheet. Most of the sheet is thermoformed and is used in many applications such as food, electronic, pharmaceutical, and other types of packaging.

Recycle content can range from 0 to 100% of bottle flake for any of these applications. The recycled material can be in the form of bottle flake or pellets. Most applications do not require the material to be solid stated. The most critical case is then the use of 100% bottle flake.

In the Critical Guidance Document, CGD, we have two possible flow diagrams. The first flow diagram is the New PET Resin Evaluation Flow Schematic for which the  $\Delta b^*$  value is measured after a pellet caustic wash. If the  $\Delta b^*$  between washed and unwashed pellets is less than 1.0, the pellets are re-extruded and blended to create sample A1, B1 and C1. The second flow diagram is

The Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Flow Schematic



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for all other types of innovations (additives, coatings, labels, etc), including the case where the above  $\Delta b^*$  is larger than 1.0. In this second flow diagram, bottles are blown (either with the innovation or without the innovation and perhaps using control resin) and the clean flake generated from those bottles is used to create sample A1, B1 and C1.

In the scope of this Bottle-to-Sheet Protocol, the test sample blends, which will be converted into sheet, needs to be the A1, B1 and C1 samples created from the second flow diagram. Thus, even for the case of innovation resins, the flow diagram wherein bottle making, bottle grinding, and washing is done to generate flake should be used. The reason for this is to simulate the sheet extrusion process as much as possible. The melting of the material in the extruder can have a significant influence on the process and the difference in shape between a pellet and a flake is a major factor. Furthermore, crystallizing and drying a pellet or a flake could generate some different types of problems. Thus, the test samples should be in the form of flake not pellets.

The Bottle-to-Sheet evaluation program is designed to show processing and unoriented sheet performance differences between a control material and that control material containing recycle-content Innovation material. It is a **comparative** study that does not rely on the final sheet meeting absolute performance criteria.

### 2.2.1 Sheet extrusion

1. Flake obtained from the Additives, Coatings, Labels, Adhesives and Multilayer Resins Test Sample Protocol will be used to make the required Samples.

Create the following three test blends from the washed flake for study:

Sample A1:	100% Control Bottle Flake	0% Innovation Bottle Flake
Sample B1:	75% Control Bottle Flake	25% Innovation Bottle Flake
Sample C1:	50% Control Bottle Flake	50% Innovation Bottle Flake

2. Flake should be dried with desiccated air at 320°F  $\pm$ 20°F (160°C  $\pm$  12°C) for 4 to 6 hours to achieve less than 50 ppm moisture content.
3. Extrusion should be done using standard process temperatures, 500 °F to 575°F (260 °C to 302°C). Melt filtration should be done using a 40/150/40 mesh screen pack. (Note, this is a more coarse filtration than other testing.) This represents minimum filtration level currently used for sheet extrusion. Sheet thickness should be 0.015 inches (0.38 mm). Sheet samples should be labelled
  - Sample G (control, made from Sample A1 flake),
  - Sample H (made from Sample B1 flake, 25% innovation bottle) and
  - Sample K (made from Sample C1 flake, 50% innovation bottle).



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When using small extruder screw diameter, feeding problems or amperage problems could occur due to the low bulk density of the flake material. In this case, 50% of the blended material could be grinded a second time to reduce the flake size. This fine grinded material would then be remix with the other 50% to be extruded into sheet.

A minimum quantity of 30 lbs of material should be extruded for each sample in order to evaluate the above process characteristics

### 2.2.1.1 Guidelines comparing Samples H and K to Control G

- a. *There should be no significant differences required when producing samples H and K compared to G. Small differences in process settings are acceptable when producing samples H and K compared to G. Note differences.*
- b. *The following process characteristics should be compared for Samples H and K vs. Control Sample G:*
  - *extruder amps (maximum  $\pm$  10% difference)*
  - *melt drop between die and roll stack nip (no die drool or blowouts)*
  - *bank stability (no substantial change)*
  - *fuming (no increase)*
  - *roll plate out (no increase)*
  - *general sheet optical quality (no substantial visual difference)*

### 2.2.2. Sheet evaluation

#### 2.2.2.1. Impact Test

Property	Test Method	Guidance
Impact test on sheet Samples G, H, and K	ASTM-5420, geometry GC	Results for Samples H and K should be within $\pm$ 10% of result for sample G.

#### 2.2.2.2. Gel Test

Property	Test Method	Guidance
Gel count on sheet samples G, H and K	Gel count on 50 sheet samples of each 2000 to 2600mm <sup>2</sup> .	If failed G sample total 0, 2 or fewer failed H samples are acceptable. If 3 or more H samples are failed and 2 or fewer K samples are failed, repeat the complete test.



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	Viewed without magnification from 12 inches away. Count any sample with a defect greater than 0.015 inches as failed.	<p>If failed G samples total 1, 4 or fewer failed H samples are acceptable. If 5 or more H samples are failed and 4 or fewer K samples are failed, repeat the test.</p> <p>If failed G samples total 2, 6 or fewer failed H samples are acceptable. If 7 or more H samples are failed and 6 or fewer K samples are failed, repeat the test.</p> <p>If failed G total 3, clean the extruder and repeat plaque preparation.</p>
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### 2.2.2.3. Speck Test

Property	Test Method	Guidance
Black specks count on sheet samples G, H, and K	<p>Black specks count on 50 sheet samples of each 2000 to 2600mm<sup>2</sup>.</p> <p>Viewed without magnification from 12 inches away. Count any sample with a speck greater than 0.015 inches as failed.</p>	<p>If failed G samples total 0, 2 or fewer failed H samples are acceptable. If 3 or more H samples plaques are failed and 2 or fewer K samples are failed, repeat the complete test.</p> <p>If failed G samples total 1, 4 or fewer failed H samples are acceptable. If 5 or more H samples are failed and 4 or fewer K samples are failed, repeat the test.</p> <p>If failed G samples total 2, 6 or fewer failed H samples are acceptable. If 7 or more H samples are failed and 6 or fewer K samples are failed, repeat the test.</p> <p>If failed G samples total 3, clean the extruder and repeat plaque preparation.</p>

### 2.2.3 Conclusion. PET Bottle-to-Sheet Protocol

A New Resin, Additive, Coating, Label, Adhesive or Multilayer Resin that meets all of the above test criteria for Sample C (50% innovation, as part of the Critical Guidance in Section 1.20) and Sample K (25% innovation, as part of Section 2.20) would be considered acceptable



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for introduction into the recycle stream for sheet making. The Innovation material may still be considered acceptable for sheet making if the Sample K test criteria are not all met but all of the Sample C and Sample H test criteria are met and the Innovation material under evaluation is not expected to be present in the recycle stream at the 50% innovation concentration of Sample K even in localized recycling environments. If most, but not all, of the above test criteria are met for Samples C and K, further explanation or testing may be required to demonstrate acceptability for recycling.

**If a Bottle-to-Strapping study is being performed, then continue with the following steps.**

### **2.30 Bottle-to-Strapping Protocol (BtSt)**

Strapping is a high performance product made from high molecular weight PET. High tensile strength needed to hold items in place is achieved by the orientation of high molecular weight resin. Strapping manufacturers consider that if PET is suitable for making biaxially oriented bottles and can be solid state polymerized to a target intrinsic viscosity, a measure of molecular weight, that it will be suitable for making strapping. The target intrinsic viscosity, ItV, is 0.95 dl/gram when measured by solution viscosity.

The Critical Guidance Document considers solid stating performance as part of the protocol and has incorporated within its guidelines provisions for performance at 15 hours of solid stating as a relative comparison between innovation samples and control samples. The Critical Guidance Document does not define an absolute ItV value to be achieved.

The strapping industry recognizes that variation occurs in recycled PET including the solid stating rates of various resins, the influences of additives, the activity of solid stating catalysts after the life cycle of the bottle, and the ItV of the cleaned flake product. As recognized in the Control Resins definition, there are two populations of intrinsic viscosity for PET bottles.

This Bottle-to-Strapping Protocol calls for the innovation bottle sample to demonstrate the ability of achieve a minimum intrinsic viscosity after a defined number of hours of solid state polymerization. Further testing to web extrusion and orientation are not needed. It is suggested for purposes of economy that the Bottle-to-Strapping solid stating examination be conducted as an extension of the Critical Guidance solid stating examination.

#### **2.3.1 Sample**

##### **Sample Blending and Preparation**

If the New Resin protocol is being followed, pellets will be blended at this step. Otherwise flake will be used to make the required Samples.



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1. Create the following three test blends from the washed flake for study:

Sample A1:	100% Control Material	0% Innovation Material
Sample B1:	75% Control Material	25% Innovation Material
Sample C1:	50% Control Material	50% Innovation Material
2. Dry Samples A1, B1 and C1 at 320°F ± 20°F (160°C ± 12°C) for at least 4 hours to <50 ppm moisture.
  1. Extrude under conditions determined by the control sample using a 40/250/40 mesh screen pack.
  2. Pelletize each of the sample blends. These are the samples prepared for the Critical Guidance examination
    - Sample A2, pellets made of Sample A1, control
    - Sample B2, pellets made of Sample B1, 25% innovation material
    - Sample C2, pellets made of Sample C1, 50% innovation material

The Critical Guidance Document (CGD) calls for the ItV of the Innovation Blends to be within 0.04 dl/g of the control after 8 hours and 0.075 dl/g of the control after 15 hours of solid stating at a temperature of at least 205°C and full vacuum.

In considering the practicality of laboratory testing and minimizing test expense, we suggest the investigator collect Critical Guidance data and samples as dictated by that protocol. Extend the solid state polymerization for all three samples for 39 hours to demonstrate attainment of 0.95 dl/gm ItV. 39 hours reflects the experience of the industry and should be readily met by favorable innovations and control resins.

### 2.3.1.1 Test Protocol Schedule Suggestion

- Load rotary vacuum dryer at noon, Day 1. Set temperature and pull vacuum.
- At 3:00pm, heating fluid temperature reaches minimum 190°C. Start time 0, Day 1
- 8 hr sampling is taken at 11:00pm, Day 1 (may require two people for safety, Estimate time to achieve 0.80 dl/gm for test material preparation for Critical Guidance evaluation and Bottle-to-Bottle Protocol evaluation or conduct second solid state polymerization to make sample. Collect sample for Critical Guidance solid stating evaluation
- 15 hr sampling taken at 6:00 am, Day 2  
Collect sample for Critical Guidance solid stating evaluation  
Measure ItV and estimate solid stating rate and time to 0.95 dl/gm for each sample.
- 22 hr sampling taken at 1:00pm, Day 2



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Measure ItV and estimate solid stating rate and time to 0.95 dl/gm for each sample.

- 26 hr sampling taken at 5:00pm, Day 2  
Collect sample and measure ItV based on earlier estimates of needed time.
- 32 hr sampling taken at 11:00pm, Day 2  
Collect sample and measure ItV based on earlier estimates of needed time.
- 39 hr sampling taken at 6:00am, Day 3  
Collect final sample and measure ItV if 0.95 dl/gm not already achieved.  
Samples now defined as **Samples A2<sub>39</sub>, B2<sub>39</sub> and C2<sub>39</sub>**

### **2.3.2 Guidelines comparing Samples B2<sub>39</sub> and C2<sub>39</sub> to Control A2<sub>39</sub>**

- a. There should be no significant differences in operating parameters (vacuum or dry inert gas, heat transfer fluid temperature and flow rate) between tests for the various samples. Note differences.***
- g. All samples after as many as 39 hours of solid stating treatment should exhibit intrinsic viscosities at or above 0.95 dl/gm.***
- h. Failure of the Sample A2<sub>39</sub> to exceed 0.95 dl/gm indicates the process conditions were not adequate.***

### **2.3.3 Conclusion. PET Bottle-to-Strapping Protocol**

A New Resin, Additive, Coating, Label, Adhesive or Multilayer Resin that meets all of the above test criteria for Sample C (50% innovation, as part of the Critical Guidance in Section 1.20) and Sample C2<sub>39</sub> (50% Innovation, as part of Section 2.30 ) would be considered acceptable for introduction into the recycle stream for strapping. The Innovation material may still be considered acceptable for strapping if the Sample C2<sub>39</sub> test criteria are not all met but all of the Sample B2<sub>39</sub> test criteria are met and the Innovation material under evaluation is not expected to be present in the recycle stream at the 50% innovation concentration of Sample C2<sub>39</sub> even in localized recycling environments. If the above test criteria are not met for either Samples B2<sub>39</sub> and C2<sub>39</sub>, further explanation or testing may be required to demonstrate acceptability for recycling.

**If a Bottle-to-Fiber study is being performed, then continue with the following steps.**

### **2.40 Bottle-to-Fiber Protocol (BtF)**

Fiber making, primarily but not exclusively staple fiber, is a major end use for recycled PET. The polymer requirements for staple fiber are met or exceeded by the requirements to make bottles and sheet. The fiber concerns for filterable contamination are met by the PET Critical





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Guidance Document filtration analysis. Fiber concerns about ability to heat set fibers are addressed in the Critical Guidance observation about non-sticking in dryers and the ability to make good bottles. Fiber concerns about gels and spinning defects are addressed by the limits in Bottle-to-Sheet testing. Fiber concerns about modulus and tenacity are met by the ability to make satisfactory bottles in the Bottles-to-Bottle protocol. Fluorescence is one area of concern for fiber not addressed in other testing. Bottles that contain fluorescing resins or additives can be disruptive to the use of those postconsumer bottles for fiber. Quantifying fluorescence is beyond this protocol. Innovation samples should not have a substantial increase in fluorescence compared to control samples. Photographic evidence can show lack of increased fluorescence.

## 2.4.1 Samples

The Critical Guidance Samples A5, B5, and C5 plaques can be used to examine fluorescence.

## 2.4.2 Testing

### 2.4.2.1 Fluorescence

Property	Guidance
Fluorescence	For plaque Samples B5 and C5, no substantial increase of emission fluorescence intensity for excitation or absorption wavelength between 320 and 390 nm compared to the fluorescence intensity for Sample A5. Photographic evidence is sufficient.

### 2.4.3 Conclusion. PET Bottle-to-Fiber Protocol

A New Resin, Additive, Coating, Label, Adhesive or Multilayer Resin that meets all of the above test criteria for Sample C (50% innovation, as part of the Critical Guidance in Section 1.20) and Sample C5 (50% Innovation, as part of Section 2.40) would be considered acceptable for introduction into the recycle stream for staple if the innovation has also been judged acceptable for making bottles and making sheet. The Innovation material may still be considered acceptable for staple if the Sample C5 test criteria are not all met but all of the Sample B5 test criteria are met and the innovation is judged acceptable for making bottles and sheet and the Innovation material under evaluation is not expected to be present in the recycle stream at the 50% innovation concentration even in localized recycling environments. If the above test criteria are not met for either Sample B5 or Sample C5, further explanation or testing may be required to demonstrate acceptability for recycling.



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## **Appendix A** **Control resins**

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

<b>APR Control Resin Listing</b>	<b>Type</b>	<b>IV</b>	<b>L*</b>	<b>b*</b>	<b>Color Test Method</b>
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
<b>European PET Bottle Platform</b>					
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.



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Moisture Correction Values				
	Dryest sample IV			
	0.70	0.75	0.80	0.85
Delta ppm H <sub>2</sub> 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 =

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

**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.