Critical Guidance Protocol for Clear PET Articles with Labels and Closures

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Introduction – Scope, significance and use

This is a comprehensive laboratory scale evaluation that can be used to assess the compatibility of PET packaging design features such as labels, closures, dispensers and attachments with common commercial scale recycling processes. This protocol is only applicable to clear PET articles. Product developers, as well as those who specify products, can employ this test to maintain and improve the quality and productivity of PET recycling.

The evaluation is conducted with molded articles made from clear PET and employs the packaging design feature of interest, (the "innovation material"). Clear PET refers to natural color PET with no colorants added at the molding process. Molded articles are most often expected to be PET packaging articles: injection stretch blow molded containers, extrusion blow molded containers, or thermoformed sheet products; but it also applies to any article that would be sorted as clear PET in the collection and sorting system.

Data developed by an independent third-party laboratory following this protocol can be used in petitions to APR's Critical Guidance Recognition Program. Petitions require data for a control material, and for the innovation articles blended with molded control articles.

In certain cases, before Critical Guidance Protocol test results can be submitted to APR for consideration of guidance recognition, all pre-requisite tests, including sortation potential protocols and degradable additives testing, must be passed and such evidence must be presented with any guidance applications. In addition, the Program Administrator may ask for additional exposure testing and performance testing as are pertinent to the innovation. To determine when pre-requisite testing is needed, please refer to the APR PET Rigid Design® Guide and the following test protocols and resources.

For products or innovations that employ metal decoration or which contain metal components:

- RES-SORT-03a Metal Sorting Resource
- SORT-S-03 Metal Sortation Protocol

For items less than 5 cm in 2 dimensions:

- RES-SORT-02 Size Sortation Resource
- SORT-S-02 Size Sortation Protocol
- SORT-P-00 Compression Practices

For dark colors and label coverage (see definition in Design® Guide):

- RES-SORT-01 NIR Sorting Resource
- SORT-S-01 NIR Sortation Protocol
- SORT-S-04 Color Sortation Protocol

For materials that might be employed or marketed as degradable additives for plastics and which might be expected to display time dependent behavior or change with environmental exposure where appearance or physical properties can change over time see APR Position Paper on Degradable Additives.

- APR Position Paper - Degradable Additives

This list is not inclusive. The Critical Guidance Protocol is not appropriate for package constructions that are not in alignment with the APR Design® Guide for Plastics Recycling text in cases where additional conditions are specified in the relevant Design® Guide section.

The final molded part of this evaluation is an injection molded plaque. Any impact of an innovation on specific end market applications such as sheet, bottles, or fiber are not fully addressed by the Critical Guidance evaluation. APR offers Applications Guidance Tests that can be used to evaluate any impact of an innovation on these specific end uses if there is reason to suspect that the innovation will impact the performance of recycled PET in these applications. Applications guidance can be conducted after completing the Critical Guidance Protocol.

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Although test data generated by a company is the property of that company and may be used for other purposes besides APR Critical Guidance Recognition, the test protocols are the property of APR and APR requires that they be used as written in their entirety. It should also be noted that partial test results that may under-report negative impacts from an innovation or design feature could misrepresent APR's intention or position and will be called out by APR when these incidents come to APR's attention.

Method summary and flow diagram

Molded articles used for control can either be made from a named control resin or by qualifying a resin for control using the PET Heat History and Color Evaluation Test.

Innovation articles are created according to the "Preparation of PET Articles for Evaluation" Practice which explains how to create articles for evaluation that include labels, closures or attachments. Innovation articles are usually mixed with control articles that do not have the design feature of interest; this mix is granulated to make a flake blend sample used in the evaluation.

The evaluation involves:

- Granulation, wash and elutriation of each set of control bottles and articles which employ the innovation.
- Extrusion and pelletization of both sets of material.
- Injection molding plaques from the pellets made from each material.

Experience shows that labels and closures are not likely to impact solid stating and so evaluation of IV build is not required in this evaluation.

Inks and adhesives can impact the costs of managing a wash system and waste disposal. Therefore, for innovations involving printing inks or labels with adhesives, it is recommended that the Wash Water Evaluation be conducted.

This procedure offers some optional steps that can be valuable in certain investigations. Examples are:

- The oven bake test for flake, and flake color evaluation to evaluate for contamination before extrusion of flake.
- A materials balance to confirm that materials are recovered as expected as sinking or floating solids, or in elutriation.

A flow diagram in Appendix I illustrates the testing steps.

Reference Documents

The following documents are referenced in this Critical Guidance Protocol:

PET-P-00: PET Standard Laboratory Practices
APR PET Screening Test Methods:
- PET-S-04: PET Package Materials Balance Test
- PET-S-01: Labels for PET - Wash Water Test
- PET-S-08: PET Flake Clumping Test
- PET-S-10: PET Flake Oven Bake Test
- PET-S-02: PET Flake or Pellet Discoloration Test
- PET-S-09: Testing of PET Plaques for Color, Haze and Inclusions

ASTM Methods
- ASTM D4603-18 Standard Test Method for Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET) by Glass Capillary Viscometer
- ASTM D1238 – 13 Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

Method steps

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The process steps below can be conducted with reference to the PET-P-00: PET Standard Laboratory Practices, PET-P-01 through PET-P-08, and the flow diagram in Appendix I:

1. Secure molded control articles.
2. Prepare innovation articles according to the practice for Preparation of PET Articles for Evaluation.
3. Create a blend of control and innovation articles as described in the Preparation of PET Articles for Evaluation
4. Separately grind control articles to create flake sample A and then grind the blend of control and innovation articles to create flake sample B.
5. Separately wash flake samples A and B.
6. When flake sample B contains labels or direct printed surfaces, retain a sample of wash water created from washing flake sample B for evaluation.
7. Elutriate flake samples A and B.

8. Desiccant dry flake samples A and B separately and then extrude and melt filter each sample to recover pellet samples A and B.
9. Crystallize the resulting pellets to create crystallized pellet samples A and B.
10. Desiccant dry and injection mold plaques from each of crystallized pellets A and B to create amorphous plaques A and B.

Measurements, report, and guidance values

Wash and elutriation evaluations

Property	Test method	APR Guidance Preferred values	Additional Guidance
Required values			
Clumping test with flake sample B	PET Flake Clumping Test, PET-S-08	<1 wt% retention on screen	
Wash water evaluation report	Labels for PET - Wash Water Test, PET-S-01	Observe and report only, no guidance values	Required only for label and direct printing evaluations
Optional values			
Flake bake test	PET Flake Oven Bake Test, PET-S-10		
Flake color	PET Flake or Pellet Discoloration Test, PET-S-02		
Materials balance	PET Package Materials Balance Test, PET-S-04		

Extrusion evaluation

IV loss - The table below calls for reporting the Extrusion IV loss when Flake samples are extruded to pellet samples. The following steps are used to report this value using Path 1 and samples A and B for illustration:

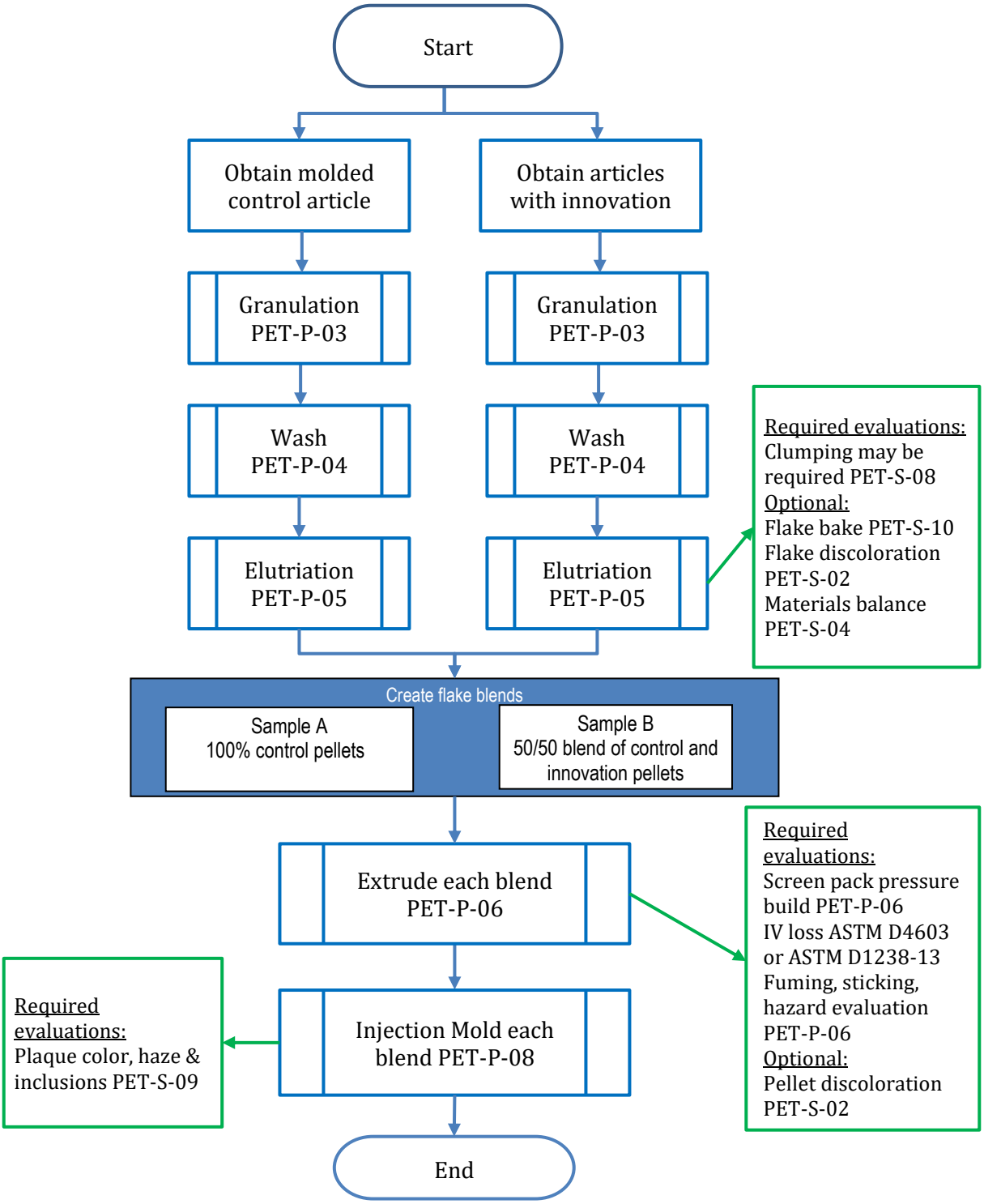
- Measure the IV of flake created from control articles as well as from innovation articles.
- Calculate the arithmetic mean IV of flake blend sample B and employ the mean value as the IV of the blend of control and innovation.
- Measure the IV of the resulting pellets for each blend after extrusion.
- Measure the IV loss for sample A with extrusion and call that value A'. This is the IV loss for the control.
- Measure the IV loss for Sample B with extrusion and call that value B'. This is the IV loss for the 50:50 blend of innovation and control.

Property	Test method	APR Guidance Preferred values
Required values		
IV loss	ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B	Difference in A' and B' is 0.025 units or less
Screen pack pressure build	Steps given in the Melt Filtration Practice, PET-P-06	End pressure is no greater than 25% over starting pressure value
Observation for fuming or odor at feed throat and die exit	Visual evaluation, no method	No unusual fuming or odors observed
Observation for material sticking in drier or feed throat	Visual evaluation, no method	No material sticks in drier of feed throat
Observation for impact on hazards or safety	Visual evaluation, method	No safety or hazard conditions observed
Optional values		
Pellet color	PET Flake or Pellet Discoloration Test, PET-S-02	

Evaluation of molded plaques

Property	Test method	APR Guidance Preferred values
Required values		
L value of plaques B	Testing of PET Plaques for Color, Haze and Inclusions, PET-S-09	>82
a* value of plaques A and B	PET-S-09	Less than 1.5 units difference between plaques A and B
b* value of plaques A and B	PET-S-09	Less than 1.5 units difference between A and B
% haze of plaques A and B	PET-S-09	Control not to exceed 9% value, and test not to exceed more than 10% units greater than control.
Inclusions and specks in plaques T5	PET-S-09	If A = 0; B is 2 or less If A = 1; B is 4 or less If A = 2; B is 6 or less
IV loss when pellets re molded to plaques	ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B	The value of IV loss for sample B is no greater than 0.025 units when compared to sample A

Appendix I Protocol Flow Diagram for Qualified PET Articles with Labels and Closures



DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	
2	April 11, 2019	Revised Haze Guidance Preferred Values as approved by PTC in March 2019
3	June 3, 2021	Added language clarifying need for pre-requisite testing
4	August 17, 2021	Added expanded disclaimer language
5	August 30, 2024	Changed naming convention for the sortation protocol tests from SORT-B-XX to SORT-S-XX; Updated hyperlinks to match new website