

Critical Guidance Protocol for Clear PET Articles with Labels and Closures

Document number – PET-CG-02

Creation or Revision Date: April 11, 2019

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.

The test is not appropriate for package constructions that will not pass through sorting steps at a Materials Recovery Facility and be collected into PET bales for recycling, nor for packages that are not in alignment with the APR Design® Guide for Plastics Recycling text. This test is not appropriate for materials that employ time dependent behavior where appearance or physical properties are expected to change over time. If it is questionable that the test article doesn’t meet these criteria, the appropriate APR sorting potential protocol or degradability test should be conducted prior to conducting this test.

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.

Disclaimer: *This document has been prepared by the Association of Plastic Recyclers as a service to the plastic industry to promote the most efficient use of the nation's plastic recycling infrastructure and to enhance the quality and quantity of recycled postconsumer plastic. The information in this document is offered without warranty of any kind, either expressed or implied, including WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, which are expressly disclaimed. 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. Participation in the Recognition Program is purely voluntary and does not guarantee compliance with any U.S. law or regulation or that a package or plastic article incorporating the innovation is recyclable or will be recycled.*

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:

APR PET Standard Laboratory Practices, PET-P-00

APR PET Screening Test Methods:

PET Package Materials Balance, PET-S-04

Labels for PET - Wash Water Evaluation, PET-S-01

PET Flake Clumping Evaluation, PET-S-08

PET Flake Oven Bake Evaluation, PET-S-10

Measurement of PET Flake or Pellet Discoloration, PET-S-02

Evaluation of PET Plaques for Color, Haze and Inclusions, PET-S-09

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

Safety Statement: APR Test and Practice documents do NOT CLAIM TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. These Tests and Practices may require the use of electrically powered equipment, heated equipment and molten polymers, rotating motors and drive assemblies, hydraulic powered equipment, high pressure air, and laboratory chemicals. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH AND FOLLOW APPROPRIATE SAFETY AND HEALTH PROCEDURES WHEN UNDERTAKING THESE TESTS AND PRACTICES THAT COMPLY WITH APPLICABLE FEDERAL, STATE AND LOCAL REGULATORY REQUIREMENTS. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance of these Tests and Practice documents by any party.

Sortation qualification - If there is any concern that the test article will not sort properly in either a materials recovery facility or plastic reclaimer, the applicable APR sortation potential test(s) must be conducted prior to proceeding with the critical guidance test. Criteria for determining this can be found in the APR Design Guide® For Plastics Recyclability. Specific factors to consider are small size, more two dimensional than three dimensional, containing metal or metalized inks and material components that could influence NIR sortation. Molded articles that do not provide a “preferred” level of sortation performance are not candidates for Critical Guidance review.

The process steps below can be conducted with reference to the PET 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 |
|-----------------------------------|--|---|---|
| <u>Required values</u> | | | |
| Clumping test with flake sample B | PET Flake Clumping Evaluation, PET-S-08 | <1 wt% retention on screen | |
| Wash water evaluation report | Labels for PET - Wash Water Evaluation, PET-S-01 | Observe and report only, no guidance values | Required only for label and direct printing evaluations |
| <u>Optional values</u> | | | |
| Flake bake test | PET Flake Oven Bake Evaluation, PET-S-10 | | |
| Flake color | Measurement of PET Flake or Pellet Discoloration, PET-S-02 | | |
| Materials balance | PET Package Materials Balance, 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:

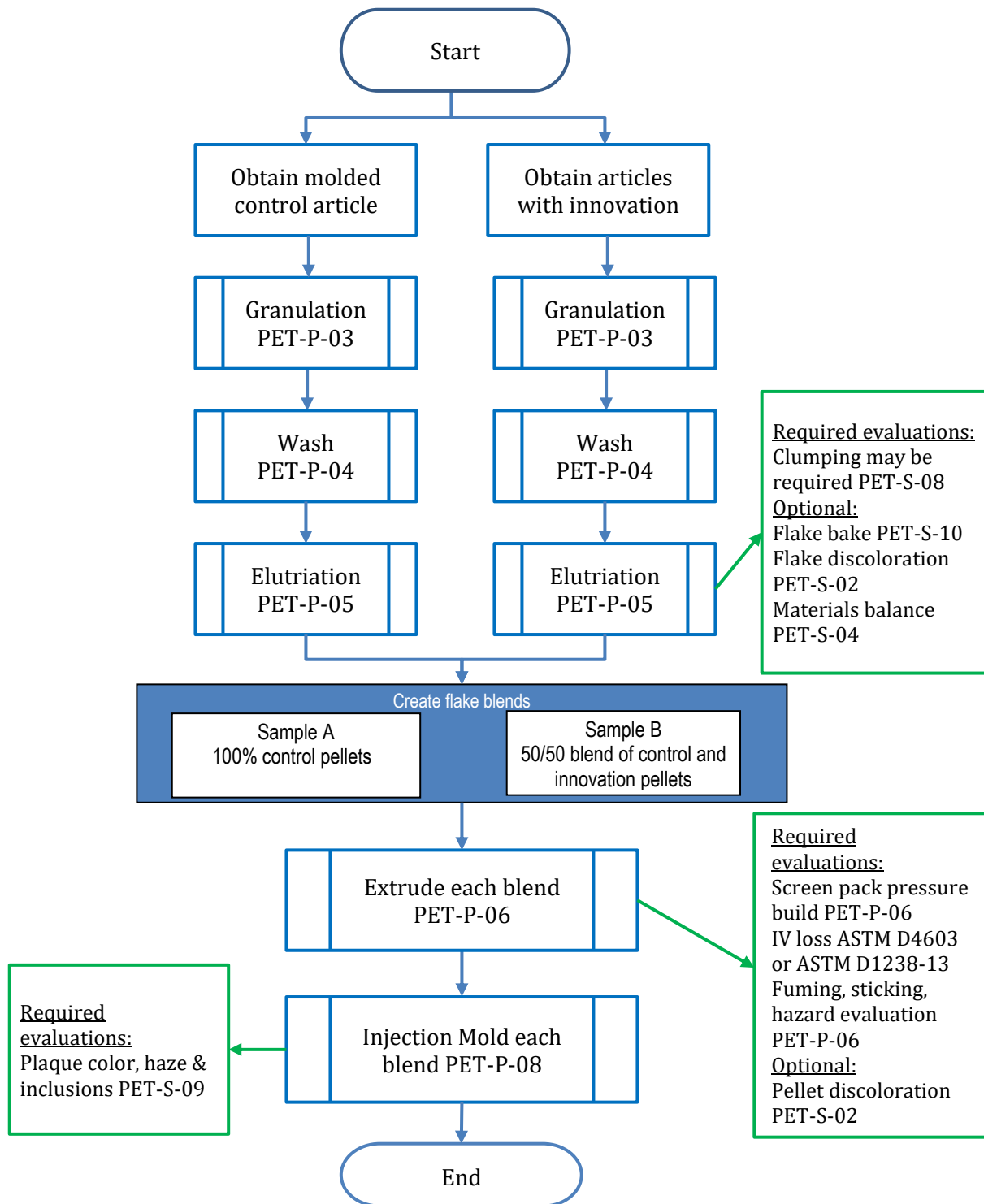
- Measure the IV of flake created from control articles as well as from innovation articles.
- Calculate the arithmetic mean IV of flake blend sample B and employ the mean value as the IV of the blend of control and innovation.
- Measure the IV of the resulting pellets for each blend after extrusion.
- 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 |
|--|--|--|
| <u>Required values</u> | | |
| 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-P06 | 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 |
| <u>Optional values</u> | | |
| Pellet color | Measurement of PET Flake or Pellet Discoloration, PET-S-02 | |

Evaluation of molded plaques

| Property | Test method | APR Guidance Preferred values |
|---|--|--|
| <u>Required values</u> | | |
| L value of plaques B | Evaluation 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 |
| | | |
| | | |