Benchmark Test for Clear PET Resin and Molded Articles

Introduction – Scope, significance and use

This is a laboratory scale evaluation that can be used to assess the compatibility of new PET resins, as well as molded PET articles that employ new resins, additives, coatings, multi-layer constructions or blends with some of the common commercial scale recycling process steps. This test is only applicable to clear articles. Clear PET refers to natural color PET with no colorants added during the molding process. 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 can be used with any pelletized PET resin or with molded articles made with PET that contain a design feature of interest, the “innovation material”. Molded articles are most often expected to be PET packages: 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.

This test has most value when used to understand how molded articles behave in the wash, float/sink and elutriation steps. The test is also valuable for understanding any impact of resin drying and melt heat history on IV drop as well as the formation of color, haze or inclusions in molded PET.

This test does not include an assessment of melt extrusion and melt filtration nor IV build with solid stating that are necessary components of the Critical Guidance Test for PET Resin and Molded Articles. This test does not consider the effects of package design on sortation in a material recovery facility or reclaiming process.

The test calls for a control material, and for the innovation material being tested in a blend at 50 wt% with a control resin. For information purposes, investigators can run the innovation material at other blend ratios with the control PET resin, if desired. The test method shows a 25/75 blend value for illustration.

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Method summary and flow diagrams

**Molded articles, Path 1** - This benchmark test provides two pathways for testing resins and molded articles. For molded articles with or without layers, coatings, additives, adhesives or blends, articles are made with the innovation material, and similar articles are also made solely with a control PET resin without the innovation for comparison. These articles are then separately:

- Granulated, washed, sink/floated, dried, and elutriated.
- Blends are created from the washed and elutriated flake. Required blends are 100% control material; and a 50/50 blend of control material and test material. Optional blends can be used if desired by the investigator. An example is given in the test method for a 75/25 blend of control with test material.
- These flake blends are crystallized if necessary, and then injection molded into plaques for evaluation.

Measurements are made during or after each of the process steps following the required test methods.

**Pelletized resins, Path 2** - For pelletized innovation PET resins a simpler path is available. The heat history of molding an article is modeled by extruding and re-pelletizing each of the control and test resins. Blends of control and test material for injection molding can be made without the need to wash and elutriate samples.

For both Paths there are optional tests that can be run to obtain additional information. A flow diagram is show in Appendix I and Appendix II.

**Reference Documents**

The following documents are referenced in this Protocol:

**APR PET Standard Laboratory Practices, PET-P-00**

**APR PET Screening Test Methods:**
- PET Package Materials Balance, PET-S-04
- 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 for Molded Articles – Path I

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Path 1 Method Steps

The following steps can be conducted with reference to the PET Practices PET-P-01 to PET-P-08 (included in document PET-P-00) as well as the flow diagram in Appendix I.

1. Obtain molded innovation articles and control articles to use in the evaluation. The amount of material will depend upon the equipment and scale used in each laboratory.
2. For each of the test and control articles, separately
   a. Granulate the articles.
   b. Wash the resulting flake including rinse and float/sink steps.
   c. Dry the flake.
   d. Elutriate the flake.
3. Prepare the following required blends:
   a. 100% control flake - Sample A
   b. 50/50 blend of control flake with flake from the innovation article – Sample B
4. Prepare any optional blends chosen by the investigator, for example: 75/25 blend of control flake and innovation flake – Sample C
5. Samples may be crystallized if necessary to prevent sticking when desiccant dried.
6. Samples may be ground to a finer size to feed well into an injection molding unit.
7. Desiccant dry each flake blend and injection mold the blends to create amorphous plaque samples: A plaques, B plaques and C plaques.

Method steps for Pelletized Resin – Path 2

Path 2 for resins

The following steps can be conducted with reference to the PET Practices PET-P-01 to PET-P-08 as well as the flow diagram in Appendix II.
1. Separately dry each of the control and innovation resins in a desiccant drier at 160° C. Then, extrude the pellets in an extruder with a strand die and recover the re-pelletized material. This step adds a drying and melt heat history to simulate making a molded article with the resins.

2. Crystallize the resin samples.

3. Prepare the following required blends from the re-pelletized samples:
   a. 100% control pellets – Sample A pellet blend
   b. 50/50 blend of control pellets with pellets from the test article – Sample B pellet blend
   c. Prepare any optional blends chosen by the investigator, for example: 75/25 blend of control pellets and test pellets – Sample C pellet blend

4. Desiccant dry each blend and injection mold the blends to create amorphous plaque samples: A plaques, B plaques and C plaques.

Measurements, Report and Guidance Values

Wash and elutriation evaluations

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
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<tbody>
<tr>
<td>Required values</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>PET flake clumping test when required</td>
<td>PET Flake Clumping Evaluation, PET-S-08</td>
<td>&lt;1 wt% retention on screen and foil for each of the un-weighted and weighted evaluations</td>
<td>Required when the following are involved: an adhesive or organic coating, a non-crystalline thermoplastic component, or a crystalline material with MP less than 225° C</td>
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<td>Optional values</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Flake bake test</td>
<td>PET Flake Oven Bake Evaluation, PET-S-10</td>
<td></td>
<td>Can reveal contamination before later evaluations</td>
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<tr>
<td>Flake color</td>
<td>Measurement of PET Flake or Pellet Discoloration, PET-S-02</td>
<td></td>
<td>Can reveal contamination before later evaluations</td>
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<tr>
<td>Materials balance</td>
<td>APR Wash Practice, PET-P-03, or PET Package Materials Balance, PET-S-04</td>
<td></td>
<td>Can reveal contamination before later evaluations</td>
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Plaque injection molding evaluations

IV loss - The table below calls for reporting the IV loss when samples are injection molded into plaques. 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 plaques for each blend after molding.
- Measure the IV loss for sample A with molding and call that value $A'$. This is the IV loss for the control.
- Measure the IV loss for Sample B with molding and call that value $B'$. This is the IV loss for the 50:50 blend of innovation and control.

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
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<tbody>
<tr>
<td>IV loss when molding plaques A</td>
<td>ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°,</td>
<td>Difference in $A'$ and $B'$ is 0.025 dg/l or less</td>
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<tr>
<td>and B</td>
<td>or ASTM D1238 – 13 method B</td>
<td></td>
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<tr>
<td>Observe drier and feed for any</td>
<td>Visual observation</td>
<td>No sticking or residue observed</td>
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<tr>
<td>sticking material or residue</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Observe injection process for</td>
<td>Visual observation</td>
<td>No unusual fumes or hazards observed</td>
</tr>
<tr>
<td>any unusual fumes or hazardous</td>
<td></td>
<td></td>
</tr>
<tr>
<td>conditions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L color value of Plaques B</td>
<td>Plaque Color Measurement, PET-S-09</td>
<td>&gt;82</td>
</tr>
<tr>
<td>a* value of plaques B</td>
<td>Plaque Color Measurement</td>
<td>Less than 1.5 units difference compared to Plaques A</td>
</tr>
<tr>
<td>b* value of plaques B</td>
<td>Plaque Color Measurement</td>
<td>Less than 1.5 units difference compared to Plaques A</td>
</tr>
<tr>
<td>% haze of plaques B</td>
<td>Plaque Color Measurement</td>
<td>Control not to exceed 9% value, and test not to exceed more than 10% units greater than control.</td>
</tr>
<tr>
<td>Inclusions and specks in 50</td>
<td>Plaque Color Measurement</td>
<td>If $A = 0$; $B$ is 2 or less</td>
</tr>
<tr>
<td>plaques B</td>
<td></td>
<td>If $A = 1$; $B$ is 4 or less</td>
</tr>
<tr>
<td></td>
<td></td>
<td>If $A = 2$; $B$ is 6 or less</td>
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Appendix I
Path 1: Benchmark Flow Diagram for clear molded articles that employ resins, additives, coatings, layers, adhesives or blends

Start

Obtain molded control article
Granulation PET-P-03
Wash PET-P-04
Elutriation PET-P-05

Obtain molded articles with innovation
Granulation PET-P-03
Wash PET-P-04
Elutriation PET-P-05

Create flake blends
Required 100% control flake Sample A
Required 50/50 blend of control and innovation flake Sample B
Optional 75/25 blend of control and innovation flake Sample C

Injection Mold each blend PET-P-08

End

Required evaluations:
- IV loss ASTM D4603 or ASTM D1238-13
- Fuming, sticking, hazard evaluation PET-P-06
- Plaque color, haze & inclusions PET-S-09

Optional:
- Flake bake PET-S-10
- Flakes discolored PET-S-02
- Materials balance PET-S-04
Appendix II
Path 2: Benchmark Flow Diagram for Clear PET Resins

Start

Obtain control PET resin
Obtain innovation PET resin

Extrude & pelletize to add heat history
Extrude & pelletize to add heat history

Create pellet blends
- Required
  - 100% control pellets
  - Sample A
- Required
  - 50/50 blend of control and innovation pellets
  - Sample B
- Optional
  - 75/25 blend of control and innovation pellets
  - Sample C

Injection Mold each blend PET-P-08

End

Required evaluations:
- IV loss ASTM D4603 or ASTM D1238-13
- Fuming, sticking, hazard evaluation PET-P-06
- Plaque color, haze & inclusions PET-S-09
### DOCUMENT VERSION HISTORY

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<th>Version</th>
<th>Publication Date</th>
<th>Revision notes</th>
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<td>1</td>
<td>November 16, 2018</td>
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<td>2</td>
<td>April 11, 2019</td>
<td>Revised Haze Guidance Preferred Values as approved by PTC in March 2019</td>
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