Benchmark Evaluation for Clear PET Articles with Labels and Closures

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Introduction – Scope, significance and use

This is a laboratory scale evaluation that can be used to assess the compatibility of PET packaging design features such as labels, closures, dispensers and attachments with some of the common commercial scale recycling process steps. This test 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 packages: injection stretch blow molded containers, extrusion blow molded containers, or thermoformed sheet products; but may include any article that would be sorted as clear PET in the collection and sorting system.

This test is 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 during injection molding on the formation of color, haze or inclusions in molded PET.

This test does not include an assessment of flake extrusion and melt filtration that is a necessary component of the Critical Guidance Protocol for PET Articles with Labels and Closures. This test does not consider the effects of package design on sortation in a MRF or reclaiming process.

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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.
- Injection molding plaques directly from washed flake made from each material.

Inks and adhesives can impact the costs of managing a wash system and waste disposal. Therefore, if inks or adhesives are observed in the wash water it is recommended that the Wash Water Evaluation for articles with labels and direct printing be performed.

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 injection molding of flake.
- A materials balance to confirm that materials are recovered as expected as sinking or floating solids.

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

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The following steps are taken with reference to the PET Practices, PET-P-01 through PET-P-08 (included in document PET-P-00) and the flow diagram shown in Appendix I:

1. Secure molded control articles.
2. Prepare these 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, adhesives or printed surfaces, retain a sample of wash water created from washing flake sample B for evaluation.
7. Elutriate flake samples A and B.
8. If necessary, crystallize flake samples A and B prior to desiccant drying to prevent any sticking in the drier.
9. If necessary to allow feeding to the injection molding unit, grind flake samples A and B to a finer flake size.
10. Desiccant dry and injection mold plaques from each of elutriated flake samples A and B to create amorphous plaques A and B.
Measurements, report, and guidance values

Wash and elutriation evaluations

<table>
<thead>
<tr>
<th>Property</th>
<th>Test method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Required values</td>
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<td></td>
<td></td>
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<tr>
<td>Clumping test with flake sample B</td>
<td>PET Flake Clumping Evaluation, PET-S-08</td>
<td>&lt;1 wt% retention on screen for each evaluation</td>
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<tr>
<td>Wash water evaluation report</td>
<td>Labels for PET - Wash Water Evaluation, PET-S-01</td>
<td>Required only for label and direct printing evaluations</td>
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<tr>
<td>Optional values</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>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>Can reveal contamination before later evaluations</td>
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<tr>
<td>Materials balance</td>
<td>PET Package Materials Balance, PET-S-04</td>
<td>Can reveal contamination before later evaluations</td>
<td></td>
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</table>

Evaluation of molded plaques

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 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>Test method</th>
<th>APR Guidance Preferred values</th>
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<tbody>
<tr>
<td>IV loss when molding plaques</td>
<td>ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B</td>
<td>Difference in A’ and B’ is 0.025 dg/l or less</td>
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<tr>
<td>L value of plaques B</td>
<td>Plaque Color Measurement, PET-S-09</td>
<td>&gt;82</td>
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<tr>
<td>a* value of plaques A and B</td>
<td>Plaque Color Measurement</td>
<td>Less than 1.5 units difference between plaques A and B</td>
</tr>
<tr>
<td>b* value of plaques A and B</td>
<td>Plaque Color Measurement</td>
<td>Less than 1.5 units difference between A and B</td>
</tr>
<tr>
<td>% haze of plaques A and 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>
</tbody>
</table>
| Inclusions and specks in plaques T5 | Plaque Color Measurement                          | If A = 0; B is 2 or less  
If A = 1; B is 4 or less  
If A = 2; B is 6 or less |
Appendix I Benchmark Flow Diagram
Natural PET Articles with Labels and Closures

Start

Obtain molded control article
Granulation PET-P-03
Wash PET-P-04
Elutriation PET-P-05
Injection Mold PET-P-08
End

Prepare molded articles with innovation
Granulation PET-P-03
Wash PET-P-04
Elutriation PET-P-05
Injection Mold PET-P-08

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

Required evaluations:
Clumping may be required PET-S-08
Optional:
Flake bake PET-S-10
Flake discoloration PET-S-02
Materials balance PET-S-04
## DOCUMENT VERSION HISTORY

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<th>Version</th>
<th>Publication Date</th>
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<tr>
<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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