Critical Guidance Protocol for HDPE Natural Bottles with Labels

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Introduction – Scope, significance and use

This is a comprehensive laboratory scale evaluation, or protocol, that can be used to assess the compatibility of HDPE rigid container packaging innovations with reclamation systems sourcing post-consumer material. For definitions, reference “Plastics Recycling Testing Terminology” at https://plasticsrecycling.org/apr-design-guide/resources/testing-terminology

This test is advised for HDPE natural homopolymer and natural copolymer bottles, jars, and jugs. This test can be used to evaluate the impact of labels and adhesives.

In APR Processing Practices and Test Protocols, the term “article” is used to mean the package submitted for testing (except in the special case where a resin pellet is tested and is referred to simply as “resin pellet”). The article incorporates the “innovation” or “design feature” which requires test data for an APR recyclability classification. Generally, an “innovation” is a modification or addition to a package construction that provides functional improvements and a “design feature” is an element of design geared toward ease of consumer use or marketing benefit. However, in APR documents the two terms may be used interchangeably.

Data developed by an independent third-party laboratory following this protocol can be used in petitions for APR’s Critical Guidance Recognition. Petitions require data for an innovative article compared to a control.

The test is not appropriate for package constructions that will not pass through APR’s Sorting Potential Protocols addressing NIR sortation, size, and metallic components. These benchmark tests, labeled SORT-B-01 through B-03, are found at https://plasticsrecycling.org/apr-design-guide/test-methods

The test is also not appropriate 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 whether the test article meets these criteria, the appropriate APR sorting potential protocol or degradability test should be conducted prior to conducting Critical Guidance Evaluation.
Method Summary

This Critical Guidance protocol provides two pathways for testing HDPE resins and articles with flow diagrams found on pages 8 and 9 for printing, labels, and adhesives. Controls are comprised of similar articles made solely with a control PE resin without the innovation. Reference document APR Polyolefin Standard Laboratory Processing Practices, O-P-00 through O-P-07 for complete details.

Path 1 - Path 1 is utilized for complete HDPE packages in the form of bottles, jars and jugs that include the innovation being tested. A flow diagram for Path 1, on page 8 illustrates that these articles are separately processed through these steps:

- Granulation
- Caustic Wash at 75°C with 0.5% NaOH and 0.3% surfactant.
- Drying
- Elutriation
- Blends created from the processed materials. APR Critical Guidance protocols require blends of 50% control and 50% innovation to be compared with 100% control material. Optional blends can be used if desired by the investigator at 75% control and 25% innovation blend.
- Blends dried, extruded and pelletized
- Pellets subjected to testing and evaluation
- Injection molding of test bars for ESCR testing is optional

Path 2 - Path 2 is utilized for testing resins and resin blends that incorporate the innovation being tested but have not been converted into package form. A flow diagram for Path 2, on page 9 illustrates that these articles are separately processed through these steps:

- Control and test dry blends processed for a single melt history to represent the initial production.
- Blends created from the processed materials. APR Critical Guidance protocols require blends of 50% control and 50% innovation to be compared with 100% control material. Optional blends can be used if desired by the investigator at 75% control and 25% innovation blend.
- Blends dried, extruded and pelletized
- Pellets subjected to testing and evaluation
- Injection molding of test bars for ESCR testing is optional
Reference Documents

The following documents are referenced in this Critical Guidance Protocol:

APR Polyolefin Standard Laboratory Processing Practices, O-P-01 through O-P-07

APR Screening Test Methods:
Polyolefin Packaging Article Sink or Float Evaluation, O-S-01

ASTM Pellet Test Methods:
ASTM D3418 Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
ASTM D1238 Melt Flow Rates of Thermoplastics by Extrusion Plastometer
ASTM D792 Density and Specific Gravity (Relative Density) of Plastics by Displacement
ASTM D1505 Density of Plastics by the Density-Gradient Technique
ASTM D5630 Ash Content in Plastics
ASTM D6980 Determination of Moisture in Plastics by Loss in Weight
ASTM D7399 Determination of the Amount of Polypropylene in Polypropylene/Low Density Polyethylene Mixtures Using Infrared Spectrophotometry
ASTM D6290 Color Determination of Plastic Pellets

Optional ASTM Test Methods:
ASTM D1693 Environmental Stress-Cracking of Ethylene Plastics
Method Steps for Extrusion Blends and Evaluation

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Path 1 Method Steps

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagram labeled Path 1, and details of each step are presented in the APR Polyolefin Standard Laboratory Processing Practices, O-P-01 through O-P-07 (included in document O-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.

1. Obtain control and test article to use in the evaluation for Path 1. For a list of acceptable control resins, refer to Practice O-P-01.
2. For each of the test and control articles for Path 1, separately:
   a. Granulate articles
   b. Sink-float the control and test articles per the Screening Test O-S-01
   c. Caustic Wash and drying
      i. at 75°C with 0.5% NaOH and 0.3% surfactant
   d. Elutriation of granulated material
3. Prepare the following required blends:
   a. 100% processed control - Sample A
   b. 50/50 processed control and innovation – Sample B
   c. Any optional blends chosen by the investigator, for example: 75/25 processed control and innovation – Sample C
4. Extrude blends and melt filter to create the samples: A pellets, B pellets and C pellets.
   a. Extruded pellets to be evaluated for DSC, melt flow, density, ash content, moisture content, FTIR, and color (only for natural pellets).
5. Injection Mold test part for ESCR testing of each blend if desired (optional)
Path 2 Method Steps

If the form submitted for testing is resin instead of containers, Path 1 method steps 1 and 2 are unnecessary. Steps 3, 4, and 5 for resin samples are illustrated in the flow diagram labeled Path 2. Details of each step are presented in the APR Polyolefin Standard Laboratory Processing Practices, O-P-01 through O-P-07 (included in document O-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.
Measurements, Report and Guidance Values

Path 1 - Incoming Flake Analysis

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sink-float</td>
<td>O-S-01</td>
<td>100% Floatability for Olefin material</td>
<td>Any non-PO labels, closures, layers, attachments, or other materials must either sink in water and be removed or must be compatible with PE. It is unacceptable for a non-PE material to stick to or otherwise not separate from the PE substrate and cause the PE to sink, resulting in yield loss, or stay with the PE and contaminate the PE.</td>
</tr>
</tbody>
</table>

Path 2 - 1st Melt History Extruded Pellet

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screen Pack Pressure Build</td>
<td>Practice O-P-06</td>
<td>Record and Report</td>
<td>No guidance; first step only for melt history</td>
</tr>
</tbody>
</table>
### Path 1 and 2 - Extruded Pellet Sample Evaluation

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Required values</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Screen Pack Pressure Build</td>
<td>Practice O-P-06</td>
<td>End pressure no greater than 25% over starting pressure value</td>
<td></td>
</tr>
<tr>
<td>Melt Flow Rate</td>
<td>ASTM – D1238</td>
<td>&lt; 0.75 g/10 minutes delta to the control</td>
<td></td>
</tr>
<tr>
<td>Density</td>
<td>ASTM – D792 or ASTM 1505</td>
<td>Lower limit of test material with 50% innovation shall be no less than 0.941 g/cm³</td>
<td>Control no less than 0.941 g/cm³</td>
</tr>
<tr>
<td>Ash</td>
<td>ASTM – D5630</td>
<td></td>
<td>Perform if density &gt;0.98. Record and Report</td>
</tr>
<tr>
<td>Volatiles/Moisture</td>
<td>ASTM – D6980</td>
<td>&lt; 0.5%</td>
<td></td>
</tr>
<tr>
<td>DSC</td>
<td>ASTM – D3418</td>
<td>Primary Peak not to exceed 150°C</td>
<td>Record primary and secondary peaks temperature and J/g. Calculate the J/g delta from primary peak to secondary peak if present.</td>
</tr>
<tr>
<td>FTIR</td>
<td>ASTM – D7399</td>
<td>&lt; 5% PP Content in test sample B (up to 10% allowable in innovation)</td>
<td></td>
</tr>
<tr>
<td>Pellet Irregularity</td>
<td>Porosity, roughness, grainy, gloss etc.</td>
<td>Record, Report, and Photograph</td>
<td></td>
</tr>
<tr>
<td>Extrusion Process Irregularity</td>
<td>Unusual sticking, fumes, odor or build-up occurring at the feed throat or die exit of the extruder</td>
<td>Record and Report</td>
<td></td>
</tr>
<tr>
<td>Optional value</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pellet Color Variations or Inconsistencies</td>
<td>ASTM – D6290</td>
<td>Only for natural pellets</td>
<td>Can reveal contamination before later evaluation</td>
</tr>
</tbody>
</table>
### Path 1 and 2 – ASTM Part Evaluation

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melt Flow Rate</td>
<td>ASTM – D1238</td>
<td>&lt; 0.75 g/10 minutes delta to the control</td>
<td></td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>ASTM – D638</td>
<td>No more than a 25% Δ to the control.</td>
<td></td>
</tr>
<tr>
<td>Elongation at Yield</td>
<td>ASTM – D638</td>
<td>No more than a 25% Δ to the control.</td>
<td></td>
</tr>
<tr>
<td>Notched Izod</td>
<td>ASTM – D256</td>
<td>No more than a 50% Δ to the control.</td>
<td></td>
</tr>
<tr>
<td>Flexural Modulus</td>
<td>ASTM – D790</td>
<td>No more than a 25% Δ to the control.</td>
<td></td>
</tr>
</tbody>
</table>

**Optional value for Critical Guidance; Required for Applications Guidance and Bottle to Bottle Testing**

<table>
<thead>
<tr>
<th>Property</th>
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<th>APR Guidance Preferred values</th>
<th>Additional Guidance</th>
</tr>
</thead>
<tbody>
<tr>
<td>ESCR</td>
<td>ASTM D1693</td>
<td></td>
<td>Record and Report – Preferred not to exceed 25% failures to the control for max. time reached.</td>
</tr>
</tbody>
</table>
Path 1: Flow Diagram for HDPE Rigid Containers incorporating innovation

Start

Obtain HDPE Control Container
Granulation O-P-03
Caustic Wash O-P-04
Air Separation O-P-05
Create Blends

Required A
100% Control Sample
Optional C
75% Control Sample
25% Innovation Sample
Required B
50% Control Sample
50% Innovation Sample
Extrude and Pelletize each Blend O-P-06

Injection Molding ASTM Test Parts of each Blend 0-P-07

End

Required Evaluation: Screen pack pressure build
Required Evaluations:
Melt Flow Rate
Tensile Strength
Elongation at Yield
Notched Izod
Flexural Modulus
Optional:
ESCR

Required Evaluation:
Sink/Float

Caustic Wash O-P-04
Air Separation O-P-05

Required Evaluations:
Melt Flow Rate
Density
Ash (if density >0.98)
Moisture
%PP (FTIR)
Pellet discoloration (natural only)
Screen pack pressure build
Pellet Appearance
Pellet Production
Performance

Extrude and Pelletize each Blend O-P-06

Injection Molding ASTM Test Parts of each Blend 0-P-07

End
Path 2: Flow Diagram for HDPE Rigid Resin incorporating innovations

1. **Start**

2. **Obtain HDPE Resin Control**
   - Optional Evaluation: Screen pack pressure build
   - Extrude O-P-06

3. **Obtain HDPE Resin with Innovation**
   - Extrude O-P-06

4. **Create Blends**
   - Required A: 100% Control Sample
   - Optional C: 75% Control Sample, 25% Innovation Sample
   - Required B: 50% Control Sample, 50% Innovation Sample

5. **Extrude and Pelletize each Blend O-P-06**
   - Required Evaluation: Screen pack pressure build

6. **Injection Molding ASTM Test Parts of each Blend O-P-07**
   - Required Evaluations:
     - Melt Flow Rate
     - Tensile Strength
     - Elongation at Yield
     - Notched Izod
     - Flexural Modulus
   - Optional: ESCR

7. **End**

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**Required Evaluations:**
- Melt Flow Rate
- Tensile Strength
- Elongation at Yield
- Notched Izod
- Flexural Modulus
- ESCR

**Optional Evaluations:**
- Screen pack pressure build

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<tr>
<th>Version</th>
<th>Publication Date</th>
<th>Revision notes</th>
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<tbody>
<tr>
<td>1</td>
<td>12-5-2019</td>
<td>Made minor clarifying changes, approved by OTC on 10/2/19</td>
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