

Critical Guidance Protocol for HDPE Colored or Natural Bottles with Resin Additives, Barriers, Layers or Closures

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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.

HDPE rigid containers typically consist of natural homopolymer, natural copolymer, and colored copolymer bottles, jars, and jugs. This test can be used to evaluate the impact of HDPE rigid container packaging innovations and components including multi-layer constructions, coatings, additives, new HDPE resin controls, new innovative HDPE resins, multi-material compositions, lids, dispensers and closures.

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.

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.

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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 articles that employ multi-layer constructions, coatings, additives, new HDPE resin controls, new innovative HDPE resins, multi-material compositions, lids, dispensers and closures. 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
 - Commercial Basic Wash and 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
1. Injection molding or compression molding of test bars for required ASTM tests of each blend (ESCR testing optional). Injection molding is recommended but compression molding may be used for low melt index polymers. In this case ASTM D4976 should be referenced.
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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
2. Injection molding or compression molding of test bars for required ASTM tests of each blend (ESCR testing optional). Injection molding is recommended but compression molding may be used for low melt index polymers. In this case ASTM D4976 should be referenced.

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

ASTM Part Test Methods:

ASTM D638 Tensile Properties of Plastics

ASTM D256 Determining the Izod Pendulum Impact Resistance of Plastics

ASTM D790 Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Material

ASTM D4976 Polyethylene Plastics Molding and Extrusion Materials

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.

3. 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.
4. 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. Commercial basic wash and dry
 - d. Elutriation of granulated material
5. 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
6. 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).
7. Injection molded or compression molded test part for required ASTM tests of each blend (ESCR testing optional). Injection molding is recommended but compression molding may be used for low melt index polymers. In this case ASTM D4976 should be referenced.

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

Property	Method	APR Guidance Preferred values	Additional Guidance
<u>Required values</u>			
Sink-float	O-S-01	100% Floatability for Olefin material	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.

Path 2 - 1st Melt History Extruded Pellet

Property	Method	APR Guidance Preferred values	Additional Guidance
<u>Optional Values</u>			
Screen Pack Pressure Build	Practice O-P-06	Record and Report	No guidance; first step only for melt history

Path 1 and 2 - Extruded Pellet Sample Evaluation

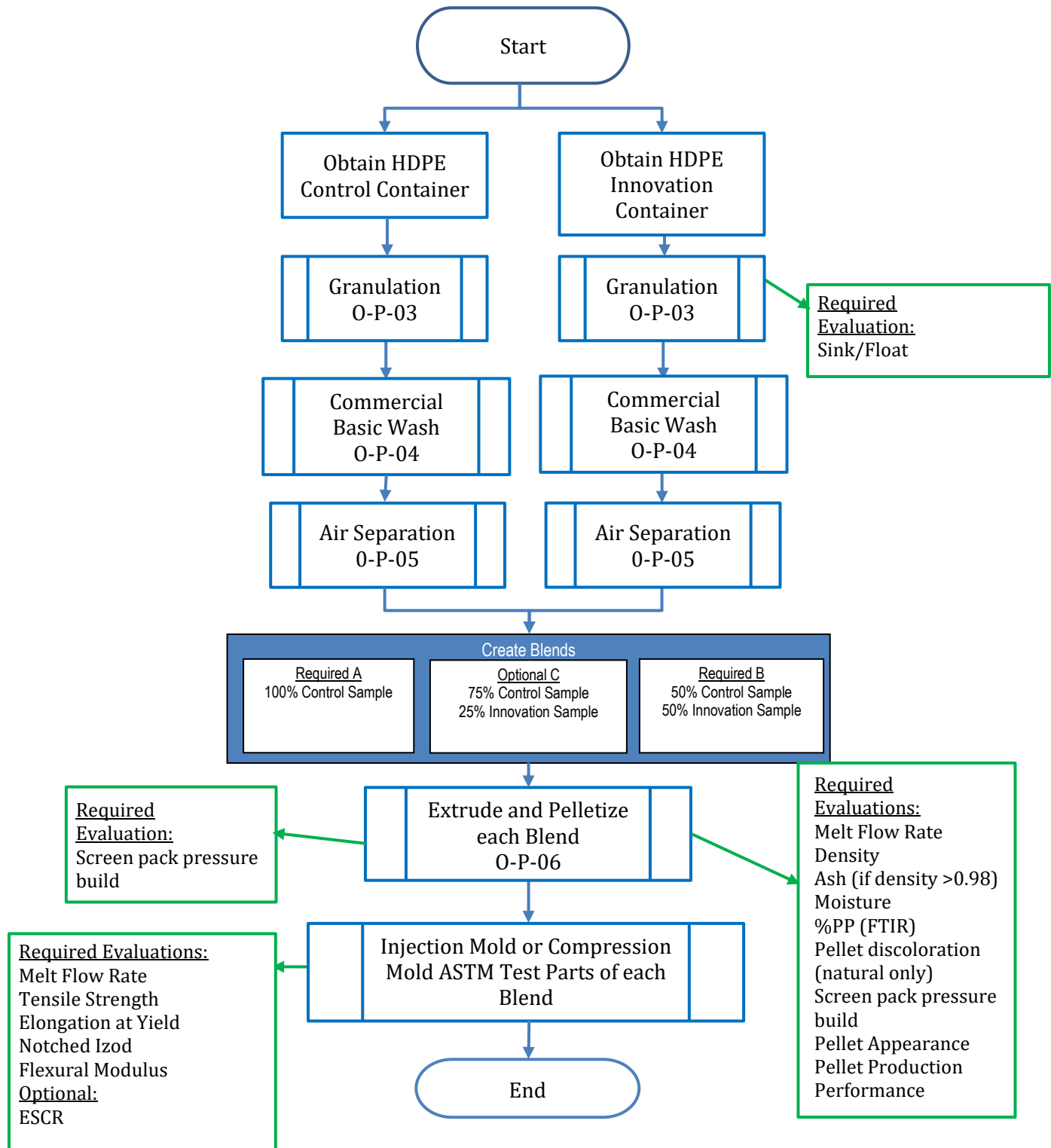
Property	Method	APR Guidance Preferred values	Additional Guidance
<u>Required values</u>			
Screen Pack Pressure Build	Practice O-P-06	End pressure no greater than 25% over starting pressure value	
Melt Flow Rate	ASTM – D1238	< 0.75 g/10minutes delta to the control	
Density	ASTM – D792 or ASTM 1505	Lower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

Path 1 and 2 – ASTM Part Evaluation

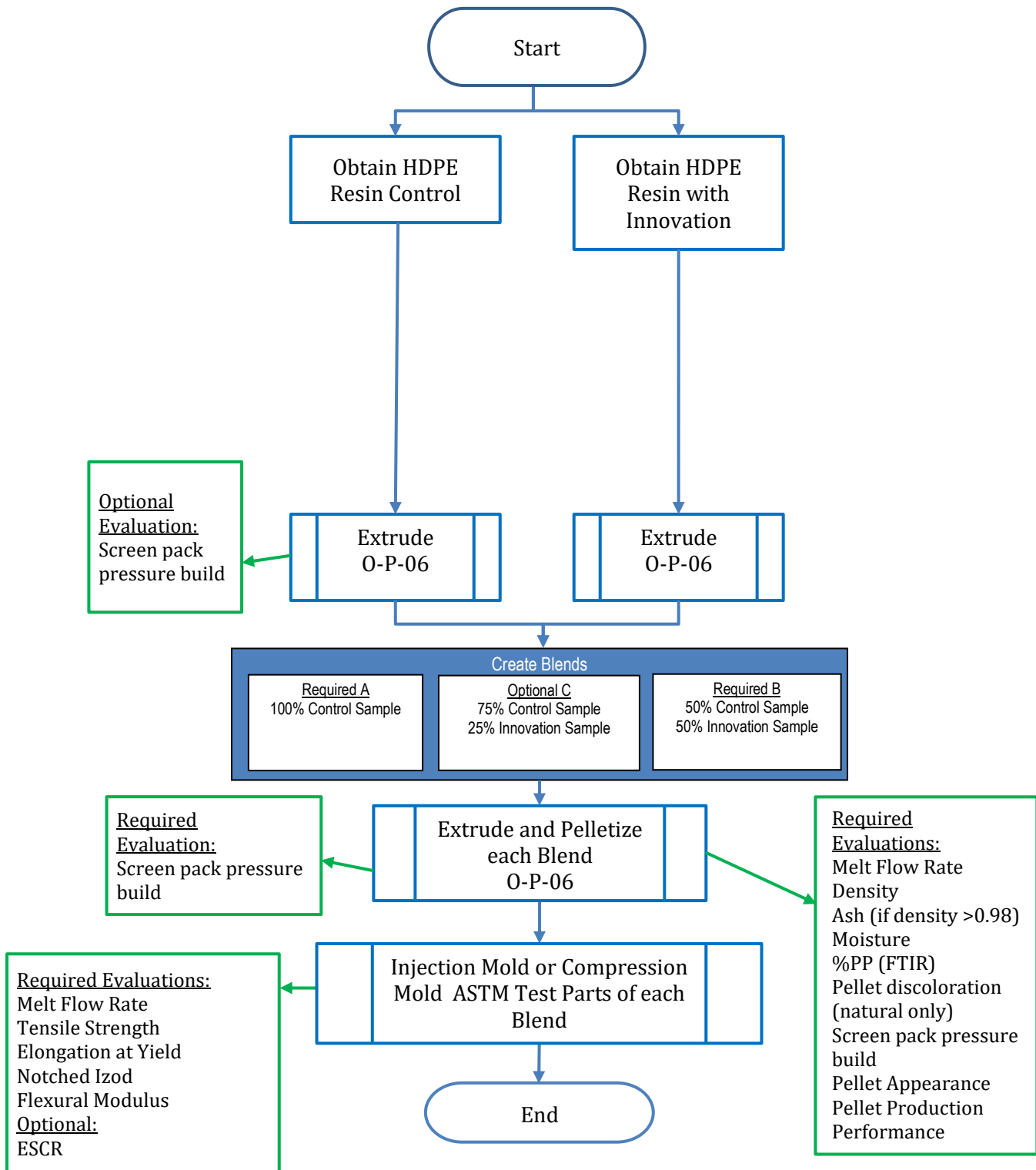
<u>Property</u>	<u>Method</u>	<u>APR Guidance Preferred values</u>	<u>Additional Guidance</u>
<u>Required values</u>			
Melt Flow Rate	ASTM – D1238	< 0.75 g/10 minutes delta to the control	
Tensile Strength at Yield	ASTM – D638	No more than a 25% delta increase or decrease, control to test samples.	
Elongation at Break	ASTM – D638	No more than a 50% delta decrease, control to test sample, with a minimum value of 200%	
Notched Izod	ASTM – D256	Control and test samples should have the same break type or show greater ductility for test sample.	Record and report break type (no break, partial, hinge, complete) and values for the control and test.
Flexural Modulus	ASTM – D790	No more than a 25% delta decrease, control to test samples.	

Property	Method	APR Guidance Preferred values	Additional Guidance
<u>Optional value for Critical Guidance; Required for Applications Guidance and Bottle to Bottle Testing</u>			
ESCR	ASTM D1693		Record and Report – Preferred not to exceed 25% failures to the control for max. time reached.

Path 1: Flow Diagram for HDPE Rigid Containers incorporating innovation



Path 2: Flow Diagram for HDPE Rigid Resin incorporating innovations



DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	12-5-2019	Made minor clarifying changes, approved by OTC on 10/2/19
2	7/24/2020	Test and specification changes, Approved by OTC on 6/4/2020