Critical Guidance Protocol for HDPE Rigid Containers

Document number – HDPE-CG-01 **Revision date**: September 3, 2024

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 are typically sorted into one of two streams listed as natural or colored stream. These streams can be processed differently in the commercial world and this protocol will reflect those differences in terms of paths. This test can be used to evaluate the impact of HDPE rigid container packaging innovations and components, examples including but not limited to: Additives, Barriers, Layers, Copolymers, Caps, Closures and Labels. This test may be utilized to potentially gain recognition for the natural stream, the colored stream or both if results meet certain criteria.

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.

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.

In certain cases, before Critical Guidance Protocol test results can be submitted to APR for consideration of guidance recognition, all pre-requisite tests, including sortation potential protocols and degradable additives testing, must be passed and such evidence must be presented with any guidance applications. In addition, the Program Administrator may ask for additional exposure testing and performance testing as are pertinent to the innovation. To determine when pre-requisite testing is needed, please refer to the APR Design® Guide for HDPE Rigid-Colored and HDPE Rigid-Natural and the following test protocols and resources.

For products or innovations that employ metal decoration or which contain metal components:

- RES-SORT-03a Metal Sorting Resource
- SORT-S-03 Metal Sortation Protocol

For items less than 5 cm in 2 dimensions:

- RES-SORT-02 Size Sortation Resource
- SORT-S-02 Size Sortation Protocol
- SORT-P-00 Compression Practices

For dark colors and label coverage (see definition in Design® Guide):

- RES-SORT-01 NIR Sorting Resource
- SORT-S-01 NIR Sortation Protocol

For materials that might be employed or marketed as degradable additives for plastics and which might be expected to display time dependent behavior or change with environmental exposure where appearance or physical properties can change over time see APR Position Paper on Degradable Additives.

This list is not inclusive. The Critical Guidance Protocol is not appropriate for package constructions that are not in alignment with the APR Design® Guide for Plastics Recycling text in cases where additional conditions are specified in the relevant Design® Guide section.

Method Summary

This Critical Guidance protocol provides two pathways for testing HDPE resins and articles with flow diagrams found on pages 10-13 for articles that employ multi-layer constructions, coatings, additives, new HDPE resin controls, new innovative HDPE resins, multi-material compositions, lids, dispensers and closures as innovation samples. 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 – Paths 1-A, Path 1-B and Path 1-C are utilized for complete HDPE rigid packages that include the innovation being tested.

- Path 1-A is followed when evaluating against a HDPE Colored Stream
- Path 1-B is followed when evaluating against a HDPE Natural Stream
- Path 1-C is followed when evaluating for both of the above streams

A flow diagram for Path 1-A, Path 1-B and Path 1-C on pages 10-12 illustrates that these articles are separately processed through these steps:

- Granulation
- Wash against the correct path (Based on O-P-04 – Wash Selection)
- Additional part production and natural color testing required only for Path 1-C
- 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 or compression molding (ASTM D4703 Procedure C) of test bars for required ASTM tests of each blend. Injection molding is recommended for materials above a 1 MI. Compression molding is recommended for materials below a 1 MI. This determination is to be made based upon the Control MI and held consistent for all sample preparation with a project for comparison consistency.

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 for both Natural and Colored HDPE stream. A flow diagram for Path 2, on page 13, 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 or compression molding (ASTM D4703 Procedure C) of test bars for required ASTM tests of each blend. Injection molding is recommended for materials above a 1 Melt Index (MI). Compression molding is recommended for materials below a 1 MI. This determination is to be made based upon the Control MI and held consistent for all sample preparation with a project for comparison consistency.

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 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
ASTM D792 Density and Specific Gravity (Relative Density) of Plastics by Displacement
ASTM D4703 Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

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. Wash using the correct method (Based on O-P-04 – Wash Selection)
    d. If following Path 1-C, wash material to move forward with Critical Guidance Testing utilizing the Commercial wash process. Additionally, wash materials against the Caustic wash process to proceed with the part production and natural color testing.
    e. Elutriation of granulated and washed 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. Step 4 only required for Path 1-C. If following Path 1-A or Path 1-B, skip to Step 5.
    a. Injection/Compression mold ASTM samples from caustic wash + elutriated flake to be evaluated for natural color testing. Injection Molding recommended for this step for better mixing of materials resulting in more uniform color properties.
    b. Evaluate color properties of produced parts.
5. 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 index, ash content(if applicable), moisture content, FTIR, and color tests.

6. Injection molding or compression molding (ASTM D4703 Procedure C) of test bars for required ASTM tests of each blend. Injection molding is recommended for materials above a 1 MI. Compression molding is recommended for materials below a 1 MI. This determination is to be made based upon the Control MI and held consistent for all sample preparation with a project for comparison consistency.
    a. Produced parts to be evaluated for melt index, density, tensile strength at yield, elongation at break, notched izod, flexural modulus and color properties.

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, 5, and 6 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
Required values			
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
Optional Values			
Screen Pack Pressure Build	Practice O-P-06	Record and Report	No guidance; first step only for melt history

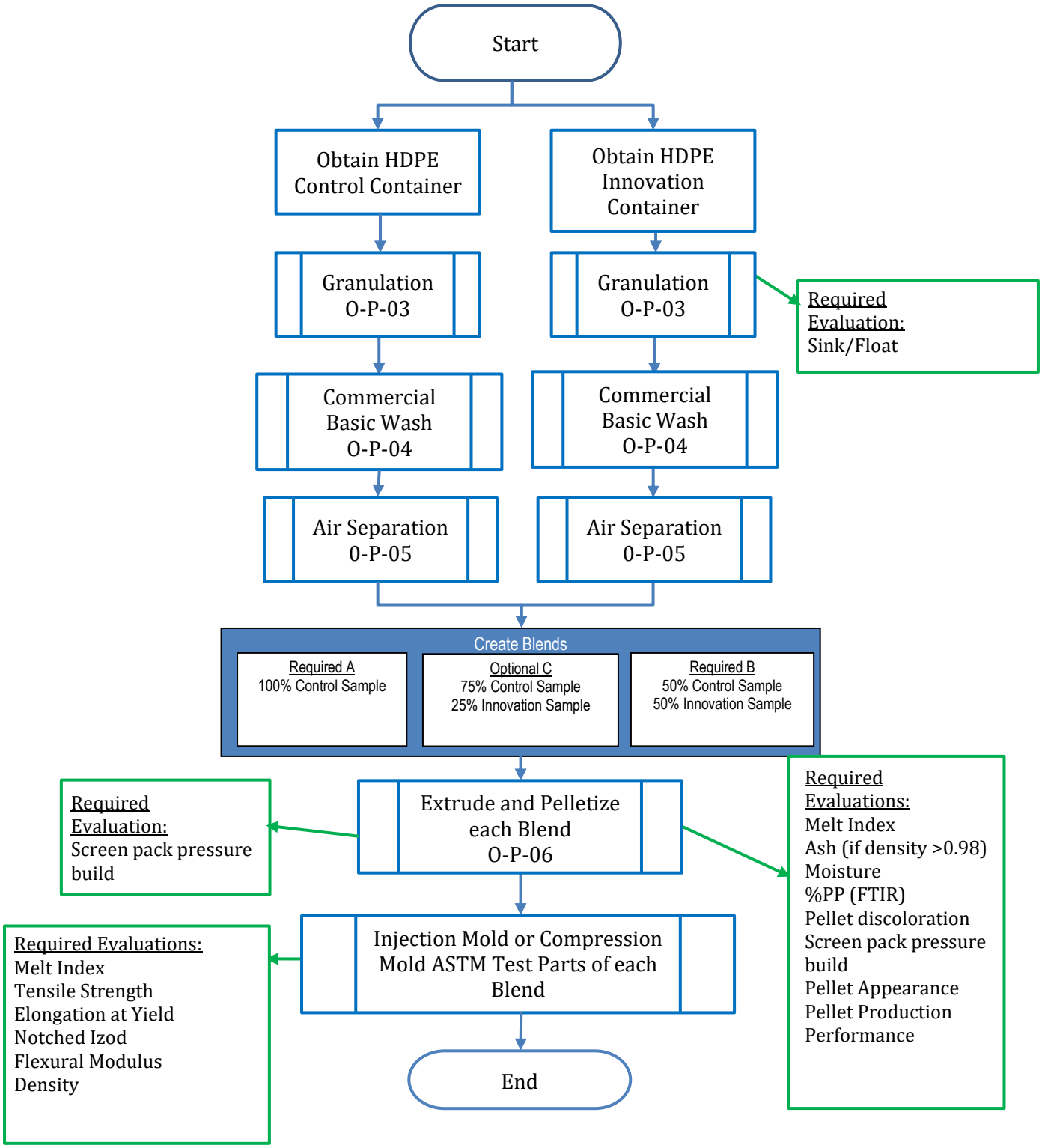
Path 1 and 2 - Extruded Pellet Sample Evaluation

Property	Method	APR Guidance Preferred values	Additional Guidance
Required values			
Screen Pack Pressure Build	Practice O-P-06	End pressure no greater than 25% over starting pressure value	
Melt Index	ASTM – D1238	< 0.75 g/10minutes delta to the control	
Ash	ASTM – D5630		Perform only if density >0.98. Record and Report
Volatiles/Moisture	ASTM – D6980	< 0.5%	
DSC	ASTM – D3418	Primary Melting Peak on 2nd Heat not to exceed 140 C	Record primary and secondary melting 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)	Report as < 5% or > 5%
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
Pellet Color Variations or Inconsistencies (Only for Path 1-B, and Path 2)	ASTM – D6290	Guidelines established for Natural Claims: ΔE < 3.5 between control and test	Not meeting color guidelines renders material ineligible for Natural Stream, but still eligible for Color Stream

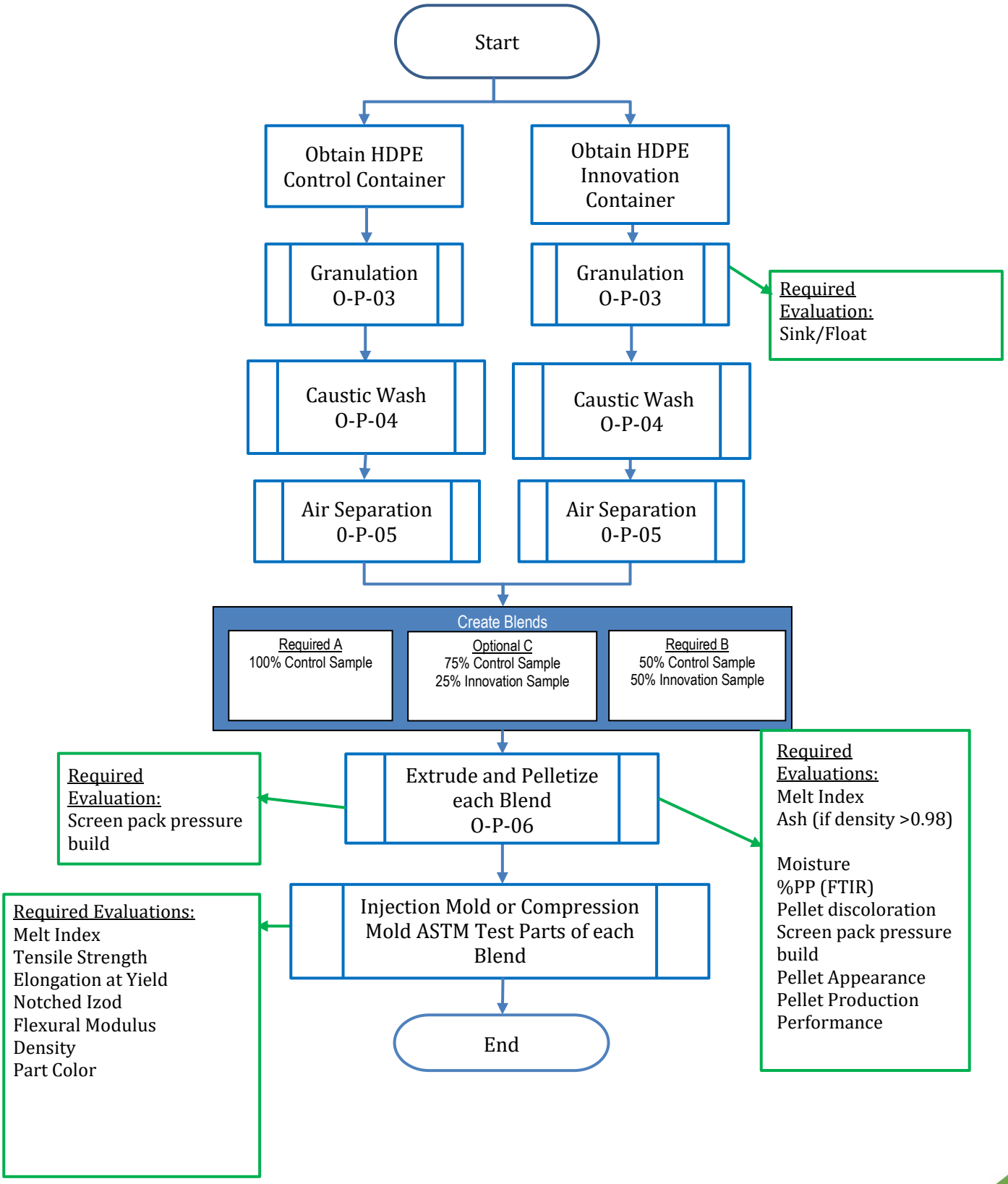
Path 1 and 2 – ASTM Part Evaluation

Property	Method	APR Guidance Preferred values	Additional Guidance
Required values			
Melt Index	ASTM – D1238	< 0.75 g/10 minutes delta to the control	
Density	ASTM – D792	Lower limit of test material with 50% innovation shall be no less than 0.941 g/cm^3	Control no less than 0.941 g/cm3
Tensile Strength at Yield	ASTM – D638	No more than a 25% decrease, control to test samples.	
Elongation at Break	ASTM – D638	No more than a 50% delta decrease, control to test sample	
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% decrease, control to test samples.	
Part Colors (Only for Path 1-B, 1-C and Path 2)	Modified ASTM – D290	Guidelines established for Natural Claims: ΔE < 3.5 between control and test	Not meeting color guidelines renders material ineligible for Natural Stream, but still eligible for Color Stream

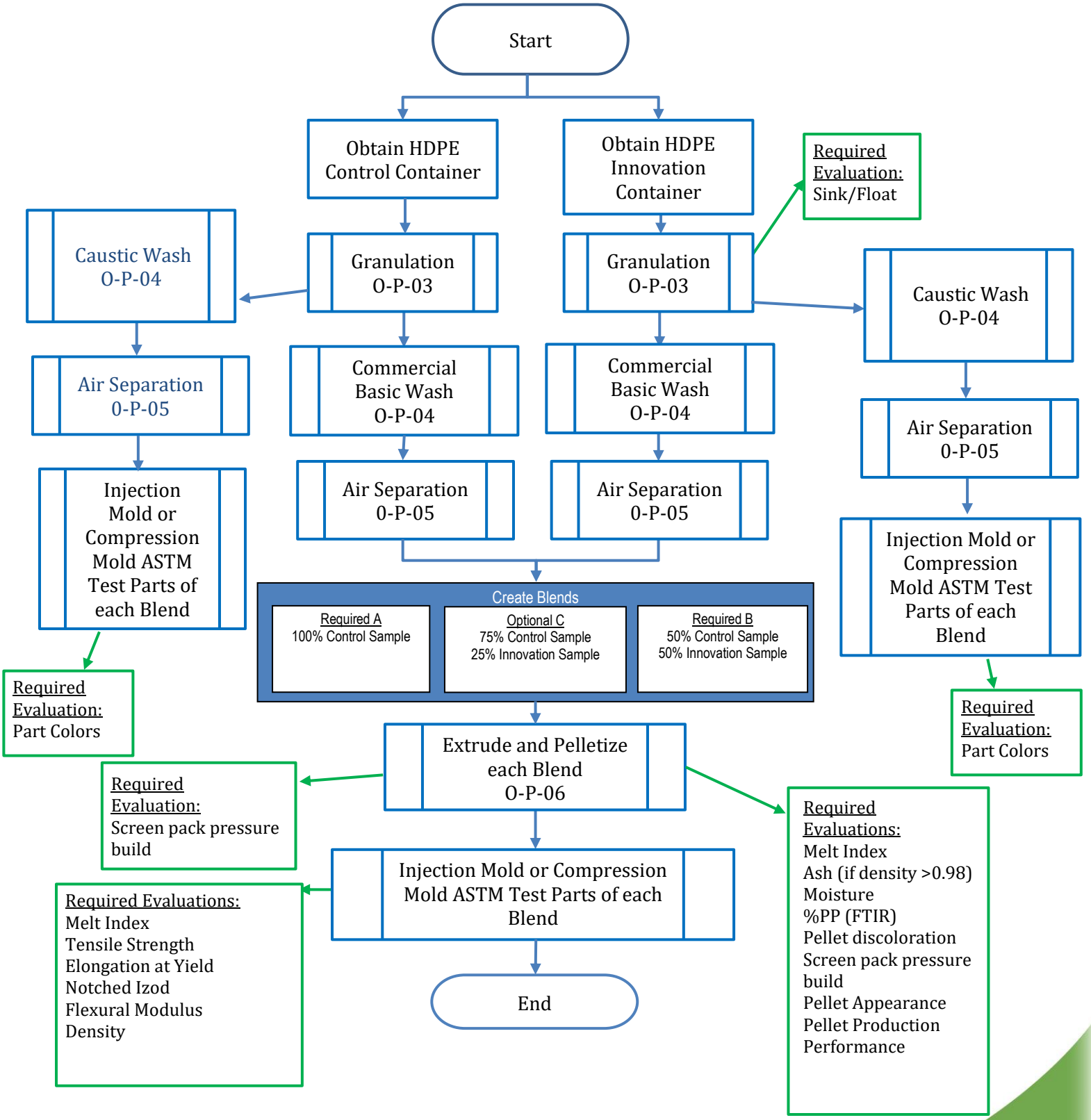
Path 1-A(Colored): Flow Diagram for HDPE Rigid Containers with Commercial Wash



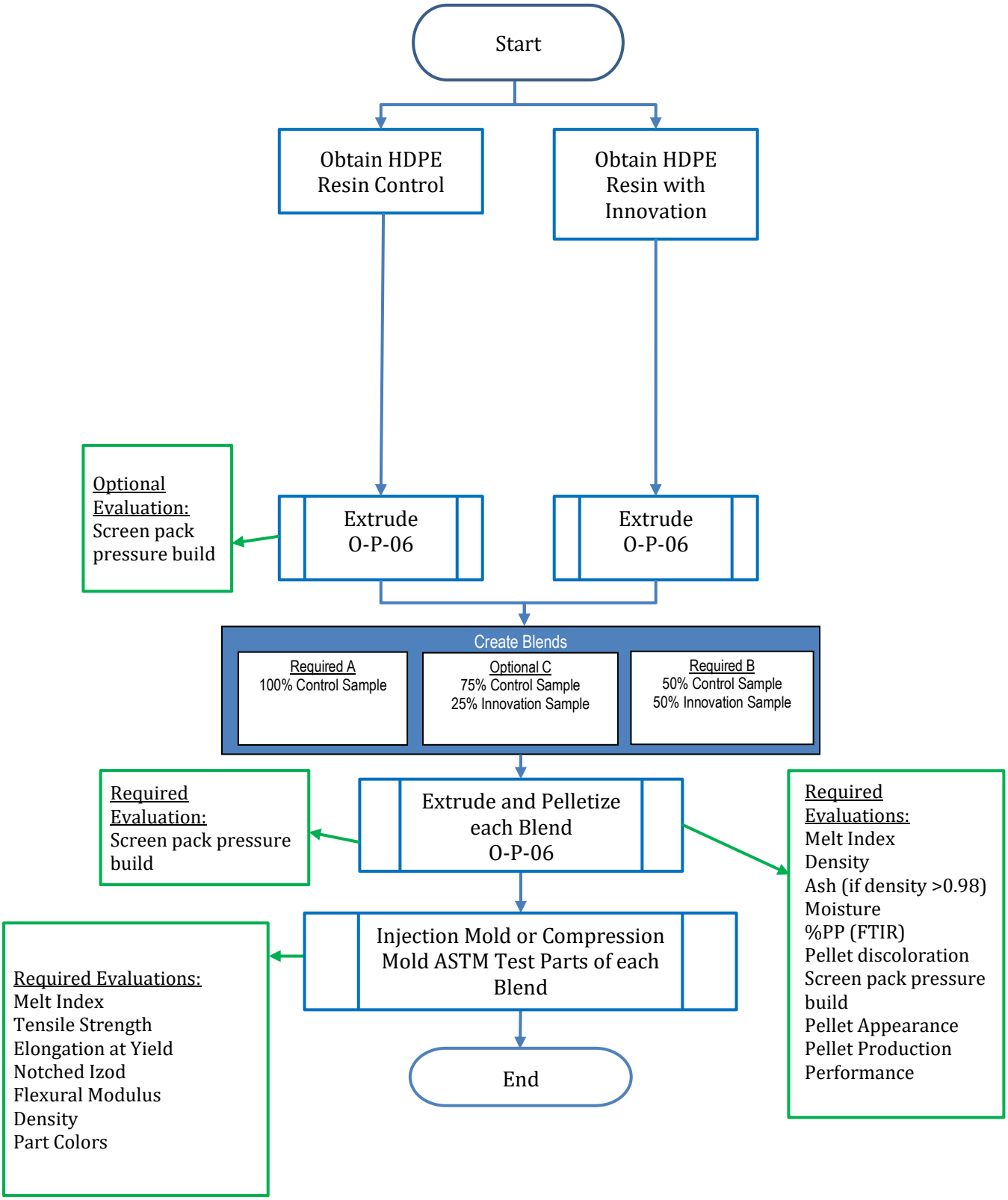
Path 1-B(Natural): Flow Diagram for HDPE Rigid Containers with Caustic Wash



Path 1-C(Colored & Natural): Flow Diagram for HDPE Rigid Containers with Both Caustic and Commercial Washes



Path 2(Colored & Natural): 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
3	June 3, 2021	Added language clarifying need for pre-requisite testing
4	August 16, 2021	Added expanded disclaimer language
5	February 7, 2022	Added changes approved by OTC including additional pathway and change to density guidance; combined all 3 HDPE CG tests into one
6	March 21, 2022	Edits per OTC; new copyright date
7	September 3, 2024	Changed naming conventions on sortation testing protocols from SORT-B-XX or SORT-S-XX; Update hyperlinks to match new website