

Critical Guidance Protocol for PP Rigid Containers

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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 PP Rigid container packaging innovations with reclamation systems sourcing post-consumer material.

PP rigid containers typically consist of natural and color homopolymer, copolymer, or random copolymer. Containers are typically in forms of tubs, lids, bottles, and other molded/shaped/formed articles. This test can be used to evaluate the impact of PP rigid container packaging components. Examples including multi-layer constructions, coatings, additives, printing, labels, adhesives, new PP resin controls, new innovative PP 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. 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 a control material, and for the innovation articles blended with extruded articles.

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 should be presented with any guidance applications. To determine when pre-requisite testing is needed, please refer to the [APR PP Design® Guide text and tables](#) and following test protocols and resources:

For metal and metal containing:

- [APR RES-SORT-1 Metal Sorting Resource](#)
- [APR SORT-B-03 Metal Sortation Protocol](#)

For items less than 5 cm in 2 dimensions:

- [APR RES-SORT-3 Size Sortation Resource](#)
- [APR SORT-B-02 Size Sortation Protocol](#)
- [APR SORT-PR-01 Compression Practices](#)

For dark colors (see definition in Design® Guide)

- [APR RES-SORT-2 NIR Sorting Resource](#)
- [APR SORT-B-01 NIR Sortation Protocol](#)

For materials that employ time dependent behavior or environmental exposure where appearance or physical properties are expected to change over time:

- [APR HDPE-S-03 HDPE/PP Degradable Additives Test](#)

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 further testing is specified in the relevant Design® Guide section.

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The results of testing using APR's Critical Guidance Test Protocols along with a Critical Guidance Technical Review are intended to qualify a company's innovation for APR's Critical Guidance Recognition only. The complete testing protocol process requires a review of the test results by a Technical Review Team convened by APR. If test results are not reviewed by an APR Technical Review Team, no APR recognition is possible. APR does not give permission for its name to be used to claim, or to imply in any way, that APR has recognized or approved the design feature or innovation that was tested when APR has not reviewed the test results

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Although test data generated by a company is the property of that company and may be used for other purposes besides APR Critical Guidance Recognition, the test protocols are the property of APR and APR

requires that they be used as written in their entirety. It should also be noted that partial test results that may under-report negative impacts from an innovation or design feature could misrepresent APR's intention or position and will be called out by APR when these incidents come to APR's attention.

Method Summary

This Critical Guidance protocol provides two pathways for testing PP resins and articles with flow diagrams found on pages 8 and 9 for articles that employ multi-layer constructions, coatings, additives, printing, labels, adhesives, 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 PP 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 PP 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
 - Small 100g caustic wash required when labels with adhesives or printing are present
- 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
- Pelletized material is injection molded into ASTM test parts for mechanical properties evaluation

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 melt history to represent the initial production heat history.
- 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
- Pelletized material is injection molded into ASTM test parts for mechanical properties evaluation

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 D1895 Apparent Density, Bulk Factor, and Pourability of Plastic Materials

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 Materials

Optional ASTM Test Methods:

ASTM D1693 Environmental Stress-Cracking of Ethylene Plastics

Method Steps for Extrusion Blends and Injection Molding 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 materials per the Screening Test O-S-01
 - c. Commercial Basic Wash and drying
 - i. Small caustic wash required when labels or printing is present
 - 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 optional C pellets.
 - a. Extruded pellets to be evaluated for DSC, melt flow, density, ash content, moisture content, bulk density, FTIR, and color.
5. Injection mold part for mechanical evaluation of each blend
Injection molded part to be evaluated for flexural modulus, tensile strength, notched Izod and optional ESCR.

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 PP. It is unacceptable for a non-PP material to stick to or otherwise not separate from the PP substrate and cause the PP to sink, resulting in yield loss, or stay with the PP and contaminate the PP. |

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 Evaluation

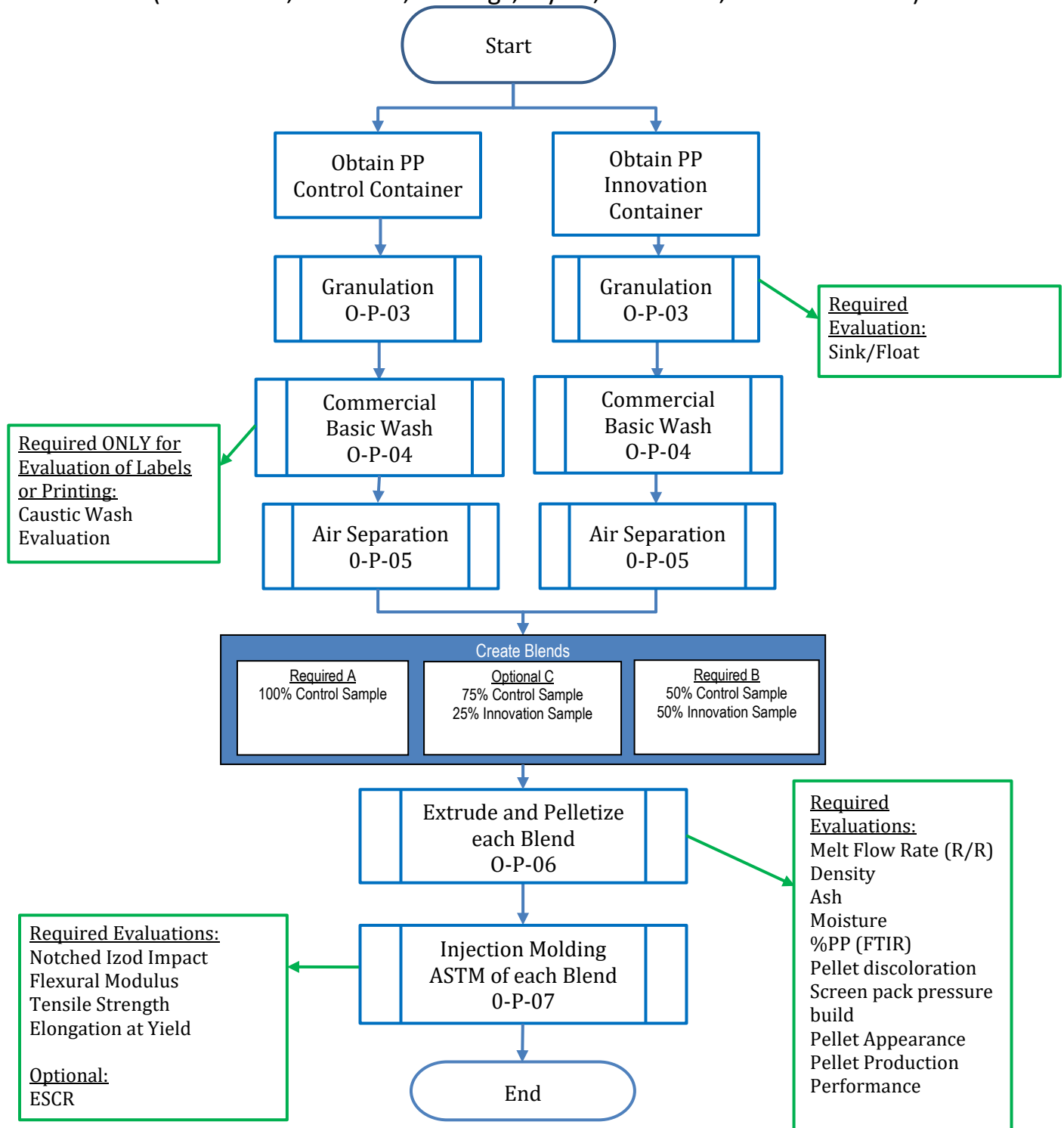
| Property | Method | APR Guidance Preferred values | Additional Guidance |
|----------------------------|--------------------------|---|--|
| <u>Required values</u> | | | |
| Screen Pack Pressure Build | Practice FPE-P-06 | End pressure no greater than 25% over starting pressure value | |
| Melt Flow Rate | ASTM – D1238 | | Record and Report |
| Density | ASTM – D792 or ASTM 1505 | <1 g/cc for control and test | |
| Ash | ASTM – D5630 | | Record and Report |
| Volatiles/Moisture | ASTM – D6980 | < 0.5% | |
| Bulk Density | ASTM – D1895 | >480 kg/m ³ | |
| DSC | ASTM – D3418 | Primary Peak not to exceed 180C | Record primary and secondary peaks temp and J/g. Calculate the J/g delta from primary peak to secondary peak if present. |
| Calculated Weight % | Basic | < 10% PE Content | |

| | | | |
|--|--|--------------------------|--|
| 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 |

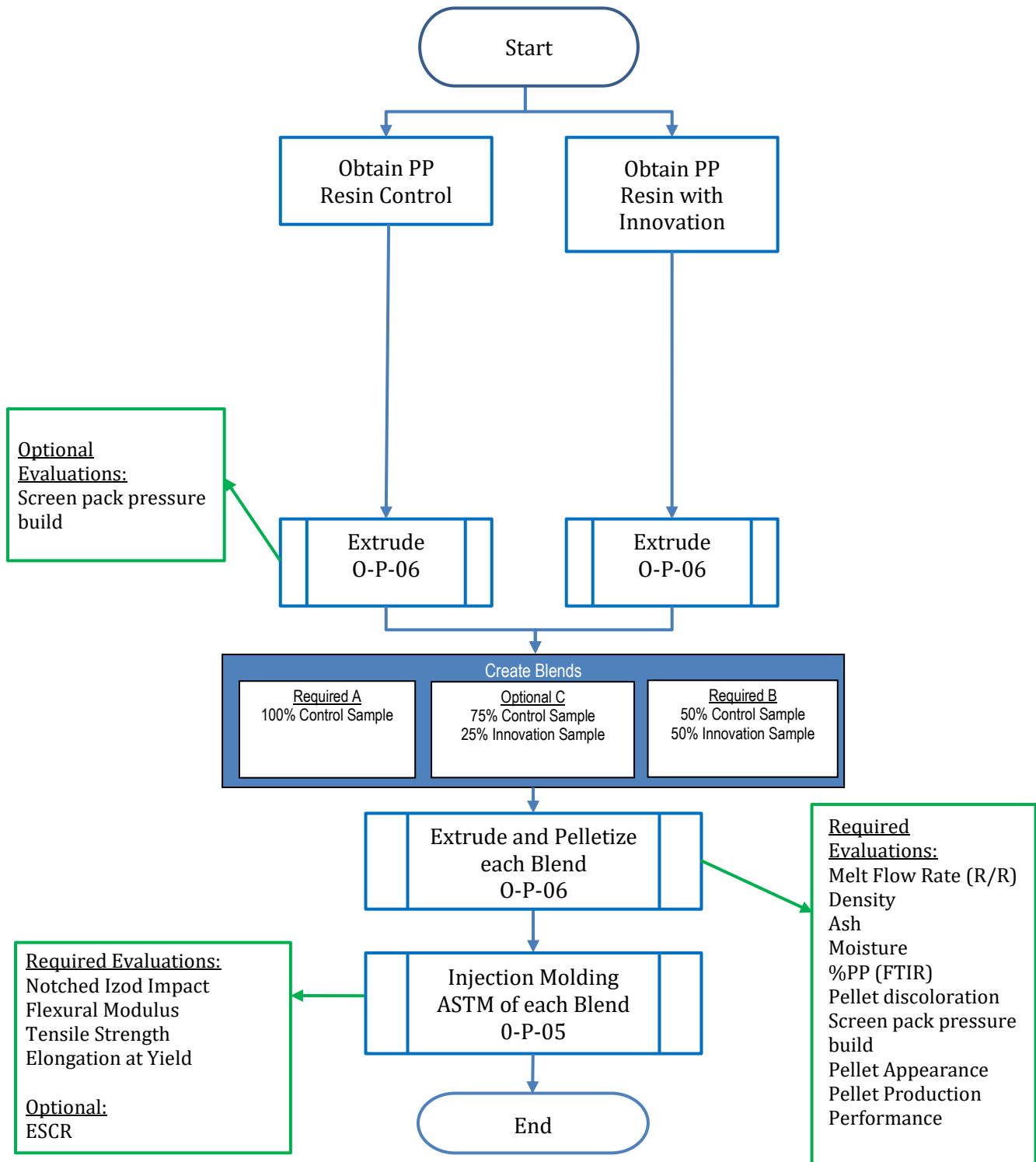
Path 1 and 2 – ASTM Part Evaluation

| Property | Method | APR Guidance Preferred values | Additional Guidance |
|------------------------|---------------|---|--|
| <u>Required values</u> | | | |
| Met Flow Rate | ASTM – D1238 | | Record and Report |
| Tensile Strength | ASTM – D638 | No more than a 25% Δ to the control. | |
| Elongation at Yield | ASTM – D638 | No more than a 25% Δ to the control. | |
| Notched Izod | ASTM – D256 | No more than a 25% Δ to the control. | |
| Flexural Modulus | ASTM – D790 | No more than a 25% Δ to the control. | |
| <u>Optional values</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 “PP Rigid” that employ qualified
(New resins, additives, coatings, layers, adhesives, labels or blends)



Path 2: Flow Diagram for “PP Rigid” Resin
 (Only an Option When Innovation is submitted as a blended pellet for Pre-Application)



DOCUMENT VERSION HISTORY

| Version | Publication Date | Revision notes |
|---------|------------------|--|
| 1 | 12-5-2019 | Made minor clarifying changes, approved by OTC on 10/2/19 |
| 2 | May 17, 2021 | Added language clarifying need for pre-requisite testing |
| 3 | August 17, 2021 | Changed MFR requirement for pellet and ASTM part testing to “Test and Report” in both Pathways 1 and 2; added expanded disclaimer language |
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