Critical Guidance Protocol for Clear PET Resin and Molded Articles

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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 new PET resins, as well as molded PET articles that employ new resins, blends, additives, coatings, adhesive layers, or multi-layer constructions with common commercial scale recycling processes. These new materials are referred to as innovations. This protocol is only applicable to clear PET articles. Clear PET refers to natural color PET with no colorants added at the molding process. 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 can be used with any pelletized PET resin or with molded articles made with PET. Molded articles are most often expected to be: injection stretch blow molded containers, extrusion blow molded containers, or thermoformed sheet products; but it also applies to any article that would be sorted as clear PET in the collection and sorting system.

Data developed by an independent third-party laboratory following this protocol can be used in petitions to APR's Critical Guidance Recognition Program. Petitions require data for a control material, and for the innovation articles blended with molded control articles.

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 PET Rigid Design® Guide 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
- SORT-S-04 Color 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.

- APR Position Paper - 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.

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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 and flow diagrams

Molded articles, Path 1 - This Critical Guidance protocol provides two pathways for testing resins and molded articles. For molded articles, including molded articles that employ layers, coatings, additives, adhesives or blends; articles are made with the innovation material, and similar articles are also made solely with a control PET resin without the innovation for comparison. A flow diagram shown in Appendix I illustrates that these articles are separately processed through these steps:

- Granulated, washed, sink/floated, dried, and elutriated.
- Blends are created from the washed and elutriated flake. Required blends are 100% control material; and a 50/50 blend of control material and test material. Optional blends can be used if desired by the investigator. An example is given in the test method for a 75/25 blend of control with test material.
- These blends are dried, extruded and pelletized.
- Pelletized material is solid stated, and the solid stated pellets are injection molded into plaques for evaluation. (Solid stated pelletized material of 0.80 dL/g that are made from this Path can also be used to satisfy the Application Test Protocols.)

Measurements are made in each of the process steps using specified tests that are required for the Critical Guidance evaluation. An investigator may run optional tests, if desired, to obtain additional information that is not required for the Critical Guidance evaluation.

Pelletized resins, Path 2 – For qualified pelletized innovation PET resins a simpler path is available. The heat history of molding an article is modeled by extruding and re-pelletizing each of the control and test resins. Blends of control and test material can be made without the need to wash and elutriate samples. This second pathway is show in Appendix II.

Reference Documents

The following documents are referenced in this Critical Guidance Protocol:

PET-P-00: PET Standard Laboratory Processing Practices

APR PET Screening Test Methods:

- PET-S-04: PET Package Materials Balance Test
- PET-S-08: PET Flake Clumping Test
- PET-S-10: PET Flake Oven Bake Test
- PET-S-07: PET IV Build Rate Test
- PET-S-02: PET Flake or Pellet Discoloration Test
- PET-S-09: Testing of PET Plaques for Color, Haze and Inclusions

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
- ASTM D3418 - 15 Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry

Method steps for Molded Articles – Path I

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Sortation qualification for molded articles

If there is any concern that the test article will not sort properly in either a materials recovery facility or plastic reclaimer, the applicable APR sortation potential test(s) must be conducted prior to proceeding with the Critical Guidance protocol. Criteria for determining this can be found in the APR Design Guide® for Plastics Recyclability. Specific factors to consider are small or very large size, more two dimensional than three dimensional, containing metal or metalized inks and material components that could influence NIR sortation. Molded articles that do not provide a "preferred" level of sortation performance are not candidates for Critical Guidance review.

Path 1 Method Steps

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagram in Appendix I, and details of each step are presented in the PET-P-00: PET Standard Laboratory Practices, PET-P-01 through PET-P-08 (included in document PET-P-00):

1. Obtain molded innovation articles and control articles to use in the evaluation. The amount of material will depend upon the equipment and scale used in each laboratory.
2. For each of the test and control articles, separately:
    a. Granulate the articles.
    b. Wash the resulting flake including rinse and float/sink steps.
    c. Dry the flake.
    d. Elutriate the flake.
3. Prepare the following required blends:
    a. 100% control flake - Sample A
    b. 50/50 blend of control flake with flake from the innovation article – Sample B

   c. Prepare any optional blends chosen by the investigator, for example: 75/25 blend of control flake and innovation flake – Sample C
4. Extrude, melt filter and re-pelletize to create the samples: A pellets, B pellets and C pellets.
5. Solid state the pellet samples for an initial 8 hour time period to create samples: A SSP Pellets, B SSP pellets and C SSP pellets. These samples are used for the IV Build Rate Evaluation.
6. Solid state a second sample of the blends to an IV of 0.80 dl/g to create samples used to make injection molded plaques: A SSP 0.8 pellets, B SSP 0.8 pellets and C SSP 0.8 pellets
   a. This material if produced in sufficient quantity will be satisfactory for use in any of the Application Testing Protocols that might optionally be performed.
7. Injection mold the 0.80 IV blends to create amorphous plaque samples: A plaques, B plaques and C plaques.

Method steps for Pelletized Resin – Path 2

When pelletized resins are being evaluated, the creation of blends can be different than when the starting point is a molded article. Otherwise, the process and evaluation steps are the same.

Pre-test to qualify a resin for Path 2

1. In a first step, wash a sample of the pelletized test material – about 100 grams - in a 1 wt% solution of NaOH at 85° C for 15 minutes, then rinse the resin with tap water and air dry the sample.
2. Measure the color values of the pellets before and after exposure to the hot caustic wash.
3. If the change in b* value of the washed pellets compared to the un-washed pellets is less than one b* unit, blends can be prepared using Path 2 outlined below which does not require making molded articles.
4. If the change in b* value of the washed pellets compared to the un-washed pellets is greater than one b* unit, the resin is treated as though there is an additive in the resin and molded articles must first be made and tested using Path I above.

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagram in Appendix II, and details of each step are presented in the PET Practices, PET-P-01 through PET-P-08:

1. Following the Extrusion Practice, PET-P-06, separately dry each of the control and innovation resins, then extrude the pellets in an extruder with a strand die and recover the re-pelletized material. This step adds a drying and melt heat history to simulate making a molded article with the resins. There is no requirement for melt filtration when extrusion is only to add heat history.
2. Prepare the following required blends from the re-pelletized samples:
   a. 100% control pellets – Sample A pellet blend
   b. 50/50 blend of control pellets with pellets from the test article – Sample B pellet blend
   c. Prepare any optional blends chosen by the investigator, for example: 75/25 blend of control pellets and test pellets – Sample C pellet blend
3. Extrude, melt filter and re-pelletize to create the samples: A pellets, B pellets and C pellets.

4. Solid state the pellet samples for an initial 8 hour time period to create samples: A SSP Pellets, B SSP pellets and C SSP pellets. These samples are used for the IV Build Rate Evaluation.
5. Solid state a second sample of the blends to an IV of 0.80 to create samples used to make injection molded plaques: A SSP 0.8 pellets, B SSP 0.8 pellets and C SSP 0.8 pellets
    a. This material if produced in sufficient quantity will be satisfactory for use in any of the Application Testing Protocols that might optionally be performed.
6. Injection mold the 0.80 IV blends to create amorphous plaque samples: A plaques, B plaques and C plaques.

Measurements, Report and Guidance Values

Pellet pre-test results when Path 2 is employed

Report b* value of test pellets before and after wash.

Wash and elutriation evaluations - (applies to Path 1 evaluations only)

Property	**Method**	**APR Guidance Preferred values**	**Additional Guidance**
Required values			
PET flake clumping test when required	PET Flake Clumping Test, PET-S-08	<1 wt% retention on screen and foil for each of the un-weighted and weighted evaluations	Required when the following are involved: an adhesive or polymeric coating, a non-crystalline thermoplastic component, or a crystalline material with MP less than 225° C
Optional values			
Flake bake test	PET Flake Oven Bake Test, PET-S-10		Can reveal contamination before later evaluations
Flake color	PET Flake or Pellet Discoloration Test, PET-S-02		Can reveal contamination before later evaluations
Materials balance	PET Package Materials Balance Test, PET-S-04		Can reveal contamination before later evaluations

Extrusion evaluation

IV loss - The table below calls for reporting the Extrusion IV loss when Flake samples are extruded to pellet samples in Path 1, or where pellet blend samples are extruded to pellet samples Path 2. The following steps are used to report this value using Path 1 and 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 pellets for each blend after extrusion.
- Measure the IV loss for sample A with extrusion and call that value A'. This is the IV loss for the control.
- Measure the IV loss for Sample B with extrusion and call that value B'. This is the IV loss for the 50:50 blend of innovation and control.

Property	Method	APR Guidance Preferred values	Additional Guidance
Required values			
Extrusion IV loss in extruding flakes samples A and B to amorphous pellets A and B	ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B	Difference in A' and B' is 0.025 units or less	
Screen pack pressure build	Steps given in the Melt Filtration Practice, PET-P06	End pressure is no greater than 25% over starting pressure value	
Observation for fuming or odor at feed throat and die exit	Visual evaluation, no method	No unusual fuming or odors observed	
Observation for material sticking in drier or feed throat	Visual evaluation, no method	No material sticks in drier of feed throat	
Observation for any impact on safety or hazardous conditions	Visual evaluation, method	No safety or hazard conditions observed	
Optional value			
Pellet color	PET Flake or Pellet Discoloration Test, PET-S-02		Can reveal contamination before later evaluations

Solid stating evaluation

Property	Method	APR Guidance Preferred values
	Required values	
IV build rate	PET IV Build Rate Test, PET-S-07	IV Delta of control and normalized value of test material: At 8 hours < 0.04 units and sample B SSP demonstrates 0.90 IV within 15 hours' time
Melting point of solid stated pellets B at 0.80 IV by DSC measurement	ASTM D3418 - 15 10° C/minute. On second melt after 1st melt rapid quench to create amorphous material.	Between 225 and 255° C
Unusual sticking or residue in solid stating unit	Visual observation, no method	No sticking or residue
	Optional values	
Pellet discoloration	PET Flake or Pellet Discoloration Test, PET-S-02	
Acetaldehyde content	ASTMF2013-10	

Evaluation of molded plaques

Property	Method	APR Guidance Preferred values
	Required values	
L color value	Testing of PET Plaques for Color, Haze and Inclusions, PET-S-09	>82
a* value of plaques B	PET-S-09	Less than 1.5 units difference compared to Plaques A
b* value of plaques B	PET-S-09	Less than 1.5 units difference compared to Plaques A5
% haze of plaques B	PET-S-09	Control not to exceed 9% value, and test not to exceed more than 10% units greater than control.
Inclusions and specks in 50 plaques B	PET-S-09	If A = 0; B is 2 or less If A = 1; B is 4 or less If A = 2; B is 6 or less
IV loss when pellets are molded to plaques	ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B	The value of IV loss for sample B is no greater than 0.025 units when compared to sample A

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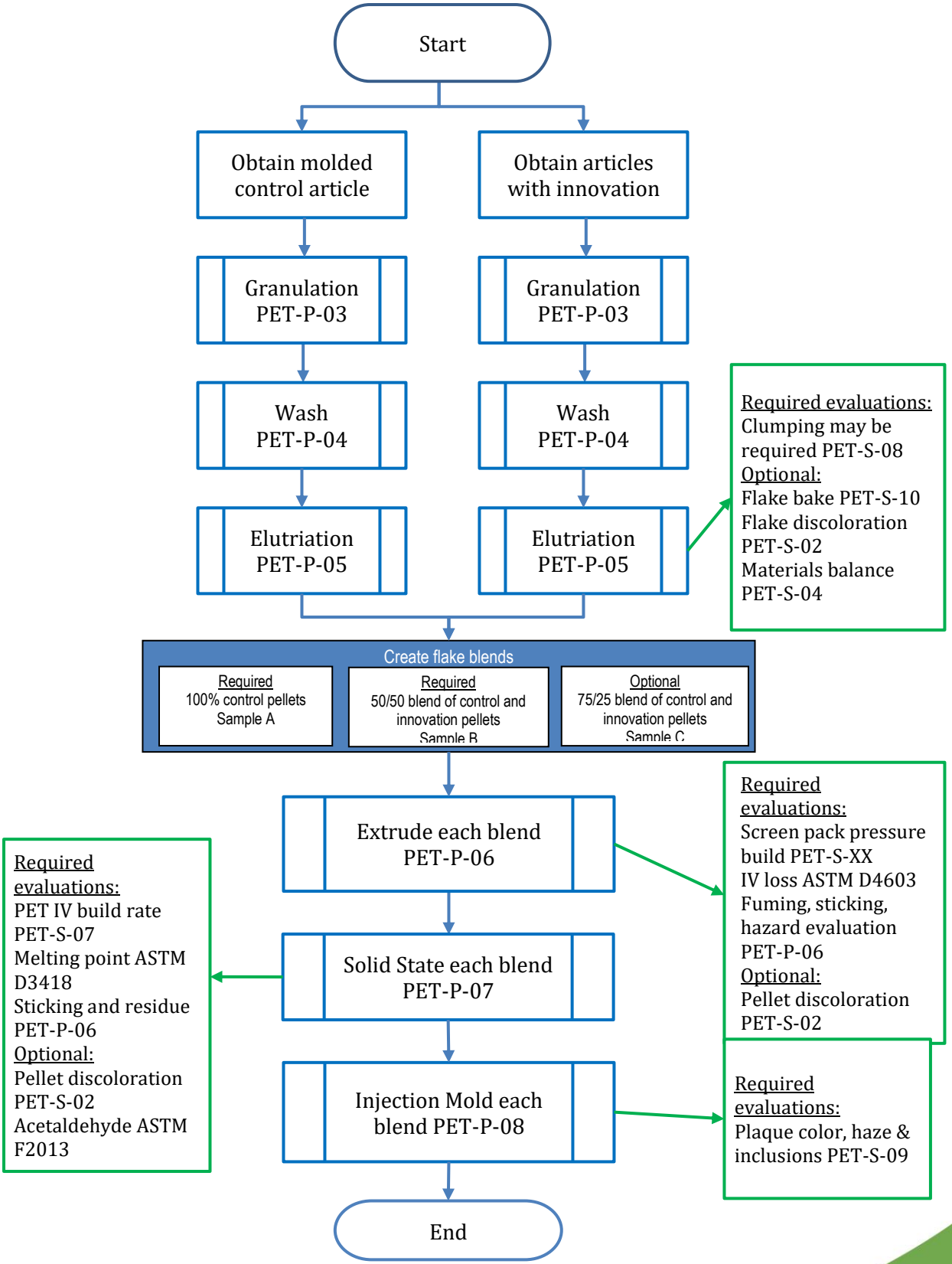
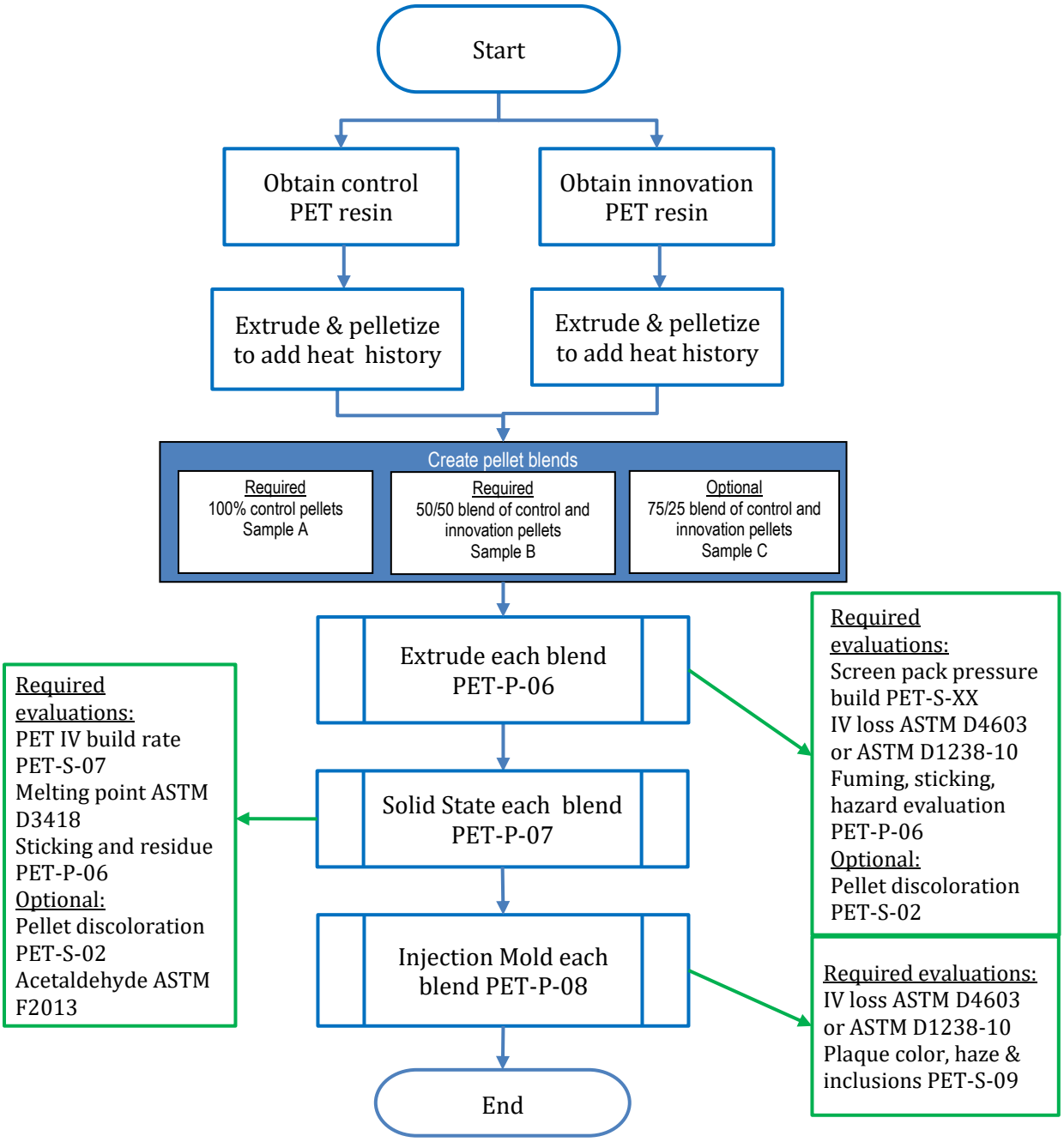
Appendix I Path 1: Flow Diagram for clear molded articles that employ qualified new resins, additives, coatings, layers, adhesives or blends



Appendix II Path 2: Flow Diagram for Qualified Clear PET Resins



DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	Original
2	April 11, 2019	Revised Haze Guidance Preferred Values as approved by PTC in March 2019
3	June 3, 2021	Added language clarifying need for pre-requisite testing
4	August 17, 2021	Added expanded disclaimer language
5	August 30, 2024	Changed naming conventions on sortation testing protocols from SORT-B to SORT-S; Updated hyperlinks to match new website