Applications Guidance Protocol for Recycled PET

Document number – PET-A-01 **Revision date** - September 3, 2024

Introduction – Scope, significance and use

The recycled PET (rPET) Applications tests are designed to evaluate how an innovation material might affect end-use applications that use rPET. The end-use applications this protocol will study are:

- rPET-to-Bottle
- rPET-to-Sheet
- rPET-to-Strapping
- rPET-to-Fiber

Before the Applications Guidance for Recycled PET tests can be used, the innovation material must first have been tested following APR's Critical Guidance Protocol for Clear PET Resin and Molded Articles, PET-CG-01, and found to have met all critical guidance criteria. 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 Applications Guidance for Recycled PET protocol is intended to build on the results of the PET-CG-01 test. Therefore, the Innovator is encouraged to consider the possibility that they will want to perform one or all of these applications tests before beginning the PET-CG-01 testing. By doing so, the quantities of materials used in the PET-CG-01 test can be increased in order to provide the required amount of materials needed to complete the PET Applications test. And since the applications tests build on information gained from the data obtained from the PET-CG-01 protocol, some issues pertinent to the sheet applications are addressed in the rPET-to-Bottle Protocol and some issues pertinent to the staple fiber applications are addressed in the rPET-to-Bottle and rPET-to-Sheet Protocols.

Data developed by an independent third-party laboratory following this protocol can be used in petitions to APR's Critical Guidance Recognition Program. The APR would consider a full petition for Recycling Guidance Recognition for meeting or exceeding all of the strictest guidance as outlined in the Critical Guidance and Applications Guidance Protocols.

The APR rPET-to-Bottle recycle evaluation is aligned with the EPBP Bottle-to-Bottle protocol so that an Innovator (under some conditions) might be able to satisfy the requirements of both protocols by careful selection of control materials and processing steps to ensure that the requirements of each protocol are met.

The guidance contained in this document does not include time as a variable. Innovations which include time as a factor will require additional considerations.

Moreover, the inability of an innovation to meet specified values does not imply recycling failure, but should be a clear message that significant technical challenges might exist under certain circumstances and mitigation of the issue may be needed to avoid degrading the value of the stream of recyclable bottles.

The Applications Guidance for Recycled PET protocol uses materials produced during the PET-CG-01 test. For example:

- Washed and elutriated flake is used to make sheet.
- 0.80 dL/g solid stated pellets are used to mold preforms and blow bottles.
- The rate of IV build will be used to determine suitability for use in strapping.
- The molded plaques are examined for fluorescence to meet fiber requirements.

All processing steps required to produce the above materials are referenced in PET-CG-01.

Reference Documents

The following documents are referenced in this Applications Guidance for Recycled PET Protocol:

APR PET Standard Laboratory Practices, PET-P-00

- Injection Molding Preforms, PET-P-09
- Blow Molding Bottles, PET-P-10
- Sheet Extrusion, PET-P-11

APR Screening Tests

- IV Build Rate Test, PET-S-07
- Testing of PET Plaques for Color, Haze and Inclusions, PET-S-09
- Molded Plaque Fluorescence Test, PET-S-13

APR PET Critical Guidance Protocol for Clear PET Resin and Molded Articles, PET-CG-01

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

ASTM F2013-10 (2016) Standard Test Method for Determination of Residual Acetaldehyde in Polyethylene Terephthalate Bottle Polymer Using an Automated Static Head-Space Sampling Device and a Capillary GC with a Flame Ionization Detector

ASTM D5420-98a Standard Test Method for Impact Resistance of Flat, Rigid Plastic Specimen by Means of a Striker Impacted by a Falling Weight (Gardner Impact)

ISBT Methods

Bottle test methods are available with membership from the International Society of Beverage Technologists, http://www.bevtech.org/.

CarboQC Operating Manual

Available for users of CarboQC through Anton-Paar.

Safety Statement: *APR Test and Practice documents do NOT CLAIM TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. These Tests and Practices may require the use of electrically powered equipment, heated equipment and molten polymers, rotating motors and drive assemblies, hydraulic powered equipment, high pressure air, and laboratory chemicals. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH AND FOLLOW APPROPRIATE SAFETY AND HEALTH PROCEDURES WHEN UNDERTAKING THESE TESTS AND PRACTICES THAT COMPLY WITH APPLICABLE FEDERAL, STATE AND LOCAL REGULATORY REQUIREMENTS. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance of these Tests and Practice documents by any party.*

rPET-to-Bottle Protocol

Introduction

The rPET-to-Bottle evaluation program is designed to show processing and bottle performance differences between a control material and that control material containing recycle-content Innovation material. It is a ***comparative*** study that does not rely on the final blown bottles meeting absolute performance criteria.

The rPET-to-Bottle program is designed to allow for a comparative property study of Carbonated Soft Drink (CSD) bottles ranging in size from 0.5L to 2L. The optimal study will be one where the initial control resin selected for use in the CG screening will be selected from the list of APR or EPBP approved CSD and non-Water Bottle Innovation Control PET resins as described in PET-CG-01. The Control CSD virgin resin called for in Step 2 below ideally would be identical to the CG control resin.

It is recognized that if the Innovation being studied in PET-CG-01 was a non-CSD resin or an additive that was incorporated into a non-CSD resin, then the 0.80±0.2 dL/g material that is produced in the CG study (Solid State Processing), **may not be ideal** ***for CSD bottle performance.*** However this non-CSD base material can still be blended with an APR or EPBP approved CSD control begin the rPET-to-Bottle evaluation. When this is the case, it is important to recognize that the resulting control bottles and Innovation recycle-content bottles may not perform ideally in all of the CSD tests. Since the bottle test performance of the Innovation recycle-content bottles will be compared to the control bottles, it will still be possible to judge the Innovation's acceptability for the recycle stream if the bottle test criteria are met. Because the non-CSD control materials are currently found in the recycle stream, then any new materials similar to these that do not result in significant differences in recycle-content bottle performance are, therefore, also expected to be acceptable.

Method Steps

The following steps are followed to mold preforms and blow bottles 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 Standard Laboratory Processing Practices, PET-P-01 through PET-P-10 (included in document PET-P-00):

1. Use the 0.80 dL/g Control and 50:50 Innovation/Control pelletized resin samples obtained from the solid stating step from PET-CG-01. The amount of material will depend upon the equipment and scale used in each laboratory.
2. For each of the test and control samples, prepare the following:
    a. Sample A is the Control virgin resin blended with 50% by weight Control pellets produced from PET-CG-01
    b. Sample B is the Control virgin resin blended with 50% by weight Innovation pellets produced from PET-CG-01
3. Desiccant dry each blend sample separately.
4. Injection mold preforms with the prepared Samples A and B at Step 2 separately.
    a. Test preforms
5. Blow bottles using a reheat/stretch blow molding machine.
    a. Test bottles

Measurements, Report and Guidance Values

Evaluation of Preforms

Property	Method	APR Guidance Preferred values
Required values		
Processing Conditions	PET-P-09	Process B under identical conditions as A. If changes are necessary, note differences.
IV loss when pellets are molded to preforms	ASTM D 4603 solution IV with phenol/tetrachloroethane 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
Acetaldehyde content	ASTM F2013-10	Sample B ≤35% increase over Sample A
Inclusions and specks in 50 preforms of each blend variable	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

Evaluation of Bottles

Property	Method	APR Guidance Preferred values
Required values		
Processing Conditions	PET-P-10	Process B under identical conditions as A. If changes are necessary, note differences.
Black specks, particulates or gels	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
Color & Haze L* a* b*	PET-S-09	Report measurement, no guidance
Bottle dimensions: Height Upper Label Panel Dia. Lower Panel Dia.	ISBT	B should be ± 5% of A
Section Weights: Base Panel Shoulder	ISBT	B should be ± 5% of A
Material Distribution Base Foot Label	ISBT	Report measurement, no guidance
Total Bottle Weight Capacity	ISBT	B should be ± 5% of A
Burst Strength (Burst Pressure)	ISBT	B <10% decrease from A

Top Load (Maximum Load empty)	ISBT	B <10% decrease from A
Drop Impact @ 40°F Bottle axis vertical Bottle axis horizontal	ISBT	 B no more than 1 additional failure than A B no more than 1 additional failure than A
Stress Crack Resistance Average Time	ISBT	B <25% reduction in time compared to A
Shelf-Life CO_2 Loss by FTIR Septum Test CarboQC Test	 ISBT ISBT Anton-Paar	 B ≤ 5% decrease compared to A B ≤ 5% decrease compared to A B ≤ 5% decrease compared to A
Thermal Stability Height Change in Fill Line Upper Panel Increase Lower Panel Increase Number of Rockers	ISBT	 B ≤ 10% increase compared to A B ≤ 10% increase compared to A B ≤ 10% increase compared to A B ≤ 10% increase compared to A 0

rPET-to-Sheet Protocol

Introduction

Today, considerable amounts of both recycled bottle flake and pellets are converted into sheet. Most of the sheet is thermoformed and is used in many applications such as food, electronic, pharmaceutical, and other types of packaging.

Recycle content can range from 0 to 100% of rPET for any of these applications. The recycled material can be in the form of bottle flake or pellets. Most applications do not require the material to be solid stated. The most critical case is then the use of 100% bottle flake or pellets.

In the scope of this rPET-to-Sheet Protocol, the test sample blends, which will be converted into sheet, need to be made using the washed and dried Control and Innovation flake samples or non-solid stated extruded pellets that are produced from the PET-CG-01 test. Note that both the Control and Innovation samples must be in the same form (either flake or pellet) for testing. The reason for this is to simulate the sheet extrusion process that directly extrudes flake or pellets at a 50% by weight blend level. The melting of the material in the extruder can have a significant influence on the process and the difference in shape between a pellet and a flake is a major factor. Furthermore, crystallizing and drying a pellet or a flake could generate some different types of problems. Thus, preferably the test samples should be in the form of flake. However, since a considerable amount of rPET is also converted into sheet from pellets, this test does allow for the use of pelletized rPET where flake may not be available.

The rPET-to-Sheet evaluation program is designed to show processing and unoriented sheet performance differences between a control material and that control material containing recycle-content Innovation material. It is a ***comparative*** study that does not rely on the final sheet meeting absolute performance criteria.

Method Steps

The following steps are followed to produce the flake samples required for evaluation; these steps are illustrated in the flow diagram in Appendix II, and details of each step are presented in the PET-P-00 Standard Laboratory Processing Practices, PET-P-01 through PET-P-11 (included in document PET-P-00). Note that if the rPET-to-Sheet evaluation is to be performed, it will be necessary to increase the quantities of materials used when performing the PET-CG-01 test.

1. Use the washed and dried Control and Innovation flake samples that are obtained from the PET-CG-01 study.
    a. The amount of material will depend upon the equipment and scale used in each laboratory.
    b. If the PET-CG-01 study was not performed, and only a rPET-to-Sheet study is to be done, then follow all the process steps as outlined in PET-CG-01 through the air drying of the washed and rinsed flake.
        i. Melt-filtered extruded flake may be used in place of flake if necessary.
2. Prepare the following test blend samples:
    a. Sample A is the Control flake (or pellets) from PET-CG-01
    b. Sample B is the Control flake (or pellets) blended with 50% Innovation flake (or pellets) from PET-CG-01
3. Desiccant dry each sample separately.

4. Extrude each sample with melt filtration to produce sheet with a target thickness of 0.015±0.002 inches.

Measurements, Report and Guidance Values

Evaluation of Sheet

Property	Method	APR Guidance Preferred values
	Required values	
Processing Conditions	PET-P-11	Process B under identical conditions as A. If changes are necessary, note differences.
Extruder amps	No Method	Maximum ± 10% difference between A and B
Melt Drop between die and roll stack nip	No Method	No die drool or blowouts for A or B
Bank Stability	No Method	No substantial change between A and B
Fuming	Sensory/Visual evaluation, no method	No increase in B over A
Roll Plate Out	Visual evaluation, no method	No increase in B over A
Impact Test	ASTM D5420, geometry GC	B Maximum ± 10% difference between A and B
Gel Count 50 sheet samples of each 2000-2600mm^2	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 If A =3; repeat extrusion
Black Specks 50 sheet samples of each 2000-2600mm^2	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 If A =3; repeat extrusion

Method steps for rPET-to-Strapping Protocol

Introduction

Strapping is a high-performance product made from high molecular weight PET. High tensile strength needed to hold items in place is achieved by the orientation of high molecular weight resin. Strapping manufacturers consider that if PET is suitable for making biaxially oriented bottles and can be solid state polymerized to a target intrinsic viscosity, a measure of molecular weight, that it will be suitable for making strapping. The target intrinsic viscosity, IV, is 0.95 dL/gram when measured by solution viscosity.

PET-CG-01 considers solid stating performance as part of the protocol and has incorporated within its guidelines, provisions for performance at 15 hours of solid stating as a relative comparison between innovation samples and control samples. While the CG defines an absolute IV value of 0.90 dL/g needs to be achieved, in order to meet strapping guidelines, both Sample A and Sample B (normalized to Sample A) must each achieve a minimum IV of 0.95 dL/g.

The strapping industry recognizes that variation occurs in recycled PET including the solid stating rates of various resins, the influences of additives, the activity of solid stating catalysts after the life cycle of the bottle, and the IV of the cleaned flake product. As recognized in the Control Resins definition, there are two populations of intrinsic viscosity for PET bottles.

This rPET-to-Strapping Protocol calls for the innovation bottle sample to demonstrate the ability of achieve a minimum intrinsic viscosity after a defined number of hours of solid-state polymerization. Further testing to web extrusion and orientation are not needed. It is suggested for purposes of economy that the rPET-to-Strapping solid stating examination be conducted as an extension of the PET-CG-01 solid stating examination.

Method Steps

The following steps are followed to produce the flake samples required for evaluation; these steps are illustrated in the flow diagram in Appendix III, and details of each step are presented in the PET-P-00 Standard Laboratory Processing Practices, PET-P-01 through the solid stating processing step PET-P-07. If the Bottle-to-Strapping evaluation is to be performed, it may not be necessary to increase the quantities of materials used when performing the PET-CG-01 test.

1. Follow the processing as outlined in PET-CG-01 up to the solid stating step.
2. The following test blend samples will be studied:
    a. Sample A are the Control pellets from PET-CG-01
    b. Sample B are the Control pellets blended with 50% by weight Innovation pellets from PET-CG-01
3. Desiccant dry each sample separately.
4. Solid state each sample following the methodology outlined in PET-Practices PET-P-07.
    a. Note the time and conditions for Sample A to reach an IV of 0.95 dL/g (not to exceed 15 h. SSP time).
    b. Proceed to SSP Sample B under the same conditions used for Sample A.
    c. If Sample B has not reached an IV of 0.95 dL/g within the same time period as Sample A, solid stating can be continued for an additional time of 5 h.

Measurements, Report and Guidance Values

Evaluation of Solid Stated Pellets

Property	Method	APR Guidance Preferred values
Required values		
Processing Conditions	PET-P-07	Process B under identical conditions as A. If changes are necessary, note differences.
IV after SSP	PET-S-07	A must achieve a minimum of 0.95 dL/g B must achieve a minimum of 0.95 dL/g when normalized to A within no more than an additional 5 hours of SSP time.

Method steps for rPET-to-Fiber Protocol

Introduction

Fiber making, primarily but not exclusively staple fiber, is a major end use for recycled PET. The polymer requirements for staple fiber are met or exceeded by the requirements to make bottles and sheet. The fiber concerns for filterable contamination are met by the PET Critical Guidance Document filtration analysis. Fiber concerns about ability to heat set fibers are addressed in the Critical Guidance observation about non-sticking in dryers and the ability to make good bottles. Fiber concerns about gels and spinning defects are addressed by the limits in Bottle-to-Sheet testing. Fiber concerns about modulus and tenacity are met by the ability to make satisfactory bottles in the Bottles-to-Bottle protocol. Fluorescence is one area of concern for fiber not addressed in other testing. rPET that contains fluorescing resins or additives can be disruptive to the use of those postconsumer bottles for fiber as fluorescence interferes with the dying used in the fiber industry. Quantifying fluorescence is beyond this protocol. Innovation samples should not have a visual increase in fluorescence compared to control samples. Photographic evidence can show lack of increased fluorescence.

Method Steps

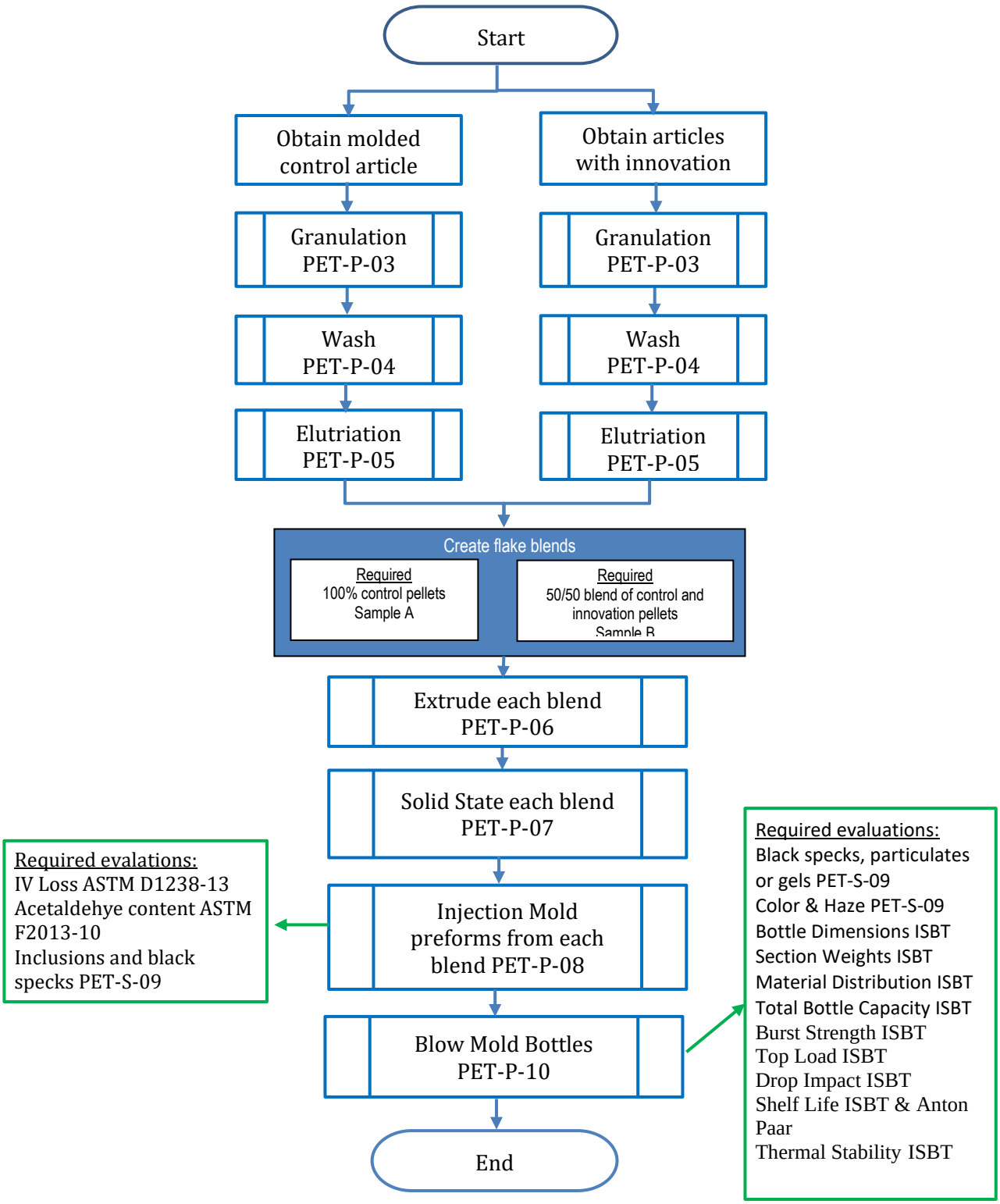
The 3mm plaques molded during the performance of the PET-CG-01 test protocol are measured for fluorescence, these steps are illustrated in the flow diagram in Appendix IV, and details of each step are presented in the PET-P-00 Standard Laboratory Processing Practices, PET-P-01 through the 3mm plaque injection molding processing step PET-P-08. No other processing steps are required.

1. Follow the processing as outlined in PET-CG-01 to obtain the injection molded plaques.
2. The following test samples will be studied:
    a. Sample A are the Control plaques from PET-CG-01
    b. Sample B are the Control plaques molded from 50% by weight Control pellets and 50% by weight Innovation pellets from PET-CG-01
3. Irradiate 5 randomly selected plaques of each variable with ultraviolet light with a wavelength between 320 and 390 nm
    a. Visually confirm that all 5 plaques are visually similar within each Sample
    b. Visually compare the fluorescence intensity of Sample B to Control Sample A.
    c. Obtain photographic images of a representative plaque from each Sample

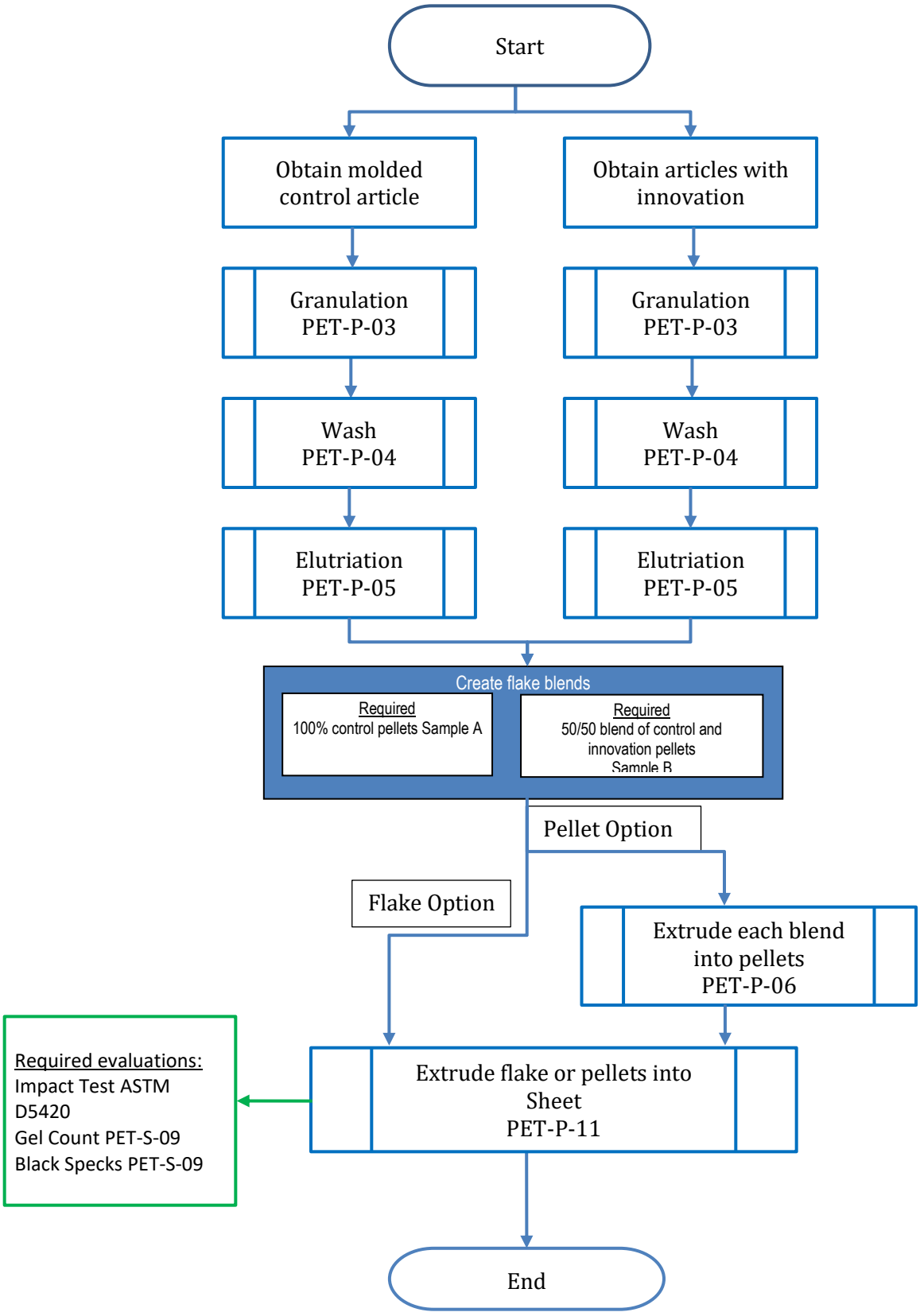
Evaluation of Plaques for Fluorescence

Property	Method	APR Guidance Preferred values
Required values		
Fluorescence	PET-S-13	No noticeable visual increase of emission fluorescence intensity for B vs. A. Photographic evidence is sufficient.

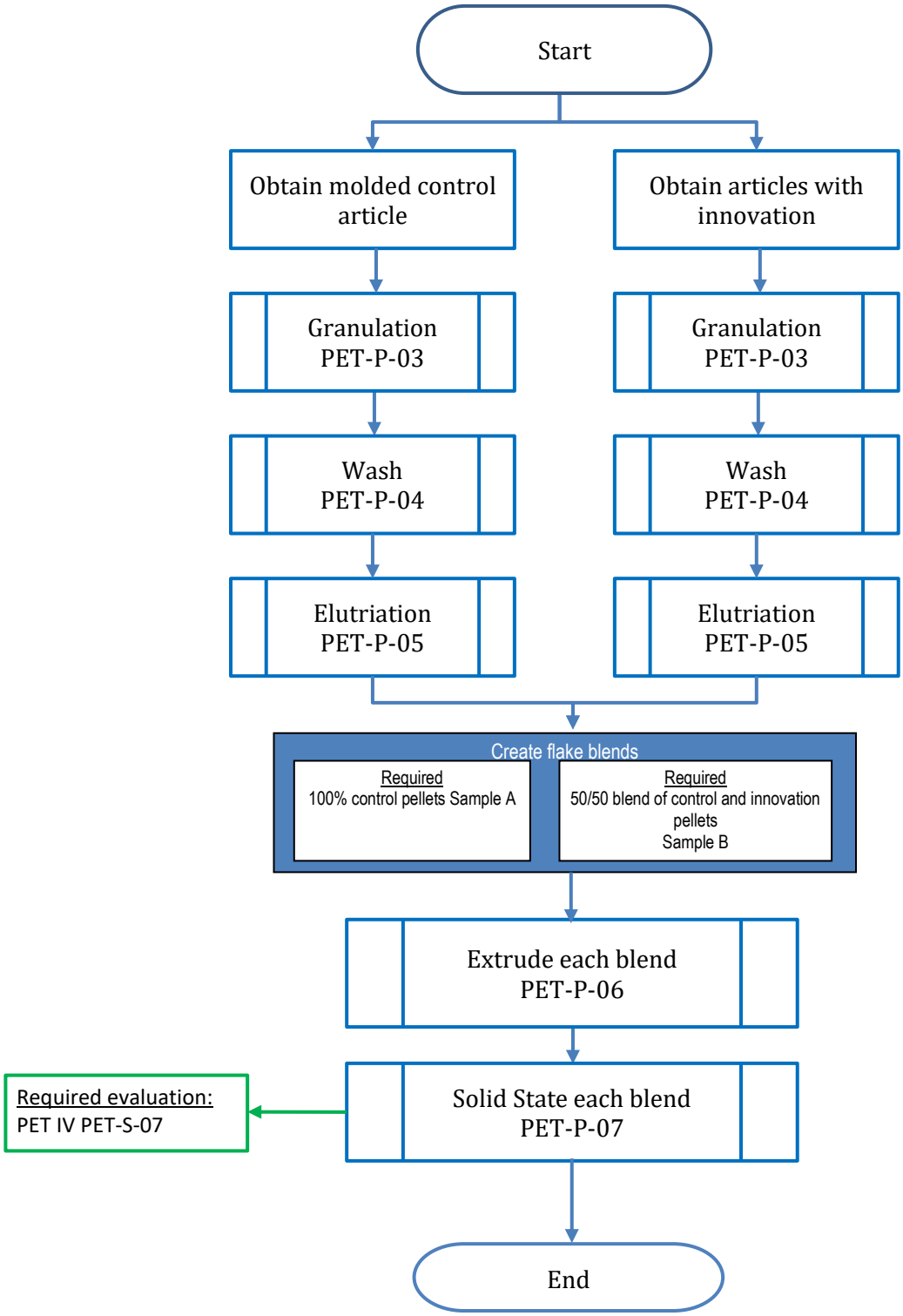
Appendix I: Flow Diagram for rPET-to-Bottle



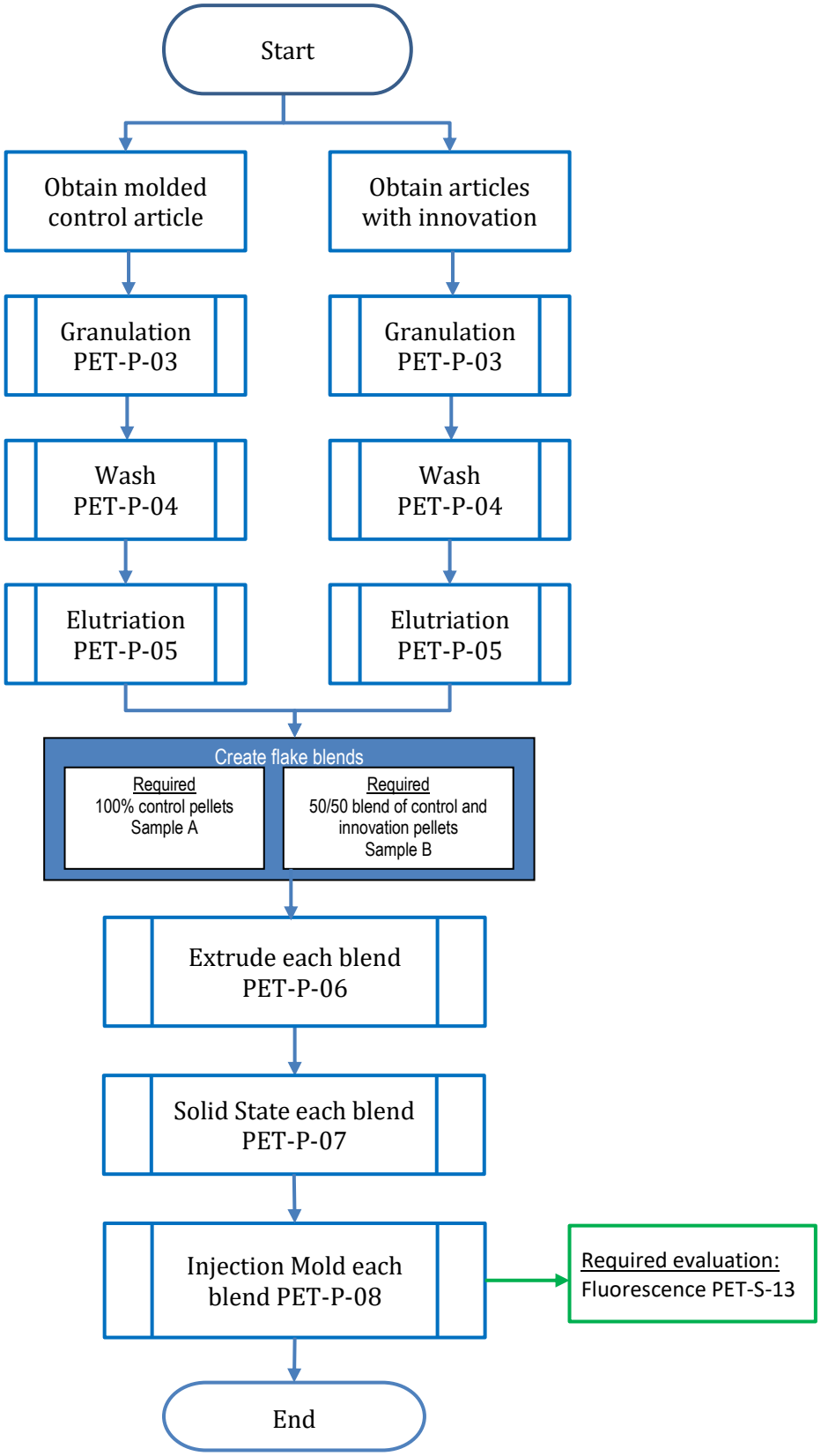
Appendix II: Flow Diagram for rPET-to-Sheet



Appendix III: Flow Diagram for rPET-to-Strapping



Appendix IV: Flow Diagram for rPET-to-Fiber



DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	February 27, 2020	Original Version
2	September 2, 2024	Added hyperlinks to reference documents to match new website