

# Critical Guidance Protocol for PE Film and Flexible Packaging

Document number – FPE-CG-01

Revision date - August 31, 2020

## Introduction – Scope, significance and use

This is a comprehensive laboratory scale evaluation, or protocol, that can be used to assess the compatibility of PE-based films and flexible packaging innovations with film reclamation systems sourcing post-consumer film from store drop-off collection points or, in some cases, curbside collection. For definitions, reference “Plastics Recycling Testing Terminology” at <https://plasticsrecycling.org/apr-design-guide/resources/testing-terminology>

This test can be used to evaluate the impact of innovative PE film packaging components for which recycling compatibility is unknown or for which data is notably lacking. As examples: mono- and multi-layer constructions, coatings, additives (including compatibilizers along with innovative material), printing inks and pigments, labels with polymer substrate (paper labels are out of scope for Critical Guidance), adhesives, or new PE resin co-polymer or multi-material compositions. This test requires assessment of the effect of the packaging in blown film. This test evaluates compatibility of the flexible packaging innovation with current, industrial-scale, film-to-film recycling processes.

Plastic film is generally defined as plastic items with a thickness of less than 10 mils (i.e., 0.010” or 0.25 mm) that are at least 95 percent (by weight) plastic with up to 5 percent other closely bonded or impregnated material, which may include printing, coatings, or fillers. Film, when used in packaging, is referred to as flexible packaging. The shape of flexible packaging typically changes when it is full of a product compared to when it is empty, whereas the shape of rigid packaging generally remains the same. In this document, the term “film” is used to mean flexible packaging which is expected to be primarily PE but may include a small percent of other polymers to meet certain desired properties.

In APR Processing Practices and Test Protocols, the term “article” is used to mean the film or flexible 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 to APR’s Critical Guidance Recognition Program. Petitions require data for a control material, and for the innovation articles blended with extruded articles.

The test is not appropriate for package constructions that will not meet APR recommendations per the protocol SORT-B-03, *Evaluation of Sorting Potential for Plastic Articles using Metal, Metallized, or Metallic Printed Components*. 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.

**Disclaimer:** *This document has been prepared by the Association of Plastic Recyclers as a service to the plastic industry to promote the most efficient use of the nation's plastic recycling infrastructure and to enhance the quality and quantity of recycled postconsumer plastic. The information in this document is offered without warranty of any kind, either expressed or implied, including WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, which are expressly disclaimed. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance upon this information by any party. Participation in the Recognition Program is purely voluntary and does not guarantee compliance with any U.S. law or regulation or that a package or plastic article incorporating the innovation is recyclable or will be recycled.*

## Method Summary

This Critical Guidance protocol provides three pathways for testing resins and articles with flow diagrams found on pages 11 through 16 for articles that employ multi-layer constructions, coatings, additives (including compatibilizers with the innovative material), printing, labels, or new PE resin co-polymer or multi-material compositions. Controls are comprised of similar articles also made solely with a control PE resin without the innovation. All processing practices are to be performed per the PE Film Standard Laboratory Processing Practices, document FPE-P-00.

To account for variations in characteristics of test material as well as laboratory capabilities, several flow diagrams are provided that describe pathways to process material and make test blends. Typically, material will be size reduced prior to extruding into pellets for blending, but these steps may be eliminated in certain cases if the film is not suitable for granulation. See method steps for further clarification.

Path 1A, 2A and 3 are utilized for complete PE control and test articles in the form of flexible or film materials.

Path 1B and 2B are utilized when the control material cannot be secured in the form of flexible or film and is extruded to match the number of heats as the film before processing.

### Path 1A and 1B

A flow diagram for Path 1A and 1B, on pages 14-15 illustrates that these articles are separately processed through these steps:

- Optional granulation
- Mechanical densification
- Blends created from the densified materials. Required blends are 100% control material, a 50/50 blend of control material and test material, and 100% innovation concentration.
- Blends dried, extruded and pelletized
- Pellets subjected to testing and evaluation.
- Pelletized material blown into monolayer film for further testing and evaluation.  
(The 100% innovation is required to be pelletized and blown into test film per FP-P-00 Film Processing Practices. Test results are to be recorded and reported; no thresholds required.)

### Path 2A and 2B

A flow diagram for Path 2A and 2B, on pages 16-17 illustrates that these articles are separately processed through these steps:

- Oven densification
- Granulation
- Blends created from the densified materials. Required blends are 100% control material, a 50/50 blend of control material and test material, and 100% innovation concentration.
- Blends dried, extruded and pelletized
- Pellets subjected to testing and evaluation
- Pelletized material blown into monolayer film for further testing and evaluation.  
(The 100% innovation is required to be pelletized and blown into test film per FP-P-06 Film Processing Practice. Test results are to be recorded and reported; no thresholds required.)

### Path 3

A flow diagram for Path 3, on page 18 illustrates that these articles are separately processed through these steps:

- Without previous densification and/or size reduction the material may be fed directly into the extruder with pressure maintained for the control and test materials.
- Blends created from the whole articles. Required blends are 100% control material, a 50/50 blend of control material and test material, and 100% innovation concentration. Care must be taken when preparing the 50/50 blend to be accurate in weighing and mixing the control with the test material.
- Blends dried, extruded and pelletized
- Pellets subjected to testing and evaluation
- Pelletized material blown into monolayer film for further testing and evaluation.

## Reference Documents

The following documents are referenced in this Critical Guidance Protocol:

APR PE Film Standard Laboratory Processing Practices, FPE-P-00 through FPE-P-07

APR Screening Test Methods:

Polyolefin Packaging Article Sink or Float Evaluation, O-S-01

PE Film Surface Impression Evaluation and Impurity Visual Inspection, Appendix A

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 Film Test Methods:

ASTM D6988 Thickness of Plastic Film Test Specimens

ASTM D1922 Propagation Tear Resistance of Plastic Film and Thin Sheeting by Pendulum Method

ASTM D882 Tensile Properties of Thin Plastic Sheeting

ASTM D1709 Impact Resistance of Plastic Film by the Free-Falling Dart Method

## Method Steps for Extrusion Blends and Blown Film

**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.

### Important Note on Control Articles and Resins:

The user should read FPE-P-01, Selection of Control Film or Control Resin (part of document FPE-P-00, PE Film Standard Laboratory Processing Practices), carefully to ensure that they understand all selection criteria for control articles or resins, prior to commencing this test protocol.

### Path 1A and Path 1B Method Steps

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagrams labeled Path 1A and 1B, and details of each step are presented in the PE Film Practices, FPE-P-01 through FPE-P-07 (included in document FPE-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.

1. Obtain control and test film to use in the evaluation for Path 1A
2. Sink-float the control and test film materials per the O-S-01
3. Obtain control resin and test film to use in the evaluation for Path 1B
4. For each of the test and control articles for Path 1A, separately:
  - a. Granulate the articles. This step is optional if granulation is not practical for the specific film article and if the mechanical densifier used can accept whole articles.
  - b. Mechanically Densify articles
5. Extrude control virgin resin if required per Path 1B, for a single heat to represent the initial film production heat history.
6. Prepare the following required blends:
  - a. 100% densified control or pelletized control - Sample A
  - b. 50/50 blend of densified control or pelletized control with the innovation article – Sample B
  - c. 100% of densified innovation – Sample C
7. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create the samples: A pellets, B pellets and C pellets.
8. Sample A, B and C extruded pellets to be evaluated for DSC, melt flow, density, ash content, moisture content, bulk density and color.

9. Extrude A, B and C sample pellets into blown film per FP-P-00 Film Processing Practices for required blown film testing.
  - a. Sample A and B extrusion blown film to be evaluated for thickness, tear, tensile strength and elongation, and dart drop. Consideration for Critical Guidance Recognition will require that Sample B blend passes ASTM testing per APR requirements in Data Tables below.
  - b. Sample C is required to be successfully blown into test film per FP-P-00 Film Processing Practices. ASTM test results are to be recorded and reported but will not count against consideration for Critical Guidance Recognition.

### Path 2A and Path 2B Method Steps

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagrams labeled Path 2A and 2B, and details of each step are presented in the PE Film Practices, FPE-P-01 through FPE-P-07 (included in document FPE-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.

1. Obtain control and test film to use in the evaluation for Path 2A
2. Sink-float the control and test film materials per the O-S-01
3. Obtain control resin and test film to use in the evaluation for Path 2B
4. For each of the test and control articles for Path 2A, separately:
  - a. Oven densify the articles
  - b. Granulate the densified materials
5. Extrude control virgin resin if required per Path 2B, to represent the initial film production heat history.
6. Prepare the following required blends:
  - a. 100% densified control or pelletized control - Sample A
  - b. 50/50 blend of densified control or pelletized control with the innovation article – Sample B
  - c. 100% of densified innovation – Sample C
7. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create the samples: A pellets, B pellets and C pellets.
  - a. Samples A, B and C extruded pellets will be evaluated for DSC, melt flow, density, ash content, moisture content, bulk density and color.
8. Extrude A, B and C sample pellets into blown film per FP-P-00 Film Processing Practices for required blown film testing.
  - a. Sample A and B extrusion blown film to be evaluated for thickness, tear, tensile strength and elongation, and dart drop. Consideration for Critical Guidance Recognition will require that Sample B blend passes ASTM testing per APR requirements in Data Tables below.
  - b. Sample C is required to be successfully blown into test film per FP-P-00 Film Processing Practices. ASTM test results are to be recorded and reported but will not count against consideration for Critical Guidance Recognition.

### Path 3 Method Steps

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagram labeled Path 3, and details of each step are presented in the PE Film Practices, FPE-P-01 through FPE-P-07 (included in document FPE-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.

1. Sink-float the test film single heat material per the FPE-S-01
2. Required blends are 100% control film; and a 50/50 blend of control material and test film. Optional blends can be used if desired by the investigator at 100% innovation concentration.
3. Prepare the following required blends:
  - a. 100% control film - Sample A

- b. 50/50 control film and innovation film– Sample B
  - c. 100% of densified innovation – Sample C
4. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create the samples: A pellets, B pellets and C pellets.
    - a. Sample A, B and C extruded pellets to be evaluated for DSC, melt flow, density, ash content, moisture content, bulk density and color.
  5. Extrude A, B and C sample pellets into blown film per FP-P-00 Film Processing Practices for required blown film testing.
    - a. Sample A and B extrusion blown film to be evaluated for thickness, tear, tensile strength and elongation, and dart drop. Consideration for Critical Guidance Recognition will require that Sample B blend passes ASTM testing per APR requirements in Data Tables below.
    - b. Sample C is required to be successfully blown into test film per FP-P-00 Film Processing Practices. ASTM test results are to be recorded and reported but will not count against consideration for Critical Guidance Recognition.



## Flake and Pellet Measurements, Report and Guidance Values

APR Guidance is for A (0%) and B (50%) Preferred Values Only – C (100%) to be recorded and reported

### Path 1A, 1B, 2A, 2B and 3 Incoming Film Analysis

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

### Path 1B or 2B Control 1<sup>st</sup> Melt History Extruded Pellet

Property	Method	APR Guidance Preferred values	Additional Guidance
<u>Optional Values</u>			
Screen Pack Pressure Build	Practice FPE-P-06	Record and Report	

### Extruded Pellet Evaluation All Paths

Property	Method	APR Guidance Preferred Values for All Samples	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	< 0.75 g/10minutes 25% delta to the control	
Density	ASTM – D792 or ASTM 1505	<0.996 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 <sup>3</sup>	
FTIR	ASTM-D7399		Identify PP homopolymer, record and report
DSC	ASTM – D3418	Primary Peak not to exceed 150°C	Record primary and secondary peaks temp. and J/g. Calculate the J/g Δ from primary peak to secondary peak if present
Pellet Irregularity	Porosity, roughness, grainy, gloss etc.		Record, Report, and Photograph
Extrusion Process Irregularity	Unusual sticking, fumes, odor or build-up at the feed throat or die exit of the extruder		Record, Report, and Photograph
<u>Optional value</u>			
Pellet Color	ASTM – D6290		Can reveal contamination before later evaluation

**Film Measurements, Report and Guidance Values**
**APR Guidance is for A (0%) and B (50%) Preferred Values Only – C (100%) to be recorded and reported**
**Evaluation of Blown Film Samples All Paths**

Property	Method	APR Guidance Preferred values for Samples A and B
Required values		
Thickness	ASTM – D6988	1.5-2.5 target range (allowable delta from target +/- 20% within range) mils per FPE-P-06
Tear Strength (MD/TD)	ASTM – D1922	No more than a 25% $\Delta$ drop to the control. Record and report % increase.
Tensile Strength (MD/TD)	ASTM – D882	No more than a 25% $\Delta$ drop to the control. Record and report % increase.
Elongation @ Yield (MD/TD)	ASTM – D882	No more than a 25% $\Delta$ drop to the control. Record and report % increase.
Dart Impact	ASTM – D1709	No more than a 25% $\Delta$ drop to the control. Record and report % increase.
Surface Impression	FPE-S-01	No more than a 25% count increase to control. Offset increase round up for count.
Impurity Visual Inspection	FPE-S-01	Record and Report for Film Appearance Rating (F.A.R.) provide in table below.

## APPENDIX A

### Evaluation of Blown Film Appearance Rating (F.A.R.) Calculation Totals for Control and Test

#### PE Film Surface Impression and Impurity Evaluation

The evaluation is to clearly display and put a significance to the amount of surface and visual impurities in a blown film sample. Direct visual comparison of film samples can be subjective in quantifying the impurities that reside in the film. Preparing a facsimile of the film sample by tracing allows a more objective way of quantifying these defects.

NOTE: The use of this test is optional. If an innovator has optical scanning equipment to measure visual impurities in blown film samples, that equipment may be used instead for Critical Guidance Recognition applications. The applicant must interpret the results of the optical scanning testing on a comparable scale with this FAR test.

#### **Test Method Summary**

Three film samples, each cut to a suggested minimum size of 9 inches by 12 inches (or other sizes with the equivalent of 108 square inches total), are to be clamped down onto a flat clean surface. Tracing paper is to be laid on top of film surface so that all wrinkles are removed from both film and paper. A crayon is then run over the entirety of the paper, capturing all impurities as impressions on the paper. The impurities are assessed by count and rating system per the PE film Critical Guidance Protocol.

Size estimates for specks, gels and fisheyes should be made by comparison with a size chart such as Tappi T564 or similar index.

#### **Equipment Required**

- Clamps
- Light box or projector

#### **Materials Required**

- Flat Clean Surface
- 9 inch by 12 inch Tracing Paper
- Triangular Crayon (label removed)
- Film Samples x 3 per variable

#### **Definitions**

- Gels: Visually uniform small clumps of cross-linked polymers
- Specks: Very small contaminants, possibly darker in color
- Fisheyes: Small round or oblong shapes with darker/more concentrated centers
- Holes: Tears in the blown film bubble starting at, or caused by, gels, specks or fisheyes, whether they cause a strand break or not. The FAR Test allows no tolerance for holes that cause film break to destruction.

## Surface Impression Evaluation

- 1) Cut 3 new film samples to the size of 9 inches x 12 inches, or sizes totaling 108 square inches.
- 2) Clean the working surface to remove existing impurities
- 3) Clamp a single film onto the flat surface in order to remove any ripples
- 4) Place the tracing paper on top of the film and hold firmly so minimal movement is made
- 5) Using a crayon on the side surface, starting in the top left corner apply minimal pressure and move to the top right corner
- 6) Start back on the left, one layer down on the blank surface repeat step 5
- 7) Repeat steps 5 and 6 until the entire tracing paper sheet has been marked up
- 8) Complete steps 2 through 7 for two additional samples per variable
- 9) Examine the tracing paper, identify and count surface defects including gels, black specks, and fisheyes.  
To identify and distinguish defects on the tracing paper, use Tappi size chart T564 or similar index.
- 10) Calculate an average surface raised impression count
- 11) Perform the FAR Impurity Visual Inspection
- 12) Fill out the FAR Evaluation Table per the PE Film Critical Guidance Protocol, and enter results below.

## Impurity Visual Inspection

- 1) Place two blown film samples totaling 108 sq/inches flat film specimens, on top of each other on the viewing plate of the projector
- 2) Place the transparent plastic plate/sheet with standard holes in contact with the film on the specimens and press firmly to remove wrinkles as far as possible
- 3) Focus the projector so that the gel images and the image of the gel size standards are clearly projected on the screen or can be viewed directly on the plastic size plate/sheet with a magnification of 1.5X to 5X
- 4) Count and record the number of gels equal to or larger than the 0.80-mm standard. For reference, the 0.8 mm standard is derived from ASTM Test Method D3351, since withdrawn.
- 5) Repeat steps above for each variable

### Control Baseline Requirements:

- Allowable defects in control must not exceed 30 gels and 5 specks or unmelts. No holes are allowed in the control.
- Gels in test material that measure larger than 0.8 mm in any dimension should not exceed 30 delta to control per APR FAR Test or optical scanning testing.
- Carbon Black, Specks and Unmelts in test material are not to exceed 5 delta to control per APR FAR Test or optical scanning testing
- Size estimates for specks, gels and fisheyes should be made by comparison with a size chart such as Tappi T564 or similar index.

### Evaluation and Scoring:

- Use table following for rating value calculation.
- **Visual Inspection Evaluation for Film Appearance Rating (F.A.R.):** \_\_\_\_\_

**PE Film Surface Impression Evaluation and Impurity Visual Inspection  
FAR Rating Table**

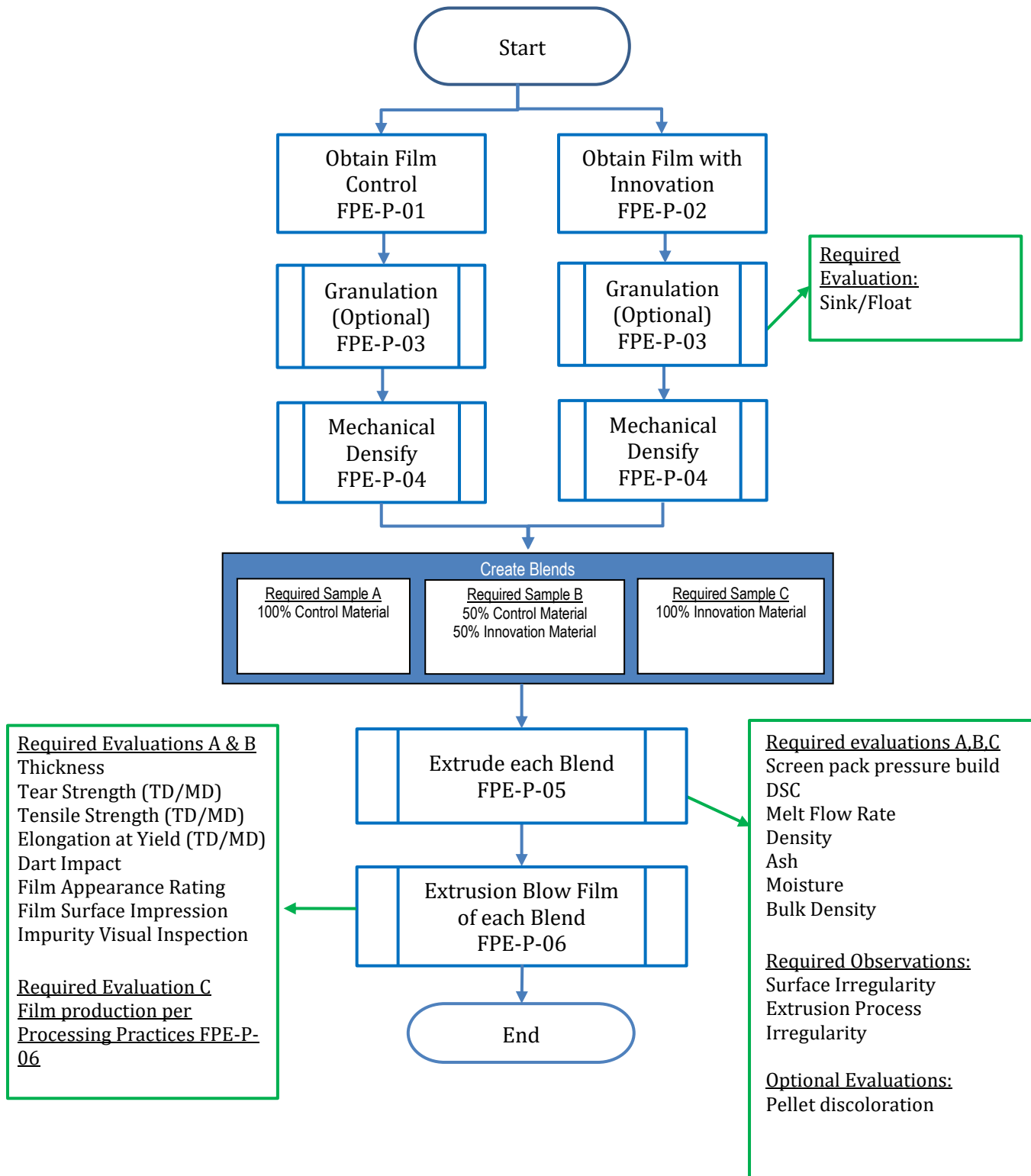
Defect Description	Rating				Test
	Very Smooth	Smooth	Rough	Very Rough	
<b>Texture</b>	0	1	2	3	
<b>Gels Larger than 0.8 mm</b>	<b>Delta to Control (0 to 30)</b>	<b>31 to 50</b>	<b>51 to 60</b>	<b>More than 60</b>	
(Defined as visually uniform small clumps of cross-linked polymers)	0	1	2	3	
<b>Carbon black particles/Specks/unmelts</b>	<b>Delta to Control (1 to 5)</b>	<b>6 to 10</b>	<b>10 to 15</b>	<b>More than 15</b>	
(Defined as very small contaminants, possibly darker in colour)	0	1	2	3	
<b>Fisheyes</b>	<b>Delta to Control 0</b>	<b>1 to 3</b>	<b>4 to 6</b>	<b>&gt; 6</b>	
(Defined as small round or oblong shapes with darker/more concentrated center)	0	1	2	3	
<b>Holes</b>	<b>None</b>	<b>1 or more</b>			
(Defined as tears in the blown film bubble starting at, or caused by, gels, specks or	0	12			0
				<b>Totals</b>	<b>0</b>

<b>Gels</b>
Control should never exceed 30
<b>Specks/unmelts</b>
Control should never exceed 5
<b>Holes</b>
Zero Tolerance

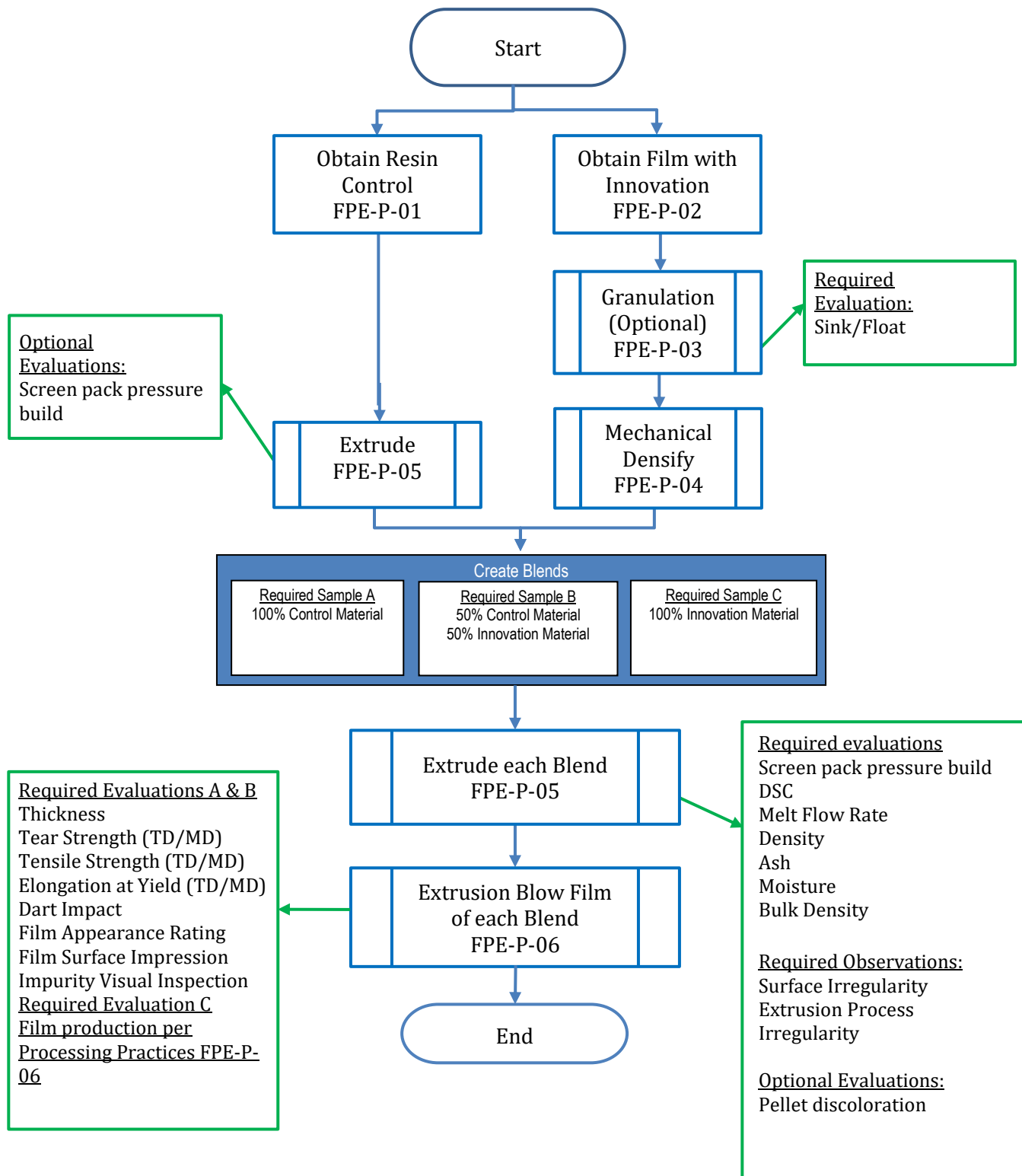
A Grade	0 to 6	Preferred
B Grade	7 to 10	Detrimental
C Grade	11 or greater	Non Recyclable



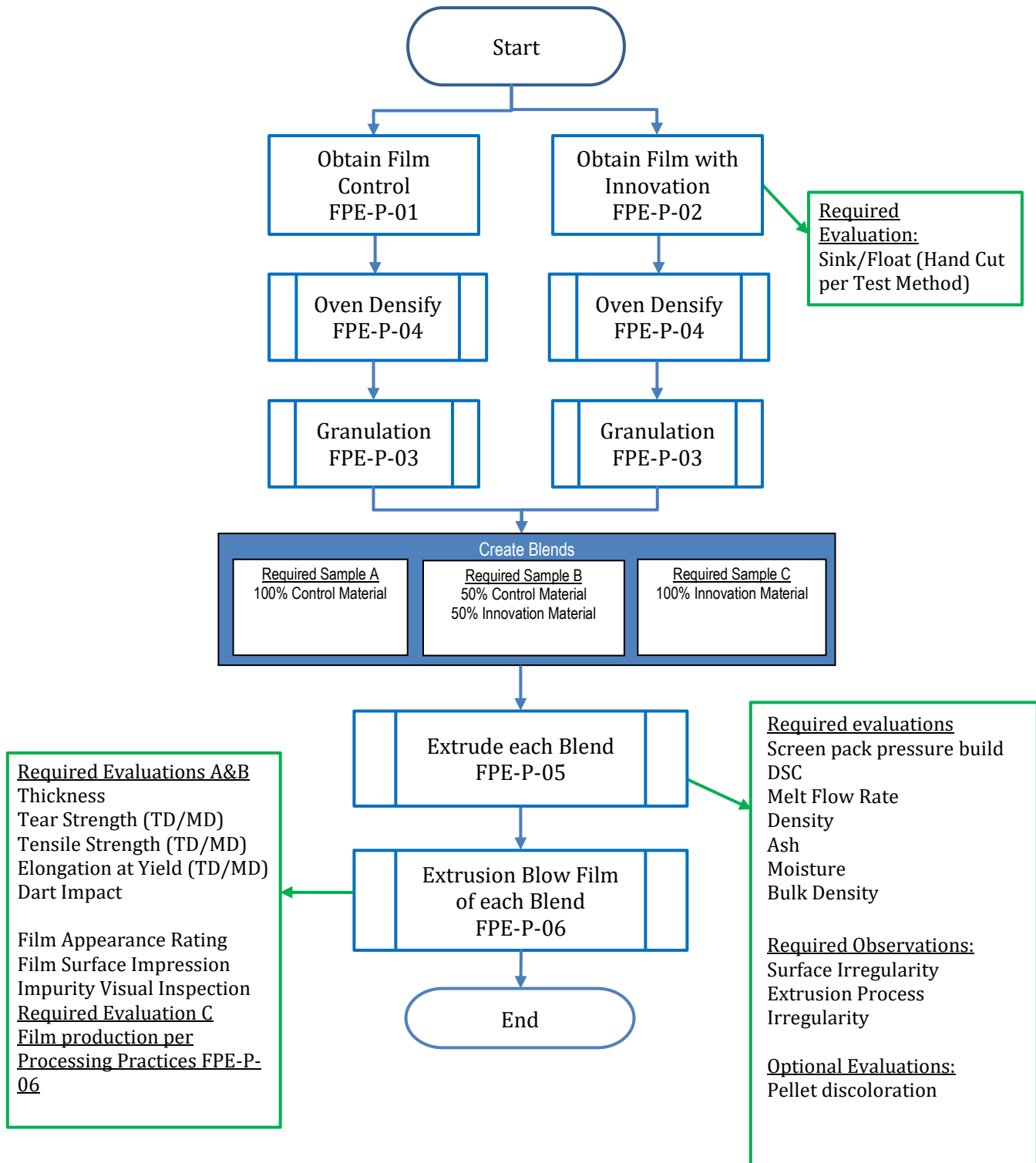
Path 1A: Flow Diagram for Control and Test PE Films that employ new resins, additives, coatings, layers, adhesives or blends (Grind then Densify)



Path 1B: Flow Diagram for Control as Resin and Test as Film (Grind then Densify)

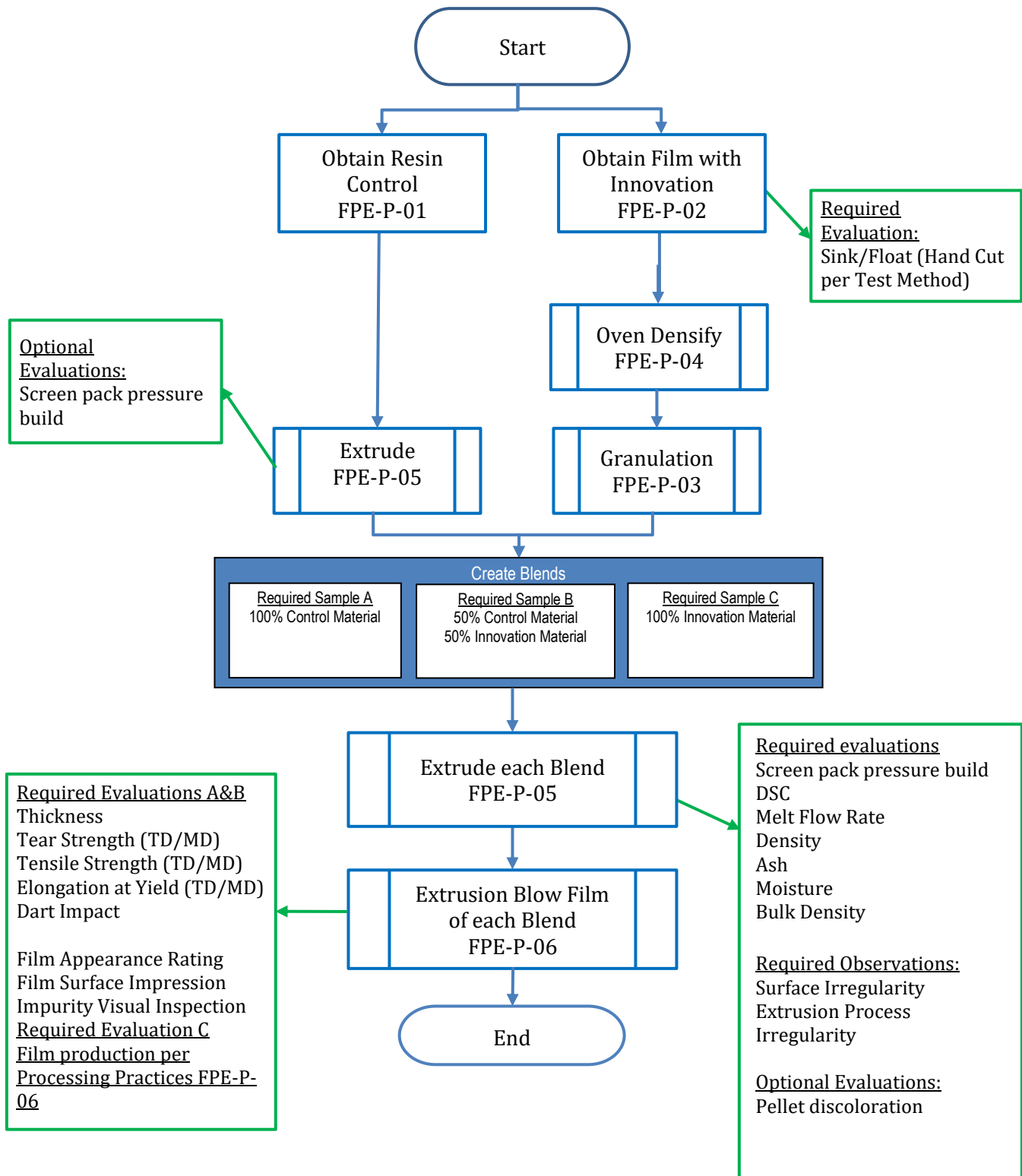


Path 2A: Flow Diagram for Control and Test PE Films that employ new resins, additives, coatings, layers, adhesives or blends (Densify then Grind)

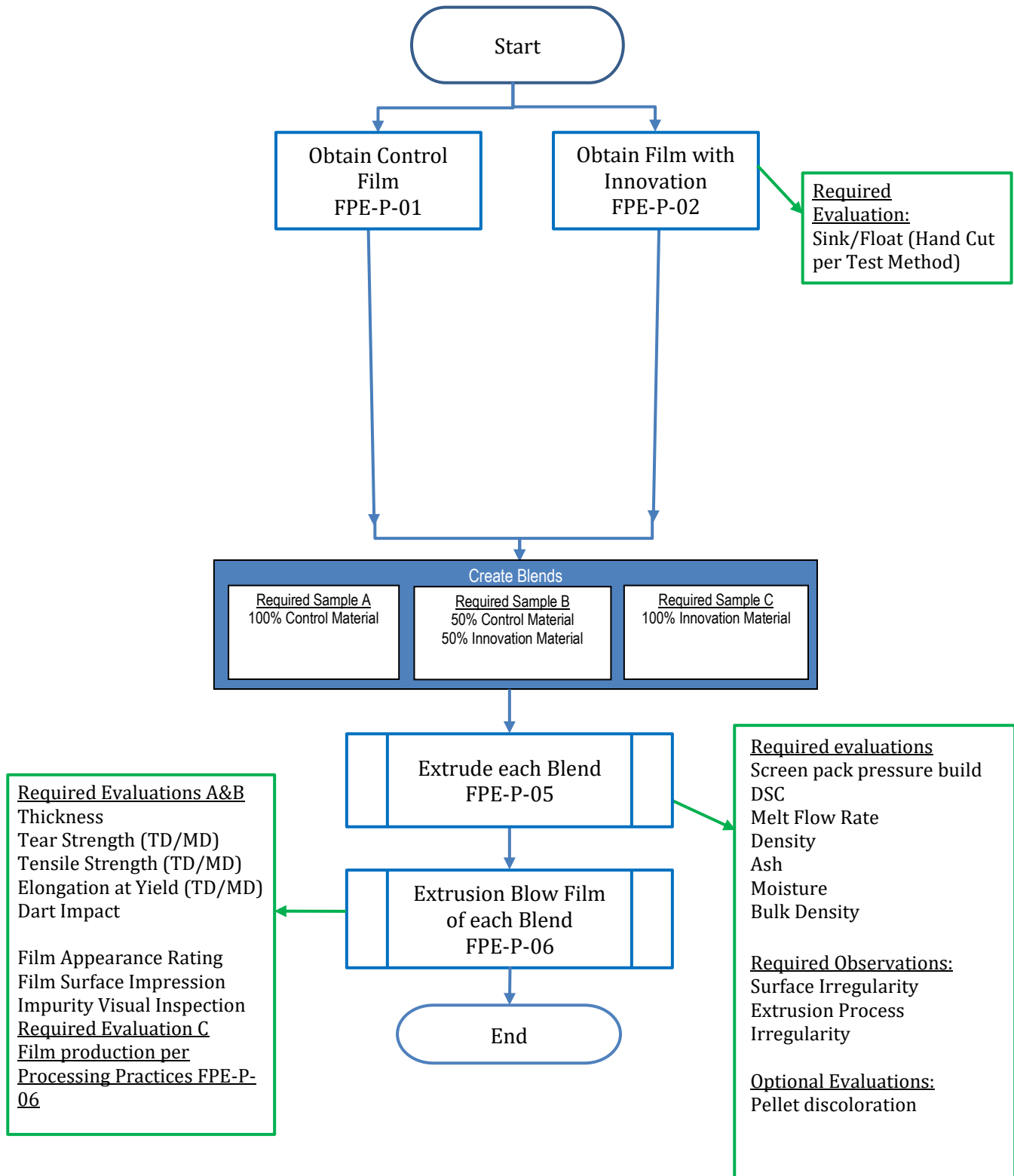




## Path 2B: Flow Diagram for Control as Resin and Test as Film (Densify then Grind)



### Path 3: Flow Diagram for Control and Test PE Films Direct Feed (No Grind or Densify)



## CURRENT VERSION HISTORY

Version	Publication Date	Revision notes
1	August 31, 2020	