APR Benchmark Polyethylene(PE) Films and Flexible Packaging Innovation Test Protocol

Document number – FPE-B-01 **Publication or Revision Date**: September 9 , 2024

Concise title – "APR Benchmark Polyethylene(PE) Films and Flexible Packaging Test Protocol"

Introduction –Scope, Significance and Use

This method presents the steps used in processing and testing for a laboratory to evaluate the compatibility of PE-based films and flexible packaging design features with certain film reclamation systems sourcing post-consumer film from store drop-off collection points or, in some cases, curbside collection. The method also provides a report form for data and guidance from APR on evaluation of the results.

This test can be used to evaluate the impact of PE film design features and technologies, including multi-layer constructions, coatings, additives, printing, labels, or new PE resin co-polymer or multi-material compositions.

This test includes assessment of the effect of the material composition in both ASTM test parts and thicker gauge blown films up to 4.0 mil to model the expected end use applications of composite lumber and thick films such as construction sheeting or industrial liners. The APR recognizes the importance of a diversity of end-use applications for recycled PE films.

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 design feature or material which requires test data for an APR recyclability classification.

The Benchmark test is not appropriate for package constructions that will not meet APR recommendations per the protocol SORT-S-03, *Evaluation of Sorting Potential for Plastic Articles using Metal, Metallized, or Metallic Printed Components*. If the sortation protocol does allow a Detrimental ruling on the article in question, it will qualify for the Benchmark protocol with the understanding that the metal sorting potential test does not allow any article to qualify as Preferred. This Benchmark 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 the evaluation.

Notes on Food Residue

The purpose of this test protocol is to evaluate the design of a flexible packaging material. While the ultimate use of the package and its contents may be dependent on design, it is not the role or intention of APR test protocols to evaluate the fitness of a package for original use or to comment on appropriate contents. However, a quality supply stream is important to the overall health and advancement of the PE film recycling industry. To this end, brands that choose to package food, cosmetics, cleaning products, or any sticky or wet substance in a flexible package should be aware of "clean and dry" requirements for store drop-off recycling as well as prohibitions on food waste and residual liquids in bale specifications used by buyers and sellers.

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.*

Method Summary

This Benchmark test protocol provides three pathways for testing resins and articles (see flow diagrams found on pages 9 through 13) 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 made solely with a control PE resin without the innovation. Typically, material will be size reduced prior to extruding into pellets for blending, but these steps may be bypassed in certain cases if the film is not suitable for granulation. See method steps for further clarification.

All method steps are to be conducted per the PE Film Standard Laboratory Processing Practices, FPE-P-00.

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 film and instead is in the form of resin pellets. If a resin pellet is selected as a control, the specific grade should be as close as possible in properties (density, MFI, molecular structure, etc) as the major PE component of the test film. Pellets are to be extruded to establish a heat history before testing. Film controls are encouraged to avoid any confounding factors when comparing a resin control to a blown film test article.

Path 1A and 1B

A flow diagram for Path 1A and 1B illustrates processing through these steps:

- Granulation (optional)
- Mechanical densification
- Sample blends created from the densified materials.
    - Sample A: 100% control material
    - Sample B: 50/50 blend of control material and test material
    - Sample C: 75/25 blend of control material and test material
- Blends dried, extruded and pelletized
- Pellet samples A and B subjected to testing and evaluation.
- Pellet samples A and B injected molded into ASTM test bars for testing and evaluation
- Pellet samples A and C blown into 3.0-4.0 mil monolayer film samples for further testing and evaluation.

Path 2A and 2B

A flow diagram for Path 2A and 2B illustrates processing through these steps:

- Oven densification
- Granulation
- Sample blends created from the densified materials.
    - Sample A: 100% control material
    - Sample B: 50/50 blend of control material and test material
    - Sample C: 75/25 blend of control material and test material
- Blends dried, extruded and pelletized
- Pellet samples A and B subjected to testing and evaluation.
- Pellet samples A and B injected molded into ASTM test bars for testing and evaluation
- Pellet samples A and C blown into 3.0-4.0 mil monolayer film samples for further testing and evaluation.

Path 3

A flow diagram for Path 3 illustrates processing 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.
- Sample blends created from the whole articles.
    - Sample A: 100% control material
    - Sample B: 50/50 blend of control material and test material
    - Sample C: 75/25 blend of control material and test material
- Blends dried, extruded and pelletized
- Pellet samples A and B subjected to testing and evaluation.
- Pellet samples A and B injected molded into ASTM test bars for testing and evaluation
- Pellet samples A and C blown into 3.0-4.0 mil monolayer film samples for further testing and evaluation.

Reference Documents

The following documents are referenced in this Protocol:

APR PE Film Standard Laboratory Processing Practices, FPE-P-00 (incorporating FP-P-01 through FPE-P-07).

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 Molded Parts Test Methods:

ASTM D638 Tensile Strength
ASTM D638 Elongation at Yield
ASTM D256 Notched Izod
ASTM D1238 Melt flow

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 Testing

The following steps are taken to prepare samples for evaluation; these steps are illustrated in the flow diagrams. Details of each step are presented in the PE Film Practices, FPE-P-01 through FPE-P-05, and FPE-P-07 (included in document FPE-P-00). The amount of material will depend upon the equipment and scale used in each laboratory.

Path 1A and Path 1B Method Steps

1. Obtain control and test film to use in the evaluation for Path 1A
2. Obtain control resin and test film to use in the evaluation for Path 1B
3. 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
4. Extrude control resin if required per Path 1B, to represent the initial film production heat history.
5. 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
6. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create pellet samples A and B.
    a. Pellet samples A and B are to be evaluated for DSC, FTIR, melt flow, density, ash content, moisture content, bulk density and color.
7. Injection mold A and B sample pellets into ASTM test parts per FP-P-00 Film Processing Practices for required part testing.
    a. Parts A and B are to be evaluated for tensile strength, elongation at yield, notched Izod, melt flow rate
8. Prepare film testing blend C using a 75/25 blend of densified control material and pellet sample B, for an effective innovation concentration of 25%. Produce thick gauge test film samples per FP-P-00 Film Processing Practices for required film testing.
    a. Film samples are to be evaluated for tear strength (MD&TD), tensile strength (MD&TD), elongation at yield (MD&TD), and dart impact.

Path 2A and Path 2B Method Steps

1. Obtain control and test film to use in the evaluation for Path 2A
2. Obtain control resin and test film to use in the evaluation for Path 2B
3. For each of the test and control articles for Path 2A, separately:
    a. Oven densify the articles
    b. Granulate the densified materials
4. Extrude control resin if required per Path 2B, to represent the initial film production heat history.

5. 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
6. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create samples: A pellets, B pellets.
    a. Pellet samples A and B are to be evaluated for DSC, FTIR, melt flow, density, ash content, moisture content, bulk density and color.
7. Injection mold A, B sample pellets into ASTM test parts per FP-P-00 for required parts testing.
    a. Parts A and B are to be evaluated for tensile strength, elongation at yield, notched Izod, melt flow rate
8. Prepare film testing blend C using a 75/25 blend of densified control material and pellet sample B, for an effective innovation concentration of 25%. Produce thick gauge test film samples per FP-P-00 Film Processing Practices for required film testing.
    a. Film samples are to be evaluated for tear strength (MD&TD), tensile strength (MD&TD), elongation at yield (MD&TD), and dart impact.

Path 3 Method Steps

1. Required blends are 100% control film; and a 50/50 blend of control material and test film.
2. Prepare the following required blends:
    a. 100% control film - Sample A
    b. 50/50 control film and innovation film– Sample B
3. Direct feed into extruder without densification or granulation. Extrude blends and melt filter per FP-P-00 Film Processing Practices to create the samples: A pellets, B pellets.
    a. Sample A, B extruded pellets to be evaluated for DSC, melt flow, density, ash content, moisture content, bulk density and color.
4. Injection mold A, B sample pellets into ASTM test parts per FP-P-00 for required parts testing.
    a. Parts A and B are to be evaluated for tensile strength, elongation at yield, notched Izod, melt flow rate
5. Prepare film testing blend C using a 75/25 blend of densified control material and pellet sample B, for an effective innovation concentration of 25%. Produce thick gauge test film samples per FP-P-00 Film Processing Practices for required film testing.
    a. Film samples are to be evaluated for tear strength (MD&TD), tensile strength (MD&TD), elongation at yield (MD&TD), and dart impact.

Flake and Pellet Measurements, Report and Guidance Values

Path 1B or 2B Control 1st Melt History Extruded Pellet

Property	Method	APR Guidance Preferred values	Additional Guidance
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
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 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^3	
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
Pellet Color	ASTM – D6290		Can reveal contamination before later evaluation

ASTM Parts Measurements, Report and Guidance Values

Path 1 and 2 – ASTM Part Evaluation

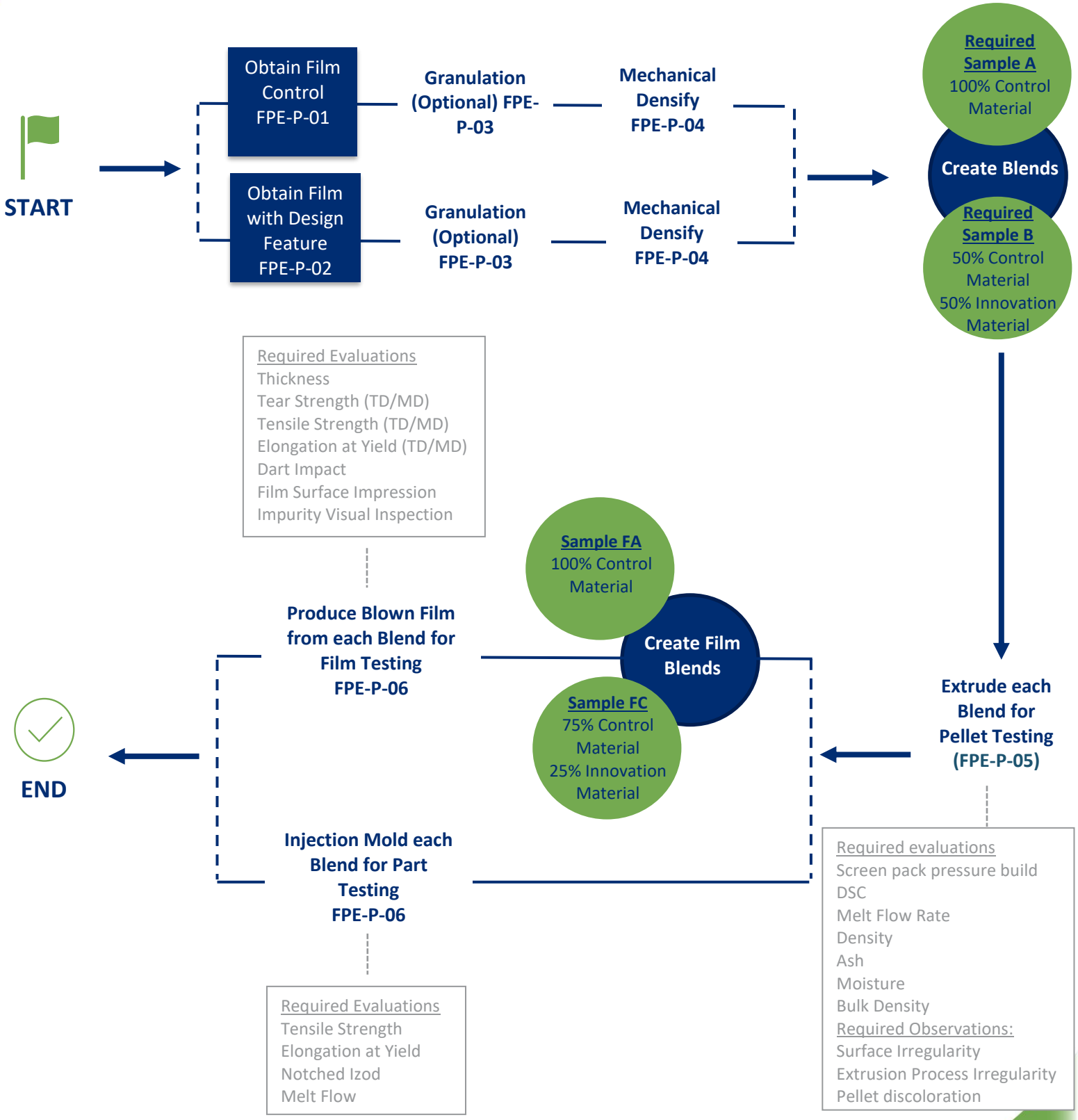
Property	Method	APR Guidance Preferred values	Additional Guidance
Melt Flow Rate	ASTM – D1238	< 0.75 g/10 minutes delta to the control	
Tensile Strength at Yield	ASTM – D638	No more than a 25% Δ decrease from control to test samples.	Record and Report any increase
Elongation at Break	ASTM – D638	No more than a 50% Δ decrease from control to test sample.	Record and Report any increase
Notched Izod	ASTM – D256	Control and test samples should have the same break type or show greater ductility for test sample.	Record and report values for the control and test.
Flexural Modulus	ASTM – D790	No more than a 25% Δ decrease from control to test samples.	Record and Report any increase

Film Measurements, Report and Guidance Values

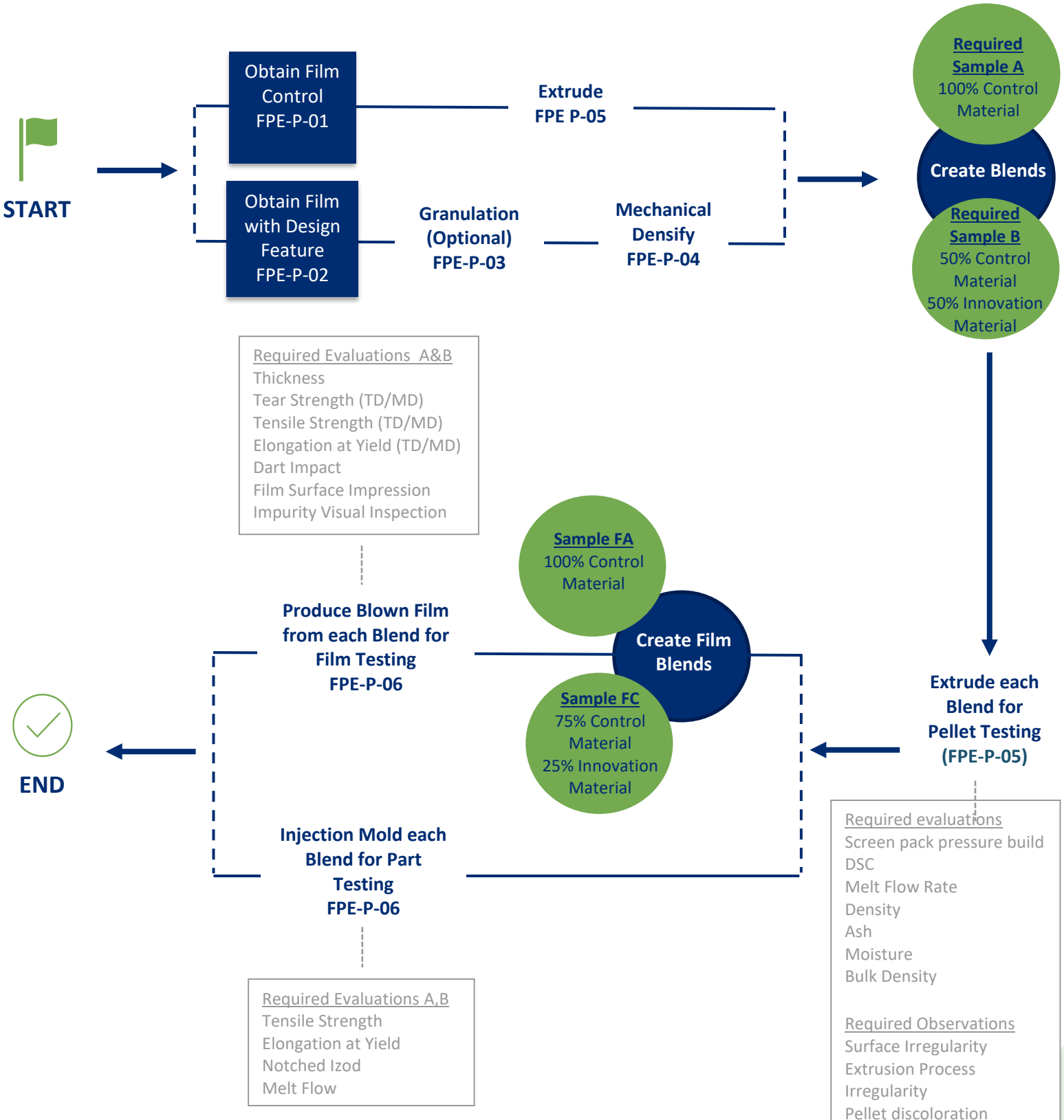
Evaluation of Blown Film Samples All Paths

Property	Method	APR Guidance Preferred values for Samples A and B
Thickness	ASTM – D6988	3.0-4.0 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% Δ drop to the control. Record and report % increase.
Tensile Strength (MD/TD)	ASTM – D882	No more than a 25% Δ drop to the control. Record and report % increase.
Elongation @ Yield (MD/TD)	ASTM – D882	No more than a 25% Δ drop to the control. Record and report % increase.
Dart Impact	ASTM – D1709	No more than a 25% Δ drop to the control. Record and report % increase.

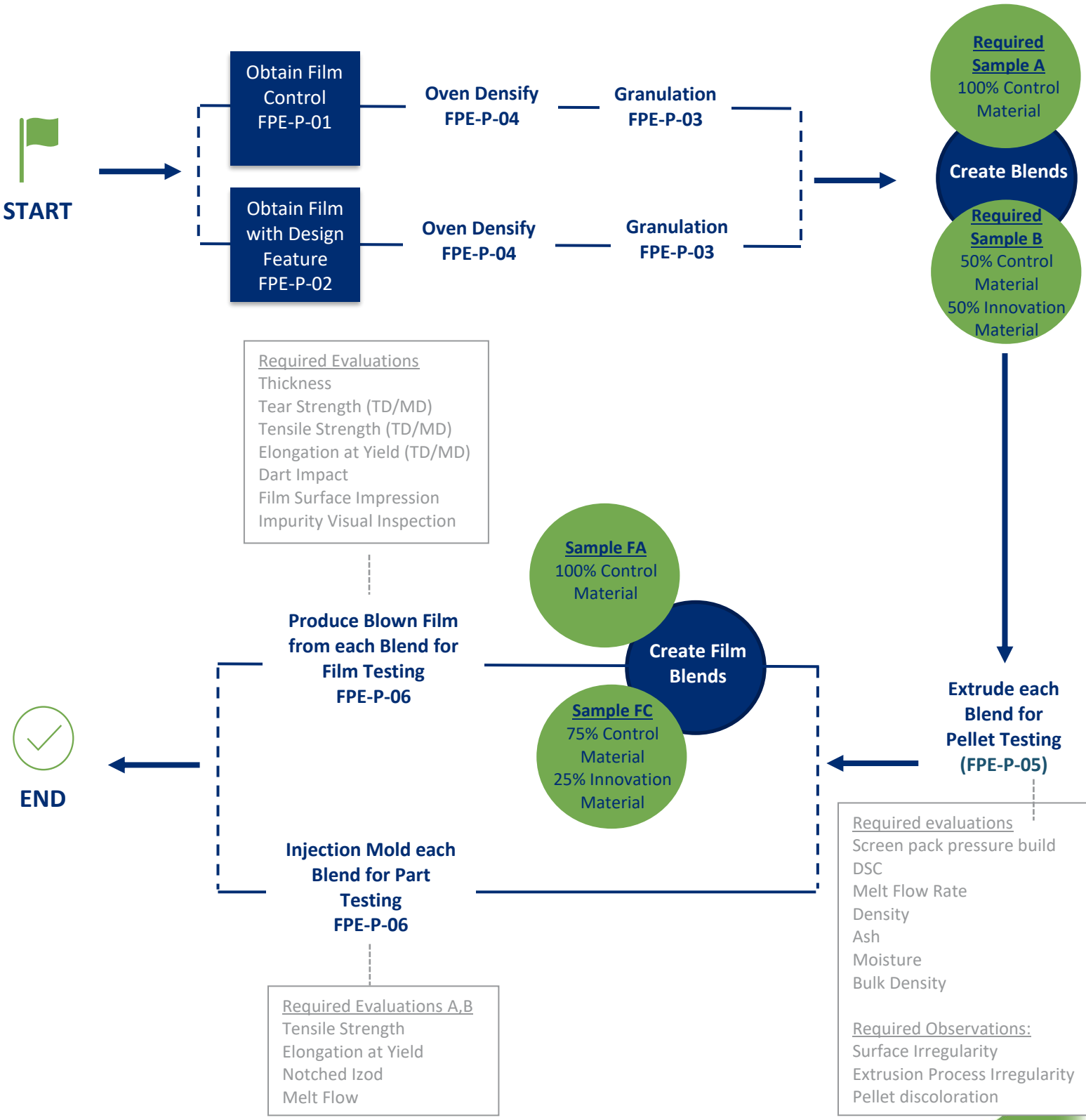
Path 1A: Flow Diagram for Control and Test PE Films (Grind then Densify)



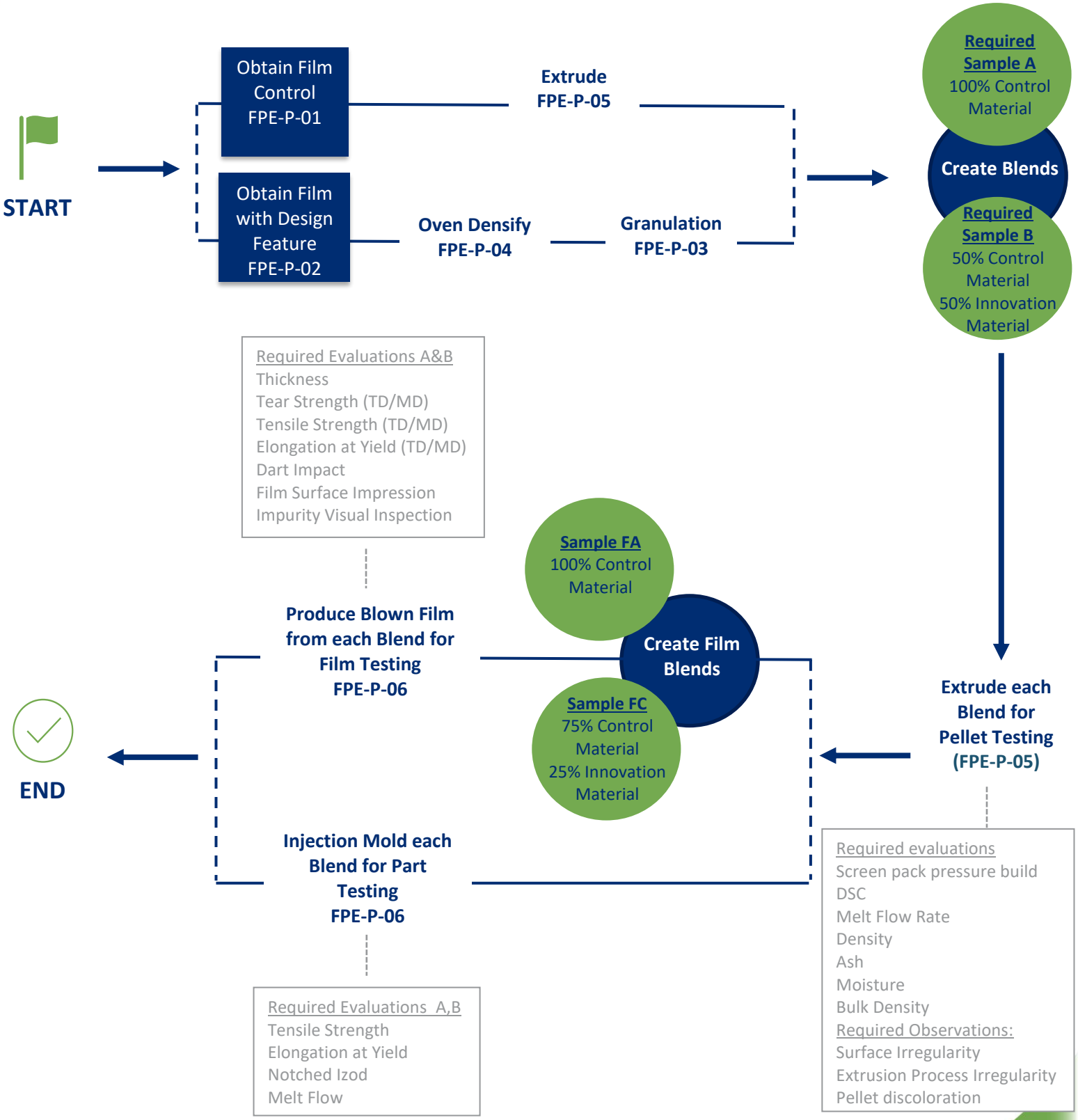
Path 1B: Flow Diagram for Control as Resin and Test as Film (Grind then Densify)



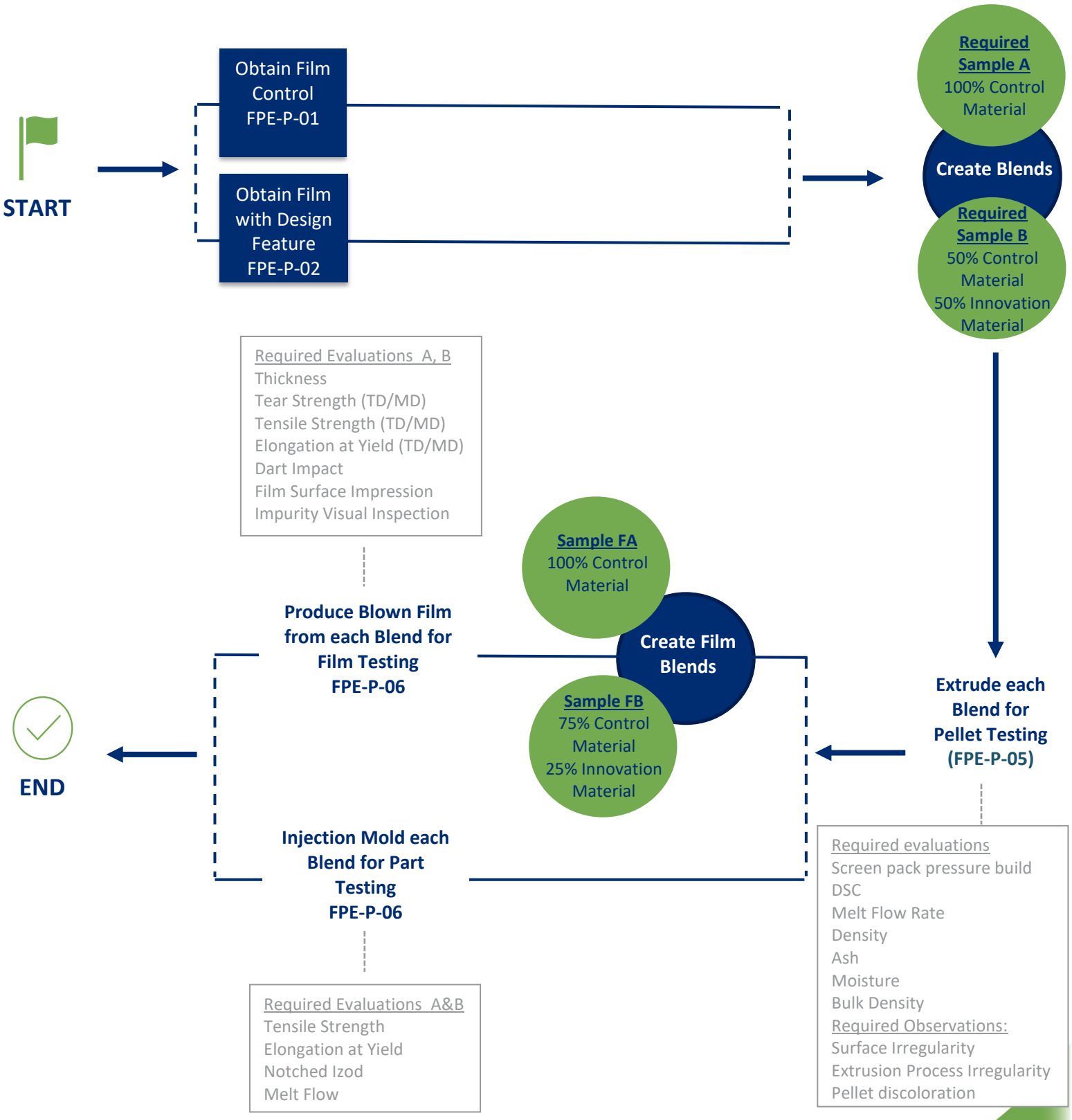
Path 2A: Flow Diagram for Control and Test PE Films (Densify then Grind)



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)



DOCUMENT VERSION HISTORY

Version	Publication Date	Change
1	April 20, 2018	
2	May 17, 2018	
3	October 24, 2018	Added © and Compatibilizer language in Scope on P. 2
4	August, 2020	Eliminated references to virgin resin controls, eliminated washing and drying, eliminated float/sink requirement, modified blend naming to clarify difference in test material concentration for parts testing and film testing; added thick blown film to test, aligned test thresholds with Critical Guidance Protocol, adjusted formatting to be consistent with Critical Guidance Protocol, revised Flow Diagrams to be consistent with Critical Guidance Protocol.
5	September 9 ,2024	Added hyperlinks to testing protocols named within document to match new website