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RECYCLABILITY EVALUATION PROTOCOL FOR PP films

Standard Laboratory Practice

Version 1.0

January 2021

CONTENTS

GLOSSARY

A.0	100% control film flakes
A.25	blend 75/25 control/innovation flakes
A.50	blend 50/50 control/innovation flakes
A.100	100% innovation film flakes
ASTM	American Society for Testing and Materials
B.0	film with 100% control pellets
B.25	film with 87.5/12.5 control/innovation pellets
B.50	film with 75/25 control/innovation pellets
B.100	film with 50/50 control/innovation pellets
EN	European Standard

Innovation: new film, flakes or pellets from innovative film which has to be tested

ISO	International Organization for Standardization
MFI	Melt Flow Index
PE	Polyethylene
PP	Polypropylene
PVC	Poly Vinyl Chloride
TC	Technical Committee
TGA	Thermo Gravimetrical Analysis

"RecyClass is an initiative aiming at enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Plastics Recyclability Evaluation Protocols will promote recyclability by encouraging industry to test new plastic technologies, materials or product before market launch and giving advice and recommendations to the companies. The Recyclability Evaluation Protocols are available for download in the PRE and RecyClass websites. Companies providing plastic packaging concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance to a Recyclability Evaluation Protocol is not a replacement for an official assessment and may not be used as a marketing tool.** All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and must be conducted by an independent laboratory approved by RecyClass which has no legal affiliation to the applicant. More information is reported in the RecyClass Internal Procedures available in the RecyClass website".

1 INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "Recyclability Evaluation Protocol for PP Films" referred to in this document as "The Protocol" describes the methodology that must be followed by the applicant at a laboratory scale in order to determine if a plastic packaging innovation is compatible with the post-consumer PP film recycling stream. The Protocol targets companies responsible for introducing a packaging product into the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the "RecyClass[1] Internal Procedures".

The Protocol analyzes whether an innovation will undergo the necessary pre-treatment, extrusion and conversion steps described in this methodology at a laboratory scale without negatively impacting the recycling process. It aims to guarantee recyclability[2] of plastics packaging while encouraging innovation in the PP film market. The overall goal is to maintain the protection of packaged goods and their marketing display functions without obstructing the proper functioning of the PP film recycling process.

This document provides guidance on the tests methodology that shall be followed, including benchmark recommendations to guide the interpretation of the results.

PP film terminology, as it is used in this document, is defined as a flexible plastic whose form changes depending on whether it is filled with a substance or not. It has a thickness of up to 250 µm and at least 85% of its weight is plastic, with up to 15% of closely bonded or impregnated material. Printing, coatings, or plastic fillers can classify as closely bonded or impregnated materials. It includes blown, cast and biaxially oriented PP films.

2 SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation introduced to the existing packaging solutions for PP films. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for clear PP films or colored PP films[3] in order to confirm that the PP innovation film is compatible with these requirements.

The following packaging solutions and/or innovations are covered by the scope of this Protocol:

1. Non-PP layers and coatings, including PE, nylon, EVOH, and others not specified.
2. Rigid PP and Non-PP attachments to the PP film tested packaging.
3. Mineral fillers and other additives that alter the density of the PP film.

1RecyClass assesses the recyclability of a plastic package providing a ranking from A to F. RecyClass also provides specific indications and recommendations on how to improve packaging design to fit current recycling technologies. More information at https://recyclass.eu/

[2] Recyclability definition according to PRE & APR: Plastics must meet four conditions for a product to be considered recyclable: 1. The product must be made with a plastic that is collected for recycling, has market value and/or is supported by a legislatively mandated program. 2. The product must be sorted and aggregated into defined streams for recycling processes. 3. The product can be processed and reclaimed/recycled with commercial recycling processes. 4. The recycled plastic becomes a raw material that is used in the production of new products.

[3] Design For Recycling Guidelines https://recyclass.eu/recyclass/design-for-recycling-guidelines/

4. Paper and PP labels
5. Inks and pigments, including direct, reverse, laminated, and other printing technologies.
6. Compatibilizers and other additives otherwise not specified.
7. Adhesives in Laminated Mono-PP-structures

Other packaging containing vacuum-deposited metalized layers, metal foil layers, degradable plastics, carbon black coating, as well as PET, PVC and PVDC layers or films shall be separately considered by the RecyClass Technical Committee in order to assess their suitability under the scope of this Protocol.

3 DISCLAIMER

A PP film recycling process is not yet established. The Protocol aims to represent as accurately as possible how the PP recycling process should work at an industrial scale to allow PP films to get recycled in high quality products. RecyClass Technical Committee reserves the right for further testing if necessary, to issue an additional opinion on the recyclability of the tested packaging.

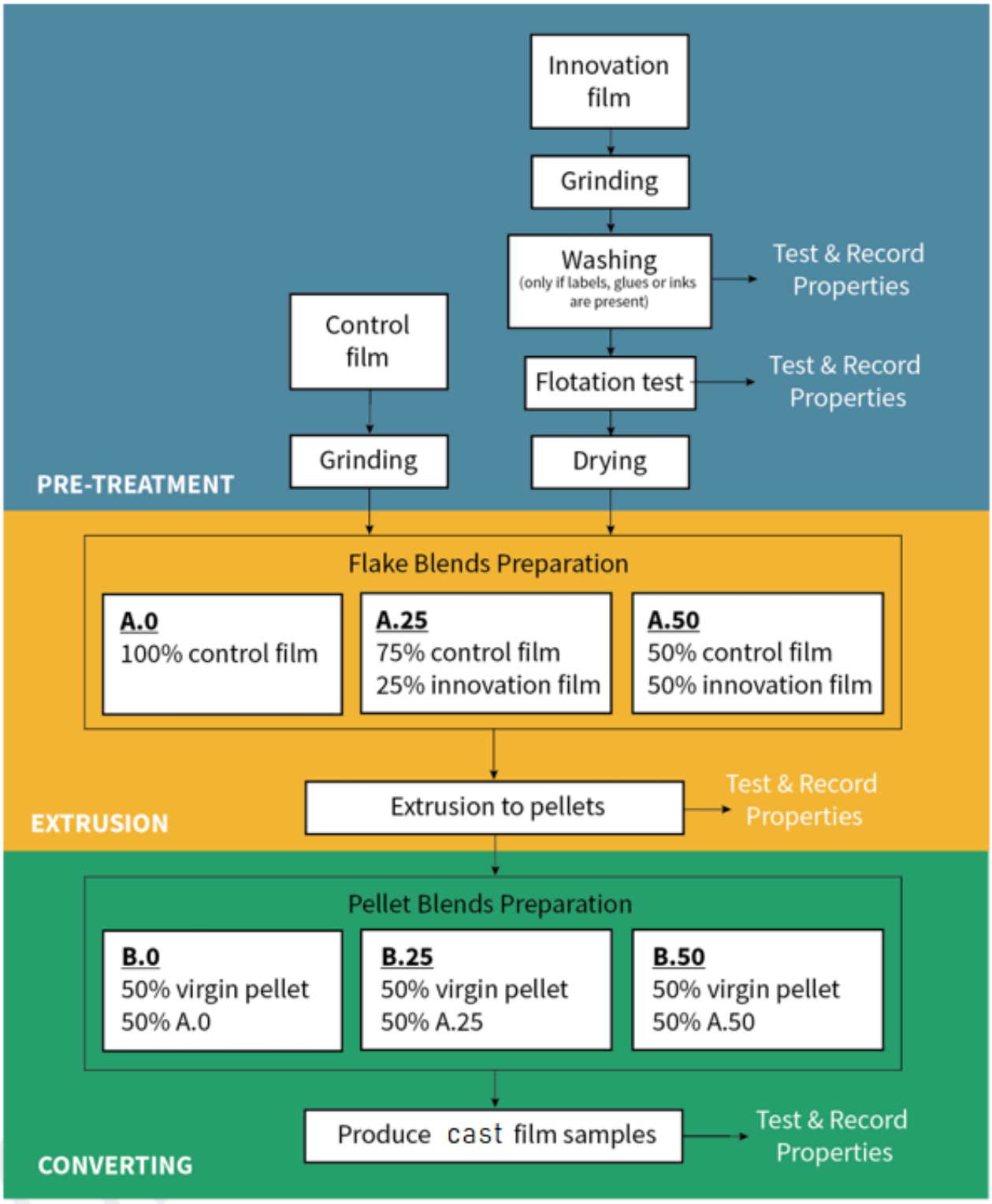
Within RecyClass, "easy-to-empty" and "easy-to-access" indexes are important factors when considering the recyclability of a package. Washing operation at a recycling facility uses mild conditions, no detergents nor strong chemicals. Consequently, any food residue constitutes an impurity for the recycling stream. RecyClass encourages testing to verify that the package is "easy-to-empty" and therefore ensures the minimum amount of leftover material at the end of its useful life. Nonetheless, this factor is beyond the scope of this Protocol.

4 LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the recycling process at a small scale to determine the suitability of an innovation material for the PP film recycling stream. The methodology described below shall be followed precisely and any modifications or problems must be noted during the testing phase. A Lab Evaluation Report compiling all the results obtained shall be prepared to report to the RecyClass Technical Committee which will interpret the final results. Any remarks during following the Protocol shall be also noted down.

See below in Figure 1 a diagram where the flow of the methodology is described.

Figure1: Methodology Diagram



4.1 CONTROL SELECTION

A PP virgin with the typical MFI for cast film applications and copolymers structure as the innovation article, ±10% MFI and ±0.005 density can be used as control for this Protocol, with/upon the approval of the PO films RecyClass Technical Committee (see Annex 1). A mixture of the resins listed in Annex 1 reflecting the innovation structure should be also proposed as control, upon the approval of the Technical Committee.

4.2 PRE-TREATMENT STEPS

4.2.1 GRINDING

Control and innovation film are separately grinded in order to fit the throat of a standard laboratory extruder.

Procedure:

- Grind separately control and innovation sample to flakes of 10 to 20 mm.
- Store in separate containers.

4.2.2 WASHING

Control and innovation film are separately washed to test the impact on wet washing operations. Washing shall only be performed if paper, labels or surface printing is present in the innovation film. If none of those are present, go directly to step 4.2.3.

Procedure:

- Prepare the wash container at a 1:24 ratio (1 g flakes vs 24 ml water) with tap water at a room temperature (+/- 20 – 25ºC). No added detergents or caustic soda.
- Wash each sample separately at a 1:24 ratio (1 g flakes vs 24 ml water) at 1.000 rpm for 10 minutes.
- Rinse each sample at the same ratio with 500 rpm for 5 minutes.

Save the wash and rinse water separately for visual observation. Record the presence of suspended particles or fibers within the water as well as any water coloration. Check and record if the glue has been diluted after the rinsing or it remains attached to film flakes.

4.2.3 FLOTATION TEST

The flotation test will determine if the flakes can be separated by density in the float/sink tank used in the recycling operation.

Procedure:

- Pour the washed flakes in a tank of water filled with water at a 1:24 ratio at a room temperature.
- Stir at 500 rpm for 10 minutes.
- Remove the tank from the magnetic stirrer.

- Collect all particles that float on the surface with a sieve.
- Collect separately the particles that sink.

Record the amount of material that float and the amount that sink in grams and %.

4.2.4 DRYING

Reduce the flake moisture according to the following procedure. A minimum of 2 Kg of material are necessary to proceed with the moisture content determination.

Procedure:

- Heat the oven to 80°C.
- Divide the granulates evenly between the 4 dishes. The dishes are sequentially numbered.
- Weigh the different dishes with the trial material before introducing them in the oven.
- As soon as the over has reached 60°C, the trials are added to the oven for 12 hours.
- Weight the material after 6 hours in the oven. Weight the material at the end, after 12 hours in the oven.
- Record the moisture content.

5 EXTRUSION

5.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of innovation PP films against control as laid out in this Protocol, a set of flake blends is prepared as described in Table 1. Blends shall be produced once the control and innovation films have separately gone through all pre-treatment steps described below.

Keep separated the control and innovation flakes obtained following the previous steps, and air dry for 24 h at ambient air. Then, according to the values reported in Table 2 prepare three different blends with 100% control (and 0% innovation), 75% control - 25% innovation, and 50% control - 50% innovation, and tag them respectively as A.0, A.25 and A.50.

Eventually, depending on the application and its market penetration, the TC can ask the Applicant also to perform the tests with a sample of 100% innovation, e.g. when the estimated market share is higher than 10% (i.e. A.100, by replacing the test with A.25).

For the purpose of the tests the Applicant should provide enough innovation and control materials which allows for the blend preparations. The laboratory carrying out the Protocol testing can define the amounts according to their best knowledge.

5.2 FLAKE BLENDS COMPOSITION

Three different blends 0%, 25% and 50% of innovation film (optionally 100% innovation film), will be prepared as described in table 1.

Eventually, depending on the application and its market penetration, the TC can ask the Applicant also to perform the tests with a sample of 100% innovation (i.e. A.100, by replacing the test with A.25).

Table 1: Flake blends composition for the production of pellets

Blend	Composition	% Control Film	% Innovation Film
A.0	100% Control film	100	0
A.25	75% Control film 25% Innovation film	75	25
A.50	50% Control film 50% Innovation film	50	50
OPTIONAL A.100	100% Innovation film	0	100

5.3 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blends preparation. The flakes will be dried at the same conditions with a desiccant bed drying unit or with hot air and extruded at temperatures of 250 °C. The extrudate will be melt filtered at 110 microns.
Control flake sample A.0 has to be extruded first. Further size reduction before extrusion is acceptable if needed to allow good feeding of the material into the extruder. See additional information in Table 2.

Table 2: Pellet production purpose & overview

Flake Compositions	Kg of blend required	Purpose of blend
A.0 100% Control flake	Per lab requirement for a 30-minute run time	All tests compared to control values
A.25 75% control with 25% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
A.50 50% control with 50% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL A.100 100% innovation	Per lab requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Extrude at a preferred melt temperature from 230–250°C with a suggested filtration screen at 110 µm. If the range is not optimal, record temperature and state reasons for alteration.
- Extrusion run time per variable, no less than 30 minutes.
- Extrusion load > 60%
- Maintain pressure increase to less than 25% from the control over a stable 15 minutes run time.

Record properties' results in table 3.

5.3.1 PELLET PROPERTIES EVALUATION

Table 3: Pellet properties evaluation

Assessment	**Result**	**Standard**	**Benchmark Recommendation**
Bulk Density (kg/m3)		ISO 60 or EN 15345	Minimum 480 kg/m^3
Density (kg/m3)		ISO 1183-1	A.25 and A.50 (and eventually A.100 lower than 0,920 g/cm^3 for natural films and lower than 0,950 g/cm^3 for colored films
Melt Index (g/10 min)		ISO 1133-1 (230 °C/2.16kg)	A.25 and A.50 (and eventually A.100) less than15% deviation in respect to A.0
Ash content (%)		ISO 3451-1 (muffle) or ISO 11358 (TGA)	A.50 lower than 1% (A.100 lower than 2%)
Filtration (110 µm)		Visual inspection	No build-up on screen
Moisture (% weight)		Moisture analyser	< 0.1%
Differential Scanning Calorimetry (°C)		ISO 11357-3	Full thermogram (0 – 240 °C)
Impurities		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (%)		10 g air-dried pellets exposed to 180°C for 10 minutes	±0.1% for A.25 and A.50 (and eventually A.100) respect to A.0

PE (%), PE-Comonomers in PP are not counted		Differential Scanning Calorimetry or optionally Spectroscopic measurement via FTIR microscope in case of doubt	No more than 2,5% for A.50 (and eventually no more than 5% for A.100)
Reflection Colour		(L*, b*, a*)	Record
Delta Pressure (MPa)		Measure it after extruding through 110 microns for the stable 30 minutes run time	No more than 25% higher pressure respect to the control sample
Extrusion process		Unusual sticking, fumes, odor and any build-up	Record

6 CONVERSION

Based on the obtained results, the RecyClass PO films Technical Committee will decide if the innovation presents some critical properties. On that basis, the Technical Committee reserves the right to further test the innovation. Otherwise, if the results are aligned with PP film recyclate specimens the Technical Committee and the Applicant will define the way to further test the innovation on the base of the main applications available on the market.

The Protocol aims to assess the highest-value recyclate application, i.e. cast film production.

For cast films production, three blends of innovation and control pellets will be produced aiming to assess different innovation concentration in the recycling stream, as reported in the following paragraph.

However, in case of failed tests the RecyClass PO films Technical Committee according with the Applicant could decide to test the innovation for injection molding. In this case, the lab will refer to the Recyclability Protocol for PP containers[4].

6.1 PELLET BLENDS COMPOSITION

Once PP pellets have been produced and tested, three additional blends of at 50% virgin – 50% blend A shall be produced for converting tests. Keep separated the pellet samples previously produced and dry it for 10 minutes at 60°C. Then according to the values reported in following Table 4 prepare three different blends with 0% innovation (50% virgin and 50% A.0 pellets), 12.5% innovation (50% virgin and 50% A.25 pellets), and 25% innovation (50% virgin and 50% A.50 pellets), and tag them as samples B.0, B.25 and B.50 respectively.

[4] The Recyclability Evaluation Protocol for PP containers is available on the RecyClass website at: https://recyclass.eu/wp-content/uploads/2020/06/2020-06-16-RecyClass-Recyclability-Evaluation-Protocol-for-PP-containers.pdf

Pellet blends will be composed of 0%, 12.5% and 25% by weight of the innovation PP films.

Eventually, depending on the application and its market penetration, the TC can ask the Applicant also to perform the tests with a sample of 100% innovation (i.e. B.100, by replacing the test with B.25).

Table 4: Pellet blends composition for application tests

Blend	Composition	% Virgin	Effective % Control	Effective % Innovation
B.0	50% Virgin 50% A.0	50	50	0
B.25	50% Virgin 50% A.25	50	37.5	12.5
B.50	50% Virgin 50% A.50	50	25	25
OPTIONAL B.100	50% Virgin 50% A.100	50	0	50

6.2 PELLET BLENDS COMPOSITION

Once new PP pellets have been produced (A.0; A.25; A.50 and eventually A.100) and tested, three additional blends of cast film at 50% virgin pellet – 50% Blend A shall be produced as described in table 5. Blends will be composed of 0%, 12.5% and 25% content (end eventually 50%) of the initial innovation film.

Table 5: Pellet blends composition for the production of cast film

Blend	Composition	% Virgin Resin	Effective % Control Film	Effective % Innovation Film
B.0	50% Virgin Pellet 50% A.0	50	50	0
B.25	50% Virgin Pellet 50% A.25	50	37.5	12.5
B.50	50% Virgin Pellet 50% A.50	50	25	25
OPTIONAL B.100	50% Virgin Pellet 50% A.100	50	0	50

6.3 CAST FILM PRODUCTION

Prepare blends for cast film extrusion as described in table 6. See more information in table 6.

Table 6: Cast film production purpose & overview

Pellet Compositions	Kg of blend required	Purpose of blend
B.0 50% A.0 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	All tests compared to control values
B.25 50% A.25 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
B.50 50% A.50 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL **B.100** 50% Virgin 50% A.100	Per lab requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Produce cast film at a melt temperature of 250 – 270°C, chill roll temperature of 50-60 °C and a thickness of 35 µm.

Report the melt temperature and the chill roll temperature profiles during the 30-min run time.

Record production properties in table 7, including information regarding structure, holes and stability of the cast film.

6.3.1 CAST FILM PROPERTIES EVALUATION

Table 7: Cast film properties evaluation

Assessment	**Results**	**Standards**	**Benchmark Recommendation**
Thickness (µm)		ISO 4593; DIN 53370	50 µm
Tear Strength (TD**) (g)		DIN EN ISO 6383-2	No more than 25% delta to control
Tear Strength (MD***) (g)		DIN EN ISO 6383-2	
Tensile Strength (TD) (MPa)		DIN EN ISO 527-3	
Tensile Strength (MD) (MPa)		DIN EN ISO 527-3	
Elongation at Yield (TD) (%)		DIN EN ISO 527-3	
Elongation at Yield (MD) (%)		DIN EN ISO 527-3	
Dart Impact (g)		ISO 7765-1	
Haze (%)		DIN EN ISO 14782	Record. Increase of haze will lower the visual aspects
Gels and Specks (amount)		5 samples of 100 cm^2 for a gel and specks count greater than 200 µm seen by the naked eye at 30 cm from sample. The number will be recorded but no standard required.	Record. All gels & specs will weaken the film quality
Surface Appearance			Record. Limit the end use application.

**Film testing results are minimum conditions. Historical data over time may require adjustments for specification change and requirements for specific applications.*

***TD: transverse direction*

****MD: machine direction*

ANNEX I – CONTROL SAMPLES SELECTION

Application	Copolymer structures	MFR at 230°C, g/10min	Melting temperature, ° C	Flexural Modulus ISO178 (MPa), measured on IM specimen 23 °C /50%RH
Cast film	Homopolymer	MFR 2=8	162	1400
Cast Film	Random Copolymer	MFR 2=8	150	1000
BOPP	Terpolymer	MFR 2=6	130	n.a.
BOPP	Homopolymer	MFR 2=3.2	161	n.a.

The reference granules are typically for cast PP films 70% Homopolymer and 30% Random-Copolymer and for BOPP films 90% Homopolymer and 10% Terpolymer.