RECYCLABILITY EVALUATION PROTOCOL

FOR LAMINATING
ADHESIVES APPLIED
ON PP FILMS

STANDARD LABORATORY PRACTICE REP-PPlam-01

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CONTENT

CONTENT	2
GLOSSARY	3
1. INTRODUCTION AND PURPOSE OF THE PROTOCOL	5
2. SCOPE OF THE PROTOCOL	6
3. DISCLAIMER	6
4. LABORATORY TEST METHODOLOGY	6
4.1 CONTROL SAMPLE SELECTION	8
4.2 VIRGIN SAMPLE SELECTION	8
5. LABORATORY TEST PROCEDURES	8
5.1 PRE-TREATMENT STEPS	8
5.1.1 GRINDING	8
5.1.2 DRYING.	9
5.2 EXTRUSION	10
5.2.1 FLAKE BLENDS PREPARATION	10
5.2.2 FLAKE BLENDS COMPOSITION	10
5.2.3 PELLET PRODUCTION	11
5.3 CONVERSION	13
5.3.1 PELLET BLENDS PREPARATION	13
5.3.2 PELLET BLENDS COMPOSITION	13
5.3.3 BLOWN FILM PRODUCTION	13
DOCUMENT VERSION HISTORY	15
ANNEX I – CONTROL & VIRGIN SAMPLES SELECTION	16
ANNEX II – MASS BALANCE FOR PRE-TREATMENT STEPS	16
ANNEX III – PE FILM SURFACE IMPRESSION EVALUATION AND IMPURITY VISUAL INSPECTION	17

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 Blend 100 % innovation film flakes

ASTM American Society for Testing and Materials

B.0 Film made of 50 % of virgin pellets and 50 % of B.0 pellets

B.25 Film made of 50 % of virgin pellets and 50 % of B.25 pellets

B.50 Film made of 50 % of virgin pellets and 50 % of B.50 pellets

B.100 Film made of 50 % of virgin pellets and 50 % of B.100 pellets

Control Sample Plain PP film (or PP resin that has already been thermally processed once) used as

benchmark

EN European Standard

Innovation Sample Film containing the innovative laminating adhesive

ISO International Organization for Standardization

MFI Melt Flow Index
PP Polypropylene

TC Technical Committee

Virgin Material PP resin that will for the first time be converted to a plastic product (no thermal pre-

treatment)

wt% Weight Percentage

DISCLAIMER

"RecyClass is a non-profit, cross-industry initiative advancing recyclability, bringing transparency to the origin of plastic waste and establishing a harmonized approach toward recycled plastic calculation & traceability in Europe. The Recyclability Evaluation Protocols will promote recyclability by encouraging the industry to test new plastic technologies, materials or products, providing recommendations on improving their recyclability before market launch.

The Recyclability Evaluation Protocols are freely available to download on the <u>RecyClass website</u>. Companies developing new plastic packaging concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance with a Recyclability Evaluation Protocol is not a replacement for an official assessment and may not be used as a marketing tool.** The RecyClass Steering Board, following the recommendations of the Technical Committees, will decide on the compatibility of the innovation with recycling according to the evaluation results, granting a Recyclability Approval Letter to the Applicant.

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and be conducted by an independent laboratory recognised by RecyClass which has no legal affiliation to the applicant.

More information is reported in the RecyClass Internal Procedures available on the <u>RecyClass</u> <u>website.</u>"

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "RecyClass¹ Recyclability Evaluation Protocol for Laminating Adhesives Applied on PP Films" referred to in this document as "The Protocol" describes the methodology the applicant must follow at a laboratory scale to determine if a laminating adhesive innovation is compatible with the post-consumer PP film recycling stream. The Protocol targets companies responsible for introducing a packaging product or a laminating adhesive into the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures² and RecyClass Recyclability Approval Quality Management & Procedures document³

The Protocol analyses whether a PP laminate will undergo the necessary pre-treatment, extrusion and conversion steps described in this methodology at a laboratory scale without negatively impacting the recycling process and the quality of the recycled PP flexible material. It aims to guarantee recyclability⁴ 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 and ensuring the highest possible quality of the recycled PP film.

This protocol is a simplified version of the Recyclability Evaluation Protocol for PP Films, with a focus on laminating adhesive, and was designed with the support of RecyClass Laminating Adhesive Working Group.

This document provides guidance on the testing 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 μ m and at least 90 % of its weight is plastic, with up to 10 % of closely bonded or impregnated material. Printing, coatings, or plastic fillers can classify as closely bonded or impregnated materials.

Please note that all units in this protocol are expressed following The International System of Units⁵, from the Bureau International des Poids et Mesures.

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

² RecyClass Internal Procedures

³ RecyClass Technology & Product Approval Quality Management & Procedures

⁴ 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.

⁵ SI Brochure - BIPM

2. SCOPE OF THE PROTOCOL

The scope of the Protocol covers any laminating adhesive introduced to the existing packaging solutions for PP films. Before initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for clear or coloured PP films¹ to confirm that the PP innovation film is compatible with the recycling stream for PP films.

This Protocol only covers the recyclability evaluation of PP laminates with laminating adhesive considered as the Innovation. The addition of any other packaging component such as inks, barrier layer or coating will lead to a recyclability assessment that must be performed according to the general Recyclability Evaluation Protocol for PP films.

3. DISCLAIMER

The Protocol is created to represent as accurately as possible how the actual PP recycling process works at an industrial scale. RecyClass Technical Committee reserves the right for further testing, if necessary, to issue a final opinion on the recyclability of the tested innovation. The Recyclability Evaluation Protocol establishes some benchmark recommendations to guide the decision-making process. However, only some of the properties listed in the protocol are provided with a benchmark recommendation, given that the evaluation is also based on the technical expertise of the Technical Committee (TC).

4. LABORATORY TESTING METHODOLOGY

This methodology aims to reproduce the recycling process at a laboratory scale to determine the suitability of an innovation for the PP film recycling stream. The methodology described below shall be followed precisely and any modifications or problems during the testing phase must be noted. Additional tests may be requested by the PO films TC for specific cases requiring particular attention. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared to report to the RecyClass PO films Technical Committee (TC), which will interpret the results. Any remarks during the laboratory tests described in the Protocol shall also be noted down.

See below in Figure 1 a diagram describing the methodology.

¹ Design for Recycling Guidelines

PRE-TREATMENT **CONTROL FILM** INNOVATION FILM GRINDING GRINDING DRYING DRYING TEST & RECORD PROPERTIES **EXTRUSION TO PELLETS** CONVERTING PELLETS BLENDS PREPARATION B.100 (optional) 50% virgin pellet 50% A.50 50% virgin pellet 50% A.100 50% virgin pellet 50% A.0 50% virgin pellet 50% A.25 PRODUCE CAST FILM SAMPLES

Figure 1: Methodology diagram

4.1 CONTROL SAMPLE SELECTION

The control sample for use following the Protocol can be selected by:

- **Option 1:** If there is a PP film known to be recyclable, consisting of the same base PP resin as the Innovation, except/apart from the laminating adhesive being evaluated, it can be selected as the control for this Protocol, with the approval of the RecyClass PO films TC. <u>This option should be the one preferred</u> to limit the number of variables in the test
- **Option 2:** The applicant can select a PP resin listed in the Annex (or another grade with similar MFI and density from alternative suppliers) to be used as a control for this Protocol, with/upon the approval of the PO films RecyClass Technical Committee. To obtain the control sample, the selected PP resin must be extruded once, following the recommendations for extrusion present in this protocol, in order to simulate the same thermal history that an actual packaging may have. The same physical form as the innovative material should be preferred.

These options will be used to make both the control flakes and the blends with innovation flakes that will contain the innovative feature(s) (barrier, adhesive, additive, coating, label, multilayer resin, etc.) for the recyclability study.

Since control material is used as reference to evaluate the impact of the innovation, A.0 and B.0 (see Figure 1) need to fulfil some minimum requirements to make the recyclability evaluation valid. RecyClass recognised testing facilities are aware of these minimum requirements and will inform both the Applicant and RecyClass in case of deviations.

For the purpose of the tests the amount of material that the Applicant should provide will depend upon the equipment and scale used in each laboratory. Usually, at least 10 kg of innovation material (as packaging) and 25 kg of control material (as packaging) will be requested to prepare blends of at least 5 kg each.

4.2 VIRGIN SAMPLE SELECTION

The virgin PE sample to be used in this Protocol can be selected from the PP resins listed in Annex I or proposed by the applicant to match the target application. The choice of the virgin must be approved by the PO films TC and used as it is (i.e. without applying any thermal pre-treatment).

5. LABORATORY TEST PROCEDURES

5.1 PRE-TREATMENT STEPS

5.1.1 GRINDING

Control (if provided as film) and innovation samples are separately ground in order to fit the feeding hopper of a standard laboratory extruder. In case the control is submitted under the shape of pellets, only the innovation sample has to be ground.

Procedure:

- Report the mass of each sample before grinding as m₀.
- Grind separately control and innovation sample to flakes of 3 to 15 mm.

- Store in separate containers.
- Report the mass of each sample after grinding as m₁.

5.1.2 DRYING

Reduce the flake moisture at 60°C without application of vacuum to release surface moisture to less than 1%.

Procedure:

- Heat the oven to 60 °C.
- Divide the flakes evenly between at least 4 dishes. The dishes are sequentially numbered.
- Weigh the different dishes with the control or innovation flakes before introducing them in the oven.
- As soon as the oven has reached 60 $^{\circ}$ C, the flakes are added to the oven without the application of vacuum until 1% moisture content is reached.
- Report the mass of each sample after drying as m₄.
- Record the moisture content.

According to the mass measured at the different steps of the pre-treatment, fill the table in Annex 2 and determine the pre-treatment yield for both control and innovation as following:

$$\eta_{PT} = \frac{m_4}{m_0}$$

Where:

η: Pre-treatment yield

m₀: mass of sample before grinding

m₄: mass of sample after drying

5.2 EXTRUSION

5.2.1 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 above.

Keep separated the control and innovation flakes obtained following the previous steps, and air dry for 24 hours in an ambient environment. Then, according to the values reported in Table 1 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.O, A.25 and A.50.

For the purpose of the tests, the Applicant should provide enough innovation and control materials which allows for the blend preparations (cf. section 4.1.).

5.2.2 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.

Depending on the application, 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

5.2.3 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blends preparation. If extrusion is not carried out directly after the previous drying stage, the flakes need to be dried under the same conditions with hot air. The level of moisture must be below 1 %. The flakes are extruded using a co-rotative twin-screw extruder at a melt temperature of 230 °C. The extrudate will be filtered with a 110 μ m filter. When needed, for low bulk density materials, a densification step can be used prior extrusion to effectively feed the extruder, under the agreement of the RecyClass PO Films Technical Committee. Densification should be done following the procedure FPE-P-04 developed by APR¹.

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. Nevertheless, the flake size should be kept constant between all samples.

The extruder has to be cleaned before starting the extrusion process. This involves pulling the screws out of the barrel and then mechanically cleaning them with brass brushes until they reach a glossy finish. The barrel has to be also mechanically cleaned with round brass brushes from the mandrel to the run-out zone.

Procedure:

- If a densification step was performed, report the equipment used, as well as condition and procedures followed.
- Dry samples A.0, A.25 and A.50 (optionally A.100) with hot air at 90 °C for 1 hour maximum to decrease surface moisture below 1%. Any agglomeration of flakes must be reported.
- Extrude at a melt temperature of 230 \pm 5 °C with a suggested filtration screen of 110 μm . If the range is not optimal, record temperature and state reasons for alteration. Melt residence time should be less than 6 minutes.
- Recommended throughput is between 5 and 10 kg/h and rotation speed between 100 and 200 rpm.
- Extrusion run time per variable, no less than 30 minutes.
- Extrusion load > 50%
- Record a 10-second video of the extrusion for each blend, to allow observations of fumes, or volatiles. The video should be centred on the dye.
- Torque and pressure over time must be monitored and reported. If continuous monitoring is not possible, 5 data points should be measured within the 30 min extrusion.
- Verify that the average pressure is less than 25 % superior to the control over a stable 15 minutes run time.

Record the resulting observations during extrusion in Table 2, and pellet properties in Table 3. The processing conditions used for all the samples must be identical. If some operating conditions need to be modified for A.25 and A.50 (optionally A.100) samples, this information must be documented in the report. A small amount of each sample (50 g) will be retained for the RecyClass PO films TC and the Applicant. The extruded pellets will be tested for pellet properties evaluation (Table 2). The pellets of the test samples will be compared with the pellets of the control sample. All pellets should meet the requirements reported in Table 2.

5.2.3.1 EXTRUSION PROCESS OBSERVATIONS

¹ APR PE Film Practices FPE-P-00

Table 2: Extrusion process observations & monitoring

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Odours	 0: No noticeable odours, even right next to the extruder. 1: Slight odour near the extruder, noticeable but not a problem for operators. 2: Strong odour in the work area, sometimes needing ventilation but still manageable. 3: Very strong odour making it uncomfortable, forcing operators to move away from the extruder or use protective equipment 	0 or 1 If odours present, report more details Report with pictures in comparison with A.0
Fumes	 0: No visible fumes observed near the extruder. 1: Slight fumes observed, disappearing quickly. 2: Moderate fumes, clearly visible and staying in the air for some time. 3: Heavy fumes, very visible and dense, making it hard for operators to stay near the extruder. 	0 or 1 If 2 or 3, report with pictures Report with pictures in comparison with A.0
Filtration (110 μm)	Visual inspection. In case of presence of build-ups, an FTIR analysis is recommended to identify the origin of the deposit.	No build-up on the screen
Average Pressure (MPa)	Average pressure after extruding through 110 µm filter for the stable 15 minutes run time, compared to 100 % control	No more than a 25 % increase to A.0
Pressure Variation (MPa)	$\left(\Delta P_{2530\text{minutes}}\Delta P_{5\text{ first minutes}}\right)$	No increase higher than 25 % compared to start

5.2.3.2 PELLET PROPERTIES CHARACTERISATION

Table3: Pellet properties characterisation

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Impurities (unmolten particles)	Visual inspection	Record
Surface appearance	Visual inspection	Record
Reflection Colour	(L*, a*, b*) + ΔE Reflectance mode, D65, 8-10°	For full compatibility: $\Delta b^* < 5$ For limited compatibility: $\Delta b^* < 8$

5.3 CONVERSION

Prior to the recyclability assessment, the RecyClass PO Films Technical Committee will decide the process to be used for conversion according to the highest value recyclate application for the innovation. In the present case, all innovative PP Films recyclate will be converted via cast film extrusion. 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.

5.3.1 PELLET BLENDS PREPARATION

Once PP pellets have been produced and tested, three additional blends of 50 % virgin – 50 % blend "A.X" (X being 0, 25, 50 or 100) shall be produced for converting tests. Keep separated the pellet samples previously produced and dry them 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.

Depending on the application and its market penetration, the TC can ask the Applicant also to perform the tests with a sample of 50 % virgin and 50 % innovation (i.e. B.100, by replacing the test with B.25).

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

5.3.2 PELLET BLENDS COMPOSITION

Three different blends of cast film at 50 % virgin pellet – 50 % Blend A shall be produced as described in Table 4. Blends will be composed of 0 %, 12.5 % and 25 % content (eventually ending at 50 %) based on the weight of the initial innovation film.

Table 4: Pellet blends	composition to	r the application tests

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

5.3.3 CAST FILM PRODUCTION

Procedure:

- Report the equipment and the die used.

- Produce cast film at a melt temperature of 230 250 °C, chill roll temperature of 50-60 °C and a thickness of $35\,\mu m$
- Report the melt temperature and the chill roll temperature profiles during the 30-min run time.
- Record production properties in Table 5, including information regarding structure, holes, and stability of the cast film.
- For "Gels & Specks" evaluation, refer to the Annex 3 "PP Film Surface Impression Evaluation and Impurity Visual Inspection". If gels are large enough, an IR analysis is requested.
- Report the morphology of the gels by performing microscopy or any other optical measurement allowing it.

5.3.3.1 CAST FILM PROPERTIES CHARACTERISATION

Table 5: Cast film properties characterisation.

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATIONS	
Thickness (μm)	ISO 4593; DIN 53370	35 ± 5 μm	
Haze (%)	DIN EN ISO 14782	Record. Increase of haze will lower the visual aspects.	
Gels and Specks (Annex 3)	5 samples of 100 cm² 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 the amount and aspect of each gels, specks, fisheyes and holes separately. Add microscope pictures of them to show size, shape, distribution.	Agels compared to B.0: <25 for full compatibility <50 for limited compatibility
Surface Appearance		Record. Limit the end	use application.

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	March 2025	Recyclability Evaluation Protocol for Laminating Adhesives applied On PP Films release

ANNEX I - CONTROL & VIRGIN SAMPLES SELECTION

Application	Copolymer structures	Grade	MFR at 230 °C, g/10 min	Melting temperature, °C	Flexural Modulus ISO178 (MPa), measured on IM specimen 23 °C / 50 %RH
Cast film	Homopolymer	HD204CF	8	164	1350
Cast film	Homopolymer	HD601CF	8	164	1400
Cast film	Heterophasic copolymer	BC918CF	3	168	1400
Cast Film	Random Copolymer	RD204CF	8	150	1100
ВОРР	Terpolymer	TD310BF	6	130	n.a.
ВОРР	Homopolymer	HC101BF	3.2	161	1350

For control material, a mono-material (mono-grade) approach remains mandatory, selecting a grade with properties as close as possible from the innovation. In the case the control material selected has an MFI out of 3 to 8 g/10min, the virgin material should be selected as homopolymer: copolymer blend that can lead to an MFI for B.25 and B.50 blends in the range of 3 to 8 g/10min (230°C, 2.16 kg).

ANNEX II – MASS BALANCE FOR PRE-TREATMENT STEPS

MASS (g)	CONTROL SAMPLE	INNOVATION SAMPLE
Before grinding: m ₀		
After grinding: m ₁		
After drying: m₄		
Pre-treatment yield: η РТ		

ANNEX III – PP FILM SURFACE IMPRESSION EVALUATION AND IMPURITY VISUAL INSPECTION

PP Film Surface Impression Evaluation and Impurity Visual Inspection

Defect description	Rating			
Texture	Very Smooth	Smooth	Rough	Very rough
Gels larger than 0.2 mm	: Defined as visually uniform small clumps of crosslinked polymers			
Carbon black particles/Specks	: Defined as very small contaminants, possibly darker in colour			
Fisheyes	: Defined as small round or oblong shapes with darker/more concentrated centre			
Holes	: Defined as tears in the cast film starting at, or caused by, gels, specks or fisheyes			

Preferred range	Limited	Non-acceptable
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For evaluation procedures, RecyClass recommends following the Appendix A "Evaluation of Blown Film Appearance Rating Calculation Totals for Control and Test" present in the APR PE Film critical guidance FPE-CG- 01^1

Defect description	B.0	B.25	B.50	B.100
Texture				
Gels larger than 0.2 mm				
Carbon black particles/specks				
Fisheyes				
Holes				

Report here the exact number of defects observed, as well as standard deviations.

¹ APR PE Film critical guidance FPE-CG-01

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