# RECYCLABILITY EVALUATION PROTOCOL

FOR PP FILLED PLASTIC COMPONENTS

STANDARD LABORATORY PRACTICE

**REP-PP Filled-A&EEE-01** 

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### GLOSSARY

A.0	100 % control flakes
A.25	Blend 75/25 control/innovation flakes
A.50	Blend 50/50 control/innovation flakes
A.100	Blend 100 % innovation flakes
ASTM	American Society for Testing and Materials
B.0	Plate made of 100 % A.0 pellets
<b>B.25</b>	Plate made of A.25 pellets
<b>B.</b> 50	Plate made of A.50 pellets
B.100	Plate made of A.100 pellets
C.0	Sheet made of A.0 pellets
C.25	Sheet made of A.25 pellets
C.50	Sheet made of A.50 pellets
C.100	Sheet made of A.100 pellets
Control Sample	PP Filled component (or PP Filled resin that has already been thermally processed once) used as a benchmark
EEE	Electrical and Electronic Equipment
ELV	End-of-Life Vehicles
EN	European Standard
Innovation Sample	Plastic components containing the innovative technology
ISO	International Organization for Standardization
MFI	Melt Flow Index
РР	Polypropylene
тс	Technical Committee
TGA	Thermogravimetric Analysis
Virgin Material	PP-filled resin that will for the first time be converted to a plastic product (no thermal pre- treatment)
WEEE	Waste from Electrical and Electronic Equipment
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 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 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 *RecyClass website*.

### 1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "RecyClass<sup>1</sup> Recyclability Evaluation Protocol for automotive and Electrical and Electronic Equipment (EEE) single PP Filled components referred to in this document as "The Protocol" describes the methodology that the applicant must follow at a laboratory scale to determine if an innovation PP Filled component is compatible with the state of the art of the automotive and EEE recycling stream. The Protocol targets companies responsible for introducing PP-filled components into the automotive and EEE markets. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures<sup>2</sup> and RecyClass Recyclability Approval Quality Management & Procedures document<sup>3</sup>.

The Protocol analyses 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 and the quality of the recycled PP filled material. It aims to prove the recyclability<sup>4</sup> of automotive and EEE plastic components while encouraging innovation in the plastic market. The overall goal is to maintain the functionality of the PP-filled plastic components without obstructing the proper functioning of the recycling process and ensuring the highest possible quality of the recycled PP with filler.

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

PP Filled terminology as it is used in this document, refers to plastic components predominantly used for automotive and EEE applications.

Please note that all units in this protocol are expressed following The International System of Units<sup>5</sup>, from the Bureau International des Poids et Mesures.

<sup>5</sup> SI Brochure - BIPM

<sup>1</sup> RecyClass assesses the recyclability of a plastic product. and provides specific indications and recommendations on how to improve the design of a plastic product to fit current recycling technologies. More information at <a href="https://recyclass.eu/">https://recyclass.eu/</a>

<sup>&</sup>lt;sup>2</sup> <u>RecyClass Internal Procedures</u>

<sup>&</sup>lt;sup>3</sup> <u>RecyClass Recyclability Approval Quality Management & Procedures</u>

<sup>&</sup>lt;sup>4</sup> Recyclability definition according to PRE: 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.

### 2. SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation introduced to the PP Filled plastics used in vehicles or EEE

The following non-exhaustive list of plastic solutions and innovations is covered by the Protocol:

- 1. PP Filled resin with a density between 1.01 and 1.09 g/cm<sup>3</sup>
- 2. Fillers
- 3. Additives
- 4. Coatings
- 5. Superficial Treatments
- 6. Polymer blends
- 7. Flame retardants
- 8. Adhesives
- 9. Pigments

PP-filled components containing degradable plastics, restricted hazardous substances, substances of very high concern (SVHCs), REACH above the threshold limits allowed by the legislation should not be covered by this Protocol. Consequently, PP-filled components containing any of these features do not fall under the scope of this Protocol.

### 3. DISCLAIMER

The Protocol is created to represent as accurately as possible how the current recycling process of technical PP-filled components from automotive and EEE works at an industrial scale. The RecyClass Automotive & EEE Technical Committee (TC) reserves the right for further testing, if necessary, to issue a final decision on the recyclability of the tested plastic.

### 4. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the recycling process at a laboratory scale to determine the suitability of an PP filled component for the automotive and EEE recycling stream. The methodology described below shall be followed precisely and any modifications or problems during the testing phase must be noted. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared to report to the Automotive & EEE 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.





#### 4.1 CONTROL SAMPLE SELECTION

The control PP-filled sample for the purpose of performing the Protocol can be selected:

- **Option 1:** If there is a PP-filled component known to be recyclable based on existing technology, consisting of the same base PP-filled materials as the Innovation, except the specific ingredient/feature being evaluated, it can be selected as the control for this Protocol, with the approval of the RecyClass Automotive & EEE TC.
- Option 2: The Applicant can select a PP-filled resin with the same critical technical specifications for MFI and density as the innovation article, ± 10 % and ± 0.005 density can be used as the control for this Protocol, upon the approval of RecyClass Automotive & EEE TC. A selection of control samples to be used is reported in Annex I. The selected material must be extruded at 230 °C to obtain the control sample. This step is necessary to realistically represent a material already used as a plastic component in the automotive or EEE sector.

A blend of different resin grades of the same polymer typology (e.g. two different ABS grades) can be used as a control material, provided these grades are also used in the innovative component. The request needs to be reviewed by the Technical Committee representatives.

These options will be used to make both the control flakes (also called "shreds," hereinafter referred to as "flakes") and the blends with innovation flakes that will contain the innovative feature(s) (filler, additive, coating, adhesive, polymer blends etc.) for the recyclability assessment.

Since control material is used as a reference to evaluate the impact of the innovation, the control needs 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 15 kg of innovation material (as a moulded component) and 25 kg of control material will be requested to prepare blends of 10 kg. More innovation material could be requested if optional tests are required by the RecyClass Automotive & EEE TC.

### 5. LABORATORY TESTING PROCEDURES

#### 5.1 PRE-TREATMENT STEPS

#### 5.1.1 GRINDING

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

#### Procedure:

- Report the mass of each sample before grinding as  $m_0$  (Annex 2).
- Grind separately control and innovation samples with a screen containing holes within the range of 10 to 15 mm.
- Store in separate containers.
- Report the mass of each sample after grinding as m<sub>1</sub> and average flake sizes.

#### 5.1.2 FLOTATION TEST

The flotation process assesses the density of the plastic component and ensures that the plastic will be correctly separated in an industrial recycling line.

For the assessment of innovation samples, the appropriate procedure must be utilized based on the characteristics of the component:

- Procedure 1: This procedure is suitable if the density of the innovation component is well-known and below 1.08 g/cm<sup>3</sup>.
- Procedure 2: This procedure should be applied in case the density of the component is close to 1.09 g/cm<sup>3</sup>
  +/- 0,01 g/cm<sup>3</sup>, or if any separable materials (e.g., labels, coatings) are present and require removal. A larger amount of innovation component should be assessed to confirm that materials are effectively separated during the process.

#### Procedure 1:

- Fill a 2 l graduated beaker with 700 ml of tap water (pH between 7 and 8).
- Add 13 % sodium chloride to the water solution (or any other salt) to increase the water density up to 1.09 g/cm<sup>3</sup>.
- Put 100 g of the innovative flakes in the water and stir at 500 rpm for 2 minutes.
- Stop the magnetic stirrer and allow the water to rest for 2 minutes.
- Take a photo of the beaker.
- Remove all particles that float at the surface with a sieve.
- Take photos of the floating and sinking fractions separately.
- Save the water for visual evaluation.

#### Procedure 2:

- Fill a vessel with tap water at a 1:6 ratio (5 kg washed flakes vs 30 l water).
- Add 0.2 wt% of dish detergent.
- Add 13 % sodium chloride to the water solution (or any other salt) to increase the water density up to 1.09 g/cm<sup>3</sup>.
- Put each sample separately in the water and stir at 750 rpm for 2 minutes.
- Stop the stirrer and allow the water to rest for 2 minutes.
- Remove all the materials that float at the surface with a sieve.
- Report the mass of the innovation sample after sink-float separation as m<sub>3</sub>f and m<sub>3</sub>s for floating and sinking fraction respectively.
- Take photos of the floating and sinking fractions separately.
- Save the water for visual evaluation.

Rinse well the flakes after performing the procedure to remove any residues of slats.

#### 5.1.3 DRYING

Reduce the flake moisture with ambient air to release surface moisture to less than 1 %.

#### Procedure:

- Dry the flakes collected after floatation with air at room temperature without applying vacuum or heat sources until 1 % moisture content is reached. If the moisture content cannot be reached under these conditions, mild heat (less than 60 °C) can be used with prior notification and approval from RecyClass.
- Report the mass after drying as m<sub>2</sub>.
- Record the moisture content.

#### 5.1.4 ELECTROSTATIC SEPARATION (OPTIONAL)

The triboelectrostatic separation divides different polymeric fractions based on their different conductivity, ensuring that the plastic will be correctly separated in an industrial recycling line.

Depending on PP filled component under assessment the Automotive & EEE TC may request this additional step to ensure that the plastic flakes will be correctly separated during the recycling process.

#### 5.2 EXTRUSION

#### 5.2.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of the innovation PP filled component 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 have separately gone through all pre-treatment steps described above.

Keep separate the control and innovation flakes obtained following the previous steps. 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.0, A.25 and A.50.

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

#### 5.2.2 FLAKE BLENDS COMPOSITION

Three different blends 0 %, 25 % and 50 % of innovation PP filled components (optionally 100 % innovation), will be prepared as described in Table 1.

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

BLEND	COMPOSITION	% CONTROL	% INNOVATION
A.0	100 % Control	100	0
A.25	75 % Control 25 % Innovation	75	25
A.50	50 % Control 50 % Innovation	50	50
OPTIONAL A.100	100 % Innovation	0	100

Table 1: Flake blends composition for the production of pellets

#### 5.2.3 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blend preparation. If extrusion is not carried out directly after the previous drying stage. The flakes need to be dried under the same conditions and extruded using a co-rotative twin-screw extruder at a melt temperature of 230 °C  $\pm$  10 °C. The extrudate will be melt filtered with about 400 µm filtration. Control flake sample A.0 must be extruded first. Further size reduction before extrusion is

acceptable if necessary 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 also has to be mechanically cleaned with round brass brushes from the mandrel to the run-out zone.

#### Procedure:

- Dry samples A.0, A.25 and A.50 (optionally A.100) in mild heat (less than 60 °C) to decrease surface moisture below 1%. Any agglomeration of flakes must be reported.
- Extrude first sample A.0 (the control blend) at a melt temperature of 230 ± 10 °C and with a 400 μm melt filter pack, for no less than 30 minutes. Melt residence time should be less than 6 minutes.
- Recommended throughput is between 5 and 10 kg/h and rotation speed is between 100 and 200 rpm, depending on the filler amount the condition can be adjusted to get the best operation condition.
- Monitor the extrusion process for heat stability.
- If the process doesn't reach steady state conditions (i.e. pressure and/or temperature increase), extrude for no less than 1 hour.
- Rapidly cool the extrudate in a water bath and fed into a pelletizer.
- The pelletizer speed has to be controlled to get a final pellet with a diameter of 3 mm.
- Monitor pressure build-up during pelletizing and report significant differences.
- Randomly select the pellets to perform all the characterisations reported in Table 2.
- Change the melt filter pack between samples for visual examination.
- Be sure to produce enough pellets for all the tests, including the conversion tests.

Record the resulting properties in Table 2. The processing conditions used for all the samples must be identical. If some operating conditions must 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 TC and the Applicant. The extruded pellets will be tested for pellet properties characterisation (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 PELLET PROPERTIES CHARACTERISATION

Samples preparation and testing conditions of PP pellets for the following characterisations must be done according to ISO 19069-2:2016 (Polypropylene (PP) moulding and extrusion materials —Part 2: Preparation of test specimens and determination of properties).

Table 2: Pellet properties	characterisation
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ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Density (kg/m <sup>3</sup> )	ISO 1183-1	A.25 and A.50 (and eventually A.100 lower than 1.09 g/cm <sup>3</sup>
Melt Index (g/10 min)	ISO 1133-1 (230 °C/2.16 kg)	No more than the following deviations respect to A0 - MFI up to $2 \rightarrow \pm 75\%$ - MFI 2 to $5 \rightarrow \pm 50\%$ - MFI 5 to $15 \rightarrow \pm 30\%$ - MFI 15 to $40 \rightarrow \pm 15\%$ - MFI > 40 $\rightarrow \pm 10\%$
Volatiles (wt%)	Heat 10 g blends (before extrusion) and pellets (after extrusion) exposed to 220 °C for 10 minutes	No increase higher than then 0.5 wt% respect A.0
Ash content (%)	ISO 3451-1 (muffle) up to 750 °C	Record
Filtration (μm)	Visual inspection. In case of the presence of build-ups, an FTIR analysis is recommended to identify the origin of the deposit.	No build-up on the screen
Moisture (wt%)	Moisture analyser	< 0.1 wt%
Melt Temperature (°C)	ISO 11357-3 (Heat-cool-heat cycle at 10 °C/min under N <sub>2</sub> from 25 °C to 240 °C with 1 minute of isotherm between each ramp)	Melt Temperature second heating < 170 °C
Surface appearance	Visual inspection	Record
Average Pressure (MPa)	Measure it after extruding through a 250-400 µm filter ((depending on the amount and type of filler under assessment) for the stable 30 minutes run time, compared to 100 % control	Average Pressure: No more than a 25 % delta increase to A.0
Pressure Variation (MPa)	(P <sub>25-30minutes</sub> - P <sub>5 first minutes</sub> )	No increase higher than 25 % compared to start in 30 minutes
Extrusion process	Unusual sticking, fumes, odour, and any build-up	Record

#### 5.3 CONVERTING

Both the control pellets and those made with the innovation must be tested for injection moulding to evaluate tensile properties, colours, as well as defects.

The Protocol aims to assess the highest value recyclate application. If possible, the converting process should be the same as the production process of each innovation component. However, the RecyClass Automotive & EEE TC could decide to test the innovation for a different application.

The injection moulding step (section 5.3.1) is the mandatory conversion step to characterise the mechanical performances and visual properties.

In the case, that the innovation component was produced by thermoforming, a sheet extrusion conversion step may be requested to evaluate the processability of the innovative blends and assess their potential use in a similar application. The three innovation blends will be assessed for sheet extrusion, as reported in section 5.3.2.

#### 5.3.1 INJECTION MOULDING

Pellets A.0, A.25 and A.50 (optionally A.100) must be tested for injection moulding to evaluate tensile properties, colours, as well as defects.

Control pellets A.0 must be moulded first.

#### Procedure:

- Dry the samples A.0, A.25 and A.50 (optional: A.100) in mild heat (less than 60 °C) to decrease surface moisture below 1 %.
- Mould sample A.0 at 200-250 °C to multipurpose specimens' type 1A according to EN ISO 527-2 and to plates with measures of about 60 x 60 x 2 mm<sup>3</sup>.
- The specimens should be completely filled without any shrinkage, overspray, or inclusions.
- Samples A.25 and A.50 (optionally A.100) must be moulded following the identical operating conditions of the control sample A.0.
- Tag the plates produced with A.0, A.25 and A.50 (optionally A.100) as B.0, B.25 and B.50 (optionally B.100), respectively.
- Small variations in operating conditions could be acceptable but must be documented in the report.
- For each material monitor the injection pressure, the heating zone temperature, mould temperature, closing force, injection time and maximum holding pressure (time)

Record the resulting properties in Table 3. Mechanical data must be analysed on the 1A specimen, while colour, inclusions and surface should be analysed on the plate. If some operating conditions must be modified for A.25 and A.50 (optionally A.100) samples, this information must be documented in the report.

#### 5.3.1.1 INJECTION MOULDED PARTS PROPERTIES CHARACTERISATION

Samples preparation and testing conditions of PP pellets for the following characterisations must be done according to ISO 19069-2:2016 (Polypropylene (PP) moulding and extrusion materials —Part 2: Preparation of test specimens and determination of properties).

#### Table 3: Injection moulded parts properties characterisation

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION	
Heat Deflection Temperature (°C at 1.8 MPa) or Vicat (°C), only applicable for automotive assessment	ISO 75 or ISO 306 VST B50	No more than a 15 % delta decrease compared to A.0	
Flexural Modulus (MPa)	ISO 178		
Tensile Modulus (MPa)	ISO 527		
Tensile Stress at Yield (MPa)	ISO 527		
Elongation at Yield (%)	ISO 527-2	No more than a 20 % delta decrease compared to A.0	
Tensile Stress at Break (MPa)	ISO 527-2	-	
Charpy Impact Strength – Notched (kJ/m²)	ISO 179-1 ISO 179-2 (optional)	-	
Reflection Colour (only to be performed for EEE white goods assessment, under request of the TC)	(L*, a*, b*) Reflectance mode, D65, 8-10°	Record	
Surface appearance/ Inclusion of materials	Visual inspection	Record	

#### 5.3.2 SHEET EXTRUSION (OPTIONAL)

The RecyClass Automotive & EEE TC can optionally decide to test the innovation component for sheet extrusion if it was produced using thermoforming, to demonstrate the possible use of the part in a similar application.

Pellets A.0, A.25, and A.50 (optionally A.100) must undergo sheet extrusion to assess their suitability for thermoforming applications. Control pellet blend A.0 must be extruded first.

#### **Procedure:**

- Dry the samples A.O, A.25 and A.50 (optional: A.100) in mild heat (less than 60 °C) to decrease surface moisture below 1 %.
- Extrude sheets at 230 ± 10 °C with a thickness of 1 mm under conditions determined for the control sample A.0. Melt residence time in the extruder should be no more than 6 minutes.
- Extrusion run time per variable, no less than 30 minutes.
- Pellets A.25 and A.50 (optionally A.100) must be extruded following the identical operating conditions of the control pellets A.0.
- Small variations in operating conditions could be acceptable but must be documented in the report.
- Tag the sheet produced with A.0, A.25 and A.50 (optionally A.100) as C.0, C.25 and C.50 (optionally C.100), respectively.

Record the resulting properties in Table 4. If some operating conditions must be modified for C.25 and C.50 samples, this information must be documented in the report.

#### 5.3.2.1 SHEET PROPERTIES CHARACTERISATION

#### Table 4: Sheet properties characterisation

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATION
Surface Appearance/ Inclusion of materials	Visual inspection	Record

### DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	July 2025	Recyclability Evaluation Protocol for PP-filled Plastic Component Release

### ANNEX I – CONTROL SAMPLES SELECTION

PP FILLED RESINS	FILLER CONTENT	APPLICATIONS	DENSITY, g/cm <sup>3</sup>	MFI, g/10 min at 230 °C 2,16 Kg
Sirmax ISOFIL H 20 TP F HS BK	20 % TD	Automotive	1.04	12

### ANNEX II – MASS BALANCE FOR PRE-TREATMENT STEPS

MASS (g)	CONTROL SAMPLE	INNOVATION SAMPLE
Before grinding: m₀		
After grinding: m1		
After drying: m <sub>2</sub>		

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