RecyClass

# RECYCLABILITY EVALUATION PROTOCOL

## FOR PP CONTAINERS

STANDARD LABORATORY PRACTICE

REP-PP-01

VERSION 6.1.0

PUBLISHED IN JUNE 2020

UPDATED IN FEBRUARY 2026

RecyClass

# CONTENT

RecyClass

# 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** | Bottle made of 50 % of virgin pellets and 50 % of A.0 pellets |
| **B.25** | Bottle made of 50 % of virgin pellets and 50 % of A.25 pellets |
| **B.50** | Bottle made of 50 % of virgin pellets and 50 % of A.50 pellets |
| **B.100** | Bottle made of 50 % of virgin pellets and 50 % of A.100 pellets |
| **C.0** | Sheet made of 50 % of virgin pellets and 50 % of B.0 pellets |
| **C.25** | Sheet made of 50 % of virgin pellets and 50 % of B.25 pellets |
| **C.50** | Sheet made of 50 % of virgin pellets and 50 % of B.50 pellets |
| **C.100** | Sheet made of 50 % of virgin pellets and 50 % of B.100 pellets |
| **Control Sample** | Plain PP container (or PP resin that has already been thermally processed once) used as a benchmark |
| **D.0** | Plaque made of 100 % control pellets |
| **D.25** | Plaque made of 75 % control and 25 % innovation pellets |
| **D.50** | Plaque made of 50 % control and 50 % innovation pellets |
| **D.100** | Plaque made of 100 % innovation pellets |
| **EN** | European Standard |
| **Innovation Sample** | Container containing the innovative technology |
| **ISO** | International Organization for Standardization |
| **MFI** | Melt Flow Index |
| **PE** | Polyethylene |
| **PP** | Polypropylene |
| **PVC** | Polyvinyl Chloride |
| **TC** | Technical Committee |
| **TGA** | Thermogravimetric Analysis |
| **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 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 *RecyClass website*. 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 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[1] Recyclability Evaluation Protocol for PP Containers" referred to in this document as "The Protocol" describes the methodology the applicant must follow at a laboratory scale to determine if a plastic packaging innovation is compatible with post-consumer PP recycling stream. The Protocol targets companies responsible for introducing a packaging product onto the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures[2] and RecyClass Recyclability Approval Quality Management & Procedures document[3].

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 material. It aims to prove the recyclability[4] of plastic packaging while encouraging innovation in the PP 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 recycling process

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

PP terminology, as it is used in this document, refers to rigid plastic containers (bottles, thermoforming, thin wall packaging) predominantly used for packaging liquids, cosmetics, and detergents, as well as food contact applications.

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

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*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* https://recyclass.eu/

[2] *RecyClass Internal Procedures*

[3] *RecyClass Recyclability 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 innovation introduced to the existing packaging solutions for PP. Before initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for natural, white and coloured PP containers[6] to confirm that the PP innovation is compatible with the recycling stream for PP containers.

The scope of this Protocol covers the following non-exhaustive list of packaging solutions and/or innovations:

1. PP resins
2. Barrier and coating materials
3. Mineral fillers and additives
4. Non-PP closure systems and lidding films
5. Non-PP liners, seals, and valves
6. Non-PP labels and sleeves
7. Adhesives
8. Printing and Inks
9. Attachments

Following the RecyClass Recyclability Methodology[7], packaging containing non-removable aluminium, metal, silicone, bio-/oxo-degradable plastics, black carbon surface, as well as PVC and PVDC layers are considered as disqualifying criteria for recyclability. Consequently, packaging containing any of these features does not fall under the scope of this Protocol.

# 3. DISCLAIMER

The Protocol is created to represent as accurately as possible how the current PP recycling process works at an industrial scale. RecyClass PP 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 establish 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).

Within RecyClass, “easy-to-empty” and “easy-to-access” indexes are essential factors when considering the recyclability of a package. At the state-of-the-art, at PP mechanical recycling facilities washing operation typically uses mild conditions and no detergents or strong chemicals. Consequently, any food or product 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 residual material at the end of its useful life. Nonetheless, this factor is beyond the scope of this Protocol.

# 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 recycling stream. The methodology described below shall be followed precisely and any

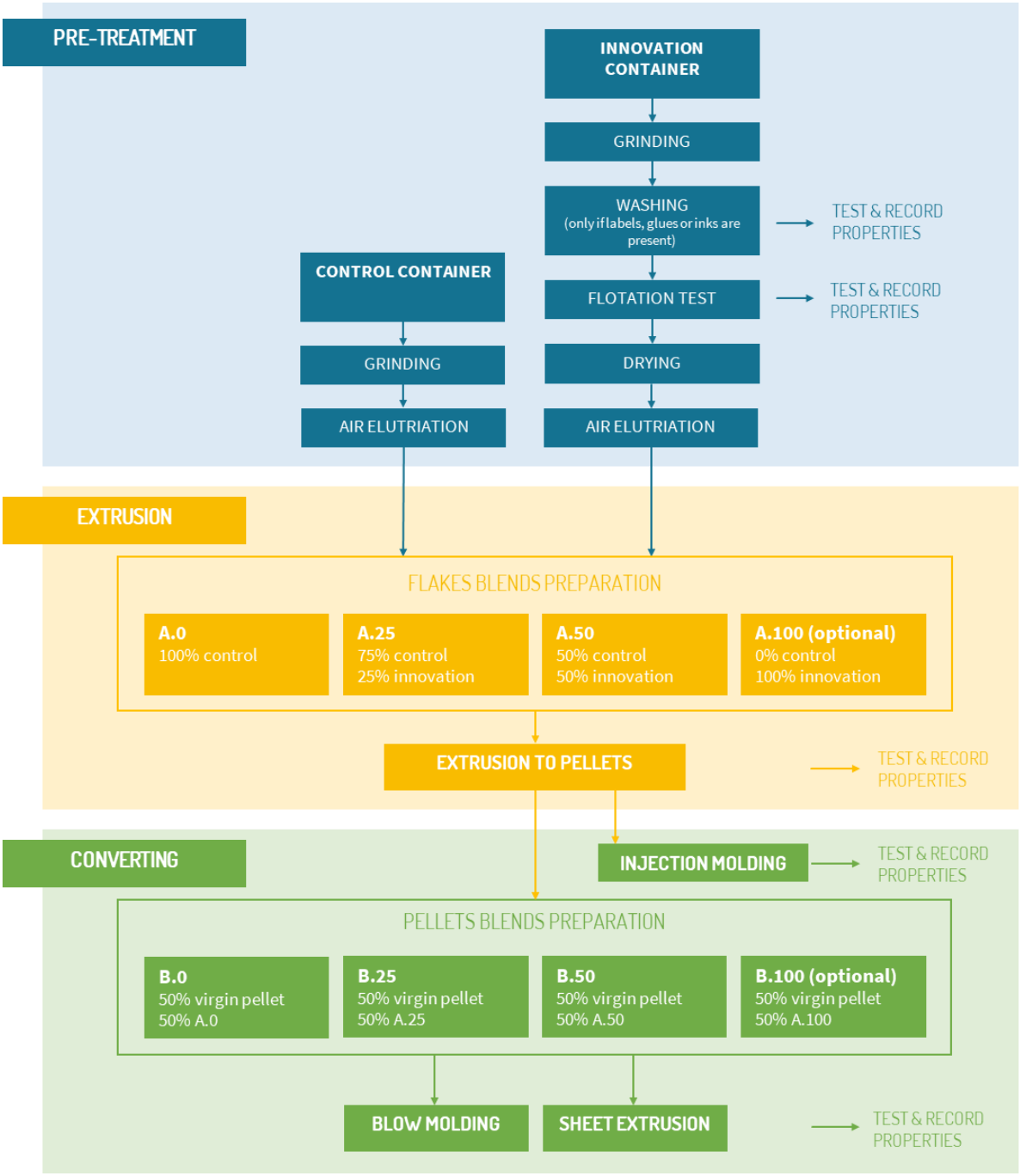
6 Design for Recycling Guidelines

7 RecyClass Methodology

modifications or problems during the testing phase must be noted. The PP TC may request additional tests in specific cases where supplementary data might be needed for a complete evaluation. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared to report to the RecyClass Technical Committee (TC), which will interpret the final 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.

*Figure 1: Methodology Diagram*



## 4.1 CONTROL SAMPLE SELECTION

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

- **Option 1:** If there is a PP container known to be recyclable, consisting of the same base PP virgin materials as the Innovation, except/apart from the specific ingredient/feature being evaluated, it can be selected as the control for this Protocol, with the approval of the RecyClass PP TC.
- **Option 2:** The Applicant can select a PP 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 PP TC. A selection of control samples to be used is reported in Annex I. The selected material must be extruded at 220 °C to obtain the control sample. This step is necessary to realistically represent a material already used as packaging by erasing its previous thermal history.

These options will be used to make both the control flakes and the blends with innovation container flakes that will contain the innovative feature(s) (additive, coating, label, adhesive, 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) 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 packaging) and 25 kg of control material (as packaging) will be requested to prepare blends of 10 kg. More innovation material could be requested if optional tests are required by the RecyClass PP TC. It is worth pointing out that the protocol should be used to test innovations as specific parts of the packaging, meaning that all the decorations or elements of the packaging do not need to be present. The objective is to evaluate the impact of a specific innovation on the recyclability of PP containers. Note that complete packaging (with labels, decoration, closures, etc.) can also be assessed under this protocol.

## 4.2 VIRGIN SAMPLE SELECTION

The virgin PP 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 material must be approved by the PP 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 container) 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. If possible, it is recommended to use a granulator rather than a grinder or shredder.

**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 $m_1$ and average flake sizes.

### 5.1.2 WASHING

State-of-the-art European PP recycling lines typically use mild washing conditions, and no detergents or strong chemicals (Procedure 1). However, in some recycling lines the washing is operated with hot wash and chemicals (Procedure 2) aiming to follow a food contact recycling process. The RecyClass PP TC representatives are requested to select a washing procedure based on the intended end-product. Both procedures take care of labels, adhesives, coatings, paper, and printing present in innovative PP containers. If none of those are present, go directly to step 5.1.3. In the case of non-removable adhesives, procedure 1 should be performed and about 1 kg of innovation flakes should be assessed via procedure 2 to evaluate washability under hot washing conditions.

The following procedures must be used for innovation samples only.

**Procedure 1:**

- Prepare the washing water in a vessel at a 1:4 ratio (10 kg flakes vs 40 l water) with tap water. No added detergents or caustic soda.
- Heat the washing water at 40 °C.
- Wash each sample separately at a 1:4 ratio (10 kg flakes vs 40 l water) at 1.000 rpm for 5 minutes.
- Save a wash sample for visual evaluations.
- Rinse the flakes in the strainer with cold running tap water and stir vigorously for 5 minutes using a manual stirring bar.
- Pour the liquid-flake mix over the de-watering screen and save the wash water.
- Take photos at each step.

Save the washing and rinsing water separately for visual observation. Record the presence of suspended particles or fibres within the water as well as any water colouration. In the case of the presence of adhesives, visually check and record whether the glue has been diluted after the rinsing or whether it remains attached to film flakes (i.e. label flakes). . If water colouration, particles, fibres or remaining glue are observed, washing and bleeding ink Quick Test procedures developed by RecyClass can be used to quantitatively assess these deviations[8].

**Procedure 2 (optional):**

- Prepare the wash solution in a vessel at a 1:4 ratio (5 kg of flakes vs 20 l water + 0.3 % surfactant and 1 % caustic soda (NaOH)). The surfactant must be dissolved in cold water before the addition of caustic soda.
- Heat the solution at 80 °C on a plate covering the vessel to minimize evaporation.
- Overhead stirrer at 1000 rpm, 2.5 cm above the bottom.
- With the stirrer on add PP flakes with their components to the solution (maintain a 1:4 ratio, i.e., 1 part of flake for 4 parts of water).
- Readjust stirrer to 1000 rpm and continue agitation for 5 minutes at 80 °C.
- Turn off and remove the stirrer. Remove the vessel from the heat plate and immediately strain the solution with test components and flakes.
- Rinse the flakes in the strainer with cold running tap water and stir vigorously for 5 minutes using a manual stirring bar. Then drain the material. Save the water for further inspection.
- Spread flakes on a sheet and dry them in an oven at 60 °C to reduce the surface moisture to less than 1 %. Separate flakes and remaining components if required. Washed and unwashed flakes will be compared for visual (and instrumental, if required) evaluations.

Save the wash and rinse water separately for visual observation. Record the presence of suspended particles or fibres within the water as well as any water colouration. Check and record whether the glue has been diluted after the rinsing

[8] RecyClass Quick Test Procedures

or whether it remains attached to film flakes (i.e. label flakes). If water colouration, particles, fibres or remaining glue are observed, washing and bleeding ink Quick Test procedures developed by RecyClass can be used to quantitatively assess these deviations.

### 5.1.3 FLOTATION TEST

Following washing, the flotation test allows flake separation by density, simulating the process of a float/sink tank used in an industrial recycling line. For proper recycling, both density separation efficiency and quality of the floating material should be optimised. Therefore:

- Combinations of polypropylene and other materials that float in water should be avoided to minimise the risk of contamination. In the case less than 100 % of the flakes would float, separation efficiency will be determined based on the innovative packaging composition.
- Non-PP components floating together with PP flakes could pose relevant concerns both in the process operations and in the quality of the recyclate, as they will be extruded along with the PP. If those elements are known to present a risk, they should be avoided. If their effect is unknown, this protocol will be used to evaluated them.

The following procedure must be used for innovation samples only.

**Procedure:**

- Homogenise the flakes collected after washing and save 50g of washed flakes to be used to estimate the efficiency of the density separation.
- Fill a vessel with tap water at a 1:6 ratio (10 kg washed flakes vs 60 l water).
- 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 $m_3f$ and $m_3s$ for floating and sinking fraction respectively.
- Take photos of the floating and sinking fractions separately.
- Save the water for visual evaluation.

The test is passed if 100 % of olefin material is floating. It means non-PP material cannot stick or not get separated from PP and cause PP to sink, resulting in yield losses or stay with PP and contaminate the PP stream.

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  The efficiency of the sink/float separation should be measured using the 50 g of flakes of innovative samples saved before density separation and a graduated beaker filled with tap water, as described by the following procedure.

**Procedure:**

- Fill a 1 l graduated beaker with 700 ml of water (pH between 7 and 8).
- Boil the water for 10 minutes, and then cool at room temperature.
- Transfer 300 ml of water to a graduated beaker.
- Put the innovative sample in the water and stir at 500 rpm for 2 minutes.
- Stop the stirrer and allow the water to rest for 2 minutes.
- Take photos of the beaker.
- Remove all particles that float on the surface with a sieve.
- Take photos of the floating and sinking fractions separately.
- Save the water for visual evaluation.
- Dry the floating fraction for 1 hour at 80 °C in a bed desiccant or 3 hours at 65 °C with air.
- Cool to room temperature, weigh and record the weight of the float fraction.
- Calculate the test efficiency as:

$$\eta = \frac{m_2 f}{m_1}\ x\ 100 = \frac{(m_1 - m_2 s)}{m_1}\ x\ 100\ [\%]$$

Where:

η: Test efficiency

$m_2f$: weight of floating fraction

$m_2s$: weight of sinking fraction

$m_1$: weight of innovative sample

### 5.1.4 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 $m_3$.
- Record the moisture content.

### 5.1.5 AIR ELUTRIATION

Control and innovation PP flakes are separately elutriated with air to remove light fraction.

**Procedure:**

- Elutriate flakes with air with one pass and with less than 2 % loss set for the control flakes.
- Report the mass of the heavy fraction and light fraction of the innovation sample as $m_4h$ and $m_4l$ respectively.
- 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 follows:

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

Where:

η: Pre-treatment yield

$m_0$: mass of sample before grinding

$m_4$: mass of heavy fraction after air elutriation

## 5.2 EXTRUSION

### 5.2.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of innovation PP container 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 separated the control and innovation flakes obtained following the previous steps, and air dry for 24 hours in an ambient e environment. Then, according to the values reported in Table 1 prepare three different dry 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 of 0 %, 25 % and 50 % of innovation PP containers (optionally 100 % innovation)flakes will be prepared as described in Table 1.

Depending on the application and its market penetration, 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).

*Table 1: Flake blends composition for the production of pellets*

| BLEND | COMPOSITION | % CONTROL FLAKES | % INNOVATION FLAKES |
|---|---|---|---|
| **A.0** | 100 % Control | 100 | 0 |
| **A.25** | 75 % Control<br>25 % Innovation | 75 | 25 |
| **A.50** | 50 % Control<br>50 % Innovation | 50 | 50 |
| **OPTIONAL A.100** | 100 % Innovation | 0 | 100 |

The different blends are prepared by manually mixing the flakes in large-capacity containers and following the procedure below:

- Introduce the specified masses of the innovation flakes and control flakes into the mixing container.
- Close the container and agitate for 2 minutes in multiple orientations so as to simulate the motion of a tumble-mixer. Ensure that the container is filled to no more than 60 % of its volume and that the mixing operation yields a suitably homogeneous flake blend.
- If the total quantity of material cannot be processed in a single batch, perform multiple mixing operations with smaller quantities rather than mixing all the material at once.
- Once mixing is complete, verify that homogeneity has been achieved. If uniformity is not confirmed, repeat the mixing cycle until the flake blend is homogeneous.

### 5.2.3 PELLET PRODUCTION

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 flakes are extruded using a co-rotative twin-screw extruder at a melt temperature of 220 °C. The extrudate will be melt filtered (about 120 µm filtration). Control flake sample A.0 has to 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**:

- Any agglomeration of flakes during drying must be reported.
- Extrude first sample A.0 (the control blend) at a melt temperature of 220 ± 5 °C and with a 120 µm melt filter pack for no less than 30 minutes. Melt residence time should be between 1 and 6 minutes.
- Recommended throughput is between 5 and 10 kg/h, and rotation speed between 100 and 200 rpm.
- 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 it into a pelletiser.
- 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.
- Torque and pressure over time must be monitored and reported. If continuous monitoring is not possible, at least 5 data points should be measured within the 30 min extrusion. The starting point must be considered 1 minute after material started to flow out of the extruder.
- 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 observations during extrusion in Table 2, and pellets properties in Table 3. The processing conditions used for all the samples must be identical. 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.

If the innovation samples are expected to render filtration issues, a dedicated filter test can be requested by the RecyClass PP TC.

### 5.2.3.1 FILTRATION TEST (OPTIONAL)

Filter contamination problems may occur when one of the components in the innovation sample is causing gels, larger particles, or releases degraded particles. Pressure drop must be monitored during pelletising since a pressure increase is an indication of the risk of filter contamination. If, based on the previous step, monitoring the pressure and visually inspecting the filter after pelletisation suggest further contamination, a dedicated filtration test should be conducted. To limit the test duration, the innovation sample will not be mixed with control PP.

About 5 kg of pellets from the pure control PP and the pure innovation PP samples will be separately extruded for a minimum of 30 minutes and filtered using a 120 µm screen pack. The 100 % control sample must be extruded first.

**Procedure**:

- Dry the control sample before extrusion with a bed desiccant for 1 hour at 80 °C or with hot air at 90 °C for 1 hour.
- Extrude the sample at a temperature of 220 ± 5 °C and with a 120 µm melt filter pack for no less than 30 minutes.
- If the process doesn't reach steady state conditions (i.e. pressure or temperature increase), extrude for no less than 1 hour.
- If required, small changes in the process parameters are admitted keeping the extrusion stable over the time but must be recorded. However, continuous adjustments of the operating parameters during the runs to overcome steady-state conditions are not admitted.
- Monitor the pressure drop during the test and register variations.

- Repeat the procedure for the innovation sample with the identical operating parameters used for the control sample filtration.

The test is passed if the pressure before the filter does not double from the starting pressure during or at the end of the analysis.

### 5.2.3.2 EXTRUSION PROCESS OBSERVATIONS

*Table 2: Extrusion process observations & monitoring*

| ASSESSMENT | STANDARD | BENCHMARK RECOMMENDATION |
|---|---|---|
| Odours | **0:** No noticeable odours, even right next to the extruder.<br>**1:** Slight odour near the extruder, noticeable but not a problem for operators.<br>**2:** Strong odour in the work area, sometimes needing ventilation, but still manageable.<br>**3:** Very strong odour, making it uncomfortable, forcing operators to move away from the extruder or use protective equipment | 0 or 1<br>If odours present, report more details<br>Report with pictures in comparison with A.0 |
| Fumes | **0:** No visible fumes observed near the extruder.<br>**1:** Slight fumes observed, disappearing quickly.<br>**2:** Moderate fumes, clearly visible and staying in the air for some time.<br>**3:** Heavy fumes, very visible and dense, making it hard for operators to stay near the extruder. | 0 or 1<br>If 2 or 3, report with pictures<br>Report with pictures in comparison with A.0 |
| Die build-ups | **0:** No dye build-ups observed.<br>**1:** Minor dye build-ups observed.<br>**2:** Frequent dye build-ups, clearly visible.<br>**3:** Significant dye build-ups, preventing proper strand formation.<br>Visual inspection. In case of presence of build-ups, an FTIR analysis is recommended to identify the origin of the deposit. | 0 or 1<br>If 2 or 3, report with pictures<br>Report with pictures in comparison with A.0 |
| Average Pressure (MPa) | Average pressure after extruding through 120 µm filter for the stable 15-minute run time, compared to 100 % control | No more than a 25 % increase to A.0 |
| Pressure Variation (MPa) | ($P_{25\text{-}30minutes}$ - $P_{5\ first\ minutes}$) | No increase higher than 25 % compared to start |

### 5.2.3.3 PELLET PROPERTIES CHARACTERISATION

Samples preparation and testing conditions of PP pellets for the following characterisations must be done according to ISO 17855-2:2024 (Polypropylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties).

*Table 3: Pellet properties characterisation*

| **ASSESSMENT** | STANDARD | BENCHMARK RECOMMENDATION |
|---|---|---|
| Density (kg/m$^3$) | ISO 1183-1 | A.25 and A.50 (and eventually A.100 lower than 0.920 g/cm$^3$ for natural containers and lower than 0.950 g/cm$^3$ for coloured containers |
| Melt Index (g/10 min) | ISO 1133-1 (230 °C/2.16 kg) | No more than a 15 % delta to A.0 |
| Volatiles (wt%) before and after extrusion | Heat 10 g blends (before extrusion) and pellets (after extrusion) exposed to 160 °C for 10 minutes | < 1.0 % weight loss |
| Ash content (wt%) | ISO 3451-1 (muffle) up to 750 °C | A.50 lower than 2 wt% |
| 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 or EN ISO 15512 or equivalent | < 0.1 wt% |
| Melt Temperature (°C) | ISO 11357-3 (Heat-cool-heat cycle at 10 °C/min under $N_2$ from 25 °C to 240 °C with 1 minute of isotherm between each ramp) | Melt Temperature second heating < 170 °C |
| Impurities | Visual inspection | Record |
| Surface appearance | Visual inspection | Record |
| PE (%) | Differential Scanning Calorimetry or Spectroscopic measurement via FTIR (method under development) | No more than 2.5 % for A.25 and 5 % for A.50 (and eventually A.100) |

## 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 same as production process of each innovation product, (1) Injection moulding, (2) blow moulding bottle, (3) extrusion sheet. However, the RecyClass PP TC could also decide to test the innovation for a different application.

Injection moulding step (section 5.3.1.) is mandatory to characterise the mechanical performances and visual properties. In case of bottles or sheets production selected for converting step, three blends of innovation and virgin pellets will be produced with the aim of assessing different innovation concentration in the recycling stream, 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) at 90 °C for 2 hours.
- Mould sample A.0 at 190-245 °C to multipurpose specimens' type 1A according to EN ISO 527-2 and to plaques with measures of about 60 x 60 x 2 $mm^3$.
- 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 plaques produced with A.0, A.25 and A.50 (optionally A.100) as D.0, D.25 and D.50 (optionally D.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 plaque. 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.

#### 5.3.1.1 INJECTION MOULDED PARTS PROPERTIES CHARACTERISATION

Samples preparation and testing conditions of PP samples for the following characterisations must be done according to ISO 17855-2:2024 (Polyethylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties).

*Table 4: Injection moulded parts properties characterisation*

| **ASSESSMENT** | STANDARD | BENCHMARK RECOMMENDATION |
|---|---|---|
| Flexural Modulus (MPa) | ISO 178 | No more than 25 % delta decrease compared to A.0 |
| Tensile Modulus (MPa) | ISO 527 | |
| Tensile Stress at Yield (MPa) | ISO 527 | |
| Elongation at Yield (%) | ISO 527-2 | |
| Tensile Stress at Break (MPa) | ISO 527-2 | |
| Elongation at Break (%) | ISO 527-2 | |
| Charpy Impact Strength ($kJ/m^2$) | ISO 179-1<br>ISO 179-2 (optional) | |

| | | |
|---|---|---|
| Reflection Colour | (L*, a*, b*) and ΔE<br>Reflectance mode, D65, 8-10° | For natural stream:<br>60<**L***, -3<**a***<0, -5<**b***<5<br>**ΔE**<5 |
| Surface appearance | Visual inspection | No black specks |
| Inclusions of extraneous material | Visual inspection | Record |

### 5.3.2 PELLET BLENDS PREPARATION

Once PP pellets have been produced and tested, three additional blends of 50 % virgin – 50 % blend A "A X" (X being 0, 25 or 50) shall be produced for the converting tests. Keep separated the pellet samples previously produced and dry them to residual moisture level inferior to 0.1 wt%. Then, according to the values reported in 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 to also perform the tests with a sample of 50 % virgin and 50 % innovation (i.e. B.100, by replacing the test with B.25).

### 5.3.3 PELLET BLENDS COMPOSITION

Three different blends 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 PP container.

*Table 5: Pellet blends composition for the application tests*

| **BLEND** | COMPOSITION | % VIRGIN RESIN | EFFECTIVE % CONTROL | EFFECTIVE % INNOVATION |
|---|---|---|---|---|
| **B.0** | 50 % Virgin Pellet<br>50 % A.0 | 50 | 50 | 0 |
| **B.25** | 50 % Virgin Pellet<br>50 % A.25 | 50 | 37,5 | 12,5 |
| **B.50** | 50 % Virgin Pellet<br>50 % A.50 | 50 | 25 | 25 |
| **OPTIONAL B.100** | 50 % Virgin Pellet<br>50 % A.100 | 50 | 0 | 50 |

### 5.3.4 BOTTLE BLOW MOULDING

In case, bottles blow moulding have been chosen by the RecyClass PP TC and the Applicant as converting step to assess the highest value recyclate application, the following procedure must be applied.

Control blend B.0 must be moulded first.

**Procedure:**

- Samples B.0, B.25 and B.50 samples (optionally B.100- should be blown moulded at 190-210 °C into a one-litre laundry detergent bottle, 1 mm tick.
- The characteristics of the bottle must be the following ones:
  Cross section: Rectangular, square, or circular.
  - The bottom corners should have radii as small as commercial laundry detergent bottles.
  - Bottle height should be typically for a one-litre laundry detergent bottle.
  - Neck may be offset.
  - The bottle must weigh 50 ± 2 g.
- The bottle must weigh 50 ± 2 g.
- Samples B.25 and B.50 (optionally B.100) must be blown following identical operating conditions of the control sample B.0.
- Small variations in operating conditions could be acceptable but must be documented in the report.

Record the resulting properties in Table 5. If some operating conditions need to be modified for B.25 and B.50 samples, this information must be documented in the report.

### 5.3.4.1 BOTTLE PROPERTIES CHARACTERISATION

*Table 6: Bottle properties characterisation*

| **ASSESSMENT** | STANDARDS | BENCHMARK RECOMMENDATION |
|---|---|---|
| Bottle Appearance | Visual defects, including surface roughness | Minimum of 10 bottles (compare with B.0) |
| Bottle Integrity | Visual inspection | |
| Bottle Dimension (mm) | Height | ± 2 % to B.0 |
| Bottle Weight (g) | Weight | |
| Bottle Capacity (mL) | Brim-full | |
| Thickness (mm) | Top, mid, and bottom side wall, shoulder, base corner | Minimum 0.3 mm for each measure |
| Top load (kg) | ASTM D2659 (no ISO available) | No more than a 10 % delta decrease to B.0 |
| Drop impact (m) | ASTM D2463, procedure B (no ISO available) | |
| Additional observation | Deposit on tooling | None observed respect to B.0 for 2 hours bottle production |

## 5.3.5 SHEET EXTRUSION

On the base of results obtained by pellet characterization, the RecyClass PP TC and the Applicant can optionally decide to test the innovation for sheet extrusion.

As reported in the section 5.3.1, 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). Tag them as C.0, C.25 and C.50, respectively.

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

Control pellet blend C.0 has to be extruded first.

**Procedure:**

- Dry samples C.0, C.25 and C.50 (optionally C.100) at 90 °C for 2 hours.
- Extrude sheets at 220 ± 5 °C with thickness of 1000 µm under conditions determined for the control sample C.0. Melt residence time in the extruder should be no more than 6 minutes.
- Extrusion run time per variable, no less than 30 minutes.
- Samples C.25 and C.50 (optionally C.100) have to be extruded following the identical operating conditions of the control sample C.0.
- Small variations in operating conditions could be acceptable but have to be documented in the report.

Record the resulting properties in Table 6. If some operating conditions have to be modified for C.25 and C.50 samples, this information must be documented in the report.

### 5.3.5.1 SHEET PROPERTIES CHARACTERISATION

*Table 7: Sheet properties characterisation*

| **ASSESSMENT** | STANDARDS | BENCHMARK RECOMMENDATION |
|---|---|---|
| Tensile modulus (GPa) | ISO 527 | No more than a 25 % decrease to C.0 |
| Tensile Stress at Yield (TD*) (MPa) | ISO 527 | |
| Tensile Stress at Yield (MD**) (MPa) | ISO 527 | |
| Tensile Stress at Break (TD*) (MPa) | ISO 527 | |
| Tensile Stress at Break (MD**) (MPa) | ISO 527 | |
| Colour | Visual inspection | No discolouration |
| Surface Appearance | Visual inspection | No black specks |
| Inclusions of extraneous material | Visual inspection | Record |

** TD: transverse direction*

*** MD: machine direction*

# DOCUMENT VERSION HISTORY

| VERSION | PUBLICATION DATE | REVISION NOTES |
|---|---|---|
| 1.0 | June 2020 | Recyclability Evaluation Protocol for PP Containers release |
| 2.0 | May 2021 | Major modifications about procedure, wording & template |
| 3.0 | January 2022 | Revised wording and removal of some testing |
| 3.1 | August 2022 | Flowchart update<br>Mandatory washing and floatation step for control sample removed<br>Wording for sample quantity requested for testing<br>Temperature for extrusion now specific to melt-temperature |
| 4.0 | January 2023 | Addition of hot washing procedure<br>Addition of mild temperature for drying<br>Addition of procedures for extrusion<br>Modifications of benchmark recommendations for injection moulded parts characterisation<br>Tensile testing for bottles removed<br>Revision of wording |
| 5.0 | January 2024 | Clarification of the unit's system to be used.<br>Addition of a mass balance report at each stage of pre-processing<br>Moisture characterisation<br>Removal of bulk and pellet size characterisation<br>Clarification on colour, volatiles, and gas content characterisations<br>Addition of ISO 19069-2:2016 standard<br>Harmonisation mechanical characterisation parameters<br>Revised wording |
| 6.0 | January 2025 | Clarification on grinding sieve size<br>Addition of torque as a variable to monitor during extrusion<br>Addition of a table to characterise the extrusion process<br>Modification on volatiles characterisation<br>Revised wording<br>Peer reviewed version |
| 6.1.0 | January 2026 | Template change<br>Addition guidance on blend preparation<br>Clarification on flotation step |

Identification of versions

v.X - Structural modification impacting the protocol.

v.X.Y – Updates to testing conditions. integration of new tests, technologies or analytical methods.

v.X.Y.Z - Editorial modifications not impacting the content of the Protocol.

# ANNEX I – CONTROL SAMPLES SELECTION

| **PP RESINS** | APPLICATIONS | DENSITY, $g/cm^3$ | MFI at 230 °C / 2.16 kg, g/10 min | FLEXURAL MODULUS, MPa | CHARPY IMPACT STRENGTH, $kJ/m^2$ |
|---|---|---|---|---|---|
| RB206MO | Bottles (Food, cosmetics) | 0.905 | 1,9 | 1100 | 7 |
| BB125MO | Bottles (industrial chemicals) | 0.905 | 1,3 | 1200 | 50 |
| RB307MO | Containers (detergents, cleaners, chemicals, oil) | 0.905 | 1,5 | 850 | 20 |
| HC205TF | Thermoforming (trays, cap, container) | 0.905 | 4 | 1700 | 5 |
| BH345MO | Thin wall packaging | 0.905 | 45 | 1300 | 6 |

# ANNEX II – MASS BALANCE PRE-TREATMENT STEPS

| **MASS (g)** | CONTROL SAMPLE | INNOVATION SAMPLE |
|---|---|---|
| Before grinding: $m_0$ | | |
| After grinding: $m_1$ | | |
| Floating fraction after sink-float separation: $m_2f$ | | |
| Sinking fraction after sink-float separation: $m_2s$ | | |
| After drying: $m_3$ | | |
| Heavy fraction from elutriation: $m_4h$ | | |
| Light fraction from elutriation: $m_4l$ | | |
| Pre-treatment yield: $\eta_{PT}$ | | |

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