RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR PP CONTAINERS

STANDARD LABORATORY PRACTICE

VERSION 5.0

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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 B.0 pellets
B.25	Bottle made of 50 % of virgin pellets and 50 % of B.25 pellets
B.50	Bottle made of 50 % of virgin pellets and 50 % of B.50 pellets
B.100	Bottle made of 50 % of virgin pellets and 50 % of B.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 benchmark
D.0	Plate made of 100 % control pellets
D.25	Plate made 75 % control and 25 % innovation pellets
D.50	Plate made 50 % control and 50 % innovation pellets
D.100	Plate made 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 an initiative working on enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Recyclability Evaluation Protocols promote recyclability by encouraging industry to test new plastic technologies, materials or product before market launch and giving advice and recommendations to the companies.

The Recyclability Evaluation Protocols are available to download on the RecyClass website. Companies providing plastic packaging concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance 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, is the decision-maker regarding the compatibility of the innovation with recycling according to the results of the evaluation, granting Recyclability Approval Letter to the Applicant.

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and must 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.*

RecyClass

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 that must be followed by the applicant at a laboratory scale in order to determine if a plastic packaging innovation is compatible with the post-consumer PP 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. It aims to prove the recyclability[4] of plastics 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. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for natural, white and coloured PP containers[6] in order to confirm that the PP innovation is compatible with these requirements.

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

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, 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 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 packaging.

Within RecyClass, “easy-to-empty” and “easy-to-access” indexes are important 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, no detergents nor 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 TEST METHODOLOGY

This methodology aims to reproduce the recycling process at laboratory scale to determine the suitability of an innovation for the PP recycling stream. The methodology described below shall be followed precisely and any modifications or problems must be noted during the testing phase. 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 be also noted down.

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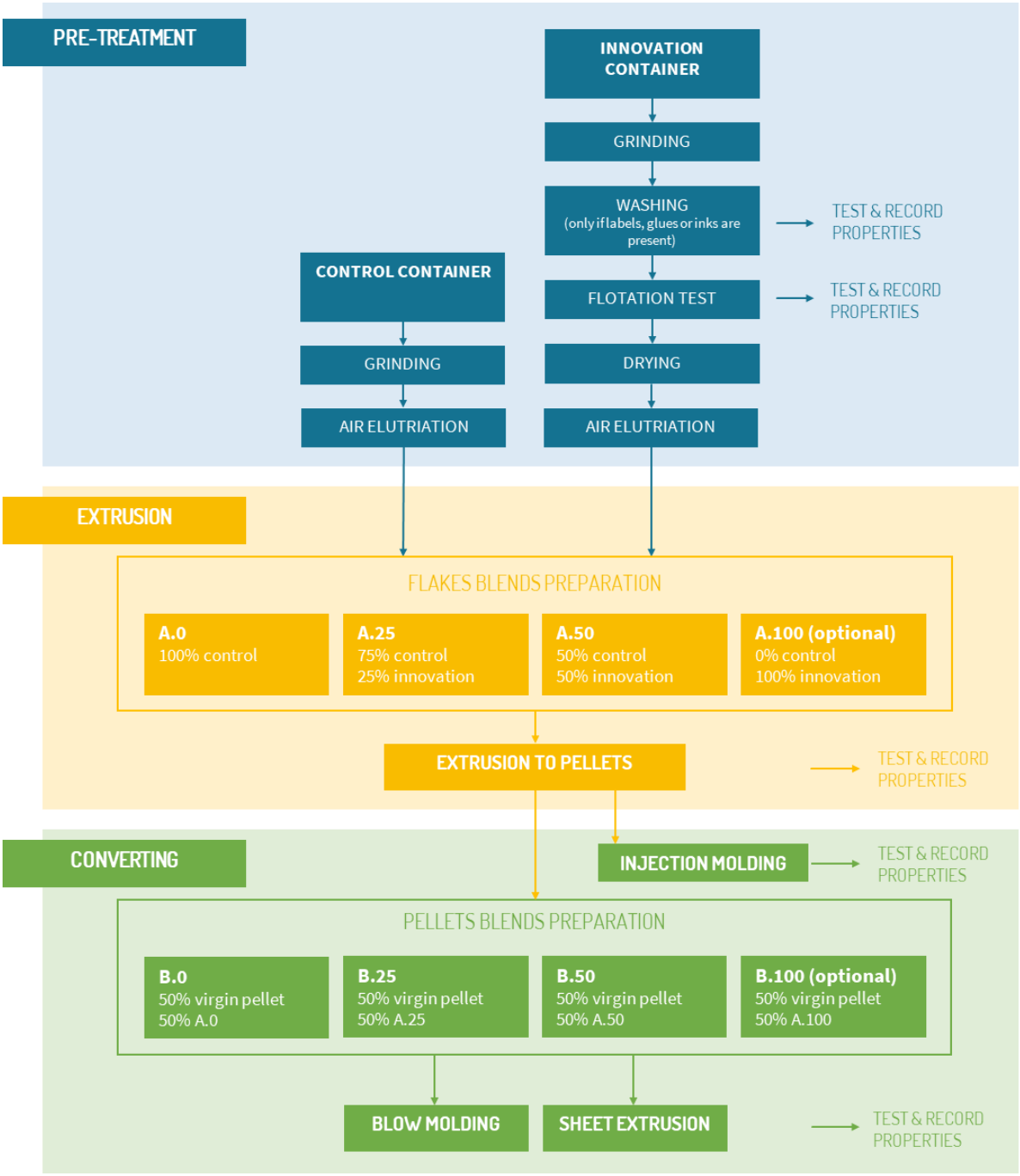
6 Design for Recycling Guidelines

[7] RecyClass Methodology

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

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 are to 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 a 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 full packaging (with labels, decoration, closures, etc.) can also be assessed according to the present 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 the Annex 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 throat 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 sample to flakes of 3 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, no detergents nor 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 bottle-making process. The RecyClass PP TC representatives are requested to select a washing procedure based upon the intended end-product. Both procedures take care of labels, adhesives, coatings, paper, and printing present in the innovation PP container. If none of those are present, go directly to step 5.1.3. In case of non-removable adhesives, procedure 1 should be performed and about 1 kg of innovation flakes should be assessed via the procedure 2, to evaluate washability under hot washing conditions.

The following procedures have to be utilized 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 manual stirring bar.
- Pour the liquid-flake mix over the de-watering screen and save the wash water.
- Report the mass of innovation sample after washing as m_2.
- 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 coloration. Check and record if the glue has been diluted after the rinsing or it remains attached to film flakes. In case water coloration, 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)). 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 stirrer on add PP flakes with its 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 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 manual stirring bar. Then drain the material. Save the water for further inspection.
- Spread flakes on a sheet and dry it in an oven at 60 °C to release 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 coloration. Check and record if the glue has been diluted after the rinsing or it remains attached to film flakes. In case water coloration, 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] RecyClass Quick Test Procedures

5.1.3 FLOTATION TEST

Following washing, the flotation process allows flake separation by density as occurring in the float/sink tank used in an industrial recycling line. For suitable recycling, both density separation efficiency and quality of the floating material should be optimized. Therefore:

- Combinations of polypropylene and other materials that float in water should be avoided in order to minimize the risk of contamination. In the case less than 100 % of the flakes would float, separation efficiency will be determined on the basis of the innovative packaging composition.
- Non-PP components floating together with PP flakes should be avoided as they cannot be further separated and are extruded with PP. This non-PP components are therefore acting as contaminants and could pose relevant concerns both in the process operations and in the quality of the recyclate.

The following procedure has to be utilized for innovation samples only.

Procedure:

- 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 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.

The efficiency of the sink/float separation should be measured using 50 g of washed flakes of innovative samples 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 tap water (pH between 7 and 8).
- Boil the water for 10 minutes, and then cool at room temperature.
- Transfer 300 ml of water in a graduated beaker.
- Put the innovative sample 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 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.
- 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:

$$\eta = \frac{W_F}{W_I}\ x\ 100 = \frac{(W_I - W_S)}{W_I}\ x\ 100\ [\%]$$

Where:

η: Test efficiency

W_F : weight of floating fraction

W_S : weight of sinking fraction

W_I : 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 the application of vacuum or heat sources until 1 % moisture content is reached. If the moisture content cannot be reached under these conditions, the application of mild heat (less than 60 °C) can be used with prior notification and approval from RecyClass.
- Report the mass after drying as m_4.
- 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 follows:

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

Where:

η: Pre-treatment yield

m_0 : mass of sample before grinding

m_4 : mass of sample after drying

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_5h and m_5l respectively.

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 at ambient air. 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.

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

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 HDPE container (optionally 100 % innovation), will be prepared as described in Table 1.

Eventually, 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	% 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

5.2.3 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blends preparation. The flakes will be dried under the same conditions with hot air and 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 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 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) with hot air at 90 °C for 1 hour maximum 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 220 ± 5 °C and with a 120 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 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 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 characterizations 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 have 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 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 the Table 2.

If filterability is seen as a potential problem for the innovative samples, 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 has to be monitored during pelletizing since a pressure increase is an indication of the risk of filter contamination. If from previous step, the monitoring of pressure-drop and the visual inspection of the filter after the pelletisation induce to further analyse contamination, a dedicated filtration test should be done. 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 has to 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 have to 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 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

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%)	Heat 10 g pellets exposed to 180 °C for 10 minutes	< 1.0 % weight loss
Ash content (%)	ISO 3451-1 (muffle) up to 750 °C	A.50 lower than 1 wt% (A.100 lower than 2 wt%)
Filtration (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 screen
Moisture (wt%)	Moisture analyser	< 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 5 % for A.25 and A.50 (and eventually A.100)
Average Pressure (MPa)	Measure it after extruding through 120 µm filter for the stable 30 minutes run time, compared to 100 % control	Average Pressure: No more than a 10 % delta increase to A.0
Pressure Variation (MPa)	($\Delta P_{25\text{-}30\text{minutes}}$ - $\Delta P_{5\ \text{first minutes}}$)	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 **have to 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 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 aiming to assess 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) have to be tested for injection moulding to evaluate tensile properties, colours, as well as defects.

Control pellets A.0 have to 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 plates with measures of about 60 x 60 x 2 mm^3.
- The specimens should be completely filled without any shrinkage, overspray, and inclusions.
- Samples A.25 and A.50 (optionally A.100) have to 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 D.0, D.25 and D.50 (optionally D.100), respectively.
- Small variations in operating conditions could be acceptable but have to 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 have 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 19069-2:2016 (Polyethylene (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
Flexural Modulus (MPa)	ISO 178	No more than a 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 ISO 179-2 (optional)	
Reflection Colour	(L*, a*, b*) Reflectance mode, D65, 8-10°	For natural stream: 60<**L***, -3<**a***<0, -5<**b***<5 **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 for 10 minutes at 60 °C. 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.

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. 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 (end eventually 50 %) by weight of the initial innovation PP container.

Table 4: Pellet blends composition for the application tests

BLEND	COMPOSITION	% VIRGIN RESIN	EFFECTIVE % CONTROL	EFFECTIVE % INNOVATION
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

5.3.4 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 has to be moulded first.

Procedure:

- The samples B.0, B.25 and B.50 (optionally B.100) should be blow moulded at 190-210 °C into 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 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) have to be blown following the identical operating conditions of the control sample B.0.
- Small variations in operating conditions could be acceptable but have to be documented in the report.

Record the resulting properties in Table 5. If some operating conditions have 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 5: 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 6: Sheet properties characterisation

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATION
Tensile modulus (GPa)	ISO 527	No more than a 25 % delta 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*

RecyClass

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 Mandatory washing and floatation step for control sample removed Wording for sample quantity requested for testing Temperature for extrusion now specific to melt-temperature
4.0	January 2023	Addition of hot washing procedure Addition of mild temperature for drying Addition of procedures for extrusion Modifications of benchmark recommendations for injection moulded parts characterization Tensile testing for bottles removed Revision of wording
5.0	January 2024	Clarification of the unit's system to be used. Addition of a mass balance report at each stage of pre-processing Moisture characterisation Removal of bulk and pellet size characterisation Clarification on colour, volatiles, and gas content characterisations Addition of ISO 19069-2:2016 standard Harmonisation mechanical characterisation parameters Revised wording

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 FOR PRE-TREATMENT STEPS

MASS (g)	CONTROL SAMPLE	INNOVATION SAMPLE
Before grinding: m_0		
After grinding: m_1		
After washing: m_2		
Floating fraction after sink-float separation: m_3f		
Sinking fraction after sink-float separation: m_3s		
After drying: m_4		
Pre-treatment yield: η_{PT}		
Heavy fraction from elutriation: m_5h		
Light fraction from elutriation: m_5l		

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