RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR PET BOTTLES

STANDARD LABORATORY PRACTICE

REP-PETbot-01

VERSION 1.1

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RecyClass

CONTENT

RecyClass

GLOSSARY

A.0	100 % control bottle flakes
A.25	Blend 75/25 control/innovation flakes
A.50	Blend 50/50 control/innovation flakes
AA	Acetaldehyde
ASTM	American Society for Testing and Materials
B.0	Blend made of 50 % of virgin pellets and 50 % of A.0 pellets
B.25	Blend made of 50 % of virgin pellets and 50 % of A.25 pellets
B.50	Blend made of 50 % of virgin pellets and 50 % of A.50 pellets
Control Sample	Plain PET bottle (or PET resin that has already been thermally processed once) used as benchmark
D.0	Injection moulded plaque made of B.0 blend
D.25	Injection moulded plaque made of B.25 blend
D.50	Injection moulded plaque made of B.50 blend
Innovation Sample	PET bottle containing the innovative technology
ISO	International Organization for Standardization
IV	Intrinsic Viscosity
PET	Polyethylene terephthalate
SSP	Solid-State Polymerisation
TC	Technical Committee
Virgin Material	PET resin that will for the first time be converted to a plastic product (no thermal pre-treatment)
wt%	Weight Percentage

DISCLAIMER

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

The Recyclability Evaluation Protocols are freely available for 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 to 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 technology or product 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 in the *RecyClass website."*

RecyClass

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "RecyClass[1] Recyclability Evaluation Protocol for PET bottles" referred to in this document as "The Protocol" describes the methodology the applicant must follow at a laboratory scale in order to determine if a plastic packaging innovation is compatible with the post-consumer PET bottle recycling streams, meaning transparent clear/light blue, transparent coloured and opaque coloured PET streams. The Protocol targets companies responsible for introducing a packaging product into the market. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass 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 guarantee recyclability[4] of plastics packaging while encouraging innovation in the PET bottle market. The overall goal is to maintain the protection of packaged goods and their marketing display functions without obstructing the proper functioning of the PET bottles recycling process. RecyClass protocol targets benchmarks that are based on requirements for bottle-to-bottle closed loop applications.

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

PET bottle terminology, as it is used in this document, is defined as a rigid plastic bottle predominantly used for packaging liquids, beverages and detergents or cosmetics.

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 Technology & Product Approval Quality Management & Procedures*

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

2. SCOPE OF THE PROTOCOL

The scope of the Protocol covers any innovation introduced to the existing packaging solutions for PET bottles. Before initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for transparent clear/light blue PET bottles, transparent coloured PET bottles and opaque coloured PET bottles[1] to confirm that the PET innovative bottle is compatible with the recycling stream for PET bottles..

1. The scope of this Protocol covers the following non-exhaustive list of packaging solutions and/or innovations: PET resins
2. Barrier materials
3. Additives that do not affect the density of the PET packaging
4. Non-PET closure systems
5. Non-PET liners, seals, and valves
6. Decorations of PET bottles
7. Adhesives for labels
8. Printing and inks

Following RecyClass recyclability methodology[2], packaging containing aluminium, metal, bio/oxo degradable plastics, black carbon surface, as well as PVC, PVDC and PC layers are considered disqualified for PET bottles recyclability. By consequence, packaging containing one 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 actual PET bottles recycling process works at an industrial scale. RecyClass PET Technical Committee reserves the right for further testing if necessary, to issue a final opinion on the recyclability of the tested innovation. The Recyclability Evaluation Protocol establishes some benchmark recommendations to guide the decision-making process. However, only some of the properties listed in the protocol are provided with a benchmark recommendation, given that the evaluation is also based on the technical expertise of the Technical Committee (TC).

Sorting behavior of PET bottles is also important to consider, since some innovations (in particular decorations) can negatively affect the sorting efficiency to the right PET stream. Therefore, it is recommended to perform a sorting test according to RecyClass Sorting Evaluation Protocol for Plastic Packaging to ensure that packaging presenting a risk of missorting is sorted in the right PET stream.

Furthermore, within RecyClass, “easy-to-empty” and “easy-to-access” indexes are essential factors when considering the recyclability of a package. Despite washing operation at a recycling facility uses hot washing conditions, with detergents and caustic soda, some residues can persist after washing. 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 leftover material at the end of its useful life. Nonetheless, this factor is beyond the scope of this Protocol.

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1 Design for Recycling Guidelines

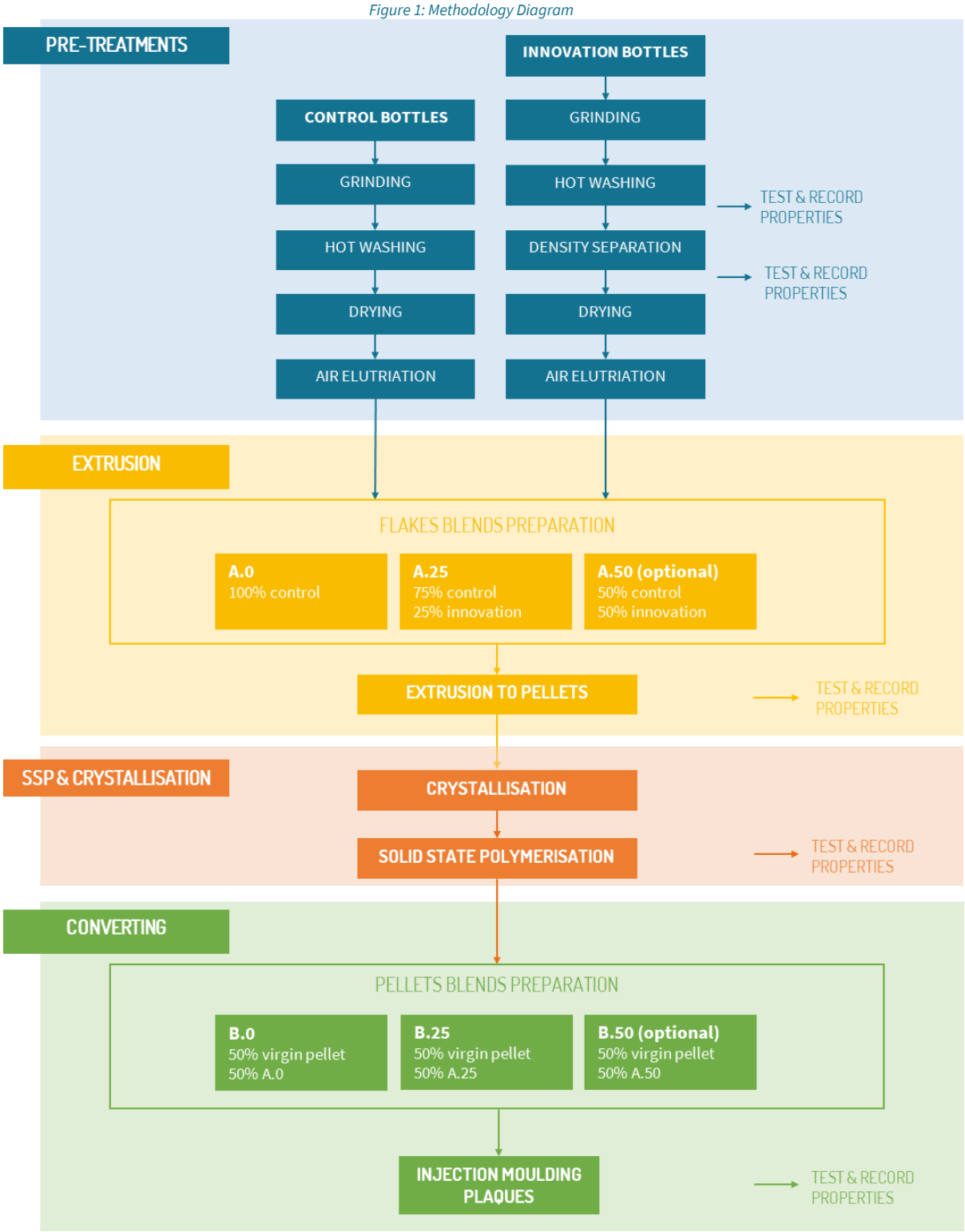
[2] RecyClass Methodology

4. LABORATORY TESTING METHODOLOGY

This methodology aims to reproduce the recycling process at laboratory scale to determine the suitability of an innovation for the PET bottle recycling streams. The methodology described below shall be followed precisely and any modifications or problems must be noted by laboratory technicians during the testing phase. Additional tests may be requested by the PET TC for specific cases requiring particular attention. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared and submitted to the RecyClass PET Technical Committee which will interpret the final results and define the compatibility or not with recycling. Any remarks during the laboratory tests described in the Protocol shall also be noted down.

See below in Figure 1 a diagram describing methodology.

Figure 1: Methodology Diagram



4.1 CONTROL SAMPLE SELECTION

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

- **Option 1:** Mono PET bottles (100 % PET) produced using the exact same PET grade as the innovative packaging. The PET grades for such options are listed in the Annex 1 and must be approved by the RecyClass PET TC.
- **Option 2:** The applicant can select a PET resin listed in the Annex to be used as control for this Protocol, with/upon the approval of the PET RecyClass Technical Committee. To obtain the control, the selected PET resin must be processed and transformed into mono PET bottles. The same physical form as the innovative material should be preferred.

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

Since control material is used as a reference to evaluate the impact of the innovation, A.0 and B.0 (see Figure 1), it 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 10 kg of innovation material (as packaging) and 25 kg of control material (as packaging) will be requested to prepare blends of at least 5 kg each. More innovation sample could be requested in case optional tests are required by the RecyClass PET Technical Committee. 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 discriminate the impact of a specific innovation on the recyclability of PET bottles. Note that complete packaging (with labels, decoration, closures, …) can also be assessed under this present protocol.

4.2 VIRGIN SAMPLE SELECTION

The virgin PET sample to be used in this Protocol can be selected from the PET 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 PET TC and used as it is (i.e., without applying any thermal pre-treatment). The virgin PET sample should not correspond to PET bottle flakes, but must be an unprocessed PET resin.

5. LABORATORY TEST PROCEDURES

5.1 PRE-TREATMENT STEPS

5.1.1 GRINDING

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

Procedure:

- Report the mass of each sample before grinding as m_0 in the table in Annex 2.
- Grind separately control and innovation sample to flakes using a 12 mm screen with a dry grinder, leading to 8-12 mm flakes.
- Report if high quantities of fines (< 1 mm) are obtained.

- Store in separate containers.
- Take photos of each fraction.
- Keep about 20 g of control and innovation flakes for melt temperature analysis, IV and colour measurement. Remove this mass from m_0 to have a fair comparison of m_1 to m_0.
- Report the mass of each sample after grinding as m_1 (Annex 2).

5.1.2 WASHING

Control and innovation samples are washed to test the impact on hot washing operations. Washing for PET bottles is commonly made in presence of caustic soda as well as detergents and is multiple steps process. The following procedures must be used for both control and innovation samples separately.

The tank to be used should respect the following conditions:

- Sized to allow PET flake and water to be mixed at a 1:4 ratio by weight.
- Will allow a recommended ratio of the water height-to-the tank width of 0.8 to 1.0 and not less than 0.6 or greater than 1.5.
- Fitted with a mixer with an impellor blade length at least 1/3 of the mixing tank diameter or width, and with variable speed capability for impeller.
- Incorporating at least three baffles when a round tank is used; baffles not necessary in a square tank

Procedure washing:

- Prepare the washing stainless steel tank for a 1:4 ratio (1 kg flakes vs 4 L solution) at 85°C with a solution of 1 wt% NaOH and 0.3 wt% MacDermid RP-24 detergent.
- Wash control and innovation samples separately at a 1:4 ratio (1 kg flakes vs 4 L solution) at 500 rpm for 15 minutes. Flakes are collected and separated from the washing solution over a vibrating table and through a centrifuge or similar equipment.
- Take photos of the washed solution and flakes after washing.

Procedure hot rinsing:

- Prepare the hot rinsing stainless steel tank for a 1:4 ratio (1 kg flakes vs 4 L solution) at 45 °C with tap water.
- Rinse washed control and innovation samples separately at a 1:4 ratio (1 kg flakes vs 4 L solution) at 240 rpm for 5 minutes.
- Flakes are collected and separated from the hot rinsing solution over a vibrating table and through a centrifuge or similar equipment.
- Take photos of the hot rinsing solution and flakes after hot rinsing.
- Report the mass of each sample after hot rinsing as m_2 (Annex 2).

5.1.3 DENSITY SEPARATION

The density separation test will determine if the flakes can be separated by density , simulating the process of a float/sink tank used in the recycling operation. Density separation will happen at the same time as the cold rinsing step.

The following procedure must be used for both control and innovation samples separately.

General Procedure:

- Pour the washed flakes in a tank filled with water at a 1:10 weight ratio at a room temperature. The tank should be high enough to enable separation of sinking and floating fractions.
- Stir at 240 rpm for 5 minutes.
- Stop the stirrer and allow the water to rest for 5 minutes.
- Remove the stirrer from the tank.
- Collect all particles that float on the surface with a sieve.
- Collect separately the flakes that sunk.

- Report the mass of the innovation sample after sink-float separation as m_3f and m_3s for floating and sinking fraction respectively (Annex 2).

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. The following procedure must be followed for the efficiency measurement on 50g sample.

Procedure to evaluate sink/float efficiency:

- 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 500 ml of water to a graduated beaker.
- Put the innovative sample in the water and stir at 240 rpm (overhead stirrer to be used) for 5 minutes.
- Stop the overhead stirrer and allow the water to rest for 5 minutes.
- Take photo 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 wash water for visual evaluation.
- Dry the sinking fraction at maximum 80°C to reach a moisture level below 1%.
- Cool to room temperature, weigh and record the weight of the sinking fraction.
- Calculate the test efficiency as:

$$\eta = \frac{m_3 s}{m_1} = \frac{(m_1 - m_3 f)}{m_1} \; x \; 100 \; [\%]$$

Where:

η: Test efficiency

m_3f : weight of floating fraction

m_3s : weight of sinking fraction

m_1: weight of innovative sample

5.1.4 DRYING

Reduce the flake moisture according to the following procedure.

Procedure:

- Dry the flakes collected after density separation with hot air without applying vacuum until 1 % moisture content is reached. The outlet temperature of the flakes should remain below 60 °C.
- Report the mass of each sample after drying as m_4 (Annex 2).
- Record the moisture content.

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

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

Where:

η_{PT}: 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 PET flakes are elutriated with air to remove the light fraction containing fines, remaining labels and potentially multilayers. A zig-zag air elutriator is recommended for this test.

Procedure:

- Elutriate with air with one pass and with about 0.1% loss set for the control flakes.
- Similar settings for the air elutriation must be applied for the innovation sample.
- Weigh the heavy fraction for innovation samples.

5.1.6 FLAKES PROPERTIES CHARACTERISATION

Flakes characterisation must be performed according to Table 1. Innovation flakes properties will be compared with the ones of the control sample. All flakes should meet the requirements reported in Table 3.

Table 1: Flake characterisation after washing

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Intrinsic viscosity (dL/g)	ISO 1133-2 (MFI measurement, 285 °C/2.16 kg)	Record
Melt temperature (°C)	ISO 11357-3:2018 (Heat-cool-heat cycle at 10 °C/min (heating and cooling) from 40 °C to 300 °C with 2 minutes of isotherm between each ramp)	Record T_m from the second heat
Colour (L*,a*,b*)	(L*, a*, b*) + ΔE Reflectance mode, D65, 8-10 °, SCI gloss setting	Record Determine $\Delta E = \sqrt{((a - a_0)^2 + (b - b_0)^2 + (L - L_0)^2)}$
Bulk Density (kg/m^3)	Annex B of EN 15344	Superior to 500 kg/m^3

5.2 EXTRUSION

5.2.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of innovative PET bottles against control as laid out in this Protocol, a set of flake blends is prepared as described in Table 2. Blends shall be produced once the control and innovation bottles have separately gone through all pre-treatment steps described above.

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

Depending on the nature of the innovation, the RecyClass PET TC can also ask the Applicant to perform the tests with a sample of 50% control - 50% innovation (i.e. A.50) (See Table 2).

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.).

Table 2: Flake blends composition for the production of pellets

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

5.2.2 PELLET PRODUCTION

Both control and innovation flakes can be mixed manually before extrusion for blends preparation. The flakes will be dried at the same conditions with a desiccant bed drying unit or with hot air at 160 °C maximum and extruded using a single screw extruder at a melt temperature of 285 °C. Note that twin screw extruder may be used under specific conditions, previously discussed with laboratories. The extrudate will be melt filtered with a 100 µm filter (40/150/40 mesh filter pack).

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.

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

Procedure:

- Dry samples A.0, and A.25 (optionally A.50) with a bed desiccant at 160 °C, to obtain a moisture level inferior to 50 ppm. Typical drying time at 160 °C is 4-6 hours.
- Extrude at a melt temperature of 285 ± 5 °C with a suggested filtration screen of 100 µm (40/150/40 mesh filter pack). Usually a 25 to 35 mm extruder with a 24:1 to 36:1 L/D is suitable for laboratory use. If the range is not optimal, record temperature and state reasons for alteration. Melt residence time should be less than 6 minutes.

- Recommended throughput is between 5 and 10 kg/h and rotation speed between 100 and 200 rpm.
- Extrusion run time per variable, no less than 30 minutes.
- Extruded strands are rapidly cooled within a water bath, then dried and fed it into the pelletiser to produce amorphous pellets. Pellets should be about 2.5 mm of diameter, and pellet weight of 1.6 to 2.0 g/ 100 pellets is suggested.
- Torque and pressure over time must be monitored and reported. If a continuous monitoring is not possible, 5 data points should be measured within the 30 min extrusion.
- Maintain pressure increase to less than 25 % from the control over a stable 15 minutes run time.
- A small amount of the dried samples will be extracted from the dryer before they enter the extruder. This will allow to evaluate the impact of the drying process on the agglomeration of the flake samples. Agglomerated flakes should represent less than 1 % of the collected sample.
- After emptying the dryer, the hopper will be checked for flakes sticking to the hopper sidewall. Agglomeration should not lead to problems emptying the hopper by gravity without additional mechanical action. The cone of the hopper should have an angle of 60-70 degrees.

Record properties' results in Table 3. The processing conditions used for all the samples must be identical. If some operating conditions toned to be modified for A.25 (and optionally A.50) sample, this information must be documented in the report. A small amount of each sample (50 g) will be retained for RecyClass PET Technical Committee and the Applicant. The extruded pellets will be tested for pellet properties evaluation (Table 3). 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 3.

5.2.2.1 PELLET PROPERTIES EVALUATION

Table 3: Pellets properties evaluations

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Intrinsic viscosity (dL/g)	ISO 1133-2 (MFI measurement, 285 °C/2.16 kg) or ISO 1628-1:2021 (IV in solution)	IV drop (before/after extrusion) of A.25 is inferior to the IV drop of the A.0 ± 0.025 dL/g (IV drops are measured comparing IV of pellets to unwashed flakes)
Filtration (100 µm)	Visual inspection	No build-up on the screen
Impurities (unmolten particles)	Visual inspection	Record
Surface appearance	Visual inspection	Record
Moisture level	Moisture analyser	Record
Reflection Colour	(L*, a*, b*) + ΔE (comparison to A.0) Reflectance mode, D65, 8-10 °, SCI gloss setting	Record Δb* of A.25 (before/after extrusion) is inferior to Δb* of A.0 ± 2 and -3.5 < b* < 2.5
Average Pressure (MPa)	Average pressure after extruding through 100 µm filter for the stable 15 minutes run time, compared to 100 % control	No more than a 25 % 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

Extrusion process	Unusual sticking, fumes, odour, and any build-up	Record

5.3 CRYSTALLISATION & SOLID-STATE POLYMERISATION

Both A.0 and A.25 (optionally A.50) amorphous pellets have to be treated through crystallisation and solid stating polymerisation (SSP) processes. These steps of the process will allow to obtain crystallised PET pellets, which will then be characterised. A.0 and A.25 (optionally A.50) amorphous pellets must be processed separately. This procedure must be used prior conversion (see section 5.3.1).

5.3.1 CRYSTALLISATION

The crystallisation step must be performed in an oven or a crystallizer at 160 °C for 1 hour. If vacuum oven is not available, nitrogen atmosphere should be used and mentioned in the report. It is important that pellets are efficiently crystallised and are not discoloured or sticking together during the crystallisation process. Store crystallized pellets in a sealed container or under dry conditions to maintain their moisture content < 2500 ppm.

5.3.2 SOLID-STATE POLYMERISATION (SSP)

The SSP step is used to prepare the samples for the injection moulding step, but also to determine the solid stating speed. For injection moulding conversion, SSP must be done as following, with similar conditions for control and innovation samples:

- SSP temperature must be set at 205 °C (sample temperature). SSP must be carried out using a rotatory vacuum unit, with a constant spinning and under vacuum during the full process. Report parameters for the SSP, like vacuum level, fill level or nitrogen flow.
- SSP process must last 8 hours, with the time starting from the moment sample reaches 200 °C.
- Samples must reach 205 °C within the first 2 hours.
- About 50 g of samples must be collected after respectively 2, 4, 6 and 8 hours of SSP for characterization.
- After 8 hours of SSP, samples must be kept at room temperature for cooling down.

5.3.2.1 SOLID STATE PELLETS CHARACTERISATION

Table 4: Solid state pellets properties evaluation

ASSESSMENT	STANDARD	BENCHMARK RECOMMENDATION
Intrinsic viscosity (dL/g)	ISO 1133-2 (MFI measurement, 285 °C/2.16 kg) or ISO 1628-1:2021 (IV in solution)	IV drop (before/after SSP) of A.25 is inferior to the IV drop of the A.0 ± 0.025 dL/g IV curve over time to determine SSP rate from 2 to 8 hours. SSP rate: No more than 10 % delta variation to control
Melt temperature (°C)	ISO 11357-3:2018 (Heat-cool-heat cycle at 10 °C/min (heating and cooling) from 40 °C to 300 °C with 2 minutes of isotherm between each ramp)	Melt temperature second heat: No more than 10 % delta variation to control

Reflection Colour	(L*, a*, b*) + ΔE (comparison to A.0) Reflectance mode, D65, 8-10 °, SCI gloss setting	Record Δb* of A.25 (before extrusion/after SSP) is inferior to Δb* of A.0 ± 3 and b* < 5
Acetaldehyde concentration (ppm)	-	No more than 35 % delta increase to control
Fluorescence	Test under UV lamp (385 nm)	Visual inspection. No fluorescence should be visible

5.4 CONVERSION

Prior the recyclability assessment, the RecyClass PET Technical Committee will decide the process to be used for conversion according to the highest value recyclate application for the innovation and to the characteristics of the material to be evaluated. In the present case, PET bottle recyclate will be converted via injection moulding for colour evaluation. At that stage, an additional dilution with virgin PET will be done to replicate the incorporation of recycled content at industrial scale.

5.4.1 PELLET BLENDS PREPARATION

Once PET pellets have been produced and went through the SSP treatment, additional blends of 50% virgin – 50% blend "A.X" (X being 0, 25, or 50) shall be produced for converting tests. Keep separated the pellet samples previously produced and dry them at 160 °C maximum to reach a moisture level below 50 ppm. Then according to the values reported in following Table 4 prepare the different blends based on the A.X samples produced before. These new blends must be tagged as samples B.0, and B.25 respectively. Eventually, depending on the application, the TC can ask the Applicant also to perform the tests with a sample of 50 % virgin and 50 % A.50 blend, which should be tagged as B.50.

For the purpose of the tests the Applicant should provide enough virgin materials which allows for the blend preparations. The laboratory carrying out the Protocol testing can define the amounts according to their best knowledge. Such virgin material must be a grade listed in the Annex 1.

Different blends made of 50 % virgin pellet – 50 % Blend A shall be produced as described in Table 5. Blends will be composed of 0 %, 12.5 % (and eventually 25 %) content of the initial PET innovation.

Table 5 Pellet blends composition for the application tests

BLEND	COMPOSITION	% VIRGIN RESIN	EFFECTIVE % CONTROL PET	EFFECTIVE % INNOVATION SAMPLE
B.0	50 % Virgin Pellet 50 % A.0	50	50	0
B.25	50 % Virgin Pellet 50 % A.25	50	37.5	12.5
OPTIONAL B.50	50 % Virgin Pellet 50 % A.50	50	25	25

5.4.2 PLAQUE INJECTION MOULDING

Plaques will be produced via injection moulding in order to perform colour characterization of both control and innovative samples. Production of the 3 mm plaques should be done as following:

Procedure:

- Dry the samples B.0, B.25 (and optionally B.50) at 160 °C maximum to reach a moisture level below 50 ppm.
- Mould sample B.0 at 275-285 °C into plaques with measures of about 3.0 mm thickness.
- The mould temperature should be set between 15 and 20 °C, while melt residence time should be between 4 and 6 minutes.
- The run time is variable, but should be not less than 30 minutes. The specimens should be completely filled without any shrinkage, overspray, and inclusions.
- Samples B.25 (optionally B.50) must be moulded following the identical operating conditions of the control sample B.0.
- Tag the plaques produced by B.0, and B.25 (optionally B.50) as D.0, and D.25 (optionally D.50), respectively.

5.4.2.1 INJECTION MOULDED PLAQUES PROPERTIES CHARACTERISATION

Table 6: Injection moulded plaques properties evaluation

ASSESSMENT	STANDARDS	BENCHMARK RECOMMENDATIONS
Colour measurement (Transmittance mode)	L*, a*, b* and ΔE (compared to B.0) D65 illuminant, 8-10 °angle, SCI gloss settings	L* > 87 a* > -3 Δb* < 1.5 (compared to B.0) Δb* < 1 is recommended and 1 < Δb* < 1.5 is acceptable
Haze (%)	DIN EN ISO 14782 Measurement at 550 nm	< 8 %
Inclusions of extraneous material		Record. Limit the end use application.

RecyClass

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	January 2024	RecyClass Recyclability Evaluation Protocol for PET Bottles release
1.1	January 2025	Revised wording Modification IV characterisation and benchmarks Addition flakes DSC measurement Benchmark recommendations modification for colour SSP process clarification Peer reviewed version

RecyClass

ANNEX I – CONTROL & VIRGIN SAMPLES SELECTION

TYPE OF RESIN	**PET RESINS***	Melting Point, °C	Intrinsic viscosity, dL/g
PET	Equipolymers Lighter C93	247	0,80
PET	Indorama RAMAPET N1	247	0,80
PET	Indorama RAMAPET N180	245	0,80
PET	Indorama RAMAPET N1S	247	0,82
PET	Indorama 1708 CC (US only)	247	0,80
PET	Lotte PET Cool	245	0.80
PET	Plastiverd Global	245	0.80

**Other PET grades with similar intrinsic viscosity and melting point from alternative suppliers can also be accepted.*

ANNEX II – PRE TREATMENTS

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}		

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