RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR PS CONTAINERS

STANDARD LABORATORY PRACTICE

VERSION 3.0

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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	Sheet made of 50 % of virgin pellets and 50 % of B.0 pellets
B.25	Sheet made of 50 % of virgin pellets and 50 % of B.25 pellets
B.50	Sheet made of 50 % of virgin pellets and 50 % of B.50 pellets
B.100	Sheet made of 50 % of virgin pellets and 50 % of B.100 pellets
Control Sample	Plain PS container (or PS resin that has already been thermally processed once) used as benchmark
C.0	Plate made of 100 % control pellets
C.25	Plate made 75 % control and 25 % innovation pellets
C.50	Plate made 50 % control and 50 % innovation pellets
C.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
PS	Polystyrene
PVC	Polyvinyl Chloride
TC	Technical Committee
TGA	Thermogravimetric Analysis
Virgin Material	PS 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 PS 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 PS 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 PS market. The overall goal is to maintain the protection of packaged goods and their marketing display functions without obstructing the proper functioning of the PS recycling process.

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

PS terminology as it is used in this document, refers to rigid plastic containers predominantly used for yogurt pots and other food and beverage products, except XPS and EPS.

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 PS. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for PS containers[6] in order to confirm that the PS 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. PS resins
2. Barrier materials
3. Mineral fillers and additives that increase the density of PS packaging
4. Non-PS closure systems
5. Non-PS liners, seals, and valves
6. Non-PS labels and sleeves
7. Adhesives
8. Inks

Following the RecyClass Recyclability Methodology[7], packaging containing aluminium, metal, foam, 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 PS recycling works at an industrial scale. RecyClass PS 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 PS 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 PS 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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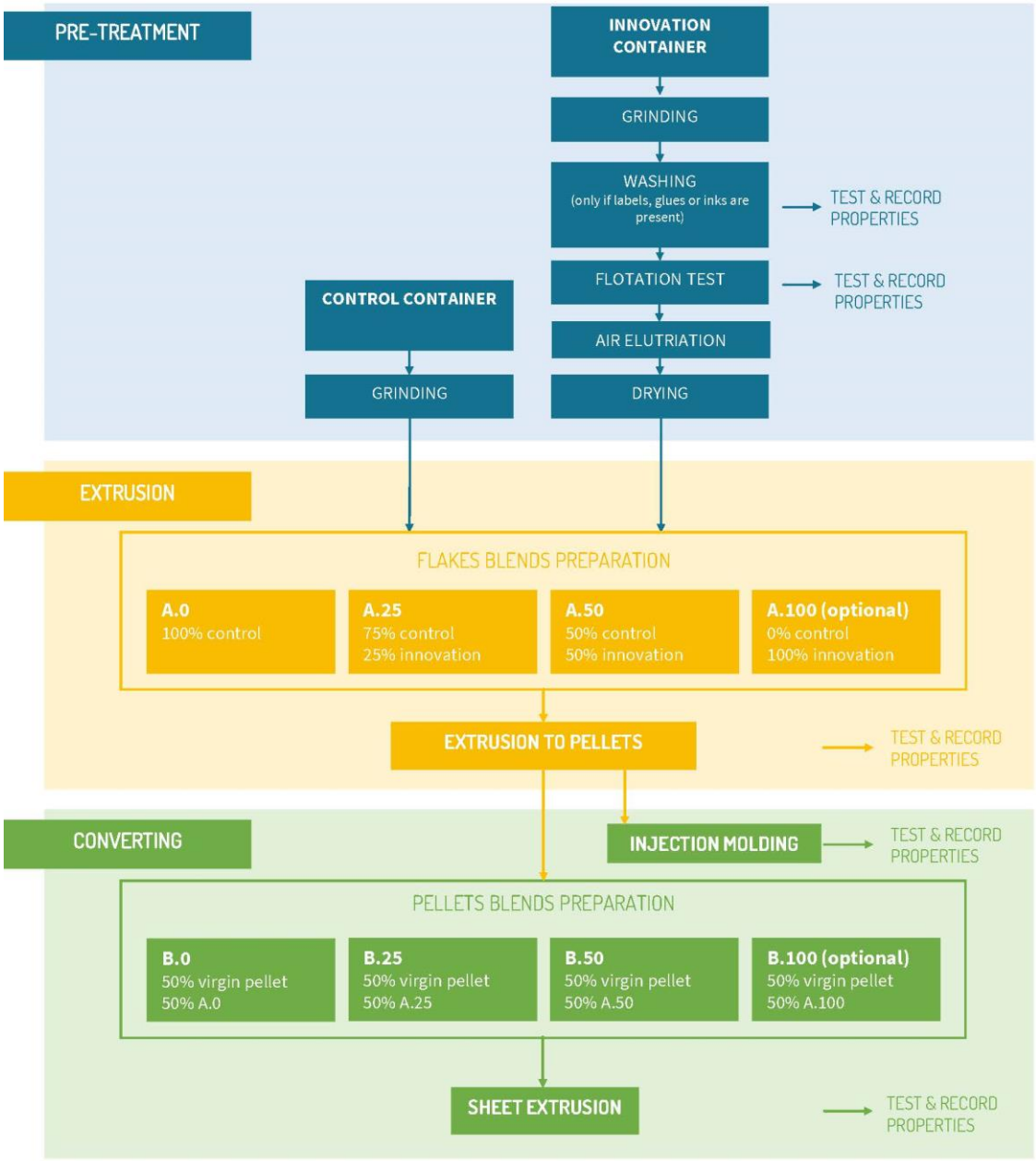
6 Design for Recycling Guidelines

[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 PS sample for the purpose of performing the Protocol can be selected:

- **Option 1:** If there is a PS container known to be recyclable, consisting of the same base PS 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 PS TC.
- **Option 2:** The Applicant can select a PS resin with the same critical technical specifications for MFI and density as the innovation article, ± 0.01 density can be used as the control for this Protocol, upon the approval of RecyClass PS TC. For MFI, please refer to the Annex 2 table summarizing the MFI range accepted depending on the innovation. A selection of control samples to be used is reported in Annex I. A mixture of the resins listed in Annex 1 reflecting the innovation structure can also be proposed as control, upon the approval of the Technical Committee. The selected material must be extruded at 230 ± 10 °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) need to fulfil some minimum requirements to make the Recyclability Evaluation valid. RecyClass recognised testing facilities are aware of these minimum requirements and will inform both the Applicant and RecyClass in case of deviations.

For the purpose of the tests the amount of material that the Applicant should provide will depend upon the equipment

and scale used in each laboratory. Usually, at least 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 PS 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 HDPE 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 PS sample to be used in this Protocol can be selected from the PS 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:

- Grind separately control and innovation sample to flakes of 3 to 15 mm.
- Store in separate containers.
- Recording the masses.

5.1.2 WASHING

Control and innovation PS samples are separately washed to test the impact on wet washing operations. The procedures take care of labels, adhesives, coatings, paper and printing present in the innovation PS container. If none of those are present, go directly to step 5.1.3.

The following procedures have to be utilized for innovation samples only.

Procedure:

- Prepare the washing water in a vessel at a 1:4 ratio (5 kg flakes vs 20 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 (5 kg flakes vs 20 l water) at 1,000 rpm for 5 minutes.
- Rinse the flakes in a strainer with cold running tap water and stir vigorously for 5 minutes using manual stirring bar.
- Then drain the material.
- 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. In the case of presence of adhesives, 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].

5.1.3 SEPARATION BY DENSITY

Following washing, the separation by density process allows flake separation by density as occurring in the float/sink tank used in an industrial recycling line. Non-PS components floating together with PS flakes cannot be further separated and are extruded with PS. This poses relevant concerns both in the process operations and in the quality of the recyclate, undermining its applications.

[8] RecyClass Quick Test Procedures

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

Procedure

STEP 1:

- Fill a vessel with tap water at a 1:12 ratio (5 kg washed flakes vs 30 l water).
- Add 0.2 wt% of dish detergent.
- Put each sample separately in the water and stir at 500 rpm for 4 minutes.
- Stop the stirrer and allow the water to rest for 10 minutes.
- Remove all the materials that float at the surface with a sieve.
- Take photos of the floating and sinking fractions separately
- Take photos of the water and save a wash for visual evaluation.

STEP 2:

- Fill a vessel with tap water at a 1:12 ratio (5 kg sinking fraction of Step 1 vs 30 l water).
- Add 0.2 wt% of dish detergent.
- Add 12 % of sodium chloride to the water solution (or any other salt) to increase the water density up to 1.08 g/cm^3.
- Put the sank samples from STEP 1 in the water and stir at 500 rpm for 4 minutes.
- Stop the stirrer and allow the water to rest for 10 minutes.
- Recover all the materials that float at the surface with a sieve.
- Rinse the flakes in a strainer with cold running tap water and stir vigorously for 5 minutes using manual stirring bar. Then drain the material.
- Take photos of the floating and sinking fractions separately
- Take photos of the water and save a wash for visual evaluation.

The test is passed if 100 % of polystyrene material is floating after the 2nd step. It means non-PS material cannot stick or not get separated from PS and cause PS to sink, resulting in yield losses or stay with PS and contaminate the PS 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. Repeat the procedure for washed and dried innovation flakes, with and without closure and labels.

Procedure:

STEP 1

- 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.
- Add a drop of dish detergent.
- Put the innovative sample in the water and stir at 500 rpm for 4 minutes.
- Stop the magnetic stirrer and allow the water to rest for 10 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 wash for visual evaluation.

STEP 2

- 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.
- Add a drop of dish detergent.
- Add 12 % of sodium chloride to the water solution (or any other salt) to increase the water density up to 1.08 g/cm^3.
- Put the sank samples from STEP 1 in the water and stir at 500 rpm for 4 minutes.
- Stop the magnetic stirrer and allow the water to rest for 10 minutes.
- Take photo of the beaker.
- Recover all particles that float at the surface with a sieve.
- Rinse the flakes in a strainer with cold running tap water and stir vigorously for 5 minutes using manual stirring bar. Then drain the material.
- Take photos of the floating and sinking fractions separately.
- Save the wash 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.
- Repeat the procedure with 50 g of the innovation flakes without caps and labels (if any).
- Calculate the test efficiency as

$$\eta = \frac{W_{F2}}{W_I}\ x\ 100 = \frac{W_I - (W_{S2} + W_{F1})}{W_I}\ x\ 100\ [\%]$$

Where:

ɳ: Test efficiency

W_{F1} : weight of floating fraction of step 1

W_{F2} : weight of floating fraction of step 2

W_{S2} : weight of sinking fraction of step 2

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 can be used with prior notification and approval from RecyClass.

5.1.5 AIR ELUTRIATION

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

Procedure:

- As for the second step, elutriate flakes with air with one pass and with less than 2 % loss set for the control flakes.

5.2 EXTRUSION

5.2.1 FLAKE BLENDS PREPARATION

For each sample obtained, to evaluate and record the properties of innovation PS 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 PS 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 a desiccant bed drying unit or with hot air and extruded using a co-rotative twin-screw extruder at a melt temperature of 230 °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. See additional information in Table 2.

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.

Table 2: Pellet production purpose & overview

FLAKE COMPOSITIONS	KG OF BLEND REQUIRED	PURPOSE OF BLEND
A.0 100 % Control flake	Per laboratory requirement for a 30-minute run time	All tests compared to control values
A.25 75 % control with 25 % innovation	Per laboratory requirement for a 30-minute run time	Required for comparison to control values
A.50 50 % control with 50 % innovation	Per laboratory requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL **A.100** 100 % innovation	Per laboratory requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Dry samples A.0, A.25 and A.50 (optionally A.100) with a bed desiccant for 1 hour at 80 °C or with hot air at 80 °C for 1 hour.
- Extrude first the sample A.0 (the control blend) at a melt temperature of 230 ± 10 °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.
- 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 3.
- 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 3. 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 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 the Table 3.

If filterability is seen as a potential problem for the innovative samples, a dedicated filter test should be requested by the RecyClass PS 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 PS.

About 5 kg of pellets from the pure control PS and the pure innovation PS 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 hot air at 80 °C for 1 hour.
- Extrude the sample at a temperature of 230 ± 10 °C and with a 120 µm melt filter pack, for no less than 30 minutes.
- 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 EVALUATION

Samples preparation and testing conditions of PS pellets for the following characterisations must be done according to ISO 24022-2:2020 Polystyrene (PS) moulding and extrusion materials, Part 2: Preparation of test specimens and determination of properties.

Table 3: Pellet properties evaluation

ASSESSMENT		STANDARD	BENCHMARK RECOMMENDATION
Bulk Density (kg/m^3)		ISO 60	Superior to 600 kg/m^3
Density (kg/m^3)		ISO 1183-1	Between 1 and 1.08 g/cm^3
Melt Index (g/10 min)		ISO 1133-1 (200 °C/5 kg)	Between 3 and 9 g/10 min
Ash content (wt%)		ISO 3451-1 by TGA	A.25, A.50 and A.100 respectively lower than 1.5, 3 and 5 wt%
Filtration (µm)		Visual inspection	No build-up on screen
Moisture (wt%)		ISO 11358-1	<0.1 wt%
Impurities		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (wt%)		10 g air-dried pellets exposed to 200 °C for 10 minutes	± 0.1wt% for A.25 and A.50 respect to A.0
Average Pressure (MPa)		Average Pressure it after extruding through 120 µm for the stable 30 minutes run time, compared to 100 % control	No more than a 10 % delta increase to A.0
Pressure Variation (MPa)		($\Delta P_{5\ last\ minutes}$ - $\Delta P_{5\ first\ minutes}$)	No increase higher than 25 % compared to start in 30 minutes

5.2.4 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 75 °C for 2 hours.
- Mould sample A.0 at 210-250 °C to multipurpose specimens' type 1A according to EN ISO 527-2 and to plates with measures of about 60 x 60 x 2 mm^3.
- 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 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 C.0, C.25 and C.50 (optionally C.100), respectively.
- Small variations in operating conditions could be acceptable but have to be documented in the report.
- For each material monitor the heat stability and the injection pressure.

Record the resulting properties in Table 4. 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.

5.2.4.1 INJECTION MOULDED PARTS PROPERTIES EVALUATION

Samples preparation and testing conditions of PS samples for the following characterisations must be done according to ISO 24022-2:2020 Polystyrene (PS) moulding and extrusion materials, Part 2: Preparation of test specimens and determination of properties.

Table 4: Injection moulded parts properties evaluation

ASSESSMENT		STANDARD	BENCHMARK RECOMMENDATION
Flexural modulus (MPa)		ISO 178	No more than 10 % delta decrease compared to A.0
Tensile Strength at Yield (%)		ISO 527	No more than 25 % delta decrease compared to A.0
Tensile Stress at Break (MPa)		ISO 527	No more than 25 % delta decrease compared to A.0
Charpy impact test strength (kJ/m^2)		ISO 179-1	No more than 10 % delta decrease compared to A.0
Reflection Colour		(L*, a*, b*)	Record
Surface Appearance		Visual inspection	No black specks
Inclusions of extraneous material		Visual inspection	Record

5.3 CONVERTING

Since the Protocol aims to assess the highest value recyclate application, sheet extrusion will be a priority. In any case, three blends of innovation and control pellets will be produced aiming to assess different innovation concentration in the recycling stream, as following reported.

5.3.1 PELLET BLENDS PREPARATION

Once PS pellets have been produced and tested, three additional blends of 50 % virgin – 50 % blend A 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 5, 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 % innovation (i.e. B.100, by replacing the test with B.25).

5.3.2 PELLET BLENDS COMPOSITION

Three different blends at 50 % virgin pellet – 50 % Blend A shall be produced as described in Table 5. Blends will be composed of 0 %, 12.5% and 25 % content (end eventually 50 %) by weight of the initial innovation PS container.

Table 5: 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

5.3.3 SHEET EXTRUSION

The Applicant has to submit its innovation primarily to sheet extrusion to test the recyclate obtained by the innovation in a closed-loop application.

Control pellet blend B.0 has to be extruded first. See more information in Table 6.

Table 6: Sheet production purpose & overview

PELLET COMPOSITIONS	KG OF BLEND REQUIRED	PURPOSE OF BLEND
B.0 50 % A.0 pellet and 50 % Virgin pellet	Per laboratory requirement for a 30-minute run time	All tests compared to control values
B.25 50 % A.25 pellet and 50 % Virgin pellet	Per laboratory requirement for a 30-minute run time	Required for comparison to control values
B.50 50 % A.50 pellet and 50 % Virgin pellet	Per laboratory requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL B.100 50 % A.100 pellet and 50 % Virgin pellet	Per laboratory requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Dry samples B.0, B.25 and B.50 (optionally B.100) at 60 °C for 10 minutes.
- Extrude sheets at melt temperature of 230 ± 10 °C with thickness of 800 µm under conditions determined for the control sample B.0. Temperature of the rollers should be fixed between 30 and 45 °C.
- Extrusion run time per variable, no less than 30 minutes.
- Samples B.25 and B.50 (optionally B.100) have to be extruded 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 10. If some operating conditions have to be modified for B.25 and B.50 samples, this information must be documented in the report.

5.3.3.1 SHEET PROPERTIES CHARACTERISATION

Table 7: Sheet properties evaluation

ASSESSMENT	RESULTS	STANDARDS	BENCHMARK RECOMMENDATION
Thickness			Variations lower than 3 %
Tensile Modulus (MPa)		ISO 527-3 or ISO 24022-2:2020	No more than a 10 % delta decrease to B.0
Tensile Stress at Yield (TD*) (MPa)		ISO 527-3 or ISO 24022-2:2020	No more than a 25 % delta decrease to B.0
Tensile Stress at Yield (MD**) (MPa)			
Tensile Stress at Break (TD*) (MPa)			
Tensile Stress at Break (MD**) (MPa)			
Colour		Visual inspection	No discolouration
Surface Appearance		Visual inspection	No black specks
Fisheyes		Visual inspection	Record the amount and size distribution per m^2. No fisheyes should be bigger than 1 mm^2.
Inclusions of extraneous material		Visual inspection	Record

**TD: transverse direction*

***MD: machine direction*

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DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	February 2022	Recyclability Evaluation Protocol for PS Containers release
1.1	August 2022	Mandatory washing and floatation step for control sample removed Wording for sample quantity requested for testing Temperature for extrusion now specific to melt-temperature
2.0	January 2023	Modification of option 2 for control selection Modification of the water/flake ratio, resting time and rinsing for the separation by density step Mild conditions accepted for drying Temperature and procedure for extrusion and injection modified
3.0	January 2024	Clarification on the units' system to be used Addition of ISO 24022-2:2020 for characterization of PS samples Correction of typos and revised wording

ANNEX I – CONTROL SAMPLES SELECTION

Type of PS	**PS**	Density, g/cm³	MFI, g/10min (190 °C/2,16 kg)
GPPS	Styrolution Taxed 1050	1.04	2.8
GPPS	Styron™ 660	1.04	7.0
GPPS	EDISTIR® N3560	1.05	3.8
GPPS	EDISTIR® N3840	1.05	10
HIPS	STYRON™ C-TECH	1.04	6.3
HIPS	Styrolution PS 476L	1.05	5.5
HIPS	EDISTIR® R850E	1.04	4

ANNEX 2 – MFI RANGE FOR CONTROL SAMPLE (OPTION 2)

MFI of PS grade in innovation (g/10min, 190 °C, 2.16 kg)	**Range of MFI accepted for control selection** (g/10min, 190 °C, 2.16 kg)
<4	± 1 compared to PS present in innovation
Between 4 and 8.5	± 2 compared to PS present in innovation
Between 8.5 and 15	± 3 compared to PS present in innovation
>15	± 5 compared to PS present in innovation

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