RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR HDPE CONTAINERS

STANDARD LABORATORY PRACTICE

VERSION 4.0

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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 HDPE container (or HDPE 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	Thermo Gravimetrical Analysis
Virgin Material	HDPE 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 aiming at enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Recyclability Evaluation Protocols will 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 for download in 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 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, is the decision-maker about the compatibility of the innovation with recycling according to the results of the evaluation, granting a technology or product 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 in the *RecyClass website."*

RecyClass

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "RecyClass[1] Recyclability Evaluation Protocol for HDPE 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 HDPE recycling stream. 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 Technology & Product 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 HDPE market. The overall goal is to maintain the protection of packaged goods and their marketing display functions without obstructing the proper functioning of the HDPE recycling process.

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

HDPE terminology as it is used in this document, refers to rigid plastic containers predominantly used for packaging liquids, cosmetics and detergents, as well as food contact applications.

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 HDPE. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for natural and coloured HDPE containers[5] in order to confirm that the HDPE innovation is compatible with these requirements.

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

1. HDPE resins
2. Barrier materials
3. Mineral fillers and additives that increase the density of the HDPE packaging
4. Non-PE closure systems and lidding films
5. Non-PE liners, seals, and valves
6. Non-PE labels and sleeves
7. Adhesives
8. Printing and inks

Following RecyClass methodology[6], packaging containing non-removable aluminium, metal, silicone, degradable plastics, black carbon surface, as well as PVC and PVDC layers are considered as disqualified for 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 current HDPE recycling works at an industrial scale. RecyClass HDPE 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 HDPE 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 leftover 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 HDPE 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 which will interpret the final results. Any remarks during the laboratory tests described in the Protocol shall be also noted down.

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

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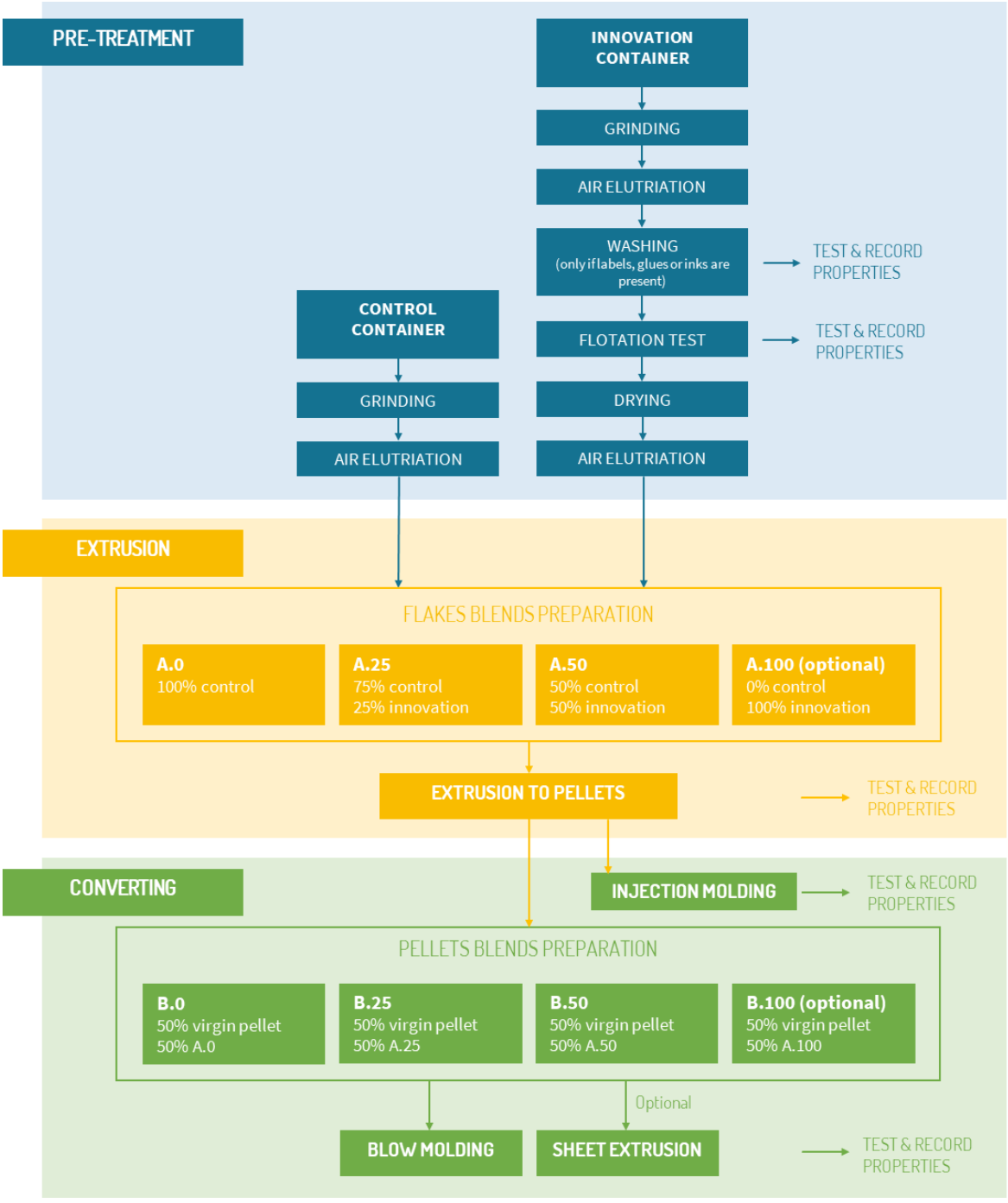
5 Design for Recycling Guidelines

[6] RecyClass Methodology

Figure 1: Methodology Diagram



4.1 CONTROL SAMPLE SELECTION

The control HDPE for use following the Protocol can be selected by:

- **Option 1:** If there is an HDPE container known to be recyclable, consisting of the same base HDPE 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 HDPE TC.

- **Option 2:** The Applicant can select an HDPE resin with the same critical technical specifications for MFI and density as the innovation article, ±0.02 MFI and ±0.005 density can be used as the control for this Protocol, with/upon the approval of RecyClass HDPE 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 …) 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 each. More innovation material could be requested if optional tests are required by the RecyClass HDPE Technical Committee. 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 discriminate the impact of a specific innovation on the recyclability of HDPE containers. Note that full packaging (with labels, decoration, closures, …) can also be assessed according to the present protocol.

4.2 VIRGIN SAMPLE SELECTION

The virgin HDPE sample to be used in this Protocol can be selected from the HDPE 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 submitted under the shape 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:

- Record the weight of the samples.

- Grind separately control and innovation sample to flakes of 3 to 15 mm.

- Store in separate containers.

- Recording the weight losses and average flake sizes.

5.1.2 AIR ELUTRIATION

Control (if provided as container) and innovation HDPE flakes are separately elutriated with air to remove light fraction.

Procedure:

- Elutriate with air with one pass and with less than 2% loss set for the control flakes. More innovation failures may occur if this step is omitted.

5.1.3 WASHING

At the state of the art, European HDPE 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 a following food contact bottle-making process. The RecyClass HDPE Technical Committee representatives are requested to select a washing procedure based upon the intended end-use application. Both the procedures take care of labels, adhesives, coatings, paper and printing present in the innovation HDPE container. If none of those are present, go directly to step 5.1.4. 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 (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 20l water) at 1.000 rpm for 5 minutes.

- 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 wash and rinse waters 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 the 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[7].

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.

[7] RecyClass Quick Test Procedures

- With stirrer on add HDPE 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 the 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.

5.1.4 FLOTATION TEST

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

- Combinations of polyethylene and other materials that float in water should be avoided in order to minimize the risk of contamination. In the case not 100% of the flakes would float, separation efficiency will be determined on the basis of the innovative packaging composition.
- Non-PE components floating together with HDPE flakes should be avoided as they cannot be further separated and are extruded with HDPE. This non-PE 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 (5 kg washed flakes vs 30 l water).

- Put each sample separately in the water and stir at 500 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.

- Take photos of the floating and sinking fractions separately.

- Save the water for visual evaluation.

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 photos 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

5.1.5 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 previous notification and approval from RecyClass.

5.1.6 AIR ELUTRIATION

Innovation HDPE flakes are elutriated with air to remove the 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 HDPE 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 h 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 at the same conditions with a desiccant bed drying unit or with hot air and extruded using co-rotative twin-screw extruders at a melt temperature of 220 °C. The extrudate will be melt filtered (about 120 microns 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 has to be also 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 90 °C for 1 hour.

- Extrude first the 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.

- 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 collect 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 properties' results 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 RecyClass 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 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 HDPE Technical Committee.

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 pelletization 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 HDPE.

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

Procedure:

- Dry the control sample before to be extruded with a bed desiccant for 1 hour at 80 °C or with hot air hot air at 90 °C for 1 hours.

- Extrude the sample at a melt temperature of 220 ± 5 °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

Table 3: Pellet properties evaluation

ASSESSMENT	RESULT	STANDARD	BENCHMARK RECOMMENDATION
Bulk Density (kg/m^3)		Annex B of EN 15344	Superior to 550 kg/m^3
Density (kg/m^3)		ISO 1183-1	Between 0,941 and 0,970 kg/m^3
Melt Index (g/10 min)		ISO 1133 (190 °C/2,16kg)	Between 0,2 and 0,9 g/10min
Ash content (wt%)		ISO 3451-1 (muffle) or ISO 11358 (TGA)	A.50 lower than 2 wt%
Filtration (µm)		Visual inspection	No build-up on screen
Moisture (wt%)		Moisture analyser	Record
Melt Temperature (°C)		ISO 11357-3 (Heat-cool-heat cycle at 10°/min from 25°C to 240°C with 1 minutes of isotherm between each ramp)	Melt Temperature second heating < 140° C
Impurities		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (wt%)		10 g air-dried pellets exposed to 160°C for 10 minutes	±0,1 wt% for A.25 and A.50 respect to A.0
PP (%)		Differential Scanning Calorimetry or Spectroscopic measurement via FTIR	No more than 2% for A.25 and A.50
Average Pressure (MPa)		Average Pressure after extruding through 120 microns 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)		($\Delta P_{25\text{-}30\ minutes}$ - $\Delta P_{5\ first\ minutes}$)	No increase higher than 25% compared to start in 30 min
Extrusion process		Unusual sticking, fumes, odour, and any build-up	Record

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 (optionally 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 run time is variable, but should be not less than 30 min. 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 by 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 heat stability and the injection pressure.

Record properties' results 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

Table 4: Injection moulded parts properties evaluation

ASSESSMENT	RESULT	STANDARD	BENCHMARK RECOMMENDATION
Flexural Modulus (MPa)		ISO 178	No more than 25% delta decrease to A.0
Tensile Stress at Yield (MPa)		ISO 527	
Tensile Stress at Break (MPa)		ISO 527-2	
Elongation at Break (%)		ISO 527-2	
Charpy Impact Strength (kJ/m^2)		ISO 179-2/1eA	
Reflection Colour		(L*, a*, b*) and ΔE	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 CONVERTING

Since this Protocol aims to assess the highest value recyclate application, blow moulding will be a priority as converting process. However, the RecyClass HDPE Technical Committee could decide to test the innovation for sheet extrusion. 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 HDPE pellets have been produced and tested, three 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 for 10 minutes at 60°C. Then according to the values reported in following 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 also to 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 HDPE 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 BOTTLES BLOW MOULDING

The Applicant has to submit its innovation primarily to blow moulding to test the recyclate obtained by the innovation in a closed-loop application, i.e. a bottle-to-bottle process.

Control blend B.0 has to be moulded first.

Table 6: Bottle 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:

- The samples B.0, B.25 and B.50 (optionally B.100) should be blow moulded at 170-180 °C into one litre straight-wall generic base monolayer 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 motor oil bottles.

    - Bottle height should be typically for one litre motor oil bottle.

    - Neck may be offset

    - The bottle must weigh 50± 5 grams.

- 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 properties' results in Table 7. 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 BOTTLE PROPERTIES EVALUATION

Table 7: Bottle properties evaluation

ASSESSMENT	RESULTS	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	±5% 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

5.3.4 SHEET EXTRUSION (OPTIONAL)

On the base of results obtained by pellet characterization, the RecyClass HDPE Technical Committee 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 also to 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. See more information in Table 9.

Table 9: Sheet production purpose & overview

PELLET COMPOSITIONS	KG OF BLEND REQUIRED	PURPOSE OF BLEND
C.0 50% A.0 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	All tests compared to control values
C.25 50% A.25 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values

C.50 50% A.50 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL **C.100** 50% A.100 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Optional, to evaluate the impact of higher concentration of innovation on recycling.

Procedure:

- Dry samples C.0, C.25 and C.50 (optionally C.100) at 60°C for 10 minutes.

- Extrude sheets at a melt temperature of 220 ± 5°C and with a thickness of 800 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 properties' results in Table 10. If some operating conditions have to be modified for C.25 and C.50 samples, this information must be documented in the report.

5.3.4.1 SHEET PROPERTIES EVALUATION

Table 10: Sheet properties evaluation

ASSESSMENT	RESULTS	STANDARDS	BENCHMARK RECOMMENDATION
Flexural Modulus (MPa)		ISO 178	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*

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DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	July 2019	Recyclability Evaluation Protocol for HDPE 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	Modification 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

ANNEX I – CONTROL SAMPLES SELECTION

HDPE RESINS	DENSITY, g/cm³	MFI at 190° C / 2,16 kg, g/10min
ENI Versalis Eraclene BC82	0,954	0,25
Chevron Phillips Marlex® HHM 5502BN	0,955	0,35
Dow UNIVAL™ DMDA-6230 NT 7	0,949	0,25
INEOS Rigidex® HD5502S	0,954	0,20
INEOS Rigidex® HD5802BM	0,958	0,30
LYONDELLBASELL Hostalen GF4750	0,950	0,40
REPSOL Alcudia® 5503	0,955	0,25

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