RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR HDPE CONTAINERS

STANDARD LABORATORY PRACTICE

VERSION 2.0

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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 with 100% control pellets
B.25	bottle with 87,5/12,5 control/innovation pellets
B.50	bottle with 75/25 control/innovation pellets
B.100	bottle with 50/50 control/innovation pellets
C.0	sheet with 100% control pellets
C.25	sheet with 87,5/12,5 control/innovation pellets
C.50	sheet with 75/25 control/innovation pellets
C.100	sheet with 50/50 control/innovation pellets
D.0	plate with 100% control pellets
D.25	plate with 75/25 control/innovation pellets
D.50	plate with 50/50 control/innovation pellets
D.100	plate with 100% innovation pellets
EN	European Standard
Innovation:	new container, flakes or pellets from innovative container which has to be tested
ISO	International Organization for Standardization
MFI	Melt Flow Index
PE	Polyethylene
PP	Polypropylene
PVC	Poly Vinyl Chloride
TC	Technical Committee
TGA	Thermo Gravimetrical Analysis

DISCLAIMER

"RecyClass is an initiative aiming at enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Plastics 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 PRE and RecyClass websites. 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.**

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and must be conducted by an independent laboratory approved 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 "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[1] Internal Procedures".

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[2] 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 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[3] 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
5. Non-PE liners, seals, and valves
6. Non-PE labels and sleeves
7. Adhesives
8. Inks

Packaging containing aluminium, metal, foam, degradable plastics, black carbon surface, as well as PVC shall be separately considered by the RecyClass HDPE Technical Committee in order to assess their suitability 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 an additional 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 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 a small scale to determine the suitability of an innovation material 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 Lab Evaluation Report compiling all the results obtained shall be prepared to report to the RecyClass Technical Committee which will interpret the final results. Any remarks during following 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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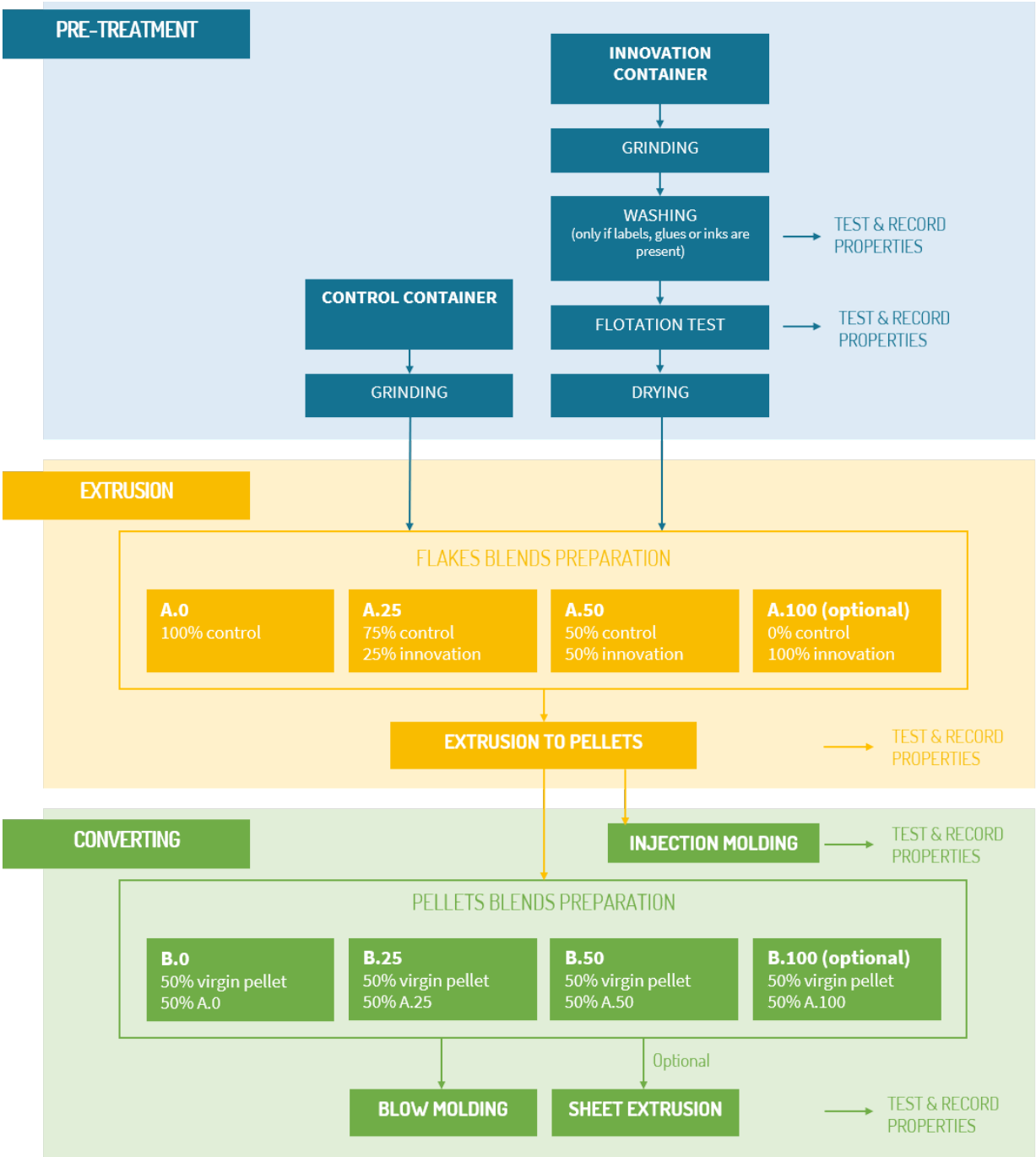
3 Design for Recycling Guidelines https://recyclass.eu/recyclass/design-for-recycling-guidelines/

Figure1: Methodology Diagram



4.1 CONTROL SELECTION

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

- **Option 1:** If there is a HDPE container on the market, similar to the innovation and is known to be recyclable, it can be selected as the control for this Protocol, with/upon the approval of the RecyClass HDPE TC.

- **Option 2:** 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 3:** The Applicant can select a 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.

These options are to be used to make both the control flakes and the blends with innovation container flakes that will contain the additive, coating, label, adhesive or multilayer resin for the recycle study.

A selection of control samples to be used is reported in Annex I.

For the purpose of the tests the Applicant should provide at least 10 kg amount of innovation material (as packaging) and 25 kg amount of control material (as packaging) which allows for blend preparations of 5 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 innovation to be tested is not limited to the main body of the packaging but to all its parts. Therefore, the innovation has to be submitted to the laboratory procedures with labels, adhesives, closure system, liners, seals, valves. If it can be correctly argued that labels and adhesives have no impact on the innovation, the innovation samples can be processed without the presence of labels and adhesives.

4.2 PRE-TREATMENT STEPS

4.2.1 GRINDING

Control and innovation HDPE containers are separately grinded in order to fit the throat of a standard laboratory extruder.

Procedure:

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

- Store in separate containers.

- Recording the masses and running times.

4.2.2 AIR ELUTRIATION

Control 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.

4.2.3 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 few recycling lines the washing is operated with more aggressive conditions (Procedure 2) aiming to a following food contact bottle-making process. The RecyClass HDPE Technical Committee and the Applicant 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 4.2.4.

The following procedures have to be utilized for both control and innovation samples, separately.

Procedure 1:

- Prepare the wash 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 wash 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 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.

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%w Triton X-100 and 1% caustic soda (NaOH)). Triton X-100 must be dissolved in cold water before the addition of caustic soda.

- Heat the solution at 90°C on a plate covering the vessel to minimize evaporation.

- Overhead stirrer at 1000 rpm, 2.5 cm above the bottom.

- With stirrer on add 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 90°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 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 fibers 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.

4.2.4 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, packaging design with combinations of polyethylene and other materials that sink in water should be avoided. Non-PE components floating together with HDPE flakes cannot be further separated and are extruded with HDPE. This poses relevant concerns both in the process operations and in the quality of the recyclate, undermining its applications such as containers, pipes and sheets.

The following procedure has to be utilized for both control and innovation samples, separately.

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 wash 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. Repeat the procedure for washed and dried innovation flakes, with and without caps and labels.

Procedure:

- Fill a 1 l graduated beaker with 700 ml of tap water (pH between 7 and 8).

- Boil the water for 10 minutes, and then cool at room temperature.

- Transfer 300 ml of water in a graduated beaker.

- Put the innovative sample in the water and stir at 500 rpm for 2 minutes.

- Stop the magnetic stirrer and allow the water to rest for 2 minutes.

- Take photo of the beaker.

- Remove all particles that float at the surface with a sieve.

- Take photos of the floating and sinking fractions separately.

- Save the wash for visual evaluation.

- Dry the floating fraction for 1 hours 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 (weight of sinking fraction) / (weight of innovative sample) x 100 (in %), separately for the innovation samples with and without caps and labels.

4.2.5 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. Stop the time.

4.2.6 AIR ELUTRIATION

Control and innovation HDPE 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. EXTRUSION

5.1 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 below.

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, e.g. when the estimated market share is higher than 10% (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. The laboratory carrying out the Protocol testing can define the amounts according to their best knowledge.

5.2 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 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).

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

5.3 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 at temperatures 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. 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 lab requirement for a 30-minute run time	All tests compared to control values
A.25 75% control with 25% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
A.50 50% control with 50% innovation	Per lab requirement for a 30-minute run time	Required for comparison to control values
OPTIONAL **A.100** 100% innovation	Per lab 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 with a bed desiccant for 1 hour at 80 °C or with hot air at 90 °C for 1 hour.

- Extrude for first the sample A.0 (the control blend) at a temperature of 220 °C and with a 120 µm melt filter pack, for no less than 30 minutes.

- 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 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.

5.3.1 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 temperature of 220 °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.

5.3.2 PELLET PROPERTIES EVALUATION

Table 3: Pellet properties evaluation

ASSESSMENT	RESULT	STANDARD	BENCHMARK RECOMMENDATION
Bulk Density (kg/m^3)		Annex B of EN 15344	No less than 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 (%)		ISO 3451-1 (muffle) or ISO 11358 (TGA)	A.50 lower than 2%
Filtration (µm)		Visual inspection	No build-up on screen
Moisture (% weight)		Moisture analyser	Record
Differential Scanning Calorimetry (°C)		ISO 11357-3	Melting point 130-140° C
Impurities		Visual inspection	Record
Surface appearance		Visual inspection	Record
Volatiles (%)		10 g air-dried pellets exposed to 160°C for 10 minutes	±0,1% for A.25 and A.50 respect to A.0
PP (%)		Differential Scanning Calorimetry or optionally Spectroscopic measurement via FTIR microscope in case of doubt	No more than 2% for A.25 and A.50
Delta Pressure (MPa)		Less than 25% higher Δ pressure after extruding through 110 microns filter for the stable 15 minutes run time, compared to 100% control	No build-up on screen and no more than 25% delta to control
Extrusion process		Unusual sticking, fumes, odour, and any build-up	Record

5.3.3 INJECTION MOULDING

Pellets A.0, A.25 and A.50 (optionally A.100) have to be tested for injection moulding to evaluate tensile properties, colours, as well as defects.

Control pellets A.0 have to be moulded first.

Procedure:

- Dry the samples A.0, A.25 and A.50 (optional: A.100) at 90 °C for 2 hours.

- Mould sample A.0 at 190-245 °C to multipurpose specimens' type 1A according to EN ISO 527-2 and to plates with measures of about 60 x 60 x 2 mm^3.

- The 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. If some operating conditions have to be modified for A.25 and A.50 samples, this information must be documented in the report.

5.3.4 PROPERTIES EVALUATION

Table 4: Injected moulded parts properties evaluation

ASSESSMENT	RESULT	STANDARD	BENCHMARK RECOMMENDATION
Flexural Modulus		ISO 178	Minimum 600 N/mm^2
Tensile Strength at Yield		ISO 527	Minimum 18 N/mm^2
Tensile Stress at Break (MPa)		ISO 527-2	No more than -25% delta to control
Elongation at Break (%)		ISO 527-2	
Charpy impact test strength		ISO 179-2/1eA	
Charpy impact test energy		ISO 179-2/1eA	
Reflection Colour		(L*, b*, a*)	Record
Gloss		ASTM D2457	Record
Surface appearance		Visual inspection	No black specks
Inclusions of extraneous material		Visual inspection	Record

Mechanical data must be analysed on the 1A specimen.

Colour, inclusions, and surface should be analysed on the plate.

6. CONVERSION

Based on the obtained results, the RecyClass HDPE Technical Committee will decide if the innovation presents some critical properties. On that basis, the Technical Committee reserves the right to further test the innovation. Otherwise, if the results are aligned with HDPE recyclate specimens the Technical Committee and the Applicant will define the way to further test the innovation on the base of the main applications available on the market.

Since the Protocol aims to assess the highest value recyclate application, blow moulding will be a priority. However, the RecyClass HDPE Technical Committee according with the Applicant 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.

6.1 PELLET BLENDS PREPARATION

Once HDPE pellets have been produced and tested, three additional blends of at 50% virgin – 50% blend A shall be produced for converting tests. Keep separated the pellet samples previously produced and dry it 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 100% innovation (i.e. B.100, by replacing the test with B.25).

6.2 PELLET BLENDS COMPOSITION

Three different blends at 50% virgin pellet – 50% Blend A shall be produced as described in table 4. Blends will be composed of 0%, 12,5% and 25% content (end eventually 50%) by weight of the initial innovation 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

6.3 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 lab requirement for a 30-minute run time	All tests compared to control values
B.25 50% A.25 pellet and 50% Virgin pellet	Per lab requirement for a 30-minute run time	Required for comparison to control values
B.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 **B.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:

- The samples B.0, B.25 and B.50 should be blow moulded at 170-180 °C into one litre straight-wall generic base monolayer bottle, 1 mm tick.

- The cross section can be rectangular or square.

- The bottom corners should have radii as small as commercial motor oil bottles.

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

- Neck may be offset.

- The bottle must weigh 50 ± 5 grams.

- Samples B.25 and B.50 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.

6.3.1 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	Minimum of 10 bottles (compare with B.0)
Reflection Colours		L*, a*, b*	Record
Gloss		ASTM D2457	Record
Bottle Dimension		Height	±5% respect to B.0
Bottle Weight		Weight	±5% respect to B.0
Bottle Capacity		Brim-full	±5% respect to B.0
Thickness		Top, mid, and bottom side wall, shoulder, base corner	Minimum 0,3mm for each measure
Top load		ASTM D2659 (no ISO available)	<10% decrease respect to B.0
Drop impact		ASTM D2463, procedure B (no ISO available)	<10% decrease respect to B.0
Flexural Modulus		ISO 178	Minimum 600 N/mm^2
Additional observation		Deposit on tooling	None observed respect to B.0 for 2 hours bottle production

6.3.2 TENSILE PROPERTIES TESTING

Tensile properties have to be tested for bottle application to minimize the influence of a subsequent processing and obtain a more realistic comparison.

Table 8: Bottle tensile properties evaluation

ASSESSMENT	RESULTS	STANDARDS	BENCHMARK RECOMMENDATION
Stress at Yield (MPa)		ISO 527-2	No more than -25% delta to control
Stress at Break (MPa)		ISO 527-2	
Elongation at Break (%)		ISO 527-2	
Elongation at Yield (%)		ISO 527-2	
Strength (MPa)		ISO 527-2	
Elongation at Strength (%)		ISO 527-2	

6.4 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 6.1, 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.

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 at 60°C for 10 minutes.

- Extrude sheets with thickness of 800 µm under conditions determined for the control sample C.0.

- Extrusion run time per variable, no less than 30 minutes.

- Samples C.25 and C.50 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.

6.4.1 SHEET PROPERTIES EVALUATION

Table 10: Sheet properties evaluation

ASSESSMENT	RESULTS	STANDARDS	BENCHMARK RECOMMENDATION
Flexural Modulus		ISO 178	Minimum 600 N/mm^2
Tensile Strength at Yield		ISO 527	Minimum 18 N/mm^3
Tensile Stress at Break (MPa)		ISO 527-2	No more than -25% delta to control
Elongation at break (%)		ISO 527-2	
Colour		Visual inspection	No discolouration
Surface Appearance		Visual inspection	No black specks
Inclusions of extraneous material		Visual inspection	Record
Colour		L*, a*, b*	Record
Gloss		ASTM D2457	Record

ANNEX I – CONTROL SAMPLES SELECTION

HDPE RESINS	DENSITY, g/cm³	MFI at 190° C / 2,16 kg, g/10min	ESCR, h
ENI Versalis Eraclene BC82	0,954	0,25	> 60
Chevron Phillips Marlex® HHM 5502BN	0,955	0,35	45
Dow UNIVAL™ DMDA-6230 NT 7	0,949	0,25	180
INEOS Rigidex® HD5502S	0,954	0,20	6
INEOS Rigidex® HD5802BM	0,958	0,30	> 200
LYONDELLBASELL Hostalen GF4750	0,950	0,40	High
REPSOL Alcudia® 5503	0,955	0,25	100

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