RECYCLABILITY EVALUATION PROTOCOL

FOR LABELS & ADHESIVES ON HDPE CONTAINERS

STANDARD LABORATORY PRACTICE REP-HDPE-02

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DISCLAIMER

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

The Recyclability Evaluation Protocols are freely available to download on the RecyClass website. Companies developing new plastic concepts are encouraged to use them to self-assess the impact of their solutions on recyclability and highlight potential issues. **However, compliance 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, will decide on the compatibility of the innovation with recycling according to the evaluation results, granting a Recyclability Approval Letter to the Applicant.

All tests must follow the Evaluation Protocols recommended by the RecyClass Technical Committees and be conducted by an independent laboratory recognised by RecyClass which has no legal affiliation to the applicant.

More information is reported in the RecyClass Internal Procedures available on the RecyClass website."

1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "Recyclability Evaluation Protocol for Labels & Adhesives on HDPE containers" referred to in this document as "The Protocol" describes the methodology that may be followed by the Applicant at a laboratory scale in order to determine if adhesive and label combinations are compatible with the post-consumer HDPE recycling stream.

Please note that for the rest of the Protocol, the terminology 'labels' refers to either label or sleeve, according to the material submitted.

The Protocol aims to evaluate the behaviour of labels and adhesives during the grinding, washing and floatation processes by performing a test at laboratory scale. The applicant shall proceed with the Protocol as established in the Assessment Process for Applicants of Recyclability Evaluation in the RecyClass Internal Procedures¹ and RecyClass Technology Approval Quality Management & Procedures document²

In case the combination of label and adhesive tested with the Protocol does not meet the requested assessment criteria, the sample can be submitted to the Recyclability Evaluation Protocols of RecyClass to demonstrate that, even though not releasable, the combination does not negatively affect HDPE recyclability in terms of process and recyclate quality. This option can only be considered for coloured packaging. For natural and white packaging decorations must be released to avoid a contamination of the material obtained after the recycling process.

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

Adhesive terminology as it is used in this document refers to adhesive for labels only.

¹<u>RecyClass Internal Procedures</u>

² RecyClass Technology Approval Quality Management & Procedures

2. SCOPE OF THE PROTOCOL

The scope of the testing Protocol covers any materials related to labels and adhesives applied to the HDPE packaging. Prior to initiating the evaluation, the Applicant is required to review the Design for Recycling Guidelines for natural and white HDPE containers³ in order to confirm that the material is compatible with these requirements.

The following non-exhaustive list of materials are covered by the scope of this Protocol:

- 1. Adhesives for labels (wet labelling adhesive, pressure sensitive adhesive, and non-pressure sensitive hotmelt)
- 2. Printed labels

The Protocol provides guidance on the behaviour of the labels, and adhesives during the HDPE recycling process. The label should detach from the packaging without leaving any adhesive residue on the washed packaging flakes. The adhesive behaviour is under investigation by RecyClass, and further recommendations and guidance will be given in the near future concerning the preferred behaviour for the adhesive. It should be noted that adhesive dissolving or dispersing in the water represents extra effort for the water treatment system. **Printed labels must be also tested according to the bleeding inks Protocol to evaluate inks behaviour.**

3. LABORATORY EQUIPMENT

- Laboratory grinding machine⁴ with the following suggested characteristics:
 - Screens from 15 to 20 mm.
 - \circ 3 rotating blades, 1 to 3 knives per blade.
 - o 2 fixed blades.
 - \circ ~ The rotating speed should be between 400 rpm and 650 rpm.
- Washing vessel/equipment preferably with deflectors (4 x 90°)
- Overhead stirrer with selectable rotation speed.
- Paddle stirrer.
- Analytical balance with an accuracy of 0.001 g.
- Strainer (mesh size of 0.5 x 0.5 mm).
- Moisture Analyzer (res. 0.001 %).
- Digital camera.
- Optical Microscope.

³ Design for Recycling Guidelines <u>https://recyclass.eu/recyclass/design-for-recycling-guidelines/</u>

⁴ Dry or wet grinding.

4. LABORATORY TEST PROCEDURE

This Protocol aims to reproduce the grinding, washing and floatation steps of the recycling process at a small scale to determine the suitability of an adhesive and label combination for the HDPE recycling stream. The Protocol described below shall be followed precisely and any modifications, remarks or problems must be noted in the report during the testing phase. A Laboratory Evaluation Report compiling all the results obtained shall be prepared.

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



Figure 1. Flow process diagram.

4.1 SAMPLES SELECTION

For the purpose of the tests, the Applicant should provide a number of containers, where the labelled area weighs at least 200 g, typically between 10 and 30. Containers need to be supplied clean and without closures.

The assessment can be performed in both finished or semi-finished packaging.

In the case of semi-finished packaging, it is required to use natural or white 1-liter, round bottles. RecyClass will provide standard bottles to the Applicants.

Applicants supplying the materials to be tested will need to provide them with the labels already applied and fully printed. Manual application is accepted. Labels must be fully printed with bright colours (yellow, cyan, magenta, black)

in order to facilitate the distinction between rigid and label flakes during the assessment. Labels must cover at least 70 % of the container surface.

A minimum time of 48 hours after application of the labels should be considered prior to shipping the samples to the testing facility. Once the materials have arrived at the testing facility, a storage time of 48 hours must be considered.

Storage conditions should be around 20 °C and a relative humidity of 50 %.

4.2 PROCEDURE

4.2.1 STEP 1: CALCULATION OF THE DIFFERENT FRACTIONS

Procedure:

- Take 5 specimens of clean, dry bottles.
- Weigh the labelled bottles. Record the weights and assign the code BLB to the average of the measurements. Take pictures.
- Cut the labelled parts of the bottle as close as possible from the side of the label(s). Weigh the label part(s) of the bottle (panels). Record the weights and assign the code B_{LP} to the average of the measurements. Take pictures.
- Weigh the bottles without panels and assign the code B_{WP}.
- Remove the label by hand from the 5 panels and clean them with a suitable solvent to remove the adhesive (e.g., Isopropanol). Dry the panels. Take pictures.
- Weigh the dry panels without labels. Record the weight and assign the code B_P to the average of the measurements. Take pictures.
- Calculate the weight corresponding to the label and adhesive as $B_{LP} B_P = B_{LA}$
- Calculate the following fractions:

Table 1. Fractions to be calculated.

Fraction of full bottle ($\chi_{B_{LB}}$)	$B_{LB}/B_{LB}=1$
Fraction of the labelled panels $(\chi_{B_{LP}})$	B _{LP} / B _{LB}
Fraction of the bottle without the labelled panels ($\chi_{B_{WP}}$)	Bwp/ Blb
Fraction of the labelled panel without the label (χ_{B_p})	Bp/ Blb
Fraction label ($\chi_{B_{LA}}$)	Bla/ Blb

4.2.2 STEP 2: GRINDING

Procedure:

- Labelled area should weigh around 200 g, which is equivalent to 10 to 30 bottles approximately (depending on the labelled area). Report as N the number of bottles to be ground. Please make sure N is a round number.
- Weigh the N bottles that are going to be ground. Assign the code G_0 .
- Grind sample B (bottles). Use preferably a screen size between 15 and 20 mm.
- Collect the ground fraction, assign it code B_G. Weigh the B_G fraction, assign the code G_T to the recorded weight. Take pictures.

- Calculate the losses B_L:

$$G_0 - G_T = B_{LG}$$

Losses % = $\frac{B_{LG}}{G_0} \times 100$

- If losses represent more than 5 %, Collect the materials losses from the grinder, fraction B_L . Weigh the B_L fraction and assign the code GL_T to the recorded weight. Take pictures.

4.2.3 STEP 3: CLASSIFICATION OF FLAKES AND GRINDING RELEASABILITY RATE CALCULATION

- Separate and quantify (weigh) the ground sample following the tables below.

Table 2. Quantification of the B_G fraction

Quantification of the B_G fraction		
Contaminated flakes and fines	G1	
(Container flakes with labels, container flakes with rest of adhesive, clogged flakes, fines)		
Label flakes	G ₂	
Clean container flakes	G ₃	
Total (Should be equal to G_T , with a tolerance of 0.2 %)		

- Losses should be lower than 5 %. If the losses are higher than 5 % after grinding, separate and quantify the losses:

Table 3. Quantification of the B_L fraction.

Quantification of the B_L fraction		
Contaminated flakes and fines	GL1	
(Container flakes with labels, rest of adhesives, clogged flakes, fines)		
Label flakes	GL2	
Clean container flakes	G _{L3}	
Total (Should be equal to G_{LT} , with a tolerance of 0.2 %)		

- Sum the weight of clean flakes: $M_{BG} = G_3 + L_3 + G_2 + GL_2$. In case G_{L3} and G_{L2} were not calculated because losses were below 5 %, consider only G_3 and G_2 .
- Calculate the label releasability rate of the grinding process:

$$\eta_{BG} = \frac{M_{BG} - ((G_T + G_{LT}) \, x \, \boldsymbol{\chi}_{B_{WP}})}{(G_T + G_{LT}) x \, \boldsymbol{\chi}_{B_P} + (G_T + G_{LT}) x \, \boldsymbol{\chi}_{B_{LA}}} \, x \, 100$$

4.2.4 STEP 4: WASHING & FLOTATION

4.2.4.1 WASHING

At the state of the art, European HDPE recycling lines typically use cold washing conditions, no detergents nor strong chemicals. The following procedure must be applied to the Flakes.

Procedure:

- Keep all fractions together and operate the washing step.

- Prepare the washing solution, which is tap water, in a vessel at a 1:4 ratio of flakes:water. Determine the current pH-value of the water. Do not add detergents or caustic soda.

- In order to simulate better the friction involved in this process at industrial scale, it is suggested to use a vessel with deflectors to promote turbulence in the wash water $(4 \times 90^{\circ})$.

- Heat the washing solution to 40 °C.

- Wash the flakes by stirring at 1000 rpm for 5 min.

- Filter the wash with a strainer, collect the wash water and rinse the flakes with cold running tap water for 5 minutes under vigorous stirring with a manual stirring bar. Collect the rinse water.

- Determine the pH-value of the wash water. Be aware of the correct temperature for measuring. If necessary, first cool down the wash water to room temperature (20 – 23 °C).

- Observe the colour of the wash water solution. If any noticeable change of colour or transparency occurs, report it, and document the colour with a photograph. In order to highlight the differences, take a photograph of the solution in a beaker against a neutral background

4.2.4.2 FLOTATION (ONLY FOR PACKAGING WITH NON-PO COMPONENTS)

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 suitable recycling, packaging design with non-PO components, such as paper labels, floating together with polyolefins flakes must be avoided.

The following procedure must be utilized on the washed flakes to separate the container flakes from the paper labels.

Procedure:

- Fill a vessel with tap water at a 1:6 ratio.
- Put the flakes in the water and stir at 750 rpm for 2 min.
- Stop the stirrer and allow the water to rest for 2 min.
- 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.
- Use only the floating fraction to continue with STEP 5.

4.2.5 STEP 5: FLAKES DRYING

Reduce the flake moisture with ambient air to release surface moisture to less than 1 %.

Procedure:

- Dry the flakes collected with air at room temperature without the application of vacuum or heat sources until at least 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.

- Evaluate the moisture content with a moisture analyser.

- Record the moisture content.

4.2.6 STEP 6: CLASSIFICATION OF FLAKES AND WASHING RELEASABILITY RATE CALCULATION

Procedure:

- Weigh the flakes after drying (B_W fraction), assign letter W_T .
- Spread the flakes and classify them according to the following table.
- Sum the weight of clean flakes: $M_{BW} = W_3 + W_2$.
- -

Table 4. Flakes separation and quantification if the B_W fraction

Flakes separation and quantification of the B _w fraction		
Contaminated flakes and fines	W1	
(Container flakes with labels, rest of adhesives, clogged flakes, fines)		
Clean label flakes	W ₂	
Clean container flakes	W ₃	
Total (Should be equal to W_T , with a tolerance of 0.2 %)		

- Calculate the label releasability rate after the washing procedure.

$$\eta_{BW} = \frac{M_{BW} - (W_T \, x \, \chi_{B_{WP}})}{W_T \, x \, \chi_{B_P} + \, W_T \, x \, \chi_{B_{LA}}} \, x \, 100$$

- Calculate the total losses of the procedure as

$$G_0 - W_T = B_{LT}$$

Losses % = $\frac{B_{LT}}{G_0} \times 100$

Assessment criteria:

For filmic PO-based labels: The tested label and adhesive combination can be considered as "releasable" and pass the procedure if the label releasability rate after washing is higher than 90 %.

For paper labels:

- The removability rate must be 95 %

- No paper labels still attached to HDPE flakes in the floating fraction.

For released labels, even partially, the report should report the adhesive behaviour: water-soluble or releasable (i.e., remains on the label). Visual inspection of the dried label flakes and residual stickiness should confirm **whether** the adhesive remains on the label or get released into water.

The wash water should be observed, as reported in step 4. **Turbid** wash water may indicate **the adhesive, additives** or inks being only partially dissolved or dispersed in the wash water.

If the characterization of the adhesive's behaviour is not possible, the report must mention it.

4.2.7 STEP 7: FIBER LOSSES EVALUATION (ONLY FOR PAPER LABELS)

This section only applies to paper labels.

The last step is to verify the fibre losses that can occur during the washing step of paper labels. The following procedure must be performed for the wash water.

Procedure:

- Weigh the dried strainer of 190 $\mu m.$ Record the weight before filtering as "FW $_{\text{BF}}$ ".
- Filter the wash water collected step 4.
- Dry the strainer in the oven 1 h at 120 °C.
- Weigh the strainer. Record the weight after filtering as "FW_{AF}".
- Calculate the weight changes of the filter as "FW" (FW_{AF} FW_{BF}).
- Repeat the procedure with the rinse water.
- Weigh the dried strainer of 190 μ m. Record the weight before filtering as "FR_{BF}".
- Filter the wash water collected step 4.
- Dry the strainer in the oven 1 h at 120 °C.
- Weigh the strainer. Record the weight after filtering as "FRAF".
- Calculate the weight changes of the filter as "FR" ($FR_{AF} FR_{BF}$).

Assessment criteria: The tested paper label can be considered as "without fibre loss" and pass the procedure if all the following criteria are met:

- Fiber losses: FW/ $B_{LA} \times 100 < 4\%$ and FR/ $B_{LA} < 4\%$.

5. REPORT CONTENT

The report should contain the following information:

- Reference to the Procedure:

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- A full and complete identification of the material tested, including:
 - Label: size, structure, and density.
 - **Adhesive**: amount per surface unit and technology (wet labelling adhesive, pressure sensitive adhesive, or non-pressure sensitive hotmelt)
 - **Container**: main polymer, thickness of the walls, colour.
- Description and photographs of the set-up.
- Description of the samples during each step (especially on colour changes, haze, deposits, sinking or nondetached label fragments, residual stickiness).
- The photographs indicated in the test procedure. Additional photographs are welcome whenever useful for documenting specific situations.
- Details of any deviation from the test method, as well as any incident which may have influenced the results. Report the pH measurements.
- In case of released labels, characterization of the adhesive behaviour (water soluble or water releasable); even for partial label releasability (releasability rate > 0%).
- Test figures. Use the tables indicated throughout the report.

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
2.0	July 2025	Addition of paper labels to the Protocol
1.1	March 2025	Correction of the formula to calculate the label releasability Correction of wording and typos
1.0	July 2024	Release of the Recyclability Evaluation Protocol for Labels & Adhesives on HDPE Containers v 1.0

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