WASHING QUICK TEST PROCEDURE

FOR LABELS APPLIED ON PS CONTAINERS

STANDARD LABORATORY PRACTICE

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DISCLAIMER

"RecyClass is an initiative aiming at enhancing and evaluating the recyclability of plastic packaging through a technical perspective. The Plastics Recyclability Evaluation Protocols and Quick Test Procedures 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 and Quick Test Procedures 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.**

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.

The Quick Test Procedures can be either performed internally for R&D purposes or performed by external bodies such as independent laboratories or certification bodies to assess the results.

More information is reported in the RecyClass Internal Procedures available in the <u>RecyClass website.</u>"

1. INTRODUCTION AND PURPOSE OF THE PROCEDURE

The "Washing Quick Test Procedure for Labels applied on PS Containers" referred to in this document as "The Procedure" 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 PS recycling stream.

The Procedure aims to evaluate the behaviour of labels, sleeves, and adhesives during the washing process by performing a quick test at lab scale. The results of the quick test may not be considered as a formal approval by RecyClass. For a complete assessment, further tests are required to highlight all possible effects of adhesives and labels on the recyclability¹. The Recyclability Evaluation Protocols of RecyClass prevail over the other side-procedures, as the following washing procedure. Please contact RecyClass for more information on protocols for complete assessment².

In case the combination of label and adhesive tested with the Procedure does not meet the requested assessment criteria, the combination can be submitted to the Recyclability Evaluation Protocols of RecyClass to demonstrate that, even though not washable, the combination does not negatively affect PS recyclability in terms of process and recyclate quality.

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 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 Recyclability Evaluation Protocols & Quick Test Procedures: <u>https://recyclass.eu/recyclability/test-methods/</u>

2. SCOPE OF THE PROCEDURE

The scope of the Procedure covers any materials related to labels, sleeves and adhesives introduced to the existing PS packaging solutions. Prior to initiating the evaluation, the Applicant is required to review the Design for Recycling Guideline for PS containers³ in order to confirm that the material is compatible with these requirements.

The following materials are covered by the scope of this Procedure:

- 1. Adhesives for labels (wet labelling adhesive, pressure sensitive adhesive, and non-pressure sensitive hotmelt)
- 2. Unprinted labels (filmic & paper)
- 3. Printed labels (filmic & paper)
- 4. Unprinted sleeves
- 5. Printed sleeves

The procedure provides guidance on the behaviour of the labels, sleeves, and adhesives during the PS recycling process. The label or sleeve should detach from the packaging and with no adhesive remaining on the washed packaging flakes. The adhesive layer should preferably remain on the label; or be dissolved in the water. Adhesive dissolving in the water represents extra effort for water treatment. **Printed labels or sleeves must be tested additionally according to the bleeding inks procedure to evaluate the potential issue coming from the inks.**

Additionally, non-PS labels or sleeves must be separable by flotation from the PS containers to ensure no contamination of the recycling stream by another material than PS. Lastly, the paper labels must not lose fibres in the water (pulping phenomena).

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

3. LAB EQUIPMENT

- Accurate cutting device (scissors, blade, scalpel)
- Round beaker of 600 mL (diameter: height ratio of 1, wide form, 9 cm diameter)
- Paddle stirrer (30 mm x 15 mm and 2 rectangular paddles). See picture.
- Overhead stirrer with freely selectable rotation speed
- Dish detergent
- Sodium chloride (or another salt)
- Analytical balance with an accuracy of 0,0001 g
- Strainer (mesh size of 0,5 x 0,5 mm)
- Sieve



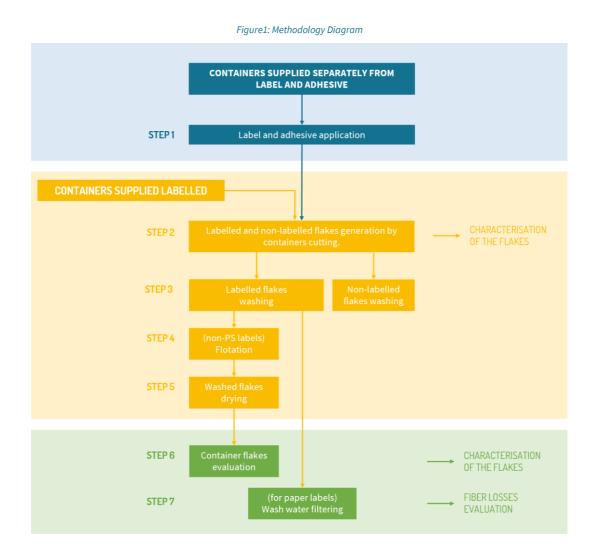
³ Design for Recycling Guidelines https://recyclass.eu/recyclass/design-for-recycling-guidelines/

- Moisture Analyzer (res. 0.001 %)
- Paint strainer 190 μm
- Oven

4. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the washing step of the recycling process at a small scale to determine the suitability of an adhesive and label combination 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 all the results obtained shall be prepared. Any remarks during following the Procedure shall be also noted down.

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



4.1 SAMPLES SELECTION

The control container for use following the Procedure must be the same container as the one with the applied label and adhesive. Two options are available:

- **Option 1:** If the container is provided without label and adhesive, the first step will consist of applying the label with the same way as the final packaging (as commercialized)⁴. Then the container will be cut into smaller parts according to each area. Area containing label will be used to obtain the labelled flakes; area without label will be used to obtain the control flakes.

- **Option 2:** If the label and adhesive are already applied on the container, the container will be cut into smaller parts according to each area. Area containing label will be used to obtain the labelled flakes; area without label will be used to obtain the control flakes.

For the purpose of the tests, the Applicant should provide at least 1kg of containers. In case of option 1, non-labelled containers must be provided with a sufficient amount of label and adhesive that cover the containers. Considering both options, if the label covers a large part of the container (>50%), a larger amount of containers may be needed to obtain a sufficient amount of control material from the parts without label. More labelled material may also be required if the label coverage is not sufficient to obtain the amount of labelled flakes and/or if additional tests are required.

4.2 PROCEDURE

4.2.1 STEP 1: LABELS & ADHESIVES APPLICATION (OPTIONAL)

Adhesive and label must be applied to the containers, with the same way as the final commercialized packaging.

Procedure:

- Report label size and structure, including the label stock and the amount of adhesive per surface unit. Document the label design with one or more photographs.

- Apply the adhesive and label to the containers, following the recommendations of the adhesive and label suppliers.
- If required, dry or cure the applied adhesive according to the recommendations of the adhesive supplier.
- Document labelled containers with one or more photographs.
- Store the labelled containers for a minimum of 72 hours.

4.2.2 STEP 2: CONTAINERS CUTTING & FLAKES CHARACTERIZATION

Once the containers have been stored for at least 72 hours, the labelled flakes and the control flakes must be generated.

⁴ In the case of hotmelt type labelling, please note that application by the lab may not be representative if the labels are applied by hand without hotmelt applicator.

Procedure:

- Cut out with scissors the part of the containers where the label is applied. Avoid generating fines. Flakes should be shaped approximately as squares with an edge length between 10-12mm. These flakes will constitute the labelled sample.

- Cut out with scissors the part of the containers where no label is applied. Avoid generating fines. Flake should be approximately as squares with an edge length between 10-12mm. These flakes will constitute the control sample.

- A minimum of 50 flakes for each labelled and control samples must be generated. Labelled flakes must be cut in different locations of the labelled area and cut from different containers. Record the total weight of the labelled flakes as "A".

- Document the labelled and control flakes with one or more photographs.

- Count and record as "B" the number of labelled flakes.

4.2.3 STEP 3: WASHING

At the state of the art, European PS recycling lines typically use cold washing conditions, no detergents nor strong chemicals. The following procedure must be applied to both control and labelled flakes, separately.

Procedure:

- Prepare the washing solution, that is tap water, in a beaker (50 flakes in 200 mL water in a 600 mL beaker). Determine the current pH-value of the water. Do not add detergents or caustic soda.

- Heat the washing solution to 40 °C.

- Wash each sample separately by stirring at 1.000 rpm for 5 min with a paddle stirrer.

- 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.

- 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 two wash water solutions. 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 both solutions in the beaker alongside each other in front of a light (white) and dark (black paper) background.

4.2.4 STEP 4: FLOTATION (FOR NON-PS LABELS)

For non-PS label material (including filmic & paper labels): Following the washing, the flotation process allows flake separation by density as occurring in the float/sink tank used in an industrial recycling line. The following procedure must be applied to both control and labelled flakes, separately.

Procedure: step 1

- Prepare the flotation solution, that is tap water, in a beaker (50 flakes in 300 mL water in a 600 mL beaker). Boil the water for 10 minutes, and then cool at room temperature.

- 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 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.

Procedure: step 2

- Prepare the flotation solution, that is tap water, in a beaker (50 flakes in 300 mL water in a 600 mL beaker). Boil the water for 10 minutes, and then cool at room temperature.

- Add a drop of dish detergent and add 12% of sodium chloride to the water solution (or any other salt) to increase the water density up to 1.08 g/cm³.

- 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 2 minutes.
- Take photo of the beaker.
- Recover 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.

Observe the colour of the wash water solutions of both steps. 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 both solutions in the beaker alongside each other in front of a light (white) and dark (black paper) background.

4.2.5 STEP 5: DRYING

Labelled flakes: The last step before the assessment is to reduce the flakes moisture content to less than 1 wt%.

Procedure:

- Dry (1) the flakes collected after washing step in the case of PS label material or (2) the floating flakes collected after STEP 2 of the flotation procedure, with air at room temperature for 24 h, without the application of vacuum or heat sources in a room with forced air ventilation at 22 °C and 50 % r.h. If it is not possible to control the humidity, then dry the flakes at 22°C for 96 h without the application of vacuum or heat sources in a room with forced air ventilation.

- Evaluate the moisture content with a moisture analyser.

- Store the dried flakes for 24 h.

4.2.6 STEP 6: FLAKES SEPARATION & ASSESSMENT

Labelled flakes: Evaluate the efficiency of adhesive and label removal.

Procedure:

- Separate the container flakes from the label flakes (if not separated by flotation). Count the container flakes to check that the numbers match with the count after cutting, i.e., no flake has been lost in the process.

- Qualitatively inspect the container flakes for residual stickiness from the adhesive. If any flakes still have attached label pieces, document them with a photograph. Evaluate the stickiness by touch and report the average level of the flake's stickiness: not sticky (0); slightly sticky (1); or sticky (2).

- Quantitatively inspect the container flakes:

- Count the number of flakes without residual stickiness of the adhesive nor any residual label. Report the total number of these clean flakes as "C".
- Count the number of flakes with residual stickiness of the adhesives and/or with residual label attached. Report the total number as of unclean flakes as "D".

- Document the flakes (with and without residual labels) with one or more photographs.

- Calculate the efficiency of the adhesive and label removal as follow:

Removal efficiency =
$$(\frac{C}{B}) * 100$$

For removed 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 3. **Turbid** wash water may indicate **the adhesive 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.

Non-PS label material: Evaluate the efficiency of separation by density with PS flakes.

Procedure:

- Separate the container flakes from the non-PS label flakes (not separated by flotation).
- Weight the container flakes, and report as "E".
- Weight the remaining non-PS labels flakes, and report as "F".
- Document the residual non-PS labels with one or more photographs.
- Calculate the efficiency of the separation of non-PS label as follow:

Separation efficiency =
$$(\frac{A - E - F}{A - E}) * 100$$

Assessment criteria: The tested label and adhesive combination can be considered as "removable" and pass the procedure if:

- for PS label materials the removal efficiency is 90% for filmic PS labels

- for any other label materials (PE, PP, paper, others) the removal efficiency is 100% and separation efficiency after the flotation step is 100%.

4.2.7 STEP 7: WATER FILTERING & FIBER LOSSES EVALUATION (FOR PAPER LABELS)

For paper label material only: 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 of labelled flakes.

Procedure:

- Weigh the dried paint strainer 190 $\mu m.$ Record the weight before filtering as "G".
- Filter the wash water collected step 3.
- Dry the paint strainer in the oven 1 h at 120 °C.
- Weigh the paint strainer. Record the weight after filtering as "H".
- Calculate the weight changes of the filter as "I" (H G).

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

- no measurable weight changes: I = 0,00 g

- no naked-eye visual residue on the filters

- no noticeable wash water and rinse water colouration

5. REPORT CONTENT

The report should contain the following information:

- Reference to the Procedure: Washing Quick Test Procedure for Labels Applied on PS Containers - v. 1.0

- 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)

- Description and photographs of the set-up.

- Description of the samples during each step (especially on colour changes, haze, deposits, sinking or non-detached 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 removed labels, characterization of the adhesive behaviour (water soluble or water releasable); even for partial label washability (removal efficiency > 0%).

- Test figures. Use the table 1 below as reference.

Table 1: Washing results

VARIABLE	VALUE	REFERENCE IN THE PROCEDURE
Total weight of the labelled flakes after cutting	X,xx g	A
Total number of labelled flakes after cutting	XX	В
Total number of clean flakes after drying	XX	С
Total number of unclean flakes after drying	XX	D
Removal efficiency	XX%	
Average level of flakes stickiness	0/1/2	
(for non-PS labels) Weight of PS container flakes	X,xx g	E
(for non-PS labels) Weight of label flakes	X,xx g	F
(for non-PS labels) Separation efficiency	XX%	
(for paper labels) Filter weight before filtration	X,xx g	G
(for paper labels) Filter weight after filtration	X,xx g	Н
(for paper labels) Weight changes of the filter	X,xx g	1

DOCUMENT VERSION HISTORY

VERSIC	N PUBLICATION DATE	REVISION NOTES
1.0	May 2022	Washing QT procedure for film labels applied on PS containers released

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