RecyClass

# RECYCLABILITY EVALUATION PROTOCOL

## FOR LABELS & ADHESIVES ON PET BOTTLES

STANDARD LABORATORY PRACTICE

REP-PETbot-02

VERSION 1.0

PUBLISHED ON JANUARY 2024

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# CONTENT

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# DISCLAIMER

"RecyClass is an initiative aiming on 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 with a Recyclability Evaluation Protocol is not a replacement for an official assessment and may not be used as a marketing tool.** The RecyClass Steering Board, following the recommendations of the Technical Committees, is the decision-maker regarding the compatibility of the innovation with recycling according to the results of the evaluation, granting Recyclability Approval Letter to the Applicant.

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

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

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# 1. INTRODUCTION AND PURPOSE OF THE PROTOCOL

The "Recyclability Evaluation Protocol for Adhesives & Labels on PET Bottles" 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 label and adhesive combinations are compatible with the post-consumer PET bottle recycling streams, meaning transparent clear/light blue, transparent coloured and opaque coloured PET streams. 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[1] and "RecyClass Recyclability Approval Quality Management & Procedures document[2]

The Protocol aims to evaluate the behaviour of labels, and adhesives during the washing process by performing a quick test at laboratory scale. It aims to guarantee recyclability[3] of plastics packaging while encouraging innovation in the label & adhesives market used for PET bottle applications. The overall goal is to ensure the removability of the label/attachment of the PET bottle without obstructing the proper functioning of the PET bottles recycling process. RecyClass protocol targets benchmarks that are based on requirements for bottle-to-bottle closed loop applications.

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

PET bottle terminology, as it is used in this document, is defined as a rigid plastic bottle predominantly used for packaging liquids, beverages and detergents or cosmetics.

[1] *RecyClass Internal Procedures*

[2] *RecyClass Technology & Product Approval Quality Management & Procedures*

[3] *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.*

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# 2. SCOPE OF THE PROCEDURE

The scope of the Protocol covers any labels, adhesives, attachments introduced to the existing packaging solutions for PET bottles. Prior to initiating the evaluation, the applicant shall review the Design for Recycling Guidelines for transparent clear/light blue PET bottles, transparent coloured PET bottles and opaque coloured PET bottles[1] in order to confirm that the PET innovative bottle is compatible with these requirements.

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

1. Adhesives for labels (pressure sensitive adhesives, non-pressure sensitive hotmelts, wet labelling adhesives)
2. Unprinted labels
3. Printed labels

The Protocol provides guidance on the behaviour of the labels, and adhesives during the PET recycling process. The label should detach from the packaging and with no adhesive remaining on the washed PET flakes. The adhesive layer should remain on the label. Adhesive dissolving in the water represents extra effort for water treatment and are therefore not recommended. While adhesive remaining on the PET flakes may lead to discoloration of the recycled PET material. Printed labels must be tested additionally according to the bleeding inks procedure to evaluate the potential issue coming from the inks.

# 3. DISCLAIMER

The Protocol is created to represent as accurately as possible how the actual PET bottles recycling works at an industrial scale. RecyClass Technical Committee reserves the right for further testing if necessary, to issue a final opinion on the recyclability of the tested packaging. Note that, the Recyclability Evaluation Protocol for Adhesives & Labels applied on PET Bottles establishes some benchmark recommendations to guide the decision-making process. However, not all the properties listed in the protocol are provided with a benchmark recommendation, being the evaluation also based on the technical expertise of the Technical Committee.

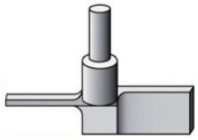
Sorting behavior of PET bottles is also important to consider, since some labels/decorations can negatively affect the sorting efficiency to the right PET stream. Therefore, it is recommended to perform a sorting test according to RecyClass Sorting Evaluation Protocol for Plastic Packaging to ensure that packaging presenting a risk of missorting is sorted in the right PET stream.

*1* Design for Recycling Guidelines

# 4. LAB EQUIPMENT

The following equipment are necessary to perform the full Protocol:

*Figure 1: Equipment necessary to the procedure*

| GLASSWARE | CHEMICALS | OTHER EQUIPMENT |
|---|---|---|
| 250 ml glass crystallizers | Caustic Soda (NaOH) | Hot plate capable of heating up to 90 °C |
| 400 ml beakers (diameter: 8 cm, height: 11 cm)<br>Suitable diameter watch glass to cover beaker when heating | Surfactant MacDermid Master RP 14-LF<br>Distilled or deionized water (referred to as “distilled water”, below) | Analytical balance with an accuracy of 0.0001 g<br>Clamping device to install overhead 600 rpm stirrer. |
| Ceramic funnel with vacuum filtration | Acetone for cleaning purposes (technical grade) | Overhead stirrer with freely selectable rotation speed; Stirrer shaft with 2 rectangular paddles (30 mm x 15).<br>  |
| | | Oven that allows temperatures up to 250°C |
| | | Color measurement equipment |
| **LAB TOOLS** | | |
| Accurate cutting device (scissors, blade, scalpel) | Manual stirrer (i.e., glass rod) | Metal weight with flat surface (150 g, max 5,5 $cm^2$) |
| Thermometer | Paper filter | Clean white tile/toughened glass tile to be used as non-sticky, dry, even and free of dust and fibres solid surface |
| Moisture Analyser (0.001% precision) | Metal tweezers | Digital camera |

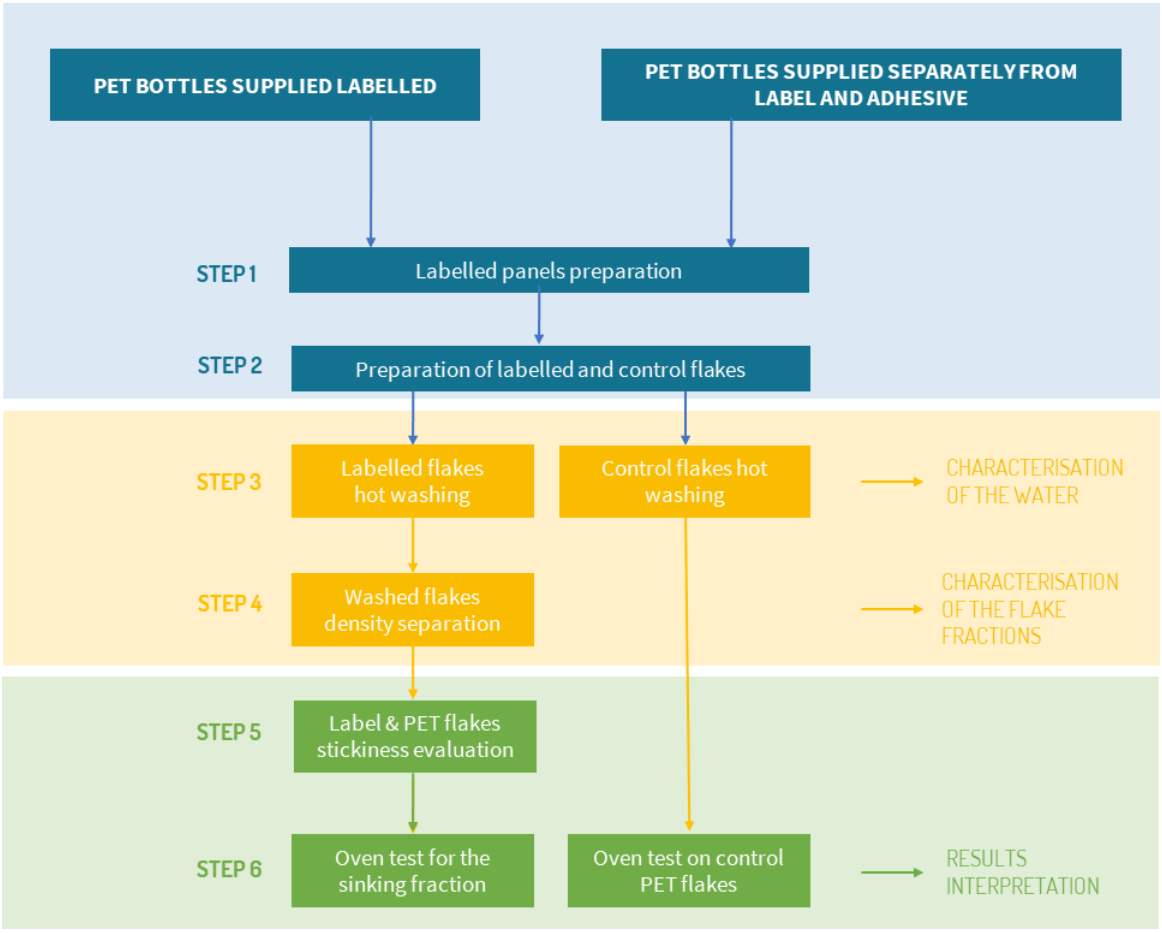
# 5. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the washing step of the PET bottles recycling process at laboratory scale to determine the suitability of an innovative adhesive or label with the PET bottle recycling streams. The methodology described below shall be followed precisely and any modifications or problems must be noted by laboratory technicians during the testing phase. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared and submitted to the RecyClass PET Technical Committee which will interpret the final results and

define the compatibility or not with recycling. 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.

*Figure 1: Methodology Diagram*



## 5.1 CONTROL SAMPLE

The control PET bottle for use following the Protocol must be the same PET container as the one with the applied label and adhesive. In order to minimise the deviations in the mass balance approach, exact same PET bottles should be used labelled, and not labelled.

For the purpose of the tests, the Applicant should provide at least 5 labelled PET bottles, with a total of labelled surface representing about 1,000cm$^2$. At least 5 non-labelled PET bottles must also be provided. 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.

## 5.2 INNOVATION SAMPLE

While both transparent and coloured labels can be used for this recyclability assessment, it is preferred to operate with coloured polyolefin labels in order to facilitate the separation and identification of the bottle and label flakes after washing.

# 6. LABORATORY TEST PROCEDURES

## 6.1 SAMPLE PREPARATION

### 6.1.1 STEP 1: PANELS PREPARATION

**Procedure:**

- Evaluate the number of bottles to be cut to obtain a minimum label surface of 600 cm$^2$. This number will be reported as Nb in the following instructions.

- Weigh at least 5 non-labelled PET bottles to obtain an average weight that will be reported as “A1”. Report as well as standard deviation.

- Weigh at least 5 labelled PET bottles to obtain an average weight that will be reported as “A2”. Report as well as standard deviation.

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

- Cut out panels from the labelled area of the side of Nb PET bottles to obtain a minimum surface of 600 cm$^2$. Uncovered panel margins surrounding the applied label must have a maximum width of 1 mm on each margin side. Please note that Nb must be a whole number, which means that the entire label must be used for the assessment. These panels will further be referred as innovation panels. For clarity, look at Figure 2.

- Record the total weight of the labelled panels as “B”.

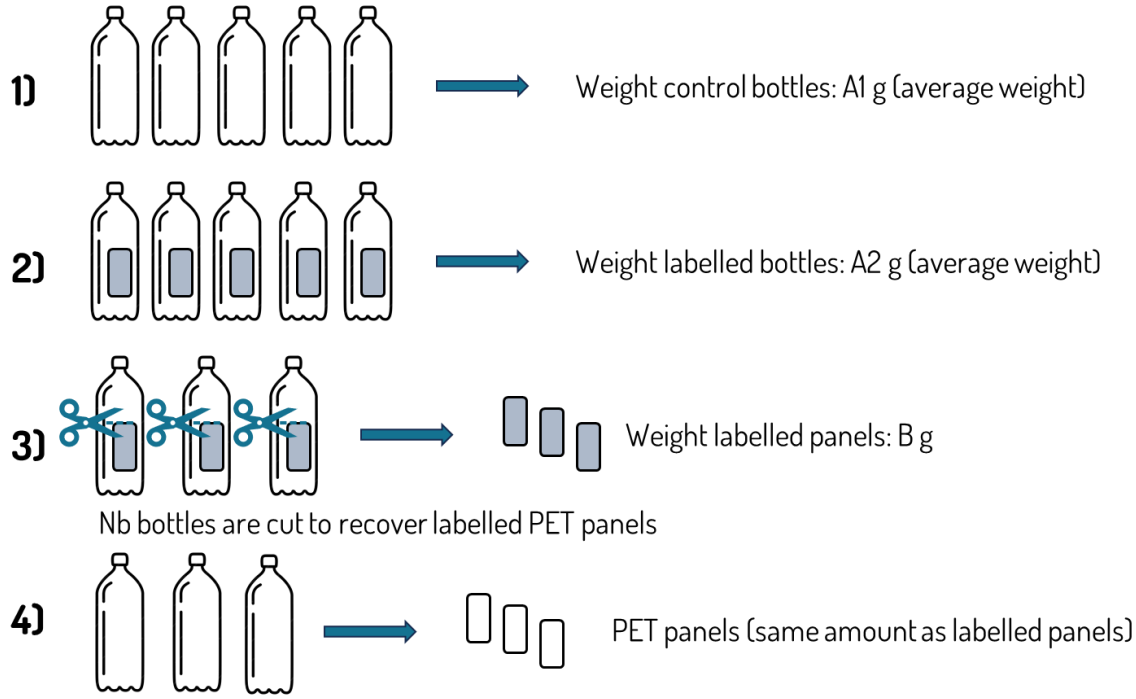
- Document labelled panels with one or more photographs.

- Cut out panels from the non-labelled area of the side of the PET bottles to obtain a minimum surface of 600 $cm^2$. These panels will further be referred as control panels.

- The weight of the PET present on the innovation panels, must be reported as "A" (in Annex 1) and calculated as following:
A = A1 – (A2-B/Nb)

- Determine the total weight of the label and adhesive present on the innovation panels, reported as "E", as following:
C = B – A

*Figure 2: Illustration of sample preparation*




### 6.1.2 STEP 2: FLAKES PRODUCTION

Control and innovation samples are separately cut in order to prepare controlled-size flakes.

**Procedure:**

- Cut control panels in a controlled environment. Avoid generating fines. Flakes should be shaped approximately as squares with an edge length of about 10 mm. These flakes will constitute the control flakes.

- Cut innovation panels in a controlled environment. Avoid generating fines. Flakes should be shaped approximately as squares with an edge length of about 10 mm. These flakes will constitute the innovation flakes.

- Weigh the innovation flakes after cutting, and report the value as "C2". The weight loss after cutting innovation panels should be inferior to 0.02 % by weight, meaning that: $\Delta C=(C2-C)/C < 0.02$ %.

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

## 6.2 WASHING & SEPARATION BY DENSITY STEPS

### 6.2.1 STEP 3: WASHING

At the state of the art, European PET recycling lines typically use hot washing conditions (about 85 °C), caustic soda and detergents, in a multiple step washing process. The following procedure must be applied to both control and innovation flakes, separately.

**Procedure hot-washing:**

- Prepare the washing beakers (400 mL) for a 1:4 ratio (10 g flakes vs 40 mL solution) at 85 °C with a solution of 1 wt% NaOH and 0.3 wt% MacDermid RP 24 detergent.

- Wash each sample separately at a 1:4 ratio (10 g flakes vs 40 mL solution) at 500 rpm for 15 minutes. Position the stirrer at about 5 mm from the bottom of the beaker.

- Make sure that the labels do not stick to the stirrer, nor the side of the beaker. It is important to ensure free agitation of flakes and label particles.

- Record the washing process with a series of photographs. Any stickiness, odour, suspended particles, or discoloration shall be recorded, and illustrated with pictures.

- In the case of discolouration of the washing water, a bleeding ink quick test is recommended to be performed. Take pictures of the washing solutions used for innovation and control flakes side by side.

- Collect the floating and sinking fractions together by filtration on a ceramic funnel connected to a vacuum pump. Use small amounts of distilled water and a suitable tool to collect any remaining flakes at the bottom of the beaker or on the stirrer. No flakes must be lost in the transfer process.

### 6.2.2 STEP 4: DENSITY SEPARATION

Since no density separation is expected for the control sample, only innovation sample should be going through the density separation step.

**Procedure density separation:**

- Prepare the beakers (400 mL) for a 1:8 ratio (10 g flakes vs 80 mL distilled water) at room temperature with distilled water.

- Gently stir the solution manually for about a minute, and let the solution settle for 10 minutes. Ensure that no flakes or labels remain on the stirrer, and that no flakes are lost in the process.

- After the settling process, take pictures of the beakers containing the flakes/labels.

- Carefully collect the floating fraction, and place it on filter paper to partially dry it. Then, transfer the floating fraction within a crystallizer.

- Collect the sinking fraction by filtration on a ceramic funnel connected to a vacuum pump. Place the collected flakes on filter paper for gross removal of water. Then, transfer the sinking fraction within a crystallizer.

- Dry both fractions separately in an oven at 85 °C for 1 hour. Then, let the flakes come back to room temperature.

- Perform the stickiness evaluation after drying for both the floating and sinking fractions according to the procedure reported in section 6.3.1.

- Record the total weight of the floating fraction as “Df”. Take photos of the fraction.

- If any labels are trapped within the sinking fraction, or if some labels are only partially removed, take picture of the corresponding flakes. Then, carefully try to separate these labels from the PET flakes. Do not force if the label cannot be separated from the PET flake.

- Record the total weight of labels that could be separated from the sinking fraction as “Ds”. Take photos of the fraction. This fraction can then be added to the floating fraction.

- PET flakes that were easily separated from the remaining labels in the sinking fraction can be added to the clean PET flake sinking fraction.

- Keep separated the 3 following fractions: 1) PET flakes with non-separable label, 2) Labels that could be easily removed or were already removed from the PET flakes in the sinking fraction, 3) Clean PET flakes from the sinking fraction, or that were easily separated from labels trapped in the sinking fraction.

- Record the total weight of the PET flakes with non-separable label from the sinking fraction as “X”. Take photos of the fraction.

- Record the total weight of the clean PET flakes from the sinking fraction, as “E”. Take photos of the fraction.

- Determine the weight of the corrected floating fraction, reported as “H”, as following: D = Df + Ds.

## 6.3 CHARACTERISATIONS

### 6.3.1 STEP 5: STICKINESS CHARACTERISATION

The stickiness of flakes after washing must be evaluated to identify any remaining traces of adhesives, or any re-activation of the adhesive after drying. Results generated with this test will only be used for informative purposes. The following procedure must be followed for such evaluations:

**Stickiness characterization procedure:**

- Spread flakes with a clean tweezers on a clean surface.

- Attach a clean PET flake with strong adhesive tape to the part of the metal weight that will be in contact with PET flakes to be tested, in order to test PET-PET contact instead of metal-PET.

- Place the 150 g metal weight on the surface of ten randomly chose different flakes (PET or label depending on the fraction) for 5 seconds each, without applying additional pressure. Make sure that the entire surface of the flake is in contact with the PET attached to the weight.

- The PET flake attached to the weight shall be cleaned with a slight amount of acetone after testing each flake. For each test, the PET flake attached to the metal weight should be dry and free of any adhesive or acetone residual.

- When lifting the metal weight, if the flake does not remain attached to the weight, this is considered as a positive result. On the opposite, if the flake remains attached to the weight, this is considered as a negative result.

- Report the amount of negative results on top of the 10 flakes tested, and fill the Annex 2 with the result.

### 6.3.2 STEP 6: OVEN TEST

An oven test will need to be performed for the remaining clean PET flakes sinking fraction (both for control and innovation) to see any potential discoloration of the PET flakes and presence of remaining adhesives.

**Procedure:**

- Preheat the oven at 220 °C.

- Take about 10 grams of the control material, and the clean PET flake sinking fraction from the innovation sample and put them on two different crystallisers. Spread the fractions evenly in the crystallisers.

- Perform a colour measurement before the heat treatment under the following conditions (Reflectance mode, D65, 8-10 °, SCI gloss setting) for both control and innovation PET flakes. Report the results in the Annex 3.

- Place the samples in the oven at 220 °C for 1 hour.

- Let the samples cool down at room temperature.

- By transferring each fraction on a white surface, evaluate flakes for any discolouration, black specks, stickiness, curling behavior.

- Perform a colour measurement after the heat treatment under the following conditions (Reflectance mode, D65, 8-10 °, SCI gloss setting) for both control and innovation PET flakes. Report the results in the Annex 3.

- Document the innovation and control flakes with photographs on a white and black background.

# 7. MASS BALANCE & RESULTS INTERPRETATION

## 7.1 MASS BALANCE

### 7.1.1 RECOVERY & DISSOLVED ADHESIVE RATIO CALCULATIONS

Additional ratios and efficiencies shall be calculated based on the weight measured during the testing. The following calculations presented in Table 2 shall be followed for the innovation material.

*Table 2: Calculation of recovery & dissolved adhesive ratios.*

| **PARAMETER** | REFERENCE | DESCRIPTION | CALCULATION |
|---|---|---|---|
| Non-recovered label and adhesive (g) | K | Amount of label and adhesive not recovered in the floating fraction | K = C - D |
| Label and adhesive recovery ratio (%) | L | Recovery efficiency of label and adhesive in the floating fraction from the initial sample | L = (D/C)*100 |
| Label/PET separation efficiency (%- | M | Efficiency of separation of the label from the PET flakes, based on the non-separated fraction X | M = (1 – (X/B))*100 |
| PET flakes loss (%) | N | Amount of PET material loss in the testing process (non-sinking, fines removed by the washing process, …) | N = ((A-E)/A)*100 |

| | | | |
|---|---|---|---|
| Non-recovered material (g) | P | Determine the amount of material loss compared to everything collected in the floating and sinking fractions | P = B – (D + E + X) |
| Amount of the initial label and adhesive fraction not recovered (%) | Q | Ratio of the non-recovered floatable material compared to the total weight of the "expected" floating fraction | Q = ((P-(A-E))/C)*100 |
| Concentration of "dissolved" fraction (ppm) | R | Assuming a water density of 1 g/cm$^3$, this determines the concentration of dissolved adhesive during the process | R = (P-(A-E))/water volume |

## 7.2 RESULTS INTERPRETATION

All results coming from the mass balance approach as well as the different characterisations must be reported in the Annex 1.

Ideally, E should be equal to A, D equal to C, and X equal to 0, which would mean that PET and labels were perfectly separated with all adhesive remaining on the label facestock. Nevertheless, due to moisture uptake, loss of fines, measurements error, some small deviations can be observed.

As success criteria for this recyclability evaluation, the following conditions shall be met:

- ✓ **M > 97 %** : Less than 3 wt% of the label should remain attached to PET flakes.
- ✓ **N < 2 %, Q < 2 %** : Less than 2 wt% of PET and label material shall be lost in the process.
- ✓ **Adhesive should remain on the label facestock.**
- ✓ **No discoloration of the washing water.**
- ✓ **No coloration of the flakes after oven test.**

Note that all the parameters will be considered by the RecyClass PET Technical Committee to assess the recyclability of the solution tested.

# 8. REPORT CONTENT

The report should contain the following information:

- Reference to the Procedure and its version: *Washing Quick Test Procedure for Labels & Adhesives Applied on PET Bottles – 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 testing equipment.

- 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.

- Summary tables present in Annex 1, Annex 2 and Annex 3, filled with results.

- Date and place of the test.

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# DOCUMENT VERSION HISTORY

| VERSION | PUBLICATION DATE | REVISION NOTES |
|---|---|---|
| 1.0 | January 2024 | RecyClass Recyclability Evaluation Protocol for Labels & Adhesives Applied on PET Bottles release |

# ANNEX 1 – SUMMARY TABLE

*Table 2: Flake characterisation before and after washing.*

| VARIABLE0 | VALUE | REFERENCE IN THE PROCEDURE |
|---|---|---|
| **Total weight of the panels** | X,xxxx ± 0.xx g | A = A1 – (A2 – B/Nb) |
| **Innovation sample weight** | X,xxxx g | B |
| **Total weight of labels and adhesives** | X,xxxx g | C = B - A |
| **Weight of the floating fraction** | X,xxxx g | Df |
| **Weight of the removable labels from the sinking fraction** | X,xxxx g | Ds |
| **Corrected weight of the floating fraction** | X,xxxx g | D = Df + Ds |
| **Weight of the PET flakes in the sinking fraction** | X,xxxx g | E |
| **Weight of the non-separable labels/PET fraction** | X,xxxx g | X |
| **Non-recovered label and adhesive (g)** | X,xxxx g | K = C – D |
| **Label and adhesive recovery ratio (%)** | X,xx % | L = (D/C)*100 |
| **Label/PET separation efficiency (%)** | X,xx % | M = (1 – (X/B))*100 |
| **PET flakes loss (%)** | X,xx % | N = ((A - E)/A)*100 |
| **Non-recovered material (g)** | X,xx g | P = B – (D + E + X) |
| **Amount of the initial label and adhesive fraction not recovered (%)** | X,xx % | Q = ((P-(A-E))/C)*100 |
| **Concentration of "dissolved" fraction (ppm)** | X ppm | R = (P-(A-E))/water volume |

# ANNEX 2 – STICKINESS RESULTS

*Table 3: Stickiness characterisation summary table*

| VARIABLE | NUMBER OF NEGATIVE RESULTS | LEVEL OF REACTIVATION* |
|---|---|---|
| **Stickiness level of floating fraction after drying** | 0 to 10 | No reactivation/Light reactivation/Strong reactivation |
| **Stickiness level of sinking fraction after drying** | 0 to 10 | No reactivation/Light reactivation/Strong reactivation |

* If 0 negative results: No reactivation; if 1 to 4 negative results: Light reactivation; if more than 4 negative results: Strong reactivation.

# ANNEX 3 – COLOR MEASUREMENTS RESULTS

*Table 4: Colour measurement summary table*

| **VARIABLE** | CONTROL MATERIAL | | INNOVATION MATERIAL | |
|---|---|---|---|---|
| | Before Oven | After Oven | Before Oven | After Oven |
| **L*** | | | | |
| **a*** | | | | |
| **b*** | | | | |
| **ΔE (before-after oven)** | | | | |
| **ΔE to control** | | | | |

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