RecyClass

RECYCLABILITY EVALUATION PROTOCOL

FOR INKS APPLIED ON DECORATIONS FOR PET BOTTLES

STANDARD LABORATORY PRACTICE

REP-PETbot-03

VERSION 1.0

PUBLISHED ON MAY 2025

RecyClass

CONTENT

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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 product, providing recommendations on improving their recyclability before market launch.

The Recyclability Evaluation Protocols are freely available for download in 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 Technology 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 Inks applied on Decorations for PET Bottles" referred to in this document as "The Protocol" describes the methodology that the applicant must follow at a laboratory scale in order to determine if inks are compatible with the post-consumer PET bottle recycling streams, meaning transparent clear/light blue, transparent coloured and white opaque 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 Technology Approval Quality Management & Procedures document[2]

The Protocol aims to evaluate the behaviour of inks, varnishes and coatings 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 inks market used for PET bottle applications. The overall goal is to ensure that inks will not be bleeding or washed-off from the PET bottle decorations to avoid any contamination 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.

Inks terminology as it is used in this document refers to the EuPIA definition:

a. Mixtures of colourants with other substances which are applied on materials to form a graphic or decorative design together with or without
b. Other coloured or uncoloured overprint varnishes/ coatings or primers which are normally applied in combination with a. in order to enable the printed design to achieve specific functions such as ink adhesion, rub resistance, gloss, slip/friction, durability etc.

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.

2. SCOPE OF THE PROCEDURE

The scope of the Protocol covers any coatings, inks, varnishes introduced to the existing packaging solutions for PET bottles. Before 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 white opaque PET bottles[1] in order to confirm that the PET innovative bottle is compatible with the recycling stream for PET bottles.

The scope of this Protocol covers the following non-exhaustive list of packaging solutions and/or innovations:

1. Direct printed PET bottles
2. Printed labels applied on PET bottles
3. Printed sleeves applied on PET bottles
4. Other printed decorations applied on PET bottles

The Protocol provides guidance on the behaviour of the inks during the PET recycling process. The inks should not bleed neither get washed off during the washing and floatation steps, remaining on the label or sleeve. Bleeding inks dissolves in the water, while washable inks are dispersed into particles in the wash water. Both of these types of inks represent extra effort for water treatment, as inks will be considered here as contaminant. Printed labels must be tested additionally according to the REP-PETb-02 testing procedure to evaluate if the adhesive would be washable or not.

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 innovation. The Recyclability Evaluation Protocol for Inks applied on Decorations for PET Bottles establishes some benchmark recommendations to guide the decision-making process. However, only some of the properties listed in the protocol are provided with a benchmark recommendation, given that the evaluation also based on the technical expertise of the Technical Committee (TC).

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.

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 with thermocouple

1 Design for Recycling Guidelines

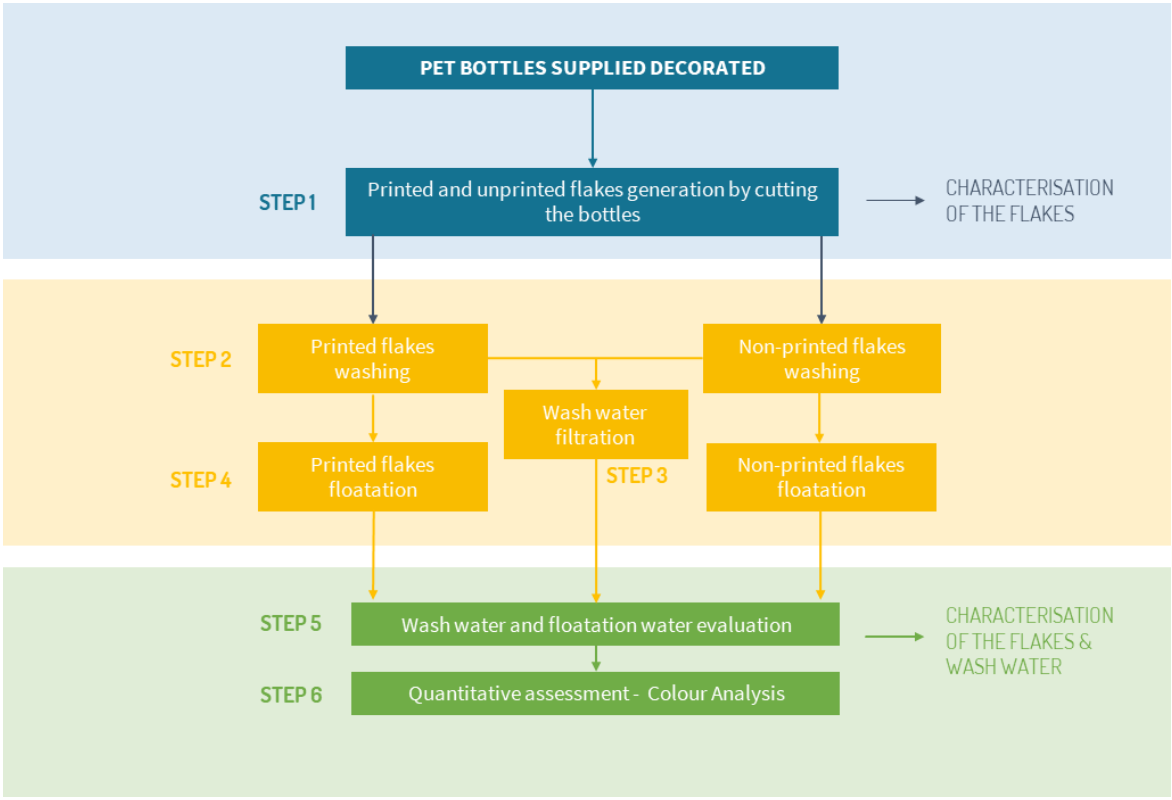
600 ml beakers (diameter: 8 cm, height: 15 cm) & 800 ml beakers (diameter: 10 cm, height: 13.5 cm)	Surfactant MacDermid Master RP 24 or 34 / BASF Gardoclean S 5150/2 / CHT Tubiwash SKP	Clamping device to install overhead 600 rpm stirrer.
Suitable diameter watch glass to cover beaker when heating	Distilled or deionized water (referred to as "distilled water", below)	Overhead stirrer with freely selectable rotation speed; Stirrer shaft (see Annex 1 for allowed geometries)
Ceramic funnel with vacuum filtration	Acetone for cleaning purposes (technical grade)	Color measurement equipment (UVVis spectrophotometer) & Laser Diffraction (LD) (optional)
LAB TOOLS		
Accurate cutting device (scissors, blade, scalpel)	Manual stirrer (i.e., glass rod)	Digital camera
Thermometer	Whatman® qualitative filter paper, Grade 5 (2.5 µm pore size)	Oven heating up to 100°C

5. LABORATORY TESTING 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. Additional tests may be requested by the PET TC for specific cases requiring particular attention. A Laboratory Evaluation Report compiling objectively all the results obtained shall be prepared and submitted to the RecyClass PET TC 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 describing methodology.

Figure 1: Methodology Diagram



5.1 CONTROL & INNOVATION SAMPLE SELECTION

The control PET bottle for use following the Protocol must be the same PET container as the one with the applied printed decoration. In order to minimise the variables in the test, exact same PET bottles should be used decorated and not-decorated. The control material can either be a non-decorated PET bottle, or can be selected as an area of the innovation PET bottle that is not decorated.

The innovation sample corresponds to PET flakes containing the corresponding printed decoration. In the case of detachable decorations, like sleeves or wrap around labels, the PET flakes and decoration flakes must represent the same surface. For labels, printed labelled PET flakes must be used.

For the purpose of the tests, the Applicant should provide at least 5 decorated PET bottles, with a total of printed surface representing about 1,000cm^2. The Applicant should preferably use one of the artworks available on Annex 2 or any similar artwork with presence of CMYK colours. At least 5 non-decorated PET bottles must also be provided. More printed decoration material may also be required if the printing coverage is not sufficient to obtain the amount of printed flakes and/or if additional tests are required.

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

Preferably, the bottle should be tested with only one type of decoration. In the case of a final packaging, all labels should be used for the testing.

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 printed surface of 600 cm^2. This number will be reported as Nb in the following instructions.

- Report the nature of the printing, including the amount and chemistry of inks used. Document the decoration design with one or more photographs.

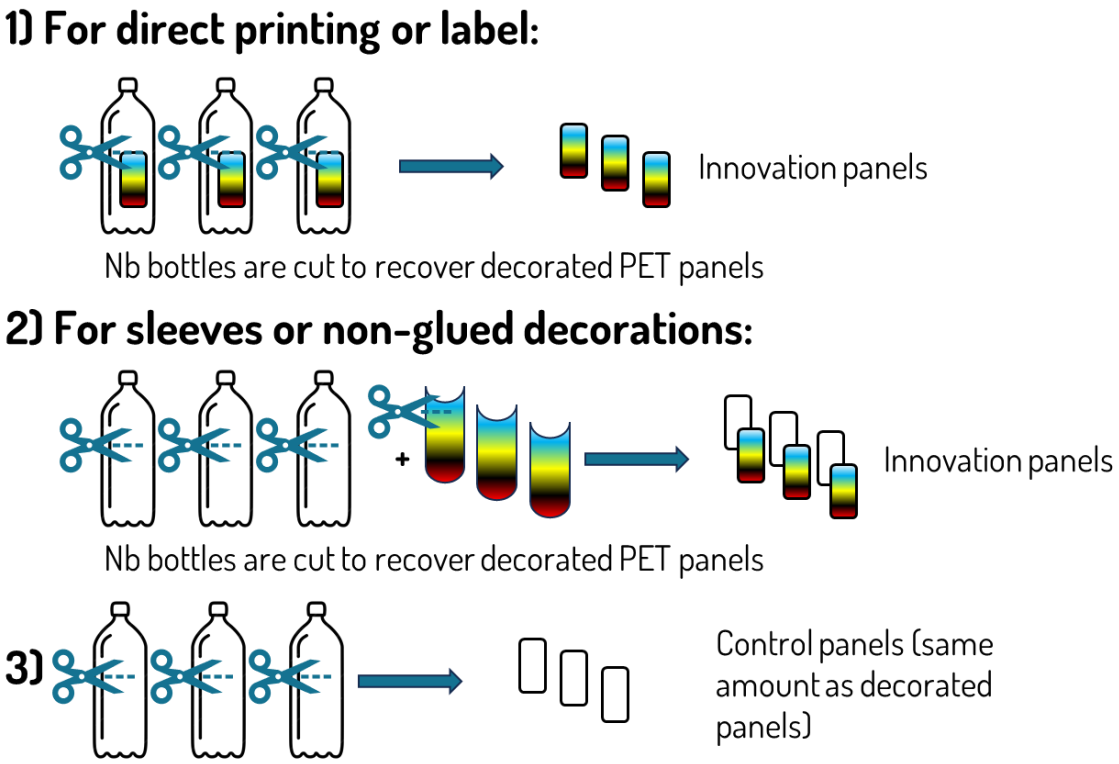
- For direct printing and printed labels: cut out panels from the printed/decorated area of the side of Nb PET bottles to obtain a minimum surface of 600 cm^2. Uncovered panel margins surrounding the applied label must have a maximum width of 1 mm on each margin side. Please note that Nb must be a whole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

- For printed sleeves or other non-glued decorations: cut out panels from the printed area of the sleeve of Nb PET bottles to obtain a minimum surface of 600 cm^2. Please note that Nb must be a whole number, which means that the entire label or printed area must be used for the assessment. These panels will further be referred as innovation panels. For clarity, look at Figure 2.

- Document printed panels with one or more photographs.

- Cut out panels from the non-printed/decorated 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.

Figure 2: Illustration of sample preparation



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 innovation flakes.

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

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

6.2 WASHING & SEPARATION BY DENSITY STEPS

6.2.1 STEP 2: WASHING

At the state of the art, European PET recycling lines typically use hot washing conditions (about 85 °C), caustic soda and surfactant, 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 (100 flakes vs 40 mL solution) at 85 °C with a solution of 1 wt% NaOH and 0.3 wt% of surfactant (see Figure 1 for surfactant selection).

- Wash each sample separately at a 1:4 ratio (100 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 flakes do not stick to the stirrer, nor the side of the beaker. It is important to ensure free agitation of flakes.

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

- Use a strainer to separate the flakes from the wash water. No flakes must be lost in the transfer process. Save the wash water in a beaker. Afterwards rinse the flakes with cold running tap water for 5 minutes under vigorous stirring with a manual stirring bar.

- Take a photograph of both wash water solutions obtained for control and printed flakes in beakers alongside each other in front of a light (white) background.

- Take a similar photograph in front of a dark (black paper, brown paper box) background.

- Report any noticeable change of colour or transparency, or any dispersed floating particles.

6.2.2 STEP 3: WASH WATER FILTRATION

In order to better identify the nature of any colouration of the water, both wash water used for control and innovation must be separately filtered, separately.

Procedure water filtration:

- Before filtration, save about 30 mL of wash water into a vial. To get this sample, stir gently the solution for 5 seconds and then drop the amount of solution within the vial without using pipette or any other sampling tool.

- Filter the wash water using a Whatman® qualitative filter paper, Grade 5 on a ceramic funnel connected to a vacuum pump. Use small amounts of distilled water and a suitable tool to collect any remaining deposit at the bottom of the beaker or on the stirrer.

- After filtration, save about 30 mL of wash water into a vial. To get this sample, stir gently the solution for 5 seconds and then drop the amount of solution within the vial without using pipette or any other sampling tool.

- Take a photograph of both wash water solutions obtained for control and printed flakes in beakers alongside each other in front of a light (white) background.

- Take close photographs of the filters, and any residues present. Identify residues present on the filters (inks particles, paper fibres, PET fines, …).

- Recover and weight the residues present on the filters. Record the total weight of the residue as “Rc” for control and “Ri” for innovation.

6.2.3 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 (100 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.

- Take pictures of the water solutions after separation by density. Report any presence of particles or impurities.

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

- Keep separated the 2 following fractions: 1) Printed/decorated flakes, 2) Clean PET flakes from the sinking fraction.

- Take pictures of the printed/decorated flakes to see if the artwork and inks were damaged during the washing and separation by density processes.

6.3 CHARACTERISATIONS

6.3.1 STEP 5: QUALITATIVE ASSESSMENT

Qualitative assessment must be carried out to evaluate inks behavior during the washing and floatation steps.

Procedure:

- Compare the washing water (before and after filtration) and the floatation water of both innovation and control samples. Note any noticeable colouration, or impurities present in the solutions.

- Observe the dried printed flakes. Note any noticeable discolouration of the flakes compare to non-washed flakes.

6.3.2 STEP 6: COLOUR CHARACTERISATION OF WATER

Quantitative colour characterization of the wash water after filtration should also be performed using the 30mL samples collected. No strong coloration of the water, neither presence of dispersed particles should be observed. A colour evaluation of the wash solutions can be carried out using the standard illuminant D65 and the 10° standard observer on a UVvis-spectrophotometer. In this case, the following procedure must be followed to quantitatively assess the colour change of both solutions in the same test conditions.

Wash water colour characterization procedure:

- Use plastic cuvettes and tap-water as reference solution.

- Perform a full UVVis-scan recording the extinction of clean tap water.

- Perform a full UVVis-scan recording the extinction of each filtered wash water.

- Display the obtained L*a*b* values obtained from the measurement.

6.3.3 OPTIONAL STEP: PARTICLE SIZE CHARACTERIZATION

In the case that dispersed inks particles would be observed before filtration, it is recommended to perform a Laser Diffraction (LD) characterization to understand the dispersion and the size of size of the particles present in the solution.

7. RESULTS INTERPRETATION

All results coming from the colour measurement (step 6) must be reported in the Annex 3.

Ideally, no colouration of the water used for the washing and separation by density steps should be observable. No ink particles should be generated during the washing process, and the flakes should keep the same visual aspect and artworks after washing.

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

- ✓ **Variations of the L*, a* and b* values should be kept into the following range:**
  - ΔL* between -2 and 2 compared to the control material
  - Δa* between -1 and 1 compared to the control material
  - Δb* between -1 and 1 compared to the control material
- ✓ **ΔE < 4 compared to the control material.**
- ✓ **No observable inks particle dispersed in the washing solution.**
- ✓ **No observation of damaged printed area or discoloration of the artwork**

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: *Recyclability Evaluation Protocol for Inks applied on Decorations for PET Bottles – V1.0*

- A full and complete identification of the material tested, including:

  - **Inks:** Colour, nature, surface/reverse printing
  - **Label or sleeve**: size, structure, and density.
  - **Adhesive if present**

- Description and photographs of the testing equipment.

- Description of the samples during each step (especially on colour changes, deposits, residual particles, …).

- 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 3, filled with results.

- Date and place of the test

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DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	May 2025	Recyclability Evaluation Protocol for Inks applied on Decorations for PET Bottles

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ANNEX 1 – RECOMMENDED ARTWORKS FOR INNOVATION

ANNEX 2 – ALLOWED STIRRER PALES GEOMETRIES

Table 2: Acceptable stirrer pales geometries

GEOMETRY TYPE	PICTURE	STIRRER DIMENSIONS
Cross shaped paddle with four 45° angled angular blades (ref: https://www.bola.de/en/Propeller-Stirrer-Shafts-with-4-Blades-BOLA)		L = 75 mm H = 20 mm

ANNEX 3 – SUMMARY TABLE

Table 1: Colour characterisation results

WASH WATER AFTER FILTRATION	CLEAR TAP WATER	CONTROL SAMPLE	INNOVATION SAMPLE
L*			
a*			
b*			
ΔE (compared to control)			

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