QUICK TEST PROCEDURE FOR BLEEDING INKS

PRINTED ON HDPE & PP CONTAINERS

STANDARD LABORATORY PRACTICE

VERSION 1.1

PUBLISHED ON JANUARY 2022

CONTENT

CONT	ENT		2
1.	INTRO	DDUCTION AND PURPOSE OF THE PROCEDURE	4
2.	SCOP	E OF THE PROCEDURE	5
3.		QUIPMENT	
4.		RATORY TEST METHODOLOGY	
4.1		IPLES SELECTION	
4.2	PRO	DCEDURE	7
4	.2.1	STEP 1: Containers cutting & Flakes characterization	7
4	.2.2	STEP 2: Washing	7
4	.2.3	STEP 3: Flotation	
4	.2.4	STEP 4: Drying	8
4	.2.5	STEP 5: Bleeding behaviour Assessment	8
5.	REPO	RT CONTENT	. 10
DOCL	JMENT	VERSION HISTORY	. 11
ANNE	X – RES	SULTS SUMMARY	.12

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 RecyClass website."

1. INTRODUCTION AND PURPOSE OF THE PROCEDURE

The "Quick Test Procedure for Bleeding Inks applied on HDPE & PP 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 inks used on HDPE or PP packaging are compatible with the post-consumer HDPE or PP recycling stream.

The Procedure evaluates printed containers, labels and sleeves by performing quick tests, mainly related to washing process. The results of the quick tests 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 recyclability. 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.

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 definition³:

- 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.

However, the term "Printing inks" does not include coatings which are applied with the prime objective of enabling the material or article to achieve a technical function such as heat sealing, barrier, corrosion resistance etc., as opposed to a graphic effect, even though they may be coloured. These "coatings" with a purely technical function should be considered separately.

¹ Quick Test Procedures: https://recyclass.eu/recyclability/test-methods/

² Recyclability Evaluation Protocols: https://recyclass.eu/recyclability/test-methods/

³ EuPIA definition of inks and other associated definitions can be found in the EuPIA glossary.

2. SCOPE OF THE PROCEDURE

The scope of the Procedure covers any materials related to inks introduced to the existing HDPE or PP packaging solutions. Prior to initiating the evaluation, the Applicant is required to review the Design for Recycling Guidelines for HDPE and PP containers⁴ in order to confirm that the material is compatible with these requirements.

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

- 1. Direct printed HDPE rigid packaging
- 2. Direct printed PP rigid packaging
- 3. Printed labels applied on HDPE or PP rigid packaging
- 4. Printed sleeves applied on HDPE or PP rigid packaging5. IML applied on HDPE or PP rigid packaging
- 6. Paper decoration applied on HDPE or PP rigid packaging

The procedure provides guidance on the behaviour of inks during the HDPE and PP recycling process. The inks should not bleed during the washing and flotation steps, remaining on the container, label, or sleeve. Bleeding inks dispersed in the water represents extra effort for water treatment, as inks are water-contaminants. Effect of the combination adhesive and label must be tested in addition according to the washing QT procedure to evaluate potential issues coming from the adhesive.

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) or another overhead stirrer able to produce a vortex. See picture.
- Overhead stirrer with freely selectable rotation speed
- Analytical balance with an accuracy of 0,0001 g
- Strainer (mesh size of 0,5 x 0,5 mm)
- Moisture Analyzer (res. 0.001 %)
- Oven
- UVVis-spectrophotometer (optional)



⁴ Design for Recycling Guidelines https://recyclass.eu/recyclass/design-for-recycling-guidelines/

4. LABORATORY TEST METHODOLOGY

This methodology aims at reproducing the washing and flotation steps of the recycling process at a small scale to determine the suitability of inks applied on packaging for the HDPE or PP recycling stream. The methodology described below shall be followed precisely and any modifications or problems must be noted during the testing phase.

An Evaluation Report compiling all the results obtained shall be prepared by the laboratory to explicit the results. Any remarks during following the Procedure shall be also noted down. An Annex to this document is also available on RecyClass website to summarize all the collected data and do the assessment

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

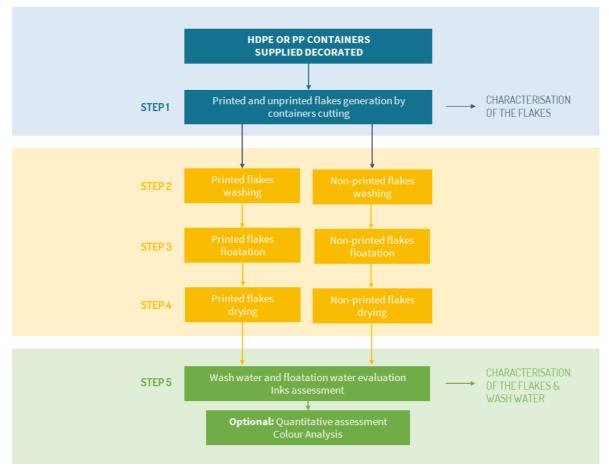


Figure1: Methodology Diagram

4.1 SAMPLES SELECTION

The control container for use following the Procedure must be the same unprinted container as the printed one. For the purpose of the tests, the Applicant should provide at least 1kg of containers. If the printed area covers a small part of the container (<50%), control flakes can be cut from the unprinted part of the container. More printed material may be required if the printing coverage is not sufficient to obtain the amount of printed flakes and/or if additional tests are required. In the opposite, if the printed area covers a large part of the container (>50%), control flakes must be cut from the control container (same unprinted container as the printed one).

4.2 PROCEDURE

4.2.1 STEP 1: CONTAINERS CUTTING & FLAKES CHARACTERIZATION

The first step consists in generating HDPE or PP flakes from the containers. Both control and printed flakes must be cut.

Procedure:

- Cut out with scissors the printed parts of the containers. Avoid generating fines. Flakes should be shaped approximately as squares with an edge length between 10-12mm. These flakes will constitute the printed sample.
- Cut out with scissors the unprinted parts of the containers. 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 printed and control samples must be generated. Printed flakes must be cut in different locations of the printed area and cut from different containers. Record the total weight of the printed flakes as well as the control flakes (See Annex Table 2).
- Document the printed and control flakes with one or more photographs.

4.2.2 STEP 2: WASHING

At the state of the art, European HDPE and PP 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 (Annex Table 2). Do not add detergents or caustic soda.
- Heat the washing solution to 40 °C.
- Wash each sample separately by stirring up to 1.000 rpm (choose a speed which produces a vortex which rises from the surface until the ground of the beaker) for 5 min with a paddle stirrer.
- Filter the wash with a strainer, collect the wash water. Afterwards rinse the flakes with cold running tap water for 5 minutes under vigorous stirring with a manual stirring bar.
- Save the wash water solution.
- Determine the pH-value of the wash water (Annex Table 2). Be aware of the correct temperature for measuring. If necessary, first cool down the wash water to room temperature (20 23 °C).

After the previous procedure has been followed for both control and printed flakes, separately, the following procedure must be respected.

Procedure:

- 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 (Annex Table 4).
- Take a similar photograph in front of a dark (black paper, brown paper box) background (Annex Table 4).
- Report any noticeable change of colour or transparency.

4.2.3 STEP 3: FLOTATION

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 utilized for both control and printed flakes, separately.

Procedure:

- Fill a beaker with tap water (ratio: 50 flakes in 200 mL water in a 600 mL beaker).
- Put the washed flakes in the water and stir at 500 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.

Observe the colour of the two flotation 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 two photographs of both solutions in the beaker alongside each other respectively in front of a light and dark background (Annex – Table 4).

4.2.4 STEP 4: DRYING

The last step before the assessment is to reduce the flakes moisture content to less than 1wt%. The following procedure must be utilized for both control and printed flakes, separately.

Procedure:

- Dry the floating flakes collected after washing 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 (Annex Table 2).
- Store the dried flakes for 24 h.

4.2.5 STEP 5: BLEEDING BEHAVIOUR ASSESSMENT

Qualitative assessment must be carried out to evaluate inks behaviour during the washing and flotation steps.

Procedure: Qualitative assessment

- Compare the washing water (before and after filtration) and the flotation water of both printed and control samples. Note any noticeable colouration of the wash.
- Observe the dried printed flakes. Note any noticeable discolouration of the flakes compare to non-washed flakes.

<u>Assessment criteria</u>: The printing inks are considered as "non-bleeding" and pass the procedure if all the following criteria are met:

- No noticeable discoloration is observed for both washing water and flotation water
- No noticeable discolouration of the flakes is observed after drying.

In order to further investigate possible bleeding inks and wash water colouration, a colour evaluation of the wash solutions can optionally be carried out using the 2120D Standard method 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.

Procedure: Quantitative assessment (optional)

- Use plastic cuvettes and tap-water as reference solution.
- Perform a full UVVis-scan recording the extinction of each filtered wash water.
- Display the obtained spectra of the control and the innovation flakes in the protocol.
- Extract the absorption maxima area (x y nm) from the spectrum. Note this in the protocol. Have a look in Figure 2 and describe the colour within the area as yellow, orange, green etc. .

Interpretation:

- Colourless samples will result in an UVVis spectrum showing no absorption maxima (linear spectrum).
- Coloured samples will result in an UVVis spectrum showing absorption maxima in a defined range. Figure 2 illustrates the absorbed colour on the UVVis spectrum and its complementary observed colour.

Figure 2: UVVis spectroscopy: Observed color

Visible color

5. REPORT CONTENT

The report should contain the following information. RecyClass provides a template to summarize the results of the QT test procedure, that can be found in Annex of this document.

- Reference to the Procedure: Quick Test Procedure for Bleeding Inks Printed on HDPE and PP Containers v.1.0.
- Date and place of the test
- A full and complete identification of the material tested (Annex Table 1), including:
 - Inks: type, amount (gsm and wt%) and colour. Use of lacquers and/or varnish must be specified.
 - If printed label: size, structure, density, and adhesive used (amount by surface unit and technology type: Wet labelling adhesive, Pressure sensitive adhesive, or non-pressure sensitive hotmelt).
 - If printed sleeve: size, structure, and density.
 - If IML: size, structure, and density.
- Description of the samples during each step (especially on colour changes, haze, deposits, sinking or non-detached label fragments, residual stickiness) (Annex Table 4).
- 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 and moisture content (Annex Table 2).
- In the case of a quantitative assessment: Details of the settings for the UVVis-spectrophotometer, the spectra for the reference, and the filtered wash water solutions measurements (Annex Table 3).
- Results of the QT procedure compared to the assessment criteria (Annex Table 5).

DOCUMENT VERSION HISTORY

VERSION	PUBLICATION DATE	REVISION NOTES
1.0	January 2022	Quick Test Procedure for Bleeding Inks applied on HDPE & PP Containers release
1.1	September 2022	Addition of the Annex at the end of the document

ANNEX - RESULTS SUMMARY

1. STRUCTURE OF THE PRINTED PACKAGING

Please report the details of the printed packaging assessed with the QT procedure and a photograph of the printed container.

Table 1: Structure of the printed packaging

	Table 1. Structur	e of the printed packaging
Packaging <mark>XX</mark>		
	Туре	
Inks	Amount (gsm and wt%)	
	Colour	
	Туре	
Lacquers/varnish (if any)	Amount (gsm and wt%)	
· · · · · · · · · · · · · · · · · · ·	Colour	
	Size	
	Structure	
Printed labels (if	Density	
any)	Adhesive used	
	Wet labelling adhesive / PSL /	
	non-pressure sensitive hotmelt	
Printed sleeves	Size	
(if any)	Structure	
	Density	
	Size	
IML (if any)	Structure	
	Density	
Photograph of the printed container		



2. SUMMARY OF THE RESULTS

Please report below the results obtained during the testing of the printed packaging.

Table 2: Evaluation of the flakes, pH and moisture content

Packaging <mark>XX</mark>			
Step	Variable	Control value	Printed value
1. Containers cutting and	Total weight of flakes	X.xx g	X.xx g
flakes characterization	Number of flakes		X
2. Washing	Initial pH value of the water	X	X
2. Washing	pH-value of the wash water	X	X
4. Drying	Moisture content of samples	Х%	X%
8	Total weight of flakes after drying	X,xx g	X,xx g
Other relevant parameter			

Table 3: Optional UWis-spectrophotometer quantitative assessment

Packaging XX				
UVVis-				
spectrophotometer				
settings				
Reference spectra				
Water solution spectra				
(washing step)				
Water solution spectra				
(flotation step)				

Table 4: Step-by-step description & Photographs

Declaration W					
Packaging XX					
Step	Description of the samples (colour change, haze, deposits, sinking, label fragments, residual stickiness)		Photographs		
Containers cutting and flakes	Control				
characterization	Printed				
2. Washing	Control				
2	Printed				
3. Floatation	Control				
3. Houtation	Printed				
4. Drying	Control				
	Printed				

Please report below any other remarks and observations that may help understanding the testing of the printed packaging:

3. PRINTED PACKAGING ASSESSMENT

Please report below the final assessment of the printed packaging tested with the QT procedure.

Table 5: Assessment criteria

Packaging XX					
Assessment criteria	Benchmark recommendation	Results	Fail / Pass		
Visual inspection of the water	No noticeable discoloration for both washing water and flotation water		✓ ×		
Visual inspection of the flakes	No noticeable discolouration of the flakes after drying		✓ ×		

c/o Plastic Recyclers Europe Avenue de Broqueville 12 1150 Brussels – Belgium

Phone: +32 2 786 39 08 info@recyclass.eu

www.recyclass.eu