QUICK TEST PROCEDURE FOR BLEEDING INKS

PRINTED ON PE & PP FILMS

STANDARD LABORATORY PRACTICE

VERSION 1.0 PUBLISHED ON JANUARY 2023

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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 recognised 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 PE & PP Films" 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 PE or PP flexible packaging are compatible with the post-consumer PE or PP recycling stream.

The Procedure evaluates printed flexibles, and labels 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 assessment.

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

² Recyclability Evaluation Protocols

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 PE or PP flexible packaging solutions. Prior to initiating the evaluation, the Applicant is required to review the Design for Recycling Guidelines for PE and PP Films⁴ in order to confirm that the material is compatible with these requirements.

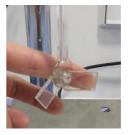
The following materials are covered by the scope of this Procedure:

- 1. Direct printed PE flexible packaging
- 2. Direct printed PP flexible packaging
- 3. Printed labels applied on PE or PP flexible packaging
- 4. Paper decoration applied on PE or PP flexible packaging

The procedure provides guidance on the behaviour of inks during the PE and PP recycling process. The inks should not bleed during the washing and flotation steps, remaining on the film, 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). See picture.
- Overhead stirrer with freely selectable rotation speed
- Magnetic Stirrer
- Analytical balance with an accuracy of 0,0001 g
- Strainer (mesh size of 0,5 x 0,5 mm)
- Oven
- Moisture Analyzer (res. 0.001 %)
- Cellulose filters with a pore size of 12-25 μm (e.g. Whatman™ Ashless Filter Paper No. 589/1 filter)
- UVVis-spectrophotometer (optional)



⁴ RecyClass Design for Recycling Guidelines

4. LABORATORY TEST METHODOLOGY

This methodology aims to reproduce the washing and flotation steps of the recycling process at a small scale to determine the suitability of inks applied on packaging for the PE or PP flexible recycling stream. The methodology described above 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 lab to explicit the results. Any remarks during following the Procedure shall be also noted down. An Annex to this document is also available at the end of the document 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.

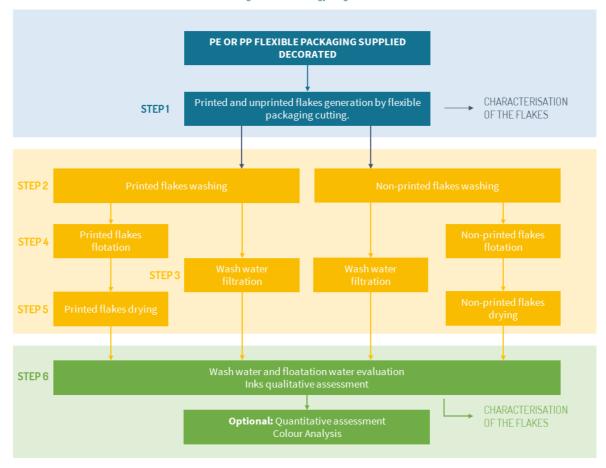


Figure 1: Methodology Diagram

4.1 SAMPLES SELECTION

The control film for use following the Procedure must be the same unprinted film as the printed one. For the purpose of the tests, the Applicant should provide at least 1kg of films. If the printed area covers a large part of the film (>50%), a larger amount of films may be needed to obtain a sufficient amount of control material from the unprinted areas. More printed 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.

4.2 PROCEDURE

4.2.1 STEP 1: FILMS CUTTING & FLAKES CHARACTERIZATION

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

Procedure:

- Cut out with scissors the printed parts of the films. 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 films. 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 films. Record the total weight of the printed flakes as well as the control flakes as "A" (See Annex Table 2).
- Document the printed and control flakes with one or more photographs.
- Count and record as "B" the number of printed flakes.

4.2.2 STEP 2: WASHING

At the state of the art, European PE and PP recycling lines typically use cold washing conditions, no detergents nor strong chemicals. The following procedure must be utilized for both control and printed flakes, separately.

Procedure:

- Prepare the washing solution, that is tap water, in a beaker (50 flakes in 120 mL water in a 600 mL beaker) at room temperature (20-25°C). Determine the current pH-value of the water (Annex Table 2). Do not add detergents or caustic soda.
- 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 10 min with a paddle stirrer.
- Save the wash water solution
- Rinse each sample at the same ratio with 500 rpm for 5 minutes.
- Determine the pH-value of the wash water (Annex Table 2).

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: FILTRATION

Following the washing, the filtration step is present to identify the presence of particles in suspension within the wash water solutions. The following procedure must be utilized for both wash water used for control and printed flakes, separately.

Procedure:

- For wash water used for printed flakes only: filter the wash water using a cellulose filter with a pore size of 12-25µm (e.g. Whatman™ Ashless Filter Paper No. 589/1 filter).
- Collect the residues and save for visual evaluation
- Take a photograph of both filtered 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)
- For the quantitative assessment, save few millilitres of the filtered solutions.
- Report any noticeable change of colour or transparency

4.2.4 STEP 4: 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 (50 flakes in 120 mL water in a 600 mL beaker).
- Put the wash of the labelled flakes in the water and stir at 500 rpm for 10 min.
- Remove the tank from the magnetic stirrer. Collect all the materials that float at the surface with a sieve.
- Collect separately all the particles that sink
- 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).

4.2.5 STEP 5: 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:

- Heat the oven to 80°C.
- Weigh the dish with the trial material before introducing it in the oven.
- As soon as the oven has reached 80°C, the trial is added to the oven for 12 hours.
- Weight the material after 6 hours in the oven. Weight the material at the end, after 24 hours in the oven.
- Evaluate the moisture content with a moisture analyser (Annex Table 2).
- Store the dried flakes for 24 h.

4.2.6 STEP 6: FLAKE SEPARATION & 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.
- Observe the eventual residues obtained after filtration. Note any small particles that may have come from the label or its inks.

<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.
- No residue is noticed after water filtration.

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:

Visible color

- 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

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 PE and PP Films 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).
- 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 2023	Quick Test Procedure for Bleeding Inks applied on PE & PP Films release

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 film.

Packaging XX Type Inks Amount (gsm and wt%) Colour Type Lacquers/varnish Amount (gsm and wt%) (if any) Colour Size Structure Printed labels (if Density any) Adhesive used Wet labelling adhesive / PSL / non-pressure sensitive hotmelt Photograph of the printed film

Table 1: Structure of the printed packaging

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. Films cutting and flakes	Total weight of flakes	X.xx g	X.xx g
characterization			Х
2. Washing	Initial pH value of the water	X	X
2. Washing	pH-value of the wash water	Х	X
4. Drying	Moisture content of samples	Х%	Х%
	Total weight of flakes after drying	Х,хх д	Х,хх д
Other relevant parameter			

Table 3: Optional UVvis-spectrophotometer quantitative assessment

Packaging <mark>XX</mark>
LIVA/io
UVVis-
spectrophotometer
settings
D. (
Reference spectra
Water solution spectra
(washing step)
Water solution spectra
(flotation step)
(notation step)

Table 4: Step-by-step description & Photographs

Packaging XX

Step	Description of the samples (colour change, haze, deposits, sinking, label fragments, residual stickiness)		Photographs	
Films cutting and flakes	Control			
characterization	Printed			
2. Washing	Control			
2	Printed			
3. Filtration	Control			
	Printed			
3. Floatation	Control			
	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		✓ X		
Filtration of the water	No noticeable residue on the filter		✓ X		

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