

SOP - Assessment protocol of the releasability of plastic labels from rigid PP and PE packaging in the recycling process



NTCP

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This is the revised version of the protocol developed for KIDV which includes hot washing, published on 1 July 2024.



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1 Introduction

Commissioned by the Netherlands Institute for Sustainable Packaging, The National Test centre of Circular Plastics (NTCP) has created a protocol which is designed to assess the behaviour of label releasability in cold and optionally hot water, simulating a step in the recycling process. It has been established to provide guidelines on how to measure, with a simple procedure, the adhesive residues and labels stuck on plastic packaging after it has gone through the shredding and washing processes. According to the description of KIDV Recycle Check a plastic packaging is divided into cap, label and container with the container being the main component of the packaging. To determine the label releasability the following conditions should be met: the label should detach from the packaging, and there should be no glue residues on the container. The adhesive layer should preferably remain on the label or otherwise be dissolved in the water. By using the procedure in this protocol, it is not necessary to assign a trained or experienced professional, or use specialized equipment. As this protocol contains a simple but detailed procedure, an explanation on how to interpret the results, and several tables for filling in key parameters. This test can therefore be executed by an internal laboratory or by an independent test laboratory. The results of this experiment will provide insight into the evaluation and quantification of the effect of label releasability in the washing process with cold and by extend hot water. Please note, that the results of these experiments cannot be used for certification, for a full assessment or questions please contact NTCP.

2 Scope

The scope of this test protocol is the release of glued plastic labels from rigid PP and PE packaging.

When adhesives are used to attach labels to packaging, recyclers attempt to remove the labels and adhesive from the packaging before it is recycled. For rigid PP and PE packaging with plastic labels this is done by washing the shredded packaging with cold water and if necessary/preferred hot water and chemicals. Four situations, or combinations thereof, can occur.

1. The label with adhesive remains on the container.
2. The label is released from the container and the adhesive remains on the label.
3. The label is released from the container and the adhesive remains on the container.
4. The label is released from the container and the adhesive dissolves in the washing water.

This protocol is designed to identify and quantify the four situations during the following steps of the label releasability process:

- Shredding of container material containing glued plastic label material;
- Cold washing of the shredded material;
- Optionally an additional hot wash of the cold washed material.

3 Material

To perform the protocol, the following materials are needed:

- Digital camera/Phone
- Heavy duty scissors (e.g. metal cutter)
- External micro meter ($\pm 10 \mu\text{m}$)
- Ruler
- Laboratory scale ($500 \text{ g} \pm 0.1 \text{ g}$)
- Shredder able to shred plastic material to 20 mm flakes (standard industrial size). For example, the Shini granulator model SG-3048E used by NTCP.
- Cylindrical laundry net (net for washing clothing with two nets, with approx. dimensions of height=12.5 cm, diameter=6.5 cm), this net must be filled with approx. 30% of the maximum volume of the net. The inside mesh size should be between 0.5 and 1 mm. The outside mesh side should be bigger than 1 mm.
- Washing machine that can control temperature and water volume ($T = 30 \text{ }^\circ\text{C}$ and $V = 12 \text{ L}$).
- Microscope 250x magnification
- Packaging with one label applied in a representative way; preferably samples from the production line (5-15 pcs.).
- Attached Excel sheet to fill out the obtained data and to execute relevant calculations. (NTCP_SOP_2021.001.v03_KIDV_Cold&Hot_Label_Releasability calculation sheet)

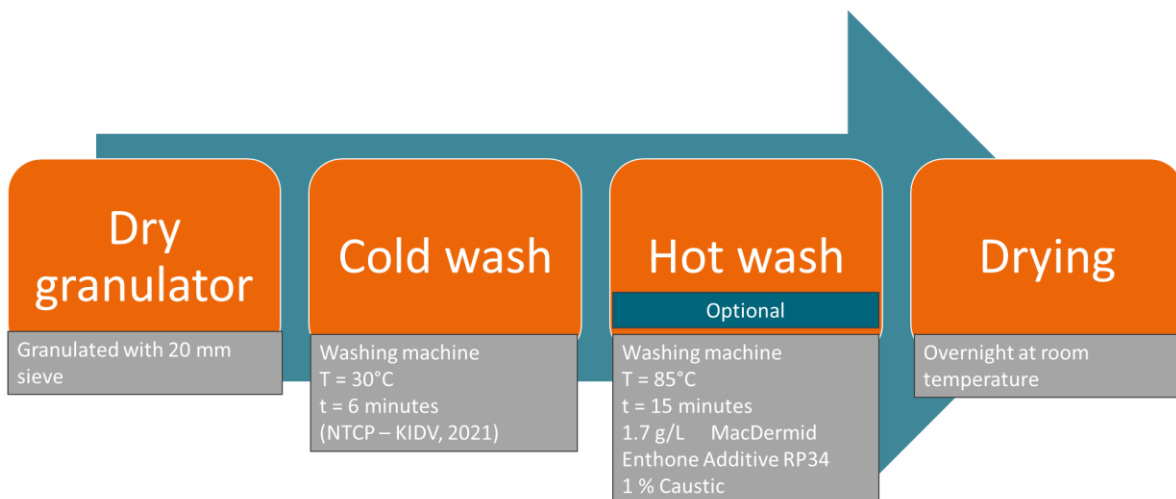
Additional hot wash materials

- Measuring Beaker
- MacDermid Enthone Additive RP34
- Sodium Hydroxide (NaOH) 30%

4 Washing Programs

The Full description of the used washing programs can be found in Appendix A.3/4

5 Overall procedure and parameters



6 Experimental procedure

The protocol was established according to the following steps (See Figure 1), each of these steps will be explained in detail after the list of steps:

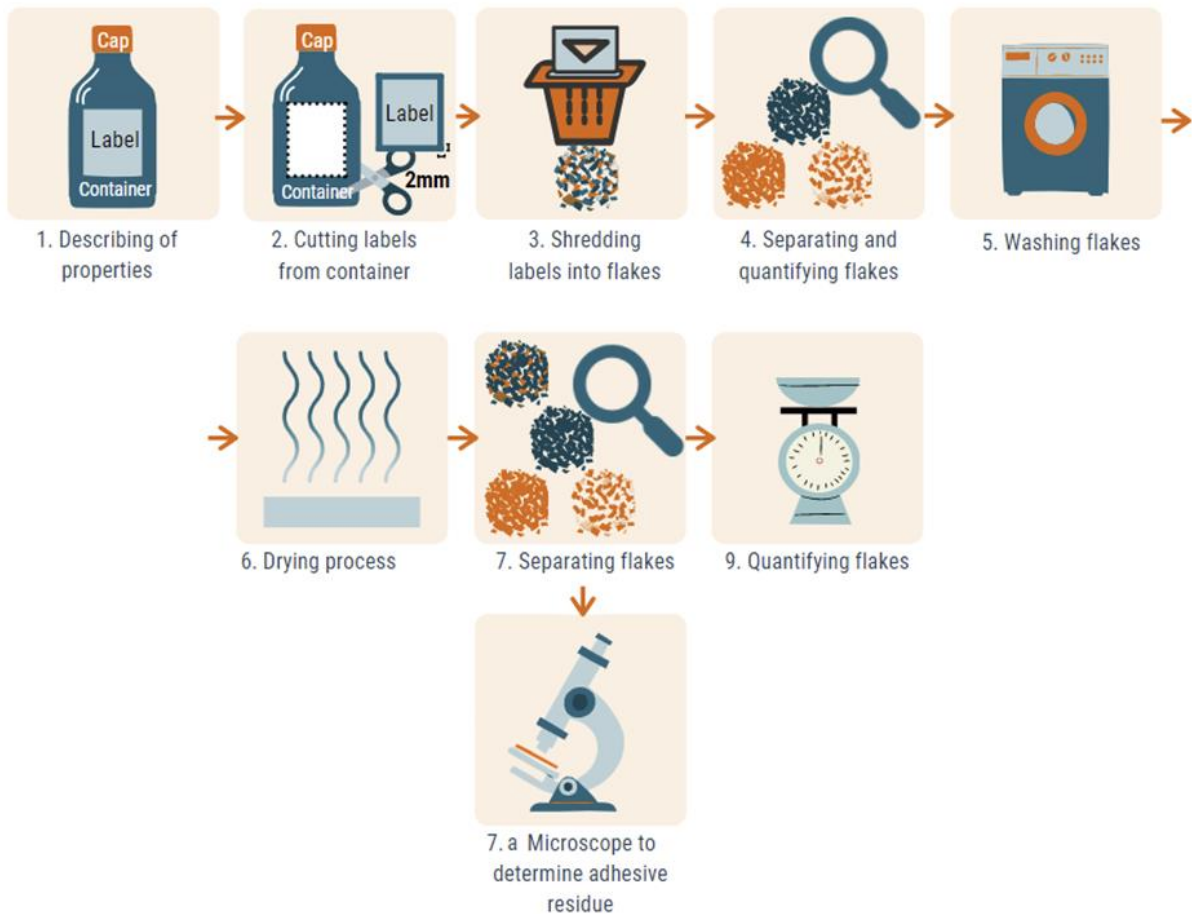


Figure 1 Schematic representation of the label release protocol.

As this protocol mimics the washing step of the recycling process at small scale, it takes into account that the labels are applied mechanically and not by hand. It is important to test one combination of packaging, adhesive and label at the time to prevent mix-ups. A detailed description of each step is described below. The obtained data from these steps can be filled out in the attached Excel file, which executes the needed calculations automatically.

Note: It is possible to combine the cold and hotwash in one cycle, if cold wash label releasability is not of interest, by skipping step 6, and 7 and therefore performing the hotwash straight after the cold wash.

Step 1. Container properties:

1. Determine the packaging properties following Table 1 in the Excel file attached to the protocol. Either by visual inspection and/or by extracting information from the suppliers specification sheet.
2. Take a picture of the packaging with the label. Peel off the protective layer of an individual label and take a picture of the side where the adhesive was applied, This can be applied uniformly over the surface area or in a specific pattern.
3. Capture the results in Table 1 in the attached NTCP adhesives protocol Excel file.

Step 2. Cut the label from the container:

4. Cut the container with label using scissors or cutters. It is important to cut around the label leaving a ± 2 mm gap to avoid pressing the label onto the packaging (Figure 2). To realistically shred the material ± 100 g (approximately 15-40 panels, consisting of container + label) is needed.

Note: For a reproducibility test, it is needed use the same mass per sample.

5. Take a picture of the sample and record the mass using a tray and assign it with "m_cutpanels".

Note: Do not write on the sample.

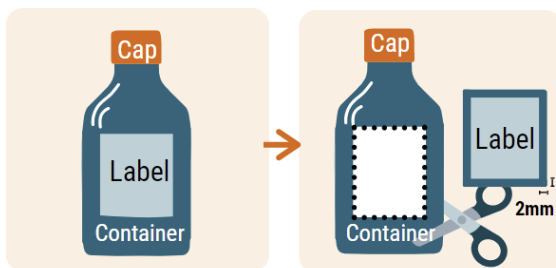


Figure 2 Procedure to cut the container material containing the label.

Step 3. Shredding process:

6. Shred the sample "m_cutpanels" to 20 mm flakes (this size is used as the standard industrial size) in approximately 5-10 min.
7. Weigh and take a picture of the sample after shredding, name this sample "m_shred".
8. Collect the material losses from the shredder machine and sieve, weigh these losses and name it "m_shredloss".

Step 4. Flake separation and quantification of the shredding process (Effect of the shredding process):

9. Separate and quantify the shredded sample "m_shred" according to Table 2 in the attached NTCP adhesives protocol Excel file a representation of the various categories can be found in **Appendix A.2**.
10. Take the collected losses "m_shredloss" and separate and quantify it according to Table 3 in the attached NTCP adhesives protocol Excel file.

Step 5. Cold washing process:

11. Carefully, put the shredded sample “**m_shred**” into the laundry net. The amount of sample must be 30% of the total net volume as shown in Figure 3.



Figure 3 30 % filled laundry net.

12. Place the laundry net filled with “**m_shred**” in the washing machine, set the water volume to 12 Liter, the temperature at 30 °C, and total washing time/residence time for the sample to 11 minutes. **The full washing program can be found in Appendix A.3**

Step 6. Drying process:

13. Carefully remove the flakes from the laundry net and spread them as much as possible on a flat surface. If the container and labels are sticking together do not detach by hand.
14. Dry the sample overnight at room temperature.
15. Take a picture of the sample on the tray and weigh the sample, name it “**m_wash_dried**”.

Step 7. Flake separation and quantification of the cold washing process (Effect of the cold washing process & mass balance closure):

16. Separate and quantify the washed sample “**m_wash_dried**” according to Table 4 in the attached NTCP adhesives protocol Excel file.
17. Visually inspect the container flakes on residual adhesive. If there is any adhesive on the container flake, it can be recognized by labels sticking to the container flakes, flakes sticking to fingers during handling, or light reflecting differently from the flake.

Step 7a. Optional microscope check

18. When in doubt, additional check can be performed by microscope. Place the flakes under the microscope to assess whether any adhesive is still present on the plastic container flake as shown in Figure 4.
19. Take a picture of the microscope measurement of each doubtful flake.

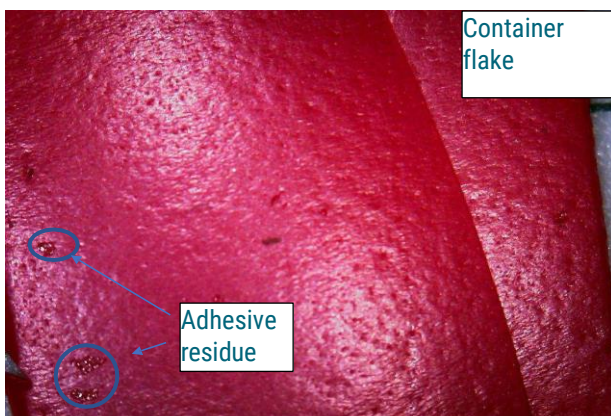


Figure 4 Picture of a sample taken by the microscope.

Step 8. Optional Hot wash

20. Carefully, put the cold washed sample "**m_wash_dried**" (or "m_wash" if step 6,7 were skipped) back into the laundry net. The amount of sample must be 30% of the total net volume as shown in Figure 3.
21. Dose 20 mL of detergent (MacDermid Enthone Additive RP34) and 250 mL Caustic (NaOH 30%) into the detergent compartment of the washing machine.
22. Place the laundry net filled with "**m_wash_dried**" in the washing machine, set the water volume to 12 Liter, the temperature at 85 °C, and total washing time/residence time for the sample to 15 minutes. **The full washing program can be found in Appendix A.4**

Step 8a. Hot wash Drying process:

23. Carefully remove the flakes from the laundry net and spread them as much as possible on a flat surface. If the container and labels are sticking together do not detach by hand.
24. Dry the sample overnight at room temperature.
25. Take a picture of the sample on the tray and weigh the sample, name it "**m_hot_dried**".
26. Unhide row 94-109, and 130 in the calculation sheet and fill in "**m_hot_dried**" mass.

Step 8b. Flake separation and quantification of the hot washing process (Effect of the hot washing process & mass balance closure):

27. Separate and quantify the washed sample "**m_hot_dried**" according to Table 5 in the attached NTCP adhesives protocol Excel file.
28. Visually inspect the container flakes on residual adhesive. If there is any adhesive on the container flake, it can be recognized by labels sticking to the container flakes, flakes sticking to fingers during handling, or light reflecting differently from the flake.

Step 9. Final Calculation

29. Use the formulas from chapter 6 to quantify the total mass of the label releasability (Table 6) and the percentage of label releasability (Table 7) in the attached NTCP adhesives protocol Excel file.

Threshold: If the efficiency of Label releasability is > **90 %** the label is releasable.

7 Calculation

The label releasability after shredding for each sample is calculated using the following equation in the attached NTCP adhesives protocol Excel file:

$$\text{Label releasability after shredding} = \frac{(\text{container flakes}) + (\text{label flakes})}{(\text{shredded sample}) - (\text{fines})}$$

The overall label & adhesive release label releasability is calculated in the attached NTCP adhesives protocol Excel file as:

$$\text{Label releasability after washing} = \frac{(\text{container flakes}) + (\text{label flakes})}{(\text{dried sample}) - (\text{fines})}$$

The overall label & adhesive release label releasability after hot washing is calculated in the attached NTCP adhesives protocol Excel file as:

$$\text{Label releasability after hot washing} = \frac{(\text{container flakes}) + (\text{label flakes})}{(\text{hot dried sample}) - (\text{fines})}$$

8 Test report

The test report must contain the following information:

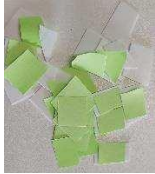



- Date, place and the name of the person/company who performed the protocol.
- Reference to this protocol (This protocol cannot be used as a certificate).
- Filled the grey cells in the tables 1, 2, 3, 4, 5 and 6 in the attached NTCP adhesives protocol Excel file. In addition, in the Appendix a Table contains the letters, the definition and, the formula's used in the protocol.
- The photos in the tables must have a reasonable quality and visibility.
- Remarks on observations during the process.

Appendix

A.1 Table of abbreviations

shortform	Definition
m_cutpanels	Cut panels (not shred)
m_shredloss	Losses after shredding
m_shred	Sample after shredding
m_shred_con+lab	Container + label flakes after shredding
m_shred_lab	Label flakes after shredding
m_shred_con	Container flakes after shredding
m_shred_con+glue	Container with glue flakes after shredding
m_shred_clog	Clogged flakes after shredding
m_shred_fines	Fines after shredding
m_shred_con+lab	Container + label flakes losses after shredding
m_shredloss_lab	Labels flakes losses after shredding
m_shredloss_con	Container flakes losses after shredding
m_shredloss_con+glue	Container flakes with glue flakes losses after shredding
m_shredloss_clog	Clogged flakes losses after shredding
m_shredloss_fines	Fines losses after shredding
m_wash_con+lab	Container + label flakes after washing
m_wash_con	Container flakes after washing
m_wash_lab	Label flakes after washing
m_wash_clog	Clogged flakes after washing
m_wash_con+glue	Container with glue flakes after washing
m_wash_fines	Fines after washing
Eff_shred	label releasability efficiency of the shredding
Eff_overall	label releasability efficiency of the overall protocol of shredding and washing
m_wash_dried	Total after drying and after washing

A.2 Example of material classification.

Quantification of the flakes type after shredding	Reference in the procedure	Picture
Shredded sample	m_shred	
Container + labels flakes	m_shred_con+lab	
Label flakes	m_shred_lab	
Container flakes	m_shred_con	
Container with glue flakes	m_shred_con+glue	
Clogged flakes (flakes that are stuck together)	m_shred_clog	
Fines	m_shred_fines	

A.3 Washing Program Cold wash

5 - <3kg Cw Delab					
1 Pre-wash					
Temperature [Celsius]	30	RPM	46 RPM	Inlets	1,2,5,6
Level	9				
Time [min]	2 min			Signal	No
On time [sec]	12 sec				
Off time [sec]	3 sec	Detergents	None		
Drain					
RPM	46 RPM	Drain valve	Valve 1		
Time	0.5 min				
2 Rinse					
		RPM	46 RPM	Inlets	1,2
Level	9				
Time [min]	2 min			Signal	No
On time [sec]	12 sec				
Off time [sec]	3 sec	Detergents	None		
Spin					
RPM	250 RPM	Drain valve	Valve 1		
Time	1 min				
3 Final rinse					
		RPM	46 RPM	Inlets	1,2
Level	9				
Time [min]	2 min			Signal	No
On time [sec]	12 sec				
Off time [sec]	3 sec	Detergents	None		
Spin					
RPM	720 RPM	Drain valve	Valve 1		
Time	2 min				

A.4 Washing programs Hot wash

6 - <3kg Hw delab.

1 Wash					
Temperature [Celsius]	85	RPM	46 RPM	Inlets	3,8
Level	9				
Time [min]	15 min			Signal	No
On time [sec]	12 sec				
Off time [sec]	3 sec	Detergents	None		
Drain					
RPM	46 RPM	Drain valve	Valve 1		
Time	0.5 min				
2 Rinse					
Level	9	RPM	46 RPM	Inlets	1,2
Time [min]	2 min				
On time [sec]	12 sec			Signal	No
Off time [sec]	3 sec	Detergents	None		
Spin					
RPM	250 RPM	Drain valve	Valve 1		
Time	1 min				
3 Final rinse					
Level	9	RPM	46 RPM	Inlets	1,2
Time [min]	2 min				
On time [sec]	12 sec			Signal	No
Off time [sec]	3 sec	Detergents	None		
Spin					
RPM	720 RPM	Drain valve	Valve 1		
Time	2 min				