

## CHI-C8-SEPPET

### Label and Adhesive Separation Test for PET-based Packaging

Version 5.0 / December 2022

This CHI test method was developed by cyclos-HTP for the evaluation of the separation of labels and adhesives in lab scale. The results can be used to assess the recycling compatibility of adhesives or pressure-sensitive labels or other adhesive applications on PET packaging with the state-of-the-art recycling process as reference.

The testing procedure comprises the following steps:

#### 1. Materials

- 1.1. Sample of PET with a pressure-sensitive label and an adhesive application with unknown recycling compatibility
- 1.2. Granulator to grind the samples into flakes
- 1.3. Sodium hydroxide (NaOH) for the preparation of a caustic solution
- 1.4. Surfactant (washing tenside) specified for cleaning solutions in PET recycling, selected and released by cyclos-HTP institute
- 1.5. Attrition cell with stirring blades and heater able to reach high forces and accurate heating to 80°C (see Figure 1)
- 1.6. Sieve with pore size < 2 mm
- 1.7. Buchner-funnel and equipment for vacuum filtration
- 1.8. Air drying oven, capable of heating up to 220°C
- 1.9. Optional: Photo Spectrometer with CIE L\*a\*b\* software or photometer capable to measure the transmission rate of visible light.

#### 2. Preparation of samples and wash solution

- 2.1. Cut or grind the packaging sample (label/adhesive/PET) into flakes with edge lengths of approx. 10-15 mm
- 2.2. The samples before and after cutting must be documented photographically
- 2.3. The sample density incl. PET and label in the wash solution should be  $\geq 100$  g per litre to reach high stock density and high forces. The corresponding sample quantity is prepared.
- 2.4. Prepare an alkaline solution containing distilled water, NaOH and 0.3% by weight surfactant (washing tenside)
- 2.5. Add NaOH until the conductivity of the washing solution is between 110 and 115 mS/cm. The conductivity of the washing solution should be min. 110 mS/cm and the NaOH concentration should be between 1.3% and 1.7%.
- 2.6. The pH value of the alkaline solution should be preferably between 12 and 13

#### 3. Test procedure

- 3.1. Measure the quantity of alkaline solution (100g /litre) in accordance with the quantity of the sample and heat it to 80°C without sample.
- 3.2. After the test temperature is reached, the sample is carefully added to the solution. The hot wash procedure is started.
- 3.3. The batch with the sample is stirred at 80°C for 15 minutes with a special configuration of the blades, so that the lower blades push the liquid upwards and the upper blades push downwards when stirring. (See figure 1), this achieves higher shear forces and turbulences, comparable with the real recycling process. The stirring speed is adjusted at 600 rpm so that the flakes undergo a certain amount of shear.
- 3.4. After 15 minutes the washing procedure is stopped. Check if the labels have detached from the PET.

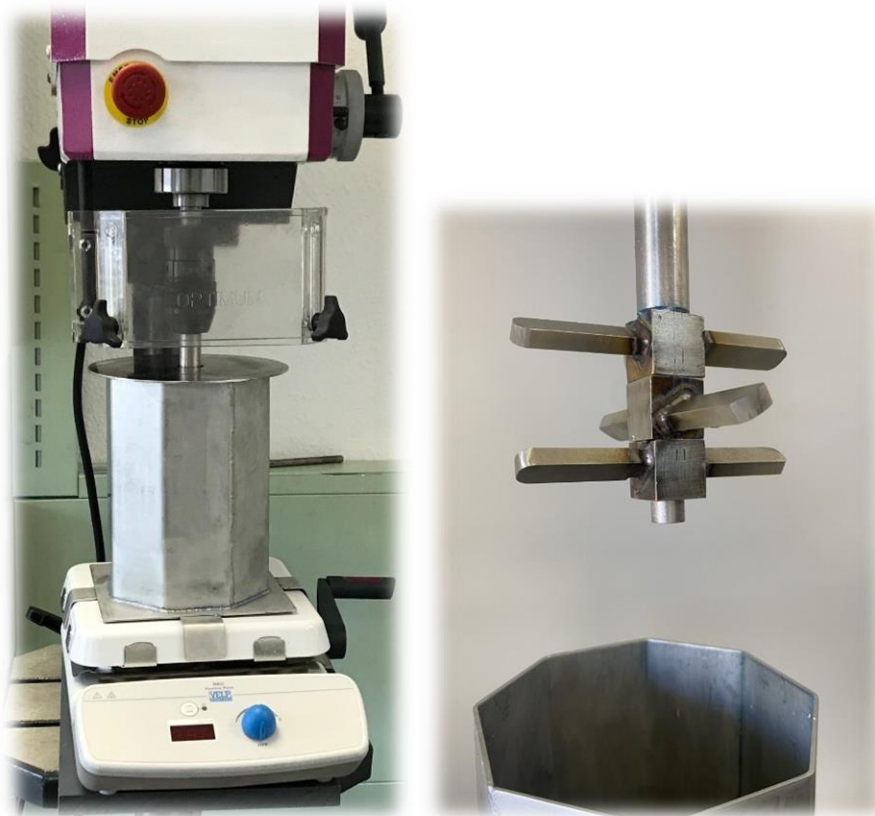
- 3.4.1. In case of PO-based labels, the label flakes should float and the PET flakes should sink to the bottom.
- 3.4.2. In case of paper-based labels, the fibrous material should be mostly disintegrated and the loose fibres should float or levitate in the liquid.
- 3.5. Carefully and completely remove the detached label material and wash it once under running water; observe whether all label pieces have detached from the PET.
- 3.6. If not all the label pieces have detached from the PET, the test for this sample is stopped and repeated if necessary; if the result is confirmed, no recycling compatibility can be testified for the respective sample or the adhesive used.
- 3.7. The PET flakes are sieved, rinsed twice with approx. 200 ml of clear water and after draining transferred to a container
- 3.8. (optional) If the adhesive has only been detached and not dissolved, the PET flakes must be washed carefully with water:
- 3.9. Collect sinking PET flakes in a Buchner-funnel, filter by vacuum without paper filter and let water flow on it.
- 3.10. Collect sinking PET flakes in a beaker and stir vigorously by hand, carefully tilt the washing solution with floating adhesive.
- 3.11. A part of the washed and sinking PET flakes is kept for the visual comparison with the final samples after the oven roasting test
- 3.12. The PET flakes are roasted in a preheated oven at 220°C for 60 minutes; the tray with the sample is then cooled in a desiccator.
- 3.13. A part of the roasted PET flakes is kept for the visual comparison with the washed flakes before the oven test.
- 3.14. The samples from the steps 3.11 and 3.13 are compared and documented photographically; colour changes from the original sample, especially in the case of dark brown discolouration (see examples in Figure 2 and Figure 3), are documented.
- 3.15. By demand or to fulfil customer specifications the colour of the PET flakes can also be evaluated quantitatively:
  - 3.15.1. As the preferred method the samples are measured with a photo spectrometer using the CIE L\*a\*b\* method. PET flakes from the same sample without adhesives and labels should be used as reference.
  - 3.15.2. As an alternative method the greying of the PET can be evaluated by measuring the transmission rate of visible light with a photometer.

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#### Version history:

<i>Version No.</i>	<i>Date</i>	<i>Reason/Content of revision</i>
1.0	Oct 2019	First version of test method
2.0	Feb 2020	Updated test program and parameters
3.0	Aug 2020	Updated evaluation criteria; additional parameter settings; improved photo documentation
4.0	Sep 2021	Changes of test procedure
4.2	Feb 2022	Updated photo documentation
4.3	Feb 2022	Update for washing solution
5.0	Dec 2022	Changes of the test procedure, additional parameter settings

#### 4. Appendix



**Figure 1:** Attrition cell and stirring blades - equipment for washing tests



**Figure 2:** PET sample without discolouring after roasting (test passed)



**Figure 3:** PET sample with discolouring by remaining adhesive