European PET Bottle Platform

Quick Test QT 507

Bleeding label

Website version

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This test has been published by the European PET Bottle Platform and was devised by experts in the European plastics, packaging and recycling industries. The test is an indicative test only and is based on the best of our knowledge at the time of publication. It does not necessarily guarantee compliance with the different recycling schemes.. Users are therefore advised to discuss the results with the EPBP who will check for specific and up-to-date information.

The European PET Bottle Platform cannot accept responsibility or be held liable for any loss or damage arising out of or in connection with the test results, their accuracy, or incomplete or misleading conclusions.

1. Introduction

The objective of the European PET Bottle Platform (EPBP) is to evaluate technologies and products to allow new PET bottle innovations whilst optimizing the environmental and economic consequences for the recyclability of PET.

EPBP has formulated guidelines to evaluate the influence of bottle innovations - such as barrier materials, resin formulations, additives and non-PET components in or on PET bottles - on R-PET recycling processes. Barrier materials can be applied as a coating, introduced in a co-injected multilayer configuration or blended with the matrix material. Additives can be incorporated into the base material during polymerization or added during injection molding in the form of liquid or solid master-batches. Other non-PET components can be labels, glue, sleeves, caps, printings, etc.

Laboratory analyses on the recyclability of new innovative PET bottles or non-PET components can be relatively expensive and usually have a time delay between sampling and getting useable results of several months. Besides, assessing the test results is a difficult task that requires training and experience. This is often seen as an inconvenience.

For this reason, EPBP has developed a series of rapid and low-cost techniques for the quick assessment of PET bottles. All quick tests include a complete explanation of the scope, techniques, equipment and test conditions, as well as a “summary interpretation” explaining how to use the test results. Quick tests can either be executed at the internal laboratory facility or by an independent test laboratory with minimal investment in equipment.

The results of the quick tests are purely indicative, and may not be considered as an advice, a recommendation or a formal approval by EPBP. For a complete assessment, further tests are required to highlight all possible effects of innovative PET bottles on the recyclability of collected bottles into r-PET, the processing of the r-PET into products and the final product properties. Please contact EPBP for more information.

SAFETY PRECAUTIONS
This guideline is intended for use by qualified personnel who recognize safety hazards and are familiar with the safety precautions required in regard to application of this guideline. The appropriate laboratory safety procedures must be used before, during, and after testing operations.

2. Quick Test QT 507

Scope
Several systems can be used to label a PET bottle. The following test protocol is designed to provide guidance on the staining of the ink and/or the adhesives on PET bottles during the recycling process. Inks and label adhesives that leave remnants of the ink, label and/or adhesive should be avoided.

Principle
Label adhesives and inks are readily removed during the hot pre-wash of the recycleprocess. Bottles or flakes are put into a hot caustic solution, where the adhesives break theirbond with the PET surface detaching the labels from the PET bottles and inks may be dissolved and dispersed. The efficiency of the adhesive/ink removal can be determined by the mass loss for the test sample. The chemical resistance of the adhesive or ink can be measured by the level of discoloration of the caustic solution and by the staining of flakes inthe wash solution. As this process is a simulation of the washing process, the quality of the flake may improve as the wash residence time is averaged at 45min. Proof of label quality will be checked by the chemical stability of the inks on the label and by the colour of the extract solution and flakes.

Apparatus
- 2 Beakers of 800 ml, The Beaker internal diameter (id) should be 9 cm.
- The slurry height to diameter ratio is about 0.8
- Hot plate, or similar equipment capable of heating up to 90°C
- 125 mm watch glass to cover beaker when heating
- Clamping device to install overhead stirrer.
- Overhead 600 rpm stirrer
- Thermometer
- Sieve, non-aluminum, fine mesh
- 2% caustic soda solution
- Whatman™ Ashless Filter Paper No. 589/1
- Distilled or deionized water
- Color measurement: Minolta spectrophotometer 3500, 3600, or equivalent.
- Oven to dry the PET flakes

Short Test Description
The test label(s) is circulated with PET flakes in a hot caustic wash solution for 15mins. The test is stopped and a small sample of the clear supernatant liquor is taken for liquid color testing. The remaining label, flake and wash solution is heated and mixed twice more and samples taken, giving 3 data points for the wash solution. At the end a rinsed flake sample is taken. Samples are compared to reference samples without labels.

Samples
Labels: Cut 200cm^2 labels into pieces of ~1cm^2. Test labels should have full ink coverage using dark ink.

PET flakes: approximately 100g of clear PET flakes are required as a control sample and 100g to be used for the test for discoloration along with the label in the caustic wash solution. The flakes must be sieved to ensure that particle size is greater than 3 mm; this willensure elimination of fines from the test.

Procedure

Important - test wash the control PET, the label and the test PET separately.

Control Sample Procedure

- Completely submerge 100g PET flakes control sample in 800 ml 2% caustic soda solution at 85°C, switch on stirrer to 500 rpm and stir for 15 minutes.
- Switch off the stirrer and allow the flakes to settle.
- Remove 30ml of supernatant liquor and retain for color measurement. Mark as “control solution 1”
- With the remaining PET flake and control solution repeat the test two more times for another 15 minutes at the same conditions and mark the samples “control solution 2” and “control solution 3” accordingly. Rinse control PET flake sample two times in 800ml stirred cold demineralized water for 3 minutes
- Dry the flakes at a maximum drying temperature of 105°C in an oven. Retain the dry control sample to be measured on the colorimeter, mark as “control flake”

Test Samples Procedure

- Completely submerge 100g PET flakes and 200cm^2 label samples in 800 ml 2% caustic soda solution at 85°C, switch on stirrer to 500 rpm and stir for 15 minutes.
  (Make sure that the labels do not stick to the stirrer, to the sides of the beaker or to each other, it is important to ensure free agitation of flakes and label particles).
- Switch off the stirrer and allow the test flakes/labels to settle.
- Remove the 30 ml of the clear supernatant liquor and mark it as “first solution”, retain for color evaluation.
- With the remaining PET flake/label test solution repeat the test for another 15 minutes at the same conditions.
- Switch off the stirrer and allow the test flakes/labels to settle. Remove 30 ml of the clear supernatant liquor and mark it as “second solution”, retain for colour evaluation.
- With the final PET flake/label test solution repeat the test for another 15 minutes at the same conditions.
- Switch off the stirrer and allow the test flakes/labels to settle. Remove 30 ml of the clear supernatant liquor and mark it as “third solution”, retain for colour evaluation. Retain the wash water. Rinse test PET flake/label sample two times in 800ml stirred cold demineralized water for 3 minutes and remove the labels. (Floating labels can be easily decanted from the rinsing solution; sinking labels may be removed after drying.)
- Dry the flakes at a maximum drying temperature of 105°C and retain the dry sample for colour evaluation, mark as “test flake”.

Evaluation of the PET flakes and the Wash solution

- Using Whatman™ Ashless Filter Paper No. 589/1 filter the remainder of the washsolution to recover any small particles that may have come from the label or its inks. Dry the residue and carefully remove any PET flake contamination

- The colour measurements of the PET flake and solutions need to be carried out using the same test conditions and should be repeated at least 5 times with separate and individual samples to provide statistical certainty. The report must specify all test readings (such as L*, a*, b*(or Hunter scale)) for all samples and an average should be calculated per sample

- The colour results of the control PET vs the label & PET washed flake should be compared per colour axis reading (L*, a* & b*, (or Hunter scale)). Absolute levels of the

b* and L* readings can lead to a direct rejection of the flake on colour quality. A b* reading over 3.0 tends to be too yellow. L* readings are much influenced by the thickness of the flakes and too low a value can create a darker color in an application, however no absolute value reading should be assumed at this point.

- The colour measurement of the wash solution is simpler, needing only a single pass through the colorimeter. The three colour axis readings should be determined for the "control", "first", "second" and "third" wash solutions. Specific larger deposits which may have been present in the solution should be noted as ink or label particles and can have an added effect on the colour of the solution. The colors of the ink and label should be compared to the maximum discoloration of the wash solution of the third wash to correlate the relation to its origin.

- The wash solution and filtered residues should be analyzed for presence of chemicals or heavy metals[1], which may have come from the ink. A suitable test method is atomic absorption (AA) or inductively coupled mass spectrometry (ICP-MS).

Results

There may not be any color difference between the control PET and the PET flakes/ labels following the 3 wash cycles. The color change trend of the wash solution from the "first" to the "third" may not show a large discoloration increase. Results of the color measurementsand the changes in a* or b* reading may be used to further validate the level of color bleeding.

The remainder of the wash water filtrate may be analyzed to show possible impact on the water treatment facility and the detect the presence of chemical substances or heavy metals which may have an impact of downstream cleaning and end applications requirements.

Test report

The test report includes the following information:

- Reference to the EPBP Quicktest QT 507 and its release date.
- A complete identification of the material tested.
- Description and detailed photographs of the samples before, during and after testing (especially on color changes, haze, deposits, etc.)
- The details of the settings for the colorimeter, its reference standards and the results of the PET flake and wash solution measurements.
- Details of any deviation from the test method, as well as any incident which may have influenced the results
- Listing the potential chemicals in the filtered residue of the wash solution
- Date and place of the test.

Remark

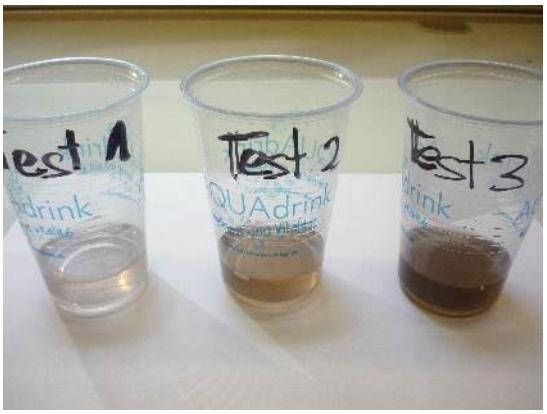
This quick test is designed as a quality indicator to monitor a single critical parameter in PET recycling. Other specific tests are needed to carry out a full screening for possible effects of innovation in PET products on the recyclability of collected bottles into rPET, the processing of rPET into products and final product properties. Please contact EPBP for more information.

[1] Lead, Hexavalent Chromium, Cadmium, Mercury

Photos

Photo 1: setup with sample immersed in caustic soda solution

Photo 2: Minolta spectrophotometer



Photos 3-4 Examples of labels for each of the 3 steps and the discoloration of the wash solution in 3 steps

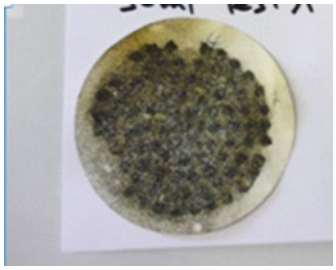


Photo 5 Example of filter residue after filtration of the 3rd step confirms the black label ink and solution color

Photo 6-8: Different effects of colour on the wash solution from 1st to 3rd step

Photo 5 Different colours are possible due to difference in label ink colours

Photo 6 Different examples of flake discolouration by different colours of label

Flow diagram of testing