European PET Bottle Platform

Quick Test QT508

Labels & Adhesives testing procedure

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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 PET recycling process. 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 lengthy (sometimes with lead times of several months between sampling and results. Assessing test results can in some cases be complex and requires a certain level of expertise, which can be considered inconvenient for early assessments

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” detailing how to use the test results. Quick tests can either be executed at the internal laboratory facilityor by an independent test laboratory with minimal investment in equipment.

Conclusions from quick tests results 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 QT508

Scope

Several systems can be used to label a PET bottle. The following test protocol is designed to provide guidance on the behavior of pressure sensitive label systems during the recycling process. The label should detach from the bottle and float and the adhesive layer should remain on the label without reactivation, with a minimal amount dissolved in the caustic wash and no adhesive remaining on the washed PET flake.

Adhesive reactivation is defined as the behavior of a hydrophobic adhesive that is releasable (i.e., stays on the label) and after the labels are dried (i.e., moisture<5%) the adhesive is again sticky, causing label flakes re-attachment/re-tackification on the PET flakes or equipment surface

This test protocol is meant to be carried out in combination with an oven test (refer to EPBP QT500).

Background

Pressure sensitive labels are applied to PET bottles with light pressure and without activation or heat. The surface of the label intended to come into contact with the bottle is covered with a layer of adhesive that forms a bond when pressure is applied. Labels are typically supplied in rolls, with a release liner that protects the adhesive and assists label handling.

The adhesive should be designed to allow the release of the label during the hot (pre)wash of the recycle process and to allow subsequent sink/float separation of label residuals from PET flakes. Ideally the adhesive:

- should remain onthe label when it detaches from the bottle,
- should leave no residues on the PET flakes which may lead to discoloration during the processing of recycled PET. The amount of adhesive dissolved into the process water should be reduced to a minimum, unless the adhesive, due to its chemical nature,
- is intentionally designed to be dissolved and not suspended into water/alkali without creating issues to the recycling process water treatment plant.
- Water releasable adhesives, instead, should remain on the label without reactivation.

Principle

The following quick test has been designed to assess the behavior of the adhesive when PET bottles with pressure sensitive labels applied are submitted to a simulation of the recycling process. The test is carried out using the bottles panel only, i.e., the section that the bottle the label is applied. This allows easier cutting with scissors, without generating fines.

Apparatus

- Digital camera
- Heavy duty (e.g., electrician) scissors
- Lab scale with an accuracy of 0.0001 g
- 250ml and 100 ml crystallizers
- 400 ml beakers.

- Hot plate stirrer, or similar equipment capable of heating up to 90°C
- Suitable diameter watch glass to cover beaker when heating
- Clamping device to install overhead 600 rpm stirrer.
- Impeller stirrer (preferred option). If an impeller stirrer is not available, a magnetic stirrer can be used, the magnet should be 4-5 cm long in order to provide adequate stirring
- Thermometer
- Caustic soda solution (2.0 % Sodium hydroxide)
- Distilled or deionized water (referred to as “distilled water”, below)
- Ceramic funnel with vacuum filtration
- Manual stirrer (i.e., glass rod)
- Filter paper
- Lab oven with air circulation, with a maximum temperature of 250°C
- Metal tweezers
- Acetone for cleaning purposes (technical grade)
- Metal weight with flat surface (150 g, max 5,5 cm^2)
- Clean white tile/toughened glass tile to be used as non-sticky, dry, even and free of dust and fibers solid surface

Short Test Description

Lab blown or commercial bottles can be used for the testing purposes. The procedures are defined below:

Procedure for commercial samples:

Samples
The commercial samples (ready to be delivered or already delivered to the retailer) are first cured for a minimum time of 21 days at room temperature or 5 days at 40°C or 3 days at 50°C..Sample panels (PET plus label) are cut from PET bottles and weighed on a lab scale. The sample panels are carefully cut into 10 mm square pieces using scissors to avoid generation of fines, and the pieces are transferred to the lab simulation of the recycling process.
The test should be carried out with labels with commercial print because this affects whether the labels are sinking.

Results and result analysis
After the process, the floating (label) and the sinking (PET) fractions are collected, dried and weighed on a lab scale with 0.0001 gr accuracy
The sum of the weights of the sinking and swimming fraction should be equal to the weight of the initial panels (PET with label)
If the sum of the weights of the sinking and floating fractions is lower than the initial sample weight, this is an indication that part of the adhesive (or other components) has dissolved into the process water.
The QT508 test is further complemented by an oven test (according to procedure QT500) to be performed on the sinking (PET) fraction, using an unprocessed sample as reference, to better highlight any glue residuals left on the panel.

Procedure for lab samples (standard bottles can be supplied by the accredited lab).

Sections of the PET bottle panels are cut and weighed on a lab scale with 0.0001 g accuracy. The sample pressure sensitive labels are then applied to the sample panels, allowing sufficient time for the adhesive to cure (minimum curing time 21 days at room temperature or 5 days at 40°C or 3 days at 50°C).The weight of the label is calculated by difference between the weight of the labelled panel and a control panel without labels. The sample panels (panel plus label) are carefully cut into10-12 mm square pieces using scissors to avoid generation of fines, and the pieces are transferred to the lab simulation of the recycling process. The test should be carried out with 100% solid-colored labels with commercial print because this facilitates identification of residual labels glued to PET flakes.

After the process, the floating (label) and the sinking (PET) fractions are collected, dried and weighed. The weight of the sinking fraction should be equal to the weight of the initial panels (without labels).There should be no residual label pieces on or at the edges of the PET flakes, indicating that all the labels have detached.

If the sum of the weights of the sinking and floating fractions is lower than the initial sample weight, this is an indication that part of the adhesive (or other components) has dissolved into the process water.

The QT508 test is further complemented by an oven test (according to procedure QT500) to be performed on the sinking (PET) fraction, using an unprocessed sample as reference, to better highlight any glue residuals left on the panel. This test is to be documented separately according to the QT500 procedure.

Samples

- PET Bottles
- Test Labels (100% solid colored)

Procedure

- Cut out the panel of the bottles until you reach a minimum surface of 600 cm^2 of covered surface by the label i.e., the part of the bottle where the label is applied. (sample “A”)
- Weigh the cut panels and add the panel weights (accuracy as defined above)
- Cut out an additional sample with the surface of 600 cm^2, approximately the same amount of the sample “A” (as defined in the first boolet point). This sample is meant to be put aside to be used as reference for the oven test.
- Report label size and structure, including the density and the amount of adhesive by surface unit (if known from supplier). Document the label color and design with one or more photographs.
- Apply a test label to each panel.
- Uncovered panel margins surrounding the applied label must have a max. width of 3 mm on each margin side.

- Weigh and record each labelled PET panel separately and sum the bottle walls weight. Record this weight as “B”.
- Allow labels to cure at room temperature for 21 days.
- Weigh and record each labelled PET bottle wall panel separately and sum the bottle walls total weight. Record the weight as “C”. Document sample with one or more photographs.
- Calculate the weight loss during curing and record it as “D”:

$D = B – C$

- A significant weight loss during curing “D” is an indication of the presence of volatile compounds in the adhesive. While this is not a parameter that will affect recycling per se, a significant weight loss is worth investigating.
- Calculate the total weight of the labels + adhesive and record it as “E”:

$E = C - A$

- Cut labelled PET bottle wall panel with scissors in a controlled environment. Avoid generating fines and the loss of non-labelled flaked sample. Flake samples should be approximately square and in the range of 10 mm. Check the weight after cutting to ensure that there are no losses (maximum weight difference +/- 0,02%). Document the sample with one or more photographs.
- Using the 400ml beaker, completely submerge the flakes in the caustic solution at 85°C. The ratio between the flakes and the caustic solution should be 40 ml of solution for each 100 individual flakes/100 cm^2 covered surface. Switch on stirrer to 500 rpm and leave 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. Record the process with a series of photographs. Any stickiness should be recorded.
- Observe the caustic solution colour after the test. If any noticeable discoloration, report it and document the caustic solution colour with a photograph. In order to highlight differences, take a sample of the solution (be careful not to include flakes) and place it into an appropriate container. Place alongside a reference sample of fresh (caustic solution in a similar container and take a picture of the two containers against a neutral background. Discoloration may indicate a potential ink bleeding issue. It may be worth considering a further investigation with a label bleeding quick test (see EPBP quick test QT 507 for reference)
- Collect the sinking and floating fractions by filtration on a ceramic funnel connected to a vacuum pump. Use small amounts of distilled water and a suitable tool to collect flakes on the bottom of the beaker and from the stirrer. Do not lose flakes in the transfer process.
- Carefully remove the flakes from the funnel and place them in a beaker containing distilled water at room temperature. The ratio between the flakes and distilled water should be 40 ml of solution for each 100 individual flakes/100 cm^2 covered surface. Gently stir the solution manually for about a minute, and allow to settle for 10-15 minutes. Ensure that no flakes or labels remain on the stirrer. When settling has occurred, document with one or more photographs.
- Carefully collect the floating fraction, place it on filter paper to allow gross removal of water and then transfer it into a crystallizer of suitable size. Qualitatively inspect the flakes for residual stickiness of the adhesive.

- Individually spread dried flakes with a clean metal tweezers on a clean white tile/toughened glass tile
- Evaluate residual stickiness by placing a metal weight (stainless steel) on the surface of ten randomly chosen different flakes and labels for 5 seconds each, without putting additional pressure. The metal pin surface which is placed in contact with the flake must be 1- 2 cm^2. The metal weight must have a flat surface and a weight of 150 g. If the adhesive is reactivating, the flake will stick on the surface of the metal weight once it is lifted. The weight should be cleaned with a slight amount of acetone after touching one flake before it is placed on the next one. The weight has to be totally dry and free of residual acetone before putting it on the next flake.
- Report the results as following:
    - Strong reactivation: more than 4 sticking flakes
    - Reactivation: 1 to 4 sticking flakes
    - No reactivation: 0 sticking flakes.
- Collect the sinking fraction by filtration on a ceramic funnel connected to a vacuum pump. Transfer it on filter paper to allow gross removal of water and then transfer into it a crystallizer of suitable size. Be careful not to lose flakes in the process.
- Dry each fraction in an oven at °85C for 6 hours.
- Cool down in desiccator (desiccator equipment is optional).
- Evaluate residual stickiness after drying.
- Weigh the floating fraction and report the weight as “F”. Document the sample with one or more photographs.
- Qualitatively inspect the floating fraction for residual stickiness of the adhesive.
- If any PET flakes are glued with label pieces in the sinking fraction, document them with a photograph, then carefully separate them from the label. Weigh them and report the labels weight as G.
- If G > 0, then calculate the new weight of the floating fraction as

H = F + G

Otherwise assume H = F

- Weigh the sinking fraction and report the weight as “J”. Document the sample with one or more photographs. Record any labels that did not separate from the PET flake. Take a separate photograph of any labels that did not separate from the PET flakes. Check the flakes surface and verify that it is not sticky.
- If G > 0, then calculate the new weight of the sinking fraction as

Y = J - G

- if it is impossible to separate the PET and the label then weigh the third fraction as X.

- Run QT500 on the sinking fraction and the reference sample*. As a consequence, QT500 will have to be run with a lower amount of material.*
- Calculate the difference between the initial label and adhesive weight and the recovered floating fraction and report it as “K”

K = E – H

- Calculate the ratio between the floating fraction and the initial label and adhesive weight and express it in percentage. Report the value as “L”

L = (H/E) x 100

- If K > 0, then calculate the ratio between the non-recovered floating fraction and the initial label and adhesive weight and express it in percentage. Report the value as “M”

M = (K/E) x 100

- Calculate the difference between the initial panel weight and the recovered sinking fraction and report it as “N”

  N = A – Y

  Calculate the non-recovered (dissolved) weight amount and report it as “P”

  P = C – (H + Y+X)

- • If P > 0, then calculate the ratio between the dissolved fraction and the initial label and adhesive weight and express it in percentage. Report the value as “Q”. Calculate also the concentration of the dissolved fraction in ppm, assuming the water density as 1 g/ml)

Q = (P/E) x 100

R = P/water volume

(Express R as ppm of adhesive, assuming water density as 1 g/ml)

Mass balance results

The ideal result is the one where J = A and E = H with minor differences, that can be explained with deviations due to experimental setup (e.g. reproducibility in weighting). Such results indicate full label detachment, with the adhesive remaining on the label and no adhesive or other label component dissolved in the washing water.

Success criteria:

J-A<0.5% (0.5% adhesive on the flakes

A-J<3% 3% flake loss

Same for E and H. Thus E-H<1% Labels sinking

H-E<3% flake loss

Any significant deviation from the ideal result will need to be investigated. In particular:

- If J > A and no label residue can be seen, this could be an indication that some residual adhesive is left on the flakes. The oven test carried out on the sinking (PET) fraction should confirm this (refer to EPBP QT500).
- If P > 0, then an amount of sample has been dissolved in the caustic washing water. If no discoloration of the caustic washing solution is observed, this could be either label

surface coating (if present) or adhesive. The amount of dissolved adhesive or label coating should not exceed 500 ppm. Adhesive concentrations above the threshold require investigation of the adhesive chemistry and solubility, to exclude that long-term accumulation effects could generate issues to the recycling plant operation and recycled PET flakes quality

- In any case, the situation where K > 0 by a value that cannot be explained with the analytical setup is an indication of incomplete sink float separation of the label.
- The fate of the non-recovered label amount should be investigated, by drawing hypotheses based on the test results and, if appropriate, by performing other

investigations to get a confirmation. It is important to ensure that the non-recovered fraction is not detrimental to recycling.

Test report

The test report must include the following information:

- Reference to the EPBP Quick test QT508
- A full and complete identification of the material tested for the bottle and the label
- Description of the samples before, during and after testing (especially on colour changes, haze, deposits, sinking or non-detached label fragments, residual stickiness, etc.)
- The photographs indicated in the test procedure should be taken and be supplied as electronic files for reference. Photographs should be of reasonable quality, with good lighting and suitable background. Additional photographs are welcome whenever useful for documenting specific situations. To keep the report file to a size reasonable for e-mail circulation, pictures can be resized upon incorporation in the report and repetitive pictures can be omitted, provided the report contains a list of all the pictures taken with file name and picture description the listed photographs are made available as separate JPG files.
- Details of any deviation from the test method, as well as any incident which may have influenced the results
- Test figures and residual stickiness evaluation. Use the Table 1 and 2 as reference.
- Documentation and reporting of the QT500 oven test following the requirements of the QT500 test procedure.
- 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 complete a full screening for possible effects of innovation on the recyclability of collected bottles, the processing of r-PET into products and the final product properties. Please contact EPBP for more information.

Table 1: Adhesive releasability in combination with reactivation

Adhesive releasability in combination with reactivation				
Priority	**Adhesive**	**Reactivation**	**Assessment**	**Conditions**
1	**Stays on the label**	**0**	**No reactivation**	
2	**Stays on the label**	**1to 3**	**reactivation**	
3	**Stays on the label**	**4 or 5**	**Strong reactivation**	

Table 2: Mass balance calculations

Variable	**Value**	**Reference in the procedure**
Total weight of the panels	X.xxxx g	**A**
Sample weight before curing	X.xxxxg	**B**
Sample weight after curing	X.xxxx g	**C**
Weight loss during curing	X.xxxx g	**D = B-C**
Weight of labels and adhesive	X.xxxx g	**E =C-A**
Weight of the floating fraction	X.xxxx g	**F**
Weight of residual labels in the sinking fraction	X.xxxx g	**G**
Corrected weight of the floating fraction	X.xxxx g	**H = F+G**
Weight of the sinking fraction	X.xxxx g	**J**
Corrected weight of the sinking fraction	X.xxxx g	**Y = J-G**
Non-separable labels and PET	X.xxxx g	**X**
adhesive lost from the label (floating fraction)	X.xxxx g	**K = E-H**
Ratio of the adhesive plus label fraction after and before washing	X.xx %	**L =** (H/E) x 100
Ratio between the non-recovered floating fraction and the initial label and adhesive weight	X.xx %	**M =** (K/E) x 100
Difference between the initial panel weight and therecovered sinking fraction	X.xxxx g	**N = A-**

		Y
Non-recovered (dissolved) weight amount	X.xxxx g	**P =** C – (H + Y+X)
Ratio between the dissolved fraction and the initial label andadhesive weight	X.xx %	**Q =** (P/E) x 100
Amount of dissolved fraction	ppm	**R = P/water volume**
Residual stickiness evaluation (S = Strong reactivation, R = reactivation, N = no reactivation) As per table 1		

Definitions

- **Adhesive:** any material that is used for the labelling system application on the PET bottle
- **Water soluble adhesive:** any applied adhesive capable of chemically dissolving in water or alkali under the specified washing conditions in the recycling process
- **Releasable adhesive:** any applied adhesive capable of releasing on at least one side of its bond under the specified washing conditions in the recycling process. For our application releasable has the meaning that the adhesive will remain on the label.
- **Floating:** adhesive that agglomerates, has a density <0.95g/cm^3 , thus staying on the surface of the wash tanks
- **Dissolvable adhesive:** adhesive that under the washing conditions is diluted in the washing water.
- **Dispersible adhesive:** adhesive that is distributed across the volume of the wash tanks in very fine particles.
- **Reactivation:** the behaviour of a hydrophobic adhesive that is releasable (i.e. stays on the label) and after the labels are dried (i.e. moisture<5%) the adhesive is again sticky, causing label flakes re-attachment/re-tackification on the PET flakes or equipment surface.