European PET Bottle Platform

PET Recycling Test Protocol

Website version

September 2017

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 polymerisation or added during injection moulding in the form of liquid or solid master-batches. Other non-PET components can be labels, glue, sleeves, caps, printings, etc. The protocol is designed to evaluate PET packaging solutions that generally end up in the PET recycling stream and that can possibly influence the quality of - or even disturb - the recycling system.

EPBP has developed a protocol for testing innovative PET bottles. This protocol is based on the latest knowledge in common practice on the recycling processes and the possible impact of the innovation in PET bottle technology on the potential to recycle PET bottles into products with the highest added value. EPBP has used the Petcore 2007 protocols and the American Plastic Recyclers (APR) protocols as guidance. The following paragraphs are important to understand the principles of the new protocol.

Non-PET components in new or innovative PET bottles may affect specific properties of R-PET which are relevant for its re-use as secondary raw material. R-PET is mainly used for applications such as bottles, film, sheet, strapping and fibre. Many of the potential effects on properties will be the same, independent of the application. Therefore the EPBP protocol focuses on testing specific properties, and can be seen as a Bottle-to-Property protocol rather than a Bottle-to-Bottle or Bottle-to-Fibre protocol.

The majority of recycling companies rely on three colour streams:

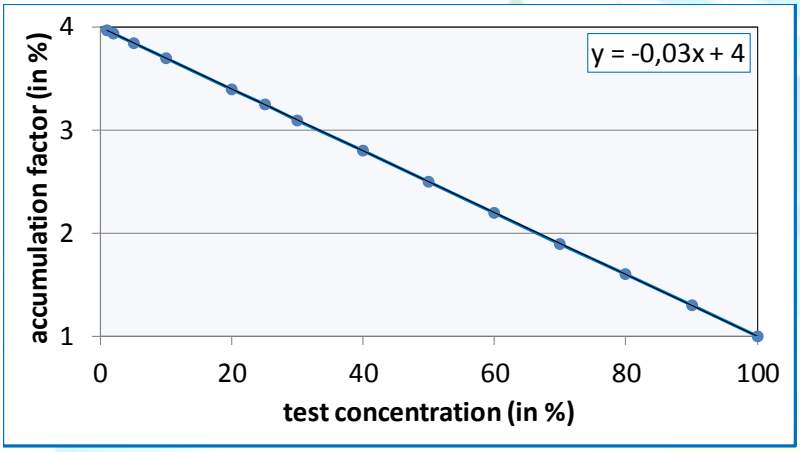
- a transparent clear/light blue stream;
- a transparent coloured stream and;
- an opaque/dark coloured stream.

Although the type of bottles and the collection system used in each country can differ, all collected PET bottles - including the innovative PET bottles - will end up in one of the groups. The capture rate is determined by market penetration, consumer behaviour, collection schemes, waste separation facilities and sorting technologies. For this reason the EPBP protocol incorporates the “sortability” of innovative PET bottles as a tool to calculate the likely concentration of innovative flakes in one of the main R-PET streams.

Innovative bottles will have different levels of market penetration. Market penetration is considered on a European level and is defined as the total market in which the innovation bottle is applied; it is not to be confused with the market share that one particular solution reaches in a given market[1]. The size of the expected market penetration will be indicated by the applicant in its written application. However, it is up to EPBP to estimate the applicable market penetration value and consequently define the testing concentration. *EPBP reserves the right to revise its decisions should a market grow beyond its initially expected market penetration.*

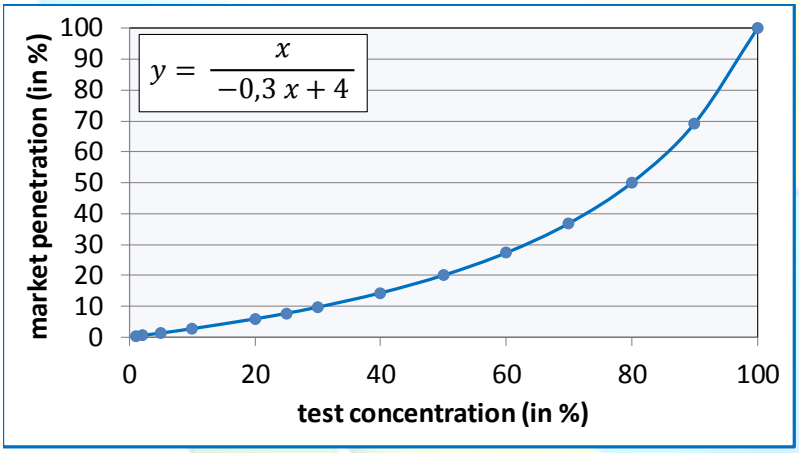
[1] *As an example PET bottles with barrier solutions may reach a market penetration of 10% of the total PET bottle market. A given producer of PET barrier resins may only reach a market share of 1%, while other technologies supply the remaining 9%. The negative impact of all technologies will impact the PET stream. Therefore each individual innovation is tested based on a 10% market penetration.*

The EPBP protocol recognizes standard testing levels of 2, 5, 10, 25, 50 and 100% of the innovative sample in the flake mix. While testing at 2% is only for applications in niche markets, higher concentrations are necessary for innovations for larger markets. Testing at other concentrations can be agreed between EPBP and the applicant. Testing in the relevant concentration range will lead to the maximum allowable concentration of innovative bottles in the recycle stream that can pass the test limits. The maximum allowable concentration is subsequently divided by an accumulation factor to take local accumulation effects into consideration and to absorb regional differences across Europe[2]. This allows the final acceptable market penetration of the innovative PET bottle to be determined (figure 1a, 1b).



test conc. (in %)	accum. factor
100	1,00
90	1,30
80	1,60
70	1,90
60	2,20
50	2,50
40	2,80
30	3,10
25	3,25
20	3,40
10	3,70
5	3,85
2	3,94
1	3,97

Figure 1a Accumulation factor as a function of test concentration



test conc. (in %)	market level (in %)
100	100,0
90	69,2
80	50,0
70	36,8
60	27,3
50	20,0
40	14,3
30	9,7
25	7,7
20	5,9
10	2,7
5	1,3
2	0,5
1	0,3

market level (in %)	test conc. (in %)
100	100,0
90	97,3
80	94,1
70	90,3
60	85,7
50	80,0
40	72,7
30	63,2
25	57,1
20	50,0
10	30,8
5	17,4
2	7,5
1	3,9

Figure 1b Maximum allowable market penetration as a function of test concentration

A higher accumulation factor may be applied if an innovation was specifically targeted at a limited local distribution (e.g. a single region or country).

[2] *Innovations may reach a market penetration in Europe of a certain percentage. However they may only be available in some countries. Consequently the innovation will accumulate at a much higher rate in the recycling stream of those countries.*

The EPBP Technical Committee will do its best to ensure that the impact of the innovation on the PET recycling systems is evaluated in an objective and structured manner, while at the same time trying to minimise the financial burden on the applicant by avoiding unnecessary testing. In order to design the "best" test program, the applicant is requested to provide as much detailed information as possible on its innovation. The EPBP Technical Committee will use the information provided by the applicant, combined with its expertise and knowledge database, to determine the optimal test program.

Important

To avoid unnecessary costs the applicant should get in contact with EPBP before committing to any testing. Tests performed by the applicant without prior agreement of EPBP are performed by the applicant at its own risk and without any guarantee that the results will allow EPBP to make an assessment.

2. Starting the procedure

EPBP has issued an Assessment Process document and an Application Form, both documents can be found on the EPBP website. The first document explains the general rules, the different steps and tools used during the test procedure. The information provided on the application form will be used to start the EPBP test procedure.

Table 1 shows the steps that will be followed to agree the individual test program:

- Step 1: It is very important to get good and complete information about the innovation, from both a technical and a market perspective. This allows the applicant and EPBP to design the most appropriate test program, and to select only the relevant tests from the total test list (table 2).
- Step 2: If sorting technologies or any other separation technique have the effect of reducing the concentration of the innovative material in the R-PET stream, an assessment of these specific additional steps (for example sorting) can be included in the test program.
- Step 3: Based on the available information, the applicant and EPBP will decide if any one property in table 2 is considered "critical". This property should be tested first to save costs.
- Step 4: In parallel to step 3, the applicant and EPBP will decide on the full test program (i.e. a selection from all tests listed in table 2) if the sample passes the critical test. Designing this program at the start enables the applicant to estimate the total cost for testing. EPBP and the applicant may agree to add other specific tests to the test program to highlight not yet identified effects.

The test protocol is designed 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 properties of the final product.

Table 1

Steps	Comments
Step 1, Input from Application Form	
Details of the innovative bottle	For example typical colour, size, weight, decoration, closure, additive, etc
Composition new component	As much as possible to understand critical effects
Addressable market	Type of applications
Volume addressable market	Size of total market including geographical concentrations
Step 2, Assessment of concentrations in recycle streams (sortability)	
Sorting Technologies (reference efficiency)	Expected efficiency
Colour sorting (95%)	To be measured if applicable
IR sorting (80%)	To be measured if applicable
Metal detection (90%)	To be measured if applicable
Sink/float (99%)	To be measured if applicable
Air elutriation (50%)	To be measured if applicable
Step 3, Testing critical properties	
Expected critical properties	For example colour
Expected concentration in clear/blue stream	
Expected concentration in dark colour stream	
Define test program for critical property	
Define test concentration	Select from 2, 5, 10, 25 and 50% innovation flakes
Step 4, Final test program	
Depending on the outcome of step 3, define final test program	Select properties to be included in the program from Properties Table 2

Specific tests must be executed using modern test equipment by an independent test laboratory with no affiliation to the Applicant. The test laboratory has to be approved by the EPBP Technical Committee. In-house testing at the facility of the Applicant is exceptionally allowed under the following conditions:

- The Applicant concerned demonstrates that they were unable to comply with the original test procedures and/or that the technical conditions do not allow any other way.
- The Applicant owns an in-house test laboratory with standard test equipment and for in-house laboratory procedures; equipment must be operated and evaluated according to similar test conditions as an independent test laboratory.
- The Platform gives special permission for in-house testing.
- The Platform appoints an auditor who will monitor the execution of the tests at the Applicant and who will certify the test results.
- The Applicant will cover the costs of the auditor.

The execution of the specific tests involves activities such as validating test environments, running the test, generating the test results and controlling the validity of the test results. Most test results are strongly affected by the precise method of testing or measuring. It is therefore vital to fully document all test methods, test conditions and measurements and to provide complementary observations if required.

Table 2

	RPET Properties	Critical test
Optical		
1	Colour and haze	L* a*, b* and haze measured on injection moulded plaques
2	Black specks and gels	Visual/camera check on products or fibre spinning and tensile testing
3	Fluorescence	Visual UV test on pellets
Processing		
4	Air elutriation efficiency	Air separation flakes
5	Sticking during drying	Agglomeration flakes
6	Feeding properties	Flow properties flakes
7	IV build-up in Solid Stating	IV on solid stated pellets
8	Rheology (viscosity, melt strength)	Fibre spinning or preform injection
9	Filter contamination	Filter test extrusion
10	Strain hardening	Fibre spinning or bottle blowing
11	Mould deposit (plate out)	Visual check after injection moulding or extrusion
Mechanical		
12	Impact resistance	Drop test and burst test on bottles
13	Stress crack resistance	Stress crack test on bottles
14	Gas barrier properties	CO_2 loss test on bottles
15	Thermal stability (creep)	Thermal stability test on bottles
16	Strength/elongation	Tensile test on fibres
17	Modulus	Tensile modulus test on fibres
18	Thermal stability (shrinkage)	Fibre shrinkage
Product Stability and Thermal Properties		
19	Melting and crystallization temperature	DSC second run on pellets or flakes
20	AA generation	AA in preforms
21	UV stability on colour	Discoloration caused by UV exposure
22	Stability during extrusion (bubbles, fumes)	Observation during pellet extrusion or injection moulding
23	IV stability/break down	IV measurement before and after extrusion or injection
24	Product stability (de-composition)	Screening test for volatile compounds such as degradation products (other than AA) or non-intentionally added substances that can leach out of the PET.
Other properties		
25	Label bleeding	To be defined
26	Label separation after hot water	To be defined
27	Glue removal	To be defined
28	Inertness	Filter test using high shear extrusion
29	Residual lipophilic content	To be defined

Remark: EPBP will not assess food contact safety (before and/or after recycling), or any other matters related to the regulatory status of the product.

3. Starting the tests

3.1 Bottle samples

The applicant will supply an agreed quantity of the virgin PET resin which is used as the reference. The applicant will also supply an agreed number of bottles with- and without the innovation for the control and test samples. The PET resin used for all samples must be the same and must be a clear PET non-reheat resin with an IV of 0.80 dl/g. Recommended PET resins are given in table 3. The use of any other PET resin shall be agreed upfront between the applicant and EPBP. The EPBP also reserves the right to use a specific resin.

Table 3

Supplier	Grade
Equipolymers	Lighter C93
Indorama (Europe)	RAMAPET N1, N180 and N1(S)
Indorama (US)	1708 CC
Lotte	LPAPETClear
Plastiverd	Global

The test bottles should be produced with a composition representative of its final use in the market. The control bottles - not containing the innovation such as barrier material, coating or additives - are produced from virgin PET, and are identical in size and weight to the test bottles. The steps to produce and test the required samples are outlined in table 4.

Table 4

Process steps		
	Pellets	
	Bottles	
	Grinding	
	Washing	
	Air elutriation	
	Flakes	
	Flake mixing (2, 5, 10, 25 and 50% test)	
	Extrusion to pellets	
Route 1	**Route 2**	**Route 3**
Solid stating for Colour	Solid stating for 0.80 IV	Crystallization pellets
Pellet blending (50/50)	Pellet blending (50/50)	Fibre spinning
Injection Molding plaques	Injection Molding Preforms	Film extrusion (gels/specks)
Testing Plaques on colour	Blow molding bottles	
	Testing bottles	
Sample code A1 to F1	Sample code A2 to F2	Sample code A3 to F3

For preform injection and bottle production, the R-PET pellets containing x% of the test sample are blended with a fixed percentage of virgin PET. To conform to previous protocols, the EPBP will use a standard mix of 50% R-PET and 50% virgin PET for plaque injection. Fibre tests are carried out using 100% innovative material.

Processing both reference and test samples at the various steps and testing of all samples must be carried out according to fixed conditions. Details of these conditions, as well as critical limits in the testing, will be communicated to the Applicant and its test facility as soon as an agreement has been reached about the test program.

3.2 Mass balance

Innovations that are not part of the PET composition – but are attached to the bottle – have to be designed to be separated from the PET bottle flake stream before they enter the extrusion step[3]. If their threshold concentration is known it will be sufficient to demonstrate their removal during the washing steps below the threshold. This must be demonstrated by a mass balance that shows where and to what extent the innovation component is separated.

Two mass balances have to be considered:

- The amount of PET entering and leaving each washing step;
- The amount of the innovation component entering and leaving each washing step.

Losses in the process (e.g. loss of fines), the loss or adsorption of volatile components (including moisture) as well as the loss of water soluble components into the wash water must be considered and be reported to explain any discrepancies in the mass balance.

The weight measurements for each washing step have to be recorded in a flow chart and summarised in a mass balance report. Any deviations and losses should be explained.

The EPBP reserves the right to request a mass balance for any application where it considers this necessary for a valid assessment.

[3] *This typically applies to caps and labels*