European PET Bottle Platform

PET Recyclability and Circularity Test Protocol

Website version

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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 circularity 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 rPET quality and processing. 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. 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 of common practice in the recycling processes and the possible impact of the innovation on the PET bottle material stream.

Non-PET components in new or innovative PET bottles may affect specific properties of rPET which are relevant for its re-use as secondary raw material. rPET is mainly used for applications such as bottles, film, sheet. Many of the potential effects on properties will be the same, independent of the application. Therefore, the EPBP protocol focuses on testing specific properties, with the main focus on Bottle-to-Bottle. In the light of circularity, the Bottle-to-Fiber protocol, is not an option anymore.

The main bottle collection schemes are:

- a transparent clear/light blue stream
- a transparent colored stream
- a white opaque stream
- an opaque/dark colored stream (not considered in the protocol for the time being)

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 behavior, 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 rPET 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*.

The circularity protocol is an amendment of the previous EPBP recycling protocol. Based on the legislative requirements the recycled PET will become a source for future recycling loops and therefore the behavior of an innovative substance must be evaluated based on its impact in multiple loops. This requires consideration of accumulation effect as well as the impact of multiple heat histories. Therefore, the Circularity Protocol will show changes in test concentrations and the addition of an accelerated impact test (AIT) for multiple recycling loops.

[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.*

2 Test concentrations

Two types of test concentrations have to be considered:

- the concentration of innovative bottles in the tests
- the concentration of a substance in the innovative bottles

2.1 Concentration of innovative bottles

Testing concentration of 25% innovative bottles is suitable for evaluation of most innovations and it is considered as the standard one. However, depending on market penetration, for some innovations, testing at higher concentrations (50% or 100% innovative bottles) is required to fully assess the impact on recycled PET quality and PET recycling processes.

Applicants can ask for testing at lower concentrations than 25% innovative bottles, supporting their request with adequate evidence. The applicant's request will be evaluated by the EPBP and the required testing concentration will be communicated to the applicant.

Testing in the relevant concentration of innovative bottles will lead to the maximum allowable concentration of innovative bottles in the recycle stream. The maximum allowable concentration is subsequently divided by a local 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).

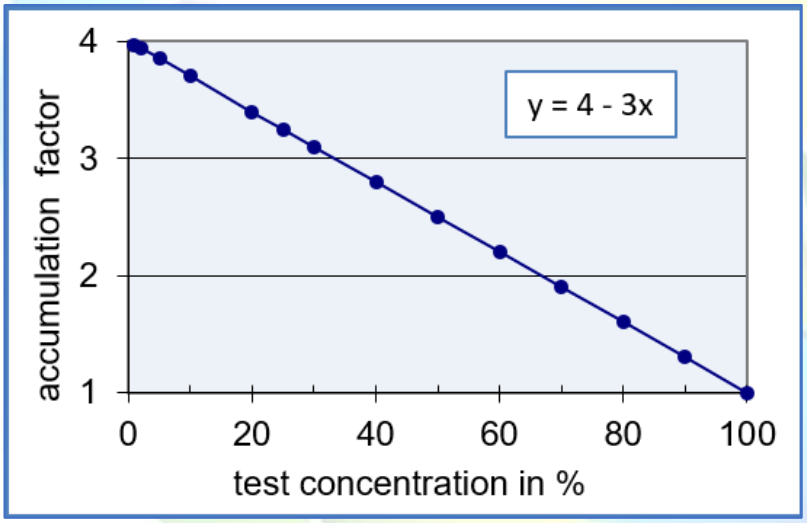
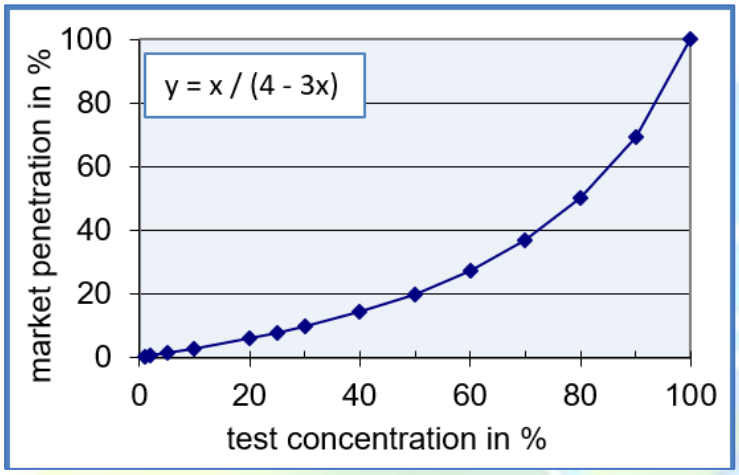


Figure 1a Local accumulation factor as a function of test concentration:

test conc. in %	local accum. factor
1	3.97
2	3.94
5	3.85
10	3.70
20	3.40
25	3.25
30	3.10
40	2.80
50	2.50
60	2.20
70	1.90
80	1.60
90	1.30
100	1

[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*



test conc. in %	market penet. in %	market penet. in %	test conc. in %
1	0.3	1	3.9
2	0.5	2	7.5
5	1.3	5	17.4
10	2.7	10	30.8
20	5.9	20	50.0
25	7.7	21	51.5
30	9.7	30	63.2
40	14.3	40	72.7
50	20.0	50	80.0
60	27.3	60	85.7
70	36.8	70	90.3
80	50.0	80	94.1
90	69.2	90	97.3
100	100	100	100

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

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

2.2 Concentration of substance

Additives, contaminants or resins of a special type tend to accumulate in the rPET resin used in multiple cycles and therefore the testing concentration of a substance in innovative bottles must not only consider the maximum intended use concentration of the substance (c0) but also the substance amount that represents the equilibrium concentration of the substance in the rPET stream due to circular PET use (c~).

The equilibrium concentration of the substance depends on the market penetration of the bottles containing the innovative substance and the amount of mechanically recycled PET that is used in a closed loop (%rPET) versus the addition of fresh PET (%vPET). Fresh PET is either virgin PET or PET from chemical recycling with the ability to remove the added substance.

For accumulation calculations a long-term maximum share of closed loop mechanical recycling of 75% is used.

The equilibrium concentration of the substance also depends on the point of introduction of an added substance. Three characteristic cases can be distinguished:

1. A substance added to the fresh PET only. An example would be a substance that is added in the PET manufacturing process. The equilibrium concentration in the rPET stream is:

   c~ = c0 * market penetration

2. A substance added to the recycled PET only. An example would be a substance that is added during the recycling process. The equilibrium concentration in the rPET stream is:

   C~ = c0 * rPET/vPET share * market penetration;

   for 75%/25% rPET/vPET share = 3 * c0 * market penetration

3. A substance added to the recycled PET and the fresh PET. An example would be an additive that is added during preform manufacturing. The equilibrium concentration in the rPET stream is

   c~ = c0 * (1 + rPET/vPET share) * market penetration;

   for 75%/25% rPET/vPET share = 4 * c0 * market penetration

The above cases can be described by one equation as follows:

c~ = c0 * (1 + a) * market penetration

whereas:

a = 0 when the substance is added only to the virgin PET

a = rPET/vPET share - 1 (at 75% rPET: a = 2) when the substance is added only to recycled PET

a = rPET/vPET share (at 75% rPET: a = 3) when the substance is added to re cycled and virgin PET

Dilution according to market penetration is already considered by mixing the innovation material with the control resin. The test concentration has to be increased accordingly. This simplifies the test concentration to:

ctest = c0 * (1 + a)

Based on the above formula the test concentration may be higher than the maximum intended use concentration. Preparing the sample at this concentration requires different strategies:

- For any components inside the PET body (like additives) by directly applying the test concentration when preparing the test sample
- For any components adhering to the PET body (like labels) by spiking to the required factor based on the residual contents after washing. This means that the residual content of the innovative substance after washing has to be measured (see Mass Balance below) and depending on the required factor additional innovative substance will have to be added. For evaluations at or below 25% test concentration the accumulation can be considered by multiplying the test concentration by (1 + a)

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 minimize 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 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.

3 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:

1. 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).
2. Step 2: If sorting technologies or any other separation technique have the effect of reducing the concentration of the innovative material in the rPET stream, an assessment of these specific additional steps (for example sorting) can be included in the test program.
3. 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.
4. 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 and/or circularity of collected bottles into rPET, the processing of the rPET into products and the properties of the final product.

In cases where multiple substances are added to innovative bottles the test program may require testing of innovative bottles with each substance independently. Such independent testing is required where one substance is masking the impact on properties of another substance.

Table 1

Steps	Comments
Step 1: Input from Application Form	
Details of the innovative bottle	For example, typical color, size, weight, decoration, closure, additive, etc.
Chemical composition of new component	As much as possible to understand critical effects
Addressable market	Type of applications
Volume addressable market	Size of total market including geographicalconcentra-tions
Step 2: Assessment of concentrations in recycle streams (sortability)	
Sorting Technologies (reference efficiency)	Expected efficiency
Color 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, color
Expected concentration in clear/blue stream	
Expected concentration in dark color stream	
Define test program for critical property	
Define test concentrations for innovative bottles for innovative substance	 Select: 2, 5, 10, 25, 50 and 100% innovation flakes Select: 1, 3 and 4 times c0
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:

1. 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.
2. 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.
3. The Platform gives special permission for in-house testing.
4. The Platform appoints an auditor who will monitor the execution of the tests at the Applicant and who will certify the test results.
5. 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	Color and haze	L* a*, b* and haze measured on injection molded plaques
2	Black specks and gels	Visual/camera check on products
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, meltstrength)	Preform injection
9	Filter contamination	Filter test extrusion
10	Strain hardening	Bottle blowing
11	Mold deposit (plate out)	Visual check after injection molding 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
Product Stability and Thermal Properties		
19	Melting and crystallizationtemperature	DSC second run on pellets or flakes
20	AA generation	AA in preforms
21	UV stability on color	Discoloration caused by UV exposure
22	Stability during extrusion(bubbles, fumes)	Observation during pellet extrusion or injection molding
23	IV stability/break down	IV measurement before and after extrusion or injection
24	Product stability (volatilization)	Screening test for the presence and/or generation of semi-volatile compounds that can leach out of the PET during processing (recycling or converting).
25	Color development in multiple recycling loops	Color evaluation based on Accelerated Impact Test (AIT)
26	NIAS formation in multiple recycling loops	NIAS evaluation based on Accelerated Impact Test (AIT)
Other properties		
27	Label bleeding	QT 507
28	Label separation after hot water	QT 508
29	Glue removal	QT 504
30	Inertness	Filter test using high shear extrusion
31	Residual lipophilic content	To be defined

Remark

EPBP will not assess food contact safety and regulatory compliance in general (before and/or after recycling), or any other matters related to the regulatory status of the product. Food contact safety and legal compliance is under the applicant's responsibility. Nevertheless, samples obtained from the execution of EPBP tests will be used to evaluate the presence of NIAS. NIAS compliance is a prerequisite for a Circularity endorsement. Prior to circularity endorsement grant, the applicant should share also with EBPB the NIAS compliance report/certificate.

4 Starting the tests

4.1 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 the same virgin PET grade, 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 (25% or higher test)		
Extrusion to pellets		
↓	↓	↓
Route 1	**Route 2**	**Circularity Evaluation**
Solid stating for color	Solid stating for 0.80 IV	Solid stating for color under air
Pellet blending (50/50)	Pellet blending (50/50)	Pellet extrusion
Injection molding plaques (black specks)	Injection molding preforms (black specks)	
Testing plaques on color	Blow molding bottles (black specks)	
	Testing bottles	
Sample code A1 to G1	Sample code A2 to G2	Sample code A3 to G3

For plaques and preforms injection and bottle production, the rPET 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% rPET and 50% virgin PET for plaque injection.

4.2 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 maximum acceptable impact 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:

1. The amount of PET entering and leaving each washing step;
2. 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 summarized 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.*