

PET materials and articles in which the recycled plastic is used behind a **Functional Barrier.**

Monitoring Report by Article 13 of Regulation (EU) 2022/1616. 10<sup>th</sup> of October 2023.

## **First monitoring programme for the project “PET materials and articles in which the recycled plastic is used behind a Functional Barrier”**

In accordance with the requirements of Article 13 of Regulation (EU) 2022/1616, we are herewith reporting the results of the first monitoring programme relative to the placing into the market of PET-base A/B/A structures, where the A and B layer consist respectively of virgin and Recycled PET (rPET).

This report should be read in conjunction with the Novel Technology notification dossier referred as “PET materials and articles in which the recycled plastic is used behind a Functional Barrier”, submitted on 5 April 2023.

The submission of the dossier was made by a consortium established by PETCORE Europe AISBL (“PETCORE”) and EUPC AISBL (“EUPC”) to assist their members using the functional barrier principles for the manufacture of PET thermoformed packaging food contact applications in complying with the above-mentioned regulation.

The present report is built out of the analysis that have been carried out by the Members of the consortium; these data are historical data, and have been obtained with different analytical approaches, nevertheless they provide a clear vision of the substances referred to in Article 13(5)(c) of the Regulation.

### **1. Description of the Functional Barrier technology**

rPET is used in food contact packaging for two main applications: direct contact with food and indirect contact with food. For indirect contact with food, the recycled PET is mildly decontaminated, and subsequently embossed between two layers of virgin PET, or PET originating from super-clean processes, suitable for direct food contact. In this case, the layer in contact with food acts as “functional barrier”, preventing any possible contaminants in the rPET to be transferred to food in a quantity that endangers human health and, therefore, making the final structure compliant with Regulation (EC )1935/2004, in particular with art 3 thereof.

The submitted dossier deals exclusively with the PET containers which include the functional barrier, where the rPET is not in direct contact with food.

Starting from pre-washed and dried flakes of PET derived from post-consumer collection that comply with the requirements of Article 6 of Regulation (EU) 2022/1616, the manufacturing of A/B/A structures include a combination of some of the following processes:

- A drying and crystallization phase of the washed flakes, which is operated usually under stirring and air flow, at temperature of 140-160°C, generated by friction or IR, for a residence time up to 6 hours.
- An extrusion phase, where flakes are melted to produce the rPET B layer with or without application of vacuum. The temperature profile is usually 270-290°C. When vacuum is applied, the vacuum conditions are typically below 100 mbar.
- The coextrusion step, in which the A layers are applied in a die. In this case the rPET of the future B layer comes in contact with the virgin PET (or mixture between virgin and EFSA assessed

PET) of the future A layers, at a temperature of typically 275-290°C for few seconds. A 3-layer sheet (A/B/A) comes out from the coextrusion process and it is immediately cooled down in a rolled stack press.

The following configurations of processes are covered by the Novel Technology dossier:

Configurations	Crystallizing/drying	Extrusion	Degassing	N of installations
X1	yes	Single Screw	No	<b>32</b>
X2	yes	Single Screw	Yes	<b>18</b>
Y1	yes	Twin Screw Co-Rotating	Yes	<b>17</b>
Y2	no	Twin Screw Co-Rotating	Yes	<b>109</b>
W	no	Single screw and satellitar	Yes	<b>1</b>

The process configurations have been identified as X1, X2, Y1, Y2 and W to simplify the text of the submission.

The final sheets are converted into trays. The sheets are heated in an oven to a temperature of 120-130°C, and the trays are formed through the application of pressure and vacuum in a mould. The total cycle takes 2-3 seconds. The trays are then immediately cooled down to an average temperature of around 30°C.

## 2. Capability of decontamination of the Functional Barrier technology

The original submitted Novel Technology dossier reported the outcomes of the decontamination efficiency associated to the above-mentioned process configurations. The decontamination efficiency was demonstrated using to model contaminating substances, referred as “surrogate contaminants”, that are normally used for testing the decontamination capabilities of PET recycling processes. Based on that data, modelling of migration of surrogate contaminants has been carried out starting from concentration of these contaminants of 3 mg/kg (EFSA assumption). The software used for the migration modelling was SML365, developed by AKTS ( Sierre- Switzerland). The concentration used for the migration modelling was calculated starting from 3 mg/kg, and applying the decontamination efficiency of each process configuration, determined via challenge test, prior to entering of the material into the die.

By using that model, we were able to calculate the maximum amount of rPET in the B layer of different A/B/A structures at which the concentration of the surrogate migrating at the highest rate of lies below the level of 0.15 microgram/kg food recommended by EFSA as safety level, by assuming that all migrating contaminants are genotoxic.

The calculation was carried out in different use conditions, i.e. 10 days at 20°C, 10 days at 40°C and 365 days at 25°C.

### 3. List of contaminating substances with molecular weight < 1000 Dalton

The list of the 20 most occurring substances in the plastic input (flakes) is reported in Table 1. The table originates from 20 analytical reports. The substances are reported in descending order of occurrence.

Table 1: List of the 20 most occurring substances in plastic input (flakes) in descending order of occurrence

substance	CAS number
1 Benzophenone	119-61-9
2 Ethylbenzene	100-41-4
3 o/m/p-xylene	95-47-6/ 108-38-3/106-42-3
4 hydrocarbons c14-c18	629-59-4/629-62-9/629-78-7/593-45-3
5 heptanone/nonanone	110-43-0/693-54-9
6 hexanal/octanal/heptanal/nonanal	66-25-1/124-13-0/111-71-7/124-19-6
7 diethyl phthalate/Di-isobutyl phthalate/other phthalates	84-66-2/84-69-5
8 limonene	138-86-3
9 toluene	108-88-3
10 acetaldehyde	75-07-0
11 terpinene/ terpinolene	99-85-4/586-62-9
12 2-propyl 1 heptanol	10042-59-8
13 diphenyl ether	101-84-8
14 N-butylbenzene sulphonamide	3622-84-2
linear and cyclic oligomers (linear C30-C60/cyclic C20-	
15 C60/cyclic with DEG C22-C52	
16 Bisphenol A	80-05-7
17 acetophenone	98-86-2
18 ethyl hexanol	104-76-7
19 2,6-di-tert-butyl-4methyl-phenol	128-37-0
20 tetrachloroethylene	127-18-6
21 hexyl cinnamal	101-86-0
22 naphthalene	81-30-1
23 tributyl phosphate	126-73-8
24 2-methyl-1,3-dioxolane	467-29-7

The list of the 20 most occurring substances in the plastic output (sheets) is reported in Table 2. The table originates from 25 analysis reports. The substances are reported in descending order of occurrence.

Table 2: List of 20 most occurring substances in plastic output (sheets) in descending order of occurrence

substance	CAS N
1 o/p-xylene	95-47-6/106-42-3
2 diethyl phthalate/Di-isobutyl phthalate/other phthalates	84-66-2/84-69-5
3 benzophenone/4-methyl benzophenone	119-61-9/134-84-9
4 hexanal/octanal/nonanal	66-25-1/124-13-0/124-19-6
5 Bisphenol-A	80-05-7
6 limonene	138-86-3
7 toluene	108-883
8 linear and cyclic oligomers (linear C30-C60/cyclic C20-C60/cyclic with DEG C22-C52)	
9 benzene	71-43-2
10 acetaldehyde	75-07-0
11 ethylbenzene/trimethyl benzene	100-41-4/95-63-6
12 2-methyl-1,3-dioxolane	467-29-7
13 ATBC/ acetyl citrate	77-90-7
14 cumene/thymol	98-82-8/89-83-8
15 benzaldehyde	100-52-7
16 heptanone/heptenone	110-43-0/1119-44-4
17 2-4-di-t butyl phenol	96-76-4
18 acetic acid	64-19-7
19 Irgafos 168/ Irgafos 168 oxidated form	119345-01-6/ 95906-11-9
20 THF/ furane/acetyl furane/furane derivatives	109-99-9/110-00-9/1192-62-7

The data collected are not standardized because of constraints given by the short time provided by the Regulation. The consortium has already started to carry out a more standardized monitoring, which will be incorporated in the next round of report.

A total of 15 Business Operators have contributed to obtain the information contained in Table 1 and 2 above, and have notified their processes. The total number of installations involved in the collection of the data reported in table 1 and 2 corresponds to those 15 Recycling Operators represents a total of 76 out of the 197 installations represented by the consortium, and covered by the New Recycling Technology notification filed on April 5<sup>th</sup>, 2023.

#### 4. List of contaminating materials in plastic input

The content of food grade PET in the plastic input (flakes after the pre-processing) is  $\geq 95\%$ . Other physical contaminants include:

- PVC  $\leq 50$  ppm
- Polyolefins  $\leq 100$  ppm
- Other plastics  $\leq 50$  ppm

- Metals  $\leq$  10 ppm
- Paper and wood fibres  $\leq$  10 ppm
- Other inert materials  $\leq$  5%

#### 5. Most likely origin of contaminants

There are several sources to which the presence of substances in the input and output plastic can be attributed.

The input would contain substances that are not fully removed by the treatment to which the bottles containers undergo in the pre-processing step

- Components of PET (oligomers)
- Chemicals originating from environmental contamination (e.g. BPA)
- Substances naturally occurring in nature (e.g. cumene, thymol, benzaldehyde, cinnamic acid esters)
- Substances, especially plasticizers, derived from glues used in labels (phthalates, benzophenone and others)
- Food and drink components (limonene)
- Decomposition of PET (ethylbenzene and derivatives, acetaldehyde, 2-methyl-1,3-dioxolane)
- Antioxidants, mostly present in polyolefins (e.g. BHT, Irgafos 168 and its decomposition products)
- Other substances of unknown origin

The substances found in the output do not significantly differ from those found in the input. A full quantitative analysis of the concentration in the input and output will be reported in the next monitoring report, it is however observed that substances present in the input plastics are largely removed by the recycling process. Especially the most volatile substances are almost completely removed.

#### 6. Measurement or estimation of the migration level

The detection of the substances present into the input plastic and in the finished sheet has been carried out by several laboratories with different methods and without direct comparison between input plastics and output plastics. Qualitative or semi-quantitative analysis of these substances has been carried out, but the differences in testing conditions (time and temperature used in test, as well as extraction solvents, non-standard equipment, insufficient substances libraries and other parameters) do not allow comparison of the outcomes. Due the complexity of the meta-analysis of the data, we decided not to report quantitative information in the present document. We expect a more systematic analysis to be carried out in the next phase of the monitoring program. The estimation of migration and comparison of the outcomes between input and output plastic was not possible for the substances reported in Table 1 and 2 . The creation of a direct link between contaminants in the input and the output plastic material is the objective of the next 6-month monitoring period.

In semi-quantitative analysis, in most cases a Detection Limit of 10 microgram/kg has been obtained.

Tests of specific migration for regulated substances in A/B/A PET structures, as well as overall migration tests, are carried out regularly by the members of the consortium. No infringement of the limits established by the law have been detected.

## 7. Sampling strategy

As the tests used to build this report belong to control campaigns carried out by the members of the Consortium in the course of the years, a uniform sampling strategy cannot be identified. Even though the lack of systematic controls does not allow 1-1 correspondence between input and output plastics, the high level of randomization provides a realistic view of the contaminants in the input flakes and in the sheets that are present in the European market. Many contaminants have a very low occurrence, therefore they may be identified as occasional contaminants, but the recurring substances carry a high probability of representing typical contaminants in the PET recycling processes, and the next monitoring campaign will focus on these substances.

## 8. Description of the analytical procedures

Various analytical methods have been used to detect the substances in input and output plastics. The analytical equipment is usually composed by

- Headspace GC for analysis of volatile substances, with or without SPME
- GC- Mass Spectroscopy for semi volatile substances, usually with MS/FID detector, and with or without QTOF
- Liquid Chromatography with Mass Spectroscopy, with or without QTOF detector, for semi volatile and volatile substances.

The conditions for extracting the substances from the rPET material were rather variable, some of the most used are reported here below.

For flakes:

- dichloromethane 3 days 40°C
- acetonitrile and dichloromethane
- ethanol 95% 10 days 60°C
- 8hr dichloromethane at reflux temperature

For sheets:

- 95% ethanol 10 days at 60°C
- hexane/ethyl acetate 5 min 60°C
- hexane /ethanol 3/1, 8h at 20°C
- dichloromethane, 3 days 40°C

## 9. Explanation of discrepancies between input and output

The differences between input and output plastic are not macroscopic. Some of the most highly occurring substances that are present in post-consumer PET are also found in the rPET. This is the case of normal constituents of PET, such as its oligomers, and decomposition products of PET, such as xylenes, acetaldehyde, ethyl benzene, 2-methyl-1,3-dioxolane etc.

Certain products occurring in food, such as limonene, are not completely removed during the recycling process, but their presence is below the safety threshold. Others such as, terpinene, terpinolene, hexyl cinnamal, which are present in small quantity in the input material, are removed during the process.

Environmental contaminants in the flakes are significantly reduced in the final sheets.

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