

First monitoring programme for the project “Manufacturing of food contact trays containing recycled PS, in expanded state, separated from food by 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 PS- based structures A2/B/A1, B/A1 and B/A1 with a barrier film, where the A and B layer consist respectively of Virgin and Expanded Recycled Polystyrene (PS and RXPS).

This report should be read in conjunction with the Novel Technology notification dossier referred as “Manufacturing of food contact trays containing recycled PS, in expanded state, separated from food by a functional barrier”, submitted on 5 April 2023.

The submission of the dossier was made by a Consortium led by Polymer Comply Europe, a consulting company established in Belgium at avenue de Cortenbergh 71, Brussels, which is acting as « developer » in accordance to the requirements set forth in article 32(2) of the 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

The requirements of Article 13 of the Regulations are treated in the paragraphs reported in this paper.

13(5)(a) Description of new technology

The process consists of four steps

- a) **Pre-processing: grinding, separation and washing** of sorted post-consumer PS, purchased from EPR collection schemes in various EU Countries
- b) **Decontamination**: extrusion of the washed flakes in a twin screw extruder with degassing, to produce pellets. In this step the flakes may be mixed with virgin PS, in various proportions
- c) **Post- processing**: production via extrusion of the foamed B layer containing up to 50% of recycled PS,
- d) **Post-processing: application of the A1 and A2 layers on the B layer** via coating and/or lamination process. In some cases only the A1 layer is applied, leading to structures B/A1, where A1 is in direct contact with food.

Pre-processing: grinding, separation and washing

The incoming PS is subjected to grinding, and subsequently to separation from non-PS materials by the use of different technologies that include metals separators, optical sorting, sifting, manual separation, washing and others. A key step is represented by flotation, which is operated to make sure that the PS entering the

decontamination has a high grade of purity. The key parameters controlling flotation consist of the water density. The flotation takes place in two washing basins: the first basin uses water at room temperature with a density between 1.05 and 1.09 kg/litre. The second basin uses hot water, which is introduced at a temperature of $80\pm 5^{\circ}\text{C}$, and the density shall be lower or equal than 1.02 kg/litre.

Decontamination:

The decontamination is carried out via extrusion with degassing in a DELCO twin screw extruders equipped with vacuum ports. The conditions of extrusion at which the decontamination take place are :

Temperature : $190\text{-}230^{\circ}\text{C}$

Residence time : 1-2 min

Vacuum level : 0.70-0.80 bar

The control parameter for this step is represented by the vacuum level; a monitoring system is in place ensuring that the value of the vacuum is constantly equal or below 0.80 bar.

Post-processing:

Production of the foamed B layer.

The layer B is produced in a single screw extruder with a temperature profile that does not exceeds 250°C . The density of the foam produced is usually below 100 kg/m^3 . The controlling parameter of this step consists of the quantity of recycled PS in layer B. Such quantity shall not exceed 50% by weight. In order to ensure that the quantity is fulfilled, the extruder is equipped with a gravimetric dosing system with continuous monitoring of the added material.

Post-processing: application of the A2 and A1 layers

The A2 and A1 layers can be applied via coating or via lamination. For the properties of the final tray, there is no difference whether these layers are applied using coating or lamination, so that both A2 and A1 layers can be indifferently applied via these two technologies. The functional barrier properties do not change with the technology used to apply it. In both cases the raw materials used to obtain the functional barrier are GPPS, HIPS or a blend of them. In case extrusion coating technology is used, the recipe of the raw materials is prepared by a dosing system. The materials constituting the A2 and A1 layers goes through a single-screw extruder and are coated on the cooled B layer. In this case the thickness of the functional barrier is determined by a control on the weight of the A1 layers.

In the case of the lamination technology, the functional barrier consists of a flexible film that is coupled to the B layer by thermal lamination. The functional barrier film is a semi-finished product that is purchased from an

10th October, 2023

external supplier. The technical parameters of the film are agreed with the supplier and a control plan on the input material is in place. Following the application of the A layers, the sheet is wound into a reel. The coils are stored at room temperature for a maximum period of 60 days. Subsequently, the reels undergo the thermoforming process to obtain trays. In the thermoforming process the film is heated in an oven to reach a temperature of 90-130°C, then formed in a mould. The forming cycle lasts usually 1-3 seconds. The key parameter for the quality of the functional barrier consists of the thickness of the A1 layer. The thickness of A1 after thermoforming should not be lower than 30 micron. The trays are then stored in a room temperature warehouse for a maximum period of 12 months until they are shipped to the customer.

13(5)(b) Decontamination Efficiency

The decontamination efficiency of the process has been determined through a “challenge test” carried out at the operations of Forever Plast.

Attachment 1 reports the technical characteristics and the outcomes of the challenge test, and includes the calculation of the decontamination efficiency.

For the decontamination efficiency, the initial concentration of the surrogate (migrants) has been assumed as 3 ppm (mg/kg). Table1 shows the decontamination percentage (4th column) of the process and the residuals concentration in the B Layer of each surrogate considering the use of 100% of R-PS (5th column) and 50% of R-PS (6th column). The simulations of the migration started considering the use of and 50% of R-PS in the layer B.

The last Column of the table, shows the updated concentration of each simulant to be coherent with the foamed structure of the XPS tray object of our study. The foaming process increases the density and consequently reduces XPX tray weight and quantity of material (plastics, contaminants, etc) for square centimeter surface of XPS tray. Concentrations of each simulants are therefore multiplied by foam density of the B layer (foam density = 0,083333 kg/cm³, about 12 time lower than compact polystyrene).

10th October, 2023

Table 1: concentration of surrogate contaminants

CAS Number	Surrogate Substances	Initial Concentration (ppm)	Decontamination Efficiency (%)	concentration in the B Layer of each surrogate considering the use of 100% of R-PS (ppm)	concentration in the B Layer of each surrogate considering the use of 50% of R-PS (ppm)	Concentrations multiplied by factor 0.083333 considering the density of the foam in layer B (ppm)
108-90-7	Chlorobenzene	3	47,0	1,59	0,794893	0,066241
119-36-8	Methyl Salicylate	3	55,7	1,33	0,664996	0,055416
112-61-8	Methyl stearate	3	81,2	0,57	0,282591	0,023549
99-62-7	1,3-diisopropyl benzene	3	81,1	0,57	0,284005	0,023667
108-88-3	toluene	3	59,9	1,20	0,601432	0,050119
119-61-9	Benzophenone	3	76,1	0,72	0,358673	0,029889

The concentrations of surrogates in column 6 have been used to simulate the migration of these substances from, respectively, structures consisting of

- A2/B/A1,
- B/A1 and
- B/A1 with a barrier film in direct contact with food

The migration modeling has been carried using the software 365SML, developed by AKTS (Switzerland)

An overview of the structures, conditions at which the simulation has been carried out, and the relevant results is reported in Table n. 2.

The density correction factor applies to layer B only, and it has been introduced as the foaming process increases the volume of the recycled PS of a factor of 12, hence reduced the density of the layer by the same factor. The density of the foam produced is always below 100kg/m³ and this aligns with 12 reduction factor used in the calculation.

The simulation has been carried out for food contact at 20°C, for 10 days, accounting for storage fresh food (e.g. red meat, processed meat, fresh pasta, cheese and others) at refrigerated conditions. The simulants used was 95% ethanol, in order to simulate contact with fatty food.

In table 2, trays 1, 2 and 3 originate from an initial sheets subject to draw ratio of 1.5; trays 3, 4 and 5 originate from an initial sheet subject to draw ratio of 2. Total sheet thickness and draw ratio have been calculated in order to obtain a thickness of the A1 layer (functional barrier layer) of 30 micron.

The outcomes of the migration modeling demonstrate that the considered structures a functional barrier layer with thickness of 30 micron is sufficient to reduce the migration of all surrogates to a level of 0.15 ppb, identified by EFSA as the safety level below which the risk from contaminants is deemed negligible.

13(5)(c),(e) Substances in Input and Output materials

Substances present in the Flakes (**input materials**):

Number of Analytical Reports: 43

Tables n. 3 and n. 4 shows all substances detected with a LoD of 0.1 ppm for volatiles and 1 ppm for non-volatiles and respective origin. The majority of substances are normally find in virgin polystyrene, such substances have been indicated as Typical. In addition the 20 Volatile and non-volatile substances most present in the flakes has been highlighted in light gray (input material)

Table 3: Non- Volatile Substances in flakes

Non- volatile Substances in flakes	CAS	Possible Origin	Typical in PS polymer	N. of occurrences	Average conc. ppm	Standard Deviation
Pentadecano	629-62-9	Paraffin oil	Typical	100%	42,3	10,9
[2.2]Paracyclophane	1633-22-3	Styrene Dimers	Typical	28%	11,3	5,4
Styrene-Acrylonitrile Dimer (DANS)		Styrene Acrylonitrile Dimers	Typical	72%	8,5	4,6
Butylated Hydroxytoluene	128-37-0	BHT: Antioxidant		33%	10,8	9,1
Ascorbic Acid 2,6 dihexadecanoato	4218-81-9	Food additive		79%	32,5	38,4
1,3-difenilpropano	1081-75-0	Styrene Dimers	Typical	100%	16,9	5,2
Styrene Dimer		Styrene Dimers	Typical	100%	458,0	124,9
Heptadecane	629-78-7	Paraffin oil	Typical	93%	8,4	2,5
C14H14N2		Styrene Acrylonitrile Trimers (An2S)	Typical	67%	71,1	91,6
Styrene-Acrylonitrile2 Trimer (TAN2S)		Styrene Acrylonitrile Trimers (An2S)	Typical	79%	29,9	30,7
Styrene2-Acrylonitrile Trimer (TANS2)		Styrene Acrylonitrile Trimers (AnS2)	Typical	95%	257,8	288,4
DimetilAdipato	627-93-0	Additive: Plasticizer		56%	211,7	382,6
Styrene Trimer		Trimer (S3)	Typical	100%	3001,9	838,1

10th October, 2023

Diotilftalato	117-81-7	Additive: Plasticizer		58%	25,1	13,0
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Table 4. Volatile Substances in the Flakes

Volatile Substances in flakes	CAS	Possible Origin	Typical in PS polymer	% presence in reports	Average conc. ppm	Standard Deviat
2-Propanol, 2-methyl	124-68-5	Washing and cleaning products		95%	0,6	0,2
Acetic acid, methoxy-, methyl ester	6290-49-9			53%	0,3	0,1
Methacrolein	78-85-3	From Methyl metacrylate polymer		5%	0,5	0,0
o-Ethylhydroxylamine	624-86-2			63%	0,6	0,3
2-Pentene, (Z)-	627-20-3			81%	0,6	0,3
Butanal	26232-84-8			7%	0,2	0,0
Acetic acid	64-19-7	Degradation		9%	1,3	0,3
Silane, dimethoxydimethyl	1112-39-6	Additive: silicone oil		84%	0,6	0,4
Cyclohexane	110-82-7	polymerisation product	Typical	100%	1,6	1,0
Pentanal	110-62-3	Fragrances		60%	0,5	0,2
Toluene	108-88-3		Typical	100%	2,8	1,2
Hexanal	66-25-1	Fragrances		91%	1,5	0,8
Cyclotrisiloxane, hexamethyl	541-05-9	Additive: silicone oil		91%	1,1	0,5
Ethylbenzene	100-41-4	Polymerisation product	Typical	98%	9,9	3,6
Benzene, 1,3-dimethyl	108-38-3		Typical	100%	2,0	0,9
Styrene	100-42-5		Typical	98%	57,7	16,9
Heptanale	111-71-7	Fragrances		14%	0,4	0,1
Benzene, (1-methylethyl)	98-82-8		Typical	98%	2,9	0,9

10th October, 2023

Benzaldehyde	100-52-7	Degradation/oxidation	Typical	65%	0,5	0,2
Benzene, propyl	103-65-1	Polymerisation product	Typical	100%	3,6	1,1
Benzene, 1,2,3-trimethyl	526-73-8	Polymerisation product	Typical	98%	0,8	0,4
Octanal	124-13-0	Fragrances		23%	0,5	0,6
Heptane, 2,2,4,6,6-pentamethyl	13475-82-6	process solvent polymer / additive		95%	2,4	7,8
Decane		HDPE		81%	0,4	0,1
1-Hexanol, 2-ethyl		degradation plasticizer		100%	4,3	1,8
D-Limonene	5989-27-5	post-consumer		100%	3,4	1,3
Acetophenone	98-86-2	degradation/oxidation		42%	0,5	0,2
Nonanal	124-19-6	fragrances		47%	0,5	0,4
Undecane		mineral oil		88%	0,6	0,4
Cyclopentasiloxane, decamethyl+	541-02-6	Additive: silicone oil		98%	0,9	0,6

Table 3. Volatile Substances find in the Flakes

Substances Present In the Granules (**Output material**)

Number of Analytical Reports n.43

The tables n. 5 and n. 6 shows all substances detected with a LoD of 0.1 ppm for volatiles and 1 ppm for non-volatiles and respective origin. The majority of substances are normally found in virgin polystyrene, such substances have been indicated as Typical. In addition the 20 Volatile and non-volatile substances most present in the granules of R-PS (output material)

Table 5: Non- Volatile Substances in granules

Non- volatile Substances in granules	CAS	Possible Origin	Typical in PS polymer	N. of occurrences	Average conc. ppm	Standard Deviation
Pentadecano	629-62-9	Paraffin oil	Typical	98%	43,1	30,2
[2.2]Paracyclophane	1633-22-3	Styrene Dimers	Typical	21%	8,2	2,7
Styrene-Acrylonitrile Dimer (DANS)		Styrene Acrylonitrile Dimers	Typical	14%	8,6	5,9
Butylated Hydroxytoluene	128-37-0	BHT: Antioxidant		23%	9,2	6,9
Ascorbic Acid 2,6 dihexadecanoato	4218-81-9	Food additive		33%	30,1	20,1
1,3-difenilpropano	1081-75-0	Styrene Dimers	Typical	100%	13,6	6,4
Styrene Dimer		Styrene Dimers	Typical	100%	347,0	224,5
Heptadecane	629-78-7	Paraffin oil	Typical	74%	19,0	34,8
C14H14N2		Styrene Acrylonitrile Trimers (An2S)	Typical	93%	74,7	57,2
Styrene-Acrylonitrile2 Trimer (TAN2S)		Styrene Acrylonitrile Trimers (An2S)	Typical	86%	30,1	16,9
Styrene2-Acrylonitrile Trimer (TANS2)		Styrene Acrylonitrile Trimers (AnS2)	Typical	74%	95,0	90,7
DimetilAdipato	627-93-0	Additive: Plasticizer		37%	39,0	55,2
Styrene Trimer		Trimer (S3)	Typical	100%	2785,0	1828,7
Diotilftalato	117-81-7	Additive: Plasticizer		37%	29,7	28,3

Table 6. Volatile Substances find in Granules

Volatile Substances in granules	CAS	Possible Origin	Typical in PS polymer	% presence in reports	Average conc. ppm	Standard Deviat
2-Propanol, 2-methyl	124-68-5	Washing and cleaning products		0%	n.d.	
Acetic acid, methoxy-, methyl ester	6290-49-9			0%	n.d.	
Methacrolein	78-85-3	From Methyl metacrylate polymer		0%	n.d.	
o-Ethylhydroxylamine	624-86-2			0%	n.d.	
2-Pentene, (Z)-	627-20-3			9%	0,6	0,3
Butanal	26232-84-8			0%	n.d.	
Acetic acid	64-19-7	Degradation		0%	n.d.	
Silane, dimethoxydimethyl	1112-39-6	Additive: silicone oil		5%	0,3	0,0
Cyclohexane	110-82-7	polymerisation product	Typical	16%	0,5	0,2
Pentanal	110-62-3	Fragrances		0%	n.d.	
Toluene	108-88-3		Typical	95%	0,9	0,4
Hexanal	66-25-1	Fragrances		2%	1,6	
Cyclotrisiloxane, hexamethyl	541-05-9	Additive: silicone oil		12%	0,5	0,5
Ethylbenzene	100-41-4	Polymerisation product	Typical	100%	3,3	2,7
Benzene, 1,3-dimethyl	108-38-3		Typical	67%	0,5	0,2
Styrene	100-42-5		Typical	100%	34,8	13,9
Heptanale	111-71-7	Fragrances		0%	n.d.	
Benzene, (1-methylethyl)	98-82-8		Typical	88%	0,9	0,5
Benzaldehyde	100-52-7	Degradation/oxidation	Typical	7%	0,3	0,1
Benzene, propyl	103-65-1	Polymerisation product	Typical	100%	1,4	0,6
Benzene, 1,2,3-trimethyl	526-73-8	Polymerisation product	Typical	51%	0,3	0,1
Octanal	124-13-0	Fragrances		2%	0,3	
Heptane, 2,2,4,6,6-pentamethyl	13475-82-6	process solvent polymer / additive		7%	0,2	
Decane		HDPE		0%	n.d.	
1-Hexanol, 2-ethyl		degradation plasticizer		0%	n.d.	

10th October, 2023

D-Limonene	5989-27-5	post-consumer		9%	0,3	
Acetophenone	98-86-2	degradation/oxidation		2%	0,1	
Nonanal	124-19-6	fragrances		0%	n.d.	
Undecane		mineral oil		0%	n.d.	
Cyclopentasiloxane, decamethyl+	541-02-6	Additive: silicone oil		0%	n.d.	

n.d. : Not Detected.

13(5)(d) Usual polymer Contaminant present in the input materials

The Usual Contaminant present in the input materials are polyolefins and PVC.

Table 7 below shown the most present physical contaminant materials; polyolefins came from the output of Nitrogen DSC Test with 10 °C/min run and 50 ml /min of Nitrogen, PVC content is performed indirectly by determining the difference between the total chlorine content and the chlorine content in ionic form.

Table 7: physical contaminants in the input material

Contaminant	Number of sample analysis	Average content
Polyethylene	43	0,18 %
Polypropylene	43	0,10 %
Total Polyolefins		0,28 %
PVC	43	81 ppm

The content of food grade PS in the plastic waste input is $\geq 95\%$.

13(5)(e) Origin of the Contaminant

The origin of the contaminants of paragraph 15(5)(c) are showed in above Tables 3-6

The origins of the contaminant of the letter 15(5)(d) are post-consumer waste polymer mixed with the post-consumer PS

13(5)(g) Description of the Strategy for sampling and Lot identification

rPS Lot identification and Lot sample

Each batch consists of 100 tons of homogeneous material.

10th October, 2023

The homogeneity of the product is guaranteed by continuous and repeated mixing of 1-5 cm flakes starting from grinding and continued in the following processing steps: separation, washing, drying, extrusion and storage in homogenizing silos before bagging. For purpose of tracing and quality identification of finished product (granules), each Lot is subject to the following actions:

- Identification with a unique tracing number,
- Identification of a sample representative of the Lot. During the entire Lot production, every 3 hours individual samples are taken, the Lot Sample is obtained by mixing all the individual samples

rPS Lot identification tracing number

Each batch of granules is identified with an 11-digit alphanumeric code as described below:

Lot: AA BBBB CCCCC

AA: Foreverplast Production Line

BBBB: Year of production

CCCCC: progressive production number

In order to allow optimal management of goods handling, each lot may be further divided into sub-lots which identify the individual trucks.

13(5)(h) Description of the Applied method for the Volatile and non-volatile Substances in the Granules

1. Sample preparation for the analysis of volatile compounds

Place 1 g of granules into a 10 mL vial for headspace with a cap and silicone/PTFE septum. Add 5-10 µl of an internal standard (chlorobenzene) methanol solution, at about 3000 ppm. The sample is thermostated at 125°C for 30' and then injected into GC-MS.

1.1. Configuration and parameters HS-GC-MS for volatile compounds analysis

Instrumentation: GC HP6890 interfaced to MS HP5975 with EI source and single quadrupole analyzer, equipped with SSL injector, FID detector and HTA 300HT headspace autosampler

Chromatographic column: Mega-JXR 25 m x 0.2 5mm x 1.50µm (or equivalent)

Thermostating: 125°C for 30'

T injector: 260°C

Injection volume: 1 mL

Injection technique: Split

Split ratio: 30:1

Column flow (He): 0.8 ml/min (constant flow mode)

Temperature ramp: initial isotherm at 40°C for 3', then T ramp (2°C/min) up to 70°C, isotherm at 70°C for 5', then T ramp (2°C/min) up to 160°C, isotherm at 160°C for 20', then T ramp (2°C/min) up to 250°C, final isotherm at 250°C for 5'

Transfer line T: 260°C

Give response factor 1 with respect to the internal standard to all compounds.

2. Sample preparation for analysis of non-volatile compounds

0.5 g of polymer is dissolved with 3 ml of an internal standard solution in CHCl₃ (di-butyl phthalate at about 15 ppm, for extraneous substances, and n-C₁₆H₃₄ at about 50 ppm, for oligomers), then precipitates with 8 ml of ethanol under mechanical agitation. The resulting solution is filtered on syringe filters with 0.45 µm pores and injected into GC (for quantitative purposes) or GC-MS (for qualitative purposes).

2.1. GC configuration for non-volatile analysis

Instrumentation: GC Thermo Trace 1300, equipped with on-column injector and FID detector and TriPlus autosampler

Chromatographic column: HP-1 25m x 0.320mm x 0.52µm (or equivalent)

Injection technique: Cold-On-Column

Injection volume: 1 µl

Column flow (H₂): 2 mL/min for 1 min', then flow ramp (0.2 mL/min/min) up to 4.2 mL/min (programmed flow mode)

Temperature ramp: initial temperature at 60°C, then T ramp (40°C/min) up to 160°C, isotherm at 160°C for 5', then T ramp (8°C/min) up to 325°C, isotherm at 325°C for 5'

Detector T: 330°C

Response factor 1 with respect to the internal standard to all compounds.

10th October, 2023

13(5)(i) Explanation of any observed discrepancies between expected contaminant levels in the material input plastic and in the output of the plant

The results observed in the output demonstrate that the efficiency of the decontamination process is high especially for volatile substances.

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