PET Flake Injection

Novel Technology Development

Data Monitoring Report

report required by Article 13 of Regulation (EU) 2022/1616

10 October 2023 – updated 30 March 2025

The data and figures presented in this report should never be regarded as definitive or isolated from their respective contexts. It is essential to acknowledge the inherent limitations of the calculation process. The values obtained are closely linked to and dependent on the limits of the analytical methods, the minute quantities of substances present, the limited number of samples, and the degree of correlation of the inputs with the outputs. This makes it impossible to obtain true values comparable to those obtained and validated in the challenge test. Consequently, only the challenge test should be regarded as the standard.

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Introduction

The novel technology *'PET Flake Injection'* was notified as required under Articles 10(2) and 10(3) of Commission Regulation (EU) 2022/1616 on 17th March 2023.

Article 13 of Commission Regulation (EU) 2022/1616 states the following:

"*a recycler operating a decontamination installation in accordance with Article 11* of the regulation *shall monitor the average contaminant level on the basis of a robust sampling strategy which samples the plastic input batches and the corresponding plastic output batches".*

The enclosed report is based on the latest information from all installations using the novel technology received in accordance with Article 13(3) for the first monitoring period and provides the information required by Article 13(5) of the Regulation.

The different subsections (a) to (j) of Article 13(5) are discussed separately.

a) Brief description of the novel technology – Art 13(5)(a)

The Flake Injection process has the capability to combine depolymerised recycled Polyethylene Terephthalate (rPET) with virgin material at different stages of a conventional PET production process for subsequent food contact use.

The input material of the Flake Injection process is previously processed PET as detailed in Table 2 of ANNEX I of COMMISSION REGULATION (EU) 2022/1616 that is deliberately depolymerized (pre-processed) before it enters the high surface area decontamination polymerisation reactor. Referring to the flow scheme Appendix I: Flake Injection – PET Production Process; previously processed PET may be introduced directly to injection point 1. or partially depolymerised with ethylene glycol, in either a stir-tank reactor or an extruder, to a defined degree of polymerisation to correspond with that of the polymer in the PET production process at the injection points labelled 2 to 6 in the flow scheme *or any points in-between*. This initial depolymerisation process of the previously processed PET allows for filtration of the intermediate polymer to remove solid contaminants before the introduction of the recycled material into a PET production process at a blend rate of up to 100% recycled content. The high surface area decontamination polymerisation technology increases the Intrinsic Viscosity (IV) of the PET polymer and removes polymerisation by-products under high vacuum of less than 20mbar, with a high temperature greater than 260°C and with a residence time greater than 30 minutes. This high surface area polymerisation technology also serves as a Decontamination Technology to efficiently remove vapourised contaminants that may have been introduced into the process further upstream by the addition of previously processed PET. Following the high surface area polymerisation and decontamination, the polymer melt is filtered for either direct use, or granulation, in the manufacture of food contact materials or articles or for introduction into a Solid State Polycondensation (SSP) process or a Conditioning Silo should further processing be needed to meet the material parameters required for its end use.

b) Summary of the reasoning on the capability of the novel technology and the recycling process(es) to manufacture recycled plastic materials and articles that meet the requirements of Article 3 of Regulation (EC) No 1935/2004 and that are microbiologically safe – Art 13(5)(b).

All references in this section are references to documents available in the dossier submitted in accordance to Articles 10(2) and 10(3) of Commission Regulation (EU) 2022/1616 on 17th March 2023.

Flake To Resin (FTR)

Ref. ANNEX II Table 1 (1) Decontamination efficiencies of the Novel Technology have been determined by Welle (2008). .

Table VI. Concentrations (determined using the HFIP extraction method) of the surrogates in the investigated PET samples of Trial 2 (cocktail A at 10 ml min^{-1}, 50% PCR flakes).

	Concentration (ppm)						
	Toluene	Chloroform	Chlorobenzene	Phenyl cyclohexane	Methyl salicylate	Benzophenone	Lindane
Calculated contamination concentration	3295	5194	1255	327	1004	885	775
Before deep-cleansing	1999 ± 28	3075 ± 47	655 ± 9	163 ± 2	<1.0	345 ± 1	133 ± 1
After deep-cleansing (final product)	<2.7	<0.8	<0.9	<0.2	<1.0	<0.2	<0.8

The study concludes that the cleaning efficiencies for the applied surrogates are above or far above 99.9%. The high cleaning efficiencies are due to the high diffusion rates of compounds in the molten PET.

Based on EFSA's criteria for safety evaluation of PET recycling processes - if a recycling process is able to reduce an input reference contamination of 3 mg/kg PET to a Cres (Residual Concentration) not higher than a Cmod (Modelled Concentration) corresponding to the relevant migration criterion, the potential dietary exposure cannot be higher than 0.0025 µg/kg bw/day and recycled PET manufactured with such recycling process is not considered of safety concern.

Ref. ANNEX II Table 1 (2) Fraunhofer_*Dossier-FTR_20061109.pdf*

Reversed Approach

Based on Safety Evaluation of Polyethylene Terephthalate Chemical Re-cycling Processes. Frank Welle. 'Reversed Approach'.

Ref. ANNEX II Table 1 (3) *Chemical_recycling_submitted.pdf*

FTR: Calculated maximum concentration (Reference Contamination – the level of contamination that the process can remove, i.e. Cmod:Cres =1) corresponding to a migration of 0.1 µg/l after storage for 365 d at 25 °C (EU cube, AP = 3.1, tau 1577 K, bottle wall thickness 200 µm, density of PET 1.4 g/cm^3). Decontamination Efficiency of 99.9%.

mm Hg (25°C)	°C	g.mol^{-1}	FTR	Reference Contamination	Decontamination Efficiency	*Cres*	*Cmod*	
Vapour Pressure	BP	Mw	Surrogate	mg/kg	%	mg/kg	mg/kg	Cmod:Cres
28.4	110.6	92.1	Toluene	**90**	99.9%	0.09	0.09	1.0
197	61.1	119.4	Chloroform	**100**	99.9%	0.10	0.10	1.0
12	131.7	112.6	Chlorobenzene	**90**	99.9%	0.09	0.09	1.0
0.0343	222.9	152.2	Methyl Salicylate	**130**	99.9%	0.13	0.13	1.0
0.04	240.1	160.3	Phenyl Cyclohexane	**140**	99.9%	0.14	0.14	1.0
0.00193	305.4	182.2	Benzophenone	**160**	99.9%	0.16	0.16	1.0
9.40E-06	311.0	290.8	Lindane	**310**	99.9%	0.31	0.31	1.0

Artenius.

EFSA-Q-2011-00969 - EFSA refused to evaluate as out of the scope of Regulation (EC) 282/2008.

Ref. ANNEX II Table 1 (7) *EFSA_Letter Related to Artenius Unique Process.pdf*

Ref. ANNEX II Table 1 (8) *Fraunhofer Institute. Challenge Test.pdf*

US FDA Guidance

Use of Recycled Plastics in Food Packaging (Chemistry Considerations): Guidance for Industry.

U.S. Department of Health and Human Services Food and Drug Administration Center for Food Safety and Applied Nutrition July 2021

VIII. Elimination of Data Recommendations for 3° Recycling Processes for PET and PEN

Based on a comprehensive review of all surrogate testing data submitted over the past decade for 3° recycling processes for PET and polyethylene naphthalate (PEN), FDA concludes that 3° recycling of PET or PEN by methanolysis or glycolysis results in the production of monomers or oligomers that are readily purified to produce a finished polymer that is suitable for food-contact use. Both 3° processes will clean the polyester sufficiently to allow it to be considered of suitable purity, even assuming 100% migration of residual surrogate to food. This is a significant difference from the surrogate testing of 2° recycling processes. Secondary recycling processes often produce PET that is insufficiently cleaned to withstand 100% migration calculations for the residual surrogates. Under these circumstances, FDA recommends additional migration tests to demonstrate that the finished PET meets the 1.5 µg/person/day EDI limit.

Based on a determination that 3° recycling processes produce PET or PEN of suitable purity for food contact use, FDA no longer recommends that such recyclers submit data for agency evaluation. Because 3° processes for polymers other than PET and PEN were not the subject of FDA reviews, recyclers who wish to engage in 3° recycling of polymers other than PET and PEN are encouraged to submit data for evaluation.

Ref. ANNEX II Table 1 (9) *Recycled-Plastics-Food-Packaging-Chemistry-ConsiderationsGuidance-04112022-1321.pdf*

c) List a list of all substances with a molecular weight below 1000 Dalton found in the plastic inputs and recycled plastic output and 20 first detected incidental contaminants – Art 13(5)(c)

As developer of the Novel Technology, PET-Europe has coordinated with the recyclers regarding the selection of the sampling strategy, the analysis to be performed and the selection of a third-party laboratory. The choice of the laboratory was based on its experience and expertise in analysing PET samples, the relevance of its analytical equipment and validated methods as well as the capability to

identify and to risk assess non-intentionally added substances (NIAS) taking into account the particularity of this specific technology.

Analysis for the detection and quantification of substances in polymer represents a major challenge, especially when they are present at very low levels i.e. ppb levels. Although significant advances are regularly reported in the literature, reliable quantification of these substances to the ppb level and without compromising the integrity of the polymer is rarely feasible and certainly not standardized even for the most qualified laboratories. What is presented in this report has been obtained with the state-of-the-art analytical equipment (Table 6) that allows the detection of minute concentrations of various organic substances present in the input and output materials. The list of substances with a molecular weight below 1000 Dalton detected in the plastic input and its recycled output is given in Appendix II. The substances were sorted in descending order by their relative occurrence in the plastic input. The analytical methods do not distinguish between incidental contaminants and PET reaction products such as PET oligomers. In this report, incidental contaminants were identified by comparing the analytical data of the input samples with virgin PET samples analyzed under the same conditions and by the same analytical methods.

Table 1 lists the 20 most frequently detected and identified incidental contaminants in the input material using the different analytical methods specified in section h.

Table 1: List of the first 20 detected incidental contaminants in the input material, their frequency of detection and average amounts in input and output samples.

Substance	CAS	INPUT		OUTPUT	
		Frequency	Average (µg/kg PET)	Frequency	Average (µg/kg PET)
Limonene	5989-27-5	90%	449.91	0%	<4.21
Isophthalaldehyde	626-19-7	90%	75.41	45%	19.41
Styrene	100-42-5	90%	11.98	9%	10.19
1-Hexanol, 2-ethyl-	104-76-7	80%	121.20	9%	<100
Benzene, 1,2,3,5-tetramethyl-	95-93-2	80%	249.38	27%	20.64
Indane	496-11-7	80%	220.07	18%	25.91
n-Propyl acetate	109-60-4	80%	56.07	64%	43.76
γ-Terpinene	99-85-4	70%	83.27	0%	<4.21
2-Decenal, (E)-	3913-81-3	60%	304.78	18%	95.66
L-.alpha.-Terpineol	10482-56-1	60%	107.20	9%	<100
Benzene, 1-methyl-3-propyl-	1074-43-7	50%	404.59	27%	70.52
1-Dodecanol	112-53-8	40%	122.07	0%	<30.3
Benzene, 1,2-diethyl-	135-01-3	40%	165.66	18%	22.22
Benzene, 1-ethyl-2-methyl-	611-14-3	40%	43.42	18%	10.71
Benzyl alcohol	100-51-6	40%	120.42	9%	294.28
Dodecanal	112-54-9	40%	604.68	9%	23.09
Benzene	71-43-2	30%	27.15	9%	10.87
Benzene, propyl-	103-65-1	30%	41.67	0%	<2.75
Ethanol, 2-phenoxy-	122-99-6	30%	105.69	9%	>1000
Methyl salicylate	119-36-8	30%	121.16	0%	<30.3
Chloroxylenol	88-04-0	20%	502.60	0%	<30.3

The frequency of detection was determined by dividing the number of samples in which a particular substance was detected by the total number of samples analysed. The average concentration of the incidental contaminants was calculated by taking into account those samples in which it was detected. If the incidental contaminant was detected but below the quantification limit, the concentration used to calculate the average concentration was the limit of quantification. If the incidental contaminant was not detected in the output (frequency of 0%), the limit of detection is reported in the Table.

This novel technology allows the input material to be introduced into the decontamination process at variable ratios of input material/virgin material. Therefore, the input material is sometimes diluted with virgin material. The concentrations provided in Table 1 are the concentrations of incidental contaminants prior to any possible dilution. However, the dilution with virgin material is taken into account for the evaluation of the decontamination efficiency (section i).

For the inorganic analysis, a summary of the obtained analytical results is given in Table 2.

Table 2. Summary of the analytical results for inorganic elements.

	INPUT		OUTPUT	
	Frequency	Average (mg/kg PET)	Frequency	Average (mg/kg PET)
Cr	100%	1.53	73%	1.21
Mn	100%	0.39	100%	0.27
Fe	90%	9.42	73%	5.13
Co	100%	0.85	100%	13.57
Ni	100%	1.91	100%	1.92
Zn	0%	<6.4	0%	<6.4
Ge	100%	0.31	100%	1.59
As	100%	0.06	100%	0.09
Zr	20%	2.68	27%	0.82
Ba	70%	2.26	36%	3.00
Sb	100%	201.11	100%	181.21
Se	0%	<3.6	0%	<3.6
Pb	50%	2.79	36%	2.83

None of the analysed primary aromatic amines (Table 7) were detected in the input or output samples. In addition, no BPA, BPF or BPS was detected in the input and output samples with targeted analysis.

d) List of contaminating materials regularly present in the plastic input – Art 13(5)(d)

Table 3 lists the contaminating materials regularly present in the PET plastic input.

Table 3. Contaminating materials regularly present in the plastic input.

Contaminating material	
PVC	<50 mg/kg input
Polyolefin (caps/labels)	<20 mg/kg input
Other Polymers	<100 mg/kg input
Metal	<10 mg/kg input
Other Inert Materials	<30 mg/kg input

e) Analysis of the most likely origin of the identified contaminants referred to in points (c) and (d) – Art 13(5)(e)

Contaminating materials

Depending on the collection and sorting process, post-consumer PET waste can contain a limited amount of other materials such as polyolefins, polyvinyl Chloride (PVC), polyamide (PA), ethylene vinyl alcohol (EVOH), polystyrene (PS) and fillers. These materials originate from the following sources:

- Polyolefins like polyethylene (PE) and polypropylene (PP) are used to manufacture bottle closures and are present in a wide range of other plastic products.
- PVC is used in the manufacturing of certain labels and sleeves for bottles.
- PS is used in disposable cups and other packaging materials.
- EVOH is used as oxygen barrier in food packaging.
- PA is often used as barrier layer in flexible packaging films.
- Fillers are used in plastic packaging materials to modify their properties and enhance their performance.

Incidental contaminants

The likely origin of the incidental contaminants detected in the input material (Table 1) is as follows:

- limonene: since a large fraction of PET bottles is used to pack flavoured beverages, the flavour substance limonene is found in nearly all post-consumer PET waste streams (Franz *et al*., 2004).
- isophthalaldehyde; indane; Benzene, 1,2,3,5-tetramethyl; benzene, 1-methyl-3-propyl; benzene, 1,2-diethyl; -benzene, 1-ethyl-2-methyl-; benzyl alcohol: could originate from the oxidation or thermal degradation of aromatic hydrocarbons present in for example plastics, inks, adhesives, antioxidants or plasticizers.
- styrene: monomer used in the manufacture of thermoplastics used in packaging materials and articles (ECHA, 2025).
- 1-hexanol, 2-ethyl: could originate from plasticizers and polymer additives used in contaminating materials (other plastics).
- N-propyl acetate: could originate from beverage, food or cosmetic residues

- γ-terpinene: major component of essential oils made from citrus fruits with strong antioxidant activity. Widely used in food, flavours and cosmetics (European Commission, 2012).
- L-alpha-terpineol: flavouring agent used in several products.
- 1-dodecanol; could originate from cosmetics or cleaning agents or from the breakdown of lubricants used in plastic manufacturing during thermal processing.
- 2-decenal and dodecanal: can result from the oxidative breakdown of fatty acids present in lubricants, slip agents or food products.
- ethanol, 2-phenoxy: could originate from cosmetics or cleaning agents or from solvents used in adhesives and inks.
- benzene: can originate from the breakdown of contaminating PVC material.
- chloroxylenol: broad-spectrum antimicrobial agent widely used in household disinfectants.

f) Measurement or estimation of the migration levels to food of contaminants present in the recycled plastic materials and articles – Art 13(5)(f)

An estimation of the migration levels was made based on the average levels of incidental contaminants in the output samples in which they were detected (Table 1) and assuming a worst case total migration to food using the average weight of 27.2g PET for a one litre PET bottle (Table 4). Since EFSA (2024) acknowledges that generally recognised diffusion migration models overestimate migration by a factor of 5 for substances ≤ 150 Da and by a factor of 10 for substances > 150 Da, this worst case total migration also overestimates migration by at least these factors.

Table 4. Worst case migration of incidental contaminants present in the output samples.

Name	MW (g/mol)	CAS	OUTPUT		TOTAL MIGRATION* Average (µg/kg food)
			Frequency	Average (µg/kg PET)	
Limonene	136.26	5989-27-5	0%	<4.21	<0.11
Isophthalaldehyde	134.13	626-19-7	45%	19.41	0.53
Styrene	104.15	100-42-5	9%	10.19	0.28
1-Hexanol, 2-ethyl-	130.23	104-76-7	9%	<100	<2.72
Benzene, 1,2,3,5-tetramethyl-	134.22	95-93-2	27%	20.64	0.56
Indane	118.18	496-11-7	18%	25.91	0.70
n-Propyl acetate	102.13	109-60-4	64%	43.76	1.19
γ-Terpinene	136.23	99-85-4	0%	<4.21	<0.11
2-Decenal, (E)-	154.25	3913-81-3	18%	95.66	2.60
L-.alpha.-Terpineol	154.25	10482-56-1	9%	<100	<2.72
Benzene, 1-methyl-3-propyl-	134.22	1074-43-7	27%	70.52	1.92
1-Dodecanol	186.33	112-53-8	0%	<30.3	<0.82
Benzene, 1,2-diethyl-	134.22	135-01-3	18%	22.22	0.60
Benzene, 1-ethyl-2-methyl-	120.19	611-14-3	18%	10.71	0.29
Benzyl alcohol	108.14	100-51-6	9%	294.28	8.00

*considering 1L beverage filled in a PET bottle of 27.2g

Table 4. Worst case migration of incidental contaminants present in the output samples (continued)

Name	MW (g/mol)	CAS	OUTPUT		TOTAL MIGRATION* Average (µg/kg food)
			Frequency	Average (µg/kg PET)	
Dodecanal	184.32	112-54-9	9%	23.09	0.63
Benzene	78.11	71-43-2	9%	10.87	0.30
Benzene, propyl-	120.19	103-65-1	0%	<2.75	<0.075
Ethanol, 2-phenoxy-	138.16	122-99-6	9%	>1000	>27.2
Methyl salicylate	152.15	119-36-8	0%	<30.3	<0.82
Chloroxylenol	156.61	88-04-0	0%	<30.3	<0.82

*considering 1L beverage filled in a PET bottle of 27.2g

The worst case estimation of the migration levels of the inorganic substances is shown in Table 5.

Table 5. Worst case migration of incidental contaminants present in the output samples.

	OUTPUT		TOTAL MIGRATION* Average (mg/kg food)
	Frequency	Average (mg/kg PET)	
Cr	73%	1.21	0.03
Mn	100%	0.27	0.01
Fe	73%	5.13	0.14
Co	100%	13.57	0.37
Ni	100%	1.92	0.05
Zn	0%	<6.4	<0.17
Ge	100%	1.59	0.04
As	100%	0.09	0.00
Zr	27%	0.82	0.02
Ba	36%	3.00	0.08
Sb	100%	181.21	4.93
Se	0%	<3.6	<0.1
Pb	36%	2.83	0.08

*considering 1L beverage filled in a PET bottle of 27.2g

g) Description of the applied sampling strategy – Art 13(5)(g)

The PET Flake Injection recycling technology is a technology that is used for over 10 years to produce PET with recycled content for food contact applications. The individual recyclers using this technology have proven records that the output produced by recycling installations applying this technology is stable and complies with the requirements of Framework Regulation (EC) 1935/2004 and Plastics Regulation

(EU) No 10/2011. Therefore, the sampling frequency of the monitoring was reduced to one sample per recycler per monitoring cycle of 6 months.

In total 10 input batches and 11 corresponding output batches were collected. The samples were analysed for the following substances:

- Volatile substances,
- Semi-volatile substances,
- Non-volatile substances,
- Inorganic substances,
- Primary aromatic amines
- Bisphenols A, F and S
- Common plastic additives.

The analysis was carried out by an independent third-party analytical laboratory.

The Laboratory was chosen based on its experience and expertise in analysing PET samples and its relevant analytical equipment and validated methods.

h) Description of the analytical procedures and methods used – Art 13(5)(h)

Samples of PET input batches and their corresponding output batches were labelled for traceability purposes and shipped in clear and hermetically sealed containers.

The sample preparation methods and analytical procedures and methods used for the analysis of the samples as well as their limits of detection and quantification are summarised in Table 6. In all cases, 3 independent replicates were analysed.

Analysis of organic substances is done through a non-targeted screening of volatile, semi-volatile and non-volatile substances with different methods (Table 6).

For volatile substances, a solid phase microextraction in headspace mode connected to GC-MS method (HS-SPME-GC-MS) is used which is a versatile technique employed in a wide range of industries and research areas to identify, quantify, and characterize volatile and semi-volatile compounds in plastic/polymer samples. The concentration of the volatile and semi volatile compounds on the SPME microfibre increases a lot the sensitivity of the method in such a way that most of the volatile substances can be detected at very low concentrations. The adsorption conditions for SPME of 20 mins@80°C specifically allow the exhaustive extraction of volatile substances present in PET without degrading the sample. The detection is done by MS and the substances identification was performed using the NIST20 database (Match > 850) and retention index values (85% tolerance) which were calculated injecting an alkane solution (C8-40) in the same conditions as the analytes. Substances were (semi-)quantified by injecting known concentrations of commercially available standards corresponding to the detected substances. Calibration curves were prepared from these standards for the quantification. In the absence of a pure standard of the identified substance, the identified substance was semi-quantified with another substance of similar chemical structure.

For semi-volatile and non-volatile substance, the samples were first extracted. The solvent and extraction conditions have been chosen to swell the polymer, without generating new substances (Nerin *et al.*, 2022). The extracts were analysed using GC/MS and LC/MS-QToF for semi-volatile and non-volatile substances, respectively. High-resolution MS detectors like the QToF provide accurate masses isotopic patterns and intensities, which can lead to theoretical information about composition of fragments

(Peters *et al*. 2019). This allows for the identification of unknown NIAS. The identification of a given substance was based on its retention time, mass spectrum and the comparison of its analysis against commercial standards. PET oligomers were quantified with the commercially available $C_{20}H_{16}O_8$ PET oligomer standard.

The application ranges of the above used non-targeted screening methods overlap but the sensitivity of the methods is different. In case the same substance was detected by different methods, the highest concentration of both analyses was reported.

For the screening for primary aromatic amines a dedicated method was used as the concentration level of interest is so low that general non-target screening methods cannot detect them (Nerin *et al*., 2022). The primary aromatic amines were analysed after migration into 3% acetic acid.

Table 7 lists the primary aromatic amines that have been analysed.

Inorganic substances were analysed using ICP-MS after microwave digestion which is considered to be a sensitive elemental analysis technique that detects trace metals and non-metals at ultralow concentrations. Inorganic elements analysed were Cr, Mn, Fe, Co, Ni, Zn, Ge, As, Zr, Ba, Sb, Se, Pb.

The independent third-party laboratory follows ISO17025 quality control measures and all analytical methods are validated.

Table 6. Applied analytical procedures and methods including their limits of detection and quantification.

	Sample preparation	Analytical method	LOD	LOQ
Non-target screening of volatile and semi-volatile substances	Cryogenic milling 0.5 mm	HS-SPME-GC/MS, extraction 15 min @80°C	Between 2.75 and 41.36 µg/kg PET	Between 9.08 and 136.5 µg/kg PET
Non-target screening of semi and non-volatile substances	Cryogenic milling, dissolution in HFIP followed by precipitation of the polymer in methanol.	UPLC-MS-QTOF	5.4 mg/kg PET	16.4 mg/kg PET
Bisphenols A, S and F		UPLC-QqQ, negative mode	27.5 µg/kg PET	/
Common non-volatile additives		UPLC-MS-MS	Between 50 and 2750 µg/kg PET	/
Targeted analysis of inorganic substances (Annex II of EU 10/2011)	Microwave digestion	ICP-MS	Between 0.01 and 6.4 mg/kg PET	/
Primary aromatic amines	Migration in 3% acetic acid, 2h@70°C	UPLC-QqQ-MS, positive mode	Between 0.19 and 8.4 µg/kg PET	Between 0.63 and 27.72 µg/kg PET

HS: Head Space; SPME: Solid phase micro-extraction; GC: Gas chromatography; MS: Mass spectroscopy; QqQ: triple quadrupole; QToF: Quadrupole- time-of-flight; UPLC: ultra-high performance liquid chromatography; ICP: Inductively Couples Plasma; HFIP: 1,1,1,3,3,3-hexafluoroisopropanol
LOD: limit of detection; LOQ: limit of quantification

Table 7. List of primary aromatic amines analysed.

Name	CAS	Name	CAS
p-Fenilendiamine	106-50-3	3,3'-Dimethylbenzidine	119-93-7
m- Fenilendiamine	108-45-2	2,6-Dimethylaniline	87-62-7
2,6-Toluendiamine	823-40-5	4,4'-Thiodianiline	139-65-1
4-Methoxy-*m*-phenylenediamine	615-05-4	2,4-Dimethylaniline	95-68-1
2,4-Toluendiamine	95-80-7	2-Naphtylamine	91-59-8
1,5-Diaminonaphtalene	2243-62-1	4,4-Methylenedi-*o*-toluidine	838-88-0
Aniline	62-53-3	4-Aminobiphenyl	92-67-1
Benzidine	92-87-5	4-Aminoazobenzene	60-09-3
o-Anisidine	90-04-0	5-Nitro-o-toluidine	99-55-8
4,4-Oxidianiline	101-80-4	2,4,5-Trimethylaniline	137-17-7
o-Toluidine	95-53-4	4-Chloro-o-toluidine	95-69-2
4-Chloroaniline	106-47-8	o-Aminoazotoluene	97-56-3
4,4-Methylenedianiline	101-77-9	3,3-Dichlorobenzidine	91-94-1
o-Dianisidine	119-90-4	4,4-Methylene-bis-(2-chloroaniline)	101-14-4
2-Methoxy-5-m-toluidine	120-71-8		

i) Analysis and explanation of any discrepancies observed between contaminant levels expected and decontamination efficiency – Art 13(5)(i).

Detected contaminant levels

Overall, the levels of incidental contaminants detected in the input samples are in the µg/kg range and are far below the conservative reference level of incidental contaminants of 3 mg/kg PET, considered by EFSA in its scientific guidance on post-consumer mechanical PET recycling processes (2024). On the other hand, the results of the individual analyses of the different samples show a relatively high variation in concentration of the individual contaminants between the different samples ranging from non-detectable levels to, very occasionally, levels above 1000 µg/kg PET. In addition, there is also not always an explicable correlation between the levels detected in the input samples and those found in the output samples. This is due to the industrial scale of the recycling operations where, unlike for a challenge study, the input batch is not perfectly homogenous combined with the fact that, in comparison, only relatively small sample sizes are used for the analysis.

The incidental contaminants detected with a high frequency in the input samples are not unexpected (see section e).

Some of the incidental contaminants or inorganic elements were sometimes also detected in the output samples, but at a lower frequency and in most cases at a lower concentration. A safety assessment was carried out based on the following considerations:

- Exposure: average total migration levels as determined in Table 4. As explained in section f, a correction factor of 5 or 10 could be used if needed.

- Hazard: the following principles were used in order of priority:
    a. For substances listed in Annex I or for inorganic elements listed in Annex II of Regulation (EU) No 10/2011, the specific or overall migration limit is applied.
    b. For the other substances, the thresholds according to the Threshold of Toxicological Concern (TTC) approach and the latest EFSA guidance (2019) were used. The substances were assigned to the corresponding toxicity classes using the Toxtree software[1]:
        i. For DNA-reactive mutagens and/or carcinogens, the threshold is 0.0025 µg/kg body weight (bw) per day;
        ii. For organophosphates or carbamates, the threshold is 0.3 µg/kg bw per day;
        iii. All other substances were classified based on the extended Cramer rule bases into Cramer class I, II, or III substances for which thresholds of, respectively 30 µg/kg bw per day, 9 µg/kg bw per day and 1.5 µg/kg bw per day

Worst case exposure assessment and hazard assessment for incidental contaminants and inorganic elements are summarised in Table 8 and 9, respectively.

Based on the above assumptions, the data indicate that the worst case total migration concentration are below the applied safety thresholds for adult, toddler and infant food consumption scenarios, for all incidental contaminants except for the aldehydes isophthalaldehyde, 2-decanal and dodecanal for which Toxtree identifies a structural alert for genotoxic carcinogenicity.

For genotoxic substances, EFSA (2024) calculated by diffusion modelling Cmod values for certain surrogate contaminants. Cmod correspond to the maximum concentration level of the substance in the PET material that would not give rise to an exposure exceeding the threshold below which the risk to human health would be negligible. The following comparison between Cmods in scenario A[2] for certain surrogate contaminants and the concentration of the three aldehydes detected in the PET output was made:

- Isophthalaldehyde (134.13 Da): the average concentration of this substance in 5 of the 11 output samples in which it is detected is 19.41 µg/kg PET which is lower than the Cmod of 40 µg/kg PET for toluene (92.1 Da) which, from a molecular weight point of view, can be considered worst case as compared to isophtalaldehyde (EFSA, 2024).
- 2-decenal (154.25 Da): the average concentration of this substance in the 2 of the 11 output samples in which it is detected is 95.66 µg/kg PET which is lower than the Cmod of 120 µg/kg PET for methyl salicylate (152.2 Da) which has a similar molecular weight as 2-decenal (EFSA, 2024).
- Dodecanal (184.32 Da): the concentration of this substance in the only output sample in which it is detected is 23.09 µg/kg PET is lower than the Cmod of 120 µg/kg PET for methyl salicylate (152.2 Da) which, from a molecular weight point of view, can be considered worst case as compared to dodecanal (EFSA, 2024).

Therefore, based on the above assumptions, migration of these substances from the PET output to the food is not expected to give rise to a dietary exposure exceeding the threshold below which the risk to human health would be negligible.

[1] Toxtree version v3.1.0, May 2018
[2] Scenario A: drinking water/infants

Table 8. Worst case exposure assessment and hazard assessment of the incidental contaminants

Name	MW (g/mol)	CAS	Frequency	Average (µg/kg PET)	TOTAL MIGRATION* average (µg/kg food)	
Limonene	136.26	5989-27-5	0%	<4.21	<0.11	Cramer I
Isophthalaldehyde	134.13	626-19-7	45%	19.41	0.53	Structural alert for genotoxic carcinogenicity
Styrene	104.15	100-42-5	9%	10.19	0.28	FCM193 w/o SML
1-Hexanol, 2-ethyl-	130.23	104-76-7	9%	<100	<2.72	Cramer I
Benzene, 1,2,3,5-tetramethyl-	134.22	95-93-2	27%	20.64	0.56	Cramer I
Indane	118.18	496-11-7	18%	25.91	0.70	Cramer III
n-Propyl acetate	102.13	109-60-4	64%	43.76	1.19	Cramer I
γ-Terpinene	136.23	99-85-4	0%	<4.21	<0.11	Cramer I
2-Decenal, (E)-	154.25	3913-81-3	18%	95.66	2.60	Structural alert for genotoxic carcinogenicity
L-.alpha.-Terpineol	154.25	10482-56-1	9%	<100	<2.72	Cramer I
Benzene, 1-methyl-3-propyl-	134.22	1074-43-7	27%	70.52	1.92	Cramer I
1-Dodecanol	186.33	112-53-8	0%	<30.3	<0.82	Cramer I
Benzene, 1,2-diethyl-	134.22	135-01-3	18%	22.22	0.60	Cramer I
Benzene, 1-ethyl-2-methyl-	120.19	611-14-3	18%	10.71	0.29	Cramer I
Benzyl alcohol	108.14	100-51-6	9%	294.28	8.00	Cramer I
Dodecanal	184.32	112-54-9	9%	23.09	0.63	Structural alert for genotoxic carcinogenicity
Benzene	78.11	71-43-2	9%	10.87	0.30	EU Drinking water limit: 1 µg/L
Benzene, propyl-	120.19	103-65-1	0%	<2.75	<0.075	Cramer I
Ethanol, 2-phenoxy-	138.16	122-99-6	9%	>1000	>27.2	Cramer II
Methyl salicylate	152.15	119-36-8	0%	<30.3	<0.82	Cramer I
Chloroxylenol	156.61	88-04-0	0%	<30.3	<0.82	Cramer III

*considering 1L beverage filled in a PET bottle of 27.2g

With regard to the inorganic substances detected in the output samples, the worst case migration level would exceed the migration limits established in Regulation (EU) No 10/2011 for antimony, cobalt, chromium, nickel, arsenic and lead.

However, regarding antimony, Welle and Franz (2011) showed that, due to the extremely low diffusion coefficients of antimony species in PET, the SML will not be exceeded under standard use of PET at room temperature and/or hotfill conditions with antimony concentrations up to 350 mg/kg. Since antimony levels in the output samples were below these levels, there would be no safety concern.

For the other inorganic substances, no such studies are available. Since the exact molecular identity under which inorganic substances are present is not known, migration modelling cannot be performed and only migration testing can rule out the risk of exceeding the migration limits. Consortium members have done migration testing on different output batches and confirmed compliance with the migration limits of Annex II of Regulation (EU) No 10/2011. In addition, verification of compliance with migration

limits established in Regulation (EU) No 10/2011 is part of the routine compliance work performed by the users of the material.

Table 9. Results of the safety evaluation of the incidental contaminants

	OUTPUT		TOTAL MIGRATION* Average (mg/kg food)	EU 10/2011 - Annex II (SML (mg/kg food))
	Frequency	Average (mg/kg PET)		
Cr	73%	1.21	0.03	ND
Mn	100%	0.27	0.01	0.6
Fe	73%	5.13	0.14	48
Co	100%	13.57	0.37	0.05
Ni	100%	1.92	0.05	0.02
Zn	0%	<6.4	<0.17	5
Ge	100%	1.59	0.04	/
As	100%	0.09	0.00	ND
Zr	27%	0.82	0.02	/
Ba	36%	3.00	0.08	1
Sb	100%	181.21	4.93	0.04
Se	0%	<3.6	<0.1	/
Pb	36%	2.83	0.08	ND

*considering 1L beverage filled in a PET bottle of 27.2g
ND: not detectable with detection limit of 0.01 mg/kg food

Decontamination efficiency

As indicated in section b, it was determined, based on the results of a challenge study, that the decontamination efficiency of the Flake Injection Novel Technology was above or far above 99.9%.

In this report, the decontamination efficiencies for the different contaminants in the samples were calculated based on the levels of contaminants in the input and output samples. For the calculation, the following rules were applied:

- Whenever the concentration in a sample is below the limit of quantification or the limit of detection, the value of the limit of quantification or the value of the limit of detection, respectively, was used as worst case approach.
- To ensure that the calculated decontamination efficiencies are not artificially increased[3] by a potential dilution of the input material with virgin material, the measured concentrations of incidental contaminants in the input material (Table 1) were corrected for the percentage virgin material used to produce the analysed batches of output material, as explained in section c.

As a result, the calculated concentration of incidental contaminants in the input material was frequently below the limit of detection of the substance. In such a case, if the substance was not detectable in the output material, the calculation generates a seemingly negative decontamination efficiency that is not relevant because it is not a real decontamination efficiency. Similarly, if the concentration of the

[3] Article 13 of Regulation (EU) 2022/1616[3] requires that residual contaminant levels in the output are determined before any dilution of the output material

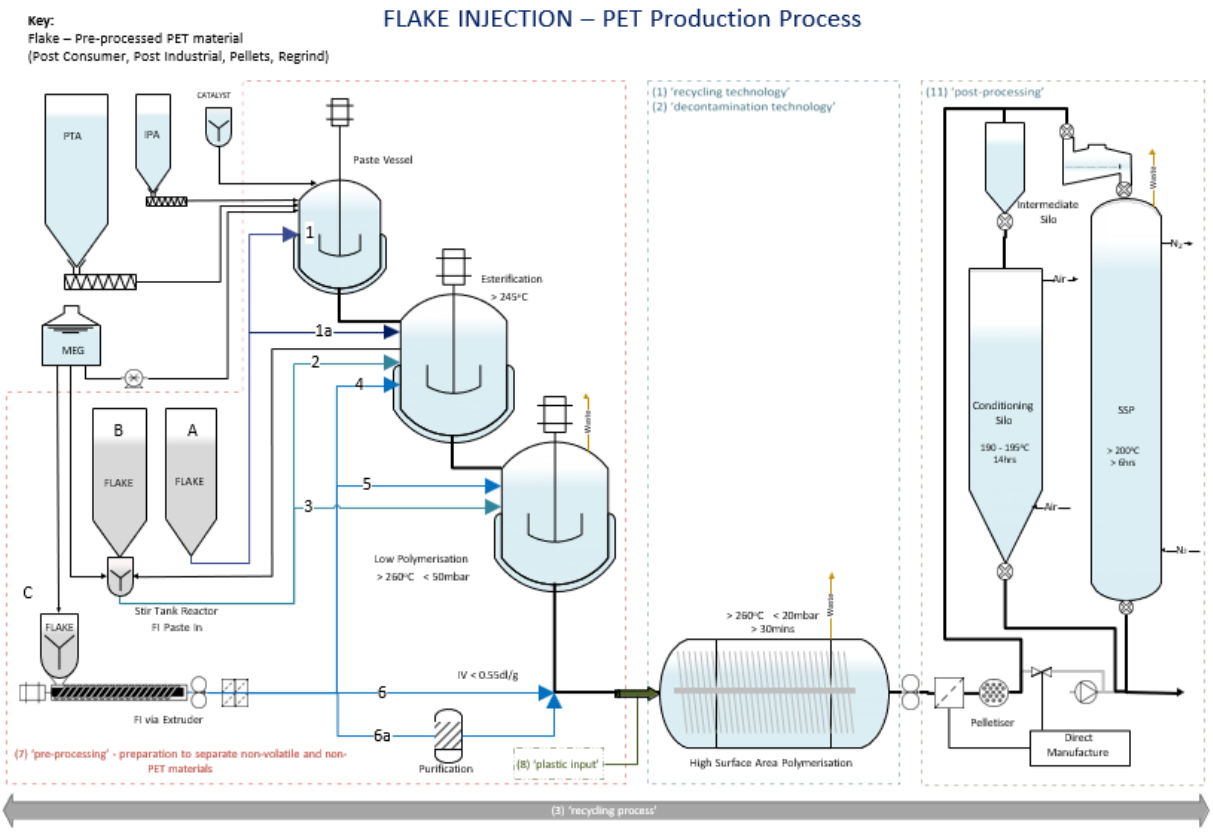
incidental contaminant is below the limit of detection or the limit of quantification in both the input and the output sample, the obtained value is also not relevant as it is not the actual decontamination efficiency.

While high decontamination efficiencies (values up to >99.5%) were found for most incidental contaminants in several input-output sample sets, the average decontamination efficiency cannot be demonstrated for all the incidental contaminants of the sample sets due to the limitations described above.

In addition, it is technically impossible to confirm a decontamination efficiency of 99.9% as reported in the Novel Technology dossier due to the analytical limitations associated with the relatively low levels of incidental contaminants detected in the input materials. Despite the very low analytical detection limits of the applied state-of-the-art analytical equipment that go down to 2.75 µg/kg PET, the contaminant levels in the input material would still have to reach 2.75 mg/kg PET to be able to confirm the reported decontamination efficiency of 99.9%. Such high incidental contaminant levels were never observed in the analysed input samples. The highest incidental contaminant level observed in the analysed input samples of this monitoring report is 1 mg/kg PET.

In conclusion, although there are a number of indications that the Flake Injection Novel Technology can achieve a high decontamination efficiency, a decontamination efficiency of 99.9% cannot be technically/mathematically demonstrated with the current low levels of incidental contaminants in the input samples and the monitoring testing methodology as defined in Article 13 of Regulation (EU) 2022/1616 mainly due to analytical limitations.

Appendix I – FLAKE INJECTION – PET Production Process



Appendix II – List of all substances with a molecular weight below 1.000 Dalton found in the plastic inputs to each of the decontamination installations and in the recycled plastic output thereof, sorted in descending order by their relative occurrence

Name	Formula	CAS	Frequency INPUT	Frequency OUTPUT
L[TPA + EG]3 + EG	C32H30O14	16033-73-1	100%	100%
2,2-Dimethyl-1-phenyl-1-propanol	C11H16O	3835-64-1	100%	100%
Butanoic acid	C4H8O2	107-92-6	100%	100%
Octanoic acid, 2-methyl-, methyl ester	C9H18O2	3004-93-1	100%	100%
Tetradecanoic acid	C14H28O2	544-63-8	100%	100%
Propylene Glycol	C3H8O2	57-55-6	100%	100%
Benzoic acid, methyl ester	C8H8O2	93-58-3	100%	100%
Hexanoic acid	C6H12O2	142-62-1	100%	100%
Pentanoic acid	C5H10O2	109-52-4	100%	100%
C[TPA + DEG]2	C24H24O10	16104-98-6	100%	100%
C[TPA + EG] + [TPA + DEG]	C22H20O9	29278-57-7	100%	100%
C[TPA + EG]2	C20H16O8	24388-68-9	100%	100%
C[TPA + EG]2 + [TPA + DEG]	C32H28O13	873422-64-1	100%	100%
C[TPA + EG]3	C30H24O12	7441-32-9	100%	100%
1,3-Dioxolane, 2-methyl-	C4H8O2	497-26-7	100%	92%
Benzaldehyde	C7H6O	100-52-7	100%	91%
p-Cymene	C10H14	99-87-6	100%	91%
Benzene, 1,2,4-trimethyl-	C9H12	95-63-6	100%	82%
Butyrolactone	C4H6O2	96-48-0	100%	73%
Mesitylene	C9H12	108-67-8	100%	55%
2-Butenal	C4H6O	4170-30-3	100%	45%
Benzene, 1,2,3-trimethyl-	C9H12	526-73-8	100%	36%
1-Propanol, 2,2'-oxybis-	C6H14O3	108-61-2	90%	82%
L[TPA + EG]2	C20H18O9	23186-89-2	90%	73%
Nonanal	C9H18O	124-19-6	90%	64%
Isophthalaldehyde	C8H6O2	626-19-7	90%	45%
Benzene, 1-ethyl-3-methyl-	C9H12	620-14-4	90%	27%
Bicyclo[3.3.1]non-3-en-2-ol, exo-	C9H14O	10060-21-6	90%	27%
Benzene, 1-ethyl-3,5-dimethyl-	C10H14	934-74-7	90%	18%
Styrene	C8H8	100-42-5	90%	9%
Limonene	C10H16	5989-27-5	90%	0%
1,4-Dioxane	C4H8O2	123-91-1	80%	91%
2-Propanol, 1,1'-oxybis-	C6H14O3	110-98-5	80%	82%
n-Propyl acetate	C5H10O2	109-60-4	80%	64%
L[TPA + EG]3	C30H26O13	16958-96-6	80%	64%

Name	Formula	CAS	Frequency INPUT	Frequency OUTPUT
Benzene, 1,2,3,5-tetramethyl-	C10H14	95-93-2	80%	27%
Indane	C9H10	496-11-7	80%	18%
1-Hexanol, 2-ethyl-	C8H18O	104-76-7	80%	9%
Naphthalene	C10H8	91-20-3	70%	36%
p-Xylene	C8H10	106-42-3	70%	18%
γ-Terpinene	C10H16	99-85-4	70%	0%
C[TPA + EG]3 + [TPA + DEG]	C42H36O17	2222729-29-3	60%	64%
Benzene, 1-methyl-4-propyl-	C10H14	1074-55-1	60%	55%
Toluene	C7H8	108-88-3	60%	55%
2-Decenal, (E)-	C10H18O	3913-81-3	60%	18%
L-.alpha.-Terpineol	C10H18O	10482-56-1	60%	9%
Ethylbenzene	C8H10	100-41-4	60%	9%
Benzene, 1-methyl-3-propyl-	C10H14	1074-43-7	50%	27%
o-Xylene	C8H10	95-47-6	50%	9%
Benzene, 1,2-diethyl-	C10H14	135-01-3	40%	18%
Benzene, 1-ethyl-2-methyl-	C9H12	611-14-3	40%	18%
Benzyl alcohol	C7H8O	100-51-6	40%	9%
Dodecanal	C12H24O	112-54-9	40%	9%
Diphenyl ether	C12H10O	101-84-8	40%	9%
1-Dodecanol	C12H26O	112-53-8	40%	0%
2-Propanol, 1-methoxy-	C4H10O2	107-98-2	30%	18%
Benzene	C6H6	71-43-2	30%	9%
Ethanol, 2-phenoxy-	C8H10O2	122-99-6	30%	9%
Benzene, propyl-	C9H12	103-65-1	30%	0%
Methyl salicylate	C8H8O3	119-36-8	30%	0%
Cyclic NPG-TPA-NPG-TPA	C26H28O8		20%	27%
Biphenyl	C12H10	92-52-4	20%	27%
1-Propanol, 2-(2-hydroxypropoxy)-	C6H14O3	106-62-7	20%	18%
Chloroxylenol	C8H9ClO	88-04-0	20%	0%
Phenol	C6H6O	108-95-2	10%	9%
1-Dodecen-3-ol	C12H24O	4048-42-4	10%	0%
Anethole	C10H12O	104-46-1	10%	0%
α-Himachalene	C15H24	3853-83-6	10%	0%
1,2,3-propanetriol 1-stearate 2,3-bisacetate	C25H46O6	33599-07-4	10%	0%

Glossary of Terms

Cmod	Modelled concentration
Cres	Residual concentration
DEG	diethylene glycol
EG	ethylene glycol
GC	gas chromatography
HPLC	high performance liquid chromatography
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
MS	Mass Spectrometry
NIAS	non-intentionally added substances
PE	polyethylene
PET	polyethylene terephthalate
PP	polypropylene
PVC	polyvinyl chloride
TPA	terephthalic acid
TTC	Threshold of Toxicological Concern

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