PET Flake Injection

Novel Technology Development

Data Monitoring Report

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

10 April 2024

Amended with additional results 14th May 2024

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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 provides a summary of the data forthcoming from the monitoring, based on the latest information from all installations using the novel technology received in accordance with paragraph 3 along with the information required by Article 13(5) of the Regulation.

a) Brief description of the novel technology

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 and is deliberately depolymerized (preprocessed) 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.

Flake To Resin (FTR)

Ref. ANNEX II Table 1 (1) Decontamination efficiency of a new post-consumer poly(ethylene terephthalate) (PET) recycling concept. FRANK WELLE. Fraunhofer Institute for Process Engineering and Packaging (IVV), Giggenhauser Straße 35, 85354 Freising, Germany.

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 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 foodcontact 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

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.

The data analysis from the analytical reports for the Input & output materials is currently ongoing along with the addition of some missing test data not yet provided by the third party laboratory. Therefore, the outcome of the analysis will be added to the report as soon as they become available which is expected in the next few weeks.

Volatiles - To be completed on receipt of the delayed analysis report

				Input	Output	
	Name	Formula	CAS	Input µg/kg PET	Outputµg /kg PET	Cleaning efficiency, %
SAMPLE1						

Volatiles (contd)

Volatiles (contd)

Non Volatiles -

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE I	5.46	429.1187	Cyclic TPA2-EG-DEG	C22H20O9	1170	870	25.6
	6.22	577.1339	Cyclic (TPA-EG)3	C30H24O12	850	570	32.9
	7.24	523.3242	Oligomer	C27H48O8	650		100.0
	6.26	621.1603	Cyclic (TPA3-EG2-DEG)	C32H28O13	543	363	33.1
	4.89	385.0915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/zC20H18O9	300	115	61.7
	5.93	385.0922	Cyclic (TPA-EG)2	C20H16O8	300	120	60.0
	5.4	473.1453	Cyclic (TPA-DEG)2	C24H24O10	283	211	25.4
	5.64	661.1533	TPA3-EG2-DEG+H2O	C32H30O14	68	30	55.9

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 2	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	1000	786	21.4
	6.22	577.1339/	Cyclic (TPA-EG)3	C30H24O12	622	455	26.8
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	404	395	2.2
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	287	94.9	66.9
	5.4	4731453	Cyclic (TPA-DEG)2	C24H24O10	193	215	-11.4
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	145	376	-159.3
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	112	63.3	43.5

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 3	5.46	429.1187	Cyclic TPA2-EG-DEG	C22H20O9	1108	1090	1.6
	6.22	577.1339	Cyclic (TPA-EG)3	C30H24O12	854	784	8.2
	6.26	621.1603	Cyclic (TPA3-EG2-DEG)	C32H28O13	430	435	-1.2
	4.89	385.0915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/zC20H18O9	305	265	13.1
	5.4	473.1453	Cyclic (TPA-DEG)2	C24H24O10	226	296	-31.0
	5.93	385.0922	Cyclic (TPA-EG)2	C20H16O8	185	135	27.0
	5.64	661.1533	TPA3-EG2-DEG+H2O	C32H30O14	72	60	16.7

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 4	5.46	4291187	Cíclico (TPA3-EG2DEG)	C22H20O9	1173	883	24.7
	6.22	5771339	Cíclico (TPA-EG)2	C30H24O12	914	654	28.4
	6.26	6211603	(TPA-DEG)2+H2O	C32H28O13	509	376	26.1
	4.89	3850915	Cíclico TPA2-EG-D(TPA-DEG)2+H2OCyclic (TPA-DEG)2Cyclic T	Fragment of 425.0843 m/z C20H18O9	339	233	31.3
	5.40	4731453	Cíclico (TPA-EG)3	C24H24O10	266	256	3.8
	5.93	3850922	Cyclic (TPA4-EG3DEG)	C20H16O8	105	115	-9.5
	5.64	6611533	Cíclico (TPA-DEG)2	C32H30O14	85	45	47.1

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 5	5.46	429.1187	Cyclic TPA2-EG-DEG	C22H20O9	1086	786	27.6
	7.24	523.3242	Oligomer	C27H48O8	970	0	100.0
	6.22	577.1339	Cyclic (TPA-EG)3	C30H24O12	950	476	49.9
	6.26	621.1603	Cyclic (TPA3-EG2-DEG)	C32H28O13	435	285	34.5
	4.89	385.0915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	290	114	60.7
	5.93	385.0922	Cyclic (TPA-EG)2	C20H16O8	290	113	61.0
	5.4	473.1453	Cyclic (TPA-DEG)2	C24H24O10	233	223	4.3
	5.64	661.1533	TPA3-EG2-DEG+H2O	C32H30O14	62	22	64.5

Non Volatiles (contd)

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 6	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	3306	223	93.3
	6.22	5771339	Cyclic (TPA-EG)3	C30H24O12	2103	73.2	96.5
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	725	23.7	96.7
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	603	60.3	90.0
	5.4	4731453	Cyclic (TPA-DEG)2	C24H24O10	581	655	-12.7
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	474	342	27.8
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	376	114	69.7
	7.24	5233242	Oligomer	C27H48O8	79	61.5	22.2

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 7	7.24	5233242	Oligomer	C27H48O8	6220	490	92.1
	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	932	2740	-194.0
	6.22	5771339	Cyclic (TPA-EG)3	C30H24O12	574	695	-21.1
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	366		100.0
	5.4	4731453	Cyclic (TPA-DEG)2	C24H24O10	227	473	-108.4
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	167	3930	-2253.3
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	117	67.6	42.2
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	58.4	373	-538.7
	3.37	1930488	(C10H9O4)n=1	C10H9O4		406	-
	3.6	3210935	Oligomer	C14H18O7		133	-

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 8	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	1050	1210	-15.2
	6.22	5771339	Cyclic (TPA-EG)3	C30H24O12	674	649	3.7
	7.24	5233242	Oligomer	C27H48O8	650	425	34.6
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	376	457	-21.5
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	237	83.8	64.6
	5.40	4731453	Cyclic (TPA-DEG)2	C24H24O10	223	286	-28.3
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	163	82.9	49.1
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	82.9	42.8	48.4

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 9	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	973	917	5.8
	6.22	577.1339/	Cyclic (TPA-EG)3	C30H24O12	608	608	0.0
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	382	417	-9.2
	7.24	5233242	Oligomer	C27H48O8	343	107	68.8
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	246	131	46.7
	5.4	4731453	Cyclic (TPA-DEG)2	C24H24O10	245	289	-18.0
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	165	147	10.9
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	94.5	82.7	12.5

	RT	Mass	Candidate	Formula	Input µg/kg PET	Output µg/kg PET	Cleaning efficiency, %
SAMPLE 10	5.46	4291187	Cyclic TPA2-EG-DEG	C22H20O9	1127	236	79.1
	6.22	5771339	Cyclic (TPA-EG)3	C30H24O12	928	1202	-29.5
	6.26	6211603	Cyclic (TPA3-EG2-DEG)	C32H28O13	422		100.0
	4.89	3850915	(TPA-DEG)2+H2O	Fragment of 425.0843 m/z C20H18O9	377	686	-82.0
	5.40	4731453	Cyclic (TPA-DEG)2	C24H24O10	210	2410	-1047.6
	5.93	3850922	Cyclic (TPA-EG)2	C20H16O8	182	749	-311.5
	5.64	6611533	TPA3-EG2-DEG+H2O	C32H30O14	112	82	26.8

Inorganic Substances – Missing some Input data to be completed on receipt of the completed delayed analysis report

SAMPLE 1

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		< LOQ	0,053
Be		< LOQ	1,083
Na		< LOQ	3,152
Mg		< LOQ	0,197
Al		< LOQ	0,571
K		< LOQ	45,840
Ca		< LOQ	12,064
Ti		< LOQ	0,533
V		< LOQ	0,040
Cr		< LOQ	3,973
Mn		< LOQ	0,003
Fe		< LOQ	70,773
Co		< LOQ	0,032
Ni		< LOQ	0,179
Cu		< LOQ	0,061
Zn		0,976	0,560
Ga		< LOQ	0,027
Ge		< LOQ	0,120
As		< LOQ	0,307
Se		< LOQ	2,493
Rb		< LOQ	0,040
Sr		< LOQ	0,016
Y		< LOQ	0,016
Zr		0,059	0,016
Nb		< LOQ	0,016
Mo		< LOQ	0,040
Ru		< LOQ	0,085
Pd		< LOQ	0,016
Ag		< LOQ	0,083
Sb		348	± 35

SAMPLE 2

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0.157	0,053
Be		< LOQ	1.083
Sc		< LOQ	0.003
Ti		0.124	0.533
V		0.013	0.04
Cr		0.014	3.973
Mn		0.009	0.003
Fe		< LOQ	70.773
Co		0	0.032
Ni		< LOQ	0.179
Cu		0.015	0.061
Zn		0.003	0.56
Ga		0.005	0.027
Ge		< LOQ	0.12
As		< LOQ	0.307
Se		0.001	2.493
Rb		0.001	0.04
Sr		< LOQ	0.016
Y		< LOQ	0.016
Zr		0.013	0.016
Nb		< LOQ	0.016
Mo		< LOQ	0.04
Ru		1.734	0.085
Pd		< LOQ	0.016
Ag		0.005	0.083
Sb		313 ± 31	

SAMPLE 3

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0,400	0,053
Be		< LOQ	1083
Sc		22408	0,003
Ti		< LOQ	0,533
V		< LOQ	0,040
Cr		< LOQ	3.973
Mn		< LOQ	0,003
Fe		< LOQ	70.773
Co		0,085	0,032
Ni		< LOQ	0,179
Cu		1301	0,061
Zn		2227	0,560
Ga		< LOQ	0,027
Ge		< LOQ	0,120
Se		< LOQ	2493
Rb		0,224	0,040
Sr		0,491	0,016
Y		0,027	0,016
Zr		0,123	0,016
Nb		< LOQ	0,016
Mo		0,059	0,040
Ru		< LOQ	0,085
Pd		< LOQ	0,016
Ag		< LOQ	0,083
Sb		379 ± 38	

SAMPLE 4

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0.072	0.053
Be		< LOQ	1.083
Sc		7.659	0.003
Ti		< LOQ	0.533
V		< LOQ	0.04
Cr		8.771	3.973
Mn		< LOQ	0.003
Fe		< LOQ	70.773
Co		59.013	0.032
Ni		3.347	0.179
Cu		3.453	0.061
Zn		8.813	0.56
Ga		0.069	0.027
Ge		46.107	0.12
As		0.925	0.307
Se		< LOQ	2.493
Rb		0.323	0.04
Sr		1.277	0.016
Y		0.019	0.016
Zr		0.093	0.016
Nb		< LOQ	0.016
Mo		0.131	0.04
Ru		< LOQ	0.085
Pd		< LOQ	0.016
Ag		< LOQ	0.083
Sb		92± 9	

SAMPLE 5

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0,157	0,053
Be		< LOQ	1083
Sc		< LOQ	0,003
Ti		< LOQ	0,533
V		< LOQ	0,040
Cr		< LOQ	3973
Mn		< LOQ	0,003
Fe		< LOQ	70773
Co		0,421	0,032
Ni		< LOQ	0,179
Cu		0,315	0,061
Zn		7256	0,560
Ga		0,091	0,027
Ge		< LOQ	0,120
As		0,496	0,307
Se		< LOQ	2493
Rb		0,301	0,040
Sr		0,408	0,016
Y		0,024	0,016
Zr		0,056	0,016
Nb		< LOQ	0,016
Mo		0,136	0,040
Ru		< LOQ	0,085
Pd		< LOQ	0,016
Ag		< LOQ	0,083
Sb		319± 32	± 32

SAMPLE 6

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li	0.189	< LOQ	0.053
Be	< LOQ	< LOQ	1.083
Sc	< LOQ	18.029	0.003
Ti	1.029	< LOQ	0.533
V	< LOQ	< LOQ	0.04
Cr	< LOQ	< LOQ	3.973
Mn	< LOQ	< LOQ	0.003
Fe	< LOQ	< LOQ	70.773
Co	0.227	0.173	0.032
Ni	21.149	< LOQ	0.179
Cu	3.301	0.275	0.061
Zn	8.109	9.357	0.56
Ga	< LOQ	< LOQ	0.027
Ge	< LOQ	< LOQ	0.12
As	0.323	< LOQ	0.307
Se	< LOQ	< LOQ	2.493
Rb	0.296	0.157	0.04
Sr	0.773	0.997	0.016
Y	0.021	< LOQ	0.016
Zr	0.069	0.069	0.016
Nb	< LOQ	< LOQ	0.016
Mo	0.061	0.117	0.04
Ru	< LOQ	< LOQ	0.085
Pd	< LOQ	< LOQ	0.016
Ag	< LOQ	< LOQ	0.083
Sb	324± 32	265± 26	

SAMPLE 7

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		1.272	0.053
Be		< LOQ	1.083
Sc		< LOQ	0.003
Ti		< LOQ	0.533
V		< LOQ	0.04
Mn		< LOQ	0.003
Fe		< LOQ	70.773
Co		1.291	0.032
Ni		< LOQ	0.179
Cu		3.299	0.061
Zn		27.653	0.56
Ga		0.2	0.027
Ge		0.144	0.12
As		2.131	0.307
Se		< LOQ	2.493
Rb		0.635	0.04
Sr		2.024	0.016
Y		0.032	0.016
Zr		0.075	0.016
Nb		< LOQ	0.016
Mo		0.2	0.04
Ru		< LOQ	0.085
Pd		< LOQ	0.016
Ag		< LOQ	0.083
Sb		243± 24	

SAMPLE 8

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0.144	0.053
Be		< LOQ	1.083
Sc		20.157	0.003
Ti		< LOQ	0.533
V		< LOQ	0.04
Cr		8.677	3.973
Mn		< LOQ	0.003
Fe		< LOQ	70.773
Co		0.184	0.032
Ni		1.715	0.179
Cu		37.013	0.061
Zn		1.613	0.56
Ga		0.04	0.027
Ge		< LOQ	0.12
As		< LOQ	0.307
Se		< LOQ	2.493
Rb		0.187	0.04
Sr		0.693	0.016
Y		< LOQ	0.016
Zr		0.056	0.016
Nb		< LOQ	0.016
Mo		0.061	0.04
Ru		< LOQ	0.085
Pd		< LOQ	0.016
Ag		< LOQ	0.083
Sb		278 ± 28	

SAMPLE 9

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		< LOQ	0.053
Be		< LOQ	1.083
Na		1.13	3.152
Mg		< LOQ	0.197
Al		< LOQ	0.571
K		0.188	45.84
Ca		< LOQ	12.064
Sc		< LOQ	0.003
Ti		< LOQ	0.533
V		< LOQ	0.04
Cr		< LOQ	3.973
Mn		< LOQ	0.003
Fe		0	70.773
Co		< LOQ	0.032
Ni		< LOQ	0.179
Cu		0.003	0.061
Zn		0.003	0.56
Ga		< LOQ	0.027
Ge		< LOQ	0.12
As		< LOQ	0.307
Se		< LOQ	2.493
Rb		< LOQ	0.04
Sr		< LOQ	0.016
Y		0.001	0.016
Zr		< LOQ	0.016
Nb		< LOQ	0.016
Mo		< LOQ	0.04
Ru		< LOQ	0.085
Pd		< LOQ	0.016
Ag		1.13	0.083
Sb		326± 32	

SAMPLE 10

Substance	INPUT mg/Kg of PET	OUTPUT mg/Kg of PET	LOQ (ug/Kg)
Li		0.323	0.053
Be		< LOQ	1.083
Ca		< LOQ	12.064
Sc		19.037	0.003
Ti		< LOQ	0.533
V		< LOQ	0.04
Cr		12.296	3.973
Mn		< LOQ	0.003
Fe		< LOQ	70.773
Co		0.037	0.032
Ni		< LOQ	0.179
Cu		1.112	0.061
Zn		6.949	0.56
Ga		0.083	0.027
Ge		< LOQ	0.12
As		0.779	0.307
Se		< LOQ	2.493
Rb		0.464	0.04
Sr		0.475	0.016
Y		0.035	0.016
Zr		0.059	0.016
Nb		< LOQ	0.016
Mo		< LOQ	0.04
Ru		< LOQ	0.085
Pd		< LOQ	0.016
Ag		< LOQ	0.083
Sb		379± 38	

Primary Aromatic Amines -

Primary Aromatic Amines were not detected in the Input or Output samples with the following exception Sample 7 .

No	Analite	Name	CAS	LOQ (µg/Kg PET)	Sample pellets
1	p-PDA	*p*-Fenilendiamina	106-50-3	79.8	< LOQ
2	m-PDA	*m*- Fenilendiamina	108-45-2	79.8	< LOQ
3	2,6-TDA	2,6-Toluendiamina	823-40-5	14.6	< LOQ
4	4-M-m-PDA	4-Methoxy-*m*-phenylenediamine	615-05-4	14.6	< LOQ
5	2,4-TDA	2,4-Toluendiamina	95-80-7	14.6	< LOQ
6	1,5-DAN	1,5-Diaminonaftaleno	2243-62-1	16.5	< LOQ
7	ANL	Anilina	62-53-3	11.3	112,83 ± 6,31
8	BNZ	Bencidina	92-87-5	41.3	< LOQ
9	o-ASD	*o*-Anisidina	90-04-0	99	< LOQ
10	4,4-DPE	4,4-Oxidianilina	101-80-4	20.1	< LOQ
11	o-T	o-Toluidina	95-53-4	33	< LOQ
12	4-CA	4-Cloroanilina	106-47-8	33	< LOQ
13	4,4-MDA	4,4-Metilenodianilina	101-77-9	21.5	< LOQ
14	o-diASD	*o*-Dianisidina	119-90-4	3	< LOQ
15	2-M-5-MA	2-Metoxi-5-m-toluidina	120-71-8	41.3	< LOQ
16	3,3-DMB	3,3-Dimetilbencidina	119-93-7	17.9	< LOQ
17	2,4-DMA	2,4-Dimetilanilina	87-62-7	3	< LOQ
18	4,4'-thioANL	4,4'-Tiodianilina	139-65-1	71.5	< LOQ
19	2,6-DMA	2,6-Dimetilanilina	95-68-1	3	< LOQ
20	2-NA	2-Naftilamina	91-59-8	7.7	< LOQ
21	4,4-MDoT	4,4-Metilenodi-*o*-toluidina	838-88-0	85.3	< LOQ
22	4-ABP	4-Aminobifenilo	92-67-1	41.3	< LOQ
23	4-AAB	4-Aminoazobenceno	60-09-3	17.3	< LOQ
24	5-N-o-T	5-Nitro-o-toluidina	99-55-8	5.8	< LOQ
25	1,4,5-TMA	2,4,5-Trimetilanilina	137-17-7	22	< LOQ
26	4-CT	4-Cloro-o-toluidina	95-69-2	63.3	< LOQ
27	AAT	o-Aminoazotolueno	97-56-3	5	< LOQ
28	3,3-DCB	3,3-Diclorobencidina	91-94-1	129.3	< LOQ
29	4,4-MCA	4,4-Metileno-bis-(2-cloroanilina)	101-14-4	4.4	< LOQ

Plastics Additives –

Plastics Additives Indicated in the following table were not detected in the Input or Output Samples

Additives	CAS	LOD (µg/kg PET)	Results Input & Output
Irgafos 168	31570-04-4	LOD=110	< LOD
TopanolCA	1843-03-4	LOD=2750	< LOD
Chimassorb 81	1843-05-6	LOD=113	< LOD
Cyasorb UV 1084	14516-71-3	LOD=850	< LOD
Tinuvin 326	05/11/3896	LOD=157	< LOD
Irganox1010	6683-19-8	LOD=83	< LOD
Tinuvin 327	01/09/3864	LOD=270	< LOD
Irgafos 38	145650-60-8	LOD=570	< LOD
Irganox 1076	2082-79-3	LOD=725	< LOD
Tinuvin P	2440-22-4	LOD=2700	< LOD
9,9-bis(methoxymethyl)fluorene	182121-12-6	LOD=75	< LOD
N,N-Bis(2-hydroxyethyl)alkylamines (C12)	942-293-6	LOD=50	< LOD
Erucamide	112-84-5	LOD=193	< LOD
Bis(2-ethylhexyl) adipate	103-23-1	LOD=82	< LOD
Tributylcitrate	77-94-1	LOD=105	< LOD
Trybutyl o-acetylcitrate	77-90-7	LOD=75	< LOD
TXIB (2,2,4- Trimethyl-1,3-pentanedioldiisobutyrate)	6846-50-0	LOD=2600	< LOD
Bis(2-ethylhexyl) sebacate	122-62-3	LOD=52	< LOD
NX8000	882073-43-0	LOD=1650	< LOD

d) List of contaminating materials regularly present in the plastic input

Table 1 lists the contaminating materials regularly present in the plastic input.

Typical Residuals		
Property	Maximum	Units
PVC	50	mg/kg
Polyolefin (caps/labels)	20	mg/kg
Other Polymers	100	mg/kg
Metal	10	mg/kg
Other Inert Materials	30	mg/kg

Table 1. Contaminating materials regularly present in the plastic input.

e) Analysis of the most likely origin of the identified contaminants referred to in points (c) and (d).

Input material

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

The likely origin of the substances detected in the input material is as follows:

- To be completed on receipt of the delayed analysis report

Output material

- To be completed on receipt of the delayed analysis report

f) Measurement or estimation of the migration levels to food of contaminants present in the recycled plastic materials and articles.

Potential migration - To be completed on receipt of the delayed analysis report

Assuming worse case 100% of migration to food and considering that the average weight of PET of one litre PET bottle is 27.2g, the potential migration would be:

Migration levels continued

g) Description of the applied sampling strategy

Samples of input batches and their resultant output batches were collected. Samples were analysed for the following substances:

- Volatile substances,
- Semi-volatile substances,
- Non-volatile substances,
- Inorganic substances,
- Primary aromatic amines.

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.

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

The analytical procedures and method used for the analysis of the samples as well as their limits of detection and quantification are summarised in Table 2.

Table 1. Applied analytical procedures and methods including their limits of detection and quantification.

	Analytical method	Sample Preparation	LOD	LOQ
Untargeted screening of volatile substances	HS-SPME-GC-MS 3min@80°C [a]			
Untargeted screening of semi-volatile substances	HS-SPME-GC-MS 3min@80°C [a]	Dissolution with HFIP		
untargeted screening of non-volatile substances	UPLC-MS-QTOF pos + neg mode [c]	Dissolution with HFIP		
Targeted analysis of primary aromatic amines	UPLC-MS-MS[d]	Extraction with 3% acetic acid		See table
Targeted analysis of inorganic substances (Annex II of EU 10/2011)	ICP-MS [e] XRF (Sb only)	Pressure digestion		See table

GC: Gas chromatography; MS: Mass spectroscopy; QToF: Quadrupole- time-of-flight; FID: Flame Ionisation Detector; LC: liquid chromatography; UPLC: *ultrahigh performance LC;* ICP: Inductively Coupled Plasma

Analysis of organic substances is done through a non-targeted screening of volatile, semi-volatile and non-volatile substances with different methods (Table 2).

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 a few ppbs (1-50 depends on the compound) can be detected for most of the volatile substances. The adsorption conditions for SPME of 3 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 mass spectra were compared with a mass spectra library (NIST or WILEY).

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 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 in paragraph 4.

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).

Inorganic substances were analysed using ICP-MS which is a sensitive elemental analysis technique that detects trace metals and non-metals at ultralow concentrations.

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

i) Analysis and explanation of any discrepancies observed between contaminant levels expected and decontamination efficiency.

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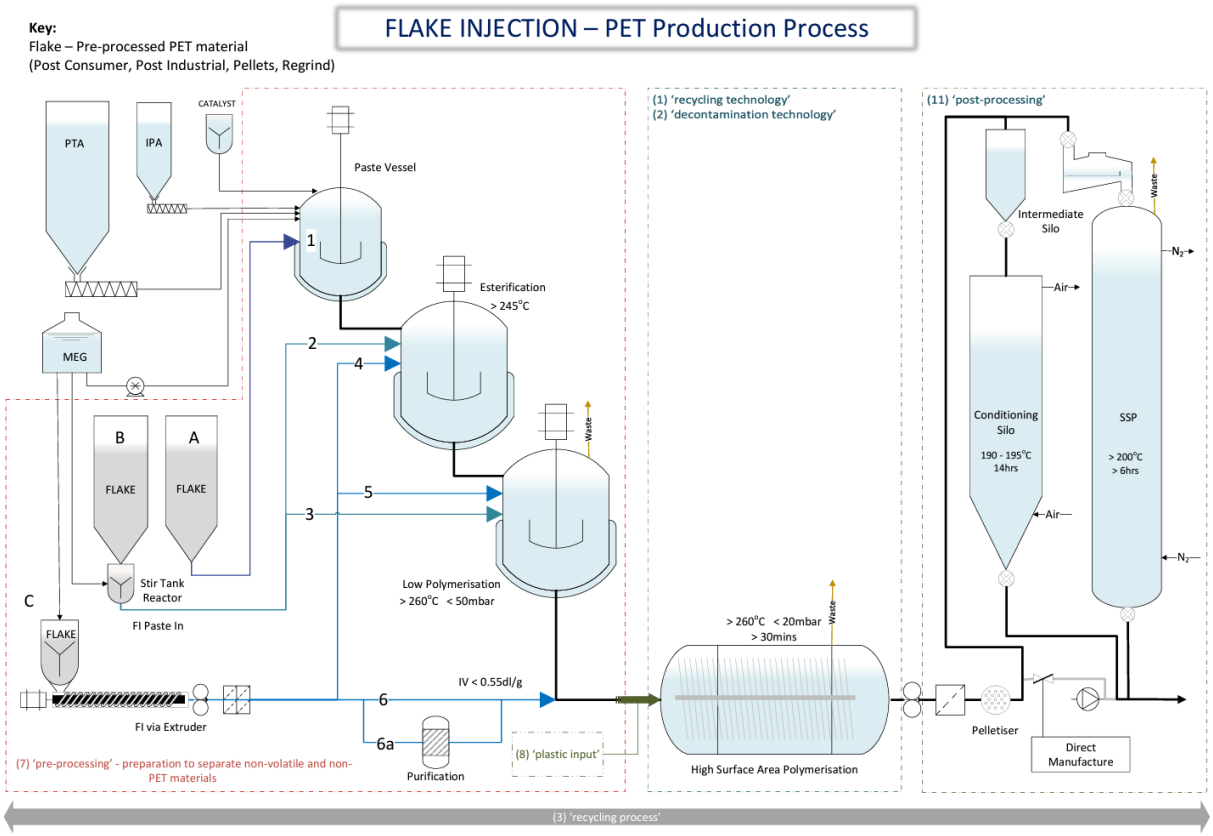
j) a discussion of the differences with previous reports published in accordance with this paragraph, if any.

To be completed on receipt of the delayed analysis report

Appendix I –.


Key:
Flake – Pre-processed PET material
(Post Consumer, Post Industrial, Pellets, Regrind)
FLAKE INJECTION – PET Production Process
(1) 'recycling technology'
(2) 'decontamination technology'
(11) 'post-processing'
PTA
IPA
CATALYST
Paste Vessel
MEG
Esterification
> 245°C
B
FLAKE
A
FLAKE
Stir Tank
Reactor
FI Paste In
C
FLAKE
FI via Extruder
Low Polymerisation
> 260°C < 50mbar
Waste
IV < 0.55dl/g
Purification
(8) 'plastic input'
(7) 'pre-processing' - preparation to separate non-volatile and non-PET materials
> 260°C < 20mbar
> 30mins
High Surface Area Polymerisation
Intermediate
Silo
Conditioning
Silo
190 - 195°C
14hrs
SSP
> 200°C
> 6hrs
Air
N2
Pelletiser
Direct
Manufacture
(3) 'recycling process'

Glossary of Terms

Cmod	Modelled concentration
DEG	diethylene glycol
EG	ethylene glycol
GC	gas chromatography
HPLC	high performance liquid chromatography
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy
IPA	isophthalic acid
MHET	mono(2-hydroxyethyl)terephthalate
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
XRF	X-ray fluorescence spectroscopy

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