Third Report on the Novel Technology

“INEOS STYROLUTION Twin Screw Degassing Extrusion”

According to Article 13(4) of the Commission Regulation (EU) 2022/1616

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1. Description of the novel technology

The INEOS STYROLUTION super-clean recycling process for polystyrene (PS), which began operating before the entry into force of Commission Regulation (EU) No. 2022/1616, consists of the following main process steps:

- Step 1: Oversorting of available PS Bales (waste specification DSD 331; > 94% article content PS) Grinding of collected post-consumer PS containers into flakes followed by an intensive wash process and drying (remark: step 1 is made by the flake suppliers)

- Step 2: Extrusion of the washed flakes using a twin screw extruder with vacuum degassing.

INEOS STYROLUTION is buying washed flake derived from post-consumer PS trays and containers from green dot systems and curbside collections in Europe. The flake suppliers use state of the art oversorting and washing processes. Oversorting ensures that non-PS and non-food articles are sorted out of the remaining recycled material to ensure that the feedstream consists of > 95% PS articles having food contact origin. After oversorting, the PS feedstock is then cut into flake.

The PS flakes are then washed using a caustic hot washing process that contains surfactants to assist in the removal of undesirable residues

The hot washing process is followed by rinsing with water and surface drying of the PS flakes. The flakes are sorted again with NIR technology in order to ensure that foreign materials from labels and closures that were formerly attached to the PS container are now taken out.

The washed flakes are then extruded using a twin screw extruder with vacuum degassing. Potential contaminants are removed during this melt degassing. The decontaminated melt is subsequently pelletized. The twin screw extruder design allows for control over the following critical decontamination parameters:

- Temperature
- Vacuum
- Residence time

The key components of the super-clean recycling process are shown in Figure 1.

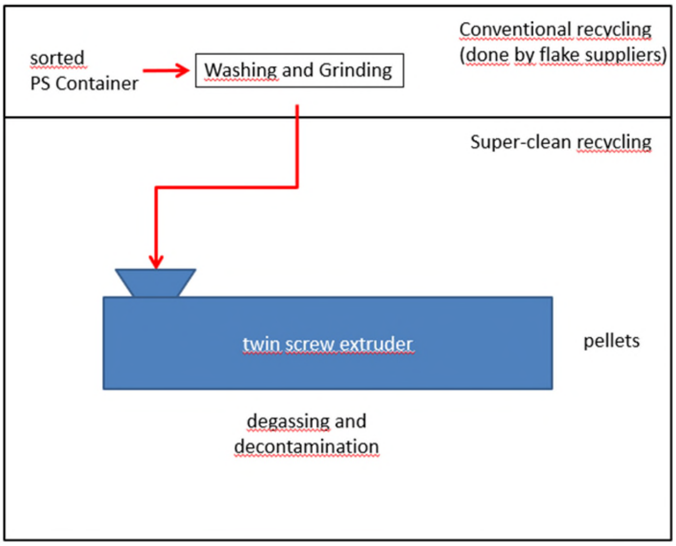

Fig. 1 Schematic of the investigated super-clean recycling process

The final pellets are intended for use in manufacturing new food contact articles with a recyclate content of up to 100%, including containers for dairy products, trays for packaging food, and beverage cups.

The novel technology developer has assessed the polystyrene (PS) recycling process in a fashion similar to that utilized in the EFSA Scientific Opinion on the *criteria to be used for safety evaluation of a mechanical recycling process to produce recycled PET intended to be used for manufacture of materials and articles in contact with food* (EFSA, 2011). As discussed in the initial novel technology development report, the recycling process has been evaluated by applying the cleaning efficiency of the recycling process, obtained from a challenge test with surrogate contaminants at highly exaggerated levels, to a conservative reference contamination level for misuse contaminants in PS to calculate the residual concentration of contaminants in recycled PS (Cres). The resulting residual concentration for each contaminant is then compared to an “allowable” concentration of each contaminant in the PS that is derived using diffusion modeling (Cmod) and by considering the conditions of use of the articles manufactured with recycled PS. Specifically, this Cmod is calculated using generally recognized conservative migration models and it corresponds to a migration which cannot give rise to a dietary exposure exceeding the threshold below which the risk to human health would be negligible. Therefore, when Cres is not higher than Cmod, it is considered that the process is able to produce an output which is not likely to be of safety concern for the defined conditions of use. Consistent with Commission Regulation (EU) 2022/1616, the notifier monitors substances in the input and output to ensure that contaminants that may be retained in the recycled output material are not expected to migrate to contacted food at levels that would present health or safety concerns.

2. Compliance with Article 3 of Regulation (EC) No 1935/2004

INEOS STYROLUTION prepared an initial report on its novel polystyrene recycling technology in accordance with Article 10 of Commission Regulation (EU) No. 2022/1616. That report included extensive reasoning, scientific evidence, and studies that demonstrated that the recycled polystyrene produced under the INEOS STYROLUTION process complies with Article 3 of Regulation (EC) No. 1935/2004. The report summarized the results of a challenge test that was used to establish the decontamination efficiency of the process for commonly utilized surrogate contaminants. The initial report also included an industry study that evaluated the residual contaminant concentration in the recycled input based on samples of post-consumer PS flake samples obtained throughout Europe. This study demonstrated that a conservative estimate of contaminants in the input stream is unlikely to exceed 1 mg/kg. The initial report also included a migration estimate for contaminants based on various use scenarios for the applications under which the recycled PS will be marketed.

The initial report demonstrated that the cleaning efficiency for the INEOS STYROLUTION polystyrene recycling process was sufficient to ensure that an exposure of 0.0025 µg contaminant/kg bw/day would not be exceeded. The 0.0025 µg contaminant/kg bw/day exposure threshold value is the level that EFSA has determined is safe even for chemicals with structural alerts raising concern for potential genotoxicity. Generally, this threshold value is low enough to address all toxicological concerns. Thus, the initial report demonstrated that any unknown contaminant potentially present in the recycled polystyrene would not result in risk of harm to consumers consuming food packaged in the modelled applications.

INEOS STYROLUTION published its first semiannual report in October 2023. The first report included sampling data demonstrating that although some impurities in the recycled PS output exceeded the 1 mg/kg assumed contaminant level, many of the substances are also present in virgin polystyrene. (Additionally, several of the compounds were suspected to be artifacts of the analytical method used to analyze the samples, and likely were not true contaminants in the recycled plastic output.) Based on the toxicity profiles of the detected substances and the expected migration of the detected substances to food, the first report demonstrated that the potential presence of the contaminants in the recycled PS did not present any health or safety concern, and the recycled PS may be considered compliant with Article 3 of Regulation (EC) No. 1935/2004.

As discussed in the last report (April 2024), INEOS/Styrolution refined the analytical methodology used for the evaluation of contaminant chemicals in the recycled PS input and output. This revised method, which is described in Section 8 below, will be used in analyzing the recycled input and output going forward.

2.1 Characterisation of contaminant levels in the plastic input and the recycled plastics

As described in the initial report, critical contaminants in post-consumer polymers might be chemicals from possible misuse of packaging containers, contaminants from containers used in non-food applications such as non-authorized additives, as well as degradation products generated during recycling (Barthélémy et al. 2014).

Statistical data assessing the frequency of misuse of food-contact PS containers for the storage of household chemicals are not currently available in the scientific literature. Accordingly, a so-called "misuse study" had been conducted by industry (Fraunhofer IVV report no. PA-2017-21 and PA-1770-23 – conducted on behalf of Styrenics Circular Solutions (SCS) and property of SCS). In this study, 49 washed post-consumer PS flake samples obtained throughout Europe were analyzed to assess whether chemicals originating from the misuse of PS containers used to store solvents, household, or garden chemicals were present in the recycled polystyrene samples. Each sample (containing approximately 35.1 flakes/gram/sample) was analyzed 6 times. Overall, 10,310 individual post-consumer PS flakes were analysed. No substances that were associated with the misuse of a container (e.g., solvent, household chemical or garden chemical) were detected in the flake samples. This testing supports that the incidence of misuse is expected to be less than 0.0097% (< 1/10,310), and that recycled polystyrene containers are not likely to be used by consumers to store hazardous substances after the first food contact-use.

For comparison, the incidence of misuse found for post-consumer PET bottles was 0.03% to 0.04%. Toluene (at a concentration of 6750 mg/kg in the contaminated PET flake) has been identified as an example of the sort of substances that are most likely filled into these misused PET bottles. In terms of consumer behavior, PET bottles are much more suitable for storage of liquids, because the bottles can be re-sealed with a closure. PS cups or trays cannot be re-sealed, and are therefore not suitable for storage of liquid chemicals. In addition, solvents such as toluene dissolve PS and destroy the container.

Therefore, the incidence for misuse of PS cups or trays for storage of hazardous chemicals is most likely much lower than that for PET, which was confirmed by the "misuse" study discussed above.

Because there was no evidence of misuse of the 10,310 individual recycled PS flakes analyzed, the input concentration chemicals attributed to the misuse of the PS cannot be directly determined from the study. However, using the incidence of contamination from this study, and the maximum sorption of surrogate contaminants from the surrogate challenge study, we can estimate the potential concentration of contaminants in a recycled PS feedstream resulting from the presence of misused PS containers therein. That is, the maximum sorption of any of the surrogate contaminants (toluene, chlorobenzene, methyl salicylate, phenyl cyclohexane, benzophenone, and methyl stearate) into PS observed after the intentional contamination of PS flake was observed for methyl salicylate, at 1,411 mg/kg. If we multiply this maximum sorption level by the incidence of contamination approximated in the misuse study (0.0097%), we estimate that the contaminant concentration of recycled PS feedstreams is approximately 0.14 mg/kg (1,411 mg/kg x 0.0097% = 0.14 mg/kg). Therefore, it would be conservative to assume a worst-case input contamination of the input flake of no more than 0.5 mg/kg, or half of that considered in the initial report.

Other contamination, such as microbiological or viral contamination, can be excluded because of the high temperatures used to process the polymer (Barthélémy et al. 2014).

3. List of substances in plastic input and recycled plastic output

Tables 1 and 2 below are lists of substances found in the plastic input (Table 1) and in the recycled polystyrene output (Table 2), sorted in descending order of the concentration in the sample. These data were generated using the modified analytical method, first discussed in the April 2024 report, to minimize the oxidation and/or degradation of certain impurities during analysis.

The tentative identity of each substance was determined by matching the fragmentation pattern for each substance with a library of known compounds. The concentration of each substance was semi-quantified using calibration data for a limonene external standard. Substances identified with an asterisk (“*”) following the chemical name were quantified using external reference calibration data for that substance (rather than using the limonene standard).

Substances highlighted in blue were also identified in virgin polystyrene samples evaluated using the same analytical methods. Because these substances are present in virgin samples, they are not considered contaminants and are not further discussed in this report.

Table 1. Substances identified in source (INPUT)

Modified Analytical Method (nitrogen)

Chemical Name	CASRN	Ave. Conc. (ppm)
styrene*	100-42-5	101.33
ethylbenzene*	100-41-4	14.41
limonene*	138-86-3	6.53
ethoxy ethene	109-92-2	6.16
1-propene	115-07-1	5.86
acetophenone*	98-86-2	4.73
hexanal	66-25-1	3.23
(1-methylethyl)-benzene	98-82-8	3.16
2-propenylbenzene	300-57-2	3.03
styrene dimer		2.84
isobutene	115-11-7	2.79
1-octene-3-one	4312-99-6	2.48
1,3-dimethylbenzene	108-38-3	2.32
heptane	142-82-5	2.30
n-propylbenzene	103-65-1	2.17
decane	124-18-5	2.05
1-ethyl-2-methyl-benzene	611-14-3	1.99
toluene	108-88-3	1.92
2-heptanone	110-43-0	1.68
cyclohexane	110-82-7	1.62
n-hexane	110-54-3	1.56
eucalyptol	470-82-6	1.49
1,3-propandiol	504-63-2	1.42
tert-butylbenzene	98-06-6	1.38
2-nonanone	821-55-6	1.36
linalool	78-70-6	1.30
2,2,4,6,6-pentamethylheptane	13475-82-6	1.27
2-butanone	78-93-3	1.25
dodecane	112-40-3	1.23
tetradecane	629-59-4	1.22
dimethylsulfide	624-92-0	1.17
1,3,5-trimethylbenzene	108-67-8	1.15
alpha-methylstyrene	98-83-9	1.11
3-heptanone	106-35-4	1.07
nonane	111-84-2	1.04

Table 2. Substances identified in rPS (OUTPUT)

Modified Analytical Method (nitrogen)

Chemical Name	CASRN	Ave. Conc. (ppm)
styrene*	100-42-5	75.11
ethylbenzene*	100-41-4	9.91
ethoxy ethene	109-92-2	4.45
limonene*	138-86-3	4.66
acetophenone*	98-86-2	4.27
2-propenylbenzene	300-57-2	3.22
1-propene	115-07-1	3.21
styrene dimer		2.81
(1-methylethyl)-benzene	98-82-8	2.31
hexanal	66-25-1	2.19
isobutene	115-11-7	2.02
1-octene-3-one	4312-99-6	1.80
heptane	142-82-5	1.68
1-ethyl-2-methyl-benzene	611-14-3	1.57
1,3-dimethylbenzene	108-38-3	1.54
n-propylbenzene	103-65-1	1.54
toluene	108-88-3	1.41
eucalyptol	470-82-6	1.36
decane	124-18-5	1.26
2-heptanone	110-43-0	1.20
tert-butylbenzene	98-06-6	1.17
tetradecane	629-59-4	1.16
1,3-propandiol	504-63-2	1.07
2-nonanone	821-55-6	0.99
linalool	78-70-6	0.98

4. List of contaminating materials regularly present in the plastic input

As discussed in INEOS/STYROLUTIONS' previous reports, the waste stream from which source material is obtained consists of PS trays and containers from green dot systems and curbside collection systems in Europe. The waste may originally contain non-food articles such as:

- Video cassettes
- Flower pots
- Hangers
- CD covers
- Clip closures (*e.g.*, freezer bags including metal wire and PS)

These materials are sorted out of the waste stream such that the input material consists predominantly of PS used in contact with food. The specifications for the input to the decontamination process are as follows:

Parameter	**Value**
Moisture	<1%
PS flakes with glue content	<0.5%
Polyolefins content	<1%
Polyamide content	<0.5%
Metals content	<0.1%
Wood, paper, cellulose	<0.5%

5. Analysis of the most likely origin of the identified contaminants

As noted above, testing has demonstrated that many of the substances found in the recycled PS are also found in samples of virgin PS. These substances are generally found at similar concentrations in both virgin and recycled samples.

Several other substances (e.g., limonene, eucalyptol) are flavoring substances that may be associated with foods that were stored in the plastic packaging that was in the source material. Other substances could be present in the input and output material from their use as components of the packaging (e.g., labels, printing inks, adhesives, etc.) that was recycled. The levels of these substances are relatively low and are comparable to the levels in other packaging materials.

6. Measurement or estimation of the migration levels to food of contaminants present

The migration of the contaminants present in the output (recycled polystyrene) were determined using diffusion modeling following the same approach for the various applications covered by the first three reports submitted on this novel technology.

That is, the Piringer-based (i.e., A_P-based) diffusion model was used to estimate migration of the various substances. Because the A_p model exaggerates migration from polystyrene, the migration values were adjusted using the temperature correction factors established by Welle (2023).[1] As noted in the initial report, the correction factors for polystyrene depend on temperature (the extent of the overprediction of the A_P-based diffusion model increases as temperature decreases), but are also influenced by both

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[1] Welle, F. Recycling of Post-Consumer Polystyrene Packaging Waste into New Food Packaging Applications—Part 1: Direct Food Contact. Recycling 2023, 8, 26. https://doi.org/10.3390/recycling8010026.

molecular weight and polarity. We have used the factor developed for toluene at the specific temperatures of interest (i.e., 4.77 for 60°C, 11.8 for 40°C, 22.9 for room temperature conditions, and 20.8 for refrigerated conditions) in determining the estimated migration for each of the contaminants in the various use scenarios for the recycled PS.[2] That is, applications considered here include packaging for yogurt and similar foods (following three different packing scenarios), meat and cheese tray applications, fish boxes, fruit and vegetable tray applications, hot and cold cup applications. The migration values for each contaminant and under each use scenario are reported in Table 3.

Table 3: Calculated migration for substances under various recycled PS use scenarios

Substance Name	CASRN	Conc. in rPS (ppm)	Predicted migration (µg/kg-food)					
			Yogurt[3]	Meat/ Cheese Tray	Fish Boxes	Fruit/ Vegetable Tray[4]	Cold Cups	Hot Cups
limonene	138-86-3	4.66	0.20	0.05	0.03	0.01	0.03	0.38
1-octene-3-one	4312-99-6	1.80	0.10	0.03	0.01	0.01	0.01	0.20
heptane	142-82-5	1.68	0.12	0.03	0.02	0.01	0.02	0.23
1-ethyl-2-methyl-benzene	611-14-3	1.57	0.10	0.02	0.01	0.01	0.01	0.18
eucalyptol	470-82-6	1.36	0.06	0.02	0.01	0.00	0.01	0.12
decane	124-18-5	1.26	0.07	0.02	0.01	0.00	0.01	0.12
2-heptanone	110-43-0	1.20	0.08	0.02	0.01	0.01	0.01	0.14
tert-butylbenzene	98-06-6	1.17	0.06	0.02	0.01	0.00	0.01	0.12
tetradecane	629-59-4	1.16	0.04	0.01	0.01	0.00	0.01	0.08
1,3-propandiol	504-63-2	1.07	0.09	0.02	0.01	0.01	0.01	0.18
2-nonanone	821-55-6	0.99	0.05	0.01	0.01	0.00	0.01	0.10
linalool	78-70-6	0.98	0.05	0.01	0.01	0.00	0.01	0.09

For the substances identified in Table 3 with a migration that results in a dietary exposure greater than 0.0025 µg/kg bw/day (the EFSA-established threshold value for genotoxic compounds),[5] we have concluded that the weight of the evidence indicates that these compounds are not genotoxic. Therefore, a dietary exposure of 1.5 µg/kg bw/day, which is the human exposure threshold value that has been used by EFSA for Cramer Class III compounds, has been used to assess the safety. Using the same exposure scenarios described in the initial report, including the consumption patterns and assumed body weights described in Table 4 (below), we have calculated the migration levels in foods that will result in exposures to the listed substances of no more than 1.5 µg/kg bw/day under each of the use scenarios.

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[2] As noted above, substances that have been identified in virgin polystyrene resin (and at levels that are similar to that found in the virgin samples) have been excluded from this analysis.

[3] The hot-filled yogurt packaging condition (60°C for 1 hour, followed by 40 days at 6°C) was found to be the worst case migration condition, and the migration estimate for only that packaging condition is included in this table.

[4] Consistent with the initial report, migration to raw, uncut/unpeeled fruit and vegetables was divided by a 10-fold correction factor as an estimate to this type of food. Prior EFSA opinions noted that the use of trays to transport, store, and display whole fruits and vegetables at room temperature or below involved conditions under which migration was unlikely to occur, noting the solid-solid contact and small surface of contact.

[5] No health or safety concerns are presented from exposures to potentially mutagenic or genotoxic substances at dietary exposures below the genotoxic threshold. Therefore, comprehensive toxicity reviews of substances that may be present in the rPS where diffusion modeling indicates extremely low migration (i.e., that results in a dietary exposure below 0.0025 µg/kg bw/day) were not conducted.

Table 4: Intended Uses and Target Migration to Ensure Exposure < 1.5 µg/kg bw/day

Application	rPS content	Representative Time / temperature scenarios	Food Consumption	Body weight	Daily consumption	Acceptable migration in food[6]
Yogurt and similar foods	100%	1 hr @ 60°C, + 40 days @ 6°C	12.3 g/kg bw/day	12 kg (toddler)	147.6 g	122 µg/kg
		8 hrs @ 40°C + 40 days @ 6°C				
		40 days @ 6°C				
Meat, poultry, fish, and cheese tray	100%	30 days @ 6°C	50 g/kg bw/day	12 kg (toddler)	600 g	30 µg/kg
Fish boxes	100%	10 days @ 5°C	50 g/kg bw/day	12 kg (toddler)	600 g	30 µg/kg
Fruit and vegetable tray	100%	30 days @ 25°C	50 g/kg bw/day	12 kg (toddler)	600 g	30 µg/kg
Cold cups	100%	1 day @ 25°C	80 g/kg bw/day	12 kg (toddler)	960 g	18.8 µg/kg
Hot cups	100%	2 hrs @ 70°C	20 g/kg bw/day	60 kg (adult)	1200 g	75 µg/kg

As demonstrated in Tables 3 and 4, the estimated migration of the contaminants in the recycled PS processed with the INEOS STYROLUTION novel technology is well below the acceptable migration level noted above (and in most cases less than 1/100th of the level), and clearly these contaminants in the recycled PS do not present any health or safety concern.

Conclusions

For all of the use scenarios described above, the estimated migration of substances in the recycled polystyrene results in a dietary exposure below the relevant toxicity threshold for each of the substances. Thus, substances that may possibly be present in the recycled polystyrene will not result in risk of harm to consumers consuming food out of the modelled containers.

7. Description of the applied sampling strategy

The technology developer operates a single recycling facility employing the novel technology. Consistent with Article 13(1) of Commission Regulation (EU) No. 2022/1616, samples from each batch of input flake from the source material and the corresponding batch of the decontaminated plastic output are collected. Each lot size is between 10 and 15 metric tons. To date, nineteen production batches have been processed using this novel technology (seven of which were produced since the last report) and each of these batches were sampled and analyzed using the described analytical methods. Replicate samples of each batch were analyzed.

8. Description of the analytical procedures and methods used

Although the analysis methodology used for the identification and quantification of potential contaminants and impurities in the recycled PS is significantly similar to the methodology that was

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[6] Example calculation:

$\langle M\rangle_{\text{Target}}$ = 1.5 µg-contaminant/kg bw/day ÷ 0.0123 kg-yogurt/kg bw/day = 122 µg/kg.
$\langle M\rangle_{\text{Target}}$ = 0.0025 µg-contaminant/kg bw/day ÷ 0.0123 kg-yogurt/kg bw/day = 0.20 µg/kg.

described in the previous semi-annual reports, one important sampling modification has been introduced for certain of the samples evaluated to further understand the source of potential contaminants in the PS feedstream. This minor, but important, difference is the sparging of the headspace of GC vials with nitrogen prior to analysis for the purposes of minimizing any oxygen-induced degradation of the samples during the analysis. The "nitrogen method" which was first introduced in the previous report, will be utilized for the assessment of future samples.

Nitrogen Method

Samples of the input and output material are screened for volatile substances using the accredited Fraunhofer IVV Method 1.334:2021-11. For each test, approximately 1.0 g of sample material is weighed, placed in a headspace vial and sparged with nitrogen to remove excess oxygen, and analyzed by headspace GC/FID. Quantification of benzene, ethylbenzene, styrene, limonene and acetophenone was achieved by external calibration.

Identification of other substances in the input and output material was conducted using mass spectrometry. Specifically, a Perkin Elmer Clarus GC-MS-System with electrospray ionization (EI), in full scan mode with mass range m/z 35-300 was used for the analysis. The identification of the substances found was performed by comparison with the NIST spectra library (NIST/EPA/NIH Mass Spectral Library 2017). Confirmation of the suggested spectra by analysis of a respective standard was not performed, so these compounds are considered tentatively identified. Other than benzene, ethylbenzene, styrene, limonene and acetophenone, which were quantified based on the external calibration, quantification of the other substances was performed using the limonene external standard.

The analytical methodology was useful in identifying low molecular weight substances (i.e., less than 300 Daltons). Higher molecular weight substances would not be expected to migrate at any significant level from recycled PS because it is a relatively low diffusive polymer.

9. Analysis and explanation of discrepancies

No discrepancies have been observed between the contaminant levels expected in the input and output of the installation and its decontamination efficiency. The data above supports a finding that the decontamination process adequately removes contaminants from the waste stream.

10. Discussion of the differences with previous reports

As discussed in Section 8, the analytical methodology now exclusively uses a nitrogen sparge of the headspace vial to remove excess oxygen which may interfere with the identification and quantification of the analytes. Future reports will employ the nitrogen method.

11. References

Welle, F. Recycling of Post-Consumer Polystyrene Packaging Waste into New Food Packaging Applications—Part 1: Direct Food Contact. Recycling 2023, 8, 26. https://doi.org/10.3390/recycling8010026.