First 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 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 by using a twin screw extruder with vacuum degassing.

INEOS STYROLUTION buys 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 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 by use of the 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.

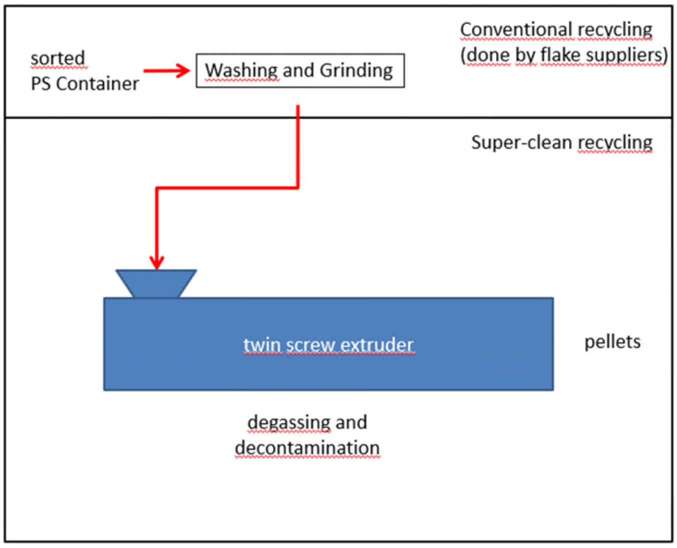

Fig. 1 Schematic of the investigated super-clean recycling process

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

The novel technology developer has assessed the mechanically recycled polystyrene (PS) 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). In that regard, the recycling process has been evaluated by measuring 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). 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 unknown contaminants in the input stream is unlikely to exceed 1 mg/kg. The initial report also included a migration estimate for unknown contaminants based on various use scenarios for the applications under which the rPS 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.

Although, as noted below, the concentration of a few 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 are suspected to be artifacts of the analytical method used to analyze the samples, and likely are not true contaminants in the recycled plastic output.) With the exception of a low-level potential contaminant, genotoxicity of the compounds has been ruled out, and the migration estimates for the contaminants discussed below demonstrate that the dietary exposure to these substances would be no more than 1/100th threshold of toxicological concern (TTC) associated with Cramer Class III compounds (i.e., 1.5 µg/kg bw/day). Thus, the potential presence of these contaminants in the rPS do not present any health or safety concern, and the rPS may be considered compliant with Article 3 of Regulation (EC) No. 1935/2004. The data that has been generated since the development of the initial report are consistent with the conclusions reached in the initial report.

3. List of substances in plastic input and recycled 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 by descending order of the concentration in the sample. As discussed in Section 8 below, 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).[1]

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

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[1] The quantification of styrene, acetophenone, limonene, and ethylbenzene reported in Table 1 was based on testing of a single sample. The quantification of the other two input flake samples was not assessed with the external calibration standard.

Table 1: Substances identified in source flake (INPUT)

Substance Name	CASRN	Ave. Conc. (ppm)
styrene*	100-42-5	138.0
ethoxy ethene	109-92-2	65.432
isobutylene	115-11-7	34.447
t-butanol	75-65-0	22.025
acetophenone*	98-86-2	20.1
1-octen-3-one	4312-99-6	13.592
limonene*	138-86-3	12.5
ethylbenzene*	100-41-4	10.5
hexanal	66-25-1	8.522
2-propenyl-benzene	300-57-2	8.424
2,6-dimethyl-4-heptanone	108-38-8	7.743
cumene	98-82-8	7.511
dimethyl disulfide	624-92-0	7.357
octanal	124-13-0	7.343
3-methylbutanal	590-86-3	6.577
2,2,4,6,6-pentamethylheptane	13475-82-6	6.315
alpha-pinene	80-56-8	5.715
cyclohexane	110-82-7	5.219
2,4-dimethylfuran	3710-43-8	5.13
2-nonanone	821-55-6	5.087
1-heptene	592-76-7	4.923
styrene dimer		4.388
toluene	108-88-3	4.052
2-heptanone	110-43-0	4.042
nonanal	124-19-6	3.616
3-heptanone	106-35-4	3.614
n-propyl-benzene	103-65-1	3.508
unknown (RT = ~1.65 min.)	--	3.464
1-pentanol	71-41-0	3.38

Table 2: Substances identified in rPS (OUTPUT)

Substance Name	CASRN	Ave. Conc. (ppm)
styrene*	100-42-5	145.3
ethoxy ethene	109-92-2	43.886
isobutylene	115-11-7	27.246
acetophenone*	98-86-2	13.300
t-butanol	75-65-0	12.834
1-octen-3-one	4312-99-6	10.205
ethylbenzene*	100-41-4	9.73
2-propenyl-benzene	300-57-2	9.043
cumene	98-82-8	7.126
limonene*	138-86-3	5.77
alpha-pinene	80-56-8	5.719
4-vinylcyclohexene	100-40-3	4.662
toluene	108-88-3	4.393
octanal	124-13-0	4.218
1-heptene	592-76-7	3.974
heptanal	111-71-7	3.921
styrene dimer	--	3.857
(1-methylpropyl)-benzene	135-98-8	3.847
dimethyl disulfide	624-92-0	3.768
2,4-dimethylfuran	3710-43-8	3.768
1-hydroxy-2-propanone	116-09-6	3.705
nonanal	124-19-6	3.036
1,3,5-trimethylbenzene	108-67-8	2.825
n-propyl-benzene	103-65-1	2.816
hexanal	66-25-1	2.741
2-methyl-3-pentanone	565-69-5	2.74
meta-xylene	108-38-3	2.510
2-ethyl-1-hexanol	104-76-7	2.430
2-heptanone	110-43-0	2.391

4. List of contaminating materials regularly present in plastic input

As discussed in Section 1 above, the waste stream consists of PS trays and containers from green dot systems and curbside collection systems in Europe. The waste may originally contain non-food articles, which are sorted out of the waste stream such that the input material entering the decontamination phase consists predominantly of PS used in contact with food. The input to the decontamination process complies with the following specifications.

Parameter	Value
Moisture	<1%
PS flakes with printing content	<0.5%
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 demostrated that many of the subtances 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, alpha pinene, various aldehydes) are flavoring substances that may be associated with foods that were previously 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.

Several substances (e.g., oxygenated compounds such as aldehydes and ketones that were found in the input and output samples) are suspected to be potential degradation products of the polymer. These substances may not be present as impurities in the recycled PS, but rather could be generated during the analysis of the polymer at high temperatures. Preliminary analysis of the same samples at lower temperatures suggest that these substances may not actually be present. The developer will continue to monitor these substances in analysis of future rPS samples produced with this novel technology to determine whether a change in the analytical method would be appropriate.

6. Estimate of migration levels of contaminants to food

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 initial report submitted on this novel technology.

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).[2] 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 molecular weight and polarity. The factor developed for toluene at the specific temperatures of interest (i.e., 4.77 for 60°C, 11.8 for 40°C, and 22.9 for room temperature conditions, and 20.8 for refrigerated conditions) was used in determining the estimated migration for each contaminant in the various use scenarios for the rPS covered by the initial report.[3] That is, applications considered here include packaging for yogurt and similar foods (following three different packing scenarios), meat and cheese tray applications, fruit and vegetable tray applications, hot and cold cup applications. (The hot cup application includes the use of the rPS at levels up to 50%.) The migration values for each contaminant and under each use scenario are reported in Table 3.

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[2] 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.

[3] 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.

Table 3: Calculated migration for contaminants under various rPS use scenarios

			Predicted migration (µg/kg-food)				
Substance Name	**CASRN**	**Conc. in rPS (ppm)**	**Yogurt[4]**	**Meat/ Cheese Tray**	**Fruit/ Vegetable Tray[5]**	**Cold Cups**	**Hot Cups (50% rPS)[6]**
acetophenone	98-86-2	13.300	0.8	0.18	0.06	0.11	0.76
1-octen-3-one	4312-99-6	10.205	0.59	0.15	0.04	0.081	0.56
limonene	138-86-3	5.77	0.31	0.08	0.02	0.04	0.29
alpha-pinene	80-56-8	5.719	0.31	0.08	0.02	0.04	0.29
4-vinylcyclohexene	100-40-3	4.662	0.31	0.08	0.02	0.04	0.29
toluene	108-88-3	4.393	0.33	0.08	0.03	0.05	0.32
octanal	124-13-0	4.218	0.24	0.06	0.02	0.03	0.23
heptanal	111-71-7	3.921	0.25	0.06	0.02	0.03	0.24
(1-methylpropyl)-benzene	135-98-8	3.847	0.19	0.05	0.01	0.03	0.18
dimethyl disulfide	624-92-0	3.768	0.28	0.07	0.02	0.04	0.27
1-hydroxy-2-propanone	116-09-6	3.705	0.33	0.08	0.025	0.045	0.31
nonanal	124-19-6	3.036	0.16	0.04	0.01	0.02	0.15
1,3,5-trimethylbenzene	108-67-8	2.825	0.17	0.04	0.013	0.024	0.16
hexanal	66-25-1	2.741	0.20	0.05	0.015	0.027	0.18
2-methyl-3-pentanone	565-69-5	2.74	0.20	0.05	0.015	0.027	0.18
meta-xylene	108-38-3	2.510	0.17	0.04	0.013	0.023	0.16
2-ethyl-1-hexanol	104-76-7	2.430	0.14	0.03	0.01	0.02	0.13
2-heptanone	110-43-0	2.391	0.15	0.04	0.01	0.02	0.14

For the substances identified in Table 3, genotoxicity has been ruled out, and 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 chemicals, has been used in this evaluation. Following the same exposure scenarios described in the initial report, including the consumption pattern and assumed body weights described in Table 4, we have calculated the migration in foods that will result in an exposure to the listed substances of no more than 1.5 µg/kg bw/day under each of the use scenarios.

One low level contaminant was found in the samples at extremely low concentrations. It is not clear whether this substance is an analytical artifact (as discussed above), generated from degradation of the polymer, or a contaminant from the input flake. Nonetheless, the estimated migration of that substance that may result from the use of the rPS in the applications considered here was calculated assuming, for the sake of conservatism, that this impurity is actually present in the output rPS material. Based on this extremely low migration (<0.15 µg/kg-food), the dietary exposure to that substance was calculated to be far lower than 0.0025 µg/kg bw/day, the EFSA-established threshold

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[4] 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.

[5] 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.

[6] Migration was estimated assuming that the maximum concentration of the rPS in the hot cup application was 50%. All other conditions assumed that the rPS was used at 100% in the considered application.

value for genotoxic compounds. Even if that substance were considered genotoxic, the substance would not present any health or safety concern from its potential low levels in the rPS material.

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[7]
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
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	50%	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 rPS processed with the INEOS STYROLUTION novel technology is no more than 1/100th of the acceptable migration level noted above, and clearly these contaminants in the rPS do not present any health or safety concern.

7. Description of 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. Currently, at least one sample from each production batch has been sampled, and replicates of each sample were analyzed using the method described below.

8. Description of analytical procedures and methods used

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 analyzed by headspace GC/FID. Quantification of 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

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[7] Example calculation:

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

was not performed, so these compounds are considered tentatively identified. Most substances were quantified using the limonene external standard. As noted in the table above, styrene, ethylbenzene, acetophenone, and limonene were quantified based on calibration standards for each of these compounds.

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 rPS because it is a relatively low diffusive polymer.

As noted above, the high temperature sampling conditions used in the analytical method used for the samples reported here may have caused some degradation of the polymer and resulted in generation of byproducts that are not otherwise expected to be found in the output material. As such, some of the substances reported here may be artifacts of the analytical procedures rather than contaminants in the source material. The developer will continue to assess this phenomenon and modify the analytical method as appropriate to avoid generation of byproducts that are not truly in the output samples. Future six-month reports will discuss any changes in the analytical approach that are implemented to address this issue.

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 differences with previous reports

This is the first 6-month report on this technology, and thus, no differences are observed.

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.