# PET Flake Injection

## Novel Technology Development

### Data Monitoring Report

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

10 October 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 | | | | | | |
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## Volatiles (contd)

## Volatiles (contd)

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

| | RT | Mass | Candidate | Formula | Input µg/kg PET | Output µg/kg PET | Cleaning efficiency, % |
|---|---|---|---|---|---|---|---|
| SAMPLE I | | | | | | | |
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## Non Volatiles (contd)

## Inorganic Substances - To be completed on receipt of the delayed analysis report

| | Substance | INPUT mg/Kg of PET | OUTPUT mg/Kg of PET | LOD |
|---|---|---|---|---|
| SAMPLE 1 | Cr | | | 0.06 |
| | Mn | | | 0.04 |
| | Fe | | | 1.37 |
| | Co | | | 0.01 |
| | Ni | | | 0.32 |
| | Zn | | | 6.4 |
| | Ge | | | 0.03 |
| | As | | | 0.02 |
| | Zr | | | 0.02 |
| | Ba | | | 0.15 |
| | Sb | | | 0.03 |
| | Se | | | 3.6 |
| | Pb | | | 0.51 |

## Primary Aromatic Amines - To be completed on receipt of the delayed analysis report

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

## Plastics Additives - To be completed on receipt of the delayed analysis report

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

## 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] | 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.

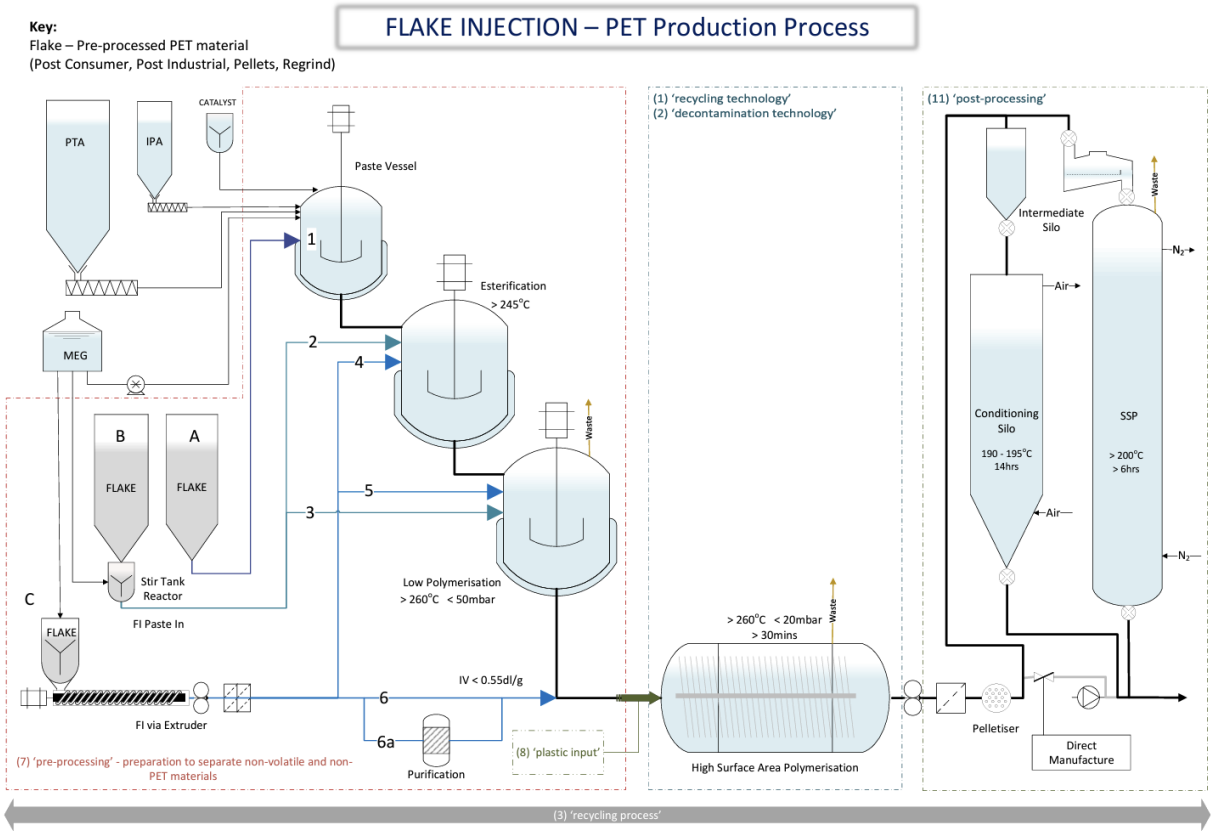
## i) Analysis and explanation of any discrepancies observed between contaminant levels expected and decontamination efficiency.

To be completed on receipt of the delayed analysis report

## 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
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
(1) 'recycling technology'
(2) 'decontamination technology'
> 260°C < 20mbar
> 30mins
High Surface Area Polymerisation
(11) 'post-processing'
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 |

# REFERENCES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