



Executive Summary

This report details the novel technology for post-consumer mechanical HDPE recycling developed by Veolia ES Plastics UK Limited at the Dagenham facility. The process, based on a recycling system developed by WRAP, focuses on recycling HDPE bottles back into food-grade materials, specifically for milk bottle applications. The technology has demonstrated consistent output quality and high decontamination efficiency, with ongoing monitoring to ensure compliance with EC 2022/1616 regulations.

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1. **Background**

a. **Purpose of the report**

The process now owned by Veolia ES Plastics UK Limited for the Dagenham facility is based on a recycling system developed by WRAP (2005) ISBN: 1-84405-225-7.

This was followed by a large-scale trial by Wrap (2007) ISBN: 1-84405-308-3.

“The development of world leading UK recycling technology allows post-consumer milk bottles to be recycled back into food contact milk bottles. Milk bottles with 30% recycled content perform identically as virgin resin bottles, have been extensively tested and have passed all EU, UK and consumer tests and are currently in production within UK dairies. The novel technology represents the first time post-consumer HDPE Milk bottles have been recycled back into Milk Bottles with full food contact status.”The process was submitted to the United States Food and Drug Administration for approval. Subsequently a No Objection Letter 108 was issued (2007).

Based on this the Dagenham facility was built (2009). The process contains the following key steps: Sorting of natural HDPE milk bottles, grinding and washing of the bottles then decontamination. Quality management of the feedstock, combined with improvements in pre-processing and decontamination reduce the risk of incidental contamination.

Since 2010 the Dagenham facility has supplied 10,000 tonnes / year to the UK dairy industry with no incidents and each batch of output material is tested for its suitability to come into contact with food.

The process was also submitted to EFSA (RECYC063) with a final safety assessment still pending on further additional data (2015).

The regulation was updated and the process was deemed a Novel Technology requiring self monitoring data and reporting based on EC 2022/1616 Article 13

The controlled version will be available online and previously superseded versions archived offline.

b. **Description of the Novel Technology**

Polymer type	Short description of the recycling technology	Specification of plastic input	Specification of output	Subject to the authorisation of individual processes	Recycling scheme applies
HDPE	Mechanical recycling	Only HDPE PCW containing maximum 1% of materials and articles that were used in contact with non-food materials or substances	Decontaminated HDPE, final materials and articles intended use for milk bottle applications	Yes	No

This a Novel Technology and there were no deviations from the requirements set out in Articles 6, 7 and 8. No recycling scheme was used. This data is consistent with the [Technical dossier](#) page 21 / 57. Confirmation of input source and traceability through the process has been independently assessed using RecyClass - Certification compliant with EN 15343:2007 Audit Report and Certificate Code: RP261-VEO-07-25-CIR-PC)

Figure 1

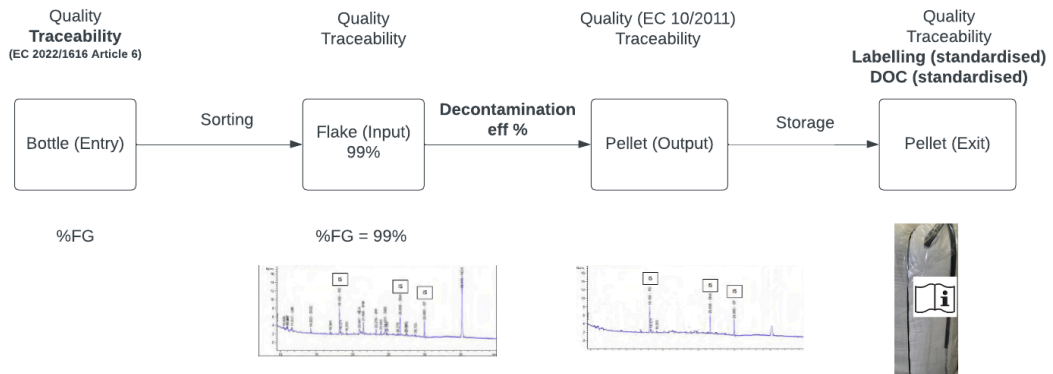
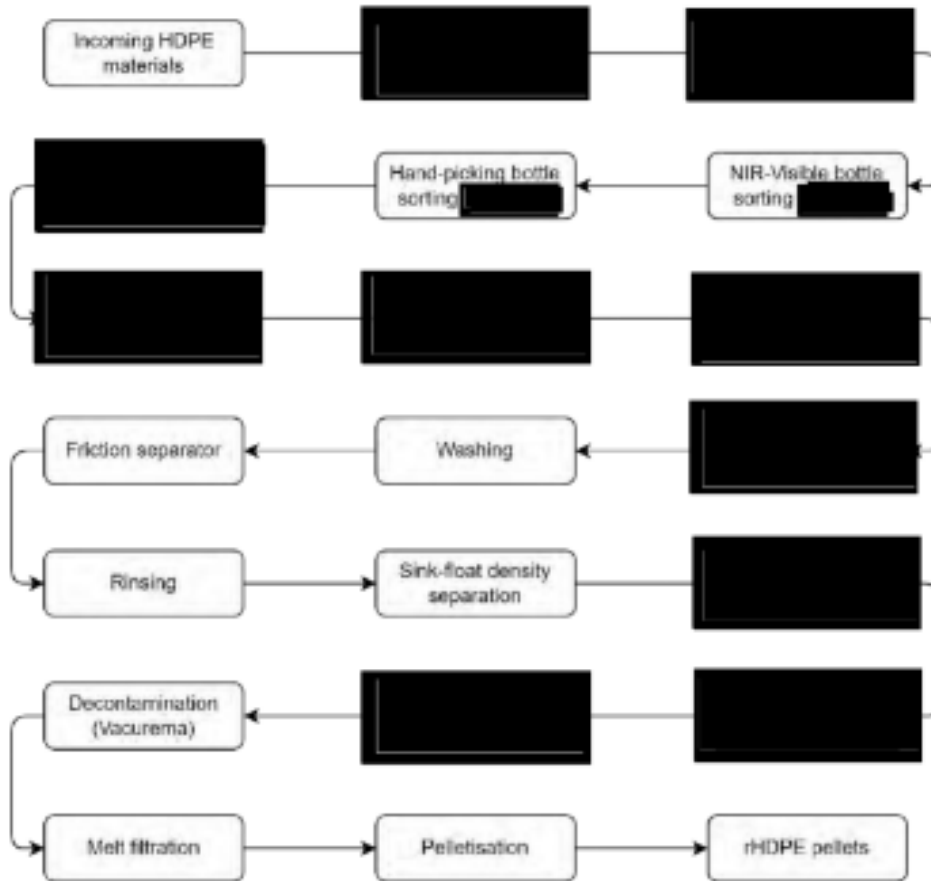


Figure 2: Veolia Plastics process flow diagram



2. Sampling strategy

The strategy is designed to ensure consistent quality and safety at each stage of the recycling process. Here's a detailed breakdown of the sampling and testing procedures:

- I. Entry Stage (Every batch):
 - A. Visual inspection of all bales
 - B. Random testing of one bale per load using ISO 15344:2008
 - C. Traceability verification through supplier pre-assessments, declarations, regulated waste transfer notes and supplier audits
 - D. Individual batch identification based on ISO 22095:2020
- II. Post-sorting (after the hand-picking bottle sorting stage) and pre-wash
 - A. Bottle count every 2 hours to confirm previous food use purity
- III. Input Stage (Every batch - random spot sample during production of that batch):
 - A. Physical characteristics:
 1. Presence of non-HDPE plastics
 2. Glue content
 - B. Chemical analysis:
 1. Quantitative analysis for volatiles (Limonene) using GC-MS(Headspace)
 2. Semi-quantitative analysis using THF extraction (ASTM D7210-21)
 3. Migration study using 50% ethanol (EC 10/2011)
- IV. Output Stage (Composite sample, minimum 1 kg/hr at 1t/hr production rate):
 - A. Physical characteristics:
 1. Melt Flow Rate at 2.16 kg and 21.6kg, density, shape and colour
 - B. Chemical analysis:
 1. Semi-quantitative analysis using THF extraction (ASTM D7210-21)
 2. Migration study using 50% ethanol (EC 10/2011)
- V. Exit Stage:
 - A. Declaration of Compliance
 - B. Compliant labelling

Sampling every batch with full traceability for at least a full year is designed to factor in seasonal, suppliers and process variations. This will provide a larger dataset for trending and reduce the overall uncertainty.

3. Monitoring

a. Entry quality

For commercial reasons results for incoming material are not discussed in this report although the records are readily available for the competent authorities to inspect at any time.

REC-001-01 Suppliers Register

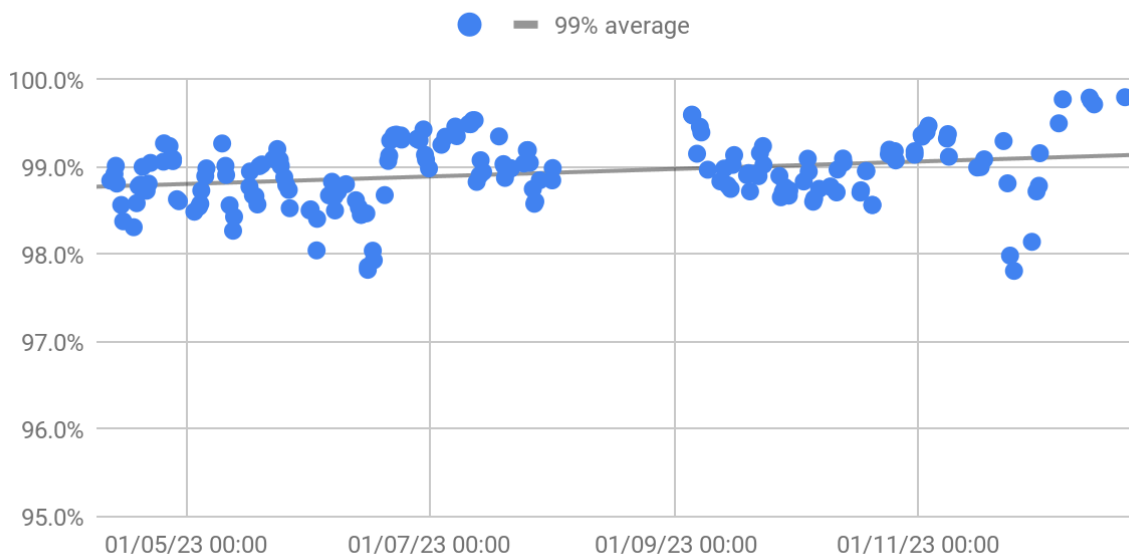
REC-002-01 Incoming HDPE Load Inspections - LAB

REC-018-02 Supplier and Storage Audit Log

Traceability of the process has been independently assessed using PRE RecyClass - Certification compliant with EN 15343:2007 audit report and certificate code: RP261-VEO-07-25-CIR-PC)

b. Pre-sorted Quality

Batch Average Food Use Purity After Sorting by Counting Method

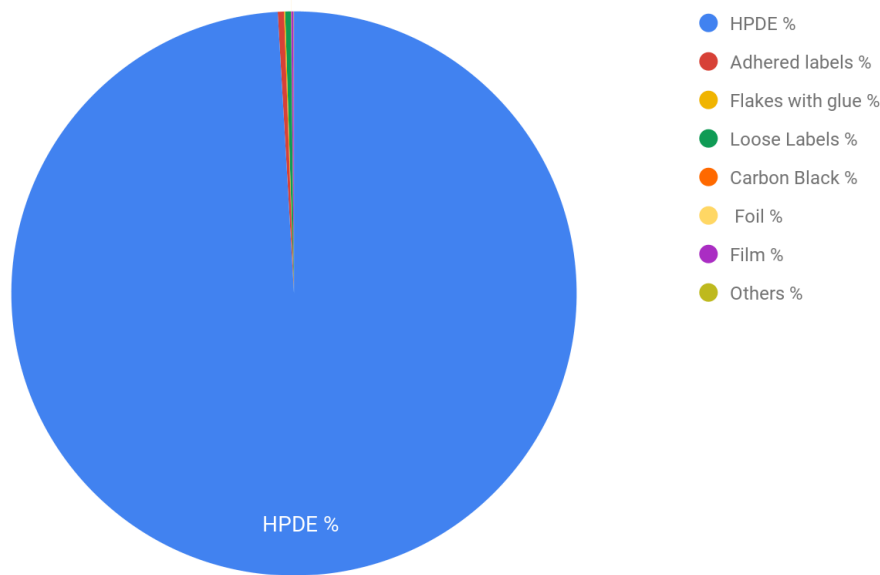


Average 99% after the initial sorting but before granulation and further sorting. This data is consistent with the [Technical dossier](#) page 26 / 57

c. Input Quality
i. Contaminating Materials in Plastic Input

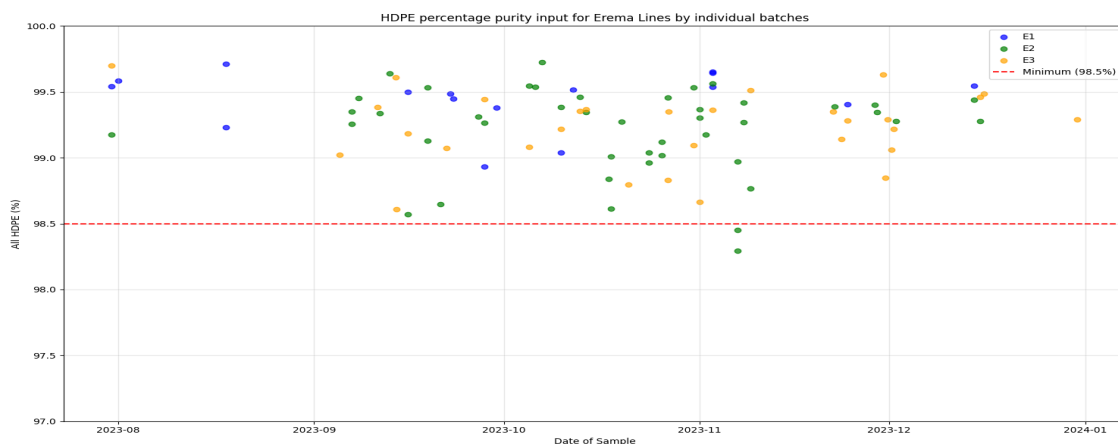
Average of input quality by mass percentage (raw data: REC-061 HDPE Flake)

From	06/06/2023	TO	10/12/2023	Number of samples		145	
HPDE %	Adhered labels %	Flakes with glue %	Loose Labels %	Carbon Black %	Foil %	Film %	Others %
99.27	0.36	0.06	0.32	0.01	0.01	0.13	0.03



Carbon black is typically from the middle layer in long life milk bottles.
 Foil is typically from the milk bottle foil seal at the top of the bottle.

99% HDPE input is consistent with the data in the [Technical dossier](#) page 27 / 57



Samples below 98.5% HDPE:

Date: 2023-11-07, Line: E2/EF2, HDPE: 98.45%, Retest: 99.42%, Average: 98.93%, Result: Passed

Date: 2023-11-07, Line: E2/EF2, HDPE: 98.29%, Retest: 99.42%, Average: 98.86%, Result: Passed

ii. Substances found in plastic inputs

Semi-quantitative analysis using THF extraction (ASTM D7210-21) on the flakes which was analysed on a GC-MS (raw data: REC-062 THF screening on flakes).

458 Input samples were checked between 10 June 2023 and 09 December 2023 this covered all the batches produced during that time, screening down to 1.5 ppm

Library/ID	Total	Washed Flake Mean Conc	Washed Flake Max Conc	Washed Flake RT	CAS#	Qual	Source
1-Docosene	320	6.96	13.31	23.39	001599-67-3	64	Oligomer
1-Hexacosene	303	4.88	14.34	26.56	018835-33-1	64	Oligomer
Cetene	270	7.48	20.16	16.85	000629-73-2	64	Oligomer
Cyclotetracosane	204	7.12	11.51	24.77	000297-03-0	62	Oligomer
5-Eicosene, (E)-	200	6.5	16.41	22.04	074685-30-6	64	Oligomer
Tris(2,4-di-tert-butylphenyl) phosphate	196	14.24	46.3	32.65	95906-11-9	60	Irgafos 168
Hexadecane	165	4.33	41.87	16.62	000544-76-3	64	Oligomer
1-Tetracosene	162	6.69	17.77	24.75	010192-32-2	64	Oligomer
1-Octadecene	151	6.64	19.61	19.03	000112-88-9	64	Oligomer
Isopropyl myristate	139	4.85	45.89	18.95	000110-27-0	62	Cosmetics
Cyclooctacosane	132	4.2	10.53	27.48	000297-24-5	64	Oligomer
Tetradecane	124	4.27	41.89	14.11	000629-59-4	64	Oligomer
3-Eicosene, (E)-	116	7.64	18.23	21.09	074685-33-9	64	Oligomer
Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	116	3.88	6.97	32.97	002082-79-3	60	Irganox 1076
Eicosane	112	2.63	11.39	20.59	000112-95-8	72	Oligomer
Octadecane	104	3.33	9.57	18.77	000593-45-3	70	Oligomer
Octanal, 2-(phenylmethylene)-	98	2.84	8.73	18.14	000101-86-0	64	Cosmetics
E-15-Heptadecenal	95	8.04	19.26	19.36	1000130-97-9	72	TBC
Isopropyl palmitate	95	2.71	10.69	21.02	000142-91-6	62	Cosmetics
D-Limonene	89	7.22	49.8	8.86	005989-27-5	78	Flavour
1-Tricosene	73	5.96	16.33	25.56	018835-32-0	70	Oligomer

* Multiply the concentration by 10 to convert the concentration in solution to concentration in the polymer as mg/kg

d. Output Quality

i. Substances found in plastic output

Semi-quantitative analysis using THF extraction (ASTM D7210-21) analysed on GC-MS

Raw data: REC-062 THF screening on pelletsW

413 samples were checked between 10 June 2023 and 09 December 2023 this covered all of the batches produced during that time, screening down to 1.5 ppm.

Library/ID	Total	Pellet Mean Conc	Pellet Max Conc	Pellet RT	CAS#	Qual	Source
1-Docosene	284	5.38	11.64	23.15	001599-67-3	72	Oligomer
Cyclotetracosane	225	4.85	10.73	24.69	000297-03-0	62	Oligomer
1-Hexacosene	208	3.58	12.37	26.11	018835-33-1	64	Oligomer
5-Eicosene, (E)-	157	5.89	16.63	21.3	074685-30-6	64	Oligomer
Tris(2,4-di-tert-butylphenyl) phosphate	139	9.45	25.52	32.59	95906-11-9	60	Irgafos 168
1-Tetracosene	135	5.02	13.06	24.67	010192-32-2	64	Oligomer
1-Octadecene	133	5.4	14.62	19.44	000112-88-9	78	Oligomer
Isopropyl myristate	129	3.16	5.35	18.89	000110-27-0	62	Cosmetics
Cetene	128	5.28	13.21	17.68	000629-73-2	70	Oligomer
3-Eicosene, (E)-	112	6.29	14.65	21.44	074685-33-9	68	Oligomer
Cyclooctacosane	105	2.96	7.07	27.14	000297-24-5	62	Oligomer
Hexadecane	95	4.92	39.78	16.57	000544-76-3	64	Oligomer
9-Eicosene, (E)-	89	6.45	19.69	20.25	074685-29-3	64	Oligomer
E-15-Heptadecenal	86	6	16.48	19.99	1000130-97-9	64	TBC
Octadecane	80	2.48	6.53	18.64	000593-45-3	72	Oligomer
Isopropyl palmitate	76	2.14	3.65	20.98	000142-91-6	60	Cosmetics
17-Pentatriacontene	72	3.55	8.66	27.65	006971-40-0	62	Oligomer
1-Tricosene	51	6.08	15.23	24.17	018835-32-0	64	Oligomer
Nonacos-1-ene	50	3.82	8.03	25.9	018835-35-3	76	Oligomer
5-Octadecene, (E)-	48	9.4	19.48	18.89	007206-21-5	72	Oligomer
1-Nonadecene	46	6.49	14.66	23.04	018435-45-5	64	Oligomer
Eicosane	44	1.89	3.62	20.4	000112-95-8	68	Oligomer

Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy- , octadecyl ester	43	2.66	5.04	32.83	002082-79-3	60	Irganox 1076
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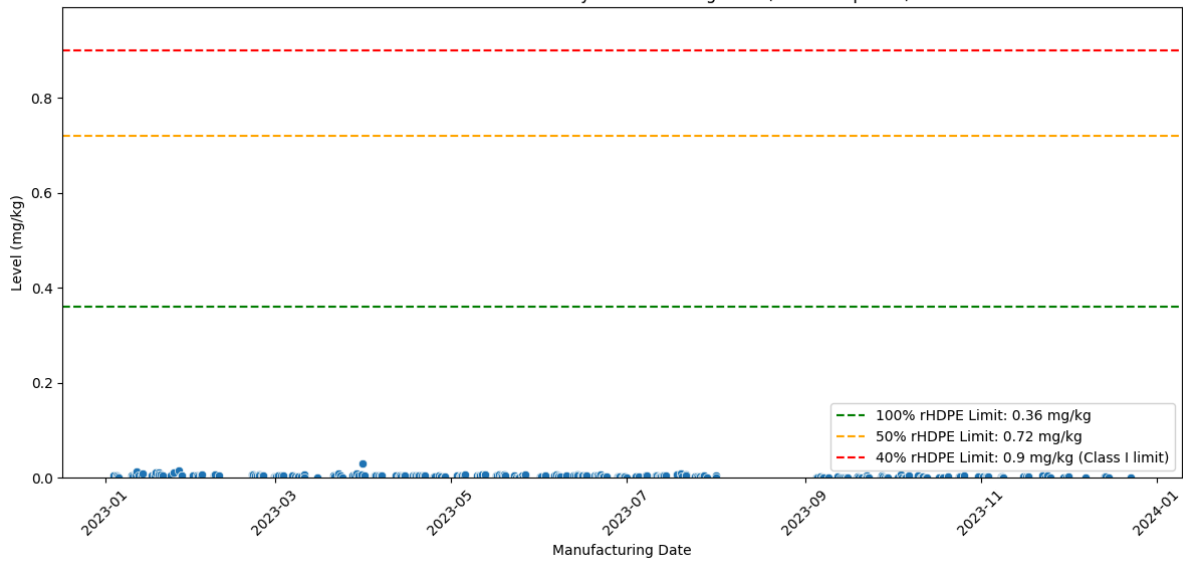
* Multiply the concentration by 10 to convert the concentration in solution to concentration in the polymer as mg/kg

ii. **Migration levels of contaminants** (Article 13(5)(f) a measurement or estimation of the migration levels);

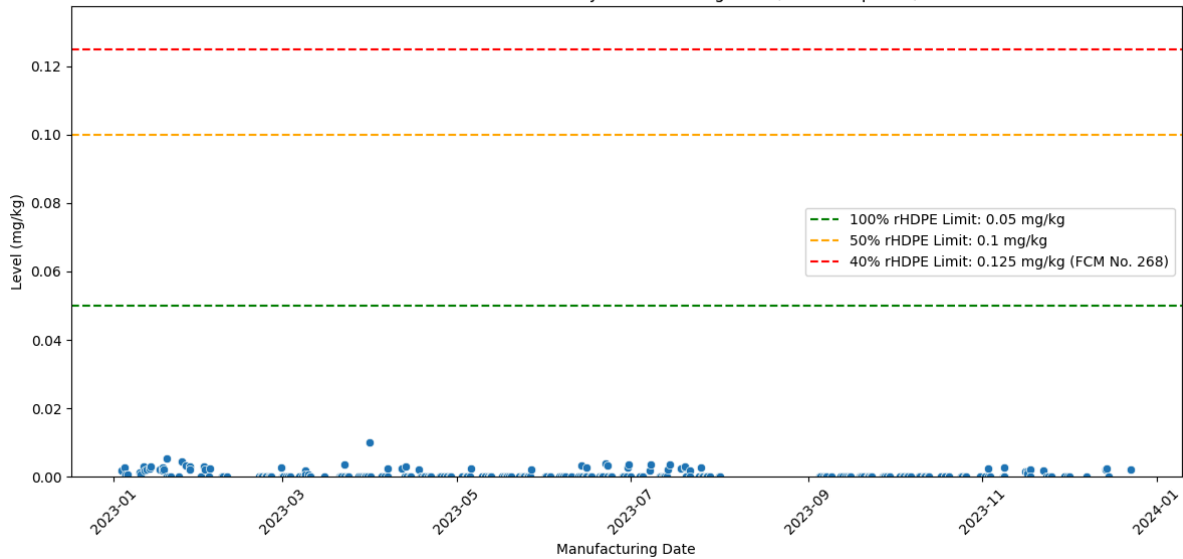
Every batch produced complied with the limits stated on the declaration of compliance when blended with virgin at 40%. This can be found on the website <https://dagenhamplastics.veolia.co.uk/>

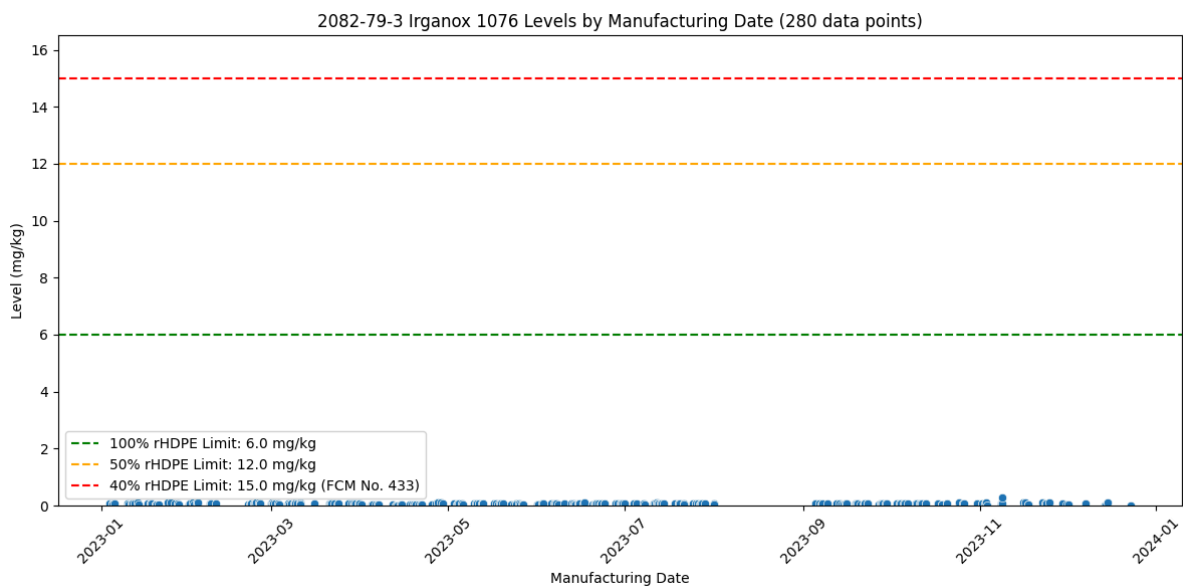
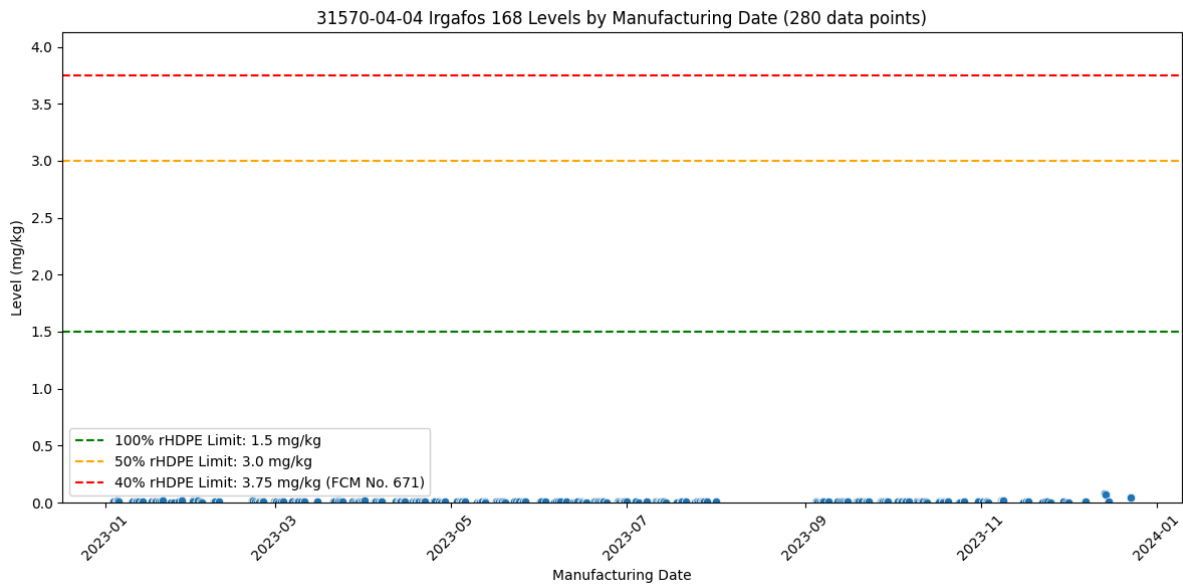
CAS	Name	mg/kg	FCM
5989-27-5	Limonene	<0.017	(Class I)
112-41-4	1-Dodecene	<0.008	268
31570-04-04	Irgafos 168	<0.040	671
2082-79-3	Irganox 1076	<0.181	433
6259-76-3	n-Hexyl salicylate	<0.045	(Class II)
5444-75-7	2-Ethylhexyl benzoate	<0.049	(Class I)
101-86-0	α -Hexyl cinnamaldehyde	<0.045	(Class II)
110-27-0	Isopropyl myristate	<0.150	(Class I)
142-91-6	Isopropyl palmitate	<0.040	(Class I)
109-43-3	Dibutyl sebacate	<0.181	242
-----	Unknown	<0.010	

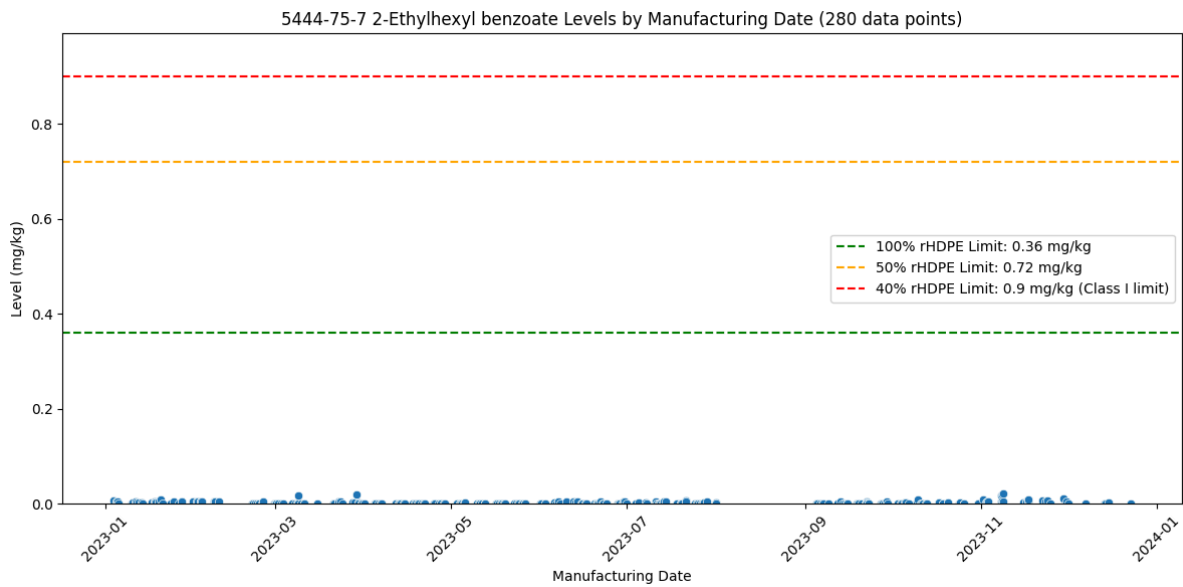
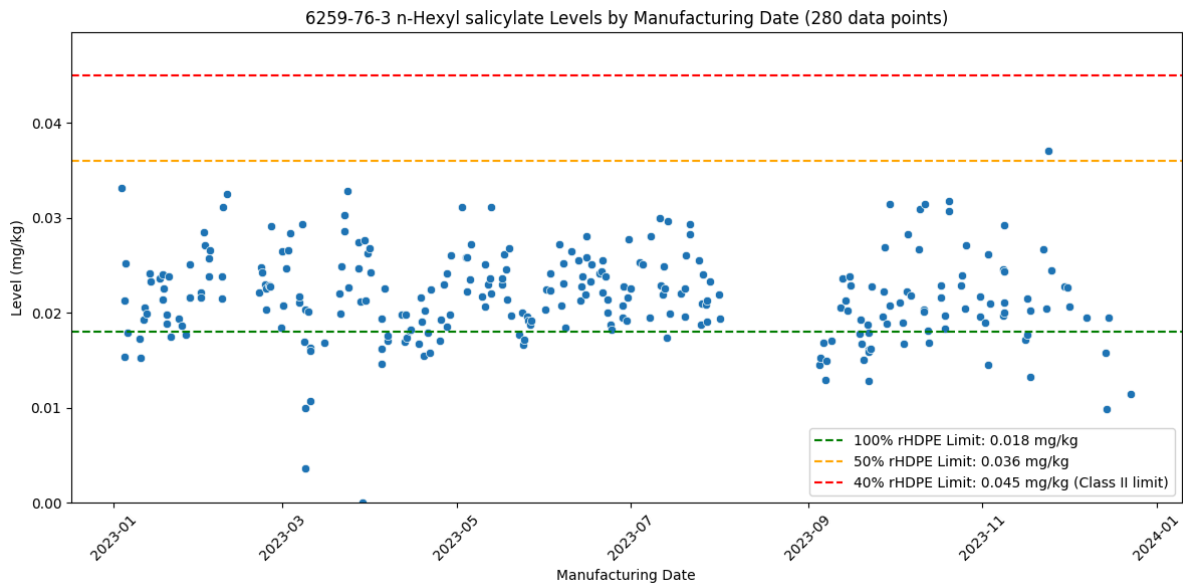
5989-27-5 Limonene Levels by Manufacturing Date (280 data points)

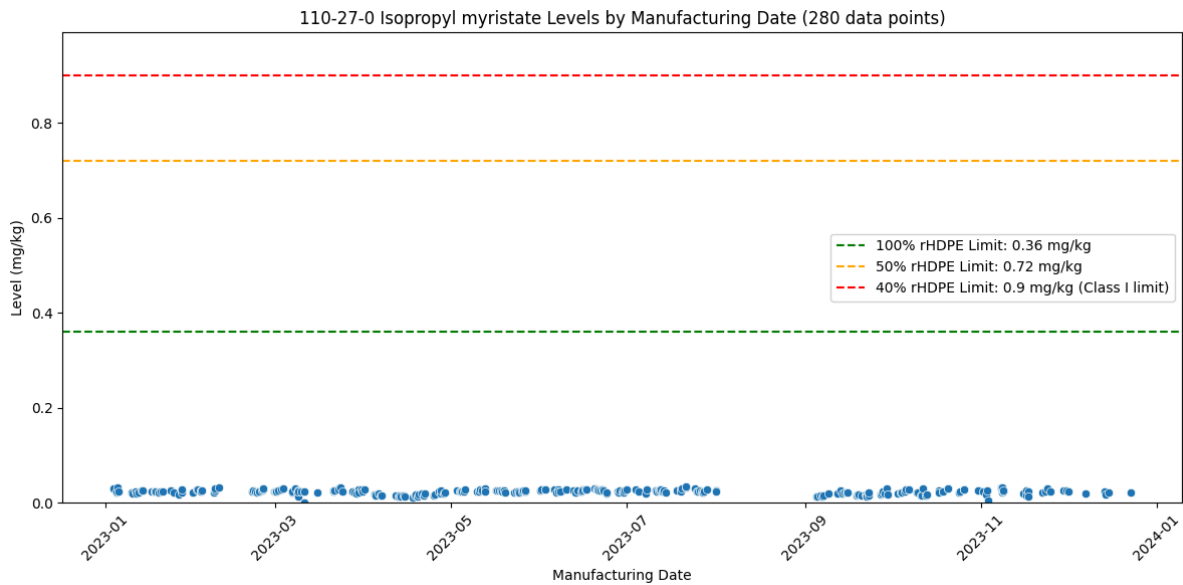
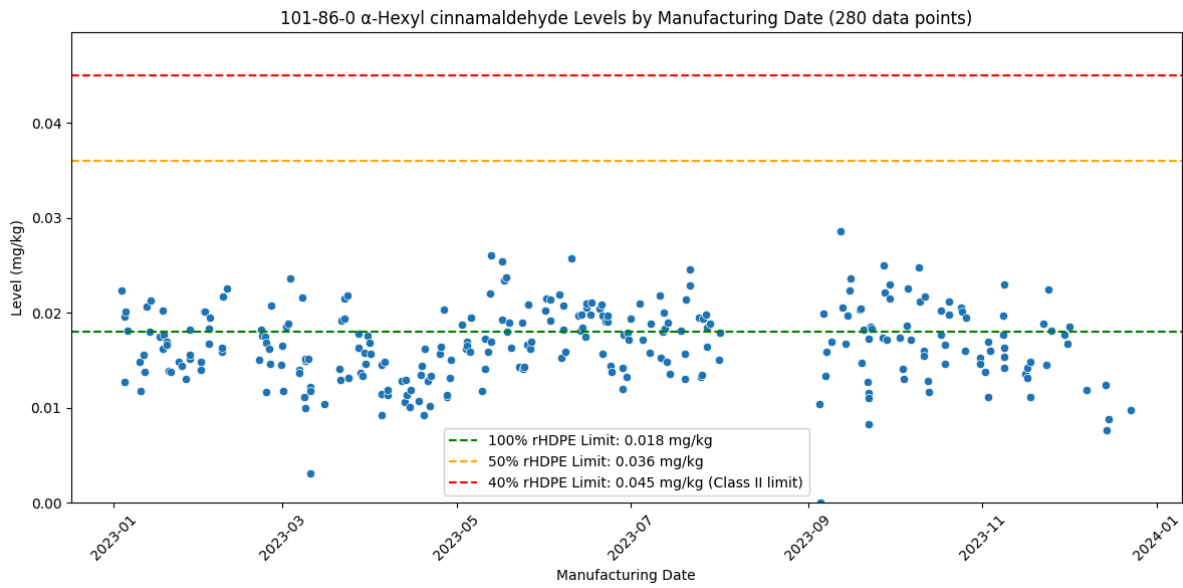


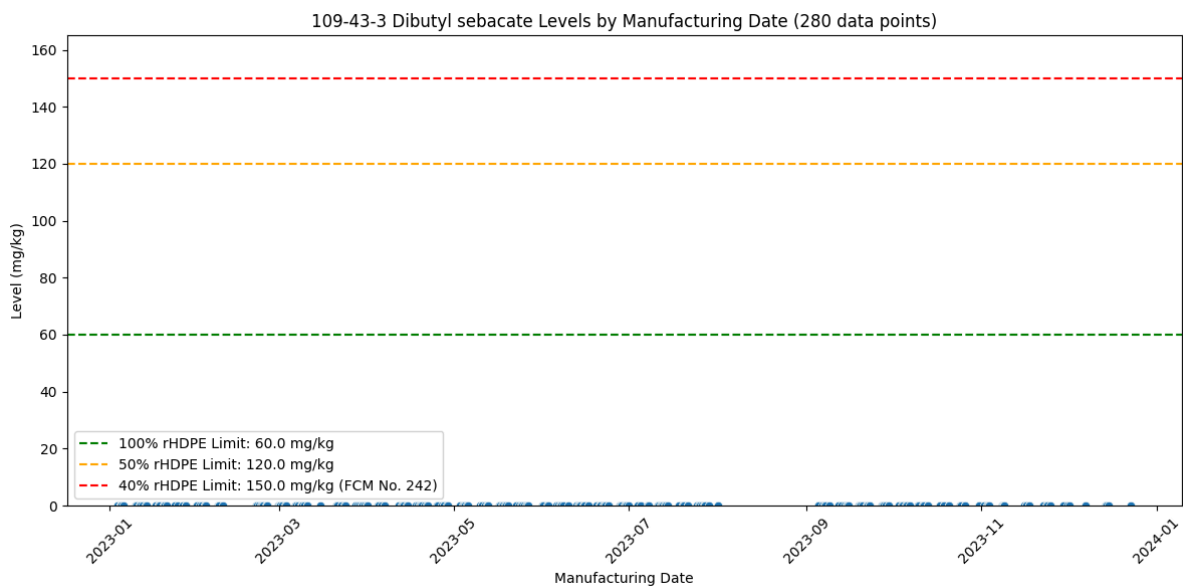
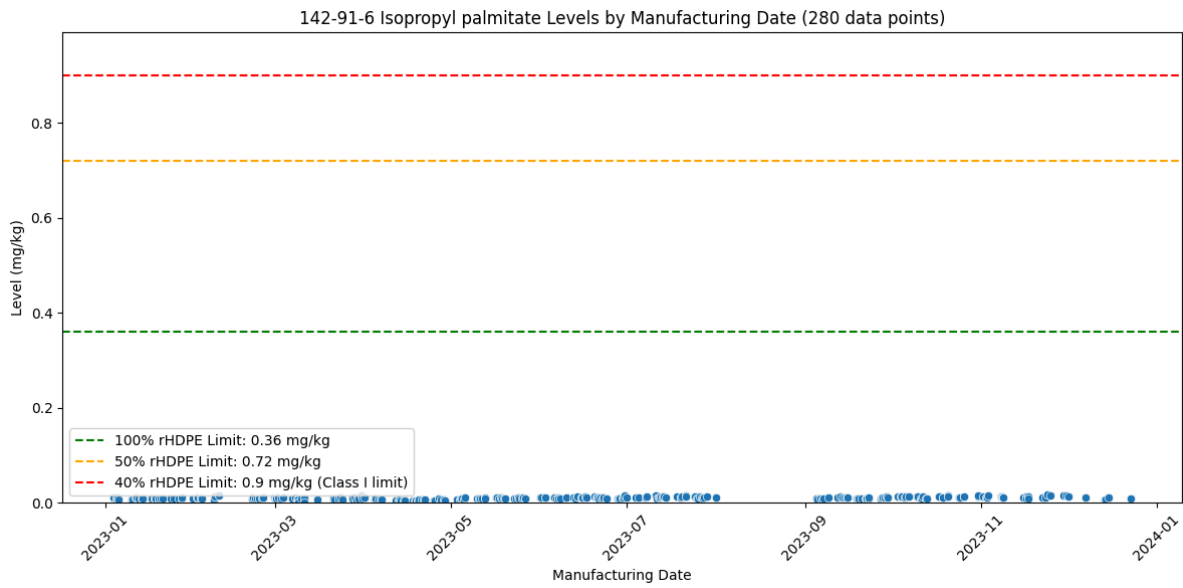
112-41-4 1-Dodecene Levels by Manufacturing Date (280 data points)

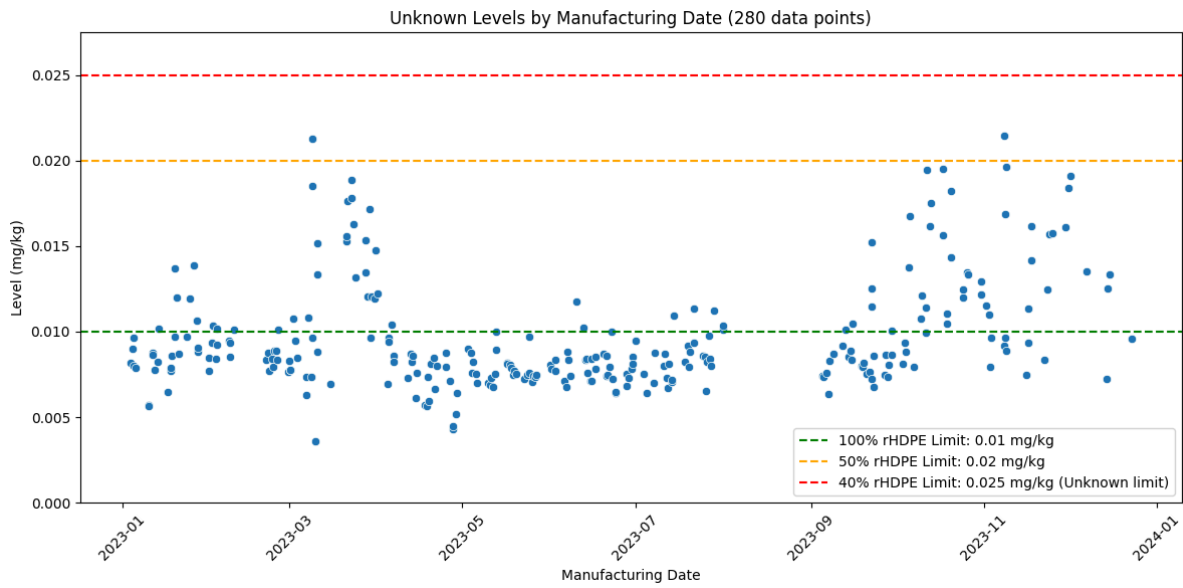








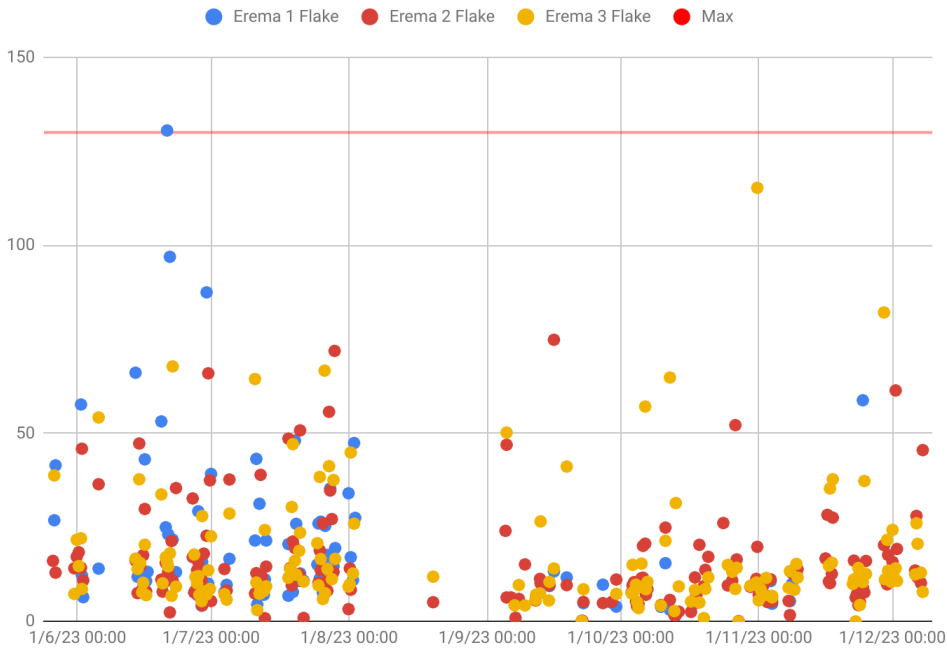




Whilst it is possible to meet the limits of 50% in most cases further work is ongoing to identify unknowns and sources of Class II compounds to maintain that margin of safety.

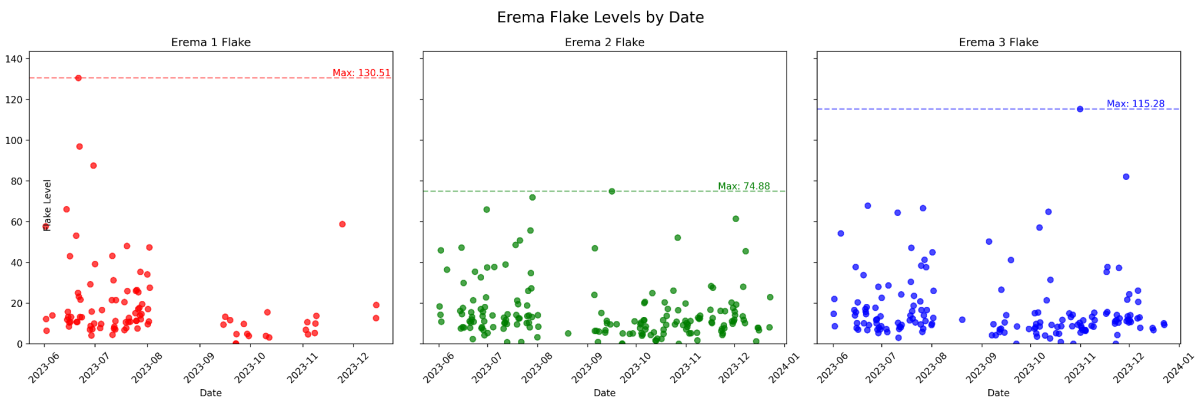
e. Decontamination efficiency

Input flake limonene concentrations (ppm) by headspace method (overlaid)

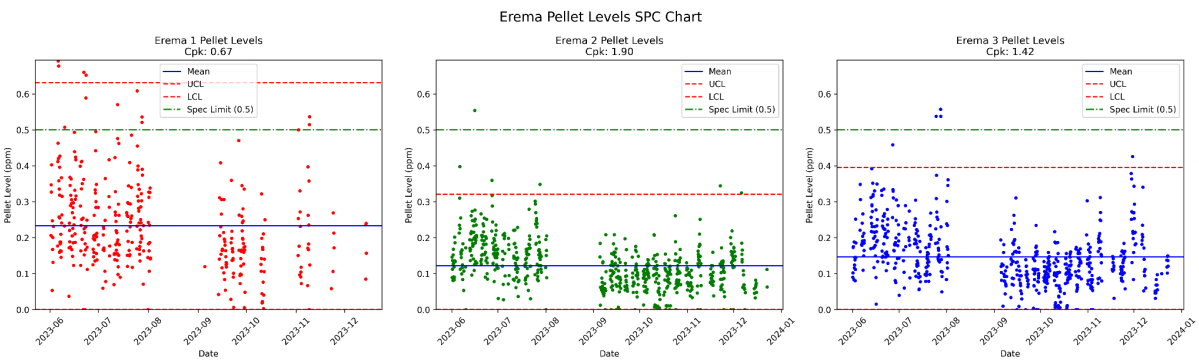


(Raw data: REC-009-01 FG rHDPE Pellet Results)

Input limonene concentrations (ppm) by headspace method (individuals plots)



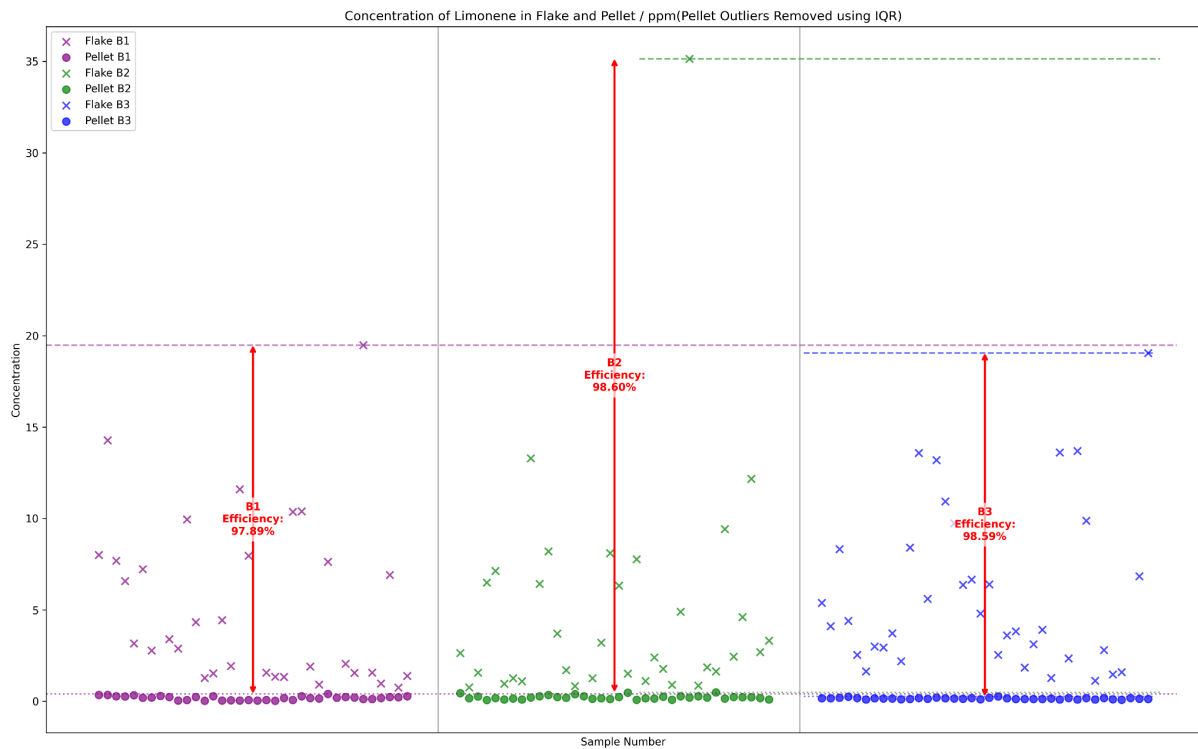
Output limonene concentrations (ppm)



(Raw data: REC-009-01 FG rHDPE Pellet Results)

Erema	Input Date Time	Output Date Time	Input Limonene	Output Limonene	Decontamination Efficiency (%)
1	2023-06-21 03:00:00	2023-06-21 12:00:00	130.51	0.25	99.8
2	2023-09-16 00:00:00	2023-09-16 21:00:00	74.88	0.12	99.8
3	2023-10-31 16:00:00	2023-10-31 21:00:00	115.28	0.07	99.9

Decontamination efficiency was also calculated for limonene using the THF extraction results



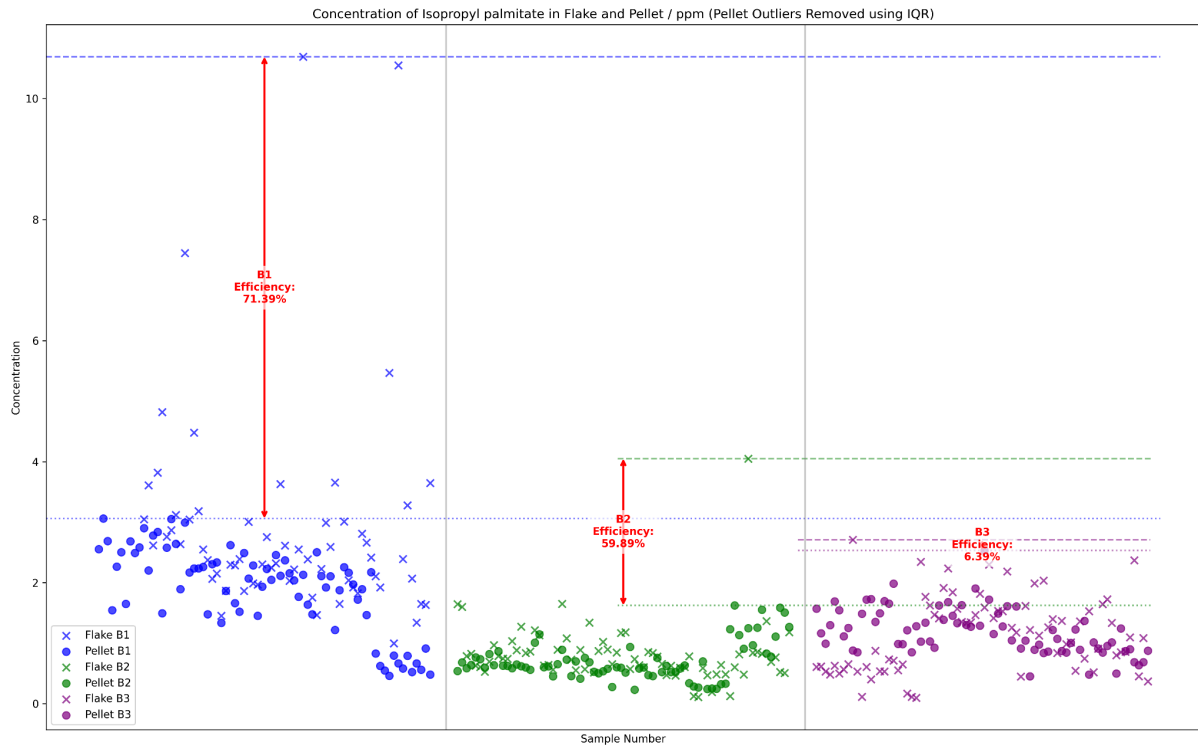
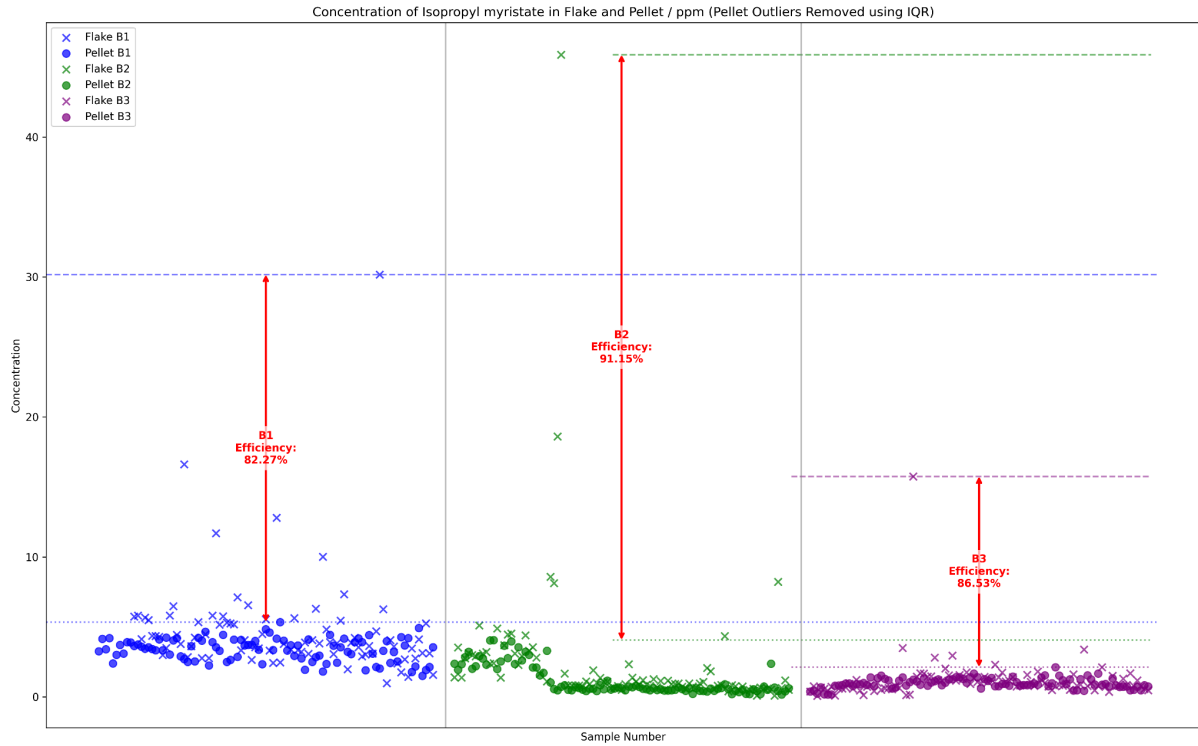


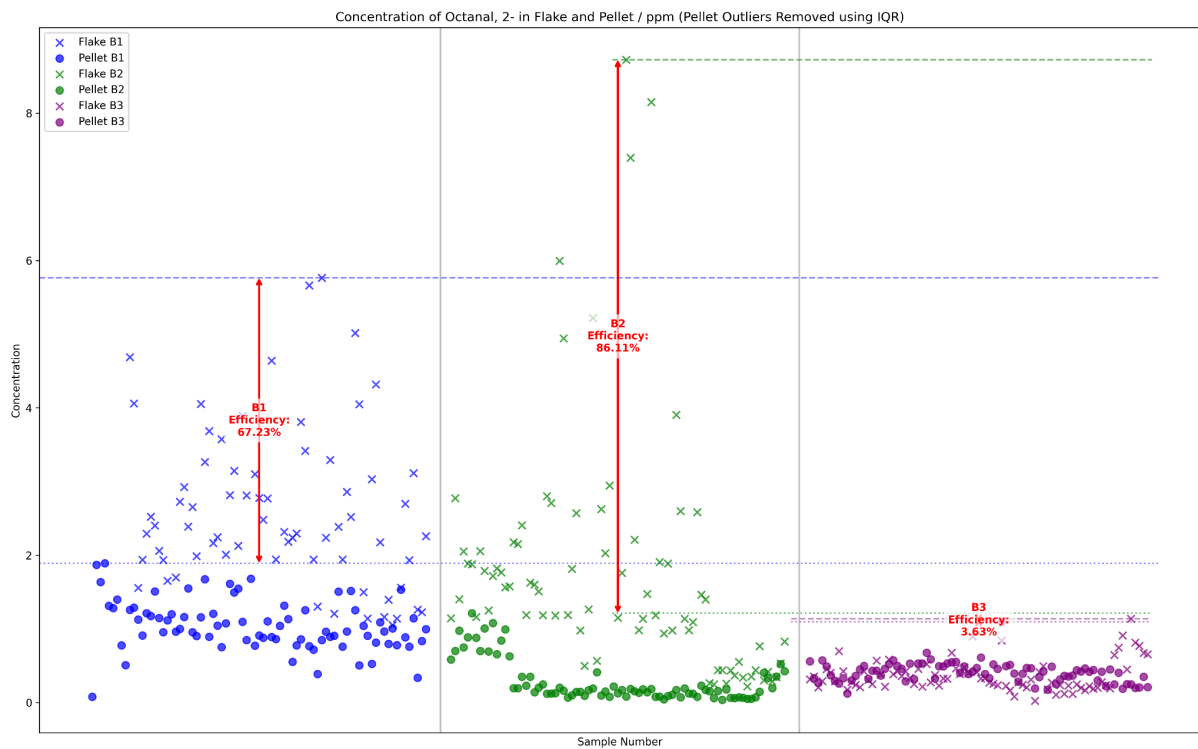
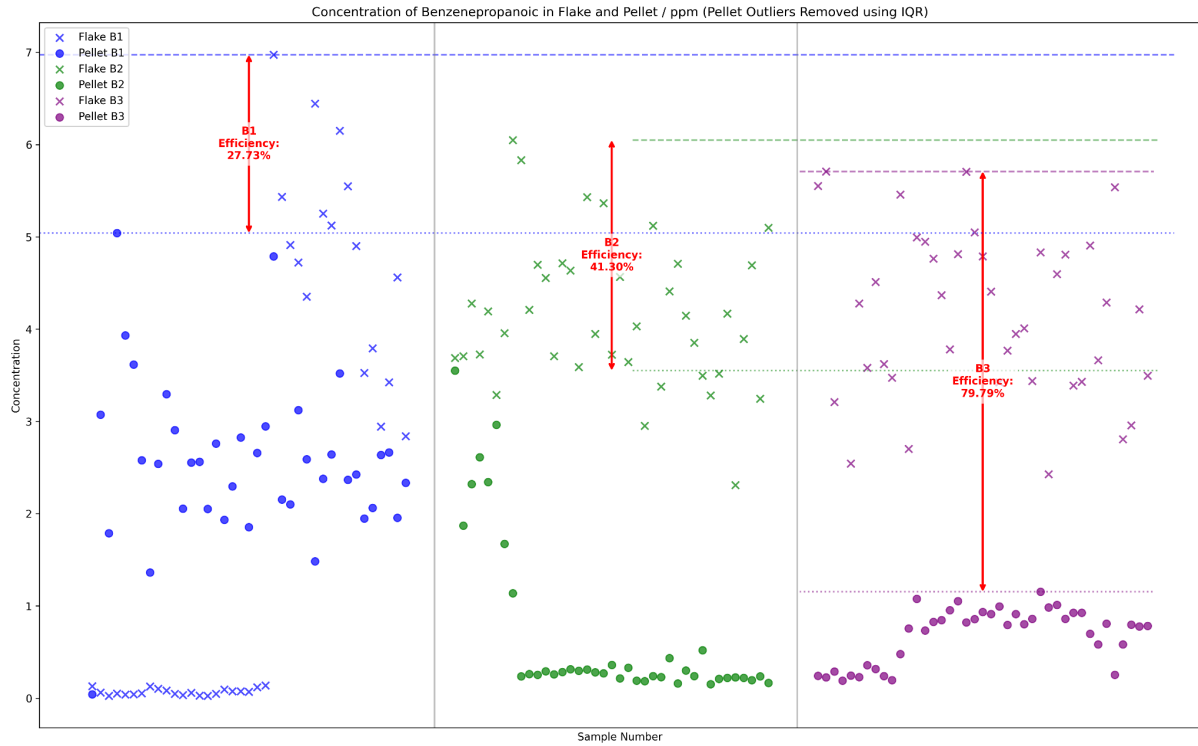
Decontamination efficiencies for a similar set of samples between June 2023 and December 2023 calculated using THF extraction were slightly lower than the headspace extraction.

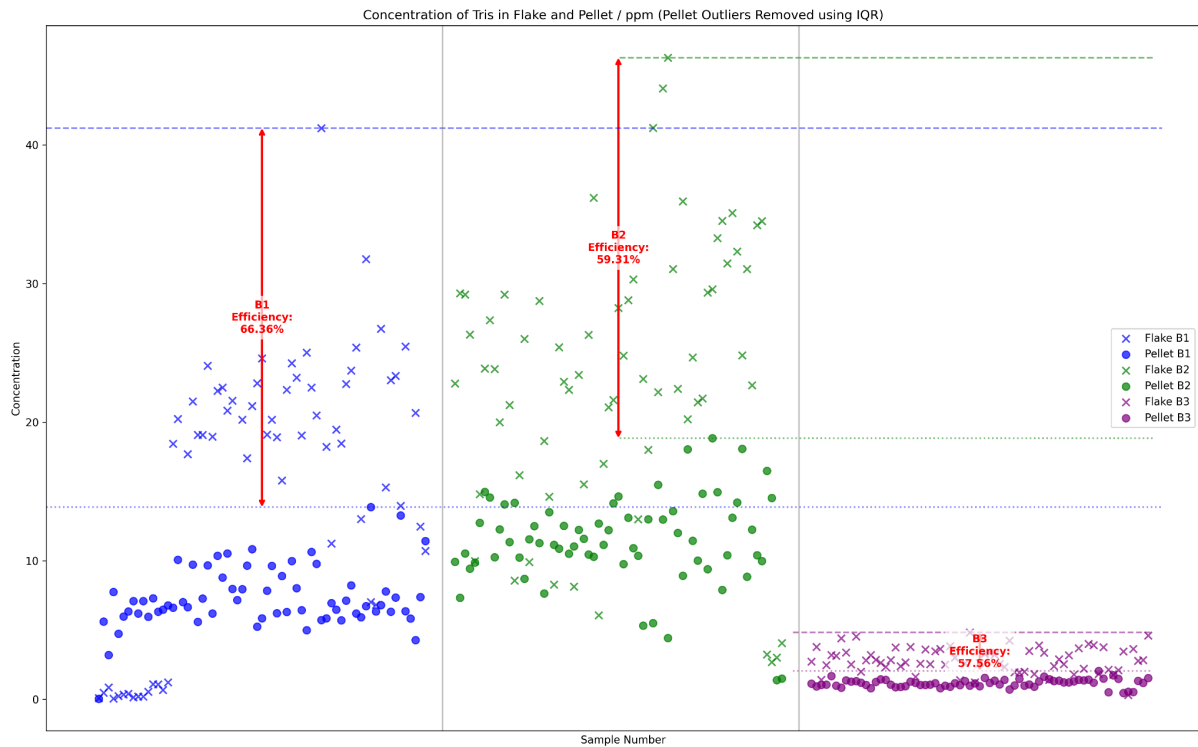
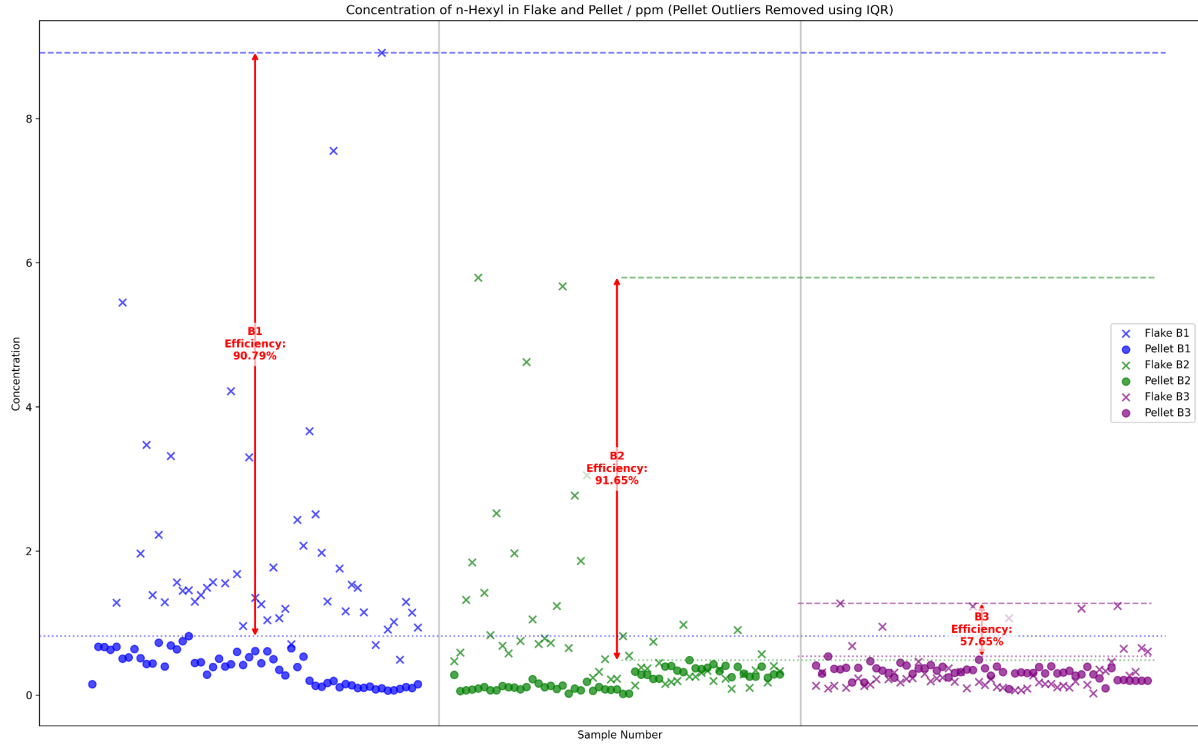
Other THF extractions were studied as they represented compounds found in the output migrations

Compound	Batch	Max Flake Sample ID	Max Flake Sample Name	Max Flake Conc.	Max Pellet Sample ID	Max Pellet Sample Name	Max Pellet Conc.	Decontamination Efficiency (%)
Limonene	B1	D-Limonene	E2WF3_1500_1 7112023TD.D	19.48	D-Limonene	B35376_E3P1 _0600_061220 23AH.D	0.41	97.89363872
Limonene	B2	D-Limonene	E2WF1_0000_2 1102023AH.D	35.15	D-Limonene	B21352_E2P2 _1200_011220 23AH.D	0.49	98.60091075
Limonene	B3	D-Limonene	E3WF2_0900_2 4102023AH.D	19.06	D-Limonene	B35361R_CO MP_E3P1_AH. D	0.27	98.59192453
Isopropyl myristate	B1	Isopropyl myristate	E1WF2_2100_1 1072023AH.D	30.17	Isopropyl myristate	E2P2_0300_16 112023TD.D	5.35	82.27196718
Isopropyl myristate	B2	Isopropyl myristate	E1WF2_0900_1 1102023TD.D	45.89	Isopropyl myristate	E3P3_0300_21 102023AH.D	4.06	91.14859834
Isopropyl myristate	B3	Isopropyl myristate	E2WF1_0300_2 0230726PG.D	15.76	Isopropyl myristate	B34365R_E3P 3_1800_21092 023TD.D	2.12	86.53106839
Isopropyl palmitate	B1	Isopropyl palmitate	E2WF2_1100_3 1102023SP.D	10.69	Isopropyl palmitate	E2P1_0300_16 112023TD.D	3.06	71.38653278
Isopropyl palmitate	B2	Isopropyl palmitate	E1WF3_1200_2 0230601SP.D	4.05	Isopropyl palmitate	B34375_E3P1 _2000_231120 23AH.D	1.62	59.89100535
Isopropyl palmitate	B3	Isopropyl palmitate	EF2WF1_0400_ 20230801SP.D	2.71	Isopropyl palmitate	B34365R_E3P 3_1800_21092 023TD.D	2.53	6.387698466
Benzenepropanoic	B1	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	E3WF3_0300_3 1102023TD.D	6.97	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	E2P2_0300_16 112023TD.D	5.04	27.72811916
Benzenepropanoic	B2	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	E1WF1_1000_1 6062023AH.D	6.05	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	E3P3_0300_20 102023AHO.D	3.55	41.30352403
Benzenepropanoic	B3	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	E3WF1_0000_0 7102023TD.D	5.71	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	B35374R_E3P 1_0600_29112 023AH.D	1.15	79.79435478
Octanal, 2-	B1	Octanal, 2-(phenylmethylene)-	E2WF1_1100_3 1102023SP.D	5.77	Octanal, 2-(phenylmethylene)-	E3P1_1300_17 1123TD.D	1.89	67.22668629
Octanal, 2-	B2	Octanal, 2-(phenylmethylene)-	E3WF3_0900_2 4102023AH.D	8.73	Octanal, 2-(phenylmethylene)-	E2P2_0300_07 102023AHO.D	1.21	86.11096294
Octanal, 2-	B3	Octanal, 2-(phenylmethylene)-	B12314_E1WF3 _2100_1809202 3AH.D	1.14	Octanal, 2-(phenylmethylene)-	B34365R_E3P 3_1800_21092 023TD.D	1.09	3.634610322
n-Hexyl	B1	n-Hexyl salicylate	E3WF3_1100_3 1102023SP.D	8.91	n-Hexyl salicylate	E3P2_0300_01 112023SP.D	0.82	90.79153649
n-Hexyl	B2	n-Hexyl salicylate	E2WF1_0900_2 4102023AH.D	5.79	n-Hexyl salicylate	B35374R_E3P 3_0600_29112 023AH.D	0.48	91.64502107
n-Hexyl	B3	n-Hexyl salicylate	EF1WF2_1900_ 20230622.D	1.27	n-Hexyl salicylate	B35374R_E3P 1_0600_29112 023AH.D	0.54	57.65187242
Tris	B1	Tris(2,4-di-tert-butylphenyl) phosphate	E2WF1_0000_0 7102023AHO.D	41.22	Tris(2,4-di-tert-butylphenyl) phosphate	E2P2_0300_16 112023TD.D	13.86	66.36339018

Tris	B2	Tris(2,4-di-tert-butylphenyl) phosphate	E2WF3_0900_20230928.D	46.30	Tris(2,4-di-tert-butylphenyl) phosphate	E2P1_1200_20230928.D	18.84	59.30868161
Tris	B3	Tris(2,4-di-tert-butylphenyl) phosphate	E1WF1_0300_20230728.D	4.83	Tris(2,4-di-tert-butylphenyl) phosphate	E1S2P3_20230725_1300.D	2.05	57.55632229







4. Analytical Procedures

- a. **Appendix 1: Migration: Quantitative Evaluation of Purity of Recycled HDPE by rapid migration test.** This method was originally developed by (Pira International Research) reference: 08A11J0703 Development of an accelerated migration test for recycled rHDPE (Pira International Research) September 2008. The method has been updated to include calibrations for known substances commonly occurring.
- b. **Appendix 2: Headspace: Quantitative Determination of Limonene Concentration in HDPE by Headspace GC-MS.** This method was originally developed by (Pira International Research) reference: 08A11J0703
- c. **Appendix 3: Extraction: Analysis of Non-Intentionally Added Substances (NIAS) in Recycled HDPE.** Based on ASTM D7210-21

5. Summary

a. Review

Summary of the reasoning on the capability of the technology to produce safe recycled materials (Article 13.5 b):

The Veolia Plastics rHDPE recycling process demonstrates a robust capability to produce safe recycled materials for food contact applications, particularly milk bottles. This conclusion is supported by several key factors: First, the process consistently achieves a high decontamination efficiency, with worst-case scenarios showing 99.8% removal of surrogate contaminants like limonene. This level of decontamination is comparable to that demonstrated in challenge tests, indicating the process's effectiveness in removing potential contaminants. Secondly, the output quality remains consistently high, regardless of input variability. Migration testing on every batch confirms compliance with EU Regulation 10/2011, with all monitored substances falling below specified limits. The process's ability to maintain this quality, even with varying input contamination levels, demonstrates its reliability in producing safe recycled materials. Additionally, the comprehensive monitoring strategy, which includes analysis of both known contaminants and potential NIAS, provides ongoing assurance of the technology's capability to meet food safety requirements.

Analysis of whether contaminant origins could lead to other undetected substances of concern (Article 13.5 e):

The majority of contaminants identified in the input material can be traced to three primary sources: residual product contents (e.g., milk components), packaging additives, and environmental contamination. While the current analytical methods effectively detect and quantify a wide range of substances, there is a possibility of undetected compounds of concern.

For instance, residual milk components could potentially harbor unidentified metabolites or breakdown products. Packaging additives, while generally well-characterized, may include proprietary formulations with undisclosed components. Environmental contaminants present perhaps the most significant unknown, as they can vary widely based on the post-consumer waste's origin and handling. To address these potential unknowns, ongoing research is focused on expanding the range of analyzed compounds and lowering detection limits. The sorting process, which achieves at least 99% food-use HDPE packaging, significantly reduces the risk of non-authorized additives. Additionally, the process's high



decontamination efficiency provides a safety margin against potential undetected substances, assuming they would behave similarly to known contaminants during processing.

b. Discussion of the difference of previous report

Compared to previous reports, this dataset demonstrates a high degree of consistency in both input quality and process performance. The decontamination efficiency remains stable, with no significant deviations from earlier findings. However, one notable difference is the increased focus on identifying and quantifying frequently occurring compounds. This shift has led to the identification of a new substance E-15-Heptadecenal, which was not prominently featured in previous reports.

The previous technical reports used decontamination efficiencies based on challenge test data. Monitoring day-to-day decontamination efficiencies are only indicative as a large number of tests need to be performed to find occasional input with elevated levels. Even on these rare occasions the input is still approximately 90% lower than the levels used in the challenge test.

c. Analysis of discrepancies in decontamination efficiency

Three methods could have been used to monitor the decontamination efficiency. Each method has advantages and disadvantages. The headspace method is quantitative and fast. This allows for more measurements throughout the batch for every batch. Limonene can be monitored as an indicator of decontamination efficiency for volatile compounds. The disadvantage of this method is that there is less confidence in detection of semi-volatile compounds. The extraction method is performed on every batch using an aggressive solvent to give more confidence in extracting all chemicals present. This technique is ideal for NIAS and satisfactory for the decontamination efficiency; it should be noted that numbers are semi-quantitative. The migration is much better suited to quantitative measurements of the output. All three methods provide valuable information about decontamination performance and efficiency.

d. Other considerations

The monitoring data focuses on decontamination efficiency

e. Recommendations

i. Developer

1. Continue to monitor data for trends

ii. Recycler

1. Continue to identify frequently occurring unknowns and quantify them using calibrated standards
2. Find the source of E-15-Heptadecenal and confirm any migration levels using a calibrated standard

iii. Reprocessors

1. Feedback on both positive and negative issues
2. Share external test reports

iv. Competent Authority Assessment

Article 13(5)(i) an analysis and explanation of any discrepancies observed between contaminant levels expected in the input plastic and in the output of the installation and its decontamination efficiency based on the reasoning provided under point (b) and the actual results under point (c).

This dataset remains similar to the initial technical report and shows batch batch consistency irrespective of any variation in the input material quality.

The data showed that routinely both input and output contamination levels were low. The main reason is due to a high quality of input material and also a consistently good decontamination stage. The low level of input contamination made it difficult to compare the maximum decontamination efficiency relative to the previous challenge test. Although the data gave an insight into what real contamination was found in the input material before decontamination and actual decontamination levels achieved. Irrespective of the input contamination levels a baseline decontamination was always achieved.

The following evaluation criteria are proposed for the Authority's future assessment of recycling processes applying this novel technology: (Article 10.3.f)

1. Input Material Characterization:
 - a. Detailed analysis of the post-consumer HDPE input, including:
 - b. Polymer composition (minimum 98.5% HDPE), plastics other than HDPE, Glue
 - c. Presence and quantification of potential migrants
 - d. Contamination levels (maximum 1% non-food contact materials for a defined blend rate with virgin material)
 - e. Composition and bulk density of the flake (range - average)
 - f. Flake dimensions, such as range and average of thickness, size distribution
2. Process Parameters Monitoring:
 - a. Continuous monitoring and recording of critical process parameters: a) Washing temperature and detergent concentration
 - b. Drying temperature and duration
 - c. Extrusion temperature profile
 - d. Vacuum levels in decontamination reactors
 - e. Residence time in each critical step of the process
3. Decontamination Efficiency:
 - a. Assessment of decontamination efficiency using challenge tests:
 - i. Challenge tests which are based on a standard methodology or matching previous challenge tests by the developer
 - ii. Requirement for the decontamination efficiency to match or be better than previous results
 - b. Regular assessment of decontamination efficiency by monitoring each batch
 - i. Using compounds consistently identified in the input e.g. limonene
 - ii. Day to day input material may show lower calculated decontamination efficiencies than the challenge test when the input quality is good because the Erema is not challenged. The focus should be on a consistent output level
 - iii. A large dataset approximately 100 results or more are needed to spot outliers

- iv. Accelerated migration results for input and output can be used to calculate decontamination efficiency. This is because most compounds identified in THF extractions are also identified in 50% migration results quantitatively
- 4. Output Quality Control:
 - a. Characterisation of the output HDPE, output including:
 - i. Melt flow index
 - ii. Colour
 - iii. Density
 - iv. Visual appearance
 - v. Form e.g. flakes, pellets, sheets
- 5. Migration Testing:
 - a. Compliance with EU Regulation 10/2011 on plastic materials and articles intended to come into contact with food
 - b. Specific migration limits (SML) for identified substances on each batch initially
 - c. Overall migration limit (OML) testing
- 6. Non-intentionally Added Substances (NIAS) Evaluation:
 - a. Screening for NIAS using advanced analytical extraction techniques (THF/GC-MS or ethanol 95% LC-MS)
 - b. Risk assessment of identified NIAS and likely sources of contamination
- 7. Instruction and labelling for reprocessors
 - a. Specify safe blend rate with virgin, intended application
- 8. Traceability System:
 - a. Implementation of a robust traceability system compliant with EU Regulation 1935/2004
 - b. Ability to trace each batch of recycled material back to its input source
- 9. Quality Management System:
 - a. Implementation of a comprehensive quality management system
 - b. Regular internal audits and third-party certifications
- 10. Long-term Performance Monitoring:
 - a. Ongoing monitoring of recycled material performance in final applications
 - b. Collection and analysis of data from end-users (e.g., dairy industry)
- 11. Continuous Improvement Plan:
 - a. Unknown compound retention times or mass fragments and concentrations should be trended to identify frequently occurring unknowns as they can significantly affect the blend rate.
- 12. Completion of a CMSS (ANNEX II) template provided by the developer highlighting critical control points, quality assessment stages and corrective actions.

6. References

- a. 08A11J0703 Development of an accelerated migration test for recycled rHDPE (Pira International Research) September 2008
- b. 08A11J0798 Studies on Recycled HDPE used for milk packaging (Pira International Research) November 2008
- c. <https://efsa.onlinelibrary.wiley.com/doi/epdf/10.2903/j.efsa.2015.4016>

7. Amendments log

- a. Initial report



8. Appendices

- a. Appendix 1: Quantitative migration of contaminants in recycled HDPE by GC-FID and GC-MS
- b. Appendix 2: Quantitative Determination of Limonene Concentration in HDPE by Headspace GC-MS
- c. Appendix 3: Analysis of Non-Intentionally Added Substances (NIAS) in Recycled HDPE

Abbreviations:

ASTM: American Society for Testing and Materials

AVG: Average

CAS: Chemical Abstracts Service

CIR: Circular Economy Certification

CMSS: Compliance Monitoring Summary Sheet

CONC: Concentration

EC: European Commission

EFSA: European Food Safety Authority

EN: European Standard

EU: European Union

FCM: Food Contact Material

FDA: Food and Drug Administration

FID: Flame Ionization Detector

GC-MS: Gas Chromatography-Mass Spectrometry

HDPE: High-Density Polyethylene

ID: Identification

ISO: International Organization for Standardization

LAB: Laboratory

MAX: Maximum

MIN: Minimum

NIAS: Non-Intentionally Added Substances

NOL: No Objection Letter

OML: Overall Migration Limit

PCW: Post-Consumer Waste

ppm: Parts Per Million

PRE: Plastics Recyclers Europe

QUAL: Quality

QC: Quality Control

REC: Record (as in REC-001-01, REC-002-01, etc.)

rHDPE: Recycled High-Density Polyethylene

RP: Recycled Plastics

RT: Retention Time

SML: Specific Migration Limit

TBC: To Be Confirmed

THF: Tetrahydrofuran

WRAP: Waste and Resources Action Programme

Appendix 1: Quantitative migration of contaminants in recycled HDPE by GC-FID and GC-MS

➤ Method

Procedure:

1. Prepare 50% ethanol solution (simulant D1) as the food simulant.
2. Use rHDPE specimens with a total surface area of approximately 0.42dm² per 35mL of simulant.
3. Expose rHDPE samples to simulant D1 at 55 ± 2°C for a minimum of 4 hours.
4. Cool samples for at least 30 minutes.
5. Add 100 ± 1µL Internal Standard Solution to test vials (except blank and NOIS).
6. Add 2 ± 0.1mL heptane to each vial.
7. Shake vigorously and allow to settle for 10 minutes.
8. Extract ≥1mL of heptane layer into GC vials.

GC-FID Analysis:

- Column: Zebtron ZB-5HT 30m x 530µm x 0.15µm
- Oven: 70°C (5 min) → 20°C/min to 320°C (4.5 min hold)
- Injector: Cool on-column, 2µL injection
- Detector: FID at 340°C

GC-MS Analysis (for unknown identification):

- Column: Rxi-5ms 30m x 250µm x 0.25µm
- Oven: 70°C (5 min) → 20°C/min to 320°C (9.5 min hold)
- Injector: Splitless, 300°C, 5µL injection
- MS parameters: Transfer line 320°C, Source 230°C, Quad 150°C, Mass range 12-700 m/z

Internal Standards:

- Pentadecane (PD)
- Bis(1-butylpentyl) adipate (DNA)
- Glyceryl trioctanoate (GT)

Calibration Substances:

1. Limonene (LIM)
2. 1-Dodecene (DOD)
3. Hexyl salicylate (HS)
4. α-Hexylcinnamaldehyde (HCA)
5. 2-Ethylhexyl benzoate (EHB)
6. Isopropyl myristate (IPM)
7. Isopropyl palmitate (IPP)
8. Dibutyl Sebacate (DBS)
9. Irgafos168 (FOS)
10. Irganox1076 (NOX)
11. Oleic acid (OA)
12. Palmitic acid (PA)

Surface Area to Volume Ratio:

- The test uses 0.42dm² of rHDPE surface area per 35mL (0.035 kg) of simulant.
- This results in a ratio of 0.42 dm² / 0.035 kg = 12 dm²/kg



- Results are reported normalized to the EU conventional food packaging ratio of 6dm²/kg.

Simulant:

- Simulant D1 (50% ethanol) is used as per EU Regulation 10/2011 for dairy products.

Calibration Range:

The calibration range is established using four levels of working calibration solutions:

Cal. Level 1: 250 ± 10µL of Cal. Soln. in 25 ± 0.5mL of 50% (v/v) ethanol

1. Concentration: Approximately 10 ppb (0.01 mg/kg)

Cal. Level 2: 500 ± 10µL of Cal. Soln. in 25 ± 0.5mL of 50% (v/v) ethanol

2. Concentration: Approximately 20 ppb (0.02 mg/kg)

Cal. Level 3: 750 ± 10µL of Cal. Soln. in 25 ± 0.5mL of 50% (v/v) ethanol

3. Concentration: Approximately 30 ppb (0.03 mg/kg)

Cal. Level 4: 1000 ± 10µL of Cal. Soln. in 25 ± 0.5mL of 50% (v/v) ethanol

4. Concentration: Approximately 40 ppb (0.04 mg/kg)

Quality Control (QC) and Limit of Detection (LOD):

1. GC Calibration Performance Criteria:

- Correlation coefficient: ≥0.99 for all calibration curves
- Calibrated limit of detection of dodecane: ≤10ppb
- 1-Dodecene in QC: Detected
- Limonene in QC: 30 ppb
- Dodecene symmetry in Cal. Level 4 (FID only) or Pentadecane symmetry: 0.8-1

2. QC Sample:

- Prepared with Limonene (30 ppb), 1-Dodecene
- Used to verify method performance

3. Limit of Detection (LOD):

- Calculated by regression analysis of the calibration curve
- Standard error in predicted concentration divided by gradient and multiplied by 3.3

4. Uncalibrated unknown substances:

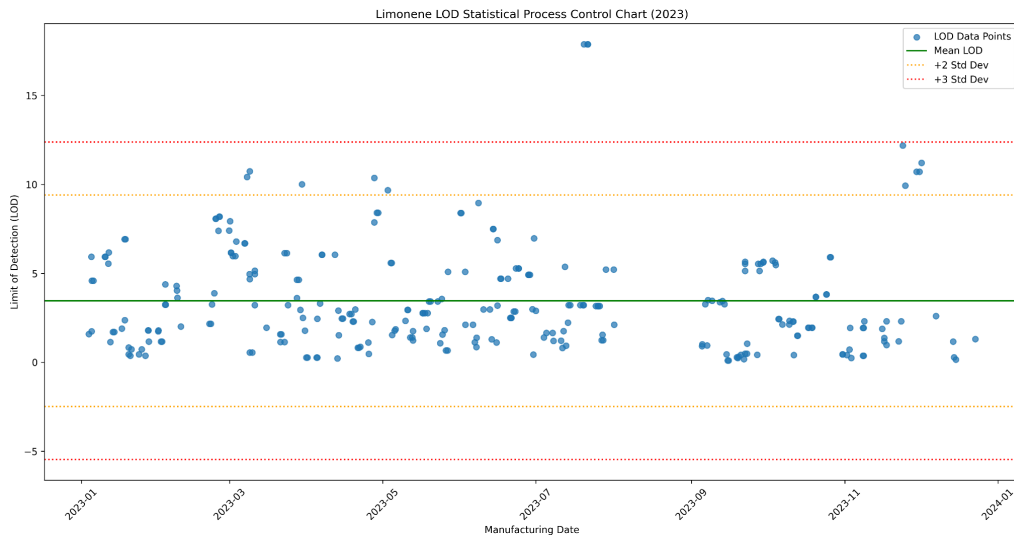
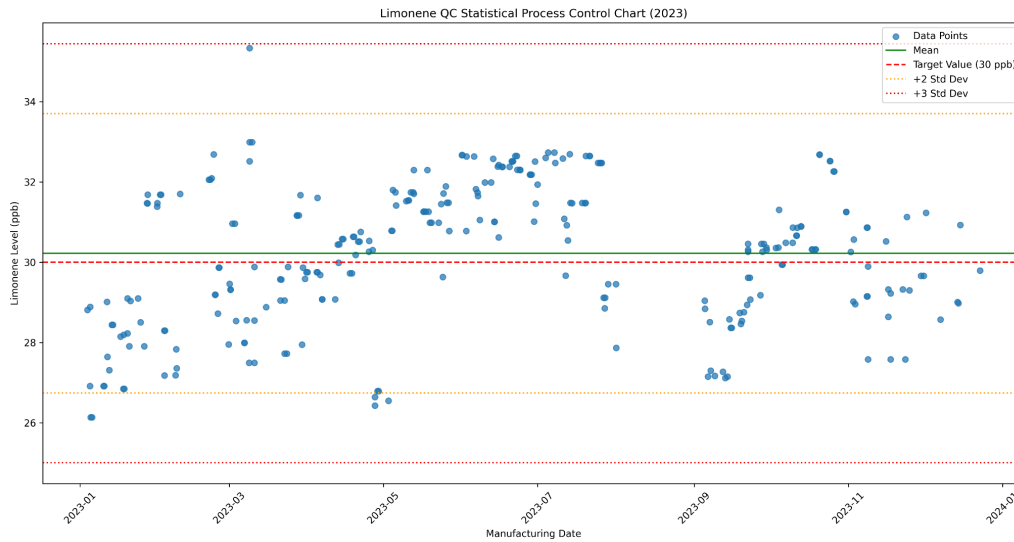
- Specific migration limit of 0.01 mg/kg applied (as per EU Reg 10/2011)
- GC-MS used for identification of unknown peaks

Calculations:

- Migration (mg/kg) calculated based on GC results and sample surface area
- Results normalized from 12 dm²/kg to 6 dm²/kg for reporting
- Maximum blend rate (%) calculated for each substance based on migration limits

➤ **Method validation and QC performance**

Number of data points: 280, Mean: 30.22 ppb, Standard Deviation: 1.74 ppb, RSD%: 5.75%, Bias from target value (30 ppb): 0.75%, Uncertainty of Measurement: 5.80%



LOD by Standard Error of the Regression

Number of LOD data points: 279, Average LOD: 3.4568 ppb ,LOD: 1.7284 ppb for 50%,

For 40% blend the LOD = 1.38272 ppb, LOQ = 2.8 ppb

The assumption in the analysis is that all substances will give a similar response with the flame ionisation detector (FID) and that the substances are amenable to extraction with heptane and GC analysis. Substances less amenable to heptane extraction and GC analysis will be polar and ionic substances that may be present in the feedstock, but will be easily removed in the washing and cleaning stages and will not therefore be expected to be present in the cleaned HDPE.

The FID responses are similar for the substances used as internal markers and this is expected to hold true also for most organics that will have an affinity for, and are compatible with HDPE, although highly halogenated substances will have a lower response.

The GC-FID analysis was conducted using cold on-column injection. This provides a more sensitive analysis than the traditional splitless technique and extends the range of substances covered up to a molecular weight of around 1000 Daltons.

This method allows for quantitative evaluation of substance migration from rHDPE, with results used to determine safe blend rates with virgin HDPE for food contact applications.

The use of simulant D1, the specific surface area to volume ratio, and the normalization of results to 6 dm²/kg ensure compliance with EU regulations. The QC measures and LOD calculations ensure the reliability and sensitivity of the analysis. The use of GC-MS for unknown identification provides additional confidence in the comprehensive assessment of potential migrants.

The calibration range covers from the lowest concentration of interest (10 ppb, which aligns with the general migration limit for non-listed substances in EU Reg 10/2011) up to 40 ppb, allowing for accurate quantification within this range. This ensures that the method can accurately quantify substances from trace levels up to concentrations that approach or exceed regulatory limits, providing a robust analytical approach for assessing migration from rHDPE materials.

Appendix 2: Quantitative Determination of Limonene Concentration in HDPE by Headspace GC-MS

> Method

Purpose:

To determine the concentration of limonene evolving from HDPE by headspace analysis using GC-MS.

Scope:

Primarily for FG rHDPE pellets, but applicable to other forms of HDPE such as flakes or purge.

Principle:

A 1g HDPE sample is heated in a sealed vial to allow gases to escape into the headspace.

The gases are analyzed by GC-MS, with limonene quantified using standard addition and an internal standard.

Equipment:

- Agilent 7890A GC
- Agilent 5975 MS
- Gerstel MPS with headspace autosampler



- MSD Chemstation
- Analytical balance (d 0.1mg)
- Various lab equipment (fridge, oven, desiccator, pipettes, vials, etc.)

Reagents:

- D-Limonene $\geq 99\%$
- Toluene $\geq 99\%$ (Internal Standard)
- Methanol
- Low limonene HDPE pellets (< 1 ppm)
- Positive control HDPE pellets

Procedure:

1. Sample Preparation:
 - Weigh 1 ± 0.02 g of sample into a 20 mL headspace vial
 - Spike with 10 ± 0.5 μL of 0 ppm calibration solution (internal standard)
 - Seal with magnetic cap and PTFE/silicone septum
2. GC-MS Analysis:
 - Place sample in autosampler tray
 - Set up sequence in MSD Chemstation
 - Run analysis (approximately 2 hours per sample)
3. Calibration:
 - Prepare calibration solutions (0, 0.5, 1, 2, 3 ppm limonene)
 - Prepare calibration samples using low limonene HDPE
 - Run calibration samples weekly or after Atune
4. QC Samples:
 - Run daily QC samples (QC Sample and 0.5 PPM)

GC-MS Parameters:

- Column: Rxi-5ms 30 m x 250 μm x 0.25 μm
- Inlet: Split, 250°C
- Oven: 40°C (2 min) \rightarrow 20°C/min to 300°C (5 min hold)
- MS: Transfer line 320°C, Source 230°C, Quad 150°C, Mass range 30-550 m/z
- Headspace: Incubator 120°C for 90 min, 2500 μL injection

Calculations:

- Limonene concentration determined by ratio of limonene to toluene response
- Calibration curve used to convert ratio to ppm
- Matrix effect factored in using x-intercept of calibration curve

Quality Control:

- Daily QC samples
- Weekly calibration
- Atune of GC-MS system before calibration

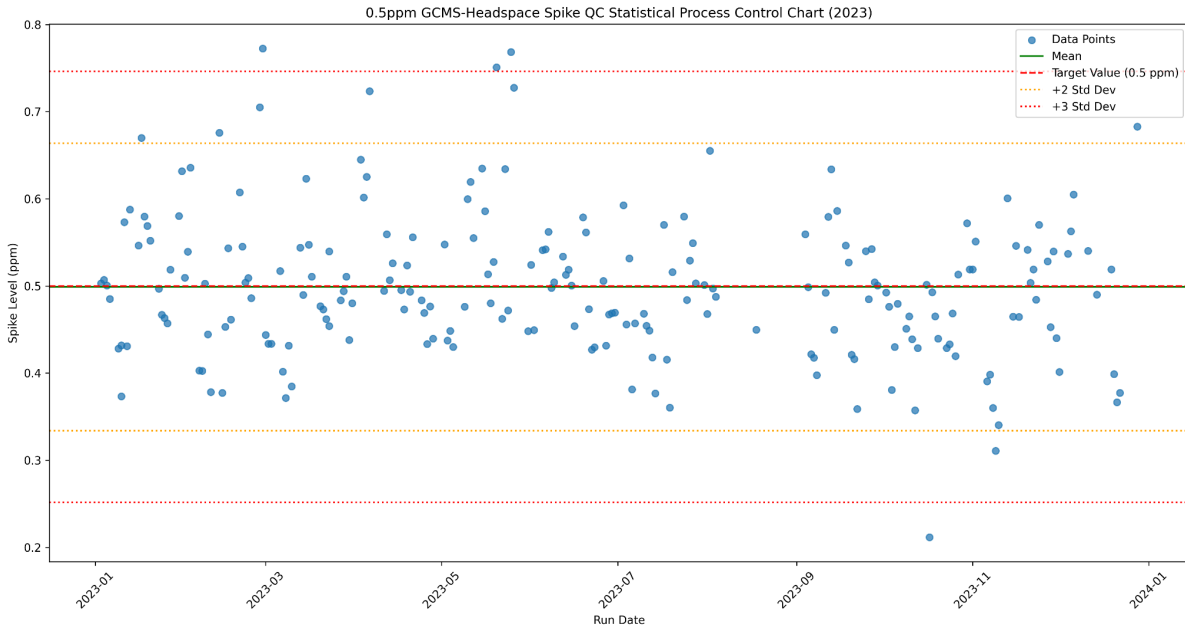
This method allows for the quantitative determination of limonene in HDPE samples, with a focus on recycled materials. The use of headspace analysis, internal standards, and matrix-matched calibration ensures accurate results for this volatile compound. The detailed QC measures and frequent system checks maintain the reliability of the analysis.

This method allows for the comprehensive analysis of decontamination performance for volatile compounds. It is quantitative and quicker than the migration testing so more frequent measurements can be made. The decontamination process can be monitored much closer to real-time. Also comparing input (flakes) and output (pellets) gives an ongoing assessment

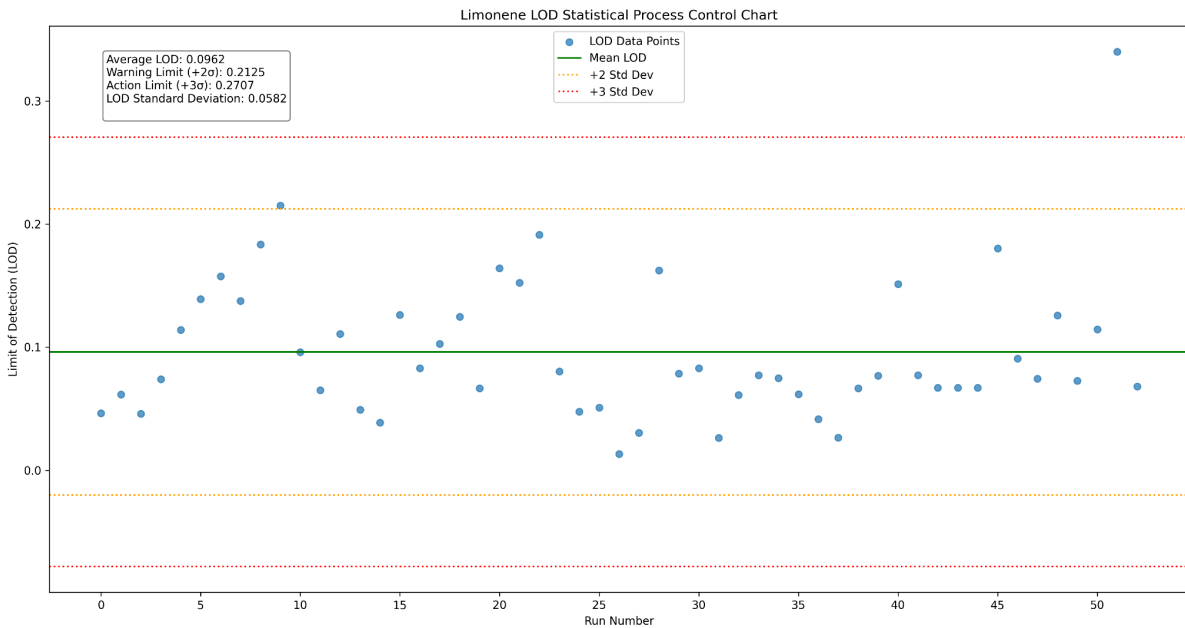
of the decontamination efficiency of the Erema process. The use of extraction, GC-MS analysis, and internal standard quantification provides a robust approach to identifying and quantifying potential contaminants in the recycled material.

➤ **Method validation and QC performance**

Number of data points: 223, Mean: 0.4989 ppm, Standard Deviation: 0.0825 ppm, RSD%: 16.53%, Bias from target value (0.5 ppm): -0.23%, Uncertainty of Measurement: 16.53%



LOD by Standard Error of the Regression, Number of LOD data points: 53, Average LOD: 0.0962 ppm, LOQ = 0.30 ppm



Appendix 3: Analysis of Non-Intentionally Added Substances (NIAS) in Recycled HDPE

➤ Method

Purpose:

To analyze Non-Intentionally Added Substances (NIAS) in recycled HDPE material before and after the extrusion process, evaluating the decontamination efficiency of the Erema process. This method is based on principles outlined in ASTM D7210-21.

Scope:

Applicable to rHDPE washed flake (input) and rHDPE pellets (output) materials.

Principle:

1. Extraction of material by heating with THF under reflux.
2. Analysis of extracts using GC-MS with injection standard quantification.

Equipment:

- Agilent 7890A GC
- Agilent 5975 MS
- Multi-extraction heating mantle with magnetic stirrer
- Gerstel MPS with headspace autosampler
- MSD Chemstation
- Various lab equipment (analytical balance, fridge, oven, glassware, etc.)

Reagents:

- Tetrahydrofuran (THF) Spectroscopic grade
- Internal Standard: Bis(1-butylpentyl) adipate (DNA)

Internal Standard Preparation:

1. Stock Solution:
 - Weigh 0.175 g of DNA into a 100 mL volumetric flask
 - Fill to the mark with THF
 - Final concentration: 1750 ppm (1.75 mg/mL)
2. Intermediate Solution:
 - Pipette 1 mL of stock solution into a 100 mL volumetric flask
 - Fill to the mark with THF
 - Final concentration: 17.5 ppm (17.5 µg/mL)

Procedure:

1. Sample Collection:
 - Collect Erema feed samples (wash flake) from all three Eremas once per shift
 - Collect corresponding output pellet samples 3 hours later
2. Extraction:
 - Weigh 2.5 ± 0.1 g of sample into a 250 mL round bottom flask
 - Add 25 ± 0.5 mL of THF
 - Reflux for minimum 1 hour at THF's boiling point (approximately 66°C)
 - Cool the extract to room temperature
 - Collect extract in 40 mL amber vial
3. GC-MS Analysis:

- Take 700 µL of cooled extract
- Add 300 µL internal standard intermediate solution (17.5 ppm)
- Final concentration of internal standard in the injection mixture: 5.25 ppm
- Total volume of injection mixture: 1 mL
- Run on GC2 using "NIAS2023 Ver1" method
- Perform extractions and analyses for both flakes and pellets

GC-MS Parameters:

- Column: Rxi-5ms 30m x 250µm x 0.25µm
- Inlet: Split, 250°C
- Oven: 40°C (2 min) → 20°C/min to 300°C (5 min hold)
- MS: Transfer line 320°C, Source 230°C, Quad 150°C, Mass range 30-550 m/z

Quality Control:

- Include blank samples treated in the same manner, excluding material
- Use well-known rHDPE as a control sample
- Prepare and analyze QC samples (details to be specified)

Data Analysis and Evaluation:

- Store all relevant data on appropriate results spreadsheet
- Retain all GC data indefinitely for reference
- Use DNA internal standard for quantification and to account for variations in extraction efficiency and instrument response
- Calculation:

The concentration of NIAS in the sample is calculated using the following formula:

$$C_NIAS = (A_NIAS / A_DNA) * (C_DNA_final / F_dilution) * (V_THF / W_sample) * RF$$

- Where:
- C_NIAS = Concentration of NIAS in the sample (µg/g)
- A_NIAS = Peak area of NIAS
- A_DNA = Peak area of DNA (internal standard)
- C_DNA_final = Final concentration of DNA in the injection mixture (5.25 µg/mL)
- F_dilution = Dilution factor of the extract (0.7 mL extract / 1 mL total = 0.7)
- V_THF = Volume of THF used for extraction (25 mL)
- W_sample = Weight of the sample (2.5 g)

RF = Response factor (assumed to be 1 if not determined experimentally)

- Example calculation:

If A_NIAS = 50000, A_DNA = 100000, and assuming RF = 1:

- $C_NIAS = (50000 / 100000) * (5.25 \mu\text{g/mL} / 0.7) * (25 \text{ mL} / 2.5 \text{ g}) * 1$
- $= 0.5 * 7.5 * 10 \mu\text{g/g}$
- $= 37.5 \mu\text{g/g}$

- Conversion Factor:

- To convert between concentration in analyzed solution (µg/mL) and concentration in polymer (µg/g):

Conversion factor = 10 (V_THF / W_sample = 25 mL / 2.5 g)

- To convert from solution to polymer: Multiply by 10
- To convert from polymer to solution: Divide by 10

Reporting:

- Report NIAS concentrations in µg/g (mg/kg) relative to the polymer sample.

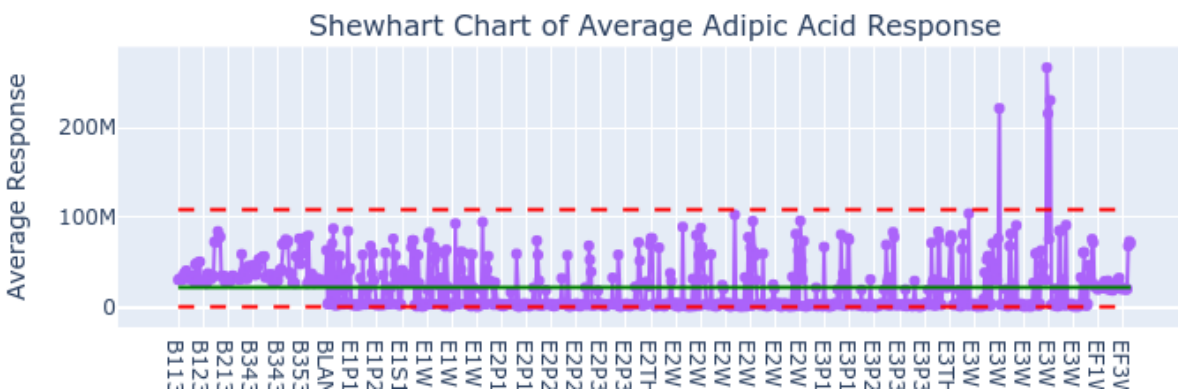
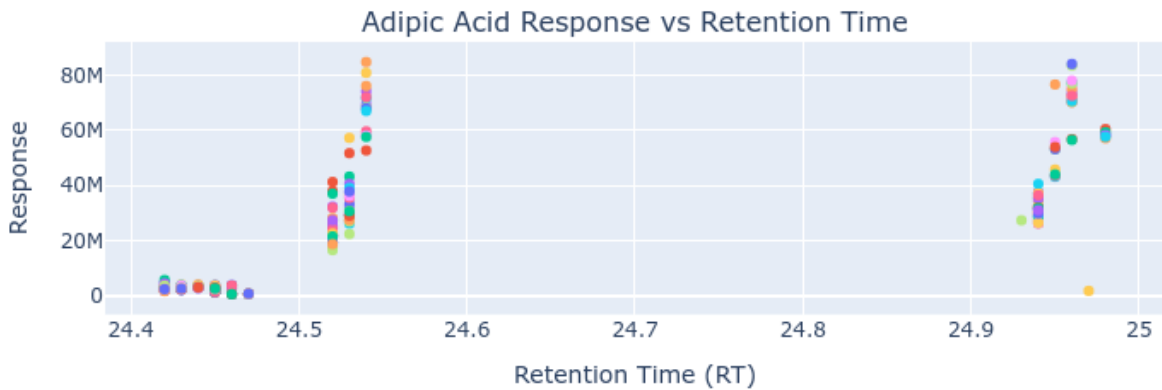
- The internal standard concentration is 52.5 µg/g in the polymer sample, corresponding to 5.25 ppm in the injection solution.

➤ **Method validation and QC performance**

This is a semi-quantitative method that has been implemented according to the ASTM D7210-21 and the original validation data.

Integration settings with a low peak threshold were used to integrate as many low peaks as possible .

Ongoing monitoring of the performance is done by statistically monitoring of the response of the DNA internal standard for variation and checking the cleanliness of the blanks making sure any peaks over 1.5ppm are noted and subtracted from the samples



Overall mean response: 21781957.68
 Upper Control Limit (UCL): 108628873.58
 Lower Control Limit (LCL): 0.00

843 samples



Current External Proficiency scores with can be found on the website
<https://dagenhamplastics.veolia.co.uk/>