

RESEARCH REPORT

Solvent Migration Tests for Resin Sample

Prepared for:

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For the attention of:

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Executive Summary

Migration tests were set up for resin sample using 50% v/v ethanol as a cosmetic simulant and test conditions of 10 days at 60°C (representing unlimited ambient storage). The migration of post-consumer contaminants into the 50% ethanol was estimated using GC-FID and the compounds identified using GC/MS.

Four compounds were found in the regulations.

A review of identified compounds were checked against the Unilever set of in-house limits and a few compounds were found in the regulations but with the level below 100ppb.

No further risk assessment is required.

Test material:

Table 1. Pellets

Smithers Sample Code	Client Sample Description	Date received
22S1891	100g Q4 PP5 Refreshed pellets (PP5-FDAMF20 Recycled Polypropylene (Q4 2021 Batch))	26/08/2022

Sampled and supplied by: Berry Global (Plasgram Limited)

Date(s) of testing: 15th September to 9th October 2022

Test(s) required: Estimation of the migration of PCW contaminants

Migration test set up in triplicate using 50% ethanol (v/v) as simulant and test conditions of 10 days at 60°C (representing unlimited ambient storage).

GC/FID to estimate levels of post-consumer contaminants.

Major peaks observed by GC/FID and identified using GC/MS.

Smithers understands that Unilever will use the results in this report to conclude safety in use for the products tested and will use the data in this report to assist with this determination and identify any substances which may need further safety consideration.

Method:

Migration test was set up in triplicate. For each replicate, 1 g of resin was weighed into a 40 ml glass vial and 25 ml of 50% ethanol simulant was added.

For reference, the exposure ratios for each resin sample are summarised in Table 2.

Table 2: Sample Exposure Ratios

Resin sample	Volume Simulant Exposed (ml)	Surface area Exposed (dm ²)*
22S1891	25	0.20

*The surface area exposed was measured by taking an average of the measured surface from ten resin pellets and multiplying this by the number of pellets exposed in 1 g sample.

After incubation, the exposed simulants were transferred into individual 25 ml volumetric flasks. Pentadecane (PD), di-n-nonyl-adipate (DNAD) and tricapylin (TCN) were used as internal markers. A combined internal standard stock was prepared (nominal concentration 1000 ppm). Dilutions of this were prepared and added at a level of approximately 80 ppb. 2 ml heptane was added to each volumetric flask and each flask shaken vigorously. The top layer was then removed and an aliquot of this transferred to individual GC vials.

The heptane extracts were injected on the GC/MS to identify major peaks. The following conditions were used for GC/MS analysis:

Gas Chromatograph	Agilent 7890A (MSD5)
Injection mode	Splitless
Injector temperature	300°C
Column head pressure	Constant Flow
Column	Restek Rxi® - 5 Sil MS, 30m x 0.25mm x 0.25µm
Temperature program	40 °C hold 2 minutes, 15°C/minute to 320°C hold 20 minutes
Mass spectrometer	Agilent 5975C
Detector mode	Scan
	Monitoring m/z 35-750

Samples were injected on the GC/FID using the conditions below, to semi-quantify peaks identified in the provided regulations.

Gas Chromatograph	Agilent FID
Injection mode	Splitless
Injector temperature	300°C
Column head pressure	Constant flow 1.9 ml/min
Column	Restek Rxi® - 5 Sil MS, 30m x 0.25 mm x 0.25 µm

Table 3: Compounds Identified in 22S1891 by GC/MS

Retention Time (min)		Compound Identification	CAS Number	Compound NIST 20 Library match (%)
GC/MS	GC/FID			
13.45	13.35	Dodecanoic acid, ethyl ester	106-33-2	91
14.73	14.61	Octanal, 2-(phenylmethylene)-	101-86-0	95
14.90	14.82	Tetradecanoic acid, ethyl ester	124-06-1	91
15.12	15.04	Isopropyl myristate	110-27-0	87
15.47	15.36	Didodecyl phthalate	2432-90-8	87
16.10	15.98	Dibutyl phthalate	84-74-2	72
16.28	16.16	Hexadecanoic acid, ethyl ester	628-97-7	98
16.35	16.23	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, ethyl ester	36294-24-3	98
17.37	17.25	E-11-Hexadecenoic acid, ethyl ester	Not available	90
17.52	17.40	Octadecanoic acid, ethyl ester	111-61-5	98
18.64	18.40	Hexanedioic acid, bis(2-ethylhexyl) ester	103-23-1	91
19.46	19.32	Bis(2-ethylhexyl) phthalate	117-81-7	90
19.71	19.55	Bumetrizole	3896-11-5	99
20.68	20.55	9-Octadecenamide, (Z)-	301-02-0	95
26.17	26.05	Tris(2,4-di-tert-butylphenyl) phosphate	95906-11-9	99

Table 4: Estimated Levels of Compounds Identified in Regulations, sample 22S1891

Retention Time (min)		Compound Identification	CAS Number	Mean concentration In ppb
GC/MS	GC/FID			
14.73	14.608	Octanal, 2-(phenylmethylene)-	101-86-0	60
15.47	15.355	Didodecyl phthalate	2432-90-8	<10
16.10	15.98	Dibutyl phthalate	84-74-2	10
19.46	19.34	Bis(2-ethylhexyl) phthalate	117-81-7	30

A targeted screen for phthalates using m/z 149 was carried out. Three phthalate compounds were previously identified from the screen in the sample. These were Didodecyl phthalate (CAS 2432-90-8), Dibutyl phthalate (CAS 84-74-2) and bis(2-ethylhexyl) phthalate (CAS 117-81-7).

A targeted screen was also carried out for poly aromatic hydrocarbons (PAHs) since these can be potential carcinogens. Characteristic ions m/z 178, 166 and 202 were used in the targeted screen. The traces (<10ppb) were found for anthracene (CAS 120-12-7).

Conclusion

Migration tests were carried out using 50% ethanol (v/v) as a simulant. 50% ethanol (v/v) is recognized as a food simulant in Regulation (EU) No. 10/2011 for fat in water emulsions (e.g. cream). 10 days at 60°C is for long term, ambient storage, a test condition specified in Regulation (EU) No. 10/2011 as amended.

Compounds listed in Table 3 were checked for compliance with the Unilever set of in-house limits. These limits are listed in the tables of the Appendices A and B.

Only compounds listed on the set of legislation specified by Unilever were compared against the Unilever limits. The specified legislation list was as given below with the web links used:

1. EU. Prohibited Substances: Annex II, Regulation 1223/2009/EC on Cosmetic Products, as amended by Regulation (EU) 2020/1683, 13 November 2020
<https://echa.europa.eu/nl/cosmetics-prohibited-substances>
2. EU. Restricted Substances: Annex III, Regulation 1223/2009/EC on Cosmetic Products, as amended by Regulation (EU) 2020/1683, 13 November 2020
<https://echa.europa.eu/nl/cosmetics-restricted-substances>
3. Table of harmonised entries in Annex VI to CLP:
<https://echa.europa.eu/nl/information-on-chemicals/annex-vi-to-clp>
Skin sensitisers are marked with * in Appendix A.

Tabulated checks are attached in Appendix A. A number of compounds were found in the regulations and they are summarised as follows:

In Annex II (1223/2009), Didodecyl phthalate (CAS 2432-90-8), Dibutyl phthalate (CAS 84-74-2) and Bis (2-ethylhexyl) phthalate were listed with the levels of ~10-30 ppb.

In Annex III (1223/2009), 2-(phenylmethylene)-Octanal is listed with the level of ~60 ppb.

In the table of harmonised entries in Annex VI to CLP, Didodecyl phthalate (CAS 2432-90-8), Dibutyl phthalate (CAS 84-74-2) and Bis (2-ethylhexyl) phthalate were listed with the levels of ~10-30 ppb.

Furthermore, no compounds need further risk assessment.

Analysed by:

18/02/22

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Chronology and changes to previous version

Document version	Summary of changes
1	This is the first version of this document
2	Amendment to typographical error from 4 days to 10 days in the executive summary section.

Appendix A: Compliance Check against Unilever limits for sample 22S1891

Compound Name	Limit in Material			<1ppm		<10ppm		<100ppm (Skin Sens 1a)	
	Limit in Migrant			<100 ppb		<100 ppb		<1000ppm (Skin Sens 1)	
Sample 22S1891	CAS No.	Est Conc.	CAS	In Annex II	Over Limit?	In Annex III	Over Limit?	In Annex VI	Over limit?
		(ppb)	Validation						
Dodecanoic acid, ethyl ester (91)	106-33-2	n/a	y	n	n	n	n	n	n
Octanal, 2-(phenylmethylene)-(95)	101-86-0	60	y	n	n	y	n	n	n
Tetradecanoic acid, ethyl ester (91)	124-06-1	n/a	y	n	n	n	n	n	n
Isopropyl myristate(87)	110-27-0	n/a	y	n	n	n	n	n	n
Didodecyl phthalate	2432-90-8	<10	y	y	n	n	n	y	n
Dibutyl phthalate	84-74-2	10	y	y	n	n	n	y	n
Hexadecanoic acid, ethyl ester (98)	628-97-7	n/a	y	n	n	n	n	n	n
Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, ethyl ester (98)	36294-24-3	n/a	y	n	n	n	n	n	n
E-11-Hexadecenoic acid, ethyl ester (90)	Not available	n/a	n/a	n	n	n	n	n	n
Octadecanoic acid, ethyl ester (98)	111-61-5	n/a	y	n	n	n	n	n	n
Hexanedioic acid, bis(2-ethylhexyl) ester (91)	103-23-1	n/a	y	n	n	n	n	n	n
Bis(2-ethylhexyl) phthalate	117-81-7	30	y	y	n	n	n	y	n
Bumetrizole (99)	3896-11-5.	n/a	y	n	n	n	n	n	n
9-Octadecenamide, (Z)- (95)	301-02-0	n/a	y	n	n	n	n	n	n
Tris(2,4-di-tert-butylphenyl) phosphate	95906-11-9	n/a	y	n	n	n	n	n	n

Figure 1: GC/FID Chromatograms for Sample 22S1891

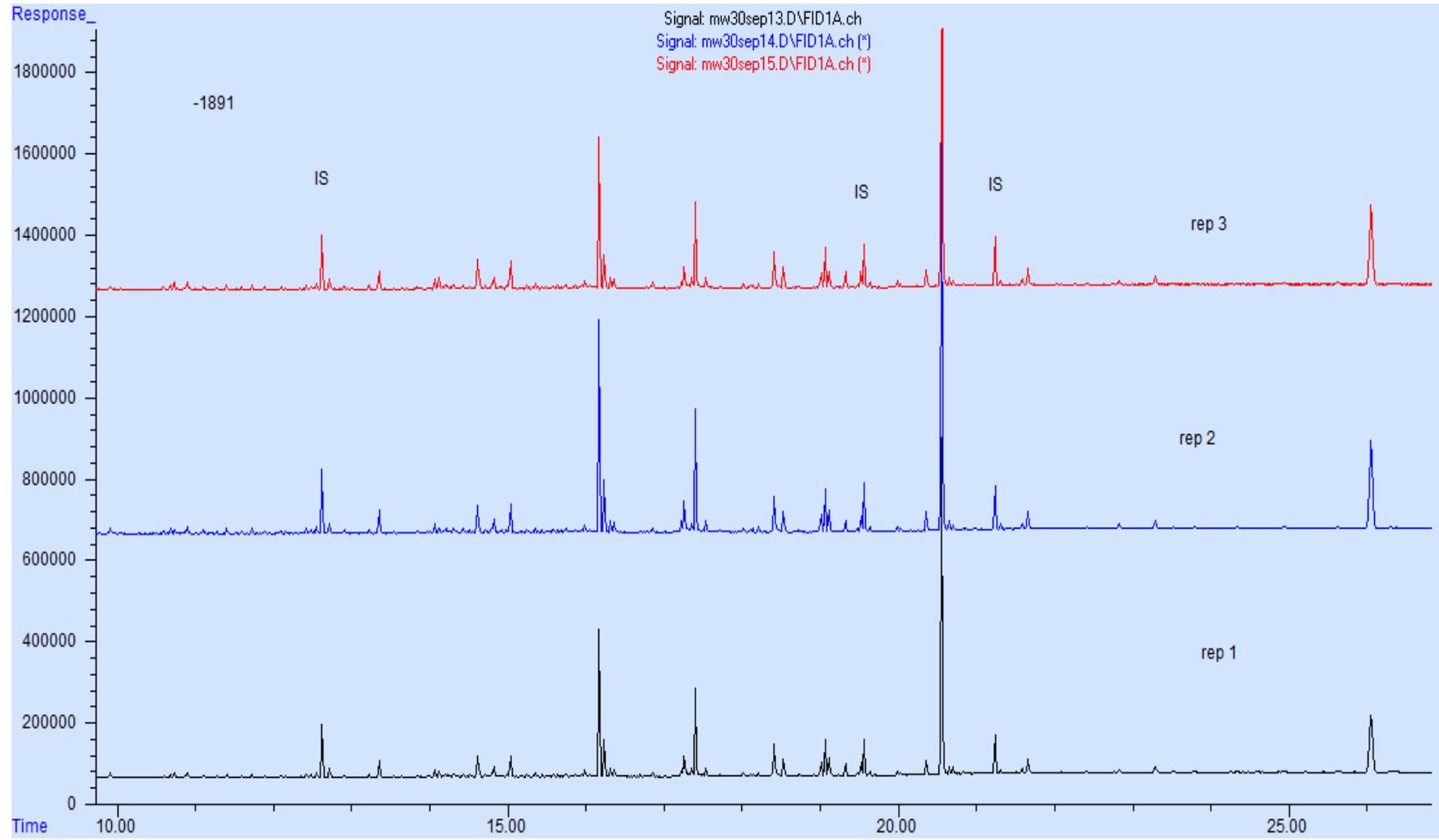


Figure 2: GC/MS Chromatograms for Sample 22S1891

