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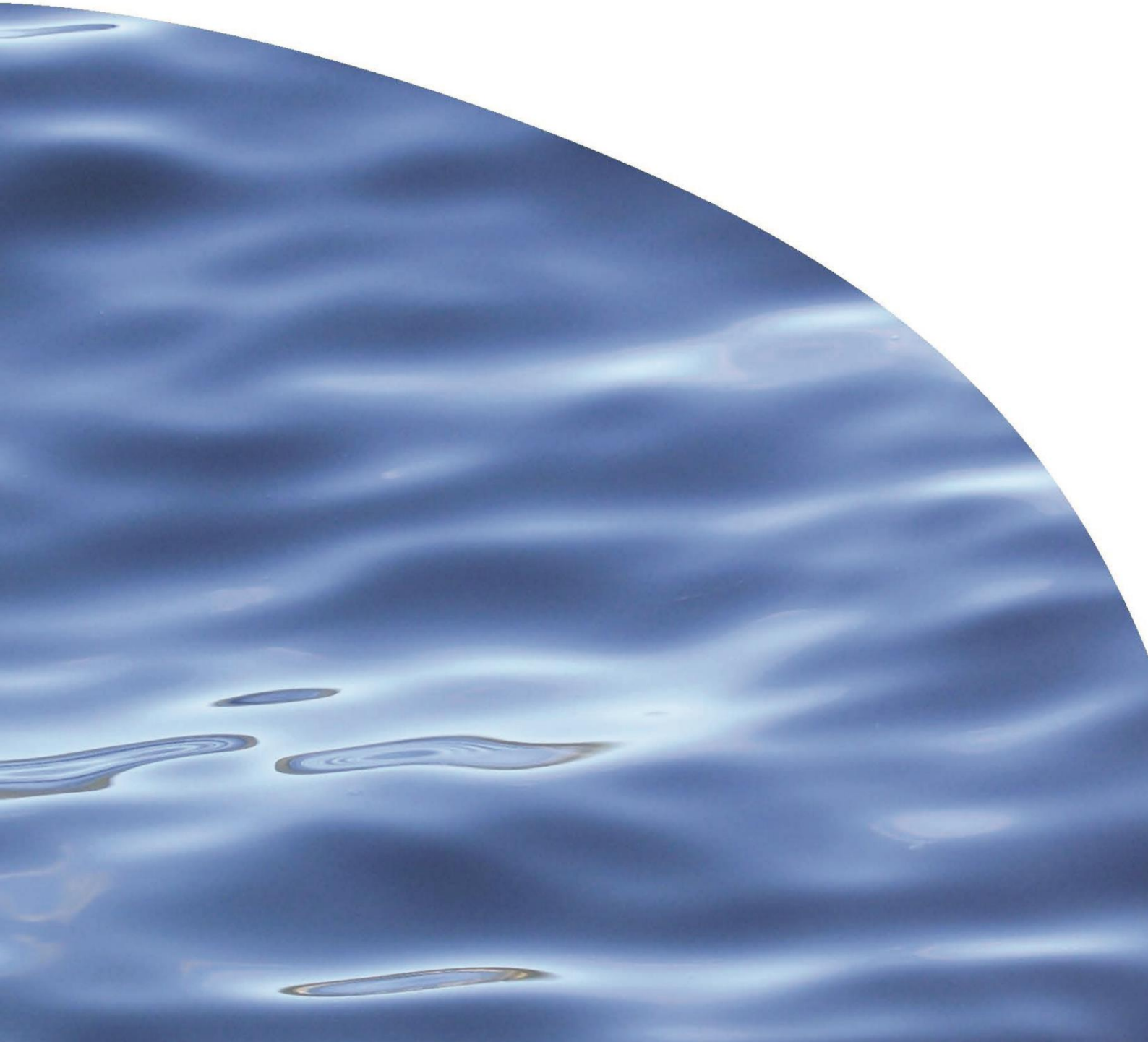

ANALYSIS OF EMERGING ORGANIC CONTAMINANTS IN EFFLUENT OF THE BELL ISLAND WASTEWATER TREATMENT PLANT

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GLOSSARY

EOCs	Emerging Organic Contaminants
WWTP	Waste water treatment plant
NRSBU	Nelson Regional Sewerage Business Unit
SPE	Solid phase extraction
GCMS	Gas chromatography mass-spectrometry
MSTFA	N-methyl-N-(trimethylsilyl)trifluoroacetamide
MTBSTFA	N-tert-butyldimethyl- silyl-N-methyltrifluoroacetamide
TBDMSCl	t-butyldimethylsilyl chloride
QA	Quality assurance
ppt	Part per trillion
MDL	Method detection limits
PNEC	Predicted no-effect concentration
ADF	Average daily flow
NOEC	No observable-effect concentration
NC	Negligible concentration
TCPP	Tris(1-chloro-2-propyl)phosphate
TDCP	Tris[2-chloro-1-(chloromethyl)ethyl]phosphate
TPP	Triphenylphosphate
TBEP	Tris(2-butoxyethyl)phosphate
TNP	Technical nonylphenol
BPA	Bisphenol A

1. INTRODUCTION

Emerging Organic Contaminants (EOCs) have been defined as synthetic or naturally-occurring chemicals or any microorganisms not commonly monitored in the environment, but which have the potential to enter the environment and cause known or suspected adverse ecological and (or) human health effects (Stewart et al. 2016). Municipal wastewater treatment plant (WWTP) effluent is recognised as a major source of EOCs into the environment. The Nelson Regional Sewerage Business Unit (NRSBU) contracted the Cawthron Institute and Northcott Research Consultants Limited (by subcontract) to analyse a suite of EOCs in the effluent from the Bell Island WWTP.

The objectives of this study were to:

- characterise EOCs present in Bell Island WWTP effluent samples
- compare the concentrations of EOCs detected with that from other WWTPs in New Zealand
- identify those EOCs whose concentrations in Bell Island WWTP effluent may pose a risk to the receiving environment.

2. METHODS

2.1. Sample delivery and extraction

Two 4-L samples of Bell Island WWTP effluent (labelled as 1/3/17 1300 hours) were delivered by courier to Northcott Research Consultants at Plant and Food Research Ruakura at 10:30 am on 2 March 2017. On arrival, the samples were acidified (pH = 2.0) by the addition of concentrated sulphuric acid, and filtered through a glass microfiber filter (47 mm, Labservice) topped with diatomaceous earth filter aid medium (Hyflo SuperCel) to remove particulate material. The sample filtrate was collected in pre-cleaned 2-L glass Schott bottle and immediately stored at -20°C to maintain stability prior to being extracted.

Chemicals in the filtered liquid effluent sample (dissolved phase) were extracted by passing through an Oasis HLB 1 g 20 mL solid phase extraction (SPE) cartridge. The acidic pharmaceuticals were eluted from the Oasis SPE cartridge as the first fraction with a mixed solvent of acetone and bicarbonate buffer (pH 10). The SPE cartridge was rinsed with a solution of 20% acetone in purified water and dried under vacuum for 5 min. Chemicals were eluted from the SPE cartridge as a second fraction with a mixed solvent of dichloromethane and methanol solvent. This fraction was purified using florosil adsorption chromatography followed by gel permeation chromatography

to remove the large amount of residual fats and lipids that were present in the sample extracts.

The purified EOC sample extract was split into two equal portions—one for analysis of non-polar semi-volatile EOCs and the other for polar EOCs, the latter requiring chemical derivatisation for analysis by gas chromatography mass-spectrometry (GCMS). The portions of split sample extract were transferred into vials, capped and sealed and stored under refrigeration for analysis.

The raw pharmaceutical solvent extracts were concentrated under a stream of nitrogen gas to remove acetone. The remaining bicarbonate solution was acidified and the pharmaceuticals extracted into diethyl ether.

One half of the EOC sample extract was exchanged into isooctane and internal standards (deuterated polycyclic aromatic compounds) were added. The extract was transferred into GC vials for the analysis of non-polar EOCs (nitro and polycyclic musk fragrances, phthalate esters, alkyl phosphate flame retardants and insect repellents).

2.2. Sample extract derivatisation

A solution of deuterated polar internal standards was added to the other half of the sample extract which was gently blown to dryness. The polar EOCs (steroid hormones, phenolic antimicrobials, paraben preservatives, and industrial alkylphenols) were derivatised to their respective trimethylsilyl ethers using a catalytic mixture of N-Methyl-N-(trimethylsilyl)trifluoroacetamide (MSTFA), ammonium iodide, and mercaptoethanol.

An internal standard mixed solution containing deuterated (-d4) monocarboxylic phthalate acid esters and ibuprofen-d3 was added to the pharmaceutical diethyl ether solvent extracts which were carefully evaporated to dryness. The polar acidic analytes were converted to their respective tertiary-butyl dimethyl silyl esters by reaction with N-tert-Butyldimethyl-silyl-N-methyltrifluoroacetamide (MTBSTFA) with 1% t-Butyldimethylsilyl chloride (TBDMSCl).

2.3. Analysis of EOCs

The analysis of the different classes of EOCs required the use of different GCMS instruments and instrumental analysis methods. Alkyl phosphate flame retardants, musk fragrances, insect repellents, industrial alkylphenols, paraben preservatives, phenolic antimicrobials and steroid hormones were analysed using an Agilent 6890N gas chromatograph coupled to a 5975 mass spectrometer operating in single ion monitoring mode. Quantitation of target EOCs was achieved by internal standard

quantitation using Agilent Chemstation MS software. Phthalate esters, monocaboxylate phthalate esters and pharmaceuticals were analysed using an Agilent 7000 series triple quadrupole GCMS operating in MS/MS mode. Quantitation of target EOCs was achieved by internal standard quantitation using Agilent Mass Hunter MS/MS software.

A total of 80 individual chemicals representing ten different classes of EOCs were analysed. These included:

- alkyl phosphate flame retardants (11 compounds)
- industrial alkylphenols (7 compounds)
- insect repellents (3 compounds)
- nitro- and polycyclic musk fragrances (10 compounds)
- paraben preservatives (5 compounds)
- pharmaceuticals (10 compounds)
- phenolic antimicrobials (5 compounds)
- phthalate esters and plasticisers (13 compounds)
- steroid hormones (16 compounds).

3. RESULTS

3.1. Recovery of surrogate standard compounds

The mean recovery of individual carbon-13 labelled surrogate standards spiked into the sample prior to extraction, and the overall mean recovery of all surrogate compounds is displayed in Table 1. The surrogate standard compounds were spiked into 2 L of prefiltered sample at an equivalent concentration of 20 ng/L (ppt). This represents a low level rate of spiking for quality assurance (QA) determinations.

Table 1. Recovery of surrogate standards for available analytes spiked into the Bell Island WWTP effluent sample.

Recovery compound	Calculated mean percentage recovery
^{13}C-methylparaben	79.4
^{13}C-ortho-phenylphenol	81.2
^{13}C-butylparaben	72.3
^{13}C-methyltriclosan	82.5
^{13}C-triclosan	96.6
^{13}C-bisphenol-A	93.2
^{13}C-estrone	88.3
^{13}C-17β-estradiol	82.5
^{13}C-17α-ethynylestradiol	86.8
Mean recovery	**84.8**

The level of surrogate standard recovery meets the acceptance requirements of quality assurance criteria (> 70% for all ^{13}C-labelled surrogates). The level of surrogate compound recovery obtained from the samples spiked at the low concentration of 20 ppt validated the performance of the analytical methodology.

3.2. Residues of EOCs

The concentration of EOCs detected in the Bell Island WWTP effluent sample are summarised in Table 2. All of the analysed EOCs together with their respective method detection limits (MDLs) are listed in Appendix 1. A total of 23 of the 80 individual EOCs analysed were detected in the effluent from Bell Island WWTP and comprised:

- 5 alkyl phosphate flame retardant
- the phenolic antimicrobial chemical triclosan
- the paraben preservative ethylparaben
- the industrial mixture of nonylphenols
- the insect repellent DEET
- the polycyclic musk fragrance galaxolide
- 6 acidic pharmaceuticals
- 7 plasticisers.

Table 2. Concentration of EOCs detected in Bell Island WWTP effluent sample along with currently predicted no-effect concentrations (PNECs) available from the international literature.

Emerging Organic Contaminant	Concentration (ng/L)	PNEC (ng/L)
Alkyl phosphate flame retardants		
Tributyl phosphate	643	370,000,000
Tris(1-chloro-2-propyl)phosphate	1884	64,000
Tris[2-chloro-1-(chloromethyl)ethyl]phosphate	148	1,000
Tri-phenyl phosphate	2.71	74
Tris(2-butoxyethyl)phosphate	256	Not available
Phenolic anti-microbials		
Triclosan	8.90	100
Paraben preservatives		
Ethylparaben	141	800
Industrial alkylphenols		
Tech-NP-equivalents	9.10	330
Insect repellents		
DEET	15.3	43,000
Musk fragrances		
Galaxolide	17.5	39,000
Acidic pharmaceuticals		
Acetaminophen	9.14	9,200
Carbamazepine	302	25,000
Diclofenac	19.35	10,000
Ibuprofen	6.05	5,000
Naproxen	158	37,000
Salicylic acid	44.4	11,200
Plasticisers		
Diethyl phthalate	82.7	900,000
Di-n-butyl phthalate	65.6	10,000
Diethylhexyl phthalate	56.0	Not available
Monomethyl-PAE	1.58	Not available
Monobutyl-PAE	5.81	Not available
MonoEH-PAE	25.1	Not available
Bisphenol A	13.4	1500-1600

4. DISCUSSION

4.1. Comparison with other WWTPs in New Zealand

The concentration of EOCs detected in the Bell Island WWTP effluent were generally within the range of concentrations reported in treated effluent discharged from WWTPs in New Zealand. However, EOCs that are typically present but were not detected in the Bell Island WWTP effluent included the disinfectant chloroxylene, preservative methyl-paraben, musk fragrance tonalide, plasticiser diethyl phthalate, the natural estrogenic steroid hormones estrone and 17β-estradiol, and the synthetic estrogenic steroid hormone 17α-ethinylestradiol.

The national survey by Northcott et al. (2013) of EOCs in the influent and effluent of 13 WWTPs is the most comprehensive dataset in New Zealand. The plants selected represented a broad range of treatment technologies, catchment population, balance of domestic to industrial inputs, and geographic distribution throughout New Zealand (Table 3). The concentrations of EOCs in the dissolved phase of effluent from these thirteen WWTPs are compared with the concentrations measured in Bell Island WWTP effluent in Table 4. The concentrations of EOCs measured in the effluent from the thirteen WWTPs are presented as the range of the minimum to maximum measured concentration and the corresponding average concentration (mean).

Table 3. Characteristics of WWTPs included in the 2012 national survey (Northcott et al. 2013).

WWTP	Description	ADF[A] (m^3)	Population	Industrial	Domestic
1	Milli-screened	20,000	55,000	25	75
2	BTF (domestic)	51,000	60,000	50	50
3	Primary sedimentation	2,330	4,000	40	60
4	Primary sedimentation	940	1,900	25	75
5	Primary sedimentation	1300	7000	5	95
6	Sedimentation and UV[B]	1,170	3,330	0	100
7	Sedimentation, activated sludge digestion, UV	45,000	140,000	10	90
8	Sedimentation, BTF, sedimentation	16,000	20,000	20	80
9	Sedimentation, BTF, sedimentation	25,000	48,000	20	80
10	Primary sedimentation	900	4,000	0	100
11	Primary sedimentation, UV	no data	700	0	100
12	Sedimentation, BTF, sedimentation	180,000	360,000	10	90
13	Sedimentation, activated sludge digestion, clarification, UV	300,000	1,000,000	40	60

[A] ADF = average daily flow, [B] UV = UV treatment of final effluent

Table 4. Comparison of the concentration of EOCs detected in Bell Island WWTP effluent with that reported for other New Zealand WWTPs as shown in Table 3.

	Concentration in ng/L (ppt)			
	Min	Max	Mean	Bell Is
Musk fragrance				
Galaxolide	24.4	902	243	17.5[A]
Alkyl phosphate flame retardant				
TBP	26.9	499	128	**643**[B]
TCPP	70.5	1024	321	**1884**
TDCP	1.92	630	222	148[C]
TBEP	N.D.	3441	783	256
TPP	6.10	3277	301	2.71
Insect repellent				
DEET	15.2	1836	220	15.3
Antimicrobial				
Triclosan	4.43	158	38.3	8.90
Paraben preservatives				
Ethyl-paraben	N.D.	39	4.11	**141**
Plasticiser				
Bisphenol-A	N.D	66.9	17.0	13.4

[A] values in green highlight represent those less than the minimum value of the range
[B] values in orange highlight represent those falling within the range of minimum to maximum
[C] values in red highlight represent those exceeding the maximum of the range

The data in Table 4 demonstrate the concentrations of the majority of EOCs in Bell Island WWTP effluent either fall within the range of concentrations or are lower than those measured in effluent samples from other New Zealand WWTPs. However, tris-butyl phosphate, Tris (1-chloro-2-propyl) phosphate and ethyl-paraben in the effluent of Bell Island WWTP exceeded the maximum concentrations found in the national survey and are highlighted in red.

The data indicate that the Bell Island WWTP achieves a level of EOC removal similar to other WWTPs in New Zealand, some of which operating secondary and tertiary wastewater treatment technologies.

4.2. What are the risks of EOCs in the effluent of Bell Island WWTP to the receiving environment?

The risk the residual EOCs in Bell Island WWTP effluent present to the receiving environment has been assessed by comparing the concentrations of the EOCs with available predicted no-effect concentrations (PNECs), an estimate of the

concentration below which exposure to a substance is not expected to cause adverse effects. For those EOCs where a PNEC is not available, the no observable-effect concentration (NOEC) was used. The results from the analyses along with available guideline limits are summarised in Table 5. Some PNECs were derived for freshwater environments that would tend to overestimate risk to marine environments. The data for the pharmaceuticals measured are not included on that table but were at concentrations orders of magnitude lower than their respective PNEC values (Li 2014). The concentrations of phthalate plasticisers were all below the available PNEC values. Diethylhexyl phthalate is the most widely used and its concentration in influent and effluent treatment plant is the highest as reported in a recent review (Deblonde et al. 2011). It should be noted that there is limited reliable data to confirm effects below the water solubility of Diethylhexyl phthalate (Oehlmann et al. 2008). Overall, the results indicate that the risk of EOCs in the Bell Island WWTP effluent can be considered negligible. It should be noted that this is based on only one sampling event.

Table 5. Bell Island concentrations of emerging organic contaminants compared to recommended limits from world-wide agencies. PNEC = predicted no-effect concentration; NOEC = no observed effect concentration. The latter are indicated by *. Order of magnitude: 1 order of magnitude is a 10-fold difference, 2 orders of magnitude is a 100-fold difference, and so forth.

Emerging organic contaminant	Abbreviation	Bell Is concentration (µg/L)	Above/below PNEC/NOEC	Order of magnitude	PNEC or NOEC* (µg/L)	Source
Tri-butylphosphate	TBP	0.643	Below	6	370,000 (algae)	OECD 2002
Tris(1-chloro-2-propyl)phosphate	TCPP	1.88	Below	3	1700 (aquatic ecosystems)	Env Canada 2016
			Below	2-3	640 (inverts) 260 (algae) 64 (fish)	European Union: EU 2008c
Tris[2-chloro-1-(chloromethyl)ethyl]phosphate	TDCP	0.148	Below	1	1.3 (aquatic ecosystems)	Env Canada 2016
				1-2	1 (seawater) 10 mg/L (freshwater)	European Union: EU 2008d
Triphenylphosphate	TPP	0.027	Below	1-2	0.16 (aquatic organisms)	Netherlands Verbruggen 2005
					0.74 (surface waters) 0.074 (marine water)	UK Environment Agency 2009
Tris(2-butoxyethyl)phosphate	TBEP	0.256	Below	2	13	Netherlands Verbruggen 2005
			Above	Same	0.13 (aquatic organisms)	
Triclosan		0.0089	Below	2	0.1 (fresh water)	European Commission Water Framework Directive Annex VIII (WFD-UKTAG 2009)

Table 5, continued

Emerging organic contaminant	Abbreviation	Bell Is concentration (µg/L)	Above/below PNEC/NOEC	Order of magnitude	PNEC or NOEC* (µg/L)	Source
ethyl-paraben		0.141	Below	X	1600 (daphnia)*	
				2	80 (vitellogenic response in medaka (*Oryzias latipes*)*	Yamamoto et al. 2011
technical nonylphenol	TNP	0.0091	Below		0.20 (water)	Europe (WHO IPCS 2004)
			Below	1	0.330	European Union EU 2002
DEET		0.015	Below	4	407 (algae, daphnia zebrafish)	Sun et al. 2016
				3	43 (aquatic organisms)	European Union EU 2010
galaxolide		0.0175	Below	3	68 (freshwater fish)	United States EPA (USEPA 2014)
					39 (marine copepods)	
			Below	2	6,800 (marine organisms)	European Union: HERA 2004 EU 2008a
Bisphenol A	BPA	0.0134	Below	2	1.5	European Union EU 2008b
				2	1.6	Japan (AIST 2007)
				1	0.175	Canada (Env Canada 2008)
				Same	0.06 (aquatic organisms)	Meta analysis Wright-Walters et al. 2011

5. CONCLUSIONS

The concentrations of EOCs measured in the effluent of the Bell Island WWTP are considerably lower than those recognised to represent a risk to freshwater and marine organisms. This suggests EOCs represent a negligible risk to aquatic organisms in the receiving environment. In addition, the effluent will be subject to dispersion and dilution upon discharge to the environment, which would further reduce the concentrations of these EOCs. EOCs entering the receiving environment are likely to be subject to loss and removal through a range of microbial and chemical degradation processes, and adsorption to sediment particles.

There is currently limited information to characterise the impacts of EOCs on the receiving environment. Therefore, it is important to keep abreast of the latest research assessing the potential risks of EOCs so that effective actions can be implemented to manage them as required.

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7. APPENDIX

Appendix. 1 List of analysed Emerging Organic Contaminants and their Method Detection Limits (MDLs) in Bell Island WWTP effluent. ND = not detected above the MDL.

Emerging Organic Contaminant	Concentration (ng/L)	MDL (ng/L)
Alkyl phosphate Flame Retardants		
Tri-isobutyl phosphate	ND	0.10
Tri-butyl phosphate	643	0.10
Tris(2-chloroethyl)phosphate	ND	0.10
Tris(1-chloro-2-propyl)phosphate	1884	0.10
Tris[2-chloro-1-(chloromethyl)ethyl]phosphate	148	0.10
Tri-phenyl phosphate	2.71	0.10
Tris(2-butoxyethyl)phosphate	256	0.10
Tris(2-ethylhexyl)phosphate	ND	0.10
Tri-o-cresyl phosphate	ND	10
Tri-m-cresyl phosphate	ND	10
Tri-p-cresyl phosphate	ND	10
Phenolic anti-microbials		
Chloroxylenol	ND	0.05
o-phenylphenol	ND	0.10
Chlorophene	ND	0.10
methyl triclosan	ND	0.05
Triclosan	8.90	0.10
Paraben preservatives		
Methyl paraben	ND	0.05
Ethyl paraben	141	0.05
Propyl paraben	ND	0.05
Butyl paraben	ND	0.05
Benzyl paraben	ND	0.05
Industrial alkylphenols		
4-t-Amylphenol	ND	0.10
4-n-Amylphenol	ND	0.10
4-t-octylphenol	ND	0.10
4-t-heptphenol	ND	0.10
4-n-octylphenol	ND	0.10
4-n-nonylphenol	ND	0.10
Tech-NP-equivalents	9.10	5.0
Insect repellents		
DEET	15.3	1.0
Picaradin	ND	1.0
Benzylbenzoate	ND	1.0

Appendix 1 continued.

Emerging Organic Contaminant	Concentration (ng/L)	MDL (ng/L)
Musk fragrances		
Cashmeran	ND	1.0
Celestolide	ND	1.0
Phantolide	ND	1.0
Musk ambrette	ND	1.0
Traseolide	ND	1.0
Galaxolide	17.5	5.0
Musk xylene	ND	1.0
Tonalide	ND	5.0
Musk moskene	ND	1.0
Musk tibetene	ND	1.0
Musk ketone	ND	1.0
Acidic pharmaceuticals		
Acetaminophen	9.14	0.10
Aspirin	ND	0.10
Carbamazepine	302	0.10
Clofibric acid	ND	0.50
Diclofenac	19.35	0.10
Ibuprofen	6.05	0.10
Ketoprofen	ND	0.10
Meclofenamic	ND	0.50
Naproxen	158	0.10
Salicylic acid	44.4	2.0
Plasticisers		
Chloro-ethoxymethane	ND	5.0
Dimethylphthalate	ND	1.0
Diethylphthalate	82.7	5.0
4-Chlorophenyl phenyl ether	ND	0.10
4-bromophenyl phenyl ether	ND	0.10
Di-n-butylphthalate	65.6	5.0
Butylbenzyl phthalate	ND	0.10
Diethylhexylphthalate	56.0	25.0
Di-n-octylphthalate	ND	5.0
Monomethyl-PAE	1.58	1.0
Monobutyl-PAE	5.81	1.0
MonoEH-PAE	25.1	1.0
Bisphenol A	13.4	0.50

Appendix 1 continued.

Emerging Organic Contaminant	Concentration (ng/L)	MDL (ng/L)
Steroid hormones		
Estrone	ND	0.02
17α-estradiol	ND	0.02
17β-estradiol	ND	0.02
Estriol	ND	0.05
Mestranol	ND	0.02
17α-ethynylestradiol	ND	0.02
Dehydroisoandrosterone (DHEA)	ND	1.0
Androstenediol	ND	0.1
19-Nortestosterone	ND	1.0
Androstenedione	ND	0.1
Testosterone	ND	0.1
19-Norethindrone	ND	1.0
6β-hydroxy-testosterone	ND	5.0
11β-hydroxy-testosterone	ND	5.0
Norgestrel	ND	1.0
16-ketotestosterone	ND	0.1