

Biosolids Sampling Project



BC Ministry of the Environment and Climate Change Strategy

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Executive Summary

Biosolids are treated and stabilized wastewater treatment solids. Throughout the world, biosolids are typically land applied, incinerated or landfilled. When applied to land, biosolids provide nutrients for plants, and enhance soil quality, including chemical and physical properties.

In BC, the land application of biosolids is regulated by the Organic Matter Recycling Regulation (OMRR). The goal of the OMRR is to protect human health and the environment. The OMRR has standards for both Class A and B biosolids. For applications of less than 5 cubic metres per parcel of land, Class A biosolids may be land applied without a land application plan, whereas a land application plan is required for land application of any volume of Class B biosolids. The regulation also facilitates the beneficial re-use of select organic materials that have value as soil amendments, including compost derived from biosolids, and biosolids growing medium (a fabricated soil product).

In response to public concern regarding the land application of biosolids, in 2015 the Ministry of Environment and Climate Change Strategy (ENV) formed a Technical Working Group (TWG) to conduct a scientific review of biosolids. The TWG was tasked with two items:

1. oversee the preparation of a literature review on the risks associated with the land application of biosolids, and compost derived from biosolids, with relevance to the Nicola Valley, and
2. develop a sampling plan for
 - a. soils from the interior of the Province; and
 - b. biosolids from BC Wastewater Treatment Plants (WWTPs).

The literature review was published in June 2016 and is available on the ENV website. The soil sampling report was initially published in October 2016, and a revised edition of the report was published in January 2019.

In April 2016, the Province announced that it would conduct a review of the OMRR. Aspects of the OMRR review related to the land application of biosolids will be informed by the scientific review. At the time of publication of this report the OMRR review is in progress.

The biosolids sampling project commenced in late 2016. This report describes the results of the biosolids sampling project. The purpose of sampling biosolids was to compare metal and indicator pathogen levels against regulatory standards in the OMRR, and standards in other jurisdictions. Results are also compared to the Canadian Council of Ministers of Environment Guidelines for Compost Quality for context purposes only, since organic matter such as biosolids has similar benefits to compost. Compost is typically created by composting several different feedstocks together. Although origin of biosolids and compost differ, both are valuable sources of organic matter.

Additionally, to address concerns that have arisen from consultation with First Nations and stakeholders, sampling was conducted for a range of non-regulated compounds including persistent

organic pollutants (POPs) and contaminants of emerging concern (CEC). POPs and CECs in biosolids are not specifically regulated in BC or most other jurisdictions, apart from a small number of European countries. However, ENV and the TWG deemed it important to measure these potential contaminants to gauge levels in BC biosolids. The European standards are used in the report to provide perspective.

Biosolids from two WWTPs (referred to as WWTP 1 and WWTP 2) were analyzed in this project. For both WWTPs, the mean concentrations of OMRR regulated metals in the biosolids were below the regulatory standards specified in Schedule 4 Quality Criteria of the OMRR. Non-OMRR regulated elements and plant macro- and micro-nutrient concentrations in biosolids do not have applicable provincial standards.

Pathogen testing for both fecal coliforms and *Salmonella* was conducted. Currently the OMRR does not have standards for *Salmonella*, however the regulation does require that biosolids undergo controlled time and temperature treatment processes for pathogen reduction. WWTP 1 samples exceeded the OMRR standards for fecal coliform levels for Class A biosolids but were below the Class B biosolids limits. *Salmonella* was isolated in one of the representative samples taken at WWTP 1. However, the hold time for this sample exceeded the test protocols and therefore the *Salmonella* test results from WWTP 1 are inconclusive. The concentrations of pathogens from WWTP 2 met the OMRR standard for fecal coliforms for Class B biosolids. *Salmonella* was detected in all samples from WWTP 2.

The detected POPs were present at low levels and were below standards from European jurisdictions where they existed with one exception. The POP, di(2-ethylhexyl) phthalate, was below the draft European Union standard but slightly above the Danish standard.

Several CECs sampled were detected. While these CECs had no standards for comparison, concentrations of select CECs were below acceptable concentrations as determined by risk assessment (Kennedy/Jenks, 2017).

Agronomic parameters, including nutrients and organic carbon, were measured to provide information on the value of biosolids as a resource for plant nutrients and soil quality enhancement. High concentrations of nutrients were noted from both WWTP sources. The results were consistent with the literature and indicated that biosolids contain high levels of organic matter and micro and macro nutrients.

It is critical to note the limitations of the scope of the project including:

- a very limited number of samples were taken,
- only two WWTP facilities were tested,
- the study was conducted over the course of only one day at each facility.

The results provide a snapshot in time and would be expected to vary if repeated. The compounds present and their concentrations in biosolids can vary temporally and geographically, therefore, sampling more frequently and over a longer time at several WWTPs would enhance the representativeness of the results. It is important not to generalize these results or attempt to extrapolate them.

The results of this sampling project indicate that the pathogen indicators and metals concentrations in the two biosolids sources were below the OMRR standards, for Class B biosolids. The CECs present in the biosolids were lower than the acceptable concentrations identified. POPs, in all cases but one, were below European standards.

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List of Abbreviations

AAFC – Agriculture and Agri-Food Canada
BDE – brominated diphenyl ether
CCME – Canadian Council of Ministers of the Environment
CEC – Contaminants of Emerging Concern
CFIA – Canadian Food Inspection Agency
CFU – colony forming unit
CWN – Canadian Water Network
E. coli – *Escherichia coli*
EDC – endocrine disrupting compound
ENV – BC Ministry of Environment and Climate Change Strategy
EPA – Environmental Protection Agency
EU – European Union
meq/100 g – milli-equivalents per 100 grams
MPN – most probable number
NATO – North Atlantic Treaty Organization
ng/g – nanogram per gram, equivalent to parts per billion (ppb)
OMRR – Organic Matter Recycling Regulation, B.C. Reg 18/2002
PAH – polycyclic aromatic hydrocarbons
PCB – polychlorinated biphenyls
PCDD – polychlorinated dibenzodioxins
PCDF – polychlorinated dibenzofurans
PDBE – polybrominated diphenyl ethers
pg/g – picogram per gram, equivalent to parts per trillion (ppt)
pH – potential of hydrogen, a numeric scale used to specify the acidity or basicity
POPs – Persistent Organic Pollutants
µg/g – microgram per gram, equivalent to parts per million (ppm)
TCDD – 2,3,7,8-tetrachlorodibenzo-*p*-dioxin
TEF – Toxic Equivalency Factor
TEQ – Toxic Equivalents
TWG – Technical Working Group
WWTP – wastewater treatment plant

1.0 Introduction

1.1 Background

Biosolids are treated and stabilized wastewater treatment solids. Throughout the world, biosolids are typically land applied, incinerated or landfilled. When beneficially applied to land, biosolids provide nutrients for plants, and enhance soil quality, including chemical and physical properties.

In BC, the land application of biosolids is regulated by the Organic Matter Recycling Regulation (OMRR). The goal of the OMRR is to protect human health and the environment. The regulation also facilitates the beneficial use of select organic materials that have value as soil amendments, including biosolids.

In response to public concern regarding the land application of biosolids, in 2015 the Ministry of Environment and Climate Change Strategy (ENV) formed a Technical Working Group (TWG) to conduct a scientific review of biosolids. The TWG was tasked with two items:

1. oversee the preparation of a literature review on the risks associated with the land application of biosolids, and compost derived from biosolids, with relevance to the Nicola Valley, and
2. develop a sampling plan for
 - a. soils from the interior of the Province; and
 - b. biosolids from BC Wastewater Treatment Plants (WWTPs).

The literature review was completed by Dr. David Burton, LRCS Land Resource Consulting Services, and published on the ENV website (BC TWG, 2016a). The first phase of the sampling plan, for soils, was completed by the TWG in 2016 and a revised version of the soils sampling report (ENV, 2016b) will be published in tandem with this report. The second phase of the sampling plan (for biosolids) commenced in late 2016. This report describes the results of the biosolids sampling project and was used to inform the review of the OMRR which is in progress at the time of publication of this report.

Sampling was conducted for a range of OMRR-regulated and non-OMRR-regulated compounds including metals, pathogens, nutrients, persistent organic pollutants (POPs) and contaminants of emerging concern (CEC). In biosolids, POPs and CECs are not specifically regulated in BC or in most other jurisdictions, other than a small number of European countries. However, it was deemed important to measure these potential contaminants to gauge levels in BC biosolids. The European standards are used in the report for benchmarking purposes.

POPs are carbon-based compounds that: persist in the environment; bioaccumulate (i.e., they are found at increasingly higher concentrations as one moves up the food chain); and may be toxic to humans and/or wildlife. POPs studied in this report include select:

- polycyclic aromatic hydrocarbons (PAHs);
- polychlorinated biphenyls (PCBs);
- phthalates; and
- dioxins and furans.

CECs consist of a variety of compounds, including endocrine disrupting compounds (EDCs) which may be present in WWTPs due to the use of pharmaceuticals, personal care products and fire retardants. Generally, CECs have been difficult to quantify because they are present in small concentrations and suitable test methods have not been developed. Concerns are emerging regarding the impact of these compounds in small concentrations in the environment. CECs are discussed by various agencies using a wide variety of names including:

- Emerging Substances of Concern – as used by Canadian Council of Ministers of the Environment (CCME), Agriculture and Agri-Food Canada (AAFC), Florida State, and Canadian Water Network; and
- Substances of Emerging Concern – as used by Environment and Climate Change Canada

The term “CEC” is used by ENV in guidance for the Contaminated Sites Regulation, the International Joint Commission (2009), and the US Environmental Protection Agency (EPA). The term CEC is used in this report to more accurately frame the discussion regarding emerging concerns about potential contaminants which may be present in domestic WWTPs.

CECs and POPs are not mutually exclusive and some CECs, such as the polybrominated diphenyl ethers in fire retardants, are also identified as POPs. Figure 1 provides examples of some POPs and CECs, and some of the possible sources.

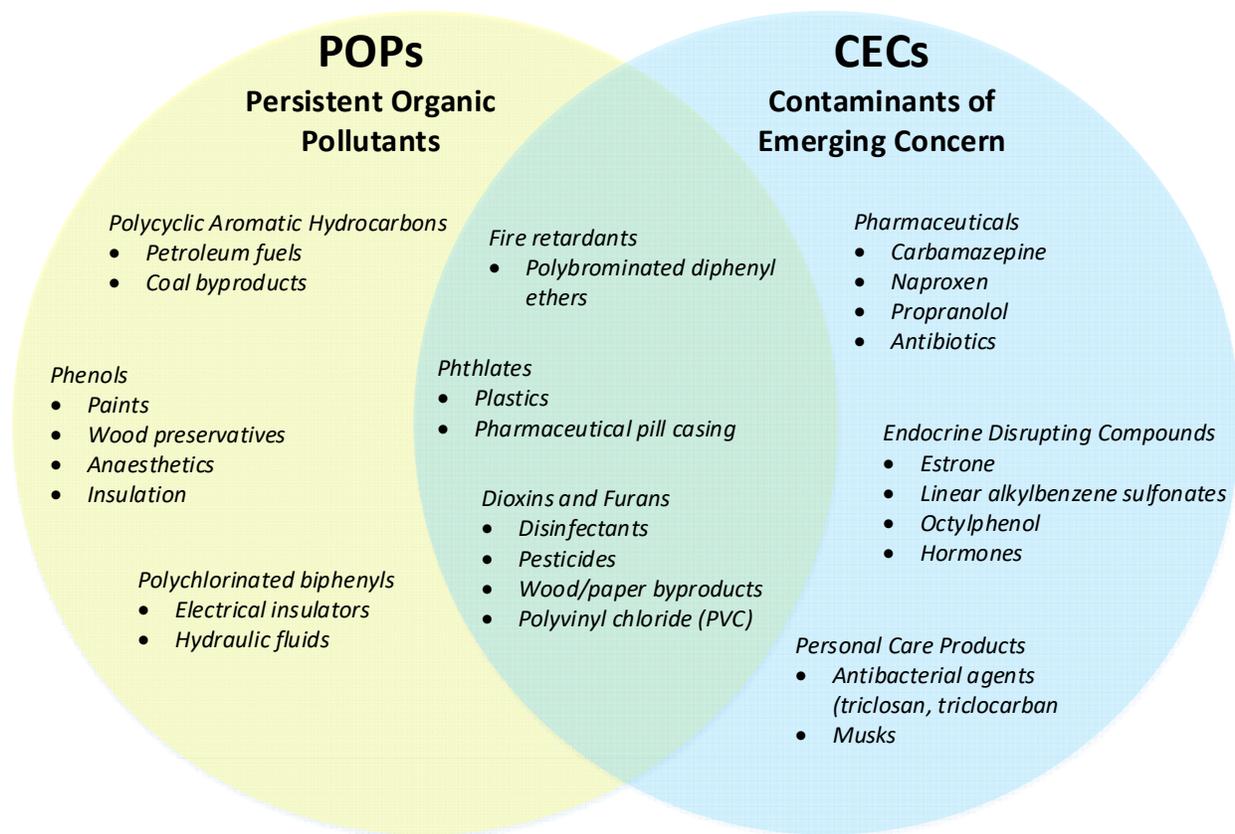


Figure 1. Examples of Persistent Organic Pollutants and Contaminants of Emerging Concern

The compounds itemized in Figure 1 are not intended to be a complete list. Continuing research may change how these compounds are categorized as either CECs or POPs.

Personal care products and medical/dental implements may also be the source of some metals, such as mercury from dental amalgam, and selenium from dandruff shampoo. Generally, these metals are not considered CECs because the environmental concerns have been already identified and are not considered to be of emerging concern.

1.2 Project Objectives

The objectives of this sampling project were to:

- 1) Compare concentrations of substances and pathogens in biosolids samples to OMRR standards; and
- 2) Obtain information regarding the nutrient content of the sampled biosolids and the levels of non-OMRR regulated compounds (specified pathogens, POPs, and CECs) in biosolids.

1.3 Project Scope

This project was limited to sampling of biosolids from two wastewater treatment plants in BC. WWTP 1 is a plant that typically produces Class A biosolids, while WWTP 2 is a plant that typically produces Class

B biosolids. Biosolids class is determined based upon the pathogen reduction processes, fecal coliform levels, and metals concentrations prescribed in the OMRR.

The biosolids analysed from WWTP 1 were processed through auto-thermal thermophilic aerobic digestion (10 to 12 days at 45 to 65°C), while the biosolids analysed from WWTP 2 were processed through mesophilic anaerobic digestion (32 days average retention time at 38°C).

The results of this study are neither intended to be nor are reflective of a rigorous scientific experiment. Only two WWTPs were sampled. Rather, the results depict a snapshot in time. Parameter detection and concentration within biosolids is expected to vary temporally and with wastewater source and the treatment process.

1.3.1 Targeted Parameters

Within the province of BC, the OMRR establishes the regulatory requirements for processing and land application of biosolids. In 2015, ENV's TWG selected several parameters to be analysed in soil and biosolids. Non-OMRR regulated parameters were included because they have standards in other jurisdictions, including some European countries, and/or because literature suggests that each of the POPs and CECs listed in Table 1 may be found in biosolids (CCME, 2012), and/or literature showed the compounds were more likely to pose a risk, (ENV, 2016a). This list was then refined based on laboratory analytical constraints, sample preservation requirements, and/or cost considerations. The TWG proposed inclusion of musks; however, due to constraints, they were not analyzed. Table 1 presents the parameters that were proposed by TWG for this study which includes parameters that are regulated under the OMRR.

Table 1 also identifies additional parameters, beyond those proposed by the TWG that were analyzed and reported by the laboratory. Many laboratory analytical packages include a set suite of parameters. The laboratory analysis provided results for parameters not included on the TWG list, for example:

- In the suite of metals, there are results for aluminum (Al), strontium (Sr) and tin (Sn), which were not initially proposed by the TWG. Not included in the results is thallium (Tl), which is not measured with the same methodology, nor included with the suite for metals. Additional testing was not requested for Tl results.
- The TWG recommended analysis of three congeners of polybrominated diphenyl ethers (PBDEs). Congeners are chemical substances related to each other by origin, structure or function. The standard laboratory analysis provided results for an additional 62 PBDEs. These results are included in Appendix 1 - Additional Contaminants of Emerging Concern Results.

The TWG did not list which compounds and congeners to target within the compound groups: PCBs; dioxins; and furans. The laboratory reported the results for compounds which are routinely included in the methodology and analytical suites for these compound groups.

Disease-causing prions and pathogenic helminth worms were not analyzed in this project, due in part to cost and to sample holding times. Several of the non-chlorinated phenols were not analyzed for due to

costs and availability of laboratory resources. However, the analysis for indicator pathogens and similar phenol compounds provided an indication of the presence/absence of these parameters.

Table 1 Parameters in Biosolids: Proposed and Analyzed

Compound Grouping	Compound Name	Proposed	Analyzed
General Parameters			
Metals	Aluminum (Al)		✓
	Antimony (Sb)	✓	✓
	Arsenic (As)	✓	✓
	Barium (Ba)	✓	✓
	Beryllium (Be)	✓	✓
	Boron (B)	✓	✓
	Cadmium (Cd)	✓	✓
	Calcium (Ca)	✓	✓
	Chromium (Cr)	✓	✓
	Cobalt (Co)	✓	✓
	Copper (Cu)	✓	✓
	Iron (Fe)	✓	✓
	Lead (Pb)	✓	✓
	Magnesium (Mg)	✓	✓
	Manganese (Mn)	✓	✓
	Mercury (Hg)	✓	✓
	Molybdenum (Mo)	✓	✓
	Nickel (Ni)	✓	✓
	Phosphorus (P)	✓	✓
	Potassium (K)	✓	✓
	Selenium (Se)	✓	✓
	Silver (Ag)	✓	✓
	Sodium (Na)	✓	✓
	Strontium (Sr)		✓
	Sulfur (S)	✓	✓
	Thallium (Tl)	✓	
	Tin (Sn)		✓
	Titanium (Ti)		✓
Vanadium (V)	✓	✓	
Zinc (Zn)	✓	✓	
Pathogens & Pathogen Indicators	Fecal coliforms	✓	✓
	enteric viruses	✓	
	<i>Salmonella</i>	✓	✓
	<i>Escherichia coli</i>	✓	✓
	Prions	✓	
	Helminth worms	✓	

Compound Grouping	Compound Name	Proposed	Analyzed
Carbon & Nitrogen	Total Carbon (C) (as total organic carbon)	✓	✓
	Total Kjeldahl Nitrogen-N (TKN)	✓	✓
	Organic Nitrogen		✓
	Nitrate	✓	
	Total Nitrogen (= TKN + nitrate + nitrite)		✓
	Ammonia – N (NH ₄ -N) (= TKN – organic nitrogen)	✓	
Physical and Chemical Parameters	pH	✓	✓
	cation exchange capacity	✓	✓
Contaminants of Emerging Concern			
Pharmaceuticals	Carbamazepine	✓	✓
	Naproxen	✓	✓
	Propranolol	✓	✓
	Ciprofloxacin	✓	✓
	Azithromycin	✓	✓
Personal Care Products	Triclosan	✓	✓
	Triclocarban	✓	✓
Polybrominated Diphenyl Ethers (PBDEs)	PBDE-47	✓	✓
	PBDE-99	✓	✓
	PBDE-209	✓	✓
Musks	Galaxolide	✓	
	Tonalide	✓	

Compound Grouping	Compound Name	Proposed	Analyzed
Persistent Organic Pollutants			
Phenols	chlorinated phenols:		
	4-Chloro-3-methylphenol		✓
	chlorophenol isomers (ortho, meta, para)	✓	✓
	dichlorophenols (2,6-, 2,5-, 2,4-, 3,5-, 2,3-, 3,4-)	✓	✓
	trichlorophenols (2,3,4-, 2,3,5-, 2,3,6-, 2,4,5-, 2,4,6-, 3,4,5-)	✓	✓
	tetrachlorophenols (2,3,5,6-, 2,3,4,5-, 2,3,4,6-)	✓	✓
	Pentachlorophenol	✓	✓
	non-chlorinated phenols:		
	2,4-dimethylphenol	✓	✓
	2,4-dinitrophenol	✓	
	2-methyl 4,6-dinitrophenol	✓	
	nitrophenol (2-, 4-)	✓	
	phenol	✓	✓
	Cresol (<i>o</i> -, <i>m</i> -, <i>p</i> -)	✓	✓
	Polycyclic Aromatic Hydrocarbons (PAHs)	acenaphthene	✓
acenaphthylene			✓
anthracene		✓	✓
benz[a]anthracene		✓	✓
benzo[a]pyrene		✓	✓
benzo[b]fluoranthene		✓	✓
benzo[j]fluoranthene			✓
benzo[k]fluoranthene		✓	✓
chrysene		✓	✓
dibenz[a,h]anthracene		✓	✓
fluorene		✓	✓
indeno[1,2,3-cd]pyrene		✓	✓
2-Methylnaphthalene			✓
naphthalene		✓	✓
phenanthrene		✓	✓
pyrene		✓	✓

Compound Grouping	Compound Name	Proposed	Analyzed
Phthalates	dibutyl phthalate (as butylbenzyl phthalate)	✓	✓
	Diethyl phthalate		✓
	Dimethyl phthalate		✓
	Di-n-butyl phthalate		✓
	Di-n-Octyl phthalate (straight chain isomer of di(2-ethylhexyl phthalate))		✓
	di(2-ethylhexyl) phthalate (as bis(2-Ethylhexyl)phthalate)	✓	✓
Polychlorinated Biphenyls (PCBs)	PCBs (specific compounds not specified by TWG)	✓	✓
	PCB-1016		✓
	PCB-1221		✓
	PCB-1232		✓
	PCB-1242		✓
	PCB-1248		✓
	PCB-1254		✓
	PCB-1260		✓
	PCB-1262		✓
	PCB-1268		✓
Dioxins	Dioxins (specific compounds not specified by the TWG)	✓	✓
	polychlorinated dibenzo-p-dioxin congeners		✓
Furans	Furans (specific compounds not specified by the TWG)	✓	✓
	polychlorinated dibenzo-furan congeners		✓

*Note: **Bold** represents parameters regulated under the OMRR.*

Some of the natural sources and industrial uses/sources, for the targeted POPs are presented in Table 2.

Table 2 Targeted Persistent Organic Pollutants and Their Sources

Persistent Organic Pollutants	Natural Source	Industrial Use or Source
Phenols	produced by plants, animals and organisms; found in coal tar	used in paints, paint remover, rubber, wood preservatives, textiles, perfumes, plastics, anaesthetic, antiseptic, and insulation
Polycyclic Aromatic Hydrocarbons (PAHs)	produced by some plants, animals and organisms	released by petroleum or coal-derived products through combustion processes (vehicle exhaust, airplanes and industrial processes)

Persistent Organic Pollutants	Natural Source	Industrial Use or Source
Polychlorinated biphenyls (PCBs)	none known	used in electrical insulators, adhesives, lubricants, hydraulic fluids, flame retardants, waterproofing materials, insulating/cooling agents
Polychlorinated dibenzo- <i>p</i> -dioxins, Polychlorinated dibenzofurans	occurring as a result of incomplete combustion (e.g., forest fires)	created when products like herbicides, pesticides, dyes, disinfectants and polyvinyl chloride are manufactured; created in the pulp and paper industry by wood pulp bleaching

The soil sampling report (ENV 2016a) recommended additional investigation of the following CECs. Some of the uses or sources of the targeted CECs are presented in Table 3.

Table 3 Targeted Contaminants of Emerging Concern and Their Sources

Category	Compound(s)	Source or Use
Pharmaceuticals	Carbamazepine	to treat seizures and nerve pain
	Naproxen	to treat pain and inflammation
	Propranolol	to treat chest pain, high blood pressure, heart rhythm disorders, and other heart or circulatory conditions
	Ciprofloxacin	antibiotic used to treat a variety of bacterial infections
	Azithromycin	antibiotic used to treat a variety of bacterial infections
	Phthalates	Plasticizer used in pill casings. Also found in: adhesives, building materials, vinyl flooring, personal care products, medical devices, detergents, packaging, toys, modelling clay, waxes, food products; naturally produced by some microorganisms such as bacteria, fungi and yeasts and by some plants
Personal Care Products	Triclosan	antibacterial and antifungal agent found in: toothpaste, soaps, detergents, toys, and surgical cleaning treatments
	Triclocarban	antibacterial agent products such as soaps and lotions
Polybrominated Diphenyl Ethers (PBDEs)	BDE-47 BDE-99 BDE-209	flame retardants used in building materials, electronics, furnishings, plastics and textiles (fabrics)
Musks	Galaxolide Tonalide	used in products such as soaps, shampoos, lotions and fragrances

Additional background on the CECs considered in this study is provided in the following sections.

1.3.1.1 Pharmaceuticals and Personal Care Products

The TWG proposed the analysis of indicator compounds rather than analyzing many chemicals in this category. The compounds selected as indicators may be frequently found in biosolids due to their presence in pharmaceuticals and consumer products.

For this study, the five compounds listed in Table 3 under pharmaceuticals were included for analysis. These five compounds are considered indicators that represent groups of chemicals present in biosolids with similar chemical characteristics including repelling water molecules. Salveson et al. (2012) used this approach to model the chemical fate of CECs in WWTPs.

Two antibiotics, ciprofloxacin and azithromycin, were selected for testing based on findings in the scientific literature related to their presence in biosolids, animal manures, and the possible induction of antibiotic resistance in soil organisms (CCME, 2009).

Kennedy/Jenks Consultants undertook a risk assessment for Metro Vancouver in 2017 of select CECs in biosolids (Kennedy/Jenks, 2017). The CECs studied included three of the same pharmaceuticals and personal care products identified by the TWG for study: triclosan, ciprofloxacin and azithromycin. Kennedy/Jenks' results for these parameters are included in this report for context purposes only.

1.3.1.2 Polybrominated Diphenyl Ethers

Polybrominated diphenyl ethers (PBDEs) are hydrophobic compounds used in flame retardants that are relatively persistent in the environment. There are 209 known congeners of PBDEs. The most common are BDE-47, BDE-99, and BDE-209, which are widely used in upholstery, textiles and other household products. Kennedy/Jenks' risk assessment results for BDE-209 (also known as "deca BDF") are included in this report in Appendix 1, for context purposes.

1.3.1.3 Endocrine Disrupting Compounds

Endocrine disrupting compounds (EDCs) are natural or artificial compounds that, at certain doses, can impact the endocrine (i.e., hormonal) system in mammals and aquatic life. EDCs such as estrone, octylphenol, and linear alkylbenzene sulfonates have been observed in effluent, sludge, and biosolids from WWTPs (Langdon et al., 2011).

Research conducted on aerated soils typical of Canadian growing regions suggests that specific estrogenic compounds (4-Nonylphenol, ethynylestradiol, estradiol, and estrone) are rapidly degraded (Lorenzen et al., 2006), therefore posing minimal risk of leaching and contamination of groundwater and aquatic systems. Based on this research, EDCs were not specifically targeted in this report; however, POPs that may impact the endocrine system, such as PCBs and dioxins, were included.

1.3.1.4 Other Contaminants of Emerging Concern

There are other types of compounds classified as EDCs which may not degrade as rapidly as those listed above including: dioxins, furans, PCBs and phthalates. These compounds were included in this study under the POPs grouping as summarized in Table 1 and Table 2.

2.0 Methodology

2.1 Sampling Protocol

Biosolids samples were collected from two separate BC WWTPs on November 1, 2016 at WWTP 1 and November 8, 2016 at WWTP 2. The samples were collected according to the Land Application Guideline

for the Organic Matter Recycling Regulation and Soil Amendment Code of Practice (Land Application Guide, ENV, 2008) and the OMRR. WWTP 1 was selected to target Class A biosolids, while WWTP 2 was selected to target Class B biosolids. Sampling was done with care to avoid cross contamination. The details of sampling are as follows:

- Sampling equipment was cleaned prior to sampling.
- Only biosolids that were not in direct contact with sampling equipment were collected for analysis.
- The sampling team used a dedicated pair of nitrile gloves for each sample, which were disposed of and replaced with clean gloves after each sample to prevent cross-contamination.
- For the targeted Class A biosolids, seven representative samples were collected after the centrifuge process from WWTP 1 for pathogen analysis as per Schedule 3 of OMRR.
- For the targeted Class B biosolids, seven grab samples were collected from WWTP 2 at one-hour intervals from the centrifuge for pathogen analysis as per Schedule 3 of OMRR.
- Three composite samples, comprised of seven grab samples each, were also collected for analysis of parameters other than pathogens, as is recommended in the Land Application Guide (Land Application Guide, ENV, 2008), from each of WWTP 1 and WWTP 2.
- These samples were placed in stainless steel bowls and covered with aluminum foil (to avoid exposure to light) until they could be transferred into the appropriate containers for submission to the laboratory. Extra care was used to not cause abrasions in the bowls, thereby avoiding metal contamination from the bowls themselves.
- From the bowls:
 - Samples for phthalates, phenols, PCB and PAH samples were placed in laboratory-supplied 125 mL clear glass sample jars.
 - Samples for pharmaceuticals/personal care products samples were placed in 250 mL amber glass jars (to avoid exposure to light) supplied by the laboratory.
- Samples were placed in containers with minimal headspace and sealed for laboratory analysis.
- All samples were transported in a cooler with ice packs.

2.2 Laboratory Analysis

Samples were transported to the ALS Laboratory (ALS) in Burnaby, BC for analysis. ALS is accredited by the Canadian Association for Laboratory Accreditation Inc. ALS performs its own internal quality assurance and quality control (QA/QC) checks. ALS's QA/QC results were reviewed by ENV and are discussed in Section 3.10 Laboratory Quality Control. The pharmaceutical and personal care product testing was subcontracted out by ALS to Axys Enviro in Sidney, BC.

2.3 Data Analysis

The units used to measure and report concentrations in this report include:

- Micrograms per gram ($\mu\text{g/g}$) which is equivalent to “parts per million” (ppm) and “milligrams per kilogram” (mg/kg).
For example, 1 $\mu\text{g/g}$ is like one drop of ink in a 50 L barrel of water.
- Nanograms per gram (ng/g), which is equivalent to “parts per billion” (ppb) or 10^{-3} $\mu\text{g/g}$.
For context, 1 ng/g (or 1 ppb) is like one second in 32 years.
- Picograms per gram (pg/g) which is equivalent to “parts per trillion” (ppt) or 10^{-6} $\mu\text{g/g}$.
For example, 1 pg/g is like one drop of ink in 20 Olympic-sized swimming pools.

Other units used to measure compounds include:

- MPN/g is the most probable number of bacteria per gram of sample. These units are calculated values based on an index table and largest volume tested in a dilution series. They are not an actual count of the existing indicator bacteria.
- milliequivalents per 100 grams of soil (mEq/100 g), which is the total positive charge that can be exchanged per 100 grams of soil.

Concentrations of parameters regulated under the OMRR were compared to the standards within the OMRR. For compounds which do not have standards under the OMRR, the results were compared to standards in other jurisdictions, where they exist. In addition, results were also compared to the Canadian Council of Ministers of Environment Guidelines for Compost Quality (CCME, 2005) for context purposes only, since organic matter such as biosolids has similar benefits to compost.

The CCME has developed two compost categories for trace element concentrations and sharp foreign matter. These categories (A and B) are based on the end use of the compost material. Category A compost can be used in any application, which differs from than land application restrictions imposed by the OMRR for Class A biosolids. Both CCME Category B compost and OMRR Class B biosolids have restricted uses.

This project compares non-OMRR regulated POPs with the regulated standards in European jurisdictions where they exist. Results for non-OMRR regulated pathogens e.g., *Escherichia coli* (*E. coli*) and *Salmonella*, were also compared with standards where they exist. The results were not compared to Contaminated Sites Regulation (CSR) standards, as CSR standards are not applicable to the biosolids product, but rather to the soil in the receiving environment.

The data results that are presented in the tables that follow are calculated means of the raw data. Where individual results were below the detection limit, the values reported in the tables are the largest detection limit of the data set. The “<” symbol is used to indicate where the concentrations were below detection limits.

3.0 Results and Discussion

There are no CCME standards for biosolids. The CCME has standards for pathogens, foreign matter and metals for two Categories of compost products: Category A (unrestricted use), and Category B (restricted use) (CCME, 2005). Category A is more stringent than Category B. Details of the derivation of the CCME standards are available in the "Support Document for Compost Quality Criteria, National Standard of Canada CANBNQ 0413-200: CCME Guidelines and AAFC Criteria" (CCME, 1996). Note that the CCME Compost guidelines for Category B Compost (maximum in product) are the same as those in the T-4-93 (1997) Trade Memorandum, except for chromium and copper. The CCME guidelines for chromium and copper were calculated in the same way as the limits for the other nine elements in the T-4-93 (1997) Trade Memorandum.

3.1 Nutrients and Other Agronomic Parameters

The mean concentrations for macro-nutrients and total organic carbon analyzed in the biosolids samples are presented in Table 4.

Table 4 Nutrients in Biosolids

Total Available Nutrients	WWTP 1 Biosolids	WWTP 2 Biosolids
Total Kjeldahl Nitrogen (%)	4.0	5.3
Total Organic Nitrogen (%)	3.60	4.97
Total Nitrogen (%)	4.29	5.64
Total Organic Carbon (%)	34.6	37
Total Phosphorus (µg/g)	28 600	17 967
Total Potassium (µg/g)	1 180	1 160

Additional analyte mean concentrations beyond those in Table 4 are provided in Table 5, and provide a rough indication of what may be available in the soil to support plant growth.

Table 5 Plant Available Nutrients and Soil Conditioners in Biosolids

Plant Available Nutrients (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids
Ammonium-N	3 603	3 383
Calcium (Ca)	2 583	4 733
Copper (Cu)	35.7	10
Iron (Fe)	1 028	1 427
Magnesium (Mg)	2 380	1 250
Manganese (Mn)	29.6	206
Phosphate-P	1 780	1 067
Potassium (K)	840	817
Sodium (Na)	2 457	500
Zinc (Zn)	197	156

Parameters presented in Table 6 include macro nutrients which have agronomic significance. The mean concentrations of plant nutrients from both WWTP 1 and WWTP 2 indicate that the biosolids may have significant value as a soil amendment. This is consistent with the findings on biosolids which have been widely reported in the literature (CWN, 2015).

Table 6 Cation Exchange Capacity and pH in Biosolids

Biosolids Parameter	WWTP 1 Biosolids	WWTP 2 Biosolids
Cation Exchange Capacity (meq/100 g)	56.6	72.7
pH	7.34	7.44

Cation exchange capacity is a measure of the ability of a substance (usually soil) to bind to positively charged ions (i.e., cations) including plant nutrients such as phosphorus. The units for measuring cation exchange capacity are milli-equivalents per 100 g of soil (meq/100 g). Biosolids, including those analysed as part of this study, have a relatively high cation exchange capacity which can be beneficial when added to soil since it can increase the ability of a soil to bind positively charged ions (e.g., phosphorus) and increase a plant's access to vital nutrients. This is particularly the case for soils with low organic matter content.

The pH reported is that of the solution created by mixing dried biosolids sample with deionized/distilled water at a 1:2 ratio of biosolids to water. The pH of land-applied biosolids can affect the mobility of metals in the soil.

3.2 Metals

Schedule 4 of the OMRR does not explicitly list standards for Class A biosolids, but instead requires that Class A biosolids meet the metals limits specified in the Canada Food Inspection Agency (CFIA) T-4-93 Trade Memorandum (CFIA, 1997). These are based on Agriculture and Agri-Food Canada (AAFC) metals standards. The OMRR Schedule 4 provides standards for Class B biosolids for 11 metals. The standards are based on an assumed annual application rate of 4,400 kilograms dry product per hectare and originate from the T-4-93 Trade Memorandum (CFIA, 1997), except for chromium, copper and mercury.

In Table 7, mean concentrations of OMRR-regulated metals in the biosolids samples are compared to the Class A and Class B biosolids standards from Schedule 4 of the OMRR, and the CCME compost guidelines for Category A and Category B. The CCME compost guidelines are provided for interest sake only, due to the similar benefits provided by both compost and biosolids use.

Table 7 OMRR-Regulated Metals in Biosolids Samples

Metals	OMRR Biosolids Standards (µg/g)		CCME Compost Guidelines (mg/kg = µg/g)		Mean Concentrations (µg/g)	
	Class A	Class B	Category A	Category B (max in product)	WWTP 1 Biosolids	WWTP 2 Biosolids
Arsenic	75	75	13	75	3.13	4.89
Cadmium	20	20	3	20	2.47	3.40
Chromium	NS	1060	210	1060	25.8	25.9
Cobalt	150	150	34	150	2.56	4.97
Copper	NS	2200	400	757	1015	463
Lead	500	500	150	500	21.3	43.6
Mercury	5	15	0.8	5	1.29	1.28
Molybdenum	20	20	5	20	5.62	6.80
Nickel	180	180	62	180	11.8	23.4
Selenium	14	14	2	14	4.25	4.54
Zinc	1850	1850	700	1850	1078	927

Note:

“NS” indicates that the limits for copper and chromium are not established in the T-4-93 Trade Memorandum. The limits for copper and chromium are calculated in the same manner as limits for the other nine elements. The trace element concentrations within the compost product are: chromium = 1060 mg/kg and copper = 757 mg/kg (CCME, 2005).

Numbers in bold exceed CCME limits for Category A and/or Category B Compost Guidelines

For both biosolids sources, the mean concentrations of metals were below the regulatory standards for Class A and Class B biosolids specified in the OMRR.

Table 7 indicates that in some cases the metals guidelines for compost are more restrictive than the OMRR standards for biosolids. This is to be expected as Category A compost has unrestricted use. Despite biosolids having restrictions on land application under OMRR, the biosolids results still met the CCME compost guidelines for just under half the regulated metals for Category A compost, and for seven metals for Category B compost, where there are multiple applications to soil.

When compared to the CCME compost guidelines, the mean concentrations of the following parameters were above the guidelines for CCME Category A compost:

- cadmium in biosolids from WWTP 2,
- copper in biosolids from both WWTP 1 and WWTP 2,
- mercury from WWTP 1 and WWTP 2,
- molybdenum, from WWTP 1 and WWTP 2,
- selenium from WWTP 1 and WWTP 2, and
- zinc from WWTP 1 and WWTP 2.

There are no requirements for biosolids to comply with CCME compost guidelines, as they are not a compost product. Compost and biosolids have different restrictions on their use.

The results in Table 7 compared to OMRR standards are depicted graphically in Figure 2, Figure 3 and Figure 4 that follow. The data has been split into three figures to reflect the different magnitudes of data and to keep the scale on the y axis legible.

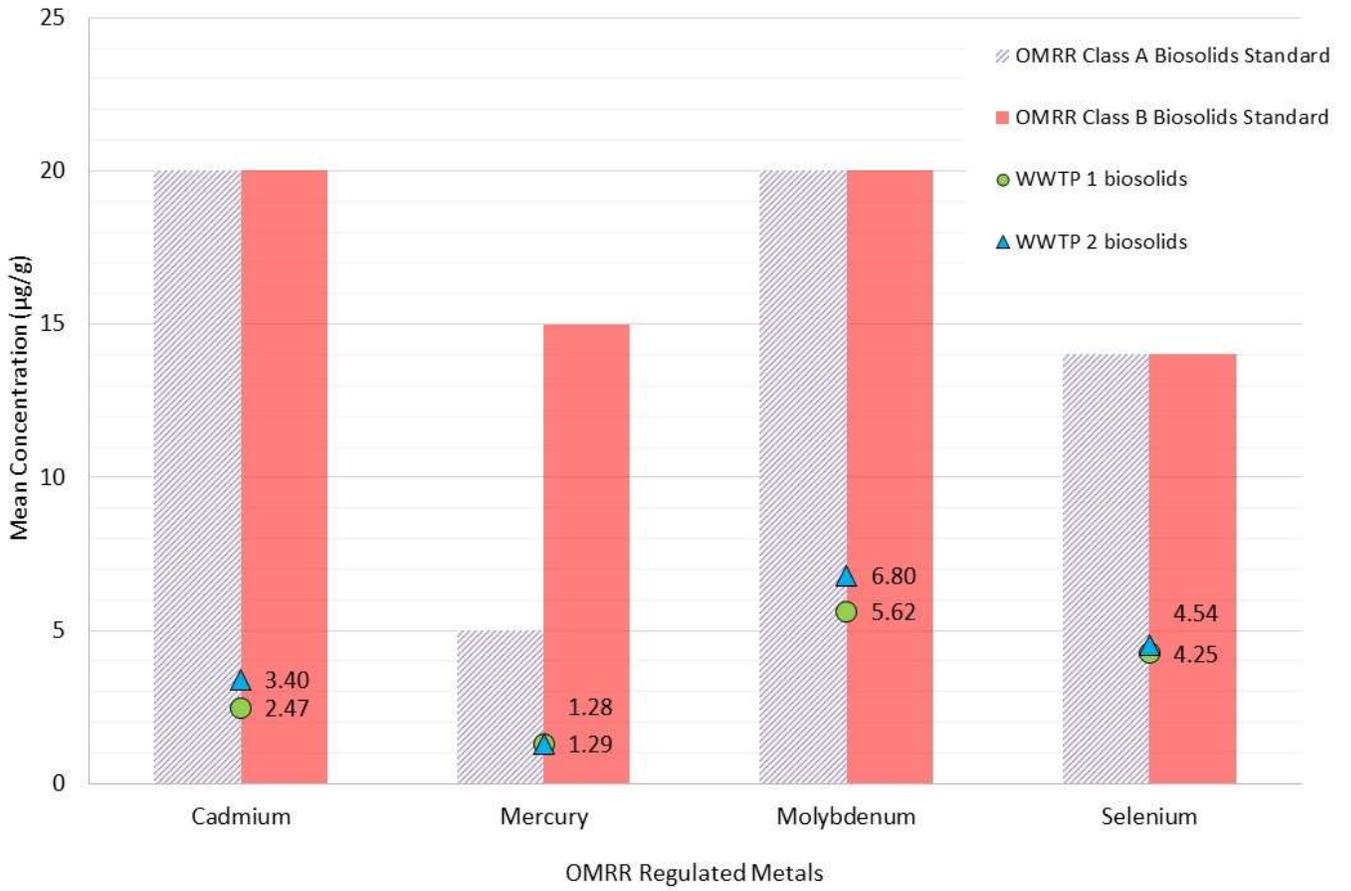


Figure 2. Cadmium, Mercury, Molybdenum and Selenium Results

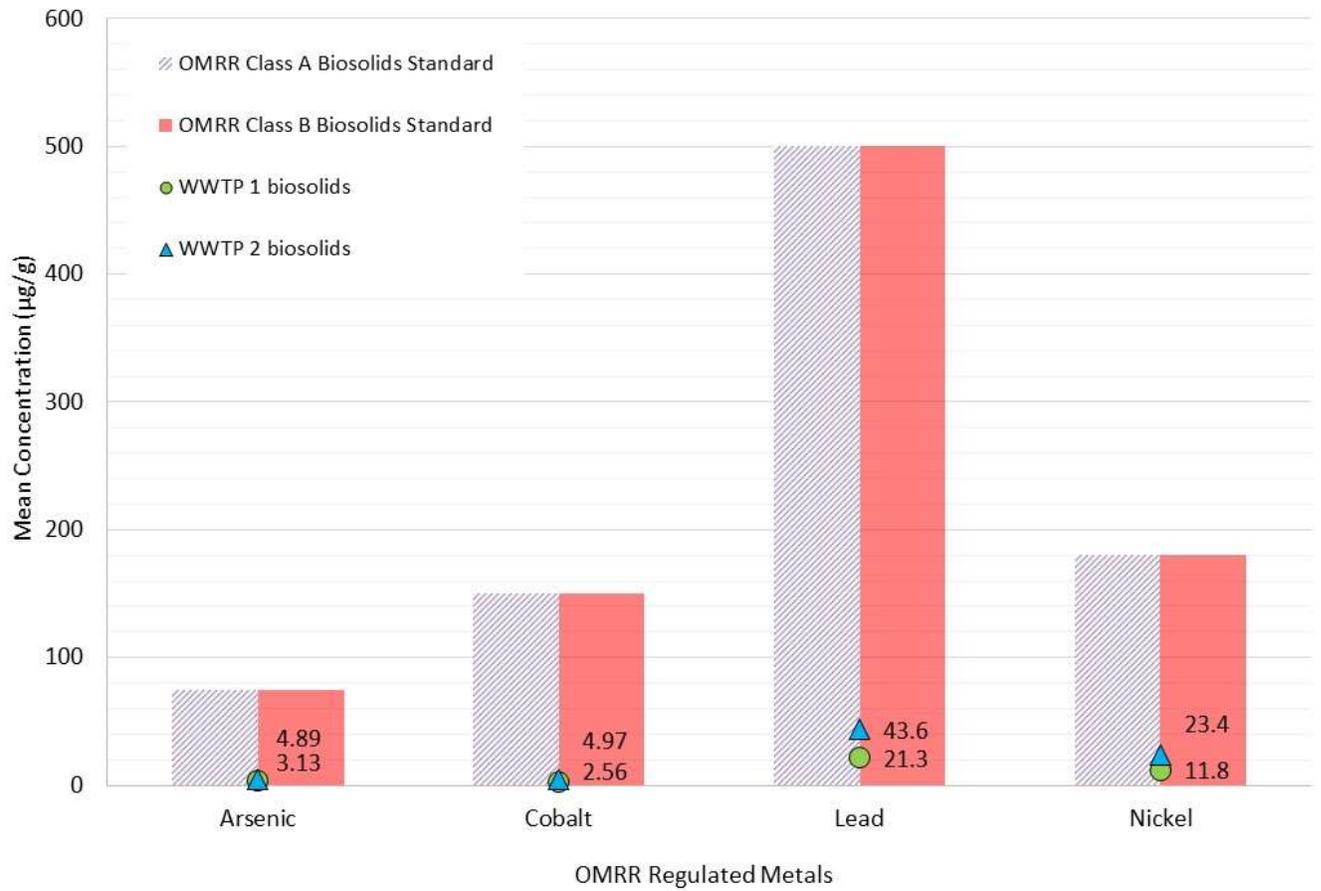


Figure 3. Arsenic, Cobalt, Lead and Nickel Results

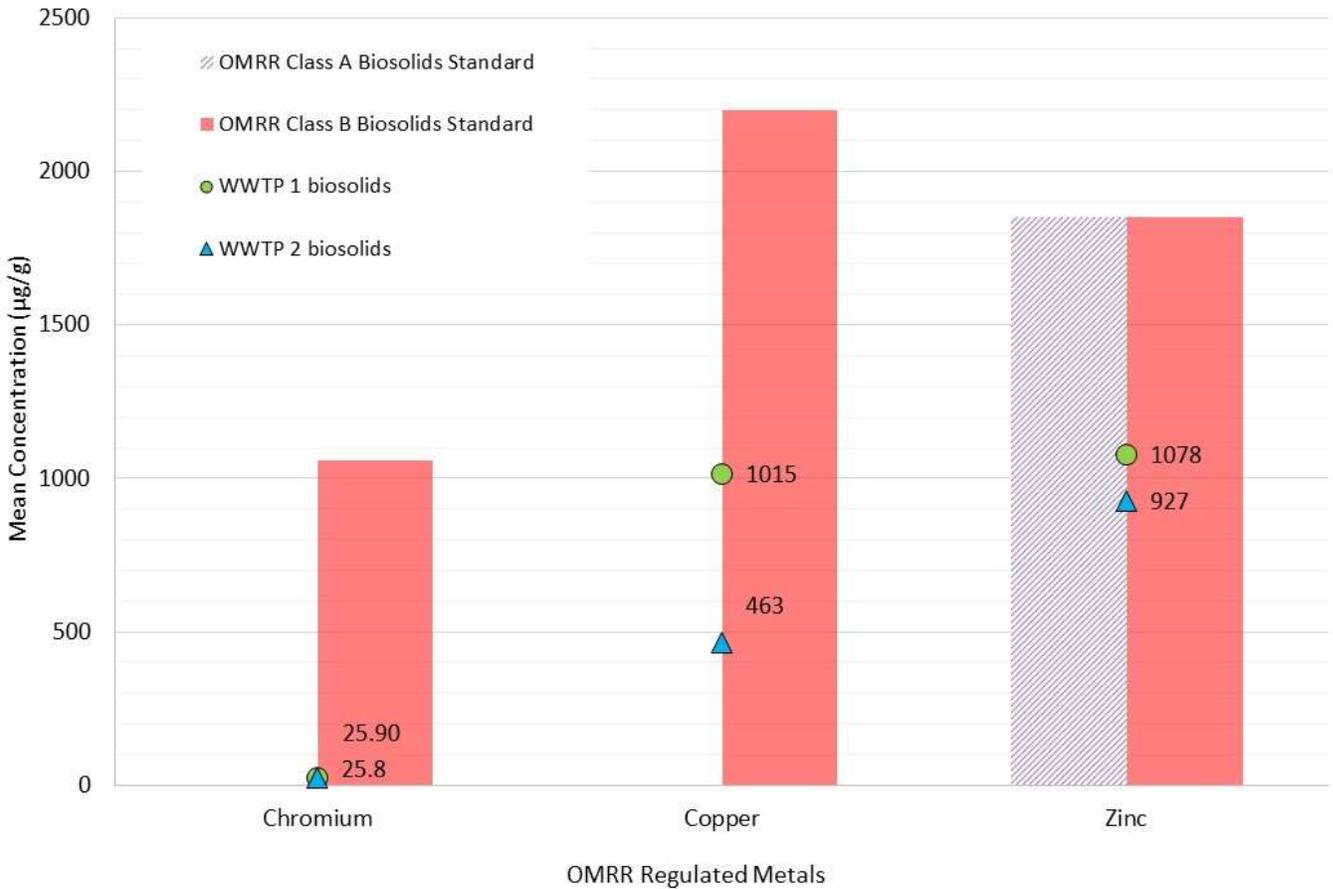


Figure 4 Chromium, Copper and Zinc Results

The OMRR Class B biosolids metals standards are risk-based standards developed to protect human health and the environment. The results show that the mean concentrations from both WWTP 1 and WWTP 2 are compliant with the OMRR Class B standards, and the Class A standards if an annual application rate of 4,400 kilograms dry product per hectare is assumed.

Non-OMRR metals and plant macro and micro nutrients were also tested in the samples and the mean results are presented in Table 8.

Table 8 Non-OMRR Regulated Metals in Biosolids

Total Metals (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids
Aluminum (Al)	8 063	4 273
Antimony (Sb)	1.80	2.29
Barium (Ba)	202	182
Beryllium (Be)	0.12	0.16
Boron (B)	27.4	14.0
Calcium (Ca)	23 300	4733
Iron (Fe)	34 367	22 367
Magnesium (Mg)	8 763	1 250
Manganese (Mn)	273	765
Silver (Ag)	3.86	6.51
Strontium (Sr)	342	94.9
Sulfur (S)-elemental	12 333	12 400
Sulfur (S)-total	18 000	13 900
Tin (Sn)	30.8	31.0
Titanium (Ti)	75.3	55.0
Vanadium (V)	15.8	11.6

The OMRR does not have standards for the metals in Table 8.

Some of the metals listed in Table 8 are nutrients for plants, including: Ca, Mg, S, Al, Bo, Fe, Mn and V. The concentrations of the parameters in Table 8 indicate that biosolids are a valuable source of plant macro- and micro-nutrients, which can improve soil fertility and plant productivity when applied to soil.

3.3 Pathogens

The OMRR prescribes testing and standards for fecal coliforms in both Class A and Class B biosolids. Based on the OMRR and the Land Application Guide, fecal coliform analysis was performed on the following samples:

- WWTP 1: In anticipation of Class A biosolids, seven composite representative samples were analyzed for fecal coliforms. The concentration of fecal coliforms must be <1,000 MPN/g of total solids (dry weight basis) for each sample to comply with the OMRR for Class A biosolids; and

- WWTP 2: In anticipation of Class B biosolids, seven grab samples were analyzed for fecal coliforms. The geometric mean of the samples must be < 2,000,000 MPN/g of total solids (dry weight basis) to comply with the OMRR for Class B biosolids.

Some jurisdictions also analyze other pathogens to assess pathogen reduction in organic matter, for example, *Salmonella* (CCME, 2012) and *E. coli* in Ontario. Therefore, the biosolids samples were also tested for *E. coli* and *Salmonella*. The results of the pathogen analysis from both WWTP 1 and WWTP 2 samples are shown in Table 9.

Table 9 Pathogen and Pathogen Indicator Analytical Results

Microbial Tests MPN/g of Total Solids (dry weight basis)	OMRR Biosolids Standards		CCME Compost Guidelines	WWTP 1 Biosolids Representative Samples	WWTP 2 Biosolids Geometric Mean of 7 Grab Samples
	Class A	Class B			
<i>E. coli</i>	NS	NS	NS	>6 817.8 >5 746.43 >5 606.27 >5 808.66 >6 094.7 >5 586.81 >5 625.87	107 229
Fecal Coliform Bacteria	<1000 for each of the 7 representative samples	<2 000 000 for geometric mean of 7 grab samples	<1000	>6 817.8 >5 746.43 >5 606.27 >5 808.66 >6 094.7 >5 586.81 >5 625.87	107 229
<i>Salmonella</i> ^a	NS	NS	<3 MPN/4 g	Not Isolated in 6 of the 7 Samples Isolated in 1 of the 7 samples, but not quantified	Isolated in each of the 7 Samples, but not quantified

Notes:

“NS” indicates that no standard exists

^a the units for the CCME Compost Guideline for salmonella differs from that in the rest of the table

Table 9 shows that none of the seven representative samples from WWTP 1 met the OMRR standards for fecal coliform bacteria for Class A biosolids. The geometric mean of the seven representative samples from WWTP 1 met the fecal coliform bacteria standard required in OMRR for Class B biosolids. The geometric mean of the concentrations of pathogens in samples from WWTP 2 in Table 9 met the OMRR standard for fecal coliform bacteria in Class B biosolids. Neither samples from WWTP 1 nor WWTP 2 met the CCME guideline for fecal coliform bacteria for compost, however, as previously discussed, this guideline is not directly applicable to biosolids.

Salmonella was isolated in one of the samples from WWTP 1. However, as will be discussed in Section 3.10 Laboratory Quality Control, the hold time for this test was exceeded and the results are inconclusive. *Salmonella* was isolated in each of the seven grab samples from WWTP 2.

The lab analysis indicated only isolation of *Salmonella* and not the identification of the species. Isolation of *Salmonella* refers to identifying its presence. When *Salmonella* is not isolated within a given sample, it means only that the microbe was not identified, and does not conclusively confirm the absence of *Salmonella* (Standard Method for Detection of Pathogenic Bacteria, 2007). For context, in the CFIA requirements for pathogens in compost (T-4-120 “Regulation of Compost under the Fertilizers Act and Regulations”), *Salmonella* must be absent (CFIA, 2018). However, the CFIA T-4-120 which is specific to compost is not directly applicable to biosolids, as previously discussed.

There are currently no standards for *E. coli* in the OMRR. For comparison, the New Zealand *E. coli* guideline for “Grade A” biosolids is < 100 MPN/g, ([NZWWA](#), 2003). “An ‘A’ grade biosolid is one in which pathogens and vector-attracting compounds, such as volatile solids, have been substantially reduced or removed by an “acceptable” pathogen reduction process”, (NZWWA, 2003). Each of the seven samples from WWTP 1 exceeded the New Zealand guideline. The *E. Coli* in each of the seven samples from WWTP 2 exceeded the New Zealand guideline for their “Grade A” biosolids.

The values for *E.Coli* and fecal coliforms are reported to be identical for all samples. This suggests that all the fecal coliforms detected in the samples were *E.Coli*. It should be noted that few strains of *E.Coli* are harmful.

3.4 Polycyclic Aromatic Hydrocarbons

Mean PAH results for biosolids from both WWTP 1 and WWTP 2 are presented in

Table 10.

Table 10 Polycyclic Aromatic Hydrocarbons in Biosolids

Polycyclic Aromatic Hydrocarbons (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids	Standards in the EU and European Countries
Acenaphthene	<0.05	<0.20	NS
Acenaphthylene	<0.20	<0.10	NS
Anthracene	0.0567	<0.20	NS
Benz(a)anthracene	0.087	<0.10	NS
Benzo(a)pyrene	<0.20	<0.20	Germany = 1 ^a France = 1-5 ^b
Benzo(b)fluoranthene	<0.20	<0.20	France = 2-5 ^b
Benzo(b+j+k)fluoranthene	<0.36	<0.28	NS
Benzo(g,h,i)perylene	<0.40	<0.30	NS
Benzo(k)fluoranthene	<0.30	<0.20	NS
Chrysene	0.117	<0.10	NS
Dibenz(a,h)anthracene	0.264	<0.30	NS
Fluoranthene	0.262	0.415	France = 4 ^b
Fluorene	<0.20	0.272	NS
Indeno(1,2,3-c,d)pyrene	<0.040	<2.0	NS
2-Methylnaphthalene	<0.040	0.209	NS
Naphthalene	0.066	<0.40	NS
Phenanthrene	0.252	0.800	NS
Pyrene	0.276	<0.40	NS
Total PAHs	1.38	1.34	Sweden = 3^b Denmark = 3^b Austria = 6^b

Notes:

“NS” indicates that no standard exists

^a draft only (Bundesgesetzblatt, 2017)

^b (National Research Council, 2002)

The results from the analysis of PAHs indicate that many of the PAHs analyzed were below the laboratory detection limit.

Table 10 also presents established standards for PAHs in four European jurisdictions and the European Union standards (National Research Council, 2002) for context.

For those PAHs detected, all mean concentrations were below the standards in other jurisdictions where they were identified.

3.5 Phenolics

Mean phenolics results for biosolids from both WWTP 1 and WWTP 2 are presented in Table 11.

Table 11 Select Phenols in Biosolids

Select Phenols (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids
4-Chloro-3-methylphenol	<0.20	<0.080
2-Chlorophenol	<3.0	<2.0
3-Chlorophenol	<0.030	<0.080
4-Chlorophenol	<0.030	<0.080
2,3-Dichlorophenol	<0.030	<0.080
2,4 & 2,5-Dichlorophenol	<0.50	<0.080
2,6-Dichlorophenol	<0.030	<0.080
3,4-Dichlorophenol	<0.080	<0.20
3,5-Dichlorophenol	<0.030	<0.10
2,4-Dimethylphenol	<2.0	<2.0
<i>o</i> -Cresol	<0.30	<0.20
<i>m</i> -Cresol	<0.20	<0.20
<i>p</i> -Cresol	126.3	<0.90
Pentachlorophenol	<0.030	<0.080
Phenol	2.09	15.8
2,3,4,5-Tetrachlorophenol	<0.030	<0.080
2,3,4,6-Tetrachlorophenol	<0.040	<0.080
2,3,5,6-Tetrachlorophenol	<0.040	<0.080
2,3,4-Trichlorophenol	<0.030	<0.080
2,3,5-Trichlorophenol	<0.040	<0.20
2,3,6-Trichlorophenol	<0.090	<0.60
2,4,5-Trichlorophenol	<0.050	<0.080
2,4,6-Trichlorophenol	<0.080	<0.080
3,4,5-Trichlorophenol	<0.20	<0.080

Most of the phenols analyzed were below the lab detection limit. The exceptions were *p*-Cresol in biosolids from WWTP 1, and phenols in biosolids from both WWTP 1 and WWTP 2. *p*-Cresol occurs naturally in some foods and beverages, as well as originates from anthropogenic sources. Phenols are commonly found in cosmetics and personal care products; some additional sources of phenols are listed in Table 2. A jurisdictional scan did not identify any standards for phenols in biosolids as a benchmark for comparison.

3.6 Phthalates

Table 12 shows the mean results of the semi-volatile organics test.

Table 12 Select Phthalates in Biosolids

Phthalates (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids	Standards in the EU and European Countries
Butylbenzyl phthalate	<2.0	<2.0	NS
Diethyl phthalate	<2.0	<2.0	NS
Dimethyl phthalate	<2.0	<2.0	NS
Di-n-butyl phthalate	<2.0	<2.0	NS
Di-n-Octyl phthalate	<20	<2.0	NS
bis(2-Ethylhexyl)phthalate	62.8	75.5	EU = 100 ^a Denmark = 50
Diisobutyl phthalate	<2.0	<2.0	NS

Notes:

“NS” signifies that no standard exists

^a draft only (National Research Council, 2002)

Most of phthalates reported in Table 12 were below the detection limit. The only phthalate detected was bis(2-ethylhexyl)phthalate in both biosolids from WWTP 1 and WWTP 2. Bis(2-ethylhexyl)phthalate is the most common phthalate and is used as a plasticizer and found in many households, and medical equipment. It is also used in the manufacture of PVC pipe, which is commonly used for irrigation piping.

Table 12 also presents the established standards for phthalates in Denmark and the European Union to provide context. The mean results for bis(2-ethylhexyl)phthalate at WWTP1 and WWTP 2 (62.8 µg/g and 75.5 µg/g, respectively) were higher than the standard for Denmark (50 µg/g), but below the EU draft standard (100 µg/g).

3.7 Polychlorinated Biphenyls

Mean PCB results for biosolids from both WWTP 1 and WWTP 2 are presented in Table 13.

Table 13 Polychlorinated Biphenyls in Biosolids

Polychlorinated biphenyl (as Aroclors) (µg/g)	WWTP 1 Biosolids	WWTP 2 Biosolids	Standards in the EU and European Countries (as congeners)
PCB-1016	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1221	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1232	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1242	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1248	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1254	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1260	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1262	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
PCB-1268	<0.030	<0.020	Germany = 0.2 ^b Germany = 0.1 ^a
Total PCB	<0.030	<0.020	Sweden = 0.4 ^b

Notes:

^a draft only (Bundesgesetzblatt, 2017), applicable to congeners 28, 52, 101, 138, 153, 180

^b (National Research Council, 2002), applicable to six specific congeners

PCBs were analyzed as individual Aroclor types and for total PCBs. Aroclor was the trade name of the commercial PCB mixture manufactured by the Monsanto Chemical Company and produced in the US from approximately 1930 to 1979. There are many types of Aroclors, and each has a distinguishing suffix number that indicates the degree of chlorination. The first two digits usually refer to the number of carbon atoms in the phenyl rings. The second two numbers indicate the percentage of chlorine by mass in the mixture. For example, Aroclor 1254 means that the mixture contains approximately 54% chlorine by weight. An exception is Aroclor 1016 which also has 12 carbon atoms but has 42% chlorine by mass.

The mean concentrations of the PCBs analyzed in both WWTP 1 and WWTP 2 were all below the detection limits which were below the standards identified in Germany. Standards for total PCBs were found in the European Union and several European countries including Austria, Czech Republic, France, Germany, and Sweden. However, these standards are not directly comparable to the Aroclor groupings given that these European standards are for congeners not necessarily equivalent to the Aroclor type.

3.8 Polychlorinated Dioxins and Furans

Toxic Equivalents (TEQs) are used to report the toxicity-weighted masses of mixtures of dioxins. Within the TEQ method, each dioxin is assigned a Toxic Equivalency Factor (TEF) which denotes a given dioxin compound's toxicity relative to that of the reference dioxin 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD). The TEQ is the sum of the product of 47 congeners of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) and their respective TEFs. There are several different TEF standards set by different agencies. The TEQs in Table 14 were calculated using the International Toxic Equivalents for dioxins and furans (I-TEQ) (NATO, 1988). The I-TEF set is recent and one of the most commonly used set of TEF. "Upper bound" indicates that any dioxins and furans that were reported as below detection limit, were assumed to be equal to the detection limit for the purposes of the calculation. This is a conservative approach to calculating the TEQ.

The mean I-TEQ, based on the analysis of dioxins and furans for biosolids from both WWTP 1 and WWTP 2, are presented in Table 14.

Table 14 Dioxin and Furan Analytical Results in Biosolids

International Toxic Equivalent (pg/g)	WWTP 1 Biosolids I-TEQ	WWTP 2 Biosolids I-TEQ	Standards in European Countries I-TEQ
Upper Bound PCDD/PCDF I-TEQ	5.29	4.74	Austria = 50 – 100 ^a Germany = 100 ^a

Notes:

^a (National Research Council, 2002)

Table 14 presents the established standards for I-TEQ in Austria and Germany to provide context (National Research Council, 2002). The upper bound I-TEQs detected in the biosolids sampled from WWTP 1 and WWTP 2 were below established and proposed standards for dioxins and furans.

For both biosolids sources, the majority of the PCDD and PCDF congeners were detectable. However, the most toxic, 2,3,7,8-TCDD was not detectable in the biosolids from either WWTP 1 or WWTP 2.

3.9 Contaminants of Emerging Concern

The risk assessment in the Kennedy/Jenks report completed for Metro Vancouver, determined the concentrations of triclosan, ciprofloxacin, azithromycin, and BDE-209 (deca-BDE) in biosolids that pose minimal risk for two biosolids exposure scenarios: adult hikers and occupational workers (Kennedy/Jenks, 2017). These modelled scenarios represent possible exposure pathways to land applied biosolids and assume worst case situations, so to be protective of the populations assessed.

The occupational worker scenario risk-based acceptable concentrations outlined in the Kennedy/Jenks' report were compared to the CEC results from WWTP 1 and WWTP 2 because it was the more

conservative of the two modelled exposure scenarios. This scenario represents an 80 kg adult whose employment is the land application of biosolids for 25 years. The scenario assumed that 100 mg of biosolids were unintentionally ingested by the occupational worker daily, in addition to dermal biosolids contact. A jurisdictional scan did not identify standards for the other CECs that could be compared to the results from WWTP 1 and WWTP 2.

The risk-based concentrations are shown in Table 15, along with the mean concentration of the CECs analyzed in biosolids from WWTP 1 and WWTP 2 and the highest detected concentration from three WWTPs in Metro Vancouver Regional District: Annacis Island, Lulu Island, and Lions Gate (Kennedy/Jenks, 2017).

Table 15 Contaminants of Emerging Concern in Biosolids

Contaminants of Emerging Concern (ng/g)		Risk-Based Acceptable Concentrations in Biosolids for Occupational Workers ^a	WWTP 1 Biosolids	WWTP 2 Biosolids	Highest Reported Concentration in Metro Van Biosolids ^b
Pharmaceuticals and Personal Care Products	Carbamazepine	-	179	201	-
	Triclosan	93 925 000	17 267	10 463	19 400
	Triclocarban	-	4 213	5 157	-
	Naproxen	-	9.04	6.2	-
	Propranolol	-	73.7	40.6	-
	Ciprofloxacin	522 000	901	4 560	4 400
	Azithromycin	1 305 000	20.3	242	582
Polybrominated Diphenyl Ethers (PBDEs)	BDE-47	-	336	182	-
	BDE-99	-	343	172	-
	BDE-209	4 899 000	1 773	336	900

Notes:

^a based on risk assessment conducted for Metro Vancouver (Kennedy/Jenks, 2017). The occupational worker scenario represents a possible exposure pathway specific to land applied biosolids originating from Metro Vancouver. It assumes exposure 220 days/year.

^b represents the highest detected concentration from three WWTPs in Metro Vancouver Regional District: Annacis Island, Lulu Island, and Lions Gate, (Kennedy/Jenks, 2017)

The differences in the mean results from the biosolids from WWTP 1 and WWTP 2 may be partly attributed to different influent sources and different production processes. The highest reported concentrations from the Metro Vancouver biosolids appear to be higher than the findings from WWTP 1 and WWTP 2 except for Ciprofloxacin, again likely due to different influent sources.

Mean concentrations of Triclosan, Ciprofloxacin, Azithromycin and BDE-209 were below the risk-based acceptable concentrations reported by Kennedy/Jenks (2017) for occupational workers in biosolids. As listed in Table 3, Triclosan is commonly used in personal care products, such as hand soaps and

toothpaste; Azithromycin and Ciprofloxacin are used in antibiotics; while BDE-209 is broadly used as a flame retardant.

The CEC results reported in Table 15 are those that were recommended for analysis by the TWG in Table 1. The biosolids concentrations of many more CECs are presented in Appendix 1. This appendix compares the CEC results from WWTP 1 to WWTP 2.

3.10 Laboratory Quality Control

ALS quality control reports indicated that the hold time of 24 hours for *Salmonella* analysis was exceeded for the samples from WWTP 1 which had an actual hold time of 45 hours. Based on this, the *Salmonella* sample results from WWTP 1 in Table 9 are inconclusive. All hold times were met for the samples collected from WWTP 2.

The laboratory's quality control report also reported multiple method blank results outside of their acceptable thresholds; however, ALS noted that no impact to data quality was expected.

4.0 Conclusions

This sampling program successfully collected biosolids samples in November 2016 from two wastewater treatment plants (WWTPs) in BC for analysis of parameters regulated under the OMRR and additional parameters identified by the TWG.

Biosolids samples from both WWTPs met the OMRR-regulated metal standards for Class A and Class B biosolids provided in Schedule 4 of the OMRR. While the study aimed to assess both Class A and Class B biosolids, none of the seven representative samples from WWTP 1 met the OMRR standards for fecal coliform levels for Class A biosolids. It is worth noting that the geometric mean of the samples collected from WWTP 1 met the fecal coliform levels required in OMRR for Class B biosolids.

Salmonella was isolated in one of the representative samples from WWTP 1 and presumed positive in three of the remaining six samples, but numbers of salmonella were not quantified. The hold time for this test was exceeded and therefore the *Salmonella* test results from WWTP 1 are inconclusive. The concentrations of pathogens from WWTP 2 met the OMRR standard for fecal coliforms for Class B biosolids. *Salmonella* was detected in the biosolids from WWTP 2 but was not quantified.

With respect to POPs, which are not regulated under the OMRR, most of the PAHs, phenolics and phthalates analyzed from both WWTP 1 and WWTP 2 were below the lab detection limits. Furthermore, the concentrations of the PCBs analyzed were all below the detection limits.

For both WWTP 1 and WWTP 2 biosolids samples, the majority of the PCDD and PCDF congeners were detected. However, the most toxic, 2,3,7,8-TCDD was not detected. The mean I-TEQ for each of the two WWTPs were below the European standards that were identified.

There is a lack of biosolids standards in other jurisdictions to provide context for many of the pharmaceutical, personal care products and polybrominated diphenyl ethers results. The results from this study were compared to the acceptable concentrations to an occupational worker, as determined by risk assessment for Metro Vancouver (Kennedy/Jenks, 2017), to provide context only. Concentrations of CECs were below acceptable concentrations where risk assessment limits were provided (Kennedy/Jenks, 2017).

This biosolids sampling project had a very limited number of samples, included only two facilities, and was conducted over the course of one day at each facility. The results provide a snapshot in time and would be expected to vary if repeated with a greater number of samples at different facilities. The chemicals present and their concentrations in biosolids can vary temporally; therefore, sampling more frequently and over a longer time period would provide a broader picture of the quality of biosolids. It is not possible to generalize these results to determine biosolids quality at other facilities, or at the same facilities at other times.

5.0 References

BC Ministry of Environment. 2008. Land Application Guidelines for the Organic Matter Recycling Regulation and the Soil Amendment Code of Practice: Best Management Practices. Produced by SYLVIS, document # 758-08. Victoria BC. March 2008. Available online at: <http://www2.gov.bc.ca/assets/gov/environment/waste-management/recycling/landappguidelines.pdf>

BC Ministry of the Environment and Climate Change Strategy. 2016a. A literature review of risks relevant to the use of biosolids and compost from biosolids with relevance to the Nicola Valley, BC Contract #CS16JHQ112. LRCS Land Resource Consulting Services. June 16, 2016. (https://www2.gov.bc.ca/assets/gov/environment/waste-management/organic-waste/biosolids/literature_review_cover_memo.pdf)

BC Ministry of the Environment and Climate Change Strategy. 2016b. Biosolids Sampling Project – Results and Analysis. October 3, 2016

Bundesgesetzblatt Jahrgang 2017 Teil Nr.65, ausgegeben zu Bonn am 2. Oktober 2017. Ein Service des Bundesministeriums der Justiz in Zusammenarbeit mit der juris GmbH. 2012. Verordnung über das Inverkehrbringen von Düngemitteln, Bodenhilfsstoffen, Kultursubstraten und Pflanzenhilfsmitteln (Düngemittelverordnung - DüMV). https://www.lfl.bayern.de/mam/cms07/zentrale_analytik/dateien/d__ngemittelverordnung.pdf

Canadian Council of Ministers of the Environment. 1996. "Support Document for Compost Quality Criteria [National Standard of Canada CAN, BNQ 0413200, Canadian Council of Ministers of the Environment (CCME) Guidelines and Agriculture and Agri-Food Canada (AAFC) Criteria]", Final Version (1996, March).

Canadian Council of Ministers of the Environment. 2005. Guidelines for Compost Quality. ISBN 1-896997-60-0 (PN 1340). CCME, Winnipeg, MB.

Canadian Council of Ministers of the Environment. 2009. Emerging substances of concern in biosolids: Concentrations and effects of treatment processes. Rep. CCME Project # 447-2009 (PN 1440). CCME, Winnipeg, MB.

Canadian Council of Ministers of the Environment. 2012. Guidance Document for the Beneficial Use of Municipal Biosolids, Municipal Sludge and Treated Septage (PN 1473). ISBN 978-1-896997-85-8.

Canadian Food Inspection Agency. 1997. Trade Memorandum T-4-93, Safety Guidelines for Fertilizers and Supplements. Government of Canada. September 20, 2017

Canadian Food Inspection Agency. 2018. Trade Memorandum T-4-120, Regulation of Compost under the Fertilizers Act and Regulations. Government of Canada. May 14, 2018

Canadian Municipal Water Consortium, Canadian Water Network. 2015. Risks Associated with Application of Municipal Biosolids to Agriculture Lands in a Canadian Context: Literature Review. Ryerson University, Toronto, ON.

Guerra, P., S. Kleywegt, M. Payne, M. L. Svoboda, H. Lee, E. Reiner, T. Kolic, C. Metcalfe, and S. A. Smyth. 2015. *Occurrence and Fate of Trace Contaminants during Aerobic and Anaerobic Sludge Digestion and Dewatering*. J. Environ. Qual. 44:1193-1200. doi:10.2134/jeq2015.01.0010

International Joint Commission. 2009. *The Challenge of Substance of Emerging Concern in the Great Lakes Basin: A review of chemicals policies and programs in Canada and the United States*, prepared by the Canadian Environmental Law Association and Lowell Center for Sustainable Production. ISBN: 978-1-926602-22-6

Kennedy/Jenks Consultants. 2017. *Biosolids Risk Assessment for Metro Vancouver*. Prepared for Metro Vancouver. May 26, 2017.

Langdon, K; Warne, M. S. J.; Smernik, R.J., Shareef, A. and Kookan, R.S. 2011. Selected personal care products and endocrine disruptors in biosolids: An Australia-wide survey. *Science of The Total Environment Journal*. 409 (6):1075–1081

Lorenzen, A., K. Burnison, M. Servos and E. Topp. 2006. Persistence of endocrine disrupting chemicals in agricultural soils. *Journal of Environmental Engineering and Science* 5(3): 211-219.

National Research Council, 2002. *Biosolids applied to land: Advancing Standard and Practices*, Committee on Toxicants and Pathogens in Biosolids Applied to Land, Board on Environmental Studies and Toxicology, Division on Earth and Life Studies, National Academy Press, Washington DC.

NATO (North Atlantic Treaty Organization). 1988. *Pilot study on international information exchange on dioxins and related compounds. Emissions of dioxins and related compounds from combustion and incineration sources*. North Atlantic Treaty Organization, Committee on the Challenges of Modern Society. Report #172. August 1988.

NZWWA (New Zealand Water & Wastes Association). 2003. *Guidelines for the Safe Application of Biosolids to Land in New Zealand*.
https://www.waternz.org.nz/Folder?Action=View%20File&Folder_id=101&File=biosolids_guidelines.pdf

Salveson, A., E. Rauch-Williams, J. Drewes, D. Drury, D. McAvoy, S. Snyder. 2012. *Trace Organic Compound Indicator Removal During Conventional Wastewater Treatment*”, CEC4R08, Alexandria, VA.

Standard Methods for the Examination of Water and Wastewater. 2007. *9260 Detection of Pathogenic Bacteria (2007)*, DOI: 10.2105/SMWW.2882.201

Appendix 1 - Additional Contaminants of Emerging Concern Results

CEC results discussed in the report were limited to those recommended by the TWG. Table A1-1 provides a complete list of the CECs analyzed in this study.

Table A1-1. Mean concentrations of additional CEC results for biosolids from WWPT 1 and WWTP 2

Contaminants of Emerging Concern (units vary)	Risk-Based Acceptable Concentrations in Biosolids for Occupational Workers ^a	WWTP 1 Biosolids	WWTP 2 Biosolids	Highest Reported Concentration in Metro Van Biosolids ^b
Pharmaceuticals and Personal Care Products (ng/g)				
1,7-Dimethylxanthine	-	<50.3	<56.2	-
10-hydroxy-amitriptyline	-	23.8	15	-
2-Hydroxy-ibuprofen	-	82.2	76.6	-
Acetaminophen	3 392 000	<12.6	<14.1	<20
Alprazolam	-	0.286	<0.281	-
Amitriptyline	-	634	215	-
Amlodipine	-	49.6	267	-
Azithromycin	1 305 000	20.3	242	582
Benzoylcegonine	-	0.54	0.483*	-
Benzotropine	-	<2.1 ^D	<2.34	-
Betamethasone	-	<6.29	<7.03	-
Bisphenol A	62 617 000	<419	2 057	2 200
Caffeine	-	83.1	18.0	-
Carbadox	-	<1.26	<1.41	-
Carbamazepine	-	179	201	-
Cefotaxime	-	<13.5	<14.6	-
Ciprofloxacin	522 000	901	4 560	4 400
Clarithromycin	-	85	47	-
Clinafloxacin	-	<19.1	<12.9	-
Cloxacillin	-	<3.14 ^H	<2.82 ^H	-
Cocaine	-	0.91*	2.96	-
DEET	-	12	10.3	-
Dehydronifedipine	-	1.18	0.593	-
Desmethyldiltiazem	-	2.58	1.2	-
Diazepam	-	1.4	0.529*	-
Digoxigenin	-	<96.9	<78.1	-
Digoxin	-	15.8*	<22.3	-
Diltiazem	-	7.93	2.51	-
Diphenhydramine	-	535	424	-
Enrofloxacin	-	2.88*	10.6	-
Erythromycin-H2O	1 305 000	16.3	5.85	63
Flumequine	-	<1.26	<1.41	-
Fluocinonide	-	<5.03	<5.62	-
Fluoxetine	52 000	111	138	164
Fluticasone propionate	-	<4.58	<3.41	-
Furosemide	-	146	37.8*	-
Gemfibrozil	-	5.12	24	-
Glipizide	-	<5.03	<5.62	-
Glyburide	-	3.6*	13.8	-
Hydrochlorothiazide	-	<16.8	<18.7	-
Hydrocortisone	-	<56.6	<153	-
Ibuprofen	522 000	184	139	300
Lincomycin	-	<2.51	<2.81	-
Lomefloxacin	-	<15.7	<12.3	-
Meprobamate	-	<3.42	<3.75	-
Methylprednisolone	-	<3.35	<3.75	-

Contaminants of Emerging Concern (units vary)	Risk-Based Acceptable Concentrations in Biosolids for Occupational Workers ^a	WWTP 1 Biosolids	WWTP 2 Biosolids	Highest Reported Concentration in Metro Van Biosolids ^b
Metoprolol	-	21.8	17.5	-
Miconazole	-	<1.26	<1.42	-
Naproxen	-	9.04	6.2	-
Norfloxacin	-	26.3	441	-
Norfluoxetine	-	22	47.2	-
Norgestimate	-	<12.2	<7.94	-
Norverapamil	-	71.3	34.8	-
Ofloxacin	1 044 000	23.7	704	800
Ormetoprim	-	<0.503	<0.562	-
Oxacillin	-	<3 ^H	<2.81	-
Oxolinic Acid	-	<30.5	<26.6	-
Paroxetine	-	60.9	90	-
Penicillin G	-	<2.51 ^H	<2.81	-
Penicillin V	-	<2.51	<2.81	-
Prednisolone	-	<30.6 ^D	<5.62	-
Prednisone	-	<83.8 ^D	<18.7	-
Promethazine	-	1.68 ^D	1.8*	-
Propoxyphene	-	<1.26 ^D	<1.34	-
Propranolol	-	73.7	40.6	-
Roxithromycin	-	<3.34	13.6	-
Sarafloxacin	-	<12.6	<15.3	-
Sertraline	-	509	85.5	-
Simvastatin	-	<83.8 ^D	<89.4 ^D	-
Sulfachloropyridazine	-	<1.26	<1.41	-
Sulfadiazine	-	<1.26	<1.41	-
Sulfadimethoxine	-	<1.54	<0.365	-
Sulfamerazine	-	<1.95	<1.51	-
Sulfamethazine	-	<7.25	<2.74	-
Sulfamethizole	-	<0.503	<0.562	-
Sulfamethoxazole	2 087 000	<3.29	<1.15	<0.6
Sulfanilamide	-	<38.5	78	-
Sulfathiazole	-	<1.49	<1.41	-
Theophylline	-	<206	<214	-
Thiabendazole	-	5.6	47	-
Trenbolone	-	<3.35	<3.75	-
Trenbolone acetate	-	<0.251	<0.281	-
Triclocarban	-	4 213	5 157	-
Triclosan	93 925 000	17 267	10 463	19 400
Trimethoprim	-	<1.26	<1.41	-
Tylosin	-	<10.9	<6.41	-
Valsartan	-	53	85.0	-
Verapamil	-	38.7	23	-
Virginiamycin M1	-	<4.26	<4.57	-
Warfarin	-	<1.26	<1.41	-
Polybrominated Diphenyl Ethers (PBDEs)(pg/g)				
BDE 10	-	<4.7	<2.9	-
BDE 100	-	71 433	36 300	-
BDE 105	-	273	90.9	-
BDE 116	-	<44	<51	-
BDE 118	-	1 714	877	-
BDE 119/120	-	1 099	510	-
BDE 12/13	-	105	77.8	-
BDE 126	-	104	32	-
BDE 128	-	372	117	-
BDE 138/166	-	2 897	1 279	-
BDE 140	-	1 653	648	-

Contaminants of Emerging Concern (units vary)	Risk-Based Acceptable Concentrations in Biosolids for Occupational Workers ^a	WWTP 1 Biosolids	WWTP 2 Biosolids	Highest Reported Concentration in Metro Van Biosolids ^b
BDE 15	-	354	1 903	-
BDE 153	-	28 400	14 433	-
BDE 154	-	26 867	13 000	-
BDE 155	-	1 750	878	-
BDE 156	-	105	29	-
BDE 17/25	-	3 123	2 013	-
BDE 181	-	256	236	-
BDE 183	-	5 850	3 380	-
BDE 184	-	245	140	-
BDE 190	-	227	166	-
BDE 191	-	136	76	-
BDE 196	-	8 867	2 183	-
BDE 197	-	15 670	4 483	-
BDE 203	-	21 867	5 643	-
BDE 206	-	62 533	12 400	-
BDE 207	-	31 267	9 917	-
BDE 208	-	27 800	8 866	-
BDE 209	4 899 000	1 773 333	336 000	900
BDE 28/33	-	7 550	4 733	-
BDE 30	-	4.6	1.8	-
BDE 32	-	23	27.3	-
BDE 35	-	44	44.6	-
BDE 37	-	174	199	-
BDE 47	-	336 333	181 667	-
BDE 49	-	12 467	6 817	-
BDE 51	-	1 125	833	-
BDE 66	-	7 890	4 313	-
BDE 7	-	73.9	38.6	-
BDE 71	-	1 313	648	-
BDE 75	-	661	350	-
BDE 77	-	64.1	47	-
BDE 79	-	<17	<6	-
BDE 8/11	-	107	105	-
BDE 85	-	14 433	7 370	-
BDE 99	-	342 667	172 333	-
HBB	-	254	122	-
PBEB	-	93.3	94	-

Notes:

^a based on risk assessment conducted for Metro Vancouver (Kennedy/Jenks, 2017). The occupational worker scenario represents a possible specific exposure pathway to land applied biosolids originating from Metro Vancouver. This scenario represents an 80 kg adult whose employment is the land application of biosolids for 25 years, working 220 days/year. It assumes that 100 mg of biosolids were unintentionally ingested by the occupational worker daily, in addition to dermal biosolids contact.

^b represents the highest detected concentration in 2017, from three WWTPs in Metro Vancouver Regional District: Annacis Island, Lulu Island, and Lions Gate, (Kennedy/Jenks, 2017)

* represents only one sample result (not an average)

- none reported

^D lab reported dilution data

^H lab reported that concentration was estimated