EMERGING SUBSTANCES OF CONCERN IN BIOSOLIDS: CONCENTRATIONS AND EFFECTS OF TREATMENT PROCESSES

Final Report – Field Sampling Program

CCME Project # 447-2009

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EXECUTIVE SUMMARY

Background to Study

The Biosolids Task Group (BTG) established by the Canadian Council of Ministers of the Environment (CCME) is mandated to study and make recommendations on biosolids management at the national level. Wastewater treatment facilities (WWTF) across Canada generate residual wastewater solids (sludge) that may require further treatment for safeguarding human health and the environment prior to their use or disposal. When treated sludge quality is appropriate for land application, it is called biosolids. Options for managing biosolids include disposal (e.g. landfill, incineration), energy recovery (e.g. thermal treatment), agricultural application as a nutrient, land reclamation and remediation (e.g. mines and quarries), forestry, and commercial product recovery (compost and pellets).

The end use of the biosolids is often governed by the constituent quality of the biosolids, such as nutrients, metals, pathogens and trace constituents. Land application of biosolids has been practiced in Canada for many decades. Currently, 11 inorganic trace/microconstituents, such as cadmium, lead and mercury, and pathogen/pathogen standards are monitored in biosolids on a routine basis, prior to land application. Other constituents thought possibly to be of concern in biosolids in the 1990's, such as PCBs, dioxins and furans, and polyaromatic hydrocarbons (PAHs) were extensively studied at that time. These classes of compounds were included in the literature review that accompanied the field study (Hydromantis *et al*., 2009). As these studies found low concentrations in biosolids, biosolids are not being tested for these constituents by most jurisdictions. Consequently, they were not included in the potential list of target analytes in this study.

At present, the risks associated with detecting in biosolids certain classes of micro-constituents (termed Emerging Substances of Concern (ESOC) herein), which include an array of pharmaceuticals, personal care products and industrial contaminants (such as plasticizers, surfactants and brominated flame retardants) are not well understood. While there is some documentation of ESOC in biosolids, no focused study has been completed yet on an inventory of ESOC in Canadian biosolids. Consequently, CCME issued a Request for Proposals to document the occurrence of ESOC in biosolids and septage; to conduct a targeted sampling program at selected representative Canadian wastewater treatment plants to provide a focused Canadian study and an inventory of ESOC in Canadian biosolids; and to assess the removal efficiencies of various treatment processes, if any. The sampling study results will contribute to the knowledge basis which will assist CCME in evaluating and managing the risks associated with ESOC in biosolids with respect to managed land application, land reclamation, and production of commercial and soil amendments.

Study Objectives

The objectives of the whole project are to:

1. Prepare a comprehensive review of research on ESOC in biosolids within Canada and elsewhere based on technical literature and wastewater sector contacts;

2. Complete a field survey and analyze Canadian biosolids and septage samples with respect to ESOC;
3. Identify the occurrence and concentration ranges of those ESOC in Canadian biosolids within the scope of this study;
4. Review and recommend treatment technologies that mitigate ESOC concentrations in biosolids;
5. Suggest Best Management Practices (BMPs);
6. Identify knowledge gaps and research needs for ESOC with respect to biosolids;
7. Produce a final report of the project to the Contract and Project Authorities.

A previous report for this project consisted of a detailed literature review of the occurrence of ESOC in municipal wastewater residual solids and biosolids from different treatment processes (Hydromantis *et al*., 2009) and corresponded to Project Objective. The following report, responding to Objectives 2 through 7, constitutes the results obtained from a detailed filed sampling program for the characterization of ESOC in residual solids and biosolids from 11 wastewater treatment facilities across Canada.

Biosolids and Sludge Treatment Processes Studied

The processes investigated in this report are summarised in **Table ES-1**. Some processes integrate many process units (e.g. biological treatment + dewatering; liming + composting) while others only cover a specific process unit (e.g.: geotextile bag dewatering, filter press dewatering). **Table ES-1** also indicates the class of stabilisation (A or B) of the final biosolids produced from these treatments according to the U.S. Environmental Protection Agency (EPA) criteria for pathogen reduction. According to U.S. EPA criteria, "treated" dewatered sludges that do not meet Class A or Class B standards are not termed "biosolids", but treated sludges. The residual wastewater solids delivered to the treatment processes studied are referred to in this report as "feed sludge".

Table ES-1. Sludge and Biosolids Treatment Processes Investigated in This Study

Process	Number in Study
Autothermal thermophilic aerobic digestion	1
Mesophilic anaerobic digestion	2
Composting	3
Alkaline stabilisation	1
Thermal drying (pelletisation)	1
Geotextile bag dewatering	1
Filter press dewatering	2

Eleven sites were selected by the BTG of CCME based on a number of considerations including the implementation of a biosolids land application program at the site, plant capacity, geographical location, and type of biosolids treatment process. Plant hydraulic capacity and extent of municipal urbanization were not identified as primary factors of interest by the BTG. Site information is provided in **Table ES-2**.

Table ES-2. Characteristics of Biosolids Sampling Sites

Municipality	Region of Canada	Wastewater Treatment	Solids Treatment Process		Comment
			Feed Sludge	Final Solids or Biosolids	
Gander, NL	Atlantic	Hydrodynamic separation	Raw solids from hydrodynamic separator	Dewatered solids from belt filter press	No stabilisation process applied
Moncton, NB	Atlantic	Primary treatment	partially stabilised biosolids from lime treatment	Composted biosolids	
N-Viro Facility, Halifax, NS	Atlantic	see "comment"	combined wastewater residuals from several locations	Alkaline-stabilised biosolids	Location does not treat wastewater, but accepts residuals from off-site
Saguenay, QC	Central	Activated sludge	Waste activated sludge (WAS)	Dewatering of WAS from belt filter press	No stabilisation process applied
Gatineau Valley, QC	Central	Septage receiving and dewatering	Dewatered septage	Composted dewatered septage with added wood chips	No stabilisation process applied
Eganville, ON	Central	Extended aeration	Waste activated sludge (WAS) or septage (separate feed streams)	Combination of aerobic digestion of WAS followed by geo-tube dewatering	Also process septage separately in geotextile bag filters
Smiths Falls, ON	Central	Conventional activated sludge plus filtration	Dewatered primary plus waste activated sludges	Heat-dried (pellets)	
Saskatoon, SK	West	Biological nutrient removal	Combined primary and secondary sludges	Liquid mesophilic anaerobically digested biosolids	
Prince Albert, SK	West	Conventional activated sludge	Dewatered primary plus waste activated sludge	Composted biosolids	
Red Deer, AB	West	Biological nutrient removal	Combined primary and secondary sludges	Mesophilic anaerobically digested biosolids plus lagoon dewatering	
Salmon Arm, BC	West	Biological nutrient removal	Combined primary and secondary sludges	Dewatered autothermal aerobically digested biosolids	

Sampling Procedures

Samples of both feed sludge before the treatment process and the resulting dewatered solids or biosolids were collected between July and November of 2009 on three separate occasions at nine of the eleven targeted Canadian municipalities; at the remaining two sites, two rounds of samples were collected rather than three due to mechanical problems with the biosolids treatment process or due to funding agreements. To account for potential losses of the ESOC in process sidestreams, such as digester supernatant or leachate from composting pads, samples of these process sidestreams were also collected concurrently and analysed in an attempt to better close mass balances around the different biosolids treatment processes.

At the treatment plant sites, sample collection devices such as spoons, rods and scoops were made of stainless steel, glass or Teflon®. Pre-cleaned sample containers were shipped from the analytical laboratories to the sites in the return shipment coolers along with sample packing instructions, gel-type freezer packages, additional packing materials and chain-of-custody forms.

From the outset, the sampling program was to be conducted by operating plant staff at each site. To ensure proper procedures for sample collection and shipment to analytical laboratories were followed, a series of internet-based presentations was provided to the operating staff. Topics covered included definition of sampling terms, acceptable materials for sampling devices, compositing of grab samples from different process streams or locations in stockpiles, proper packing of coolers used for shipment, shipping logistics and health and safety issues in sample collection. Telephone and email were also used to respond to immediate questions from the field staff during sample collection and shipment. Samples were shipped from the collection sites by overnight courier to the laboratories, with shipments no later than Thursday afternoon to avoid sitting in courier depots over weekends. On arrival at the laboratories, samples were processed and refrigerated or frozen until analysis.

Selection of Analytes

The potential list of classes of ESOC that might be analysed in this study is extensive. The literature review produced as part of this report (Hydromantis *et al*., 2009) identified many types of ESOC which have been studied, including brominated flame retardants (polybrominated diphenyl ethers and others) plastics and plasticizer agents, alkylphenols and their ethoxylates, linear alkylbenzene sulphonates, perfluorinated organic compounds, natural and synthetic hormone, pharmaceuticals, synthetic musk fragrances, antibacterial compounds, quaternary ammonium compounds, and volatile methyl siloxanes.

The literature review examined the occurrence and removal of ESOC in biosolids treatment processes, but did not examine any human health or environmental risks due to ESOC present in biosolids. It was useful guide in the selection of the analytes in that it identified the near-complete lack of data on the fate of ESOC in treatment processes other than anaerobic digestion.

Three main considerations were responsible for the selection of the ESOC targeted as analytes in this study, namely:

(1) their potential environmental and human health significance based on other literature reviews and professional judgement,
(2) the availability of suitable analytical methods to determine concentrations in sludges and biosolids, which are difficult matrices for analysis of ESOC in the ng/l to µg/L concentration range, and
(3) budgetary constraints

Of these considerations, budgetary constraints had the greatest impact in narrowing the potential ESOC test groups to be analysed. The majority of the pharmaceutical that can be detected in wastewater and sludge matrices can be captured in five different analytical lists (Grace, 2009), with each list associated with a unit cost. Based on discussions with the analytical laboratories involved in the study (AXYS Analytical Services, ALS Analytical Group and Trent University), a proposed list of target analytes was developed for the project that was deemed to meet the three considerations outlined above. The list can in general be considered to include 57 pharmaceutical compounds, 3 alkylphenolic compounds (including Bisphenol A), 11 synthetic musk fragrances, 11 metals and macronutrients including forms of nitrogen and phosphorus. Although it would clearly be desirable to include additional test groups in the sampling program, budgetary limitations precluded this. The finalised list of analytes for the study is indicated in **Table ES-3**.

Table ES-3. List of Target Analytes for Biosolids Treatment Study

Pharmaceutical Test Group 1 (Acid Positive Pharmaceuticals)		**Pharmaceutical Test Group 2 (Acid Negative Pharmaceuticals)**	**Fragrances**
Acetaminophen	Norgestimate	Furosemide	DPMI
Azithromycin	Ofloxacin	Gemfibrozil	ADBI
Caffeine	Ormetoprim	Glipizide	AHDI
Carbadox	Oxacillin	Glyburide	HHCB
Carbamazepine	Oxolinic Acid	Hydrochlorothiazide	AHTN
Cefotaxime	Penicillin G	2-Hydroxy-ibuprofen	ATII
Ciprofloxacin	Penicillin V	Ibuprofen	Musk Moskene
Clarithromycin	Roxithromycin	Naproxen	Musk Tibetene
Clinafloxacin	Sarafloxacin	Triclocarban	Musk Ketone
Cloxacillin	Sulfachloropyridazine	Triclosan	Musk Ambrette
Dehydronifedipine	Sulfadiazine	Warfarin	Musk Xylene
Diphenhydramine	Sulfadimethoxine	**Alkylphenolics**	**Metals**
Diltiazem	Sulfamerazine	Bisphenol A	Arsenic (As)-Total
Digoxin	Sulfamethazine	Octylphenol	Cadmium (Cd)-Total
Digoxigenin	Sulfamethizole	Nonylphenol	Chromium (Cr)-Total
Enrofloxacin	Sulfamethoxazole		Cobalt (Co)-Total
Erythromycin-H_2O	Sulfanilamide		Copper (Cu)- Total
Flumequine	Sulfathiazole		Lead (Pb)-Total
Fluoxetine	Thiabendazole		Mercury (Hg)
Lincomycin	Trimethoprim		Molybdenum (Mo)-Total
Lomefloxacin	Tylosin		Nickel (Ni)-Total
Miconazole	Virginiamycin		Selenium (Se)-Total
Norfloxacin	1,7-Dimethylxanthine		Zinc (Zn)-Total

Pharmaceutical Test Group 1 includes a number of frequently detected antibiotics and other relevant pharmaceutical groups (fluoroquinolones, macrolides and sulfa compounds, as well as the anti-convulsives carbamazepine and trimethoprim, the analgesic acetaminophen, and stimulants such as caffeine and diphenhydramine). As such Test Group 1 encompasses a range of compounds in biosolids that could potentially be of environmental significance. Test Group 2 is a shorter list of pharmaceuticals, but includes a number of frequently detected and widely used pharmaceuticals including the non-steroidal anti-inflammatory drugs ibuprofen and naproxen, the anti-bacterial compounds triclosan and triclocarban, and the lipid regulator gemfibozil. Both Tests Groups 1 and 2 are acidic pharmaceuticals based on the extraction procedure for analysis. The difference between the Test Groups results from the analytical technique involving positive electrospray (Group 1) or negative electrospray (Group2) ionisation mass spectrometry.

Three different laboratories were involved in the analytical program because of the diversity of the target analytes. AXYS Analytical Services Ltd. in Sydney BC performed the analysis of acid positive and acid negative pharmaceutical compounds using EPA Method 1694 (EPA, 2007). The Worsfold Water Quality Centre of Trent University in Peterborough, ON analyzed the samples for synthetic musk fragrances and alkylphenolic compounds, including Bisphenol A by liquid chromatography followed by tandem mass spectrometry (LC/MS/MS). For logistical reasons, samples destined for analysis by the Trent University laboratory were shipped from the sites to the AXYS laboratory, and then to the Trent University laboratory. ALS Laboratory Group of Waterloo, ON completed the analyses of target metals and nutrients.

Results of Sampling Program

Metals

The metals analysed are not technically considered as ESOC since they have been widely documented in the literature and regulated by provincial standards for land application. Metals were analyzed only in the first round of biosolids collected at the different survey sites to provide a high-level comparison with historical data. The results are presented in **Table ES-4**. Median values of both detected and non-detected metal concentration in this study are all below limits used by jurisdictions in Canada for biosolids. For example, limits for metals in unrestricted use of compost (among the most stringent) are provided in the table for comparison. Cadmium was detected in only two of the biosolids samples from the eleven sites. Copper, mercury and zinc were found in biosolids samples from all eleven sites. Although maximum concentration values of copper, mercury and molybdenum exceeded the limits for unrestricted use at 2, 4 and 1 sites, respectively, these elevated concentrations may still be acceptable for other beneficial uses for soil amendment.

Most metals are conservative materials through biosolids treatment processes, i.e., the processes cannot specifically reduce the mass of metals in the feed sludge. Metals may be lost from the biosolids treatment process in aqueous sidestreams such as leachates, filtrates or supernatants. Because metals cannot be removed by the biosolids treatment processes, the only method to further reduce concentrations in the biosolids, if needed, is to restrict them at the source.

Over the past three decades, very positive steps have been taken in Canada to reduce the concentrations of all metals in the biosolids. Current concentrations of cadmium, chromium, lead

Table ES-4. Metal Concentration Data in 11 Canadian Treated Sludge and Biosolids Samples

Metal (total)	No. of Detected Conc'ns (out of 11)	Concentration (mg/kg TS dw)			Conc'n Limit for Unrestricted Use (Compost) (CCME, 2005)
		Median of All Conc'ns	Median of Detected Conc'ns	Maximum Detected Conc'n	
Arsenic (As)	7	1.4	2.6	6.7	13
Cadmium (Cd)	2	<1.0	1.1	1.2	3
Chromium (Cr)	10	18.1	20.3	120	210
Cobalt (Co)	7	2.6	2.9	4.2	34
Copper (Cu)	**11**	**271**	**271**	890	400
Lead (Pb)	9	22.5	24.7	55.5	150
Mercury (Hg)	**11**	**0.68**	**0.68**	3.2	0.8
Molybdenum (Mo)	8	1.8	3.5	8.6	5
Nickel (Ni)	9	9.9	10.5	21.1	62
Selenium (Se)	6	1.3	2.2	3.2	2
Zinc (Zn)	**11**	**331**	**331**	647	700

Samples in **bold font** are detected in all samples of treated biosolids

and nickel are reduced by greater than 90% compared to the 1981 levels. The literature review associated with this field survey (Hydromantis *et al*. 2009) noted that reductions of metal concentrations, such as nickel, chromium and cadmium, were effectively accomplished in the 1980s and 1990s by source control, pre-treatment and sewer use limits. When comparing metal concentrations in composted septage (Gatineau Valley) to median biosolids values, the metal concentrations are approximately the same, an observation also reported by (Perron and Hébert, 2007) who evaluated a higher number of septage locations. All median metal concentration in sludge and biosolids, with the exception of selenium, met the current most stringent quality criteria for land application, although a limited number of exceedances were observed for copper, mercury and molybdenum on a site-specific basis. The data reinforce the success of source reduction of metals from industries, with metals contributed to biosolids now mainly originating from domestic rather than industrial sources.

Pharmaceutical, Alkylphenolic and Fragrance Compounds

The pharmaceutical analyses included lists of both acid positive and acid negative compounds; in total 57 compounds were included in the scans of the two lists. Of the 57 candidate pharmaceutical compounds, twenty were never observed above the detection level in the treated sludge and biosolids, as indicated in **Table ES-5**. Nonylphenol and four nitro musk compounds were also never detected. Sample detection limits were determined for each compound in each matrix, and as a result no single "representative" detection limit is provided.

Only four of 57 pharmaceutical compounds (7%) were found in detectable concentrations in all 31 samples of treated sludges and biosolids. These four pharmaceutical compounds included triclocarban, carbamazepine, diphenhydramine and miconazole. Two polycyclic fragrance compounds, HHCB and AHTN were also detected in all samples of treated sludges and biosolids. The frequency of occurrence and median concentrations of the organic analytes is presented in **Table ES-6**.

Table ES-5. Compounds Never Detected in Treated Sludges and Biosolids in this Study

Pharmaceuticals		Fragrances and Alkylphenolics
Acetaminophen	Penicillin G	Nonylphenol
Carbadox	Sarafloxacin	Musk Moskene
Cefotaxime	Sulfachloropyridazine	Musk Tibetene
Clinafloxacin	Sulfadiazine	Musk Ketone
Cloxacillin	Sulfadimethoxine	Musk Ambrette
Flumequine	Sulfamethazine	
Lomefloxacin	Sulfamethizole	
Norgestimate	Sulfathiazole	
Ormetoprim	Tylosin	
Oxacillin	Warfarin	

Table ES-6. Occurrence and Median Concentrations of Organic Analytes in Treated Sludges and Biosolids in this Study

Compound	% occurrence	Median conc'n (ng/g TS dw)	Compound	% occurrence	Median conc'n (ng/g TS dw)
HHCB	100%	3470	Gemfibrozil	52%	56
Triclocarban	100%	1930	Trimethoprim	42%	31.2
AHTN	100%	1340	Dehydronifedipine	42%	7
Miconazole	100%	441	Sulfamethoxazole	39%	5.2
Diphenhydramine	100%	420	Furosemide	32%	543
Carbamazepine	100%	66.6	2-Hydroxy-ibuprofen	26%	497
Triclosan	97%	6085	Enrofloxacin	23%	22.2
ATII	96%	255	Octylphenol	18%	50
Ciprofloxacin	94%	3610	1,7-Dimethylxanthine	13%	378
Ofloxacin	87%	276	Sulfanilamide	13%	63.1
Bisphenol A	86%	325	Glyburide	13%	11.5
Azithromycin	84%	205	Hydrochlorothiazide	10%	143
Fluoxetine	84%	53.9	Sulfamerazine	10%	17.9
Naproxen	81%	98.1	Virginiamycin	6%	197
Clarithromycin	74%	41.8	Digoxin	6%	192
Thiabendazole	74%	17.9	Digoxigenin	6%	128
Erythromycin-H_2O	74%	12.5	Musk Xylene	5%	530
DPMI	73%	82.5	ADBI	5%	60
Ibuprofen	68%	522	Lincomycin	3%	71.1
Diltiazem	68%	29.8	Penicillin V	3%	59.3
AHDI	64%	158	Glipizide	3%	11.4
Caffeine	61%	266	Oxolinic Acid	3%	1.9
Norfloxacin	58%	558	Roxithromycin	3%	0.8

Although 20 pharmaceuticals were found in detectable concentrations in more than 75% of the feed sludge samples, only 10 of 57 pharmaceuticals (18%) were found in more than 75% of the treated biosolids samples likely to be land applied. A greater proportion of pharmaceuticals were detected when septage was the feed sludge (49 % at Gatineau Valley) rather than from on-site wastewater processes.

A shift in the frequency distribution occurred such that a greater number of the pharmaceuticals were detected less frequently after the biosolids treatment, compared to frequency in the feed sludge samples, suggesting that on a broad overview, biosolids treatment processes reduce the number of detectable concentrations of ESOC in the feed sludge. The ability to reduce ESOC in biosolids is process dependent, however. The frequency of occurrence of fragrance compounds was relatively similar in both feed sludge and treated sludges or biosolids.

A small number (12/57) of pharmaceutical compounds were observed at concentrations exceeding 1,000 ng/g TS dw (1 mg/kg TS dw) in the final sludge or biosolids products from the test sites. The antibacterial compounds triclosan and triclocarban, and the antibiotic ciprofloxacin were the compounds most frequently detected (9 of 11 sites) above 1000 ng/g TS dw. At a few sites, the concentrations of triclosan and ciprofloxacin in the final sludge or biosolids exceeded 10,000 ng/g TS dw. The fragrance compounds HHCB and AHTN were observed at median concentrations greater than 1,000 ng/g TS in 10 and six of the eleven sites respectively. The median concentration of Bisphenol A exceeded 1,000 ng/g TS in 3 of the 11 sites tested.

Elevated concentrations of ESOC such as triclosan, ciprofloxacin, BPA, HHCB and AHTN may be one criterion used for identifying ESOC that should be considered for detailed risk assessment. There are other criteria, however, such as persistence, potential for bioaccumulation, and toxicity, that are at least as important and also need to be considered for targeting the ESOC for priority risk assessment.

Many pharmaceuticals (nearly 30 % of those tested) were not detected in the final biosolids products. For those substances that were still detected after process treatments, the statistics provided in **Table ES-6** may help scientists to evaluate whether or not these concentrations may still pose risk with land application.

Biosolids or sludge treatment processes at four of the sampling locations involve the production of sidestreams (e.g. dewatering press filtrate, compost pad leachate) that contain elevated concentrations of some of the hydrophilic pharmaceuticals, which can represent a significant percentage of the input mass of the ESOC. In a few cases, the pharmaceutical mass calculated in the filtrate was greater than the input mass (e.g., ibuprofen and carbamazepine at Eganville, acetaminophen and dehydronifedipine at Gander). Because some compound mass in the feed sludge may be transferred to the aqueous sidestream, the change in frequency of occurrence of detectable concentrations from feed sludge to treated biosolids cannot be interpreted simplistically as a reduction or removal efficiency. With the exception of the concentration of Bisphenol A in the Gander press filtrate, the mass of the BPA in the leachate represented between 1% and 7% of the mass in the feed sludge. The mass of the fragrances in the sidestreams or leachates represented less than 1% of the mass in the feed sludges. In general, there is a very

minor loss of the fragrance compounds and BPA from the feed sludge to the leachate, as would be expected of hydrophobic compounds.

It was observed that composting of sludges (aerobic treatment) generally resulted in the highest removal efficiencies of most ESOC, including pharmaceutical and fragrance compounds. Many other pharmaceuticals were effectively removed in the aerobic environment compared to the anaerobic environment. Compounds with this behaviour included azithromycin, ciprofloxacin, miconazole, triclosan, triclocarban, diphenhydramine, gemfibrozil, thiabendazole and carbamazepine. A limited number of pharmaceuticals, such as naproxen, however, survived and apparently increased through the composting process. Mesophilic anaerobic digestion of sludges, conversely, was found to substantially reduce concentrations of naproxen, as was noted in the project's literature review (Hydromantis *et al*., 2009). There was also limited evidence that anaerobic digestion may result in higher removal efficiencies of acetaminophen than composting, based on one location of each process type with quantifiable results. In general, however, anaerobic digestion was less successful in overall removal of ESOC than the composting process.

A very few compounds appeared to be susceptible to removal by both aerobic and anaerobic biological treatment. These included sulfamethoxazole, trimethoprim, diltiazem and caffeine. A limited number of pharmaceutical compounds appeared to be difficult to remove in almost all processes examined, when present at detectable concentrations. These included the diuretic furosemide, the anti-epileptic carbamazepine, and the antibiotic ofloxacin.

For the most part, the corresponding compounds in this study and the U.S. EPA's Targeted National Sewage Sludge Survey (TNSSS) are comparable in frequency of occurrence and concentrations. Of the nine compounds that can be compared directly, the ratio of the median concentrations (TNSSS/this study) of seven of the compounds falls between 0.6 and 2.7. For these seven compounds, the ratio is greater than unity for 6 compounds, indicating that median levels in U.S. sludges are slightly higher than in the Canadian sludges and biosolids examined in this study. For the other two pharmaceutical compounds (triclocarban and ofloxacin), median concentrations in the U.S. sludges were an order of magnitude higher than observed in the sludges and biosolids tested in this study. The higher median concentrations in the U.S. TNSSS than in this study may be reflective of a greater proportion of untreated sludges included in the U.S. study compared to this, as the extent of sludge treatment was not of primary consideration in the U.S. study. The results of this analysis suggest that data from the U.S. TNSSS can be used as a general indicator of compounds found in Canadian sludges and biosolids, with some compounds in Canadian samples being substantially lower than the levels in the U.S. TNSSS.

When the results of this field study were compared to the observations documented in the accompanying literature review, similar trends were noted. In both the literature and this field study, anaerobic digestion readily removed the antibiotic sulfamethoxazole and the non-steroidal anti-inflammatory drug naproxen, with ibuprofen removed to a lesser extent. Compounds such as the anti-epileptic carbamazepine, the anti-microbials triclosan and triclocarban, Bisphenol A, and the polycyclic musk fragrances HHCB and AHTN either remained unaffected by anaerobic digestion or increased in concentration through the process. Effectiveness of other sludge or biosolids treatment processes was uncertain because the literature documenting such information was sparse.

Overall Effectiveness of Processes for ESOC Removal

As a test of the different process capabilities for removing pharmaceuticals, alkylphenolic and fragrance compounds, the different removal efficiency ranges were assigned a numerical score, ranging from 1 for compounds which were removed by over 90%, to a value of 5 for compounds with calculated removal efficiencies that had a magnitude greater than -50%. (A negative removal means that the total mass leaving the biosolids treatment unit is greater than the mass entering the unit.) By summing the points assigned to each process for each compound, and dividing by the number of detections of the compound per site (i.e., counts) included in the assessment, a mean score for each process was calculated. The interpretation of this procedure considers that the lower the mean score (i.e. closer to unity), the more effective the process is at removing the pharmaceuticals. The results of this process comparison are presented in **Table ES-7**.

Table ES-7 identifies in general terms the ability of a process to reduce ESOC loading from the feed sludge to the treated sludge or biosolids. A higher score is not a criticism of the process: the treatment processes were neither designed nor implemented specifically for removal of these contaminants. The removal efficiencies are also not a reflection on the overall operation of all processes at a WWTP.

Table ES-7. Ranking of Sludge and Biosolids Treatment Processes for ESOC Removal in the Field Study

Location	Treatment Process Assessed	Score total	Number of compound (counts)	Processing /Reduction efficiency (average score)
Gatineau Valley	Biological – aerobic (Compost)	49	27	1.81
Moncton	Biological – aerobic (Compost)	57	31	1.84
Prince Albert	Biological – aerobic (Compost)	72	29	2.48
Eganville (Septage)	Physical – geotextile bag dewatering	85	28	3.04
Halifax N-Viro	Physical-chemical (alkaline stabilisation)	115	35	3.29
Red Deer	Biological – mesophilic anaerobic digestion	115	34	3.38
Salmon Arm	Biological – autothermal aerobic digestion	111	32	3.47
Saskatoon	Biological – mesophilic anaerobic digestion	118	34	3.47
Smiths Falls	Physical – thermal drying	101	27	3.74
Gander	Physical – filter press dewatering	102	27	3.78
Saguenay	Physical – filter press dewatering	108	27	4.00

Composting was the most effective treatment for reducing loadings of the target analytes in the feed sludges. Anaerobic digestion was less successful than the aerobic composting processes. One of the more surprising results from this assessment was the lower removal efficiencies of pharmaceutical compounds than might have been expected in the autothermal aerobic digestion process, considering it is an aerobic process that operates at an elevated temperature, which should result in faster removal rates. The reasons for this observed performance are not clear and have been identified below as a knowledge gap.

The geotextile bag filtration and alkaline stabilisation processes were the most effective of the non-biological processes. The processes involving solids dewatering alone were the least effective; however, because they operate only as physical separation devices, they are not designed for removal of ESOC. The low rating for theses processes is therefore not surprising. Dewatering may remain helpful in a series of biosolids treatment processes, however, to reduce concentration of water-soluble compounds.

Best Management Practices

The sampling survey reported here provided an interesting look at different biosolids and sludge treatment processes, and their ability to remove metals, pharmaceutical, fragrance and alkylphenolic compounds present in the process feed sludge streams. The different treatment processes examined, however, are not replicated sufficiently to draw statistical inferences. Some of the processes in fact were represented by only one site. It is therefore difficult to state definitively from this initial survey which processes should be categorized as "Best Management Practices".

Metals

Most of the metals of industrial significance (e.g., electro-plating and surface finishing) concentrations in biosolids have been reduced very substantially over the past two to three decades. The reductions of these metals are almost entirely due to source control measures or substitution (e.g. substituting cadmium-plating with other metals). Such measures should continue to be implemented and enforced. The two metals that were regularly observed at the highest concentrations in the biosolids and sludges are copper and zinc, which are commonly used in residential, commercial and institutional plumbing. Further reductions of these two metals in biosolids can be accomplished by a substitution of plumbing pipes and appurtenances with other materials such as polyvinyl chloride (PVC) or high density polyethylene (HDPE) if the concentrations in the biosolids warrant this expenditure

Pharmaceutical, Alkylphenolic and Fragrance Compounds

With respect to removal of pharmaceutical compounds by biosolids or sludge treatment, the technology that appears to be more effective than others is composting, an aerobic biological process that operates at thermophilic (e.g. approximately 55^{o}C) temperatures. Anaerobic digestion removes a limited number of different pharmaceutical compounds, presumably because of the different microbial consortia present in the two environments and the absence of oxygen. If greater reduction of ESOC in biosolids is determined by risk assessment to be necessary, a combination of treatments may act as a multi-barrier approach for reducing concentrations in treated biosolids. For smaller municipalities, the geotextile bag filter dewatering process may offer some reduction in pharmaceuticals at low cost.

If some pharmaceutical compounds of concern are difficult to remove by the biosolids treatment processes examined herein, consideration may be given to preventing their deposition in the sludge feed streams for the biosolids processes. This prevention concept could be implemented by two potential design and operating changes. First, many of the pharmaceutical compounds are hydrophobic, and are thus associated with primary clarifier underflows. Consequently, they are not subject to aerobic biological treatment, which could enhance their overall removal from the

incoming wastewater. Overall reduction in pharmaceutical compounds could potentially be improved without a primary clarification step, as is often practiced with the extended aeration process and aerated lagoons used at smaller municipalities. Implementation of such a practice in conventional activated sludge processes would represent a radical departure from existing design and operating philosophies, however. Alternatively, preliminary separate treatment of the primary sludge, for example by either aerobic digestion or other treatment such as ozonation, prior to mixing with secondary sludge, may provide reduced concentrations of pharmaceuticals entering the biosolids or sludge treatment processes. Pre-ozonation of the combined feed sludge may also provide some beneficial effect on removal in the biosolids treatment processes.
Source control of pharmaceutical compounds may be accomplished to some extent through pharmaceutical take-back programs and education of the public that they should not flush unused medications via toilets to the sanitary sewer system. Product substitution is likely difficult to implement, as the public needs their medications. Other ESOC, such as fragrances, surfactants and anti-microbials could be candidates for product substitution, however.

Depending on the mode of action, some pharmaceuticals can be metabolized in the body and excreted in urine. Others are excreted in feces. For those pharmaceuticals that are excreted in urine, use of toilets equipped with urine traps may help to remove the compounds from entering the wastewater stream. Such a shift in technology substitution would require a long period to implement across the country.

Knowledge Gaps and Research Needs

This study afforded an opportunity to investigate in detail the potential removal of ESOC by sludge and biosolids treatment processes commonly used in Canada. The study produced much valuable information on the fate of the ESOC selected for investigation, but as is often the case the acquisition of new knowledge leads to additional questions. Below are listed some of the knowledge gaps and research needs arising from this survey and from the literature review (Hydromantis, 2009) in no particular order of importance.

This study looked at a select group of pharmaceuticals, fragrance and alkylphenolic compounds. Due to budgetary limitations, it did not look at other classes of ESOC, including other pharmaceutical compounds, natural and synthetic human hormones, industrial chemicals (e.g. phthalate esters, polybrominated diphenyl ethers and other flame retardants, perfluorinated organic substances, alkylphenol ethoxylates, quaternary ammonium compounds), and personal care products (insect repellents, sunscreens, parabens, organic siloxanes, fabric softeners, fluorescent whitening agents, etc.). Research at full-scale similar to this study for these many types of ESOC is encouraged to round out the knowledge of ESOC behaviour in biosolids treatment processes. [At the time of preparing this report, another field study was conducted by Environment Canada under the Chemical Management Plan to analyse sample of wastewater liquid and solids process streams for a range of substances including selected pharmaceutical and personal care products, brominated flame retardants, perfluorinated organic compounds, volatile methyl siloxanes nonylphenol ethoxylates and a suite of 18 metals (Smythe, 2010).]
Some unexpected results were obtained in this study, both positive and otherwise. An unexpected result was the reduction of a number of organic ESOC by the geotextile bag filter dewatering process at the Eganville, ON treatment plant. Only one application of this type of

dewatering process was included in this sampling survey. Additional sites using this technology should be tested in a similar manner to determine if the process does offer a low-cost means of dewatering wastewater sludge with better removal efficiencies of more ESOC than other processes examined herein. Factors to consider in additional testing should include the type of feed solids (primary sludge, septage, waste activated sludge) to the process, loss of ESOC in bag filtrate, possible effect of freezing and thawing, and retention time and possible aerobic/anaerobic microbial activity in the geotextile bags.

The autothermal aerobic digestion process exhibited lower removal efficiencies of pharmaceutical, fragrance and alkylphenolic compounds than might have been expected considering it is an aerobic process that operates at an elevated temperature, which should result in faster removal rates. The possible reason may be that the relatively short detention time at the elevated temperature of thermophilic operation (e.g. approximately 55 ^{o}C) reduces the number and types of microbes that can biodegrade the ESOC. Composting is an aerobic process in which temperatures reach thermophilic conditions, which is similar to those experienced in the ATAD process. Additional studies with this type of process should be undertaken to determine this discrepancy.

It was observed that composting of sludges to produce biosolids generally resulted in the highest removal efficiencies of most ESOC. A limited number of pharmaceuticals, such as naproxen, however, survived and apparently increased through the composting process. Mesophilic anaerobic digestion of sludges was found to substantially reduce concentrations of naproxen, but was less successful in overall removal of ESOC. The ability of a combination of anaerobic digestion, followed by dewatering and composting, for example, might provide a means of reducing more of the ESOC, including other that were not tested in this program. Such a study, either at pilot-scale or at existing full-scale facilities with this treatment combination would be helpful in determining the possible benefits of different redox environments for ESOC removal.

The biological treatment processes for biosolids in general were able to reduce ESOC in the feed sludge more efficiently than were the physical (including physical-chemical) processes. Of the physical-chemical processes, the N-Viro alkaline stabilisation process appeared to offer the best performance for ESOC removal. Only one example of this process was included in this survey (i.e. the Halifax site). Moncton, NB uses a partially lime-stabilised biosolids as the feed material for the composting operation, but the focus there was on the composting process, rather than on lime stabilisation. Additional testing of lime- and alkaline-stabilisation processes for reduction of ESOC should be undertaken.

The thermal drying process (pelletisation) was not efficient in the reduction of ESOC, with the knowledge that it was not intended for that purpose. It may be possible, however, to accomplish greater reduction of ESOC to take advantage of thermal or chemical decomposition by a change in process operating conditions.

It is of high importance to evaluate whether the detected concentrations of pharmaceuticals and other ESOC in land applied biosolids could are of concern for either human health or environmental risk. The U.S. EPA is currently conducting such risk assessments (Hebert, 2010). The results of such studies will help to determine if further reductions in concentration of specific

compounds may be needed. Studies by Carballa *et al*. (2007b) indicated that pre-ozonation of the feed sludge to the anaerobic digestion process generally resulted in improved removal of several classes of ESOC. Because it is unlikely that source control can restrict inputs of pharmaceuticals to wastewater treatment plants, improving the removal of the compounds by biosolids treatment processes by pre-ozonation or other processes should be investigated, including sludge feeds to all the different biosolids treatment processes (i.e., not just anaerobic digestion).

Study Conclusions

The conclusions that follow relate to the suite of target ESOC evaluated in this study.

1. Metal contaminants in biosolids are in general unaffected by the biosolids stabilisation process employed, as compared to organic constituents. A potential exception may be mercury, which can be biologically activated in anaerobic environments, and also undergo transfer from biosolids to the gas phase by stripping or volatilisation.
2. All median metal concentration in sludge and biosolids, with the exception of selenium, met the current most stringent quality criteria for land application, although a limited number of exceedances were observed for copper, mercury and molybdenum on a site-specific basis.
3. Metal concentration of biosolids and septage were quite similar, indicating that metals in biosolids now mainly originate from domestic rather than industrial sources.
4. Although 24 pharmaceutical, alkylphenolic and fragrance compounds were found in detectable concentrations in more than 75% of the feed sludge samples, only 14 of 71 pharmaceutical, alkylphenolic and fragrance compounds (20%) were found in more than 75% of the treated biosolids samples likely to be land applied.
5. The antibacterial compounds triclosan and triclocarban, the antibiotic ciprofloxacin, and the fragrance compound HHCB were the compounds most frequently detected (9 or more of 11 sites) above 1000 ng/g TS dw.
6. For the most part, the corresponding compounds in this study and the U.S. EPA's Targeted National Sewage Sludge Survey (TNSSS) are comparable in frequency of occurrence and concentrations.
7. Biosolids stabilisation processes using some form of biological treatment are more efficient at reducing the organic ESOC concentrations than are non-biological processes.
8. Of the biological treatment processes, the composting process (aerobic) appears to be more effective in overall reduction (in number and degradation) of ESOC than does mesophilic anaerobic digestion.
9. ESOC removed efficiently by composting, but not well reduced by anaerobic digestion include compounds such as ciprofloxacin, miconazole, triclosan, gemfibrozil, thiabendazole, carbamazepine, Bisphenol A, HHCB, AHTN, AHDI, and ATII.
10. The autothermal aerobic digestion process was much less effective in reducing ESOC than was either composting or mesophilic anaerobic digestion.
11. The geotextile bag filter used for dewatering sludge and septage was capable of reducing a number of ESOC, although the exact mechanism is unclear at this time.
12. Of the physical processes (including physical-chemical) processes, the N-Viro alkaline stabilisation process appeared to offer the best performance for ESOC removal
13. The thermal drying process (pelletisation) alone was not efficient in the reduction of ESOC, acknowledging that it was not intended for that purpose.

14. Mechanical sludge dewatering processes alone are among the least effective for reducing concentrations of ESOC in the feed sludge.
15. A few pharmaceutical compounds appear to be removed readily by either aerobic or anaerobic biological treatment, including sulfamethoxazole, trimethoprim, caffeine and diltiazem.
16. A limited number of pharmaceutical compounds appeared to be difficult to remove in almost all processes examined, when present at detectable concentrations. These included the diuretic furosemide, the anti-epileptic carbamazepine, and the antibiotic ofloxacin.
17. Naproxen appears to increase substantially through aerobic composting, possibly due to biotransformation from other compounds, but it appears to be more efficiently removed by anaerobic digestion.
18. While many of the ESOC remain associated with the solid phase of the sludges or biosolids, a number of compounds can be lost in any aqueous process sidestream (e.g., dewatering filtrate, leachate, digester supernatant), including furosemide, ibuprofen and 2-hydroxy-ibuprofen, naproxen, acetaminophen, caffeine, carbamazepine, clarithromycin, dehydronifedipine, erythromycin-H_2O, sulfamethoxazole and trimethoprim.
19. Less than 1% of the mass of fragrance compounds in feed sludge resides in the process sidestreams or leachates from the treatment processes, while between 1% and 6% of the mass of Bisphenol A in the feed sludges was transferred to the process sidestreams or leachates.
20. A combination of processes (e.g. anaerobic digestion plus dewatering plus composting as at Prince Albert; lime stabilisation plus composting as at Moncton) result in the highest reductions of many ESOC.
21. The treatment efficiencies of ESOC by anaerobic digestion observed in this field study are comparable to results reported in the technical literature; published removal efficiencies of ESOC in other biosolids treatment processes are sparse.
22. The ESOC concentration data in sludges and biosolids produced in this sampling program are insufficient alone, without applying formal risk assessment methods, to determine human health or environmental risks of managed biosolids land application, land reclamation, and production of commercial and soil amendments.

Study Recommendations

1. Risk assessments should be conducted with ESOC to evaluate if they may pose risk to human health or the environment when applied to land amended with biosolids. Based on frequency and concentrations observed in the treated sludges and biosolids, candidate compounds for initial risk assessment may include triclosan and triclocarban, ciprofloxacin, the fragrances HHCB and AHTN, and BPA, although other factors such as persistence, bioaccumulation potential and toxicity also need to be considered.
2. Research at full-scale, similar to this study, for many other types of ESOC (other classes of pharmaceutical compounds, natural and synthetic human hormones, industrial chemicals (e.g. phthalate esters, brominated flame retardants, perfluorinated organic substances, alkylphenol ethoxylates, quaternary ammonium compounds), and personal care products (insect repellents, sunscreens, parabens, organic siloxanes, fabric softeners, fluorescent whitening agents, etc.) is encouraged to round out the knowledge of ESOC behaviour in biosolids treatment processes.

3. Additional sites using the geotextile bag filtration technology should be tested in a manner similar to this survey to determine if the process offers a low-cost means of dewatering wastewater sludge with substantial removal efficiencies of certain ESOC. Factors to consider in additional testing should include the type of feed solids (primary sludge, septage, waste activated sludge) to the process, loss of in bag filtrate, possible effect of freezing and thawing, and retention time in the geotextile bags.
4. Additional sampling of the autothermal aerobic digestion process at other locations should be undertaken to determine if the lower removal efficiencies of pharmaceutical, fragrance and alkylphenolic compounds observed at the one site tested (compared to other aerobic processes such as composting), was an isolated event, or is representative of the process behaviour with respect to ESOC.
5. Because only one example of lime- or alkaline-stabilisation processes was included in this survey, and because the alkaline stabilisation process appeared to offer the best performance for ESOC removal of any of the physical (including physical-chemical) processes, additional testing should be undertaken for confirmation and optimization of ESOC reduction.
6. A study examining the ability of a combination of processes (e.g. anaerobic digestion, followed by dewatering and composting; alkaline/lime stabilization followed by composting), either at pilot- or full-scale, is recommended for determining the possible benefits of different redox environments for reducing ESOC, including others that were not tested in this program.
7. Because only one example of lime- or alkaline-stabilisation processes was included in this survey, and because the alkaline stabilisation process appeared to offer the best performance for ESOC removal of any of the physical (including physical-chemical) processes, additional testing to document reduction of ESOC by this type of process should be undertaken.
8. Studies of pre-treatment of feed sludges, such as by ozonation, prior to the biosolids treatment processes should be investigated to determined the potential beneficial effects and cost-effectiveness for overall improvement in ESOC removal efficiencies.
9. Data produced by this and similar investigations need to be transferred out to appropriate departments and agencies, federal and provincial regulators, municipalities and academic researchers for risk assessment purposes.

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1. INTRODUCTION

1.1 Basis for Project

The Biosolids Task Group (BTG) established by the Canadian Council of Ministers of the Environment (CCME) is mandated to study and make recommendations on biosolids management at the national level. Wastewater treatment facilities (WWTF) across Canada generate residual wastewater solids (biosolids) that require treatment for safeguarding human health and the environment prior to their use or disposal. Options for disposal, recovery or recycling of biosolids include energy, nutrient or material recovery, landfilling, incineration, managed land application, land reclamation, and commercial product recovery (compost and pellets).

The end use of the biosolids is often governed by the constituent quality of the biosolids, such as nutrients, metals, pathogens and trace constituents. Land application of biosolids has been practiced in Canada for many decades. Currently, 11 inorganic trace/micro-constituents, such as cadmium, lead and mercury, and pathogen/pathogen standards are monitored in biosolids on a routine basis, prior to land application. Other constituents thought possibly to be of concern in biosolids in the 1990's, such as PCBs, dioxins and furans, and polyaromatic hydrocarbons (PAHs) were extensively studied at that time. These classes of compounds were included in the literature review that accompanied the field study (Hydromantis *et al*., 2009). As these studies found low concentrations in biosolids, biosolids are not being tested for these constituents by most jurisdictions. Consequently, they were not included in the potential list of target analytes in this study.

At present, the risks associated with detecting in biosolids certain classes of micro-constituents (termed Emerging Substances of Concern (ESOC) herein), which include an array of pharmaceuticals, personal care products and industrial contaminants (such as plasticizers, surfactants and brominated flame retardants) are not well understood. While there is some documentation of ESOC in biosolids, no focused study has been completed yet on an inventory of ESOC in Canadian biosolids. Consequently, CCME issued a Request for Proposals to document the occurrence of ESOC in biosolids and septage; to conduct a targeted sampling program at selected representative Canadian wastewater treatment plants to provide a focused Canadian study and an inventory of ESOC in Canadian biosolids; and to assess the removal efficiencies of various treatment processes, if any. The sampling study results will contribute to the knowledge basis which will assist CCME in evaluating and managing the risks associated with ESOC in biosolids with respect to managed land application, land reclamation, and production of commercial and soil amendments.

1.2 Objectives

The objectives of the entire project are to:

1. Prepare a comprehensive review of research on ESOC in biosolids within Canada and elsewhere based on technical literature and wastewater sector contacts;
2. Complete a field survey and analyze Canadian biosolids and septage samples with respect to ESOC;

3. Identify the occurrence and concentration ranges of those ESOC in Canadian biosolids within the scope of this study;
4. Review and recommend treatment technologies that mitigate ESOC concentrations in biosolids;
5. Suggest Best Management Practices (BMPs);
6. Identify knowledge gaps and research needs for ESOC with respect to biosolids;
7. Produce a final report of the project to the Contract and Project Authorities.

A previous report for this project consisted of a detailed literature review of the occurrence of ESOC in municipal wastewater residual solids and biosolids from different treatment processes (Hydromantis *et al*., 2009) and corresponded to Project Objective. The following report, responding to Objectives 2 through 7, constitutes the results obtained from a detailed filed sampling program for the characterization of ESOC in residual solids and biosolids from 11 wastewater treatment facilities across Canada.

2. DESCRIPTION OF BIOSOLIDS TREATMENT PROCESSES

2.1 Introduction

The purposes of biosolids treatment processes are stabilisation and odour reduction, volume reduction, pathogen inactivation, and reduction in vector attraction. Processes are often referred to as Class A or Class B solids, based on designations originally prescribed by the U.S. EPA in the U.S. Federal Register Part 503 Biosolids regulations. These designations are based primarily on reduction of pathogenic organisms in the feed wastewater sludges that are treated for production of biosolids. **Although the terminology has become widely adopted throughout North America, the quality of the designated Classes of biosolids can vary between jurisdictions in Canada.**

Class A biosolids are those of "exceptional quality" that have been subjected to rigorous levels of treatment. Because of the high level of treatment, there are few restrictions on the use of Class A biosolids. While there are six alternatives that can be used to produce Class A biosolids, a common feature is the pathogen limit requirements, which are that the density of fecal coliform in the biosolids must be less than 1,000 most probable number (MPN) per g of total solids (dry-weight basis), or the density of Salmonella sp. bacteria must be less than 3 MPN per 4 g TS (dry-weight basis) (EPA, 1994).

One of the alternatives for producing Class A biosolids involves the use of processes termed "processes for further reduction of pathogens", or PFRPs. Processes included in this alternative include composting, thermophilic aerobic digestion, heat drying, heat treatment (of slurries), pasteurization, all of which involve a specified combination of retention times and operating temperatures. Gamma and beta ray irradiation are also allowed as PFRPs.

The requirements for producing Class B biosolids are less stringent than those for Class A; the potential uses of Class B biosolids, however, are also more restricted. Criteria for Class B biosolids specify microbial limits and site restrictions. The pathogen reduction requirement for Class B biosolids is that the geometric mean of seven samples of the biosolids must be less than 2 million MPN per gram of TS (or less than 2 million colony-forming units (CFU) per gram of TS at the time of use or disposal (EPA, 1994). Processes that are accepted by the U.S. EPA for producing Class B biosolids include: aerobic digestion, air drying, anaerobic digestion composting and lime stabilisation. Site requirements for these processes typically specify a waiting period between the time of application and the start of the ultimate use of the amended site, such as animal grazing, recreational use, planting of food crops, etc.

The processes investigated in this report are summarised in **Table 1.** Some processes integrate many process units (e.g. biological treatment + dewatering; liming + composting) while others only cover a specific process unit (e.g.: geotextile bag dewatering, filter press dewatering). According to U.S. EPA criteria, "treated" dewatered sludges that do not meet Class A or Class B standards are not termed "biosolids", but treated sludges. The residual wastewater solids delivered to the treatment processes studied are referred to in this report as "feed sludge".

Table 1. Sludge and Biosolids Treatment Processes Investigated in This Study

Process	Number in Study
Autothermal thermophilic aerobic digestion	1
Mesophilic anaerobic digestion	2
Composting	3
Alkaline stabilisation	1
Thermal drying (pelletisation)	1
Geotextile bag dewatering	1
Filter press dewatering	2

2.2 Anaerobic Digestion

The anaerobic digestion process makes use of bacteria that thrive in the absence of oxygen to stabilise the volatile solids and reduce levels of pathogens. The two most common types of anaerobic digestion are mesophilic digestion (temperature maintained at approximately 35 ^{o}C, and thermophilic digestion (typically between 50 and 57 ^{o}C). Although mesophilic digestion can result in volatile solids reduction greater than 40%, it does not result in a pathogen-free product, and so is considered a Class B process. Thermophilic digestion, either alone or as part of a temperature-phased system, with sufficient retention time can produce a Class A Biosolids quality in terms of pathogen concentrations.

In mesophilic anaerobic digestion process, the feed sludge stream (primary and/or secondary sludge) is pumped to the digestion tank on a semi-continuous basis, where it is maintained for a typical average detention time of approximately 15 days or higher. The digested sludge is a liquid slurry which can be applied off-site directly as a Class B product, or dewatered to produce a drier sludge cake for off-site disposal. Biosolids can be designated as Class A following thermophilic digestion if held for a specified period of time, rather than hydraulic retention time, at a temperature above 50 ^{o}C, as provided in Alternative 1 of the Part 503 regulations (WEF, 2009).

2.3 Aerobic Digestion

2.3.1 Ambient and Mesophilic Temperatures

Aerobic digestion of wastewater solids uses aerobic micro-organisms to degrade organic matter (measured as volatile solids) and other organic components, to reduce mass and volume, and to reduce pathogenic organisms (WEF, 2009). The principle behind aerobic digestion is cellular endogenous respiration, in which the microbes are maintained in a process tank for an extended time period. During this period, the external food substrate is depleted, and so the microbes must consume their own protoplasm as the energy source for cell maintenance functions. This self-consumption, or endogenous respiration, is responsible for the observed reduction in the volatile fraction of the microbes.

Biosolids produced by aerobic digestion (mesophilic, i.e., 10 to 40 ^{o}C) are typically designated as Class B. WEF (2009) points out that the vector reduction criterion of 38% volatile solids reduction may be difficult to accomplish in aerobic digestion if the excess secondary sludge

results from a secondary treatment unit with a long solids retention time, such as extended aeration. In situations where the 38% volatile solids reduction cannot be demonstrated, a bench-scale evaluation can also be used to demonstrate acceptable vector reduction. The test involves the already aerobically digested solids, at concentration 2% or less, undergoing further aerobic digestion over a 30 day test period at 20 ^{o}C with a change in the volatile solids concentration of less than 15%. Alternatively, the reduction in vector attraction can be demonstrated by other allowable criteria, such as a soluble oxygen uptake rate (SOUR) criterion value of less than 1.5 mg of oxygen per g of total solids per hour at a temperature of 20 ^{o}C.

2.3.2 Autothermal Aerobic Digestion (ATAD)

Autothermal thermophilic aerobic digestion (ATAD) involves mixing the wastewater sludge with excess air or oxygen in an insulated reactor. Extensive mineralization of the microbes to carbon dioxide, water and nitrogen occurs in the endogenous respiration process. The endogenous respiration proceeds in a sufficiently fast exothermic reaction to maintain the process temperature between 40 to 80 ^{o}C, thereby achieving the pathogen reduction and 38% volatile solids reduction criteria to meet the Class A designation. The ATAD effluent is a liquid slurry that can either be applied off-site directly as a Class A product, or dewatered to produce a drier sludge cake for off-site disposal.

2.4 Composting

According to WEF (2009), composting is a biological process in which organic matter is decomposed under controlled aerobic conditions to produce humus. The main factors controlling the composting process are the solids content, the carbon-to-nitrogen ratio, a supply of air to maintain aerobic conditions and the process temperature.

The principal raw material used as process feed is wastewater sludge dewatered to between 14% and 30% solids, which is combined with a drier bulking agent (e.g., wood chips, sawdust, shredded yard waste) to reach a solids content of 38% to 45%. Under proper operation, after initial heating, composting is a self-maintaining, aerobic, thermophilic process. As the compost matures and then dries to 55% solids or higher, pathogenic bacteria, viruses and parasites are greatly reduced in number and concentrations. Some fungi however (e.g., *Aspergillus fumigatus*) are able to survive the composting process because they are thermo-tolerant organisms.

2.5 Alkaline Stabilisation

Alkaline stabilisation is a process used to reduce concentrations of pathogens and odour-causing microbes, and to prevent their re-growth. It also produces a more stable product that is environmentally acceptable in terms of odours and vector-attraction. Other benefits are that the increase in the material's pH resulting from the chemical reactions in the process helps to reduce short-term leaching of metals from biosolids as they are tied up as hydroxide precipitates, and that free ammonia produced by the reaction serves as a chemical disinfectant. Depending on the process operation, the resulting stabilised product can achieve either Class B or Class A designation. Class B designation typically involves a pH and time requirement, whereas an

elevated temperature requirement is typically required to meet a Class A designation (WEF, 2009).

Although alkaline stabilisation was initially performed with lime (as quicklime or slaked lime), it now can include other alkaline material such as cement kiln dust, lime kiln dust, Portland cement and incinerator fly ash. In the process, the sludge feed is mixed with the alkaline material and maintained at an elevated pH above 12 for a specified period on the order of 24 hours, to meet Class B requirements. Class A designation can be achieved by using a combination of time and temperature elevation, such as maintaining the temperature at 70 ^{o}C for 30 minutes, or at 52-62 ^{o}C for 12 hours (WEF, 2009). With this treatment, the biosolids produced are essentially free of pathogenic organisms that can be used as a soil amendment.

2.6 Heat Drying

Heat drying is a process that produces biosolids which can be classified as meeting Class A requirements for reduction of pathogens and vector attraction. As well as being used as a fertilizer or soil conditioner, the dried biosolids (termed pellets or granules) can be used as a biofuel. The quality of the granules produced, drying system used and local economic factors are likely to determine the end use of the dried biosolids.

There are two principal types of biosolids thermal drying process categories, namely convection and conduction dryers. Rotary drum dryers and fluidised bed dryers are the most common type of convection dryers in North America (WEF, 2009). Examples of conduction driers are paddle dryers, disc dryers, rotary chamber dryers, tray dryers and pressure filter/vacuum dryers. The initial three types of conduction dryers are in most common use in North America (WEF, 2009).

Drying of biosolids is affected by many operating considerations including solids flow rate, heat transfer rate, operating temperature and humidity, rate and direction of gas flow, exposed surface area and physical form of the biosolids, agitation, detention time and the biosolids support method used (WEF, 2009). Operation of the biosolids driers requires care because improperly dried granules can be subject to auto-oxidation. As well, dust created by over-drying of the granules can create explosive conditions.

2.7 Geotextile Bag Filtration

Geotextile bag filtration of municipal sludges and septage, is a relatively new technology developed within the past two decades. Use of the geotextile fabric allows for water in the sludge to drain from the bag, resulting in drier solids at the conclusion of the dewatering period. According to the literature of one manufacturer of this technology, the fabric also allows for transfer of air through to the solids, permitting aerobic decomposition to occur (Bishop Water Technologies, 2010). In typical operation, the sludge or septage feed solids concentration pumped into the filtration bag is between 1 to 5% TS. Water drained from the bags is usually collected and returned to the wastewater treatment facility. The dewatered biosolids can be land applied when the bags are opened, providing that they meet metal and pathogen requirements.

2.8 Solids Dewatering

According to WEF (2009), the objective of solids dewatering is to reduce the volume of material (residual wastewater solids) and to prepare the solids for further processing, beneficial use or disposal. The dewatering process separates water in the feed sludge to produce a cake that has the properties of a semi-solid or solid product. Commonly employed methods of dewatering include centrifuges, belt filter presses, plate-and-frame filter presses, and drying beds and lagoons. Rotary presses and screw presses are technologies applied to municipal wastewater solids in the past decade. Most dewatering processes produce a dilute sidestream (separated water) that is either returned to a sanitary sewer or directly to the head of the wastewater treatment facility. The dewatering process is not specifically intended to reduce pathogen content or vector attraction of the dewatered cake.

3. METHODOLOGY

3.1 Site Selection

Eleven sites were selected by the BTG of CCME based on a number of considerations including implementation of a biosolids land application program, plant capacity, geographical location, and type of biosolids treatment process. Plant hydraulic capacity and extent of municipal urbanisation were not identified as primary factors of interest by the BTG. Site information is provided in **Table 2**.

3.2 Experimental Plan

Samples of both sludge before the treatment process and the resulting dewatered solids or biosolids were collected between July and November of 2009 on three separate occasions at nine of the eleven targeted Canadian municipalities; at the remaining two sites, two rounds of samples were collected rather than three due to mechanical problems with the biosolids treatment process or due to funding agreements. To account for potential losses of the ESOC in process sidestreams, such as digester supernatant or leachate from composting pads, samples of these process sidestreams were also collected concurrently and analysed in an attempt to better close mass balances around the different biosolids treatment processes.

3.3 Selection of Target Analytes

The potential list of classes of ESOC that might be analysed in this study is extensive. The literature review produced as part of this report (Hydromantis *et al*., 2009) identified many types of ESOC which have been studied, including brominated flame retardants (polybrominated diphenylethers and others) plastics and plasticizer agents, alkylphenols and their ethoxylates, linear alkylbenzene sulphonates, perfluorinated organic compounds, natural and synthetic hormones, pharmaceuticals, synthetic musk fragrances, antibacterial compounds, quaternary ammonium compounds, and volatile methyl siloxanes.

The literature review examined the occurrence and removal of ESOC in biosolids treatment processes, but did not examine any human health or environmental risks due to ESOC present in biosolids. It was useful as a guide in the selection of the analytes in that it identified the near-complete lack of information on the fate of ESOC in treatment processes other than anaerobic digestion.

Three main considerations were responsible for the selection of the ESOC targeted as analytes in this study, namely:

(1) their potential environmental and human health significance based on frequency of occurrence and concentration from the study's literature review, other technical publications and professional judgement;
(2) the availability of sensitive analytical methods to determine concentrations in sludges and biosolids, which are difficult matrices for analysis of ESOC in the ng/l to µg/L concentration range; and

Table 2. Characteristics of Biosolids Sampling Sites

Municipality	Region of Canada	Wastewater Treatment	Solids Treatment Process		Comment
			Feed Sludge	Final Solids or Biosolids	
Gander, NL	Atlantic	Hydrodynamic separation	Raw solids from hydrodynamic separator	Dewatered solids from belt filter press	No stabilisation process applied
Moncton, NB	Atlantic	Primary treatment	partially stabilised biosolids from lime treatment	Composted biosolids	
N-Viro Facility, Halifax, NS	Atlantic	see "comment"	combined wastewater residuals from several locations	Alkaline-stabilised biosolids	Location does not treat wastewater, but accepts residuals from off-site
Saguenay, QC	Central	Activated sludge	Waste activated sludge (WAS)	Dewatering of WAS from belt filter press	No stabilisation process applied
Gatineau Valley, QC	Central	Septage receiving and dewatering	Dewatered septage	Composted dewatered septage with added wood chips	No stabilisation process applied
Eganville, ON	Central	Extended aeration	Waste activated sludge (WAS) or septage (separate feed streams)	Combination of aerobic digestion of WAS followed by geotextile bag dewatering	Also process septage separately in geotextile bag filters
Smiths Falls, ON	Central	Conventional activated sludge plus filtration	Dewatered primary plus waste activated sludges	Heat-dried (pellets)	
Saskatoon, SK	West	Biological nutrient removal	Combined primary and secondary sludges	Liquid mesophilic anaerobically digested biosolids	
Prince Albert, SK	West	Conventional activated sludge	Dewatered primary plus waste activated sludge	Composted biosolids	
Red Deer, AB	West	Biological nutrient removal	Combined primary and secondary sludges	Mesophilic anaerobically digested biosolids plus lagoon dewatering	
Salmon Arm, BC	West	Biological nutrient removal	Combined primary and secondary sludges	Dewatered autothermal aerobically digested biosolids	

(3) budgetary constraints.

Of these considerations, budgetary constraints had the greatest impact in narrowing the potential ESOC test groups that were to be analysed. The majority of the pharmaceutical compounds that can be detected in wastewater and sludge matrices can be captured in five different analytical lists (Grace, 2009), with each list associated with a unit cost. Based on discussions with the analytical laboratories involved in the study (AXYS Analytical Services, ALS Analytical Group and Trent University), a proposed list of target analytes was developed for the project that was deemed to meet the three considerations outlined above. The list can in general be considered to include 57 pharmaceutical compounds, 3 alkylphenolic compounds (including Bisphenol A), 11 synthetic musk fragrances, 11 metals and macronutrients including forms of nitrogen and phosphorus. Although it would clearly be desirable to include additional test groups in the sampling program, budgetary limitations precluded this. The finalised list of analytes for the study is indicated in **Table 3**.

Table 3. List of Target Analytes for Biosolids Treatment Study

Pharmaceutical Test Group 1 (Acid Positive Pharmaceuticals)		**Pharmaceutical Test Group 2 (Acid Negative Pharmaceuticals)**	**Fragrances**
Acetaminophen	Norgestimate	Furosemide	DPMI
Azithromycin	Ofloxacin	Gemfibrozil	ADBI
Caffeine	Ormetoprim	Glipizide	AHDI
Carbadox	Oxacillin	Glyburide	HHCB
Carbamazepine	Oxolinic Acid	Hydrochlorothiazide	AHTN
Cefotaxime	Penicillin G	2-Hydroxy-ibuprofen	ATII
Ciprofloxacin	Penicillin V	Ibuprofen	Musk Moskene
Clarithromycin	Roxithromycin	Naproxen	Musk Tibetene
Clinafloxacin	Sarafloxacin	Triclocarban	Musk Ketone
Cloxacillin	Sulfachloropyridazine	Triclosan	Musk Ambrette
Dehydronifedipine	Sulfadiazine	Warfarin	Musk Xylene
Diphenhydramine	Sulfadimethoxine	**Alkylphenolics**	**Metals**
Diltiazem	Sulfamerazine	Bisphenol A	Arsenic (As)-Total
Digoxin	Sulfamethazine	Octylphenol	Cadmium (Cd)-Total
Digoxigenin	Sulfamethizole	Nonylphenol	Chromium (Cr)-Total
Enrofloxacin	Sulfamethoxazole		Cobalt (Co)-Total
Erythromycin-H_2O	Sulfanilamide		Copper (Cu)- Total
Flumequine	Sulfathiazole		Lead (Pb)-Total
Fluoxetine	Thiabendazole		Mercury (Hg)
Lincomycin	Trimethoprim		Molybdenum (Mo)-Total
Lomefloxacin	Tylosin		Nickel (Ni)-Total
Miconazole	Virginiamycin		Selenium (Se)-Total
Norfloxacin	1,7-Dimethylxanthine		Zinc (Zn)-Total

Pharmaceutical Test Group 1 includes a number of frequently detected antibiotics and other relevant pharmaceutical groups (fluoroquinolones, macrolides and sulfa compounds, as well as the anti-convulsives carbamazepine and trimethoprim, the analgesic acetaminophen, and stimulants such as caffeine and diphenhydramine). As such Test Group 1 encompasses a range

of compounds in biosolids that could potentially be of environmental significance. Test Group 2 is a shorter list of pharmaceuticals, but includes a number of frequently detected and widely used pharmaceuticals including the non-steroidal anti-inflammatory drugs ibuprofen and naproxen, the anti-bacterial compounds triclosan and triclocarban, and the lipid regulator gemfibozil. Both Tests Groups 1 and 2 are acidic pharmaceuticals based on the extraction procedure for analysis. The difference between the Test Groups results from the analytical technique involving positive electro-spray (Group 1) or negative electro-spray (Group2) ionisation mass spectrometry.

3.4 Sample Collection

At the treatment plant sites, sample collection devices such as spoons, rods and scoops were made of stainless steel, glass or Teflon®. Pre-cleaned sample containers were shipped from the analytical laboratories to the sites in the return shipment coolers along with sample packing instructions, gel-type freezer packages, additional packing materials and chain-of-custody forms.

3.5 Preparation and Instructions

From the outset, the sampling program was to be conducted by operating plant staff at each site. To ensure proper procedures for sample collection and shipment to analytical laboratories were followed, a series of internet-based presentations was provided to the operating staff. Topics covered included definition of sampling terms, acceptable materials for sampling devices, compositing of grab samples from different process streams or locations in stockpiles, proper packing of coolers used for shipment, shipping logistics and health and safety issues in sample collection. Telephone and email were also used to respond to immediate questions from the field staff during sample collection and shipment.

3.6 Shipment

Samples were shipped from the collection sites by overnight courier to the laboratories, with shipments no later than Thursday afternoon to avoid sitting in courier depots over weekends. On arrival at the laboratories, samples were processed and refrigerated or frozen until analysis.

3.7 Analytical Procedures

Three different laboratories were involved in the analytical program. AXYS Analytical Services Ltd. in Sydney BC performed the analysis of acid positive and acid negative pharmaceutical compounds using EPA Method 1694 (EPA, 2007). The Worsfold Water Quality Centre of Trent University in Peterborough, ON analyzed the samples for synthetic polycyclic and nitro musk fragrances using gas-chromatography-mass spectrometry on cleaned-up biosolids extracts, as described by Yang and Metcalfe (2005). Alkylphenolic compounds, including Bisphenol A, were analysed by liquid chromatography and tandem mass spectrometry (LC-MS/MS) with an electrospray (ESI) ionization source. For logistical reasons, samples destined for analysis by the Trent University laboratory were shipped from the sites to the AXYS laboratory, and then to the Trent University lab. ALS Laboratory Group of Waterloo, ON completed the analyses of target metals and nutrients identified in **Table 4**.

Table 4. Target Analytes and Methods used for Metals and Nutrients in Biosolids

Parameter	Method
Cadmium, Cobalt, Chromium, Copper, Molybdenum, Lead, Zinc	EPA Method 3050
Arsenic, Selenium	EPA Method SW846 3050B/6020A
Mercury	EPA Method SW8467470A
Total Kjeldahl Nitrogen (TKN)	APHA Method 4500-N
Ammonia-N	APHA 4500-NH3
Nitrate, Nitrite (in Soil)	EPA 300.0
Nitrate + Nitrite	APHA 4110B
Phosphorus Total, Ortho-phosphorus (low level)	APHA Method 4500-P B E

EPA = U.S. Environmental Protection Agency
APHA = American Public Health Association

3.8 Data Analysis

In this report, the effectiveness of the different treatment processes for reducing contaminants in the final processed solids is assessed by use of contaminant mass balances rather than concentrations alone. Many concentrations are expressed on a solids basis, such as ng/g TS dw (total solids dry weight basis). In some treatment processes, there is a loss of a portion of the volatile fraction of the solids due to biological reduction, or from volatilization upon drying at elevated temperatures. The solids balance is used to determine the potential change in the total solids mass, so that when concentrations of the target analytes are expressed on a solids basis, the actual mass of the analyte leaving the process in the solids can be correctly calculated.

The behaviour of metals in the solids treatment processes is assessed somewhat differently than are the organic pharmaceutical, fragrance and alkylphenolic compounds. Metals cannot be removed from or reduced in mass in the treatment processes, with some unusual exceptions (e.g., methylation of mercury) that are not considered to be in effect at the sites tested. As a result, the metals are considered to be "conservative". Interpretation of the metals mass balances therefore leads to an estimate of the mass balance closure (i.e. how closely the input and output masses match). The organic compounds, conversely, can be transformed or "removed" by a number of biological, physical or chemical processes in the solids treatment units. As long as the masses in the input and output streams have been accounted for, it is possible to compare the total output mass with the input mass. The difference between these two quantities is the process removal efficiency. When the total output mass is smaller than the input, a positive removal is obtained. When the combined output mass is larger than the input mass, a negative removal efficiency is calculated. Negative removal efficiencies are not necessarily an indication of error in the calculation process, as reactions can occur in the treatment units that can produce more of the target analyte by biotransformation or chemical reaction, resulting in an apparent increase in mass through the process (e.g., estrone from 17β-estradiol in aerobic processes; nonylphenol from mono- or diethoxylated nonylphenol in anaerobic digestion).

The mass balance closures consider only the disappearance or reduction of a specific organic compound. Many compounds can be biologically transformed to metabolites (e.g. alkylphenol ethoxylates to alkylphenols; the cardiac drug nifedipine to dehydronifedipine) that may be of as great environmental or health significance as the original compound.

4. RESULTS

4.1 Overview

The results of the sampling program are presented in this section. The general format is a brief description of the wastewater treatment plant, a description of the biosolids or sludge treatment process of interest, the results of the sampling program in terms of frequency of detection, median concentrations in the process streams and range of concentrations observed. Mass balance estimates of the metals, pharmaceuticals, fragrance and alkylphenolic compounds are then developed. Lastly, there is a brief assessment of the capability of the process for removing the organic ESOC. Metals cannot be removed during biosolids treatment processes.

The solids produced by the different treatment processes examined in this report can be called by different terms. In the discussion following, the treated product is referred to as biosolids if it has achieved a minimum level of treatment consistent with Class B biosolids, as identified in Section 2.1. Otherwise, the processed solids are referred to as sludges.

4.2 Autothermal Thermophilic Aerobic Digestion, Salmon Arm, BC

4.2.1 Site Description

The Salmon Arm facility is a biological nutrient removal (BNR) system with primary clarification. The treated effluent is disinfected by ultraviolet (UV) light prior to discharge to Shuswap Lake. The design capacity of the existing treatment plant is 9,200 m^3/d, while the average daily dry weather flow is 4,900 m^3/d.

4.2.2 Biosolids Treatment Description

The wastewater sludge is treated by autothermal thermophilic aerobic digestion (ATAD). There are in total 6 ATAD reactor vessels at the facility. Two types of raw sludge are pumped to the ATAD tanks separately, including fermented primary sludge from the primary sludge anaerobic fermenter, and thickened waste activated sludge. During this study, the solids concentration in the feed sludge to the ATAD reactors ranged from 5.7% to 11%, while the aerobically digested sludge solids concentration ranged from 2.6% to 7.3%

After the aerobic digestion, the processed biosolids are cooled and sent to centrifuges (two) for dewatering. Dewatered biosolids are sent to agricultural lands. The centrate is discharged to a lagoon prior to being returned to the headworks of the plant liquid train The pumping rate of feed sludge to the ATAD process is 20 m^3/d, resulting in a nominal hydraulic retention time of 15 d, assuming ideal mixing conditions (no stagnant zone or short-circuiting).

For this project, the biosolids treatment process of interest was the ATAD system. The sampling locations included the ATAD digester inlet (a mixture of fermented primary sludge and thickened WAS) and the ATAD process effluent serving as feed to the dewatering centrifuges. There was no additional sampling location for a digester supernatant return to the plant headworks. A process schematic of the Salmon Arm biosolids treatment process is shown in **Figure 1**.

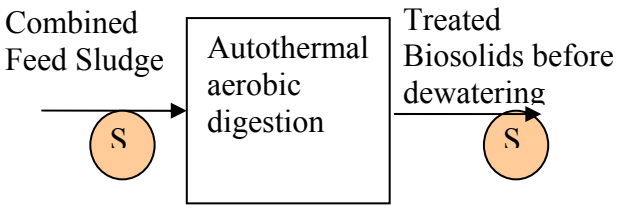

Figure 1. Schematic of Salmon Arm Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on June 29, August 11 and August 19.

4.2.3 Sampling Campaign Results

4.2.3.1 Nutrients

Based on the one set of nutrient data from grab samples, interpretation of the data is limited. Concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) were higher in the digester effluent than in the sludge feed (**Table 5**). Concentrations of total- and ortho-phosphorus were observed at lower concentrations in the aerobically digested solids than in the feed sludge. The observed differences may be due to variations in the composition of the two process streams at the time of sampling.

Table 5. Nutrients in Feed Sludge and Aerobically Digested Biosolids from Salmon Arm, BC

Parameter	Concentration (mg/L)	
	ATAD Feed Sludge	ATAD Digested Sludge
Total Solids	57,000	26,000
Nitrate-N	<2.0	<10
Nitrite-N	<2.0	<10
Total Kjeldahl Nitrogen	1,620	2,350
Ammonia as N	343	756
Phosphorus, Total	1,440	1,170
Phosphate-P (ortho)	1,010	408

4.2.3.2 Metals

Few metals were identified above the detection limits, as shown in **Table 6.** The exceptions were copper, mercury and zinc. Both samples were analyzed as liquid matrices, with concentrations reported in units of mg/L. The concentrations of copper and zinc were approximately equal in the feed sludge and aerobically digested biosolids, while the concentration of mercury in the digested biosolids was approximately three times higher than the concentration in the feed sludge.

Table 6. Metals in Feed Sludge and Aerobically Digested Biosolids from Salmon Arm, BC

Metal	Concentration (mg/L)	
	ATAD Feed Sludge	ATAD Digested Sludge
Arsenic (As)-Total	<1.0	<1.0
Cadmium (Cd)-Total	<0.10	<0.10
Chromium (Cr)-Total	<1.0	<1.0
Cobalt (Co)-Total	<0.80	<0.80
Copper (Cu)- Total	**19.7**	**20.4**
Lead (Pb)-Total	<1.0	<1.0
Mercury (Hg)- Total	**0.0130**	**0.0392**
Molybdenum (Mo)-Total	<1.0	<1.0
Nickel (Ni)-Total	<2.0	<2.0
Selenium (Se)-Total	<5.0	<5.0
Zinc (Zn)-Total	**12.7**	**13.4**
Total Solids	49,600	27,500

Data in **bold font** are above the detection limit

4.2.3.3 Pharmaceuticals

The frequency of detection and median detected concentrations of the pharmaceutical compounds in the raw sludge feed and the ATAD biosolids at the Salmon Arm facility are presented in **Table 7**. The raw analytical data for the pharmaceuticals in the process streams are provided in **Appendix Table A1**. A total of 20 pharmaceuticals were detected in the digester feed samples from all three sampling campaigns; seventeen pharmaceuticals were detected in all digester effluent samples from the three campaigns.

Table 7. Frequency of Detection and Median Concentrations of Pharmaceutical Compounds in Salmon Arm, BC Feed Sludge and Aerobically Digested Biosolids

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Furosemide	1	1	233 [a]	543 [a]	<111-233	<108-543
Gemfibrozil	**3**	**3**	**47.2**	**219**	42.2-49.9	177-245
Glipizide	0	0	NA	NA	<24.3 [b]	<68.4 [b]
Glyburide	0	0	NA	NA	<12.1 [b]	<34.2 [b]
Hydrochlorothiazide	1	0	106 [a]	NA	<55.5-106	<228 [b]
2-Hydroxy-ibuprofen	2	**3**	498	**1160**	<222-609	571-1570
Ibuprofen	**3**	**3**	**359**	**1960**	196-466	1130-3010
Naproxen	**3**	**3**	**85.8**	**278**	82.7-127	247-431
(continued)						

Table 7 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Triclocarban	**3**	**3**	**3360**	**5010**	3080-3700	4900-6700
Triclosan	**3**	**3**	**8390**	**21500**	6670-9640	21300-24000
Warfarin	0	0	NA	NA	<6.07 [b]	<17.1 [b]
Acetaminophen	2	0	333	NA	<167-367	<430 [b]
Azithromycin	**3**	**3**	**154**	**220**	112-267	219-385
Caffeine	**3**	**3**	**1270**	**4110**	1260-1360	2960-4550
Carbadox	0	0	NA	NA	<6.07 [b]	<10.7 [b]
Carbamazepine	**3**	**3**	**213**	**717**	168-423	579-2360
Cefotaxime	0	0	NA	NA	<218 [b]	<1260 [b]
Ciprofloxacin	**3**	**3**	**13000**	**6900**	9620-14400	4220-8210
Clarithromycin	**3**	**3**	**71.1**	**126**	50.4-344	73.4-249
Clinafloxacin	0	0	NA	NA	<36 [b]	<91.2 [b]
Cloxacillin	0	0	NA	NA	<13.6 [b]	<44.5 [b]
Dehydronifedipine	**3**	2	**9.19**	6.04	6.28-9.94	<2.51-7.36
Diphenhydramine	**3**	**3**	**451**	**612**	424-559	514-807
Diltiazem	**3**	**3**	**192**	**21.7**	162-480	5.94-27.7
Digoxin	0	0	NA	NA	<138 [b]	<107 [b]
Digoxigenin	0	0	NA	NA	<42.9 [b]	<130 [b]
Enrofloxacin	**3**	0	**14.1**	NA	12.5-20.4	<21.5 [b]
Erythromycin-H_2O	**3**	**3**	**27.5**	**31.9**	18.8-33.4	29.8-95.3
Flumequine	0	0	NA	NA	<6.07 [b]	<14 [b]
Fluoxetine	**3**	**3**	**127**	**96.7**	122-153	80.1-278
Lincomycin	0	1	NA	71.07 [a]	<28.3 [b]	<12.3-71.07
Lomefloxacin	0	0	NA	NA	<12.1 [b]	<21.5 [b]
Miconazole	**3**	**3**	**683**	**1350**	533-901	1160-1710
Norfloxacin	**3**	1	**410**	154 [a]	99.2-434	<57-154
Norgestimate	0	0	NA	NA	<12.1 [b]	<37 [b]
Ofloxacin	**3**	**3**	**326**	**245**	300-394	187-279
Ormetoprim	0	0	NA	NA	<2.43 [b]	<4.3 [b]
Oxacillin	0	0	NA	NA	<12.1 [b]	<21.5 [b]
Oxolinic Acid	0	0	NA	NA	<2.68 [b]	<6.15 [b]
Penicillin G	0	0	NA	NA	<27.8 [b]	<38 [b]
Penicillin V	0	1	NA	59.3 [a]	<12.1 [b]	<8.06-59.3
Roxithromycin	0	0	NA	NA	<1.21 [b]	<2.15 [b]
Sarafloxacin	0	0	NA	NA	<276 [b]	<135 [b]
Sulfachloropyridazine	0	0	NA	NA	<6.07 [b]	<10.7 [b]
Sulfadiazine	0	0	NA	NA	<6.07 [b]	<10.7 [b]
Sulfadimethoxine	0	0	NA	NA	<1.21 [b]	<66.2 [b]
Sulfamerazine	0	2	NA	27.75	<2.43 [b]	<1.61-36.3
Sulfamethazine	0	0	NA	NA	<3.44 [b]	<5.45 [b]

(continued)

Table 7 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Sulfamethizole	0	0	NA	NA	<2.75 [b]	<7.19 [b]
Sulfamethoxazole	**3**	1	**25.8**	3.41 [a]	20.3-43	<1.61-3.41
Sulfanilamide	0	2	NA	128.25	<60.7 [b]	<40.3-164
Sulfathiazole	0	0	NA	NA	<6.07 [b]	<10.7 [b]
Thiabendazole	**3**	**3**	**16**	**21.7**	14.1-18	13.4-27.5
Trimethoprim	**3**	1	**60.2**	36.4 [a]	56.5-75.3	<4.03-36.4
Tylosin	0	0	NA	NA	<81 [b]	<53.8 [b]
Virginiamycin	0	1	NA	197 [a]	<115 [b]	<127-197
1,7-Dimethylxanthine	1	**3**	1030 [a]	**1850**	<416-1030	1030-2800

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The distribution of detectable concentrations in the ATAD process feed and effluent streams from the three sampling campaigns is found in **Table 8**. There appeared to be a minor difference in the distribution of detectable concentrations in the digester feed to the process effluent. Most notably the number of compounds detected in all three campaigns declines from 20 in the digester feed to 17 in the process effluent, while the number of compounds detected in only one of the three campaigns rose from three in the digester feed to 6 in the ATAD effluent.

Table 8. Summary of Pharmaceutical Compound Detections in Feed and Aerobically Digested Sludge, Salmon Arm, BC

# Detects in process stream for 3 sampling campaigns	# Compounds in Process Streams	
	Feed	Digested
3	20	17
2	4	5
1	3	6
0	29	28
Total	56	56

4.2.3.4 Fragrances and Alkylphenolics

Concentrations and frequency of detection of fragrances and alkylphenolics are provided in **Table 9**. The raw analytical data are provided in **Appendix Table A2**. The compounds observed at the highest concentrations (e.g. greater than 1,000 ng/g TS) in the digested biosolids were Bisphenol A and the synthetic polycyclic musks HHCB, AHTN and ATII. With the exception of musk xylene observed in both digester feed sludge samples, no nitro musks were

detected in either the feed sludge or anaerobically digested biosolids. The musk xylene was reduced to below the detection limit in the digested biosolids. Additional discussion of the fragrance and alkylphenolics is found later in this section under Data Interpretation.

Table 9. Frequency of Detection, Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Feed Sludge and ATAD Biosolids from Salmon Arm, BC

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Digester Feed Sludge	Digested Biosolids	Digester Feed Sludge	Digested Biosolids	Digester Feed Sludge	Digested Biosolids
Alkylphenolics						
Bisphenol A	**2**	**2**	**785**	**1220**	520-1050	700-1740
Octylphenol	1	1	60	70	<20-60	<20-70
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	**2**	**2**	**125**	**195**	120-130	180-210
ADBI	0	0	NA	NA	<20	<20
AHDI	**2**	**2**	**565**	**370**	230-900	210-530
HHCB	**2**	**2**	**6975**	**8685**	6430-7520	8570-8800
AHTN	**2**	**2**	**3690**	**4440**	3510-3870	4230-4650
ATII	**2**	**2**	**760**	**1025**	740-780	910-1140
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	<120	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	**2**	0	**245**	NA	80-410	<70

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in both sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

4.2.4 Data Interpretation

4.2.4.1 Metals Mass Balances

Mass balances for metals around the ATAD process were determined by multiplying the volumetric throughput rate of feed sludge and digested biosolids (20 m^3/d) by the liquid-based concentrations of the metals (in units of mg/L). Only mass calculations for the detected metals are presented in **Table 10**. For copper and zinc, the masses in and out were nearly identical, indicating that the mass of the metals is conserved through the process. The mass of mercury in the ATAD effluent, at 0.784 g/d, is substantially higher than the input mass of mercury of 0.260 g/d. Possible reasons for the apparent increase in the mass of mercury in the process effluent include some mercury being present in a form that was not analysed in the digester feed,

accumulation of mercury from materials of construction of the digester, non-steady state conditions with respect to mercury, or possible sample contamination during the collection or analytical stage.

Table 10. Mass Balance and Removal Calculations for Metals in ATAD Process, Salmon Arm BC

Metal	Concentration (mg/L)		Mass of Contaminant (g/d)		% Removal
	ATAD Feed Sludge	ATAD Digested Sludge	ATAD Feed Sludge	ATAD Digested Sludge	
Copper (Cu)-Total	19.7	20.4	394	408	-4%
Mercury (Hg)	0.0130	0.0392	0.260	0.784	-202%
Zinc (Zn)-Total	12.7	13.4	254	268	-6%

4.2.4.2 Pharmaceutical Compounds Mass Balances

Because concentrations for the pharmaceutical compounds are expressed on a dry weight basis, mass balances for the pharmaceutical compounds are based on a total solids balance around the ATAD process. The solids balance around the ATAD process is estimated using the mean values of the total solids concentrations in the sludge feed and ATAD biosolids out of the process from the three sampling campaigns. The pertinent solids concentration and flow data are:

ATAD volumetric throughput = 20 m^3/d
Mean measured total solids concentration in feed sludge = 79.33 kg/m^3
Mean measured total solids concentration in digested biosolids = 50.33 kg/m^3

In the balance, it was assumed the difference in the total mass of solids entering and leaving the digester was the mass of volatile solids lost through the process. The total solids balance is depicted in **Figure 2**.

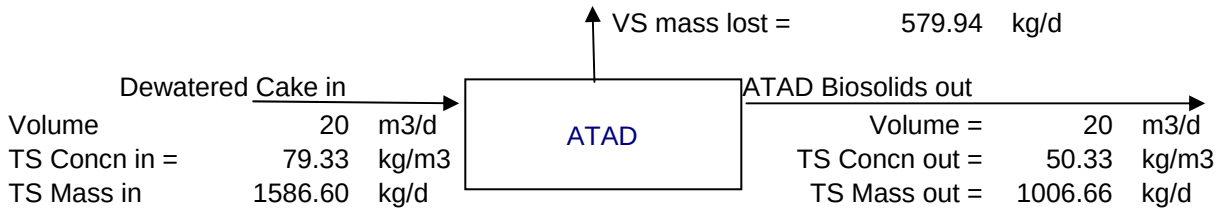

Figure 2. Solids Mass Balance around ATAD Process, Salmon Arm, BC

Concentrations of the contaminants measured on a dry weight basis (i.e. ng/g TS) were converted to a mass flow rate (mg/d) for comparison of input and output masses. The results of the mass estimates are provided in **Table 11**. Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids were not included in the **Table 11**.

Table 11. Mass Balance and Removal Calculations for Pharmaceutical Compounds in ATAD Process, Salmon Arm BC

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	
Furosemide	233	543	182	315	-73%
Gemfibrozil	47.2	219	36.8	127	-245%
Hydrochlorothiazide	106	<143	83.5	<83.8	>-0.3%
2-Hydroxy-ibuprofen	498	1160	388	673	-73%
Ibuprofen	359	1960	280	1137	-306%
Naproxen	85.8	278	66.9	161	-141%
Triclocarban	3360	5010	2621	2906	-11%
Triclosan	8390	21500	6544	12470	-91%
Acetaminophen	333	<228	262	<134	>49%
Azithromycin	154	220	120	128	-6%
Caffeine	1270	4110	991	2384	-141%
Carbamazepine	213	717	166	416	-150%
Ciprofloxacin	13000	6900	10140	4002	61%
Clarithromycin	71.1	126	55.5	73.1	-32%
Dehydronifedipine	9.19	6.035	7.17	3.50	51%
Diphenhydramine	451	612	352	355	-1%
Diltiazem	192	21.7	150	12.6	92%
Enrofloxacin	14.1	<20.1	11.1	<11.8	>-6%
Erythromycin-H_2O	27.5	31.9	21.5	18.5	14%
Fluoxetine	127	96.7	99.1	56.1	43%
Miconazole	683	1350	533	783	-47%
Norfloxacin	410	154	320	89.3	72%
Ofloxacin	326	245	254	142	44%
Sulfamethoxazole	25.8	3.41	20.1	1.98	90%
Thiabendazole	16.0	21.7	12.6	12.6	-1%
Trimethoprim	60.2	36.4	47.0	21.1	55%
1,7-Dimethylxanthine	1030	1850	803	1073	-34%

A categorisation of the removal efficiencies of the pharmaceutical compounds is presented in **Table 12**. Six pharmaceutical compounds were identified as having removal efficiencies greater than or equal to 50%, while two of the six compounds (diltiazem and sulfamethoxazole) had removal efficiencies greater than or equal to 90%. More of the calculated removal efficiencies for the different pharmaceutical compounds have negative values than positive values. On the most basic level of interpretation, this observation implies that more of the compound mass is exiting the process than arrives in the feed stream. Because biodegradation can involve the formation of metabolites that are already included measured as compounds in the feed sludge, the apparent increase through the digestion process may in some cases be due to the formation of metabolites. This bio-conversion process cannot explain all of the observed negative removal efficiencies, however; an error in the total solids concentration could also contribute to the number of negative removal efficiencies.

Table 12. Categorised Removal Efficiencies of Pharmaceutical Compounds by Autothermal Thermophilic Aerobic Digestion, Salmon Arm, BC

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Furosemide	Triclocarban	Erythromycin-H_2O	Ciprofloxacin	Diltiazem
Gemfibrozil	Azithromycin	Fluoxetine	Dehydronifedipine	Sulfamethoxazole
2-Hydroxy-ibuprofen	Clarithromycin	Ofloxacin	Norfloxacin	
Ibuprofen	Diphenhydramine	Hydrochlorothiazide	Trimethoprim	
Naproxen	Miconazole	Acetaminophen		
Triclosan	Thiabendazole			
Caffeine	1,7-Dimethylxanthine			
Carbamazepine	Enrofloxacin			
n=8	n=8	n=5	n=4	n=2

4.2.4.3 Fragrance and Alkylphenolic Compounds

Mass balance results for the fragrance and alkylphenolic compounds in the ATAD process are presented in **Table 13**. The basis for the mass balances is the same as for the pharmaceutical compounds. Only compounds with detectable concentrations in the feed sludge are listed in the table. No removal of Bisphenol A was observed through the process, while octylphenol was removed through the digester to a minor extent at 13%. Musk xylene had the highest calculated removal efficiency of >79%, and the polycyclic musk AHDI was removed by 51%. All of the other polycyclic fragrances were calculated to have only minor or zero removal efficiencies, indicating minimal biodegradation of these compounds in autothermal aerobic digestion.

Table 13. Mass Balance and Removal Calculations for Fragrance and Alkylphenolic Compounds in ATAD Process, Salmon Arm, BC

Fragrance and Phenolic Compounds	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Digester Feed Sludge	Digested Sludge	Digester Feed Sludge	Digested Sludge	
Alkylphenolics					
Bisphenol A	785	1220	618.4	714.7	-16%
Octylphenol	60	70	47.3	41.0	13%
Fragrances					
DPMI	125	195	98.5	114.2	-16%
AHDI	565	370	445	217	51%
HHCB	6975	8685	5495	5088	7%
AHTN	3690	4440	2907	2601	11%
ATII	760	1025	599	600	0%
Musk Xylene	245	<70	193	<41.0	>79%

4.2.4.4 Effectiveness of ATAD Process for ESOC Removal

Taken as a whole, the removal efficiencies calculated for the ATAD process at Salmon Arm, BC indicate that the biosolids treatment process is only partially successful in reducing the incoming mass of pharmaceutical, fragrance or alkylphenolic compounds.

4.2.5 Section Summary

Concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) were higher in the digester effluent than in the sludge feed; conversely, concentrations of total- and ortho-phosphorus were observed at lower concentrations in the aerobically digested solids than in the feed sludge. The inconsistent behaviour of the nutrients is based on only one set of samples, and may be due to variations in the composition of the two process streams at the time of sampling.

Few metals were identified above the detection limits, with the exceptions of copper, mercury and zinc. For copper and zinc, the masses in and out are nearly identical, indicating that the mass of the metals is conserved through the process. The mass of mercury in the ATAD effluent, at 0.784 g/d, is substantially higher than the input mass of mercury of 0.260 g/d possibly due to a number of potential causes outlined in the main text.

A total of 20 pharmaceuticals were detected in the digester feed samples of all three sampling campaigns; seventeen pharmaceuticals were detected in all digester effluent samples from the three campaigns. More of the calculated removal efficiencies for the different pharmaceutical compounds have negative values than positive values. The apparent increase through the digestion process may in some cases be due to the formation of metabolites; however, this bio-conversion process cannot explain all of the observed negative removal efficiencies. Six pharmaceutical compounds were identified as having removal efficiencies greater than or equal to 50%, while two of the six compounds (diltiazem and sulfamethoxazole) had removal efficiencies greater than or equal to 90%.

The compounds observed at the highest concentrations (e.g. greater than 1,000 ng/g TS) in the digested biosolids were Bisphenol A and the synthetic polycyclic musks HHCB, AHTN and ATII. With the exception of musk xylene observed in both digester feed sludge samples, no nitro musks were detected in either the feed sludge or anaerobically digested biosolids. The musk xylene was reduced to below the detection limit in the digested biosolids. No removal of Bisphenol A was observed through the process, while octylphenol was removed through the digester to a minor extent at 13%. Musk xylene had the highest calculated removal efficiency of >79%, and the polycyclic musk AHDI was removed by 51%. All of the other polycyclic fragrances were calculated to have only minor or zero removal efficiencies, indicating minimal biodegradation of these compounds in autothermal aerobic digestion.

Taken as a whole, the removal efficiencies calculated for the ATAD process at Salmon Arm, BC indicate that the biosolids treatment process is only partially successful in reducing the incoming mass of pharmaceutical, fragrance or alkylphenolic compounds.

4.3 Mesophilic Anaerobic Digestion, Red Deer, AB

4.3.1 Site Description

The Red Deer facility is a biological nutrient removal (BNR) system with ultraviolet (UV) light disinfection of the treated effluent prior to discharge to the Red Deer River. The design capacity of the existing treatment plant is 47,500 m^3/d, while the average daily dry weather flow is 37,809 m^3/d.

4.3.2 Biosolids Treatment Description

The excess wastewater sludge is treated by anaerobic digestion. There are in total three (3) primary digesters and one (1) secondary digester at the facility. Three separate types of raw sludge are pumped to the anaerobic digesters, including unfermented primary sludge from primary clarifiers, fermented primary sludge from the primary sludge anaerobic fermenter, and thickened waste activated sludge (WAS) from dissolved air flotation (DAF). The flow of feed sludge to the anaerobic digester is 350 $m^{3/}$d. The capacity of the anaerobic digester is 2,000 m^3 each, resulting in a nominal hydraulic capacity of 17 days. During this study, the solids concentration in the feed sludge to the digesters ranged from 1.1% to 9.4%, while the anaerobically digested sludge solids concentration ranged from 1.0% to 8.9%.

After the anaerobic digestion, the processed biosolids are sent to a lagoon for dewatering. Dewatered biosolids are sent to agricultural lands. The liquid decant is sent to the headworks of the plant liquid train for treatment.

For this project assessment, the biosolids treatment process of interest was the mesophilic anaerobic sludge digestion system. The two sampling locations included the anaerobic digester inlet (a mixture of unfermented primary sludge, fermented primary sludge and thickened WAS) and outlet (the digested sludge) serving as feed to the sludge dewatering lagoon. There was no additional sampling location for the dewatering lagoon supernatant return to the plant headworks. A process schematic of the Red Deer biosolids treatment process is shown in **Figure 3**.

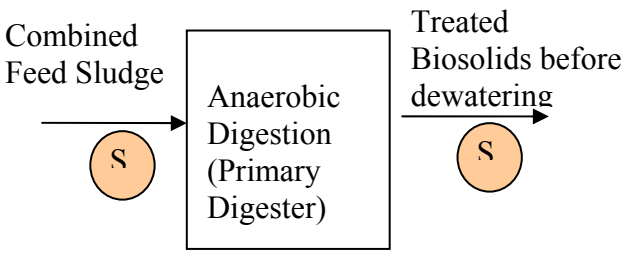

Figure 3. Schematic of Red Deer Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on June 29, July 23 and August 27, 2009, respectively

4.3.3 Sampling Results

4.3.3.1 Nutrients

Concentrations of nutrients in the combined digester feed sludge and digested biosolids are presented in **Table 14**. The concentration of ammonia-N (a component of total Kjeldahl nitrogen, or TKN) increases through the digestion process as expected, due to breakdown of proteins in the feed biomass. The concentration of TKN appears to increase slightly through the digestion process, but on a mass basis the increase is slight, on the order of a 6% difference. Concentrations of nitrite and nitrate ion are low in both the digester feed sludge and in the digester effluent (**Table 14**). Feed concentrations of these ions are believed to be low due to the denitrification process used in the liquid treatment train at the Red Deer facility, a BNR plant. Nitrate and nitrite ion concentrations are low in the digested biosolids because there is no nitrate or nitrite formation during anaerobic digestion, which occurs in the absence of oxygen.

Both total phosphorus and ortho-phosphate were observed at higher concentrations and masses in the digested biosolids compared to the feed sludge. Total phosphorus is a conservative mass, and should not change from influent to effluent. Possible reasons for the observed increase in total phosphorus through the digester include mobilization from accumulated reserves in the digestion tank, or sampling and analytical variability resulting from one set of grab samples.

Table 14. Nutrients in Feed Sludge and Anaerobically Digested Sludge from Red Deer, AB

Parameter	Concentration (mg/L)	
	Digester Feed Sludge	Anaerobically Digested Sludge
Nitrate-N	<2.0	<2.0
Nitrite-N	<2.0	<2.0
Total Kjeldahl Nitrogen	4,330	4,590
Ammonia as N	329	734
Phosphorus, Total	1,290	1,500
Phosphate-P (ortho)	206	240
Total Solids	28,600	16,700

4.3.3.2 Metals

Most metals were identified above the detection limits in the digester feed sludge, as shown in **Table 15**. Exceptions were arsenic, cobalt and selenium. Only a few metals, i.e., chromium, copper, mercury and zinc, were detected in the anaerobically digested sludge. Because metals are conservative, the reduced number of detected species in the digested sludge, compared to the feed sludge, is most likely due to the effect of the matrix on the detection limits. Concentrations of both solids were analyzed as liquid matrices, with results reported in units of mg/L. The highest and lowest concentrations of metals in the feed and digested sludges were associated with zinc and mercury, respectively. With the exception of mercury, concentrations of the metals detected in both the feed sludge and digested biosolids were similar in magnitude. The concentration of mercury in the digested biosolids sample was approximately an order of magnitude higher than in the combined feed sludge.

Table 15. Metals in Combined Feed Sludge and Anaerobically Digested Sludge from Red Deer, AB

Metal	Concentration (mg/L)	
	Digester Feed Sludge	Anaerobically Digested Sludge
Arsenic (As)-Total	<0.10	<1.0
Cadmium (Cd)-Total	**0.020**	<0.10
Chromium (Cr)-Total	**1.32**	**2.0**
Cobalt (Co)-Total	<0.080	<0.80
Copper (Cu)-Total	**5.60**	**5.8**
Lead (Pb)-Total	**1.34**	<1.0
Mercury (Hg)-Total	**0.00703**	**0.0531**
Molybdenum (Mo)-Total	**0.20**	<1.0
Nickel (Ni)-Total	**0.32**	<2.0
Selenium (Se)-Total	<0.50	<5.0
Zinc (Zn)-Total	**10.8**	**10.8**
Total Solids	28,600	16,700

Data in **bold font** are above the detection limit

4.3.3.3 Pharmaceuticals

The frequency of detection and median detected concentrations of the pharmaceutical compounds in the raw sludge feed and the anaerobically digested sludge at the Red Deer facility are presented in **Table 16.** A total of 22 pharmaceuticals were detected in the digester feed samples in all three sampling campaigns; 15 pharmaceuticals were detected in all digester effluent samples from the three campaigns. The reported concentration data from each of the three sampling campaigns are found in **Appendix Table A3**.

Table 16. Frequency of Detection and Median Concentrations of Pharmaceutical Compounds in Raw Sludge and Anaerobically Digested Sludge in Red Deer, AB.

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Furosemide	0	1	NA	93.9 [a]	<732 [b]	<88.8-93.9
Gemfibrozil	**3**	**3**	**18**	**57.5**	17.8-136	22.4-75.7
Glipizide	0	0	NA	NA	<110 [b]	<104 [b]
Glyburide	0	0	NA	NA	<54.9 [b]	<51.9 [b]
Hydrochlorothiazide	1	1	164 [a]	349 [a]	<41.8-164	<44.4-349
2-Hydroxy-ibuprofen	**3**	1	**755**	348 [a]	638-1540	<178-348
Ibuprofen	**3**	**3**	**502**	**686**	257-1330	350-1910
(continued)						

Table 16 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Naproxen	**3**	1	73.6	35.3 [a]	66.4-270	<6.66-35.3
Triclocarban	**3**	**3**	**2660**	**4410**	2440-10800	3560-4710
Triclosan	**3**	**3**	**9130**	**12700**	5340-36800	11700-13900
Warfarin	0	0	NA	NA	<27.4 [b]	<25.9 [b]
Acetaminophen	2	0	366	NA	<126-551	<1040
Azithromycin	**3**	**3**	**616**	**679**	459-1830	419-793
Caffeine	**3**	1	**2530**	175 [a]	1700-8970	<33.3-175
Carbadox	0	0	NA	NA	<27.4 [b]	<26 [b]
Carbamazepine	**3**	**3**	**260**	**987**	230-1070	430-1060
Cefotaxime	0	0	NA	NA	<567 [b]	<1320 [b]
Ciprofloxacin	**3**	**3**	**4390**	**6520**	3690-17000	4140-8450
Clarithromycin	**3**	**3**	**142**	**77.4**	62.3-442	20.2-111
Clinafloxacin	0	0	NA	NA	<116 [b]	<120 [b]
Cloxacillin	0	0	NA	NA	<54.8 [b]	<67.6 [b]
Dehydronifedipine	**3**	0	**6.17**	NA	3.75-14.3	<10.4 [b]
Diphenhydramine	**3**	**3**	**1640**	**2300**	1510-4380	1980-2360
Diltiazem	**3**	**3**	**209**	**35.2**	129-947	17.2-41.2
Digoxin	1	0	560 [a]	NA	<31.4-560	<260 [b]
Digoxigenin	1	1	257 [a]	193 [a]	<35.4-257	<27.8-193
Enrofloxacin	2	2	44.84	27.3	<6.28-82.9	<13.1-35.5
Erythromycin-H_2O	**3**	**3**	**36.7**	**20**	24.1-171	5.25-26.9
Flumequine	0	0	NA	NA	<27.4 [b]	<26 [b]
Fluoxetine	**3**	**3**	**154**	**255**	126-373	120-297
Lincomycin	1	0	28.8 [a]	NA	<14.6-28.8	<51.9 [b]
Lomefloxacin	0	0	NA	NA	<54.8 [b]	<51.9 [b]
Miconazole	**3**	**3**	**429**	**1090**	225-1720	518-1220
Norfloxacin	**3**	**3**	**2100**	**3270**	1910-10200	1810-4380
Norgestimate	0	0	NA	NA	<57.8 [b]	<51.9 [b]
Ofloxacin	**3**	**3**	**416**	**712**	263-1790	649-1290
Ormetoprim	0	0	NA	NA	<11 [b]	<10.4 [b]
Oxacillin	0	0	NA	NA	<61.5 [b]	<51.9 [b]
Oxolinic Acid	0	0	NA	NA	<11 [b]	<12.5 [b]
Penicillin G	0	0	NA	NA	<54.8 [b]	<51.9 [b]
Penicillin V	0	0	NA	NA	<54.8 [b]	<51.9 [b]
Roxithromycin	0	0	NA	NA	<7.89 [b]	<5.19 [b]
Sarafloxacin	0	0	NA	NA	<408 [b]	<346 [b]
Sulfachloropyridazine	0	0	NA	NA	<27.4 [b]	<26 [b]
Sulfadiazine	0	0	NA	NA	<27.4 [b]	<26 [b]
Sulfadimethoxine	0	0	NA	NA	<5.48 [b]	<5.19 [b]
Sulfamerazine	0	0	NA	NA	<12.9 [b]	<10.4 [b]

(continued)

Table 16 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Sulfamethazine	1	0	6.13 [a]	NA	<1.49-6.13	<10.4 [b]
Sulfamethizole	0	0	NA	NA	<11 [b]	<10.4 [b]
Sulfamethoxazole	**3**	0	**22**	NA	16.7-43.6	<10.4 [b]
Sulfanilamide	0	0	NA	NA	<274 [b]	<260 [b]
Sulfathiazole	0	0	NA	NA	<27.4 [b]	<26 [b]
Thiabendazole	**3**	2	**10**	20.15	9.66-35	<3.33-25.4
Trimethoprim	**3**	0	**58.7**	<14.9	54.8-262	<26 [b]
Tylosin	0	0	NA	NA	<110 [b]	<104 [b]
Virginiamycin	0	0	NA	NA	<373 [b]	<249 [b]
1,7-Dimethylxanthine	1	0	475 [a]	NA	<314-475	<2600 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the digested biosolids included the anti-microbials triclosan and triclocarban, the anti-biotics ciprofloxacin and norfloxacin, the stimulants caffeine and diphenhydramine, the anti-fungal miconazole.

The distribution of detectable concentrations in the anaerobic digestion process feed and effluent streams from the three sampling campaigns is found in **Table 17**. There appears to be a small shift in the distribution of detectable concentrations in the digester feed to the process effluent. Most notably the number of compounds detected in all three campaigns declines from 22 in the digester feed to 15 in the process effluent, while the number of compounds never detected in the digester feed sludge (27) increases to 34 compounds in the anaerobic digester effluent.

Table 17. Summary of Pharmaceutical Compound Detections in Combined Feed Sludge and Anaerobically Digested Sludge, Red Deer, AB

Frequency of detection in sampling campaigns (out of 3)	# Compounds in Process Streams	
	Feed	Digested
3	22	15
2	2	2
1	6	6
0	27	34
Total	57	57

4.3.3.4 Fragrance and Alkylphenolic Compounds

Results for the two sampling rounds are available and are provided in **Table 18**. The raw analytical data are provided in **Appendix Table A4**. Bisphenol A was the only one of the three alkylphenolic compounds detected in the feed and digested biosolids samples. The concentration of BPA was higher in the digested biosolids than in the feed sludge. The polycyclic musk fragrances HHCB and AHTN were detected at the highest concentrations (i.e., greater than 1000 ng/g TS) in the digested biosolids. None of the nitro musk compounds were observed above the limit of quantification. Many compounds were observed at higher concentrations in the digested sludge than in the feed sludge, but mass balances (see Data Interpretation) are required to determine whether the concentration differences are significant.

Table 18. Frequency of Detection, Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Raw Sludge and Anaerobically Digested Sludge, Red Deer, AB

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Digester Feed Sludge	Digested Sludge	Digester Feed Sludge	Digested Sludge	Digester Feed Sludge	Digested Sludge
Alkylphenolics						
Bisphenol A	**2**	**2**	**295**	**515**	140-450	280-750
Octylphenol	0	0	NA	NA	<20	<20
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	1	**2**	190	**215**	<40-190	30-400
ADBI	0	1	NA	60	<20	<20-60
AHDI	1	**2**	780	**100**	<30-780	50-150
HHCB	**2**	**2**	**3615**	**8975**	2640-4590	6830-11120
AHTN	**2**	**2**	**2090**	**4015**	1350-2830	2850-5180
ATII	**2**	**2**	**335**	**520**	120-550	210-830
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	<120	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	0	0	NA	NA	<70	<70

Data in **bold font** are detected in both sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

4.3.4 Data Interpretation

4.3.4.1 Metals Mass Balance

Mass balances for metals around the anaerobic digestion process were determined by multiplying the volumetric throughput rate of feed sludge and anaerobically digested biosolids (350 m^3/d) by the liquid-based concentrations of the metals (in units of mg/L). Only mass calculations for the detected metals are presented in **Table 19**. Metals with detected feed sludge concentrations are presented in the table even if their concentrations in the digested biosolids were less than the

detected value in an attempt to establish a minimum removal efficiency. Only for lead did this method result in a positive outcome. For the other metals, the minimum mass calculated in the digested biosolids was larger than the mass in the feed sludge, and so no estimate of a minimum removal efficiency was possible.

Table 19. Mass Balance and Removal Calculations for Metals in Mesophilic Anaerobic Digestion Process, Red Deer, AB

Metal	Concentration (mg/L)		Mass of Contaminant (g/d)		% Removal
	Digester Feed Sludge (composite)	Anaerobically Digested Sludge	Digester Feed Sludge (composite)	Anaerobically Digested Sludge	
Cadmium (Cd)-Total	0.020	<0.10	7	<35	NA
Chromium (Cr)-Total	1.32	2.0	462	700	-52%
Copper (Cu)-Total	5.60	5.8	1960	2030	-4%
Lead (Pb)-Total	1.34	<1.0	469	<350	>25%
Mercury (Hg)-Total	0.00703	0.0531	2.46	18.6	-655%
Molybdenum (Mo)-Total	0.20	<1.0	70	<350	NA
Nickel (Ni)-Total	0.32	<2.0	112	<700	NA
Zinc (Zn)-Total	10.8	10.8	3780	3780	0%

For most of the metals in **Table 19**, the calculated mass leaving the digester is larger than the mass in the feed sludge. Since the metals are conservative, with the possible exception of mercury, under steady-state conditions the mass entering and the mass leaving should be equal. Possible reasons for the observed higher exit masses of metals may include temporal variations captured in only one sampling, possible release of metals in the digester effluent following a period of accumulation in the digester, or more complete recovery of the metals in the analysis of the digested biosolids than in the feed sludge.

4.3.4.2 Pharmaceutical Compounds Mass Balance

Concentrations for the pharmaceutical compounds are expressed on a dry weight basis, and so the mass balances for the pharmaceutical compounds are based on a total solids balance around the anaerobic digestion process. The solids balance around the anaerobic digester is estimated using the mean values of the total solids concentrations in the sludge feed and digested biosolids out of the process from the three sampling campaigns. The pertinent solids concentration and flow data are:

Anaerobic digester volumetric throughput = 350 m^3/d
Mean measured total solids concentration in feed sludge = 46.3 kg/m^3
Mean measured total solids concentration in digested biosolids = 39.3 kg/m^3

The wide range of solids concentrations reported for the feed sludge and digested biosolids introduces an element of uncertainty in the mass balance determination. Without additional data to provide a basis for the balance, the mean value of the sampling campaigns was selected for the calculation. In the balance, it was assumed the difference in the total mass of solids entering and leaving the digester was the mass of volatile solids lost through the process. The total solids balance is depicted in **Figure 4**.

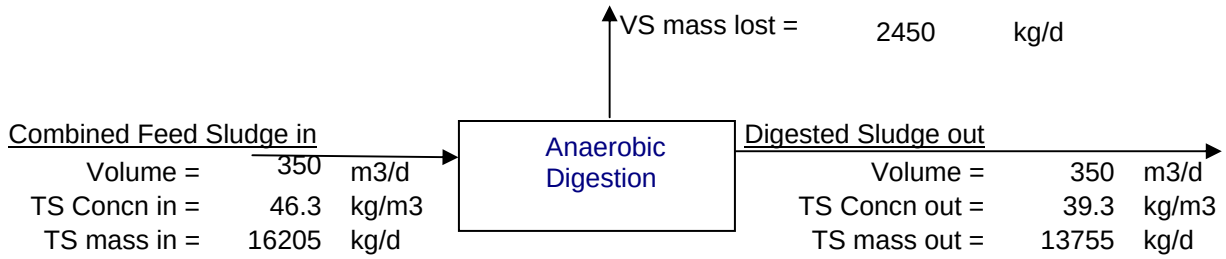

Figure 4 . Solids Mass Balance around Mesophilic Anaerobic Digestion Process, Red Deer, AB

Concentrations of the pharmaceutical compounds measured on a dry weight basis (i.e. ng/g TS) were converted to a mass flow rate (mg/d) for comparison of input and output masses. The results of the mass estimates are provided in **Table 20.** Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids were not included in **Table 20**.

Table 20. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Mesophilic Anaerobic Digestion Process, Red Deer, AB

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Raw sludge	Digested biosolids	Raw sludge	Digested biosolids	
Gemfibrozil	18	57.5	296	793	-168%
Hydrochlorothiazide	164	349	2693	4811	-79%
2-Hydroxy-ibuprofen	755	348	12399	4798	61%
Ibuprofen	502	686	8244	9458	-15%
Naproxen	73.6	35.3	1209	487	60%
Triclocarban	2660	4410	43683	60798	-39%
Triclosan	9130	12700	149933	175089	-17%
Acetaminophen	366	<594	6010	<8189	NR
Azithromycin	616	679	10116	9361	7%
Caffeine	2530	175	41548	2413	94%
Carbamazepine	260	987	4270	13607	-219%
Ciprofloxacin	4390	6520	72093	89888	-25%
Clarithromycin	142	77.4	2332	1067	54%
Dehydronifedipine	6.17	<5.94	101	<82	>19%
Diphenhydramine	1640	2300	26932	31709	-18%
Diltiazem	209	35.2	3432	485	86%
Digoxin	560	<242	9196	<3336	>64%
Digoxigenin	257	193	4220	2661	37%
Enrofloxacin	44.84	27.3	736	376	49%
Erythromycin-H_2O	36.7	20	603	276	54%
Fluoxetine	154	255	2529	3516	-39%
Lincomycin	28.8	<29.7	473	<409	>13%
Miconazole	429	1090	7045	15027	-113%
Norfloxacin	2100	3270	34486	45082	-31%
(continued)					

Table 20 (continued)

Ofloxacin	416	712	6832	9816	-44%
Sulfamethazine	6.13	<5.94	101	<82	>19%
Sulfamethoxazole	22	<5.94	361	<82	>77%
Thiabendazole	10	20.15	164	278	-69%
Trimethoprim	58.7	<14.9	964	<205	>79%
1,7-Dimethylxanthine	475	<1490	7800	<20542	NR

NR = no result

The highest removal efficiency in the process was associated with caffeine, at 94%, followed by diltiazem at 86%. Both trimethoprim and sulfamethoxazole appear to be efficiently removed as well, with calculated minimum removal efficiencies of 79% and 77%, respectively. Conversely, compounds such as carbamazepine, gemfibrozil and miconazole were associated with high negative removals of -219%, -168%, and -113%, respectively, indicating their masses in digested biosolids were much higher than in the feed sludge.

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 21**. The number of pharmaceutical compounds with positive removal efficiencies (i.e., greater than 0) at 15 was approximately the same as the number of compounds with negative removal efficiencies (i.e., less than 0) at 13. It is noted here, however, that the variability in the feed and digested solids concentrations can affect the solids balance and hence the determination of positive and negative removal efficiencies.

Table 21. Categorised Removal Efficiencies of Pharmaceutical Compounds by Mesophilic Anaerobic Digestion, Red Deer, AB

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Gemfibrozil	Ibuprofen	Azithromycin	2-Hydroxy-ibuprofen	Caffeine
Hydrochlorothiazide	Triclocarban	Dehydronifedipine	Naproxen	
Carbamazepine	Triclosan	Digoxigenin	Clarithromycin	
Miconazole	Ciprofloxacin	Enrofloxacin	Diltiazem	
Thiabendazole	Diphenhydramine	Lincomycin	Digoxin	
	Fluoxetine	Sulfamethazine	Erythromycin-H_2O	
	Norfloxacin		Sulfamethoxazole	
	Ofloxacin		Trimethoprim	
n=5	n=8	n=6	n=8	n=1

4.3.4.3 Fragrance and Alkylphenolic Compounds Mass Balances

The mass balances for the alkylphenolic and fragrance compounds for the Red Deer anaerobic digestion process are provided in **Table 22**. Only BPA was quantifiable in a mass balance in the anaerobic digester, with a calculated negative removal of -47%. Of the fragrance compounds, only AHDI was found to have a high removal efficiency of 89%. Poor or negative removal efficiencies were calculated for the remaining polycyclic musk compounds. As noted previously,

the wide range in solids concentrations in the digester feed and digested biosolids can affect the calculation of negative removal efficiencies.

Table 22. Mass Balance and Removal Calculations for Fragrance and Alkylphenolic Compounds in Mesophilic Anaerobic Digestion, Red Deer, AB

Fragrance	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Digester Feed Sludge	Digested Sludge	Digester Feed Sludge	Digested Sludge	
Alkylphenolics					
Bisphenol A	295	515	4844	7100	-47%
Fragrances					
DPMI	190	215	3120	2964	5%
AHDI	780	100	12809	1379	89%
HHCB	3615	8975	59366	123734	-108%
AHTN	2090	4015	34322	55353	-61%
ATII	335	520	5501	7169	-30%

4.3.4.4 Effectiveness of Process for ESOC Removal

The results indicate that the anaerobic digestion process, as represented by the Red Deer data, provides only a moderate barrier for reducing some concentrations of pharmaceutical compounds in feed sludge during biosolids treatment. Almost as many pharmaceutical compounds exited the digester with higher masses than in the feed sludge, as there were pharmaceutical compounds that exhibited reduced masses in the digested solids compared to their masses entering in the digester feed. The uncertainty of the solids balance caused by the variability of the solids concentrations of the feed sludge and digested biosolids may cause this assessment of the digestion process a conservative one,

4.3.5 Section Summary

The concentration of ammonia-N (a component of total Kjeldahl nitrogen, or TKN) increases through the digestion process as expected, due to breakdown of proteins in the feed biomass. The concentration of TKN appears to increase slightly through the digestion process. Concentrations of nitrite and nitrate ion are low in both the digester feed sludge and in the digester effluent. Feed concentrations of these ions are believed to be low due to the denitrification process used in the liquid treatment train at the Red Deer facility, a BNR plant. Nitrate and nitrite ion concentrations are low in the digested biosolids because there is no nitrate or nitrite formation during anaerobic digestion, which occurs in the absence of oxygen. Both total phosphorus and ortho-phosphate were observed at higher concentrations and masses in the digested biosolids compared to the feed sludge. Total phosphorus is a conservative mass, and should not change from influent to effluent. Possible reasons for the observed increase in total phosphorus through the digester include mobilization from accumulated reserves in the digestion tank, or sampling and analytical variability resulting from one set of grab samples.

Most metals, with the exceptions of arsenic, cobalt and selenium were identified above the detection limits in digester feed sludge, whereas only a few metals, i.e., chromium, copper, mercury and zinc, were detected in the anaerobically digested sludge. The highest and lowest concentrations of metals in the feed and digested sludges were associated with zinc and mercury, respectively. With the exception of mercury, concentrations of the metals detected in both the feed sludge and digested biosolids were similar in magnitude. The concentration of mercury in the digested biosolids sample was approximately an order of magnitude higher than in the combined feed sludge. For most metals, the calculated mass leaving the digester is larger than the mass in the feed sludge. Since the metals are conservative (with the possible exception of mercury), under steady-state conditions, the mass entering and the mass leaving should be equal. Potential reasons for the negative removal efficiencies were discussed in the body of the text.

The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the digested biosolids included the anti-microbials triclosan and triclocarban, the antibiotics ciprofloxacin and norfloxacin, the stimulants caffeine and diphenhydramine, the anti-fungal miconazole. There appears to be a small shift in the distribution of detectable concentrations in the digester feed to the process effluent. Most notably the number of compounds detected in all three campaigns declines from 22 in the digester feed to 15 in the process effluent, while the number of compounds never detected in the digester feed sludge (27) increases to 34 compounds in the anaerobic digester effluent. The highest removal efficiency in the process was associated with caffeine, at 94%, followed by Diltiazem at 86%. Both trimethoprim and sulfamethoxazole appear to be efficiently removed as well, with calculated minimum removal efficiencies of 79% and 77%, respectively. Conversely, compounds such as carbamazepine and gemfibrozil were associated with high negative removals of -219% and -168%, respectively, indicating their concentrations in digested biosolids were much higher than in the feed sludge. The number of pharmaceutical compounds with positive removal efficiencies (i.e., greater than 0) at 15 was approximately the same as the number of compounds with negative removal efficiencies (i.e., less than 0) at 13.

Bisphenol A was the only one of the three alkylphenolic compounds detected in the feed and digested biosolids samples. The concentration of BPA was higher in the digested biosolids than in the feed sludge. The polycyclic musk fragrances HHCB and AHTN were detected at the highest concentrations (i.e., greater than 1000 ng/g TS) in the digested biosolids. None of the nitro musk compounds were observed above the limit of quantification. Many compounds were observed at higher concentrations in the digested sludge than in the feed sludge. Only BPA was quantifiable in a mass balance in the anaerobic digester, with a calculated negative removal of -47%. Of the fragrance compounds, only AHDI was found to have a high removal efficiency of 89%. Poor or negative removal efficiencies were calculated for the remaining polycyclic musk compounds. As noted previously, the wide range in solids concentrations in the digester feed and digested biosolids can affect the calculation of negative removal efficiencies.

The results indicate that the anaerobic digestion process, as represented by the Red Deer data, provides only a moderate barrier for reducing some concentrations of pharmaceutical compounds in feed sludge during biosolids treatment. Almost as many pharmaceutical compounds exhibited higher mean concentrations in the treated biosolids as in the feed sludge. The uncertainty of the

solids balance caused by the variability of the solids concentrations of the feed sludge and digested biosolids may cause this assessment of the digestion process a conservative one,

4.4 Mesophilic Anaerobic Digestion, Saskatoon, SK

4.4.1 Site Description

The Saskatoon facility is a biological nutrient removal (BNR) plant with chlorine disinfection of the treated effluent prior to discharge to South Saskatchewan River. The design capacity of the existing treatment plant is 120,000 m^3/d, while the average daily flow is 90,000 m^3/d.

4.4.2 Biosolids Treatment Description

Excess wastewater sludge is treated by mesophilic anaerobic digestion. There are in total three (3) anaerobic digesters at the facility. Four types of raw sludge are pumped to the digesters, including unfermented primary sludge from primary settling basins, primary scum, fermented primary sludge from the primary sludge anaerobic fermenter, and thickened waste activated sludge (WAS) from dissolved air flotation (DAF). The total pumping rate of feed sludge (all sources) to the three mesophilic anaerobic digesters is 1000 m^3/d. The capacity of each digester is 7,000 m^3, resulting in a nominal hydraulic capacity of approximately 21 days, assuming ideal mixing conditions (no stagnant zone or short-circuiting). During this study, the solids concentration in the feed sludge to the mesophilic digesters ranged from 2.4% to 4.9%, while the mesophilic anaerobically digested sludge solids concentration ranged from 1.3% to 5.6%. After anaerobic digestion, the processed biosolids are sent to the biosolids operation facility for temporary storage and then wet injected to agricultural lands twice a year.

For this project assessment, the biosolids treatment process of interest was mesophilic anaerobic digestion process. The two sampling locations included combined feed sludge (i.e. fermenter sludge for nutrients and metals, and all sources for pharmaceuticals) and digested sludge. The treatment facility indicated there was no supernatant from the digesters. A process schematic of the Saskatoon biosolids treatment process is shown in **Figure 5**.

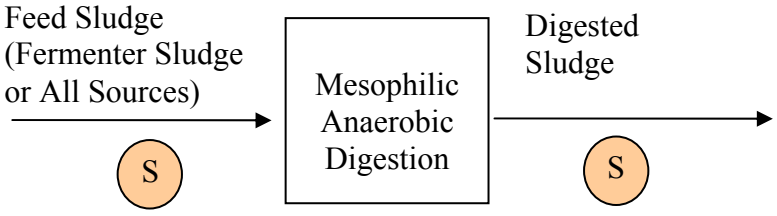

Figure 5. Schematic of Saskatoon Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on July 13, July 27 and August 11, 2009, respectively.

4.4.3 Sampling Results

4.4.3.1 Nutrients

Concentrations of nutrients in the combined digester feed sludge and digested biosolids are presented in **Table 23**. Although the concentration of total Kjeldahl nitrogen (TKN) decreased through the digestion process, the concentration of ammonia-N (a component of the TKN) increased as expected due to breakdown of proteins in the feed biomass. The organic-N component of TKN is thus significantly reduced because of anaerobic digestion. Concentrations of nitrite and nitrate ion are low in both the digester feed sludge and in the digester effluent (**Table 23**). Feed concentrations of these ions are believed to be low due to the denitrification process used in the liquid treatment train at the Saskatoon facility, a BNR plant. Nitrate and nitrite ion concentrations are low in the digested biosolids because there is no nitrate or nitrite formation during anaerobic digestion, which occurs in the absence of oxygen.

Although the concentration of soluble ortho-phosphate remained relatively constant through the digestion process, the total phosphorus (total P) concentration in the digested biosolids was lower than in the digester feed sludge. Total phosphorus is a conservative mass, and should not change from influent to effluent. Possible reasons for the observed reduction in total phosphorus through the digester include accumulation in the digestion tank, or sample variability resulting from one set of grab samples.

Table 23. Nutrient Concentrations in Combined Feed Sludges and Mesophilic Anaerobically Digested Sludge, Saskatoon, SK

Parameter	Concentration (mg/L)	
	Combined Sludges (Digester Feed)	Digested Sludge
Nitrate-N	<2.0	<2.0
Nitrite-N	<2.0	<2.0
Total Kjeldahl Nitrogen	2090	1880
Ammonia as N	120	497
Phosphorus, Total	706	434
Phosphate-P (ortho)	217	239
Total Solids	49000	56000

4.4.3.2 Metals

Most metals, with the exceptions of arsenic, cadmium and selenium, were identified above the detection limits in both the combined feed sludge and digested sludge samples, as shown in **Table 24**.

In general concentrations of the metals are approximately the same in the digester feed and digested biosolids samples. Zinc and copper were observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. Additional discussion of the metals is found later in this section under Data Interpretation.

Table 24.Metal Concentrations in Combined Feed Sludges and Mesophilic Anaerobically Digested Sludge, Saskatoon, SK

Parameter	Concentration (mg/L)	
	Combined Sludges (Digester Feed)	Digested Sludge
Arsenic (As)-Total	<0.10	<0.10
Cadmium (Cd)-Total	**0.018**	**0.018**
Chromium (Cr)-Total	**1.05**	**0.86**
Cobalt (Co)-Total	<0.080	<0.080
Copper (Cu)-Total	**4.59**	**4.74**
Lead (Pb)-Total	**0.62**	**0.52**
Mercury (Hg)-Total	**0.0119**	**0.0106**
Molybdenum (Mo)-Total	**0.13**	**0.15**
Nickel (Ni)-Total	**0.55**	**0.37**
Selenium (Se)-Total	<0.50	<0.50
Zinc (Zn)-Total	**8.96**	**9.40**
Total Solids	49000	56000

Data in **bold font** are above the detection limit

4.4.3.3 Pharmaceuticals

The frequency of detection and median and range of detected concentrations of the pharmaceutical compounds in the digester feed sludge and digested sludge at the Saskatoon facility are presented in **Table 25**. A total of 18 pharmaceuticals were detected in the digester feed sludge from all the three sampling campaigns; and 14 pharmaceuticals were detected in the digested sludge from all the three campaigns. The raw analytical data are provided in **Appendix Table A5**.

Table 25. Frequency of Detection, Median and Range of Detected Concentrations of Pharmaceutical Compounds in Combined Feed Sludge and Digested Biosolids from Saskatoon, SK

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Furosemide	0	0	NA	NA	<326 [b]	<617 [b]
Gemfibrozil	**3**	**3**	**50.4**	**75.2**	34.3-52	55.7-110
Glipizide	0	0	NA	NA	<48.9 [b]	<92.5 [b]
Glyburide	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Hydrochlorothiazide	1	1	234 [a]	143 [a]	<82.3-234	<68.9-143
2-Hydroxy-ibuprofen	1	2	535 [a]	496.5	<329-535	<276-561
Ibuprofen	**3**	**3**	**323**	**561**	308-362	365-1160
Naproxen	**3**	1	**155**	56.5 [a]	97.5-165	<10.3-56.5
(continued)						

Table 25 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Triclocarban	**3**	**3**	**1680**	**1930**	1550-1760	1850-3130
Triclosan	**3**	**3**	**4090**	**6050**	3540-4150	5590-6270
Warfarin	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Acetaminophen	1	0	1110 [a]	NA	<247-1110	<925 [b]
Azithromycin	**3**	**3**	**399**	**426**	164-621	330-480
Caffeine	3	1	1740	136 [a]	1670-2130	<71.1-136
Carbadox	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Carbamazepine	**3**	**3**	**64.7**	**131**	48.1-79.2	115-133
Cefotaxime	0	0	NA	NA	<162 [b]	<295 [b]
Ciprofloxacin	**3**	**3**	**3570**	**3610**	3390-3580	3100-6900
Clarithromycin	**3**	**3**	**71**	**38.6**	23.4-141	31.2-84.3
Clinafloxacin	0	0	NA	NA	<48.9 [b]	<92.5 [b]
Cloxacillin	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Dehydronifedipine	2	0	3.685	NA	<2.47-3.92	<9.25 [b]
Diphenhydramine	**3**	**3**	**1180**	**994**	1050-1310	984-2290
Diltiazem	**3**	**3**	**186**	**18.7**	61-201	11.5-29.9
Digoxin	0	1	NA	287 [a]	<327 [b]	<76.1-287
Digoxigenin	0	1	NA	63.1 [a]	<96.4 [b]	<35.9-63.1
Enrofloxacin	1	1	14.1 [a]	21.1 [a]	<12.3-14.1	<10.3-21.1
Erythromycin-H_2O	**3**	**3**	**58.7**	**33.1**	46.1-62.2	13.7-312
Flumequine	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Fluoxetine	**3**	**3**	**109**	**62.6**	49.6-126	55.8-130
Lincomycin	0	0	NA	NA	<37.9 [b]	<46.2 [b]
Lomefloxacin	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Miconazole	**3**	**3**	**259**	**488**	226-418	375-517
Norfloxacin	1	1	312 [a]	87.1 [a]	<61.7-312	<51.7-87.1
Norgestimate	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Ofloxacin	1	**3**	108 [a]	**109**	<61.7-108	90-232
Ormetoprim	0	0	NA	NA	<4.89 [b]	<9.25 [b]
Oxacillin	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Oxolinic Acid	0	0	NA	NA	<4.89 [b]	<9.25 [b]
Penicillin G	0	0	NA	NA	<81.6 [b]	<154 [b]
Penicillin V	0	0	NA	NA	<24.5 [b]	<46.2 [b]
Roxithromycin	0	0	NA	NA	<2.87 [b]	<4.62 [b]
Sarafloxacin	0	0	NA	NA	<144 [b]	<231 [b]
Sulfachloropyridazine	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Sulfadiazine	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Sulfadimethoxine	0	0	NA	NA	<2.45 [b]	<4.62 [b]
(continued)						

Table 25 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids
Sulfamerazine	1	1	7.21 [a]	8.03 [a]	<3.84-7.21	<3.05-8.03
Sulfamethazine	0	0	NA	NA	<4.89 [b]	<9.25 [b]
Sulfamethizole	0	0	NA	NA	<4.89 [b]	<9.25 [b]
Sulfamethoxazole	**3**	0	**33.3**	<3.05	10.3-35.8	<9.25 [b]
Sulfanilamide	0	0	NA	NA	<122 [b]	<231 [b]
Sulfathiazole	0	0	NA	NA	<12.2 [b]	<23.1 [b]
Thiabendazole	**3**	2	**13.4**	17.85	12.4-19.2	<5.17-18.8
Trimethoprim	**3**	1	**144**	12.5 [a]	74-147	<5.17-12.5
Tylosin	0	0	NA	NA	<128 [b]	<102 [b]
Virginiamycin	0	0	NA	NA	<202 [b]	<276 [b]
1,7-Dimethylxanthine	0	0	NA	NA	<1220 [b]	<2310 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the digested biosolids included the anti-microbials triclosan and triclocarban, and the antibiotic ciprofloxacin. The stimulant diphenhydramine was just below the 1000 ng/g TS concentration.

The distribution of detectable concentrations in the combined digester feed sludge and digested biosolids from the three sampling campaigns is found in **Table 26**. There appears to be a minor shift in the distribution of detectable concentrations in the digester feed sludge to digested sludge. The number of compounds detected in all three campaigns declines from 18 in the digester feed sludge to 14 in digested sludge, while the number of compounds detected in either two, one or none of the three campaigns increments by one or two compounds in each category.

Table 26. Summary of Pharmaceutical Compound Detections in Digester Feed Sludge (All Sources) and Digested Sludge from Saskatoon, SK

Frequency of detection in sampling campaigns (out of 3)	Number of Compounds in Process Streams	
	Combined Feed Sludge	Digested Sludge
3	18	14
2	1	2
1	7	9
0	31	32
Total	57	57

4.4.3.4 Fragrances and Alkylphenolics

Concentrations of fragrances and alkylphenolics are provided in **Table 27**. The raw analytical data are provided in **Appendix Table A6**. The compounds observed at the highest concentrations (e.g. greater than 1,000 ng/g TS) in both the digester feed sludge and digested biosolids were Bisphenol A and the synthetic polycyclic musks HHCB and AHTN. With the exception of musk xylene observed at the detection limit in one of the two digester feed sludge samples, no nitro musks were detected in either the feed sludge or anaerobically digested biosolids. Additional discussion of the fragrance and alkylphenolics is found later in this section under Data Interpretation.

Table 27. Frequency of Detection, Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Combined Feed Sludge and Digested Biosolids from Saskatoon, SK

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Digester Feed Sludge	Digested Biosolids	Digester Feed Sludge	Digested Biosolids	Digester Feed Sludge	Digested Biosolids
Alkylphenolics						
Bisphenol A	**2**	**2**	**6495**	**1765**	790-12200	970-2560
Octylphenol	1	**2**	20 [a]	**50**	<20-20	40-60
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	**2**	**2**	**245**	**460**	70-420	150-770
ADBI	0	0	NA	NA	<20 [b]	<20 [b]
AHDI	**2**	**2**	**230**	**325**	230-230	150-500
HHCB	**2**	**2**	**3205**	**5130**	2250-4160	4790-5470
AHTN	**2**	**2**	**1365**	**2225**	1100-1630	2190-2260
ATII	**2**	**2**	**305**	**605**	300-310	520-690
Musk Moskene	0	0	NA	NA	<50 [b]	<50 [b]
Musk Tibetene	0	0	NA	NA	<80 [b]	<80 [b]
Musk Ketone	0	0	NA	NA	<120 [b]	<120 [b]
Musk Ambrette	0	0	NA	NA	<140 [b]	<140 [b]
Musk Xylene	1	0	70 [a]	NA	<70-70	<70 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in both sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

4.4.4 Data Interpretation

4.4.4.1 Metals Mass Balances

Mass balances for metals around the anaerobic digestion process were determined by multiplying the volumetric throughput rate of feed sludge and anaerobically digested biosolids (350 m^3/d) by the liquid-based concentrations of the metals (in units of mg/L). Only mass calculations for the

detected metals are presented in **Table 28**.

From the table, it appears the anaerobic digestion process is at or near steady-state, with the mass of each metal entering the digester close in value to the mass exiting the digester. The mass closures range from 67% for nickel to 115% for molybdenum. The median mass closure value is 95%. The fact that nearly as many metals exhibit positive removal efficiencies as negative removals suggests that the mass balances are unbiased to either the feed sludge or digested biosolids, with the differences in the input and output masses likely only dependent on analytical variability.

Table 28. Mass Balance Closure Calculations for Metals in Mesophilic Anaerobic Digestion Process, Saskatoon, SK

Metal	Concentration (mg/L)		Mass of Contaminant (g/d)		Mass Closure (%)
	Raw sludge	Treated biosolids	Raw sludge	Treated biosolids	
Cadmium (Cd)-Total	0.018	0.018	18	18	100%
Chromium (Cr)-Total	1.05	0.86	1050	860	82%
Copper (Cu)-Total	4.59	4.74	4590	4740	103%
Lead (Pb)-Total	0.62	0.52	620	520	84%
Mercury (Hg)-Total	0.0119	0.0106	12	10.6	89%
Molybdenum (Mo)-Total	0.13	0.15	130	150	115%
Nickel (Ni)-Total	0.55	0.37	550	370	67%
Zinc (Zn)-Total	8.96	9.40	8960	9400	105%

4.4.4.2 Pharmaceutical Compounds Mass Balances

Concentrations for the pharmaceutical, fragrance and alkylphenolic compounds are expressed on a dry weight basis, and so the mass balances for the pharmaceutical compounds are based on a total solids balance around the anaerobic digestion process. The solids balance around the anaerobic digester is estimated using the mean values of the total solids concentrations in the combined sludge feed and digested biosolids out of the process from the three sampling campaigns. The pertinent solids concentration and flow data are:

Anaerobic digester volumetric throughput = 1,000 m^3/d
Mean measured total solids concentration in feed sludge = 34.7 kg/m^3
Mean measured total solids concentration in digested biosolids = 36.3 kg/m^3

In the balance, it was assumed the difference in the total mass of solids entering and leaving the digester was the mass of volatile solids lost through the process. The total solids balance is depicted in
Figure 6. The calculated higher mass of solids leaving the digester than the mass entering the digester is considered an artifact of the variability associated with sampling sludge and biosolids.

Concentrations of the pharmaceutical compounds measured on a dry weight basis (i.e. ng/g TS dw) were converted to a mass flow rate (mg/d) for comparison of input and output masses. The results of the mass estimates are provided in **Table 29**. Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids were not included in **Table 29**.

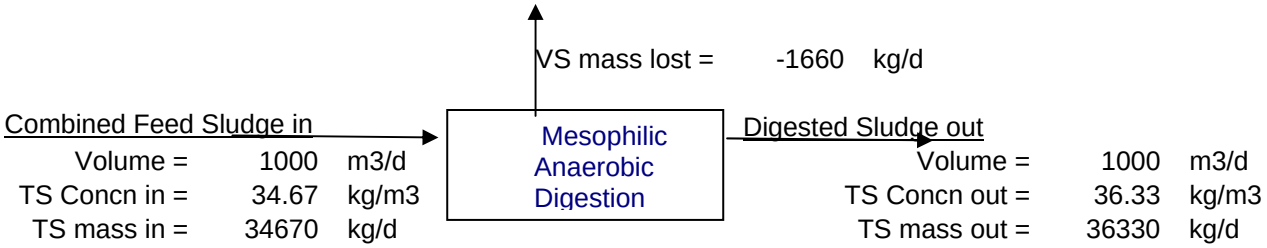

Figure 6. Solids Mass Balance around Anaerobic Digestion Process, Saskatoon, SK

Table 29. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Mesophilic Anaerobic Digestion Process, Saskatoon, SK

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Combined feed sludge	Digested biosolids	Combined feed sludge	Digested biosolids	
Gemfibrozil	50.4	75.2	1747	2732	-56%
Hydrochlorothiazide	234	143	8113	5195	36%
2-Hydroxy-ibuprofen	535	496.5	18548	18038	3%
Ibuprofen	323	561	11198	20381	-82%
Naproxen	155	56.5	5374	2053	62%
Triclocarban	1680	1930	58246	70117	-20%
Triclosan	4090	6050	141800	219797	-55%
Acetaminophen	1110	<475	38484	<17257	>55.2%
Azithromycin	399	426	13833	15477	-12%
Caffeine	1740	136	60326	4941	92%
Carbamazepine	64.7	131	2243	4759	-112%
Ciprofloxacin	3570	3610	123772	131151	-6%
Clarithromycin	71	38.6	2462	1402	43%
Dehydronifedipine	3.685	<3.05	128	<111	>13.3%
Diphenhydramine	1180	994	40911	36112	12%
Diltiazem	186	18.7	6449	679	89%
Enrofloxacin	14.1	21.1	489	767	-57%
Erythromycin-H_2O	58.7	33.1	2035	1203	41%
Fluoxetine	109	62.6	3779	2274	40%
Miconazole	259	488	8980	17729	-97%
Norfloxacin	312	87.1	10817	3164	71%
Ofloxacin	108	109	3744	3960	-6%
Sulfamerazine	7.21	8.03	250	292	-17%
Sulfamethoxazole	33.3	<3.05	1155	<111	>90.4%
Thiabendazole	13.4	17.85	465	648	-40%
Trimethoprim	144	12.5	4992	454	91%

Caffeine, trimethoprim, sulfamethoxazole were calculated to have the highest removal efficiencies (greater than 90%), while diltiazem was similarly removed efficiently at 89%. The

highest negative removal efficiencies calculated were for carbamazepine (-112%), miconazole (-97%) and ibuprofen (-82%). The negative removal efficiencies indicate that a substantially higher mass of the compounds is leaving the digester than was present in the combined digester feed sludges.

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 30.** The number of pharmaceutical compounds with positive removal efficiencies (i.e., greater than 0) at 14 was approximately the same as the number of compounds with negative removal efficiencies (i.e., less than 0) at 12.

Table 30. Categorised Removal Efficiencies of Pharmaceutical Compounds by Mesophilic Anaerobic Digestion, Saskatoon, SK

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Gemfibrozil	Triclocarban	Hydrochlorothiazide	Naproxen	Caffeine
Ibuprofen	Azithromycin	2-Hydroxy-ibuprofen	Acetaminophen	Sulfamethoxazole
Triclosan	Ciprofloxacin	Clarithromycin	Diltiazem	Trimethoprim
Carbamazepine	Ofloxacin	Dehydronifedipine	Norfloxacin	
Enrofloxacin	Sulfamerazine	Diphenhydramine		
Miconazole	Thiabendazole	Erythromycin-H_2O		
		Fluoxetine		
n=6	n=6	n=7	n=4	n=3

4.4.4.3 Fragrance and Alkylphenolic Compounds Mass Balances

Mass balance results for the fragrances and alkylphenolics are presented in **Table 31**. Only compounds with detectable concentrations in the feed sludge are listed in the table. Bisphenol A was removed at 78% efficiency through the mesophilic digester, while the removal efficiency estimated for musk xylene based on the detection limit was >19%. All of the other compounds were calculated to have negative removal efficiencies, indicating no biodegradation of these compounds in mesophilic digestion.

4.4.4.4 Effectiveness of Process for ESOC Removal

The results indicate that the anaerobic digestion process, as represented by the Saskatoon data, provides only a moderate barrier for reducing some concentrations of pharmaceutical compounds in feed sludge during biosolids treatment. Almost as many pharmaceutical compounds were associated with negative removal efficiencies through the digestion process as there were compounds with positive removal efficiencies. All polycyclic fragrance compounds were observed to have negative removal efficiencies. Bisphenol A was removed to a great extent (78%). The uncertainty of the solids balance caused by the variability of the solids concentrations of the feed sludge and digested biosolids may cause this assessment of the digestion process to be a conservative one.

Table 31. Mass Balance and Removal Calculations for Fragrance and Alkylphenolic Compounds in Mesophilic Anaerobic Digestion Process, Saskatoon, SK

Fragrance and Phenolic Compounds	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Digester Feed Sludge	Digested Sludge	Digester Feed Sludge	Digested Sludge	
Alkylphenolics					
Bisphenol A	6495	1765	140292	30888	78%
Octylphenol	20	50	432	875	-103%
Fragrances					
DPMI	245	460	5292	8050	-52%
AHDI	230	325	4968	5688	-14%
HHCB	3205	5130	69228	89775	-30%
AHTN	1365	2225	29484	38938	-32%
ATII	305	605	6588	10588	-61%
Musk Xylene	70	<70	1512	<1225	>19%

4.4.5 Section Summary

Although the concentration of total Kjeldahl nitrogen (TKN) decreased through the digestion process, the concentration of ammonia-N (a component of the TKN) increased as expected due to breakdown of proteins in the feed biomass. Concentrations of nitrite and nitrate ion are low in both the digester feed sludge and in the digester effluent. Although the concentration of soluble ortho-phosphate remained relatively constant through the digestion process, the total phosphorus concentration in the digested biosolids was lower than in the digester feed sludge. Possible reasons include accumulation of total P in the digestion tank, or sample variability resulting from one set of grab samples.

Most metals, with the exceptions of arsenic, cadmium and selenium, were identified above the detection limits in both the combined feed sludge and digested sludge samples. In general concentrations of the metals are approximately the same in the digester feed and digested biosolids samples. Zinc and copper were observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. The mass of each metal entering the digester was close in value to the mass exiting the digester. Almost as many metals exhibit positive removal efficiencies as negative removals through the digester, suggesting no net loss or gain through the digestion process.

The pharmaceutical compounds detected at the highest concentrations (above 1,000 ng/g TS) in the digested biosolids included the anti-microbials triclosan and triclocarban, and the antibiotic ciprofloxacin. The stimulant diphenhydramine was just below the 1000 ng/g TS concentration. The number of compounds detected in all three campaigns declines from 18 in the digester feed sludge to 14 in digested sludge, while the number of compounds detected in either two, one or none of the three campaigns increments by one or two compounds in each category. Caffeine, trimethoprim, sulfamethoxazole were calculated to have the highest removal efficiencies (greater than 90%) through the digestion process, while diltiazem was similarly removed efficiently at 89%. The highest negative removal efficiencies calculated were for carbamazepine (-112%), miconazole (-97%) and ibuprofen (-82%). The negative removal

efficiencies indicate that a substantially higher mass of the compounds is leaving the digester than was present in the combined digester feed sludges. The number of pharmaceutical compounds with positive removal efficiencies (i.e., greater than 0) at 14 was approximately the same as the number of compounds with negative removal efficiencies (i.e., less than 0) at 12.

The compounds observed at the highest concentrations (e.g. greater than 1,000 ng/g TS) in both the digester feed sludge and digested biosolids were Bisphenol A and the synthetic polycyclic musks HHCB and AHTN. With the exception of musk xylene observed at the detection limit in one of the two digester feed sludge samples, no nitro musks were detected in either the feed sludge or anaerobically digested biosolids. Bisphenol A was removed at 78% efficiency through the mesophilic digester, while the removal efficiency estimated for musk xylene based on the detection limit was >19%. All of the other compounds were calculated to have negative removal efficiencies, indicating no biodegradation of these compounds in mesophilic digestion.

The results indicate that the mesophilic anaerobic digestion process, as represented by the Saskatoon data, provides only a moderate barrier for reducing some concentrations of pharmaceutical, fragrance and alkylphenolic compounds in feed sludge during biosolids treatment. Almost as many of the pharmaceutical compounds were associated with negative removal efficiencies through the digestion process as there were compounds with positive removal efficiencies. All of the synthetic polycyclic musks and octylphenol had negative removal efficiencies. Only Bisphenol A and Musk Xylene were associated with positive removal efficiencies in the anaerobic digester. The uncertainty of the solids balance caused by the variability of the solids concentrations of the feed sludge and digested biosolids may cause this assessment of the digestion process a conservative one,

4.5 Composting, Prince Albert, SK

4.5.1 Site Description

The Prince Albert facility (J.W. Oliver Pollution Control Centre) is a conventional activated sludge plant with chlorination disinfection prior to discharge to North Saskatchewan River. The design capacity of the existing treatment plant is 44,415 m^3/d, while the average daily dry weather flow is 13,000 m^3/d.

4.5.2 Biosolids Treatment Description

The waste activated sludge (WAS) from the secondary clarifiers is thickened using a dissolved air flotation (DAF) and combined with primary sludge from the primary clarifiers and is then passed through a belt filter press for dewatering. The dewatered cake is hauled at a rate of 17 m^3/d to the composting facility for final treatment. The compost bulking agents such as wood chips and wood shaving are added to the dewatered cake for composting. The following is the recipe for the composting feed sludge:

- 10 parts dewatered cake
- 12 parts wood block (wood chunk)
- 8 parts fibre (wood shaving)
- 4 parts compost product (for seeding).

During this study, the solids concentration in the feed sludge (i.e. dewatered cake) to the compost bunkers ranged from 18.3% to 23%, while the compost solids concentration ranged from 43.6% to 67.4%. The compost is applied to agricultural lands.

For this project assessment, the biosolids treatment process of interest was the biosolids composting process. Three sampling locations included the feed sludge (i.e. dewatered cake) to the composting facility (i.e. dewatered cake before wood chips or wood shaving addition), compost and leachate from composting process. For the finished compost samples, as much of the fibrous wood and yard waste material as possible was removed prior to the laboratory analyses. A process schematic of the Prince Albert biosolids treatment process is shown in **Figure 7**.

The plant was considered by plant staff to be in normal operation during the two sampling campaigns. Samples were collected and shipped to the analytical laboratories on September 29 and October 15, 2009, respectively.

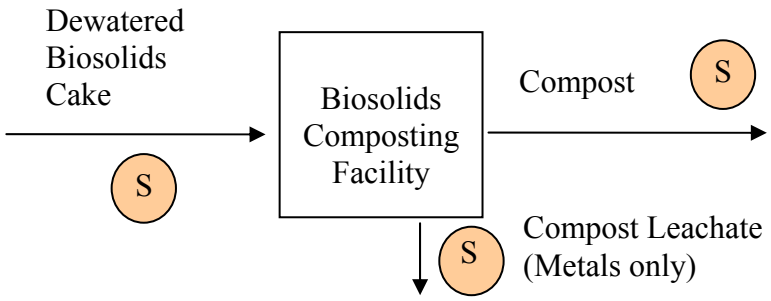

Figure 7. Schematic of Prince Albert Biosolids Process and Sampling Locations

4.5.3 Sampling Results

4.5.3.1 Nutrients

Based on the one set of nutrient data from grab samples, interpretation of the data is limited. The nutrient data as reported by the laboratory could not be directly compared because of the different concentration units used, for liquid samples (leachate) and solids samples (dewatered cake feed and compost). The concentrations of nitrate-N and of total Kjeldahl nitrogen (TKN, a measure of combined ammonia-N and organic-N) were lower in the compost than in the dewatered cake (**Table 32**). Because the concentration of ammonia-N (a component of TKN) was relatively constant, the lower concentration of TKN in the compost can be likely attributed to a loss of organic-N, either through reduction of the volatile solids during composting, or due to loss from leachate generated during the process. The observed differences may also be due to variations in the composition of the two process streams at the time of sampling. Concentrations of total phosphorus and especially ortho-phosphate were observed at lower concentrations in the compost

product than in the dewatered cake. The observed differences in concentrations of these compounds may again be due to loss in leachate from the composting pad.

Table 32. Nutrients in Dewatered Biosolids Cake and Compost from Prince Albert, SK

Parameter	Concentration		
	Dewatered Cake (mg/kg TS)	Compost (mg/kg TS)	Leachate (mg/L)
Nitrate-N	11.3	5.4	<2.0
Nitrite-N	<1.0	<1.0	<2.0
Total Kjeldahl Nitrogen	44000	23500	1360
Ammonia as N	7320	7430	1170
Phosphorus, Total	14300	11000	228
Phosphate-P (ortho)	6730	684	190
Total Solids	19.2%	61.5%	3000[a]

[a] Total suspended solids

4.5.3.2 Metals

The metals data for the different matrices as reported by the laboratory could not be directly compared because of the different concentration units used, for liquid samples (leachate) and solids samples (dewatered cake feed and compost). Most metals except cadmium were identified above the detection limits in dewatered cake and compost samples, as shown in **Table 33**. Arsenic, cobalt and selenium were not detected in the dewatered cake, but were above the detection limit in the compost. Only copper, mercury and zinc were detected in the leachate.

Table 33.Metals in Dewatered Biosolids Cake and Compost from Prince Albert, SK

Metal	Concentration		
	Dewatered Cake (mg/kg TS dw)	Compost (mg/kg TS dw)	Leachate (mg/L)
Arsenic (As)-Total	<1.0	**2.6**	<0.10
Cadmium (Cd)-Total	<1.0	<1.0	<0.010
Chromium (Cr)-Total	**3.9**	**18.1**	<0.10
Cobalt (Co)-Total	<1.0	**2.9**	<0.080
Copper (Cu)-Total	**82.1**	**275**	**0.15**
Lead (Pb)-Total	**5.8**	**20.7**	<0.10
Mercury (Hg)-Total	**1.10**	**1.14**	**0.00022**
Molybdenum (Mo)-Total	**1.6**	**3.8**	<0.10
Nickel (Ni)-Total	**3.2**	**11.0**	<0.20
Selenium (Se)-Total	<1.0	**2.1**	<0.50
Zinc (Zn)-Total	**84.2**	**299**	**0.41**
Total Solids	19.2%	61.5%	0.3%

Detected concentrations in Bold font

4.5.3.3 Pharmaceuticals

Due to site-specific constraints, there are a total of two instead of three sampling campaigns for the Prince Albert facility. The frequency of detection and median and range of detected

concentrations of the pharmaceutical compounds in the dewatered cake and compost samples at the Prince Albert facility are presented in **Table 34**. The unprocessed concentration data and detection limits in the two sampling campaigns are provided in **Appendix Table A7**.

Table 34. Frequency of Detection, Median and Range of Detected Concentrations of Pharmaceutical Compounds in Dewatered Biosolids Cake and Compost from Prince Albert, SK

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Dewatered Cake	Compost	Dewatered Cake	Compost	Dewatered Cake	Compost
Furosemide	1	1	167 [a]	817 [a]	<103-167	<153-817
Gemfibrozil	**2**	**2**	**72.0**	**54.5**	63.9-80	12-97
Glipizide	0	1	NA	11.4 [a]	<11.8 [b]	<9.9-11.4
Glyburide	0	1	NA	6.63 [a]	<5.92 [b]	<4.95-6.63
Hydrochlorothiazide	0	0	NA	NA	<39.5 [b]	<36.9 [b]
2-Hydroxy-ibuprofen	0	0	NA	NA	<158 [b]	<148 [b]
Ibuprofen	**2**	1	**217**	310 [a]	179-254	<24.8-310
Naproxen	**2**	**2**	**100**	**2600**	94.5-106	1030-4170
Triclocarban	**2**	**2**	**1880**	**1635**	1490-2270	1610-1660
Triclosan	**2**	**2**	**7300**	**3950**	6300-8300	2320-5580
Warfarin	0	0	NA	NA	<2.96 [b]	<2.77 [b]
Acetaminophen	0	0	NA	NA	<118 [b]	<110 [b]
Azithromycin	**2**	**2**	**1570**	**190.2**	1460-1680	24.4-356
Caffeine	**2**	**2**	**646**	**1033**	446-846	596-1470
Carbadox	0	0	NA	NA	<2.96 [b]	<2.75 [b]
Carbamazepine	**2**	**2**	**63.1**	**53.7**	58.2-67.9	43.6-63.7
Cefotaxime	0	0	NA	NA	<63.3 [b]	<116 [b]
Ciprofloxacin	**2**	**2**	**6595**	**2020**	5990-7200	860-3180
Clarithromycin	**2**	**2**	**41.2**	**13.2**	39.7-42.6	4.5-22
Clinafloxacin	0	0	NA	NA	<20 [b]	<238 [b]
Cloxacillin	0	0	NA	NA	<3.92 [b]	<6 [b]
Dehydronifedipine	**2**	0	**3.9**	NA	3.51-4.19	<1.53 [b]
Diphenhydramine	**2**	**2**	**2735**	**1220**	2610-2860	680-1760
Diltiazem	**2**	1	**215**	24.3 [a]	175-254	<0.5-24.3
Digoxin	0	0	NA	NA	<29.6 [b]	<27.5 [b]
Digoxigenin	0	0	NA	NA	<41.4 [b]	<59.3 [b]
Enrofloxacin	0	0	NA	NA	<11.9 [b]	<64.3 [b]
Erythromycin-H_2O	**2**	**2**	**51.3**	**12.5**	46.6-55.9	7.1-18

(continued)

Table 34 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns	Median of Detected Concentrations (ng/g TS dw)	Range of Detected Concentrations (ng/g TS dw)

	(out of 2)					
	Dewatered Cake	**Compost**	**Dewatered Cake**	**Compost**	**Dewatered Cake**	**Compost**
Flumequine	0	0	NA	NA	<2.96 [b]	<2.75 [b]
Fluoxetine	**2**	**2**	**83.0**	**55.8**	65.9-100	38.3-73.3
Lincomycin	0	0	NA	NA	<13.8 [b]	<12.8 [b]
Lomefloxacin	0	0	NA	NA	<5.92 [b]	<21.6 [b]
Miconazole	**2**	**2**	**442**	**267.5**	293-591	205-330
Norfloxacin	0	0	NA	NA	<29.6 [b]	<62.8 [b]
Norgestimate	0	0	NA	NA	<8.8 [b]	<9.84 [b]
Ofloxacin	**2**	1	**115**	53.1 [a]	92.9-137	<24.8-53.1
Ormetoprim	0	0	NA	NA	<1.2 [b]	<1.1 [b]
Oxacillin	0	0	NA	NA	<5.9 [b]	<5.5 [b]
Oxolinic Acid	1	0	1.35 [a]	<2.75	<1.18-1.35	<3.1 [b]
Penicillin G	0	0	NA	NA	<2.4 [b]	<2.2 [b]
Penicillin V	0	0	NA	NA	<5.9 [b]	<6.2 [b]
Roxithromycin	0	0	NA	NA	<2.3 [b]	<1.5 [b]
Sarafloxacin	0	0	NA	NA	<164 [b]	<498 [b]
Sulfachloropyridazine	0	0	NA	NA	<3.0 [b]	<2.8 [b]
Sulfadiazine	0	0	NA	NA	<3.0 [b]	<2.8 [b]
Sulfadimethoxine	0	0	NA	NA	<5.9 [b]	<1.8 [b]
Sulfamerazine	0	0	NA	NA	<2.6 [b]	<1.7 [b]
Sulfamethazine	1	0	7[a]	NA	<2.6-7	<5.0 [b]
Sulfamethizole	0	0	NA	NA	<1.5 [b]	<1.6 [b]
Sulfamethoxazole	**2**	0	**28.9**	NA	23.5-34.2	<1.1
Sulfanilamide	0	0	NA	NA	<29.6 [b]	<27.5 [b]
Sulfathiazole	1	0	2.93 [a]	NA	<2.8-2.9	<2.75 [b]
Thiabendazole	**2**	**2**	**22.5**	**11.38**	21.4-23.6	7.96-14.8
Trimethoprim	**2**	1	**267**	85.9 [a]	261-273	<2.8-85.9
Tylosin	0	0	<25.3	<43.85	<39.5 [b]	<77.8 [b]
Virginiamycin	0	0	<103	<35.7	<201 [b]	<54.9 [b]
1,7-Dimethylxanthine	0	0	<286	<261.5	<296 [b]	<275 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The antimicrobial triclosan was found at the highest concentrations in both the dewatered cake and biosolids, at levels of 7,300 and 3,950 ng/g TS, respectively. Other compounds detected in the finished compost with median concentrations greater than 1,000 ng/g TS were triclocarban (antimicrobial), the antibiotics azithromycin and ciprofloxacin, the non-steroidal anti-inflammatory naproxen and the stimulants diphenhydramine and caffeine.
The distribution of detectable concentrations in the dewatered biosolids cake and compost from the two sampling campaigns is found in **Table 35**. There appears to be a slight shift in the distribution of detectable concentrations in the dewatered cake and compost. The number of compounds detected in all two campaigns declines from 20 in the dewatered cake to 14 in

compost samples. The number of compounds detected in only one of the two campaigns rises from 4 in the dewatered cake to 7 in the compost, while the number of compounds detected in neither of the two campaigns also rises from 33 in the dewatered cake to 36 in the final compost.

Table 35. Summary of Pharmaceutical Compound Detections in Dewatered Biosolids Cake and Compost from Prince Albert, SK

Frequency of detection in sampling campaigns (out of 2)	Number of Compounds in Process Streams	
	Dewatered Biosolids Cake	Compost
2	20	14
1	4	7
0	33	36
Total	57	57

4.5.3.4 Fragrance and Alkylphenolic Compounds

The alkylphenolic compound and fragrance compound data are summarized in **Table 36** for the two sampling rounds for these compounds. The raw analytical data are provided in **Appendix Table A8**. Bisphenol A was the only alkylphenolic compound detected; it was present in both the feed dewatered cake and compost samples at approximately the same concentrations. The polycyclic musk HHCB was found at the highest concentration, followed by AHTN at substantially lower concentrations. Nitro musk compounds were all below the limit of quantitation.

4.5.4 Data Interpretation

4.5.4.1 Metals Mass Balances

Concentrations of the metal contaminants in the dewatered cake and final compost were expressed on a dry weight basis (i.e. mg/kg TS), while concentrations in the leachate were expressed in volumetric units (i.e. mg/L). A solids balance around the composting process (**Figure 8**) was developed using the following information:
Dewatered cake feed volumetric rate = 17 m^3/d (1 truckload)
Leachate production rate = 2000 US gal/3 day period = 2.52 m^3/d
Measured total solids concentration in dewatered cake = 19.2%
Measured total solids concentration in compost = 61.5%
Measured total solids concentration in leachate = 0.3%.

Table 36. Frequency of Detection, Median and Range of Detected Concentrations of Alkylphenolic and Fragrance Compound in Dewatered Biosolids Cake and Compost from Prince Albert, SK

Fragrance and Phenolic			
	Frequency of Detection in Sampling	Median of Detected Concentrations	Range of Detected Concentrations

Compounds	Campaigns (out of 2)		(ng/g TS dw)		(ng/g TS dw)	
	Dewatered Cake	Compost	Dewatered Cake	Compost	Dewatered Cake	Compost
Alkylphenolics						
Bisphenol A	**2**	**2**	**105**	**115**	90-120	100-130
Octylphenol	0	0	NA	NA	<20	<20
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	1	1	70	40	<40-70	<40-40
ADBI	0	0	NA	NA	<20	<20
AHDI	0	0	NA	NA	<30	<30
HHCB	**2**	**2**	**1555**	**3470**	1240-1870	3020-3920
AHTN	**2**	**2**	**580**	**545**	470-690	360-730
ATII	**2**	**2**	**85**	**110**	70-100	90-130
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	<120	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	0	0	NA	NA	<70	<70

Data in **bold font** are detected in both sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

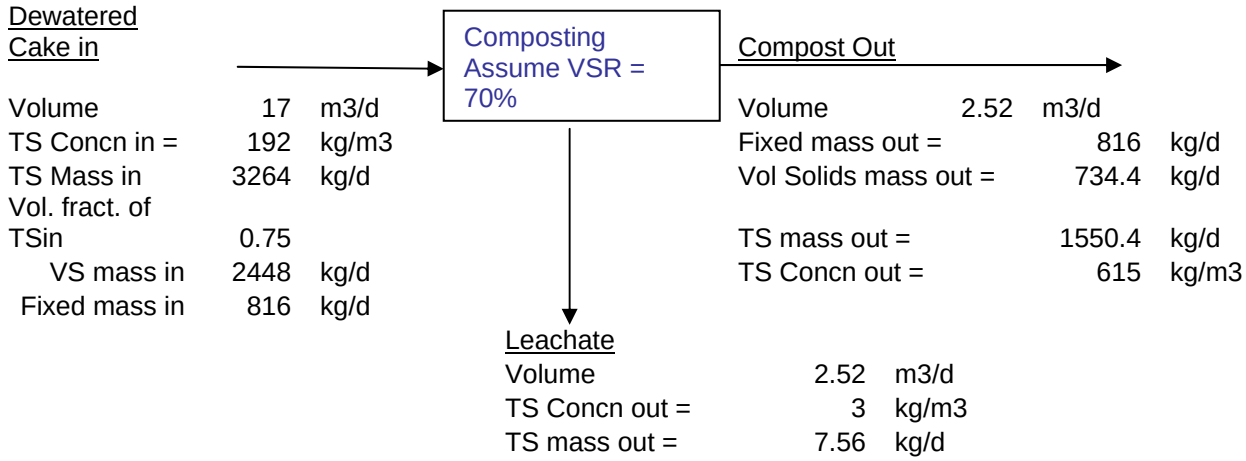

Figure 8. Solids Balance at Prince Albert, SK, Composting Process

For the balance, it was assumed that the volatile solids fraction of the dewatered cake was 0.75, and the volatile solids reduction achieved was 70%, based on professional judgement. It was further assumed that the mass of fixed solids in the dewatered cake was conserved in the compost following removal of the wood-based bulking agents.

The mass balance of metals in the composting process, using the solids balance created above, is presented in **Table 37**.

Table 37. Mass Balance of Metals through Composting Process, Prince Albert, SK

Metal	Mass (mg/d)				Max Total Out/Cake	Compost/ Cake
	Dewatered Cake	Compost	Leachate	Range Total Out[a]		
Chromium (Cr)-Total	12,730	28,062	≤252	28,062-28,314	2.20-2.22	2.20
Copper (Cu)-Total	267,974	426,360	378	426,738	1.59	1.59
Lead (Pb)-Total	18,931	32,093	≤252	32,093-32,345	1.70-1.71	1.70
Mercury (Hg)-Total	3,590	1,767	0.6	1,768	0.49	0.49
Molybdenum (Mo)-Total	5,222	5,892	≤252	5,892-6,144	1.13-1.18	1.13
Nickel (Ni)-Total	10,445	17,054	≤504	17,054-17,558	1.63-1.68	1.63
Zinc (Zn)-Total	274,829	463,570	1,033	464,603	1.69	1.69

[a] Maximum total out range = compost mass only to compost mass plus maximum mass in leachate

Mass balances were not attempted for arsenic, cadmium, cobalt and selenium because of non-detectable concentrations of these metals in the dewatered feed cake. Only three metals, copper, mercury and zinc, were observed at detectable concentrations in the compost leachate; only complete balances could be calculated for these three metals. The mass balance closures ranged from a low of 0.49 for mercury to a high of 1.69 for zinc. The mass calculated for the metals in the compost in all three cases dominated the output mass for the process, as indicated by the ratio of mass of metal in compost to mass of metal in the dewatered cake. The mass balance closures suggest that either the calculated input mass tended to be low, or the output mass in the compost tended to be high, assuming that the analytical concentrations are correct. The apparent inaccuracy of the balance is believed to be due to the imprecise solids flows, obtained primarily by anecdote from plant staff.

4.5.4.2 Pharmaceutical Compounds Mass Balances

A complete mass balance around the composting process could not be accomplished for the pharmaceutical compounds because samples of leachate were not submitted for analysis by the treatment plant. Based on mass balances for other treatment processes in this report, however, the mass of many of the pharmaceutical compound in the leachate produced is expected to be small compared to the mass still residing in the composted solids. As well, the analyses for the compost were based on the composted sludge solids with most of the fibrous wood and yard waste material removed prior to the actual analysis. Some of the pharmaceutical mass may possibly have been removed with the woody and fibrous material, either through surface adsorption or through absorption into the bulking agents along with the aqueous phase.

The level of certainty which can be attributed to the effectiveness of the composting process for reducing the mass of pharmaceutical compounds in the feed dewatered cake is reduced without mass balance estimates. It is possible, however, to compare the mass in the finished compost with the initial mass in dewatered cake, and to determine a mass that is unaccounted for in the composting process, as has been provided in **Table 38**, with the recognition that any error in the assumption of the volatile solids reduction in the composting process will affect the unaccounted masses obtained. The unaccounted mass includes mass of pharmaceuticals removed from the process in leachate returned to the plant, mass biodegraded in the composting process, and mass removed with the wood bulking agent.

Table 38. Estimates of Pharmaceutical Mass Flow in Dewatered Cake and Finished Compost, Prince Albert, SK

Pharmaceutical	Median Concentration (ng/g TS dw)		Mass of Contaminants (mg/d)		Fraction of Input Mass in Compost	% Mass Unaccounted
	Dewatered Cake	Compost	Dewatered Cake	Compost		
Furosemide	167	817	610	1,752	2.87	-187%
Gemfibrozil	72.0	54.5	263	117	0.45	55%
Ibuprofen	217	310	790	665	0.84	16%
Naproxen	100	2600	366	5,577	15.24	-1424%
Triclocarban	1880	1635	6,864	3,507	0.51	49%
Triclosan	7300	3950	26,652	8,472	0.32	68%
Azithromycin	1570	190	5,732	408	0.07	93%
Caffeine	646	1033	2,358	2,216	0.94	6%
Carbamazepine	63.1	53.7	230	115	0.50	50%
Ciprofloxacin	6595	2020	24,078	4,333	0.18	82%
Clarithromycin	41.2	13.2	150	28.4	0.19	81%
Dehydronifedipine	3.85	<1.38	14.1	<2.96	<0.21	>79%
Diphenhydramine	2735	1220	9,985	2,617	0.26	74%
Diltiazem	215	24.3	783	52.1	0.07	93%
Erythromycin-H_2O	51.3	12.5	187	26.9	0.14	86%
Fluoxetine	83.0	55.8	303	120	0.40	60%
Miconazole	442	268	1,614	574	0.36	64%
Ofloxacin	115	53.1	420	114	0.27	73%
Oxolinic Acid	1.35	<2.75	4.93	<5.90	<1.20	>-20%
Sulfamethazine	7	<3.03	25.6	<6.49	<0.25	>75%
Sulfamethoxazole	28.9	<1.05	105	<2.24	<0.021	>98%
Sulfathiazole	2.93	<2.62	10.7	<5.61	<0.52	>48%
Thiabendazole	22.5	11.4	82.1	24.4	0.30	70%
Trimethoprim	267	85.9	975	184	0.19	81%

Evaluation of the data in **Table 38** indicates that only a low fraction of the original input mass of some pharmaceuticals (e.g., azithromycin, diltiazem and sulfamethoxazole) remains in the final compost. While biodegradation is not the only mechanism responsible for reducing mass in the composting process, it is likely a major contributing removal mechanism. Conversely, there is a very significant increase in the mass of the non-steroidal anti-inflammatory naproxen (i.e., 15

times the initial mass in the feed dewatered cake). A similar phenomenon was observed in the composting process in Moncton, NB, described later in this report. The increase in the mass of naproxen through the composting process may possibly be due to metabolic formation from the breakdown of other compounds, or introduction of the compound with the wood bulking agent.

With the exception of the non-steroidal anti-inflammatory drug naproxen, the mass of the pharmaceutical compounds in the finished compost is often substantially less than the mass in the dewatered cake solids. It remains unclear, however, whether the reduced mass in the compost is a result of aerobic biodegradation, or whether there may be a significant loss of some pharmaceutical compounds in the composting process leachate, or with the removed wood bulking agent.

4.5.4.3 Fragrance and Alkylphenolic Compounds Mass Balances

As with the pharmaceutical compounds, complete mass balances could not be calculated for the alkylphenolic and fragrance compounds due to the lack of leachate samples. The mass of the compounds in the finished compost has been compared to the initial mass in dewatered cake, and the mass that is unaccounted for in the composting process, is provided in **Table 39**. It appears from this analysis that a majority of the Bisphenol A and polycyclic musks are retained with the compost. The fragrance compound DPMI was the only one in **Table 39** in which the majority of the compounds was unaccounted by the procedure used. Only the fragrance HHCB had a calculated mass in the compost greater than in the dewatered cake feed material. Because the compounds in this Table tend to be hydrophobic, there is a substantial probability that the unaccounted mass of the compounds results from biodegradation rather than from loss with the leachate.

Table 39. Estimates of Fragrance and Alkylphenolic Compounds Mass Flow in Dewatered Cake and Finished Compost, Prince Albert, SK

Pharmaceutical	Median Concentration (ng/g TS dw)		Mass of Contaminants (mg/d)		Fraction of Input Mass in Compost	% Mass Unaccounted
	Dewatered Cake	Compost	Dewatered Cake	Compost		
Alkylphenolics						
Bisphenol A	105	115	383	247	0.64	36%
Fragrance Compounds						
DPMI	70	40	256	86	0.34	66%
HHCB	1555	3470	5,677	7,443	1.31	-31%
AHTN	580	545	2,118	1,169	0.55	45%
ATII	85	110	310	236	0.76	24%

4.5.4.4 Effectiveness of Process for ESOC Removal

Based on the data for the composting operation at Prince Albert, SK, it would appear that the process has the potential to reduce the concentrations of a substantial number of pharmaceutical and fragrance compounds, and BPA. An additional sampling program in which leachate samples

were collected and analysed together with the dewatered cake and final compost would be required to confirm the ability of composting to substantially remove pharmaceutical compounds by biodegradation.

4.5.5 Section Summary

The concentrations of nitrate-N and of total Kjeldahl nitrogen (TKN, a measure of combined ammonia-N and organic-N) were lower in the compost than in the dewatered cake. Because the concentration of ammonia-N (a component of TKN) was relatively constant, the lower concentration of TKN in the compost can be likely attributed to a loss of organic-N, either through reduction of the volatile solids during composting, or due to loss from leachate generated during the process. The observed differences may also be due to variations in the composition of the two process streams at the time of sampling. Concentrations of total phosphorus and especially ortho-phosphate were observed at lower concentrations in the compost product than in the dewatered cake. The observed differences in concentrations of these compounds may again be due to loss in leachate from the composting pad, or due to sample variability.

Most metals except cadmium were identified above the detection limits in dewatered cake and compost samples. Arsenic, cobalt and selenium were not detected in the dewatered cake, but were above the detection limit in the compost. Only copper, mercury and zinc were detected in the leachate. Complete mass balances could be calculated for only three metals, copper, mercury and zinc, because only these three were observed at detectable concentrations in the compost leachate. The partial mass balance closures ranged from a low of 0.49 for mercury to a high of 1.69 for zinc. The output mass calculated for the metals in the compost in all three cases was far higher than the output mass in the leachate.

With respect to pharmaceutical compounds, the antimicrobial triclosan was found at the highest concentrations in both the dewatered cake and biosolids, at levels of 7,300 and 3,950 ng/g TS, respectively. Other compounds detected in the finished compost with median concentrations greater than 1,000 ng/g TS were triclocarban (antimicrobial), the antibiotics azithromycin and ciprofloxacin, the non-steroidal anti-inflammatory naproxen and the stimulants diphenhydramine and caffeine. There appears to be a slight shift in the distribution of detectable concentrations in the dewatered cake and compost. The number of compounds detected in both sampling campaigns declined from 20 in the dewatered cake to 14 in compost samples. The number of compounds detected in only one of the two campaigns rose from 4 in the dewatered cake to 7 in the compost, while the number of compounds detected in neither of the two campaigns also rose from 33 in the dewatered cake to 36 in the final compost. Complete mass balances around the composting process could not be accomplished for the pharmaceutical compounds because samples of leachate were not submitted for analysis by the treatment plant.

With the exception of the non-steroidal anti-inflammatory drug naproxen, the mass of the pharmaceutical compounds in the finished compost is often substantially less than the mass in the dewatered cake solids. It remains unclear, however, whether the reduced mass in the compost is a result of aerobic biodegradation, or whether there may be a significant loss of some pharmaceutical compounds in the composting process leachate, or with the removed wood bulking agent.

Bisphenol A was the only alkylphenolic compound detected; it was present in both the feed dewatered cake and compost samples at approximately the same concentrations. The polycyclic musk HHCB was found at the highest concentration, followed by AHTN at substantially lower concentrations. Nitro musk compounds were all below the limit of quantitation. A majority of the Bisphenol A and polycyclic musks are retained with the compost. The fragrance compound DPMI was the only one in which the majority of the compound mass was unaccounted by the procedure used. Only the fragrance HHCB had a calculated mass in the compost greater than in the dewatered cake feed material. Because these compounds tend to be hydrophobic, there is a substantial probability that the unaccounted mass of the compounds results from biodegradation rather than from loss with the leachate

Based on the data for the composting operation at Prince Albert, SK, it would appear that the process has the potential to reduce the concentrations of a substantial number of pharmaceutical and fragrance compounds, and BPA. An additional sampling program in which leachate samples were collected and analysed together with the dewatered cake and final compost would be required to confirm the ability of composting to substantially remove pharmaceutical compounds by biodegradation.

4.6 Geotextile Bag Filter Dewatering, Eganville, ON

4.6.1 Site Description

The Eganville sewage treatment plant (STP) is an extended aeration treatment facility with chlorination disinfection of the treated effluent prior to discharge to Bonnechere River. The design capacity of the existing treatment plant is 1,080 m^3/d, while the average daily dry weather flow is 700 m^3/d.

4.6.2 Wastewater Solids Treatment Description

The excess wastewater sludge is treated by aerobic digestion. There are in total two (2) aerobic digesters at the facility. Waste activated sludge (WAS) is pumped at an average flow rate of 4.29 $m^{3/}$d from the secondary clarifiers to the aerobic digesters for digestion. After the aerobic digestion, the processed WAS are transferred to the dewatering facility for dewatering and storage. Raw septage is also received, dewatered and stored (but separately from the aerobic digested WAS during all of these processes) at the dewatering facility.

The dewatering facility contains one sludge holding tank and a total of six geotextile bag filters (also called by their commercial trade names, “geotubes”) with two in operation at any given time and another one or two in storage (drying) mode. Polymer is added after the sludge holding tank prior to the mixing manifold. After sufficient mixing and contact time, the digested sludge or septage is then sent to the geotextile bag filter separately and stored there for about three or six months. During this study, the solids concentration in WAS ranged from 5.0% to 5.1% and in raw septage was 0.8%, while the dewatered biosolids cake solids concentration ranged from 13.6% to 15.4% and the filtrate solids concentration ranged from 0.0% to 0.06%.

After storage, the biosolids cake (i.e. dewatered digested WAS or dewatered septage) is sampled and taken to Ministry of the Environment (MOE) approved Organic Soil Conditioning Sites. The filtrate from the dewatering of the digested WAS or septage is directed back to the headworks of the STP.

For this project assessment, the biosolids treatment process of interest was the combined biosolids aerobic digestion and dewatering processes. A process schematic of the Eganville biosolids treatment process is shown in **Figure 9**. The three sampling locations included the feed sludge (i.e. WAS) to aerobic digester (S_a), or the raw septage (S_b) to dewatering facility, dewatered biosolids cake, and filtrate. Subsequent to the conclusion of the sampling period, during the data analysis period of the study, it was determined that approximately 25 % of the volume of the WAS feed was returned to the plant headworks as digester supernatant, which was not sampled for the mass balance closure. As a result, separate interpretations of the data have been developed for the campaigns with digested WAS as the feed and for the campaign with septage as the feed.

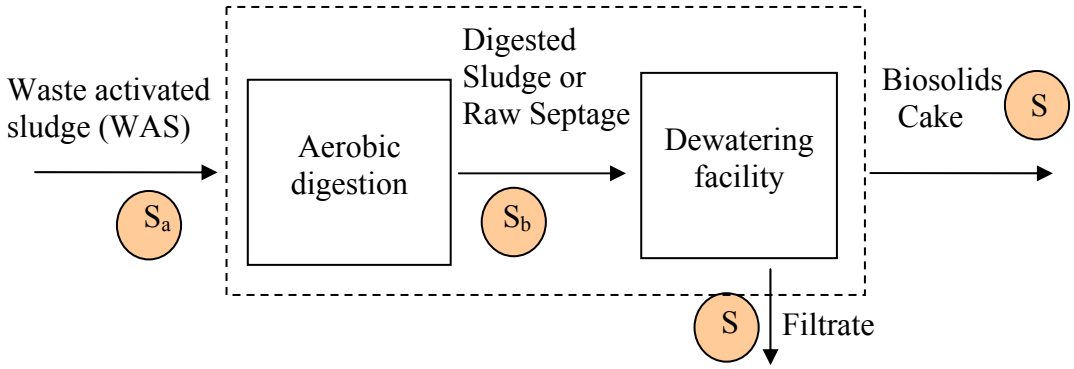

Figure 9. Schematic of Eganville Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on July 17, August 13 and August 26, respectively.

4.6.3 Sampling Results

4.6.3.1 Nutrients

The nutrient data that were reported by the laboratory are difficult to compare because of the different concentration units used, for liquid samples (WAS and filtrate) and solids samples (dewatered biosolids cake). It can be inferred from the WAS and filtrate sample concentration in **Table 40**, however, that there is a significant shift in the different nitrogen components, with a substantial reduction of ammonia-N and TKN, and a corresponding increase in nitrate-N, as would be expected with a combination of extended aeration and aerobic digestion processes. The soluble ortho-phosphate concentration in the filtrate is only slightly lower than in the WAS. The

total P concentration in the filtrate, however, is substantially less than in the WAS, possibly due to precipitation with accumulation in the dewatered cake sample.

Table 40. Nutrients in Waste Activated Sludge (WAS), Dewatered Biosolids Cake and Filtrate from Eganville, ON

Parameter	Concentration		
	WAS (mg/L)	Dewatered Biosolids Cake (mg/kg TS dw)	Filtrate (mg/L)
Nitrate-N	2.60	137	38.0
Nitrite-N	<0.50	<1.0	<0.50
Total Kjeldahl Nitrogen	406	45300	7.47
Ammonia as N	35.8	2050	4.30
Phosphorus, Total	302	32500	0.161
Phosphate-P (ortho)	0.0326	2.72	0.0265
Total Solids	11,100	151,000	740

4.6.3.2 Metals

Metals concentration data from the first sampling campaign are provided in **Table 41.** Chromium, copper, lead, mercury and zinc were detected in both the WAS and aerobically digested and dewatered biosolids cake; nickel was also detected in the biosolids cake. Copper and zinc were observed present at the highest concentrations in the WAS and dewatered biosolids cake samples. Zinc was the only metal detected in the geotextile bag filtrate, at a low concentration of 0.085 mg/L. Additional discussion of the nutrients is found in the Data Interpretation of this section.

4.6.3.3 Pharmaceuticals

The frequency of detection and median detected concentrations of the pharmaceutical compounds in the WAS/septage, dewatered biosolids cake and filtrate at the Eganville facility are presented in **Table 42**. A total of 13 pharmaceuticals was detected in the WAS samples from all the three sampling campaigns; 14 pharmaceuticals were detected in the dewatered biosolids samples from all the three campaigns and 13 pharmaceuticals were detected in the filtrate samples from all the three campaigns. The raw analytical data for the sampling campaigns at Eganville are provided in **Appendix Table A9**.

Table 41. Metals in Waste Activated Sludge (WAS), Dewatered Biosolids Cake and Filtrate, Eganville, ON

Parameter	Concentration

	WAS (mg/L)	Dewatered Biosolids Cake (mg/kg TS)	Filtrate (mg/L)
Arsenic (As)-Total	<0.10	<1.0	<0.010
Cadmium (Cd)-Total	<0.010	<1.0	<0.0010
Chromium (Cr)-Total	**0.10**	**3.7**	<0.010
Cobalt (Co)-Total	<0.080	<1.0	<0.0080
Copper (Cu)-Total	**4.89**	**140**	<0.010
Lead (Pb)-Total	**0.26**	**6.2**	<0.010
Mercury (Hg)-Total	**0.0172**	**0.193**	<0.00010
Molybdenum (Mo)-Total	<0.10	<1.0	<0.010
Nickel (Ni)-Total	<0.20	**2.4**	<0.020
Selenium (Se)-Total	<0.50	<1.0	<0.050
Zinc (Zn)-Total	**3.41**	**151**	**0.085**
Total Solids	11100	151000	740

Data in **bold font** are above the detection limit

Compounds detected at the highest concentrations in the dewatered solids from the geotextile bag filters included the antibiotic ciprofloxacin, the antimicrobials triclosan and triclocarban, and the diuretic furosemide. A number of compounds were detected in the feed WAS/septage and geotextile bag filtrate at very high concentrations, but were either non-detected or detected only at low concentrations in the dewatered cake. These compounds included caffeine and its metabolite 1,7-dimethylxanthine, acetaminophen, ibuprofen and its metabolite 2-hydroxy-ibuprofen and naproxen.

Table 42. Frequency of Detection and Median Concentrations of Pharmaceutical Compounds in Waste Activated Sludge (WAS) or Septage, Dewatered Solids Cake and Filtrate in Eganville, ON

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	WAS/ Septage	Dewatered Solids	Filtrate	WAS/ Septage (ng/g TS dw)	Dewatered Solids (ng/g TS dw)	Filtrate (ng/L)	WAS/ Septage (ng/g TS dw)	Dewatered Solids (ng/g TS dw)	Filtrate (ng/L)
Furosemide	1	1	1	987 [a]	1120 [a]	9800 [a]	<144-987	<56.7-1120	<180-9800
Gemfibrozil	0	0	0	NA	NA	NA	<35.8	<16.8	<4.77
Glipizide	0	0	0	NA	NA	NA	<143	<55.6	<19.1
Glyburide	0	1	**3**	NA	16.5 [a]	**14.1**	<71.6	<11.1-16.5	6.95-89.6
Hydrochlorothiazide	2	0	**3**	2408.5	NA	**1220**	<72-4700	<225	1090-8030
2-Hydroxy-ibuprofen	1	0	2	39000 [a]	NA	113050	<288-39000	<898	<90.8-208000
Ibuprofen	2	2	**3**	5681.4	553.5	**4870**	<54-11300	<55.3-931	174-75700
Naproxen	3	1	**3**	16.6	41.7 [a]	**1090**	13.7-3740	<11.1-41.7	7.68-23900
Triclocarban	**3**	**3**	**3**	**1410**	**2550**	**16.2**	1360-16900	1140-6580	4.08-321
Triclosan	**3**	**3**	1	**901**	**3050**	1490 [a]	817-12700	602-30600	<68.1-1490
Warfarin	0	0	1	NA	NA	20.5 [a]	<35.8	<13.9	<1.7-20.5
Acetaminophen	1	0	2	121000 [a]	NA	115355	<216-121000	<556	<68.1-226000
Azithromycin	**3**	**3**	**3**	**126**	**88.8**	**236**	94.9-230	58.9-111	135-1200
Caffeine	2	2	2	5040.25	609	49360	<54-10000	<55.4-1070	<17-97400
Carbadox	0	0	0	NA	NA	NA	<35.8	<13.9	<2.77
Carbamazepine	**3**	**3**	**3**	**43.2**	**40.2**	**897**	34.5-94	31.9-172	738-991
Cefotaxime	0	0	0	NA	NA	NA	<143	<77.7	<64.9
Ciprofloxacin	**3**	**3**	**3**	**7440**	**6470**	**118**	5330-7570	5800-26800	112-196
Clarithromycin	**3**	**3**	**3**	**32.3**	**32.3**	**195**	17.2-225	27.5-40.3	145-1770
Clinafloxacin	0	0	0	NA	NA	NA	<143	<55.6	<144
Cloxacillin	0	0	0	NA	NA	NA	<71.6	<27.8	<13.1
Dehydronifedipine	2	1	**3**	4.21	2.88 [a]	**32.2**	<2.16-5.56	<2.21-2.88	28.1-57.2
Diphenhydramine	**3**	**3**	**3**	**223**	**286**	**213**	223-900	203-525	141-798
Diltiazem	**3**	**3**	**3**	**21.6**	**5.4**	**24.5**	19.4-96.4	4.95-6.24	1.63-278

(continued)

Table 42 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	WAS/ Septage	Dewatered biosolids	Filtrate	WAS/ Septage (ng/g TS dw)	Dewatered biosolids (ng/g TS dw)	Filtrate (ng/L)	WAS/ Septage (ng/g TS dw)	Dewatered biosolids (ng/g TS dw)	Filtrate (ng/L)
Digoxin	0	1	0	NA	96.4 [a]	NA	<358	<56.1-96.4	<27.7
Digoxigenin	0	0	0	NA	NA	NA	<143	<81.2	<115
Enrofloxacin	1	0	0	11.2 [a]	NA	NA	<10.8-11.2	<27.8	<14.5
Erythromycin-H_2O	**3**	**3**	**3**	**13.6**	**10.7**	**117**	9.94-16.9	10.1-46.9	28.5-133
Flumequine	0	0	0	NA	NA	NA	<35.8	<13.9	<2.77
Fluoxetine	2	**3**	1	28.65	**45.9**	5.9 [a]	<5.4-38	28.7-101	<1.7-5.9
Lincomycin	0	0	0	NA	NA	NA	<71.6	<27.8	<18.1
Lomefloxacin	0	0	0	NA	NA	NA	<71.6	<27.8	<10.1
Miconazole	**3**	**3**	0	**221**	**350**	NA	211-328	291-376	<2.77
Norfloxacin	**3**	**3**	0	**600**	**581**	NA	458-1090	451-5590	<85.7
Norgestimate	0	0	0	NA	NA	NA	<71.6	<27.8	<13.2
Ofloxacin	2	**3**	0	598	**754**	NA	<54-763	351-975	<27.7
Ormetoprim	0	0	0	NA	NA	NA	<14.3	<5.56	<1.11
Oxacillin	0	0	0	NA	NA	NA	<71.6	<27.8	<5.55
Oxolinic Acid	0	0	0	NA	NA	NA	<14.3	<5.56	<5.7
Penicillin G	0	0	1	NA	NA	4.73 [a]	<239	<37.4	<0-4.73
Penicillin V	0	0	0	NA	NA	NA	<71.6	<27.8	<5.83
Roxithromycin	0	0	0	NA	NA	NA	<7.16	<2.78	<3.64
Sarafloxacin	0	0	0	NA	NA	NA	<372	<306	<94.4
Sulfachloropyridazine	0	0	0	NA	NA	NA	<35.8	<13.9	<27.2
Sulfadiazine	0	0	0	NA	NA	NA	<35.8	<13.9	<7
Sulfadimethoxine	0	0	0	NA	NA	NA	<7.16	<2.78	<5.92
Sulfamerazine	0	0	1	NA	NA	126 [a]	<14.3	<5.56	<0-126
Sulfamethazine	0	0	0	NA	NA	NA	<14.3	<5.56	<0.636
Sulfamethizole	0	0	1	NA	NA	22.7 [a]	<14.3	<5.56	<0-22.7
Sulfamethoxazole	**3**	2	1	**79.4**	10.8	1100 [a]	41-577	<2.21-17.4	<0-1100

(continued)

Table 42 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	WAS/ Septage	Dewatered biosolids	Filtrate	WAS/ Septage (ng/g TS dw)	Dewatered biosolids (ng/g TS dw)	Filtrate (ng/L)	WAS/ Septage (ng/g TS dw)	Dewatered biosolids (ng/g TS dw)	Filtrate (ng/L)
Sulfanilamide	0	0	0	NA	NA	NA	<358	<139	<15.9
Sulfathiazole	0	0	1	NA	NA	71.7 [a]	<35.8	<13.9	<0-71.7
Thiabendazole	2	**3**	**3**	20.15	**14.4**	**29.2**	<5.4-22.1	12.1-16.2	24.1-39.9
Trimethoprim	1	1	1	106 [a]	59.5 [a]	507 [a]	<5.4-106	<5.54-59.5	<21-507
Tylosin	0	0	0	NA	NA	NA	<143	<73.8	<37
Virginiamycin	0	0	0	NA	NA	NA	<349	<110	<278
1,7-Dimethylxanthine	1	0	2	23700 [a]	NA	38750	<540-23700	<1390	<170-69200

[a] indicates median value is from one detectable concentration only; [b] indicates highest identified detection limit for compound

Data in **bold font** are detected in both sampling campaigns; NA = not applicable (no median for all non-detectable concentrations)

The distribution of detectable concentrations in the biosolids digestion and dewatering process feed and effluent streams from the three sampling campaigns is found in **Table 43**. There appears to be a very minor shift in the distribution of detectable concentrations in the feed WAS to the dewatered digested biosolids. The number of compounds detected in all three campaigns remains approximately the same at 13 in the WAS and 14 in both the dewatered biosolids and filtrate. The greatest change is the decline of the number of compounds detected in the WAS in two of the three campaigns (7 compounds) to only three or four compounds detected twice in the dewatered biosolids and geotextile bag filtrate. The number of compounds (6) detected in only one of the three campaigns remains the same in the WAS and the dewatered biosolids cake, but rises to 10 in the filtrate, suggesting some depletion from the feed WAS. The dewatered digested biosolids exhibited a higher number of compounds never detected in any of the three samples than the WAS Feed or filtrate, possibly as a result of either some removal through the digestion process or due to different detection limits in the different matrices.

Table 43. Summary of Pharmaceutical Compound Detections in Waste Activated Sludge (WAS) or Septage, Dewatered Biosolids Cake and Filtrate in Eganville, ON

Frequency of detection in sampling campaigns (out of 3)	# Compounds in Process Streams		
	WAS/Septage	Dewatered biosolids	Filtrate
3	13	14	14
2	7	3	4
1	6	6	10
0	31	34	29
Total	57	57	57

4.6.3.4 Fragrance and Alkylphenolic Compounds

Concentrations and frequency of detection of fragrances and alkylphenolics are provided in **Table 44**. The raw analytical data from the sampling campaigns are provided in **Appendix Table A10**.

The polycyclic musks HHCB and AHTN were observed at the highest concentrations (e.g. greater than 1,000 ng/g TS dw) in the cake samples from dewatering both WAS and septage. The fragrance compounds AHDI and ATII were substantially higher in the solids samples when septage was processed (Campaign 2), compared to when WAS was processed in Campaign 1. Bisphenol A was also present at a high concentration in the dewatered septage cake. Musk xylene was detected in both the raw septage and dewatered cake sample, but not in any samples from the first sampling campaign with WAS. No other nitro musks were detected in either the feed samples or dewatered cake samples, or WAS filtrate. Results of the compounds in the filtrate from dewatering of the septage are pending. Additional discussion of the fragrance and alkylphenolics is found later in the Data Interpretation section.

Table 44. Concentrations of Fragrance and Alkylphenolic Compounds in Geotextile Bag Filter Feed, Dewatered Cake and Filtrate, Eganville, ON

Fragrance and Phenolic Compounds	Concentrations					
	Campaign 1 (WAS)			Campaign 2 (Septage)		
	WAS Feed (ng/g TS dw)	Dewatered Cake (ng/g TS dw)	Bag Filtrate (ng/L)	WAS Feed (ng/g TS dw)	Dewatered Cake (ng/g TS dw)	Bag Filtrate (ng/L)
Alkylphenolics						
Bisphenol A	290	160	<50	1550	2590	550
Octylphenol	<20	<20	20	<20	<20	<10
Nonylphenol	<140	<140	<90	<140	<140	<90
Fragrances						
DPMI	140	70	<20	210	180	<20
ADBI	<20	<20	<10	<20	<20	<10
AHDI	40	50	<10	470	370	<10
HHCB	1890	2330	830	8220	8990	570
AHTN	1110	1510	590	3930	2090	120
ATII	270	250	<10	960	550	30
Musk Moskene	<50	<50	<90	<50	<50	<90
Musk Tibetene	<80	<80	<50	<80	<80	<50
Musk Ketone	<120	<120	<90	<120	<120	<90
Musk Ambrette	<140	<140	<20	<140	<140	<20
Musk Xylene	<70	<70	<20	730	530	<20

4.6.4 Data Interpretation

4.6.4.1 Mass Balance Estimating Procedures

The mass balance for the Eganville facility is complex compared to some of the other facilities described in the report because there are two potential biosolids treatment processes to investigate, i.e. the aerobic digestion process and the geotextile bag filter storage/dewatering process. At the initiation of the sampling period, the process boundary was drawn to include the aerobic digestion and geotextile bag filter dewatering process because it was believed that the aerobically digested sludge was loaded directly to the geotextile bag filter without settling and withdrawal of supernatant. Subsequent to the conclusion of the sampling period, it was determined that approximately 25 % of the volume of the WAS feed was returned to the plant headworks as digester supernatant, which was not sampled. Thus only a partial mass balance based on the WAS can be accomplished.

Attempts at partial mass closures and mass balance estimates for the combined aerobic digestion and geotextile bag filter dewatering process are based on a total solids mass balance. The solids balance was calculated using measured total solids concentrations provided by the analytical laboratories, and process flows estimated by treatment plant personnel. Pertinent data used to develop the solids balance include:
Flow rate of WAS to aerobic digester = 4.29 m^3/d
Flow rate of aerobically digested WAS to geotextile bag filter = 3.29 m^3/d
Total solids concentration of WAS to digester = 1.11%

Total solids concentration of dewatered cake = 15.1%
Total suspended solids concentration of geotextile bag filter filtrate = 0.74 kg/m^3

Additionally, two process assumptions were made based on professional judgement, including a volatile fraction of 0.75of the WAS to the aerobic digester, and a volatile solids reduction of 67%. The calculated solids balance is depicted in **Figure 10**. The calculated mass balance indicated the geotextile bag filter captured approximately 90% of the solids in the aerobically digested WAS feed.

The solids balance, once established, was used to establish mass flows of contaminants from the WAS feed entering the aerobic digester, to the geotextile bag filter dewatered solids and filtrate. A volumetric flow rate of aerobic digester supernatant was estimated by using data from the plant staff after completion of the sampling campaigns, however, no samples of the supernatant were collected for analysis of the contaminants. The mass of contaminants removed in the supernatant could therefore not be estimated, and a complete balance through the combined processes was not possible.

4.6.4.2 Metals Mass Balances

Metals were only sampled once in this program, in the first sampling campaign. At Eganville, aerobically digested WAS was dewatered in the geotextile bag filter in the first campaign. The mass balance for metals is based on process flow rates for the feed WAS stream to the aerobic digester and for the geotextile bag filter filtrate, which are multiplied by the volumetric metal concentrations expressed in units of mg/L. The mass in the dewatered cake is calculated as the product of the dewatered cake concentration (expressed as a dry weight basis) and the mass rate of solids produced by the geotextile bag filter.

The results of the metals balance are presented in **Table 45**. Because only zinc had a detectable concentration in the geotextile bag filter filtrate, it is the only metal presented as a single value in the mass closure. When the filtrate concentrations are expressed as less than a detectable value, the actual concentration may range from the detection limit down to “zero”. It is possible to estimate a range of contaminant mass in the filtrate from the maximum at the detection limit down to 0. The calculated mass in the dewatered cake can be combined with the range of the mass in the filtrate to provide a range for the mass closure out of the geotextile bag filter.

As evident from **Table 45**, the mass closures around the combined aerobic digester and geotextile bag filter dewatering process are low, ranging from about 6 to 27% of the feed mass. Based on a comparison of the masses in the feed WAS and dewatered cake from the geotextile bag filter, a very substantial mass of the metals has been lost from or is unaccounted for in the process. The most probable unaccounted loss is in the digester supernatant, for which no analysis was performed. Alternatively, it may be possible that the mass entering the aerobic digester in the WAS feed is over-estimated, although this is considered unlikely, as it implies significant error in the WAS analytical measurements.

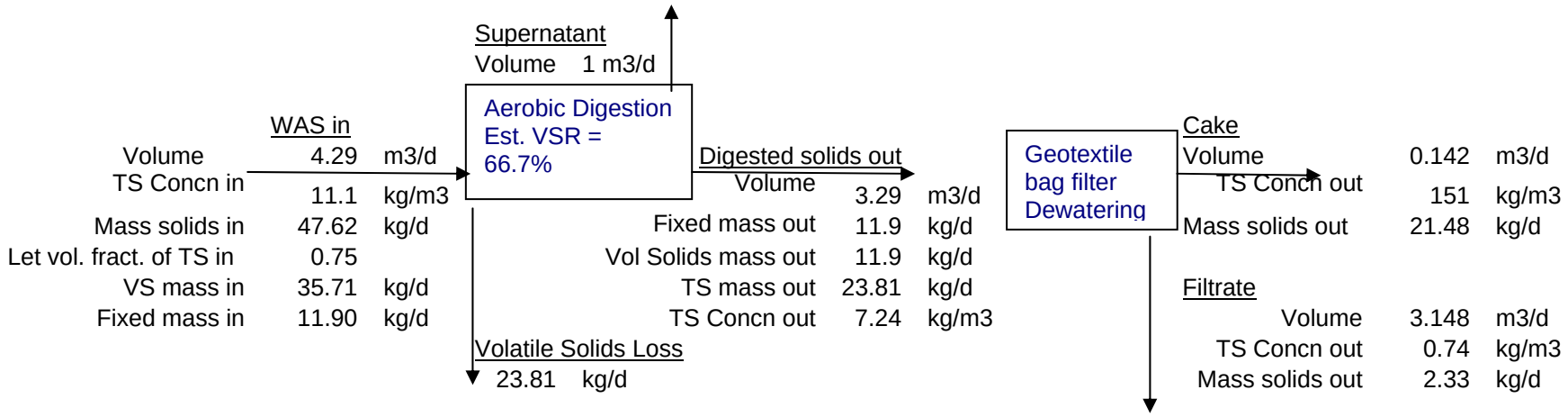

Figure 10. Total Solids Mass Balance for Combined Aerobic Digestion and Geotextile Bag Filter Dewatering Process, Eganville, ON

Table 45. Partial Mass Closure of Metals in the Combined Aerobic Digester and Geotextile Bag Filter Dewatering Process, Eganville, ON

Metal	Concentration of contaminant			Mass of contaminant (g/d)			% Closure
	WAS (mg/L)	Sludge cake (mg/kg TS)	Filtrate (mg/L)	WAS	Sludge cake	Filtrate	
Arsenic (As)-Total	<0.10	<1.0	<0.010				
Cadmium (Cd)-Total	<0.010	<1.0	<0.0010				
Chromium (Cr)-Total	0.10	3.7	<0.010	0.429	0.0826	<0.031	19.3%-26.6%
Cobalt (Co)-Total	<0.080	<1.0	<0.0080				
Copper (Cu)-Total	4.89	140	<0.010	20.98	3.13	<0.112	14.9%-15.4%
Lead (Pb)-Total	0.26	6.2	<0.010	1.12	0.139	<0.151	12.4%-26.0%
Mercury (Hg)-Total	0.0172	0.193	<0.00010	0.0738	0.0043	<0.0157	5.8%-27.1%
Molybdenum (Mo)-Total	<0.10	<1.0	<0.010				
Nickel (Ni)-Total	<0.20	2.4	<0.020				
Selenium (Se)-Total	<0.50	<1.0	<0.050				
Zinc (Zn)-Total	3.41	151	0.085	14.63	3.37	0.01	23.1%

4.6.4.3 Pharmaceutical Compounds Mass Balances

While the data presented in **Table 42** and **Table 44** are representative of the "bigger" picture at Eganville over a longer period, grouping the WAS and septage feed data together is problematic for two reasons. [Note that this issue does not apply to the assessment of the metals data, which were derived from only the first sampling campaign.] The first is that while septage is dewatered directly in the geotextile bag filters, the WAS is subjected to aerobic digestion with potential loss of some contaminants in the digester supernatant, which was not sampled. Thus the mass balance estimates for the two feed streams cannot be treated in the same manner. Secondly, the WAS is a residual of the extended aeration process at Eganville, and so the organic ESOC have already been exposed to aerobic treatment before reaching the aerobic digester. The extended aeration treatment results in significantly lower concentrations of the organic ESOC in the WAS compared to the septage as received, as demonstrated in **Table 46**.

With the exception of miconazole and norfloxacin, the concentrations in the septage are multiples higher than in the WAS, especially for the compounds ibuprofen, naproxen and caffeine.

For the two campaigns involving WAS as the feed material, only partial mass balance closures were possible. Pharmaceutical data were assessed by examining the mass of the compounds originally present in the WAS that remained in the dewatered biosolids cake, and the mass of the compound that was not accountable through the combined aerobic digestion and geotextile bag filter dewatering process. The unaccountable fraction can result from a combination of factors, including mass biodegraded in the aerobic digester, mass potentially lost in the aerobic digester supernatant, and mass potentially biodegraded in the geotextile bag filter. The results for this assessment are presented in **Table 47**. The raw analytical data for the two sampling campaigns involving WAS as a feed stream are found in **Appendix Table A9**.

Table 46. Comparison of Pharmaceutical Concentrations in WAS and Septage Feed Streams

Pharmaceutical	WAS		Septage Conc. (ng/g TS dw) (1 campaign)	Septage/ WAS
	Frequency of Detection in Sampling Campaigns (out of 2)	Median of Detected Concentration (ng/g TS dw)		
Furosemide	0	NA	987	A
Hydrochlorothiazide	1	117	4700	40.2
2-Hydroxy-ibuprofen	0	NA	39000	A
Ibuprofen	1	62.8	11300	180
Naproxen	2	15.2	3740	247
Triclocarban	2	1385	16900	12.2
Triclosan	2	859	12700	14.8
Acetaminophen	0	NA	121000	A
Azithromycin	2	110	230	2.1
Caffeine	1	80.5	10000	124
Carbamazepine	2	38.9	94	2.4
Ciprofloxacin	2	6385	7570	1.2
Clarithromycin	2	24.8	225	9.1
Dehydronifedipine	2	4.21	ND	B
Diphenhydramine	2	223	900	4.0
Diltiazem	2	20.5	96.4	4.7
Enrofloxacin	1	11.2	ND	B
Erythromycin-H_2O	2	11.8	16.9	1.4
Fluoxetine	2	28.7	ND	B
Miconazole	2	275	211	0.8
Norfloxacin	2	774	600	0.8
Ofloxacin	2	598	ND	B
Sulfamethoxazole	2	60.2	577	9.6
Thiabendazole	2	20.2	ND	B
Trimethoprim	0	NA	106	A
1,7-Dimethylxanthine	0	NA	23700	A

A = detected concentration in septage but not WAS
B = detected concentration in WAS but not septage

The fraction of the mass of each pharmaceutical in the feed WAS that resides in the dewatered biosolids provides an indication of the recalcitrance and hydrophobicity of the compound through the combined digestion and dewatering processes. In **Table 47**, the range of the input mass fraction residing in the dewatered cake is variable, from low values of 0.08 for the antibiotic sulfamethoxazole and 0.13 for the anti-angina drug diltiazem, to high values of 1.40 for the non-steroidal anti-inflammatory ibuprofen and 1.06 for the antimicrobial triclosan.

The percent of the compound in the feed mass that was not accountable through the combined aerobic digestion and dewatering process is an indication of loss from the system boundary either through biodegradation, or as mass removed in the aerobic digester supernatant. The unaccounted mass is determined by deducting the calculated masses of the pharmaceuticals in the dewatered cake and geotextile bag filter filtrate from the input mass in the WAS feed to the

Table 47. Partial Mass Closure of Pharmaceuticals in the Combined Aerobic Digester and Geotextile Bag Filter Dewatering Process, Eganville, ON

Compound	Median Concentration			Mass mg/d			Fraction of input mass in cake	% unaccounted
	WAS (ng/g TS dw)	Dewatered Biosolids (ng/g TS dw)	Filtrate (ng/L)	WAS	Dewatered Biosolids	Filtrate		
Hydrochlorothiazide	117	<185	1155	25.9	<20.4	2.95	<0.789	>9.7%-89%
Ibuprofen	62.8	176	2522	13.9	19.4	6.44	1.398	-86%
Naproxen	15.2	NA	549	3.35	<3.06	1.40	<0.915	>-33%-58%
Triclocarban	1385	1845	10.1	306	203	0.03	0.665	34%
Triclosan	859	1826	<111	190	201	<0.28	1.060	-6%
Azithromycin	110	99.9	186	24.4	11.0	0.47	0.451	53%
Caffeine	80.5	148	1320	17.8	16.3	3.37	0.917	-11%
Carbamazepine	38.9	36.1	865	8.58	3.97	2.21	0.463	28%
Ciprofloxacin	6385	6135	157	1,411	676	0.40	0.479	52%
Clarithromycin	24.5	29.9	170	5.47	3.30	0.43	0.603	32%
Dehydronifedipine	4.21	2.88	44.7	0.93	0.32	0.11	0.341	54%
Diphenhydramine	223	245	177	49.3	27.0	0.45	0.547	44%
Diltiazem	20.5	5.82	13.1	4.53	0.64	0.03	0.142	85%
Erythromycin-H_2O	11.8	10.4	80.8	2.60	1.15	0.21	0.441	48%
Fluoxetine	28.7	37.3	5.9	6.33	4.11	0.015	0.649	35%
Miconazole	275	321	<2.77	60.7	35.3	<0.01	0.582	42%
Norfloxacin	774	516	<85.7	171	56.9	<0.22	0.333	67%
Ofloxacin	598	553	<27.7	132	60.9	<0.07	0.461	54%
Sulfamethoxazole	60.2	10.8	NR	13.3	1.19	NR	0.090	<91%
Thiabendazole	20.2	15.3	26.7	4.45	1.69	0.068	0.379	61%

[a] Median is one detected concentration out of two samples

NA = not assessed ND = not detected NR = not reported

aerobic digester. The highest values for the unaccounted mass of pharmaceuticals were for sulfamethoxazole (91%) and diltiazem (85%), while the lowest percent values for unaccounted mass were for ibuprofen (-86%) and naproxen (as low as -33%), both anti-inflammatory drugs. The negative percent values reflect the fact that more of the pharmaceutical is found in the dewatered cake and filtrate than was initially present in the feed WAS.

The second sampling campaign involved septage being dewatered in the geotextile bag filter. For this one campaign, it is possible to calculate a full mass balance because all process streams were sampled. Calculated removal efficiencies of the pharmaceuticals were highly variable, as shown in **Table 48**, with high removals approaching 80% for hydrochlorothiazide, acetaminophen and sulfamethoxazole. The lowest estimated removal was observed for norfloxacin. For this campaign, removal may be attributable to biodegradation in the geotextile bag filter.

Categorised removal efficiencies of the pharmaceutical compounds in the septage by geotextile bag filter dewatering are provided in **Table 49**. Geotextile bag filter dewatering offers some removal of approximately half of the detected pharmaceuticals in the septage. Although no pharmaceuticals were removed by over 90%, six compounds were determined to have removal efficiencies in the 50-80% range, and five other compounds had removal efficiencies in the 0%-50% range. Negative removal efficiencies were calculated for 10 compounds in total.

Table 48. Mass Balance and Removal of Pharmaceutical Compounds by Geotextile Bag Filter Dewatering of Septage, Eganville, ON

Parameters	Concentration			Mass (mg/d)			% Removal
	Raw Septage (ng/g TS dw)	Dewatered Biosolids (ng/g TS dw)	Filtrate (ng/L)	Raw Septage	Dewatered Biosolids	Filtrate	
Furosemide	987	1120	9800	26.0	27.3	30.6	-123%
Hydrochlorothiazide	4700	<225	8030	124	<5.48	25.1	>75%- 80%
2-Hydroxy-ibuprofen	39000	<898	208000	1026	<21.9	650	>35%-37%
Ibuprofen	11300	931	75700	297	22.7	237	13%
Naproxen	3740	41.7	23900	98.4	1.02	74.7	23%
Triclocarban	16900	6580	321	445	160	1.00	64%
Triclosan	12700	30600	1490	334	745	0.60	-123%
Acetaminophen	121000	<225	226000	3185	<5.48	706	78%
Azithromycin	230	58.9	1200	6.05	1.43	3.75	14%
Caffeine	10000	1070	97400	263	26.1	304	-26%
Carbamazepine	94	172	897	2.47	4.19	2.80	-183%
Ciprofloxacin	7570	26800	112	199	652	0.35	-228%
Clarithromycin	225	40.3	1770	5.92	0.98	5.53	-10%
Diphenhydramine	900	525	798	23.7	12.8	2.49	36%
Diltiazem	96.4	4.95	278	2.54	0.12	0.87	61%
Erythromycin-H_2O	16.9	46.9	117	0.44	1.14	0.37	-239%
Miconazole	211	376	NQ	5.55	9.15	NQ	<-65%
Norfloxacin	600	5590	NQ	15.8	136	NQ	<-762%
Sulfamethoxazole	577	<2.25	1100	15.2	<0.055	3.44	77%
Trimethoprim	106	59.5	507	2.79	1.45	1.58	-9%
1,7-Dimethylxanthine	23700	<561	69200	624	<13.7	216	>63%-65%

NQ = not quantified by Laboratory

Table 49. Categorised Removal Efficiencies of Pharmaceutical Compounds by Geotextile Bag Filter Dewatering of Septage, Eganville, ON

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Furosemide	Caffeine	2-Hydroxy-ibuprofen	Hydrochlorothiazide	
Triclosan	Clarithromycin	Ibuprofen	Triclocarban	
Carbamazepine	Trimethoprim	Naproxen	Acetaminophen	
Ciprofloxacin		Azithromycin	Diltiazem	
Erythromycin-H_2O		Diphenhydramine	Sulfamethoxazole	
Miconazole			1,7-Dimethylxanthine	
Norfloxacin				
n=7	n=3	n=5	n=6	n=0

4.6.4.4 Fragrance and Alkylphenolic Compounds Mass Balances

The partial mass balance was calculated for the single campaign involving WAS as the feed to the combined aerobic digester and geotextile bag filter dewatering process. Results shown in **Table 50** therefore indicate the mass of a compound recovered in the dewatered cake as a fraction of the input mass, and the mass unaccounted for.

Table 50. Partial Mass Balance and Removal of Fragrance and Alkylphenolic Compounds by Geotextile Bag Filter Dewatering of WAS, Eganville, ON

Alkylphenolic or Fragrance	Concentration			Mass			% of Input Mass Un-accounted	Fract. of Input Mass in Cake
	WAS (ng/g TS dw)	Dewatered cake (ng/g TS dw)	Filtrate (ng/L)	WAS (mg/d)	Dewatered cake (mg/d)	Filtrate (mg/d)		
Alkylphenolics								
Bisphenol A	290	160	<50	64.1	17.6	<0.13	72%	0.275
Fragrances								
DPMI	140	70	<20	30.9	7.72	<0.052	75%	0.249
AHDI	40	50	<10	8.84	5.51	<0.026	37%	0.624
HHCB	1890	2330	830	418	257	2.12	38%	0.615
AHTN	1110	1510	590	245	166	1.51	32%	0.679
ATII	270	250	<10	60.0	27.6	<0.026	54%	0.462

A substantial fraction of the mass of the polycyclic musks AHDI, HHCB and AHTN (0.62-0.68) resides in the dewatered cake solids. For Bisphenol A and the other musks DPMI and ATII, however, the majority of the input mass is unaccounted for, which means that it is either lost in aerobic digester supernatant, or biodegraded. Because these compounds tend to be hydrophobic, the mass leaving the process in the aerobic digester supernatant is expected to be small; consequently, there is a reasonable possibility that they are mostly biodegraded though the

combined aerobic digester and geotextile bag filter. It is not possible to determine where the majority of the biodegradation occurs, whether in the aerobic digester or in the geotextile bag filter.

Mass balances were also calculated for the fragrance and alkylphenolic compounds in the septage feed and dewatered cakes samples of Campaign 2. The results are shown in **Table 51.**

Table 51. Mass Balance and Removal of Fragrance and Alkylphenolic Compounds by Geotextile Bag Filter Dewatering of Septage, Eganville, ON

Alkylphenolic or Fragrance	Concentrations			Mass of Contaminants (mg/d)			% Removal	Fract. of Input Mass in Cake
	Raw Septage (ng/g TS dw)	Dewater. Cake (ng/g TS dw)	Filtrate (ng/L)	Raw Septage	Dewater. Cake	Filtrate		
Alkylphenolics								
Bisphenol A	1550	2590	550	40.8	68.0	1.71	-71%	1.67
Fragrances								
DPMI	210	180	<20	5.53	4.73	<0.06	13%	0.86
AHDI	470	370	<10	12.4	9.71	<0.03	21%	0.79
HHCB	8220	8990	570	216	236	1.77	-10%	1.09
AHTN	3930	2090	120	103	54.9	0.37	46%	0.53
ATII	960	550	30	25.3	14.4	0.09	43%	0.57
Musk Xylene	730	530	<20	19.2	13.9	<0.06	28%	0.72

The data indicate there was no reduction of Bisphenol A as a result of the geotextile bag filter dewatering of septage. With the exception of the polycyclic musk HHCB, removal efficiencies for the fragrance compounds ranged from 13% to as high as 46%, as was determined for AHTN. A greater portion of the compounds from the septage feed are retained in the dewatered solids than when WAS with aerobic digestion was the feed material. The observation was particularly relevant for Bisphenol A, and the polycyclic musks HHCB and DPMI.

Table 52 provides a comparison of the fraction of input masses of the fragrance and alkylphenolic compounds that reside in the dewatered cake solids, and the fraction of feed mass that are either unaccounted (WAS feed) or removed (septage feed). With the exception of AHTN, a greater fraction of the compounds resides in the dewatered cake when septage is the feed than when the feed is WAS. Similarly, with the exception of AHTN, the input mass that is unaccounted for is greater with the WAS feed than is calculated to be removed with the septage feed. This evaluation would tend to indicate that aerobic digestion probably has a greater role in increasing the unaccounted input mass in the WAS feed than does the geotextile bag filter.

Table 52. Comparison of Mass Contaminant Fate with Different Process Feed Streams, Eganville, ON

Alkylphenolic or Fragrance	WAS		Septage	
	Fract. of Input Mass in Cake	% of Input Mass Un-accounted	Fract. of Input Mass in Cake	% Removed
Alkylphenolics				
Bisphenol A	0.28	72%	1.67	-71%
Fragrances				
DPMI	0.25	75%	0.86	13%
AHDI	0.62	37%	0.79	21%
HHCB	0.62	38%	1.09	-10%
AHTN	0.68	32%	0.53	46%
ATII	0.46	54%	0.57	43%
Musk Xylene			0.72	28%

4.6.4.5 Effectiveness of Process for ESOC Removal

Based on the data for the septage treatment, the geotextile bag filter dewatering alone appears to offer some ability to reduce the mass of pharmaceuticals and fragrance compounds (but not Bisphenol A) in the raw septage. The aerobic digestion process in addition probably offers some additional removal of compounds in WAS prior to the geotextile bag filter dewatering, although analysis of the digester supernatant would be required to determine the actual removal capability. Compounds that have a high positive value for the percent of input mass in WAS that is unaccounted are among those most likely to be favourably removed by aerobic biodegradation in the digester.

4.6.5 Section Summary

There is a significant shift in the different nitrogen components through the combined aerobic digestion and geotextile bag filter dewatering process, with a substantial reduction of ammonia-N and TKN, and a corresponding increase in nitrate-N, as would be expected with the aerobic digestion processes. Although the soluble ortho-phosphate concentration in the filtrate is only slightly lower than in the WAS, the total P concentration in the filtrate is substantially less than in the WAS, probably due to precipitation with accumulation of the solids phase in the dewatered cake sample.

Chromium, copper, lead, mercury and zinc were detected in both the WAS and aerobically digested and dewatered biosolids cake; nickel was also detected in the biosolids cake. Copper and zinc were observed present at the highest concentrations in the WAS and dewatered biosolids cake samples. Zinc was the only metal detected in the geotextile bag filter filtrate, at a low concentration of 0.085 mg/L. The mass closures of metals around the combined aerobic digester and geotextile bag filter dewatering process are low, ranging from about 6 to 27% of the feed mass, indicating that a very substantial mass of the metals has been lost from or is unaccounted for in the process. The most probable unaccounted loss is in the digester supernatant, for which

no analysis was performed. Alternatively, it may be possible that the mass entering the aerobic digester in the WAS feed is over-estimated, although this is considered unlikely

The fraction of the mass of each pharmaceutical in the feed WAS that resides in the dewatered biosolids provides an indication of the recalcitrance and hydrophobicity of the compound through the combined digestion and dewatering processes. The range of the input mass fraction residing in the dewatered cake is variable, from low values of 0.08 for the antibiotic sulfamethoxazole and 0.13 for the anti-angina drug diltiazem, to high values of 1.40 for the non-steroidal anti-inflammatory ibuprofen and 1.06 for the antimicrobial triclosan. The percent of the compound in the WAS feed mass that was not accountable through the combined aerobic digestion and dewatering process is an indication of loss from the system boundary either through biodegradation, or as mass removed in the aerobic digester supernatant. The highest values for the unaccounted mass of pharmaceuticals were for sulfamethoxazole (91.7%) and diltiazem (82.7%), while the lowest percent values for unaccounted mass were for ibuprofen (-86%) and naproxen as low as -33%), both anti-inflammatory drugs. The negative percent values reflect the fact that more of the pharmaceutical is found in the dewatered cake and filtrate than was initially present in the feed WAS.

For the second sampling campaign, involving dewatering of septage in the geotextile bag filter, it is possible to calculate a mass balance because all process streams were sampled. Calculated removal efficiencies of the pharmaceuticals were highly variable, with high removals approaching with high removals approaching 80% for hydrochlorothiazide, acetaminophen and sulfamethoxazole. The lowest estimated removal was observed for norfloxacin. For this campaign, removal may be attributable to biodegradation in the geotextile bag filter. Although no pharmaceuticals were removed by over 90%, six compounds were determined to have removal efficiencies in the 50-80% range, and five other compounds had removal efficiencies in the 0%-50% range. Negative removal efficiencies were calculated for 10 compounds in total.

The polycyclic musks HHCB and AHTN were observed at the highest concentrations (e.g. greater than 1,000 ng/g TS) in the cake samples from dewatering both WAS and septage. Bisphenol A was also present at a high concentration in the dewatered septage cake. Musk xylene was detected in both the raw septage and dewatered cake sample, but not in any samples from the first sampling campaign with WAS. No other nitro musks were detected in either the feed samples or dewatered cake samples, or WAS filtrate. The musk xylene was reduced to below the detection limit in the dewatered digested biosolids. With the exception of AHTN, a greater fraction of the compounds resides in the dewatered cake when septage is the feed than when the feed is WAS. Similarly, with the exception of AHTN, the input mass that is unaccounted for is greater with the WAS feed than with the septage feed. This evaluation would tend to indicate that aerobic digestion probably has a role in increasing the input mass that is unaccounted with the WAS feed.

Based on the data for the septage treatment, the geotextile bag filter dewatering alone appears able to reduce the mass of some pharmaceuticals and fragrance compounds (but not Bisphenol A) in the raw septage. The aerobic digestion process in addition probably offers some additional removal of compounds in WAS prior to the geotextile bag filter dewatering, although analysis of the digester supernatant would be required to determine the actual removal capability.

4.7 Thermal Drying, Smiths Falls, ON

4.7.1 Site Description

The Smiths Falls Wastewater Treatment Plant (WWTP) is a conventional activated sludge system with tertiary filtration and ultraviolet (UV) disinfection. The treated effluent is discharged to Rideau River. The design capacity of the existing treatment plant is 14,700 m^3/d, while the average daily dry weather flow is 8,250 m^3/d.

4.7.2 Biosolids Treatment Description

Combined primary sludge and waste activated sludge (WAS) are dewatered using a belt filter press. The dewatered biosolids cake enters the heat drying system (i.e. drying drum) as a single stream for drying. In the drying drum, the dewatered cake is contacted directly with hot air. The temperature varies from 250 to 450 °C at the inlet end of the dry drum. The heat dried pellets exit the drying drum between 80-130 °C. The moisture is evaporated from the surface of the pellets as the product moves along the drum.

The feed rate of dewatered biosolids cake to the heat drying system is 7.5 m^3/d and the production rate of heat dried pellets is 1.53 m^3/d. During this study, the solids concentration in the feed sludge to the heat drying system (i.e. belt press dewatered biosolids cake) ranged from 17.3% to 20.3%; while the total solids concentrations of the heat dried pellets ranged from 92.1% to 93.4%.

For this project assessment, the biosolids treatment process of interest was the heat drying process. The two sampling locations included belt press biosolids cake and heat dried pellets. A process schematic of the Smiths Falls biosolids treatment process is shown in **Figure 11**.

The plant was considered by plant staff to be in normal operation during the two sampling campaigns. Samples were collected and shipped to the analytical laboratories on September 2, and October 19, 2009 respectively. Due to prolonged shutdowns of the drying unit in the summer and autumn, plans for a third sampling campaign were cancelled in November, 2009.

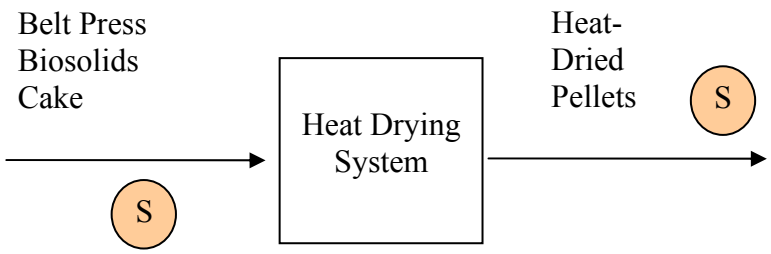

Figure 11. Schematic of Smiths Falls Biosolids Process and Sampling Locations

4.7.3 Sampling Results

4.7.3.1 Nutrients

Concentrations of nitrate-N, total Kjeldahl nitrogen (TKN) and soluble ortho-phosphate were all significantly lower in the heat dried biosolids pellets than in feed sludge (i.e. belt press dewatered cake) (**Table 53**), while ammonia-N was reduced to a lesser extent. The reduction in nitrate is possible due to a thermo-chemical reaction resulting from the high temperature (250-450 °C) drying process. The reduced concentration of TKN may be a result of destruction of the organic-N component of the TKN at the elevated drying temperature. The observed differences may also be due to some of the TKN components, including ammonia, amine and other organic nitrogen compounds, being driven off during high temperature drying process. The concentration of ortho-phosphate may be lower in the dried pellets compared to the feed sludge due to precipitation from reduced solubility at the elevated temperatures during drying. The difference in total phosphorus may either reflect the variations in the composition of the two process streams at the time of sampling, or possibly a difference in the ability of the analytical digestion process to recover all the phosphorus in the dried pellets compared to the belt press cake.

Table 53. Nutrients in Belt Press Biosolids Cake and Heat Dried Pellets from Smiths Falls, ON

Parameter	Concentration (mg/kg TS dw)	
	Belt Press Cake	Heat Dried Pellets
Nitrate-N	16.0	2.9
Nitrite-N	<1.0	<1.0
Total Kjeldahl Nitrogen	201,000	32,600
Ammonia as N	2,450	1,890
Phosphorus, Total	26,200	18,900
Phosphate-P (ortho)	20.9	1.20
Total Solids	200,000	914,000

4.7.3.2 Metals

All the metals studied were identified above the detection limits in belt press cake, as shown in **Table 54**. All the metals except cadmium were also detected in heat dried pellets. In general the concentrations of the metals in the feed dewatered sludge and dried pellets were similar. Zinc and copper were observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. Additional discussion of the metals is found later in this section under Data Interpretation.

4.7.3.3 Pharmaceuticals

The frequency of detection and median and range of detected concentrations of the pharmaceutical compounds in the dewatered cake and heat dried pellets at the Smiths Falls facility are presented in **Table 55**. The raw concentration data from the three sampling campaigns are found in **Appendix Table A11**. It should be noted that there are a total of two instead of three sampling campaigns for the Smiths Falls facility during this study due to the operational difficulties with the drier described above. A total of 23 pharmaceuticals were

detected in the dewatered cake samples from both sampling campaigns; 22 pharmaceuticals were detected in the heat dried pellets samples from both campaigns.

Table 54. Metals in Belt Press Biosolids Cake and Heat Dried Pellets, Smiths Falls, ON

Parameter	Concentration (mg/kg TS dw)	
	Belt Press Cake	Heat Dried Pellets
Arsenic (As)-Total	2.8	3.0
Cadmium (Cd)-Total	1.4	<1.0
Chromium (Cr)-Total	19.6	22.5
Cobalt (Co)-Total	2.7	2.8
Copper (Cu)-Total	319	334
Lead (Pb)-Total	51.0	54.0
Mercury (Hg)-Total	0.478	0.453
Molybdenum (Mo)-Total	3.7	4.1
Nickel (Ni)-Total	10.6	11.9
Selenium (Se)-Total	3.2	3.2
Zinc (Zn)-Total	549	565
Total Solids	200000	914000

Table 55. Frequency of Detection, Median and Range of Detected Concentrations of Pharmaceutical Compounds in Belt Press Biosolids Cake and Heat Dried Pellets, Smiths Falls, ON

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets
Furosemide	**2**	1	**305**	238 [a]	169-441	<107-238
Gemfibrozil	**2**	**2**	**9.4**	**10.8**	7.5-11.3	8.7-12.9
Glipizide	0	0	NA	NA	<11.6 [b]	<10.7 [b]
Glyburide	0	1	NA	6.39 [a]	<5.8 [b]	<5.2-6.4
Hydrochlorothiazide	0	0	NA	NA	<38.7 [b]	<35.7 [b]
2-Hydroxy-ibuprofen	0	0	NA	NA	<155 [b]	<143 [b]
Ibuprofen	**2**	**2**	**197**	**208**	183-210	204-211
Naproxen	**2**	**2**	**61.9**	**67.2**	53.9-69.8	48.2-86.2
Triclocarban	**2**	**2**	**4480**	**3960**	4470-4490	3830-4090
Triclosan	**2**	**2**	**11850**	**11485**	11800-11900	8670-14300
Warfarin	0	0	NA	NA	<2.9 [b]	<2.68 [b]
Acetaminophen	0	0	NA	NA	<116 [b]	<107 [b]
Azithromycin	**2**	**2**	**111**	**146**	95.3-127	138-153
Caffeine	**2**	**2**	**90.5**	**147**	82.7-98.3	118-175
(continued)						

Table 55 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets
Carbadox	0	0	NA	NA	<2.9 [b]	<2.68 [b]
Carbamazepine	**2**	**2**	**49.2**	**99.7**	40.8-57.6	91.3-108
Cefotaxime	0	0	NA	NA	<39.4 [b]	<52.6 [b]
Ciprofloxacin	**2**	**2**	**4755**	**2530**	3380-6130	2160-2900
Clarithromycin	**2**	**2**	**56.9**	**44.9**	30.6-83.1	31.8-58
Clinafloxacin	0	0	NA	NA	<17.2 [b]	<22.8 [b]
Cloxacillin	0	0	NA	NA	<2.79 [b]	<4.17 [b]
Dehydronifedipine	**2**	**2**	**4.5**	**9.6**	4.3-4.63	9.02-10.2
Diphenhydramine	**2**	**2**	**590**	**833**	571-609	770-895
Diltiazem	**2**	**2**	**160**	**303**	143-177	284-321
Digoxin	0	0	NA	NA	<29 [b]	<26.8 [b]
Digoxigenin	0	0	NA	NA	<28.4 [b]	<69.9 [b]
Enrofloxacin	**2**	**2**	**13.3**	**10.1**	10.2-16.4	8.05-12.2
Erythromycin-H_2O	**2**	**2**	**9.2**	**9.1**	8.9-9.5	7.2-11
Flumequine	0	0	NA	NA	<2.9 [b]	<2.7 [b]
Fluoxetine	**2**	**2**	**83.7**	**90**	70.4-97.0	83.6-96.4
Lincomycin	0	0	NA	NA	<13.5 [b]	<12.5 [b]
Lomefloxacin	0	0	NA	NA	<5.8 [b]	<5.4 [b]
Miconazole	**2**	**2**	**384**	**542**	360-408	474-609
Norfloxacin	**2**	**2**	**2145**	**1140**	1800-2490	1090-1190
Norgestimate	0	0	NA	NA	<6.7 [b]	<9.9 [b]
Ofloxacin	**2**	**2**	**282**	**167**	149-415	101-233
Ormetoprim	0	0	NA	NA	<1.16 [b]	<1.07 [b]
Oxacillin	0	0	NA	NA	<5.8 [b]	<5.4 [b]
Oxolinic Acid	0	1	NA	1.04 [a]	<1.16 [b]	<1.03-1.04
Penicillin G	0	0	NA	NA	<2.3 [b]	<2.1 [b]
Penicillin V	0	0	NA	NA	<5.8 [b]	<5.4 [b]
Roxithromycin	0	0	NA	NA	<2.4 [b]	<3.0 [b]
Sarafloxacin	0	0	NA	NA	<85.4 [b]	<159 [b]
Sulfachloropyridazine	0	0	NA	NA	<2.9 [b]	<2.7 [b]
Sulfadiazine	0	0	NA	NA	<2.9 [b]	<2.7 [b]
Sulfadimethoxine	0	0	NA	NA	<1.5 [b]	<1.5 [b]
Sulfamerazine	0	0	NA	NA	<2.1 [b]	<4.6 [b]
Sulfamethazine	0	0	NA	NA	<4.9 [b]	<6.3 [b]
Sulfamethizole	0	0	NA	NA	<1.8 [b]	<2.2 [b]
Sulfamethoxazole	**2**	**2**	**7.6**	**28.5**	3.7-11.6	18.6-38.3
Sulfanilamide	0	1	NA	63.1 [a]	<29 [b]	<25.9-63.1
(continued)						

Table 55 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets
Sulfathiazole	0	0	NA	NA	<3.0 [b]	<2.7 [b]
Thiabendazole	**2**	**2**	**7.21**	**6.75**	6.6-7.8	5.6-7.9
Trimethoprim	**2**	**2**	**31.0**	**31.2**	30-31.9	30.6-31.8
Tylosin	0	0	NA	NA	<38.7 [b]	<34.5 [b]
Virginiamycin	0	0	NA	NA	<145 [b]	<157 [b]
1,7-Dimethylxanthine	0	0	NA	NA	<290 [b]	<268 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in both sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the dried pellets included the anti-microbials triclosan and triclocarban, and the antibiotics ciprofloxacin and norfloxacin.

The distribution of detectable concentrations in the dewatered cake and heat dried pellets from all the two sampling campaigns is found in **Table 56**. The number of compounds detected in both sampling campaigns remained relatively constant at 23 in the dewatered cake and 22 in the heat dried pellets. The number of compounds detected in only one of the two campaigns rises from 0 in dewatered cake to 4 in heat dried pellets; at the same time the number of compounds not detected in either of the two campaigns declined from 34 on the feed sludge to 31 on the dried pellets. The probable cause for this observed shift is the change in detection limits associated with the dried pellets relative to the dewatered cake.

Table 56. Summary of Pharmaceutical Compound Detections in Belt Press Biosolids Cake and Heat Dried Pellets from Smiths Falls, ON

Frequency of detection in sampling campaigns (out of 2)	Number of Compounds in Process Streams	
	Dewatered Cake	Heat Dried Pellets
2	23	22
1	0	4
0	34	31
Total	57	57

Note: there are two (instead of three) sampling campaigns for Smiths Falls.

4.7.3.4 Fragrance and Alkylphenolic Compounds

The concentration data for the alkylphenolic and fragrance compounds are presented in **Table 57**.

The raw analytical data are provided in **Appendix Table A12**. Bisphenol A was detected in the belt press filter cake and heat dried pellets in only one of the two sampling campaigns. Octylphenol and nonylphenol were not detected in any samples in the two sampling campaigns. The only synthetic musks detected in samples were HHCB, AHTN and ATII; these compounds were detected in both the feed press cake and dried pellets in both sampling campaigns. The concentrations of HHCB and AHTN were approximately an order of magnitude higher in concentration compared to ATII.

Table 57. Frequency of Detection, Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Belt Press Biosolids Cake and Heat Dried Pellets from Smiths Falls, ON

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Belt Pressed Cake	Heat Dried Pellets	Belt Pressed Cake	Heat Dried Pellets	Belt Pressed Cake	Heat Dried Pellets
Alkylphenolics						
Bisphenol A	1	1	110	120	<80-110	<80-120
Octylphenol	0	0	NA	NA	<20	<20
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	0	0	NA	NA	<40	<40
ADBI	0	0	NA	NA	<20	<20
AHDI	0	0	NA	NA	<30	<30
HHCB	**2**	**2**	**3990**	**2795**	3810-4170	2740-2850
AHTN	**2**	**2**	**1540**	**830**	1340-1740	550-1110
ATII	**2**	**2**	**100**	**95**	70-130	90-100
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	<120	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	0	0	NA	NA	<70	<70

Data in **bold font** are detected in both sampling campaigns

4.7.4 Data Interpretation

4.7.4.1 Mass Balance Estimation Procedures

Concentrations for metals and pharmaceutical compounds are expressed on a dry weight basis (i.e., mg/kg TS for metals, ng/g TS for pharmaceuticals), and so the mass balances for the both types of contaminants are based on a total solids balance around the thermal drying process. The solids balance around the pellet drier is estimated using the mean values of the total solids concentrations in the dewatered sludge cake feed and dried biosolids pellets out of the process from the two sampling campaigns. The pertinent solids concentration and flow data are:

Volumetric feed rate of dewatered cake = 7.5 m^3/d

Mean measured total solids concentration in dewatered cake feed = 196 kg/m^3

Volumetric exit rate of dried pellets = 1.53 m^3/d
Mean measured total solids concentration in dried biosolids pellets = 983 kg/m^3

In the balance, it was assumed the difference in the volume of dewatered cake solids entering and leaving the drier was the volume of water evaporated through the process. The total solids balance is depicted in **Figure 12**. The calculated masses of solids in and out of the drier are approximately equal; the higher mass of solids leaving the drier than the mass entering the drier is considered an artifact of the variability associated with sampling and analysis of sludge and biosolids, and estimating the flow rates.

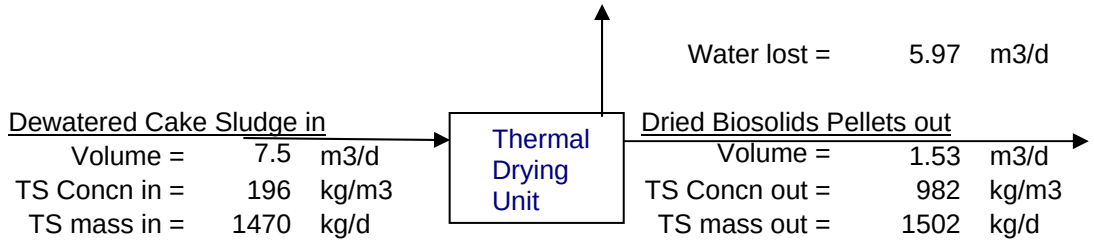

Figure 12. Solids Mass Balance around Thermal Sludge Drying Process, Smiths Falls, ON

4.7.4.2 Metals Mass Balances

Calculated mass balances for the metals are presented in **Table 58**. The mass balances indicate that in most cases, the mass of the individual metals leaving the biosolids pellet drier are slightly elevated compared to the mass of each in the feed dewatered cake. The calculated increase is likely due to minor inaccuracies or variations in the volumetric flow data. The exceptions cadmium and mercury exhibited a slight reduction in mass through the drying process. These two metals can be volatilized at elevated temperatures, and it is possible that the calculated reduction in mass may be due to volatilization.

Table 58. Mass Balance Closure Calculations for Metals in Biosolids Thermal Drying Process, Smiths Falls, ON

Metal	Concentration (mg/kg TS dw)		Mass of Contaminant (g/d)		% Closure
	Dewatered Cake	Heat Dried Pellets	Dewatered Cake	Heat Dried Pellets	
Arsenic (As)-Total	2.8	3.0	4.11	4.51	110%
Cadmium (Cd)-Total	1.4	<1.0	2.05	<1.5	<73%
Chromium (Cr)-Total	19.6	22.5	28.7	33.8	118%
Cobalt (Co)-Total	2.7	2.8	3.96	4.21	106%
Copper (Cu)-Total	319	334	468	502	107%
Lead (Pb)-Total	51.0	54.0	74.8	81.2	109%
Mercury (Hg)-Total	0.478	0.453	0.70	0.68	97%
Molybdenum (Mo)-Total	3.7	4.1	5.43	6.16	114%
Nickel (Ni)-Total	10.6	11.9	15.5	17.9	115%
Selenium (Se)-Total	3.2	3.2	4.69	4.81	103%
Zinc (Zn)-Total	549	565	805	849	106%

4.7.4.3 Pharmaceutical Compounds Mass Balances

The results of the mass estimates for the pharmaceutical compounds are provided in **Table 59**. Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids have not been included in **Table 59**.

Table 59. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Biosolids Thermal Drying Process, Smiths Falls, ON

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Belt Pressed Cake	Heat Dried Pellets	Belt Pressed Cake	Heat Dried Pellets	
Furosemide	305	238	447	358	20%
Gemfibrozil	9.42	10.8	13.8	16.2	-17%
Ibuprofen	197	207.5	288	312	-8%
Naproxen	61.9	67.2	90.7	101	-11%
Triclocarban	4480	3960	6569	5954	9%
Triclosan	11850	11485	17377	17267	1%
Azithromycin	111	146	163	219	-34%
Caffeine	90.5	147	133	220	-66%
Carbamazepine	49.2	99.7	72.2	150	-108%
Ciprofloxacin	4755	2530	6973	3804	45%
Clarithromycin	56.9	44.9	83.4	67.5	19%
Dehydronifedipine	4.47	9.61	6.6	14.5	-121%
Diphenhydramine	590	833	865	1252	-45%
Diltiazem	160	303	235	455	-94%
Enrofloxacin	13.3	10.1	19.5	15.2	22%
Erythromycin-H_2O	9.18	9.08	13.5	13.6	-1%
Fluoxetine	83.7	90	123	135	-10%
Miconazole	384	542	563	814	-45%
Norfloxacin	2145	1140	3145	1714	46%
Ofloxacin	282	167	414	251	39%
Sulfamethoxazole	7.64	28.5	11.2	42.8	-282%
Thiabendazole	7.21	6.75	10.6	10.2	4%
Trimethoprim	31.0	31.2	45.4	46.9	-3%

Removal efficiencies of the pharmaceutical compounds are lower than observed in most other biosolids treatment processes. None of the pharmaceuticals were removed at efficiencies greater than 50%. The highest removal efficiencies observed were for the antibiotics ciprofloxacin, norfloxacin and ofloxacin in the range of 39-46%. The highest negative removal efficiencies were calculated for the antibiotic sulfamethoxazole (-282%), the anti-anginal heart medication dehydronifedipine (-121-%) and the anti-epileptic carbamazepine (-108%).

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 60**. As noted above, there are no pharmaceutical compounds with calculated removal efficiencies greater than

Table 60. Categorised Removal Efficiencies of Pharmaceutical Compounds by Biosolids Thermal Drying Process, Smiths Falls, ON

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Caffeine	Gemfibrozil	Furosemide		
Carbamazepine	Ibuprofen	Triclocarban		
Dehydronifedipine	Naproxen	Triclosan		
Diltiazem	Azithromycin	Ciprofloxacin		
Sulfamethoxazole	Diphenhydramine	Clarithromycin		
	Erythromycin-H_2O	Enrofloxacin		
	Fluoxetine	Norfloxacin		
	Miconazole	Ofloxacin		
	Trimethoprim	Thiabendazole		
n=5	n=9	n=9	n=0	n=0

50% through the thermal drying process. The total number of pharmaceuticals with negative removal efficiencies (14) outnumbers the compounds with positive removal efficiencies (9). As with the metals, the mass balances indicate that in the majority of cases, the mass of the individual pharmaceutical compounds leaving the biosolids pellet drier are elevated compared to the mass of each compound in the feed dewatered cake. Assuming that in most cases the pharmaceutical mass is left unchanged through the drying process, the calculated increase in mass is likely due to inaccuracies or variations in the reported volumetric flow data.

4.7.4.4 Fragrance and Alkylphenolic Compounds Mass Balances

The mass balance and removal calculations for the fragrance and alkylphenolic compounds in the Smiths Falls heat drying system are provided in **Table 61**. Estimated removal efficiencies for the detected compounds ranged from a low of -12% for Bisphenol A to a high of 45% for the synthetic musk AHTN. The ability to assess the effectiveness of the heat drying system for removing alkylphenolic and fragrance compounds was hampered by the limited number of detectable concentrations.

4.7.4.5 Effectiveness of Process for ESOC Removal

The results indicate that the thermal drying process, as represented by the Smiths Falls data, provides only a modest barrier at best for reducing some concentrations of pharmaceutical and polycyclic fragrance compounds in feed sludge during biosolids treatment. More pharmaceutical compounds were associated with negative removal efficiencies through the drying process than were compounds with positive removal efficiencies. Bisphenol A exhibited no removal through the heat drying process.

Table 61. Mass Balance and Removal of Fragrance and Alkylphenolic Compounds by Biosolids Thermal Drying Process, Smiths Falls, ON

Compound	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/d)		% Removal
	Belt Pressed Cake	Heat Dried Pellets	Belt Pressed Cake	Heat Dried Pellets	
Alkylphenolics					
Bisphenol A	110	120	161	180	-12%
Fragrances					
HHCB	3990	2795	5851	4202	28%
AHTN	1540	830	2258	1248	45%
ATII	100	95	147	143	3%

4.7.5 Section Summary

Concentrations of nitrate-N, total Kjeldahl nitrogen (TKN) and soluble ortho-phosphate were all significantly lower in the heat dried biosolids pellets than in feed sludge (i.e. belt press dewatered cake), while ammonia-N was reduced to a lesser extent. The reduced concentration of TKN may be a result of destruction of the organic-N component of the TKN at the elevated drying temperature. The observed differences may also be due to some of the TKN components, including ammonia, amine and other organic nitrogen compounds, being driven off during high temperature drying process. The concentration of ortho-phosphate may be lower in the dried pellets compared to the feed sludge due to precipitation from reduced solubility at the elevated temperatures during drying.

All of the metals studied were identified above the detection limits in belt press cake, and all of the metals except cadmium were also detected in heat dried pellets. In general the concentrations of the metals in the feed dewatered sludge and dried pellets were similar. Zinc and copper were observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. Calculated mass balance for the metals indicate that in most cases, the mass of the individual metals leaving the biosolids pellet drier are slightly elevated compared to the mass of each in the feed dewatered cake. The calculated increase is likely due to minor inaccuracies or variations in the volumetric flow data. The exceptions cadmium and mercury exhibited a slight reduction in mass through the drying process; these two metals can be volatilized at elevated temperatures, and it is possible that the calculated reductions may be due to volatilization.

Removal efficiencies of the pharmaceutical compounds are lower than observed in most other biosolids treatment processes. None of the pharmaceuticals were removed at efficiencies greater than 50%. The highest removal efficiencies observed were for the antibiotics ciprofloxacin, norfloxacin and ofloxacin in the range of 39-46%. The highest negative removal efficiencies were calculated for the antibiotic sulfamethoxazole (-282%), the metabolite of anti-anginal heart medication dehydronifedipine (-121-%) and the anti-epileptic carbamazepine (-108%). The total number of pharmaceuticals with negative removal efficiencies (14) outnumbers the compounds with positive removal efficiencies (9). The mass balances indicate that in the majority of cases,

the mass of the individual pharmaceutical compounds leaving the biosolids pellet drier are elevated compared to the mass of each compound in the feed dewatered cake. Assuming that in most cases the pharmaceutical mass is let unchanged through the drying process, the calculated increase in mass is likely due to inaccuracies or variations in the reported volumetric flow data.

Bisphenol A was detected in the belt press filter cake and heat dried pellets in only one of the two sampling campaigns. Octylphenol and nonylphenol were not detected in any samples in the two sampling campaigns. The only synthetic musks detected in samples were HHCB, AHTN and ATII; these compounds were detected in both the feed press cake and dried pellets in both sampling campaigns. The concentrations of HHCB and AHTN were approximately an order of magnitude higher in concentration compared to ATII. Estimated removal efficiencies for the detected compounds ranged from a low of -12% for Bisphenol A to a high of 45% for the synthetic musk AHTN. The ability to assess the effectiveness of the heat drying system for removing alkylphenolic and fragrance compounds was hampered by the limited number of detectable concentrations.

The results indicate that the thermal drying process, as represented by the Smiths Falls data, provides only a modest barrier at best for reducing some concentrations of pharmaceutical and polycyclic fragrance compounds in feed sludge during biosolids treatment. More pharmaceutical compounds were associated with negative removal efficiencies through the drying process than were compounds with positive removal efficiencies. Bisphenol A exhibited no removal through the heat drying process.

4.8 Filter Press Dewatering of Septage, Gatineau Valley, QC

4.8.1 Site Description

The Gatineau Valley facility is a septage receiving and handling plant with treated effluent discharged to the Kazabazua River. The design capacity of the existing treatment plant is 85 m^3/d of septic sludge, while the average daily dry weather flow is 66 m^3/d.

4.8.2 Septage Dewatering Process Description

The facility treatment process is described as follows: septage is received, mixed and stored in bins. An estimated volume of 128 m^3/d of the mixed septage is then dewatered by rotary press. The dewatered cake is mixed with bulking agents (i.e. wood chips for dilution of cake) and the mixture is composted on a composting pad. Composting is done outside and turned on a regular basis. An average of 6.37 m^3/d of dewatered cake (called “dry mud” by facility staff), combined with 13.4 m^3/d wood chips is fed to the composting pad. The average production rate of compost is 19.1 m^3/d. The compost leachate (mainly because of rain) is also collected by the facility and a volume of 4500 m^3 was collected from April 27 to November 4, 2009, resulting in an average leachate flow of 25 m^3/d. During this study, the solids concentrations in the dry mud fed to the composting pad ranged from 35.3% to 39.5%, while the compost solids concentrations ranged from 41.3%to 47.1%, and the composting leachate solids concentrations were 0%.

The compost is moved from the pad every year. Due to delays in the use of the compost by a third party, all the compost is currently being stockpiled on the site since the first operation of the composting facility in 2005.

For this project assessment, the biosolids treatment processes of interest were biosolids dewatering and composting. The four sampling locations included the raw septage serving as feed to the dewatering rotary press, the dewatered cake (before wood chip addition) serving as feed to the composting pad, the compost product and rain water composting leachate. A process schematic of the Gatineau Valley biosolids treatment process is shown in **Figure 13**.

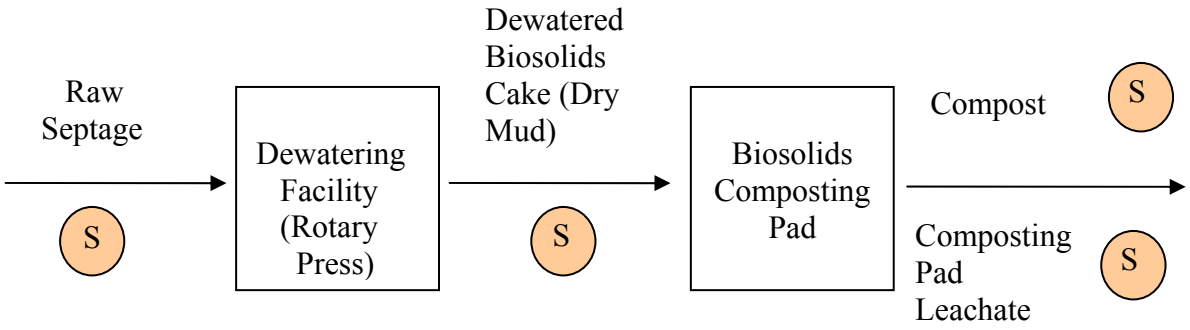

Figure 13. Schematic of Gatineau Valley Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on July 21, September 29 and October 27, respectively.

4.8.3 Sampling Results

4.8.3.1 Nutrients

Based on only the one set of nutrient concentration data from grab samples of septage and finished compost, interpretation of the data is difficult. Comparison of the concentrations is difficult because the concentration units are different for septage (mg/L) and compost (mg/kg). Some broad observations are possible, however. The effect of the aerobic composting of the dry mud is evident from the presence of nitrate-N in the compost but not in the septage (**Table 62**). The proportion of ammonia-N in the septage is much higher than in the compost, due to either loss of ammonia in the rotary press filtrate, volatilization of free ammonia from the rotary press or compost pile, the effect of the biological nitrification process, or leaching of ammonia-N by rainwater from the compost pad. The proportion of soluble ortho-phosphate, relative to total P, is much higher in the septage than in the finished compost. Potential causes of this transformation include precipitation of the soluble phosphate during the rotary press dewatering and composting processes, or leaching of ortho-phosphate by rainwater from the compost pad.

Table 62. Nutrients in Septage and Compost from Gatineau Valley, QC

Parameter	Concentration	
	Septage (mg/L)	Compost (mg/kg TS dw)
Nitrate-N	<1.0	88.0
Nitrite-N	<1.0	<1.0
Total Kjeldahl Nitrogen	545	17100
Ammonia as N	159	540
Phosphorus, Total	92.7	4120
Phosphate-P (ortho)	24.0	5.21
Total Solids	19100	420000

4.8.3.2 Metals

The first sampling round was used to provide the samples used for metal analysis. Due to a period of dry weather at the Gatineau Valley site when the first sampling campaign was completed, no sample of leachate was submitted with the raw septage and finished compost. All of the target metals were detected in the finished compost, while only half as many were observed above the detection limit in the raw septage (**Table 63**). Zinc and copper were observed at the highest concentrations of any metals in both the raw septage and finished compost. Mercury was the metal with the lowest detected concentration. Additional discussion of the metals data is found later in this section under Data Interpretation.

Table 63. Metals in Septage and Compost from Gatineau Valley, QC

Parameter	Concentration	
	Septage (mg/L)	Compost (mg/kg TS dw)
Arsenic (As)-Total	<0.10	**1.4**
Cadmium (Cd)-Total	**0.014**	**1.2**
Chromium (Cr)-Total	**0.14**	**15.4**
Cobalt (Co)-Total	<0.080	**3.4**
Copper (Cu)-Total	**5.46**	**224**
Lead (Pb)-Total	**0.28**	**22.5**
Mercury (Hg)-Total	**0.00338**	**0.686**
Molybdenum (Mo)-Total	<0.10	**1.8**
Nickel (Ni)-Total	<0.20	**11.4**
Selenium (Se)-Total	<0.50	**2.2**
Zinc (Zn)-Total	**11.5**	**475**
Total Solids	19100	420000

Samples in **bold** font are above the detection limit

4.8.3.3 Pharmaceuticals

The frequency of detection and median and range of detected concentrations of pharmaceutical compounds in the composting feed sludge (i.e. termed “dry mud” by the plant staff) the finished

compost and pad leachate at the Gatineau Valley facility are presented in **Table 64**. The raw analytical data for the three sampling campaigns are provided in **Appendix Table A13**.

Only two compounds are detected at concentrations above 1000 ng/g TS dw in the compost. The median concentration of the non-steroidal anti-inflammatory naproxen is 9890 ng/g TS, much higher than in the dry mud input to the composting process. The median concentration of the antibiotic ciprofloxacin is 1058 ng/g TS. Compounds such as triclosan, triclocarban and caffeine are greatly reduced in concentration in the finished compost compared to levels in the dry mud.

The distribution of detectable concentrations in the dry mud, compost and rain water composting leachate from all the sampling campaigns is found in **Table 65**. There appears to be a significant shift in the distribution of detectable concentrations in the dry mud, compost and composting pad leachate samples. Most notably the number of compounds detected in all three campaigns declines from 18 in the dry mud to 6 in compost, and the number of compounds not detected in any of the 3 sampling campaigns increases from 29 in the dry mud to 45 in the finished compost. A total of 18 compounds were detected in both samples of the composting pad leachate, the same number as detected in all three samples of dry mud.

Table 64. Frequency of Detection, Median and Range of Detected Concentrations of Pharmaceutical Compounds in Dry Mud, Compost and Rain Water Composting Leachate Samples from Gatineau Valley, QC

Pharmaceutical	Frequency of Detection in Sampling Campaigns			Median of Detected Concentration			Range of Detected Concentration		
	Dry Mud	Compost	Compost leachate [b]	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Compost leachate (ng/L) [b]	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Compost leachate (ng/L) [b]
Furosemide	2	0	0	539	NA	NA	<77.9-760	<425 [c]	<379 [c]
Gemfibrozil	2	0	**2**	35.1	NA	**41.3**	<2.83-66.2	<15.9 [c]	37.2-45.3
Glipizide	0	0	0	NA	NA	NA	<52.4 [c]	<63.7 [c]	<6.42 [c]
Glyburide	**3**	0	**2**	**70.9**	NA	**31.6**	27.4-801	<31.9 [c]	30.8-32.4
Hydrochlorothiazide	**3**	0	0	**235**	NA	NA	133-532	<212 [c]	<21.4 [c]
2-Hydroxy-ibuprofen	1	0	**2**	171 [a]	NA	**388**	<151-171	<849 [c]	339-437
Ibuprofen	**3**	1	**2**	**432**	29.2 [a]	**616**	266-433	<25.4-29.2	590-642
Naproxen	**3**	**3**	**2**	**133**	**9890**	**127**	50.7-665	2360-10800	76.2-177
Triclocarban	**3**	**3**	**2**	**10000**	**784**	**16.7**	2450-20700	529-2120	16.5-16.8
Triclosan	**3**	2	**2**	**38600**	781.5	**129**	27600-46400	<102-918	121-136
Warfarin	0	0	0	NA	NA	NA	<13.1 [c]	<15.9 [c]	<1.6 [c]
Acetaminophen	0	0	0	NA	NA	NA	<524 [c]	<637 [c]	<118 [c]
Azithromycin	**3**	1	**2**	**315**	10.8 [a]	**10.3**	250-694	<2.75-10.8	9.11-11.4
Caffeine	**3**	0	**2**	**1090**	<28.8	**431**	910-1240	<159 [c]	332-529
Carbadox	0	0	0	NA	NA	NA	<13.1 [c]	<15.9 [c]	<1.6 [c]
Carbamazepine	**3**	**3**	**2**	**53**	**40.2**	**561**	12.1-291	31-45.9	395-727
Cefotaxime	0	0	0	NA	NA	NA	<341 [c]	<142 [c]	<137 [c]
Ciprofloxacin	**3**	2	**2**	**13900**	1059	**75.9**	11800-18100	928-1190	58.1-93.7
Clarithromycin	**3**	0	**2**	**146**	<2.88	**11.6**	135-353	<15.9 [c]	5.34-17.8
Clinafloxacin	0	0	0	NA	NA	NA	<52.4 [c]	<135 [c]	<138 [c]
Cloxacillin	0	0	0	NA	NA	NA	<26.2 [c]	<31.8 [c]	<6.16 [c]
Dehydronifedipine	**3**	**3**	**2**	**15.6**	**8.0**	**47.6**	7.33-17.1	6.09-18.5	45.2-49.9
Diphenhydramine	**3**	**3**	**2**	**778**	**47.7**	**32.7**	652-1070	42.3-80	27.3-38
Diltiazem	**3**	0	**2**	**47.6**	NA	**9.7**	29.2-81.9	<3.18 [c]	7.63-11.7
Digoxin	0	0	0	NA	NA	NA	<149 [c]	<159 [c]	<16 [c]
Digoxigenin	1	0	0	15.6 [a]	NA	NA	<13.9-15.6	<87 [c]	<335 [c]
Enrofloxacin	1	0	0	35.3 [a]	NA	NA	<5.71-35.3	<31.8 [c]	<8.28 [c]
(continued)									

Table 64 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns			Median of Detected Concentration			Range of Detected Concentration		
	Dry Mud	Compost	Compost leachate [b]	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Compost leachate (ng/L) [b]	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Compost leachate (ng/L) [b]
Erythromycin-H_2O	**3**	0	**2**	**6.4**	NA	**7.3**	2.75-14.3	<3.18 [c]	7.03-7.6
Flumequine	0	0	0	NA	NA	NA	<13.1 [c]	<15.9 [c]	<7.22 [c]
Fluoxetine	2	2	0	67.1	9.9	NA	<2.83-95	<2.75-11.2	<1.6 [c]
Lincomycin	0	0	0	NA	NA	NA	<26.2 [c]	<31.8 [c]	<16.8 [c]
Lomefloxacin	0	0	0	NA	NA	NA	<26.2 [c]	<31.8 [c]	<3.21 [c]
Miconazole	2	**3**	0	786	**62.6**	NA	<2.87-1040	54.2-109	<6.67 [c]
Norfloxacin	**3**	0	0	**119**	NA	NA	53.7-212	<159 [c]	<16 [c]
Norgestimate	0	0	0	NA	NA	NA	<33 [c]	<31.8 [c]	<3.21 [c]
Ofloxacin	**3**	1	0	**299**	288 [a]	<15.8	185-402	<0-288	<16 [c]
Ormetoprim	0	0	0	NA	NA	NA	<5.24 [c]	<6.37 [c]	<0.64 [c]
Oxacillin	0	0	1	NA	NA	8.8 [a]	<26.2 [c]	<31.8 [c]	<3.21-8.84
Oxolinic Acid	0	0	0	NA	NA	NA	<5.24 [c]	<6.37 [c]	<10.6 [c]
Penicillin G	0	0	0	NA	NA	NA	<26.2 [c]	<31.8 [c]	<1.28 [c]
Penicillin V	0	0	0	NA	NA	NA	<26.2 [c]	<31.8 [c]	<13.2 [c]
Roxithromycin	0	0	0	NA	NA	NA	<2.62 [c]	<3.18 [c]	<0.32 [c]
Sarafloxacin	0	0	0	NA	NA	NA	<142 [c]	<311 [c]	<16 [c]
Sulfachloropyridazine	0	0	1	NA	NA	31.1 [a]	<13.1 [c]	<15.9 [c]	<1.6-31.1
Sulfadiazine	0	0	0	NA	NA	NA	<13.1 [c]	<15.9 [c]	<1.6 [c]
Sulfadimethoxine	0	0	0	NA	NA	NA	<2.62 [c]	<3.18 [c]	<0.32 [c]
Sulfamerazine	0	0	0	NA	NA	NA	<5.24 [c]	<6.37 [c]	<5.05 [c]
Sulfamethazine	0	0	0	NA	NA	NA	<7.25 [c]	<6.37 [c]	<17.9 [c]
Sulfamethizole	0	0	0	NA	NA	NA	<8.42 [c]	<6.37 [c]	<3.67 [c]
Sulfamethoxazole	1	0	0	7.8 [a]	NA	NA	<1.13-7.75	<6.37 [c]	<0.64 [c]
Sulfanilamide	0	0	0	NA	NA	NA	<131 [c]	<159 [c]	<16 [c]
Sulfathiazole	0	0	0	NA	NA	NA	<13.1 [c]	<15.9 [c]	<4.58 [c]
Thiabendazole	**3**	0	**2**	**62.7**	NA	**6.0**	29.7-65.2	<15.9 [c]	4.25-7.66
Trimethoprim	2	0	0	71.2	NA	NA	<2.83-78.4	<15.9 [c]	<70.6 [c]

(continued)

Table 64 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns			Median of Detected Concentration			Range of Detected Concentration		
	Dry Mud	Compost	Compost leachate [b]	Dry Mud (ng/g TS)	Compost (ng/g TS)	Compost leachate (ng/L) [b]	Dry Mud (ng/g TS)	Compost (ng/g TS)	Compost leachate (ng/L) [b]
Tylosin	0	0	0	NA	NA	NA	<80.4 [c]	<82.3 [c]	<21.4 [c]
Virginiamycin	0	0	0	NA	NA	NA	<67.8 [c]	<65.3 [c]	<81.9 [c]
1,7-Dimethylxanthine	1	0	**2**	645 [a]	NA	**550**	<283-645	<1590 [c]	508-591

[a] indicates median value is from one detectable concentration only; [b] only two round samplings for composting leachate

[c] indicates highest identified detection limit for compound

Data in **bold** font are detected in all sampling campaigns; NA = not applicable (no median for all non-detectable concentrations)

Table 65. Summary of Pharmaceutical Compound Detections in Dry Mud, Compost and Composting Pad Leachate Samples, Gatineau Valley, QC

Frequency of detection in sampling campaigns (out of 3[a])	# Compounds in Process Streams		
	Dry Mud	Compost	Compost Pad Leachate
3	18	6	NA
2	5	3	18
1	5	3	2
0	29	45	37
Total	57	57	57

[a] There are only two (instead of three) samples of composting pad leachate
NA = not applicable

4.8.3.4 Fragrance and Alkylphenolic Compounds

The assessment of concentration data for the two sampling campaigns for alkylphenolic and fragrance compounds is provided in **Table 66**. The raw analytical data are provided in **Appendix Table A14**. Bisphenol A was detected in both samples of dry mud and composting pad leachate, but in only one of the two compost samples. Octylphenol and nonylphenol were non-detected except for a low concentration of octylphenol in one leachate sample. The fragrance compounds HHCB and AHTN were found at levels approaching or above 1000 ng/g TS dw in the compost, and at higher levels in the dry mud prior to composting. The fragrance compounds DPMI, ADBI and AHDI were generally detected at low concentrations in only one of the two campaigns in the different matrices.

4.8.4 Data Interpretation

4.8.4.1 Metals Mass Balances

The mass balance for metals was based on a sample of the septage as received, and the final compost. Concentrations in the septage, which were reported on a volumetric basis (i.e., mg/L), are multiplied by the estimated septage feed rate to the rotary press to obtain the mass in the dry mud. Because no intermediate sample of the dewatered septage was analysed for metals, it was assumed for the mass balance that the metals in the septage are all retained in the dry mud. Concentrations of metals in the compost are reported on a dry weight basis (i.e., ng/g TS), so the mass of metals is estimated by multiplying the concentrations by the compost production rate of 3.3 m^3/d after deducting the wood chip mass.

The mass balances for the metals are provided in **Table 67**. The mass closures for the metals range from a low of 46% for copper to a high of 230% for mercury. With a median value of 94% for the mass closures, it appears that the assumption that the mass of metals in the septage was carried through entirely in the dry mud is acceptable.

Table 66. Frequency of Detection, Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Dry Mud, Compost and Composting Pad Leachate Samples, Gatineau Valley, QC

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)			Median of Detected Concentration			Range of Detected Concentration		
	Dry Mud	Compost	Pad Leachate	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Pad Leachate (ng/L)	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Pad Leachate (ng/L)
Alkylphenolics									
Bisphenol A	**2**	1	**2**	**150**	70	**200**	120-180	<80-70	130-270
Octylphenol	0	0	1	NA	NA	30	<20	<20	<0-30
Nonylphenol	0	0	0	NA	NA	NA	<140	<140	<90
Fragrances									
DPMI	1	1	1	30	40	30	<40-30	<40-40	<30-30
ADBI	1	0	0	90	NA	NA	<20-90	<20	<90
AHDI	1	1	0	80	40	NA	<30-80	<30-40	<80
HHCB	**2**	**2**	**2**	**6525**	**2215**	**75**	2530-10520	1270-3160	70-80
AHTN	**2**	**2**	**2**	**2135**	**925**	**240**	570-3700	800-1050	230-250
ATII	**2**	**2**	**2**	**190**	**120**	**15**	40-340	120-120	10-20
Musk Moskene	0	0	0	NA	NA	NA	<50	<50	<90
Musk Tibetene	0	0	0	NA	NA	NA	<80	<80	<50
Musk Ketone	0	0	0	NA	NA	NA	<120	<120	<90
Musk Ambrette	0	0	0	NA	NA	NA	<140	<140	<20
Musk Xylene	0	0	1	NA	NA	40	<70	<70	<20-40

Data in **bold font** are detected in both sampling campaigns

Table 67. Mass Balance Closure for Metals in Septage Composting Process, Gatineau Valley, QC

Metal	Concentration		Mass of Metal (g/d)		Mass Closure (%)
	Septage (mg/L)	Compost (mg/kg TS dw)	Septage	Compost	
Arsenic (As)-Total	<0.10	1.4	NA[a]	2.04	NA
Cadmium (Cd)-Total	0.014	1.2	1.80	1.74	97
Chromium (Cr)-Total	0.14	15.4	18.0	22.4	125
Cobalt (Co)-Total	<0.080	3.4	NA	4.94	NA
Copper (Cu)-Total	5.46	224	700	326	46
Lead (Pb)-Total	0.28	22.5	35.9	32.7	91
Mercury (Hg)-Total	0.0034	0.686	0.43	1.00	230
Molybdenum (Mo)-Total	<0.10	1.8	NA	2.62	NA
Nickel (Ni)-Total	<0.20	11.4	NA	16.6	NA
Selenium (Se)-Total	<0.50	2.2	NA	3.20	NA
Zinc (Zn)-Total	11.5	475	1475	691	47

[a] NA = not applicable, cannot be determined

4.8.4.2 Pharmaceutical Compounds Mass Balances

Because the pharmaceutical compounds in the dry mud and compost samples were measured on a dry weight basis, a solids balance was completed for estimation of the pharmaceutical mass balances through the septage composting process at the Gatineau Valley facility. The information used for the solids balance was
Dry mud flow rate: 6.37 m^3/d
Dry mud total solids concentration: 388.5 kg/m^3
Compost flow rate: 3.30 m^3/d
Compost solids concentration: 441 kg/m^3
Pad leachate flow rate; 25 m^3/d

In addition to the data provided above, assumptions based on engineering judgement that were used to close the solids balance included:
Volatile fraction of the dry mud: 75%
Volatile solids reduction through the composting process: 55%

The solids mass balance is depicted in **Figure 14**.

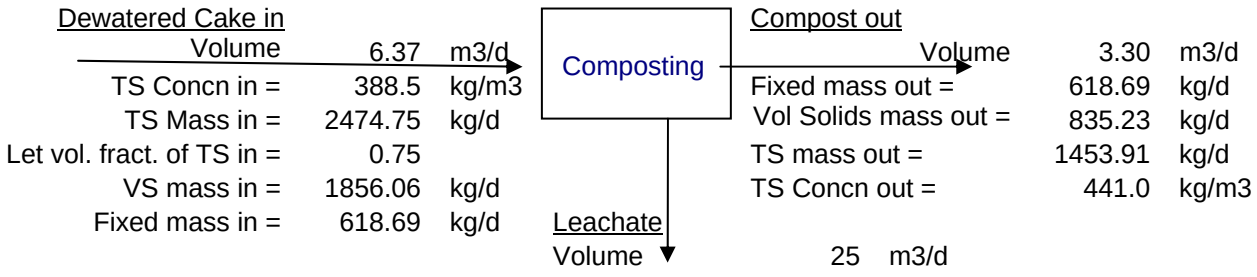

Figure 14. Solids Mass Balance around Septage Composting Process, Gatineau Valley, QC

The mass balances for pharmaceutical compounds in the composting process are presented in **Table 68.** Many of the pharmaceutical compounds detected in the dry mud prior to composting are significantly removed during the composting process. Removal efficiencies of greater than 90% are seen with a large number of the compounds. The only pharmaceutical with a large negative removal is the non-steroidal anti-inflammatory compound naproxen, with a calculated negative removal of -4270% due to a high concentrations reported in the compost. Only a few other compounds were not removed to a great extent, including carbamazepine (44.7%) and the antibiotic ofloxacin.

Table 68. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Septage Composting Process, Gatineau Valley, QC

Pharmaceutical	Concentrations (median)			Mass of Contaminants (mg/d)			% Removal
	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Compost Pad Leachate (ng/L)	Dry Mud	Compost	Compost Pad Leachate	
Gemfibrozil	35.1	<2.88	41.3	86.8	<4.03	1.03	>94.2%-98.8%
Glyburide	70.9	<5.56	31.6	175	<8.08	0.79	>94.9%-99.5%
2-Hydroxy-ibuprofen	171	<149	388	423	<216	9.7	>46.7%-97.7%
Ibuprofen	432	29.2	616	1,069	42.5	15.4	94.6%
Naproxen	133	9890	126.6	329	14,380	3.17	-4270%
Triclocarban	10000	784	16.7	24,750	1,140	0.42	95.4%
Triclosan	38600	782	129	95,530	1,136	3.2	98.8%
Azithromycin	315	10.8	10.3	780	15.7	0.26	98.0%
Caffeine	1090	<28.8	431	2,697	<41.9	10.8	>98.0%-99.6%
Carbamazepine	53	40.2	561	131	58.4	14.0	44.7%
Ciprofloxacin	13900	1059	75.9	34,400	1,540	1.9	95.5%
Clarithromycin	146	<2.88	11.6	361	<212	0.29	>41.2%-99.9%
Dehydro-nifedipine	15.6	8.0	47.6	38.6	11.6	1.2	66.8%
Diphen-hydramine	778	47.7	32.7	1,925	69.4	0.82	96.4%
Diltiazem	47.6	<0.577	9.7	118	<0.84	0.24	>99.1-99.8%
Erythromycin-H_2O	6.4	<0.577	7.3	15.7	<10.6	0.18	>31.2%-98.8%
Fluoxetine	67.1	9.9	<1.6	166	14.4	<0.040	91.3%
Miconazole	786	62.6	<6.6	1,944	91.0	<0.17	95.3%
Ofloxacin	299	288	<15.8	740	419	<0.40	43.4%
Thiabendazole	62.7	<2.88	6.0	155	<4.2	0.15	>97.2%-99.9%
1,7-Dimethy-lxanthine	645	<288	550	1,596	<40.7	13.7	>96.6%-99.1%

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 69**. The table clearly depicts that the great majority of the removal efficiencies of the pharmaceutical compounds are high, i.e., greater than 90%. Lower removal efficiencies (less than 50% removal) were only identified for naproxen, carbamazepine and ofloxacin. In addition to these calculated removal efficiencies, an additional three compounds had calculated removal efficiency ranges that were potentially greater than 90%, but could not be determined due to their having non-detectable concentrations in the finished compost. The metabolite 2-hydroxy-ibuprofen, and the antibiotics clarithromycin and erythromycin-H_20 had minimum removal efficiencies greater than 31% but potentially as high as 98-99%.

Table 69. Categorised Removal Efficiencies of Pharmaceutical Compounds by Septage Composting Process, Gatineau Valley, QC

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Naproxen		Carbamazepine	Dehydronifedipine	Gemfibrozil
		Ofloxacin		Glyburide
				Ibuprofen
				Triclocarban
				Triclosan
				Azithromycin
				Caffeine
				Ciprofloxacin
				Diphenhydramine
				Diltiazem
				Fluoxetine
				Miconazole
				Thiabendazole
				1,7-Dimethylxanthine
n=1	n=0	n=2	n=1	n=14

4.8.4.3 Fragrance and Alkylphenolic Compounds Mass Balances

The calculated mass balances and removals of the fragrance and alkylphenolic compounds are provided in **Table 70**. Very little of the mass of the compounds from the dry mud is recovered in the pad leachate. Although most of the recovered mass of the compounds resides in the compost, for most of the fragrances (except DPMI) and Bisphenol A, the majority of the incoming contaminant masses in the dry mud have been removed, presumably by biodegradation.

4.8.4.4 Effectiveness of Process for ESOC Removal

The composting process as represented by the Gatineau Valley operation provided evidence of very high removal efficiencies of the pharmaceutical compounds, and substantial removal of most polycyclic fragrances and Bisphenol A. The results may be a result of prolonged storage periods of a year or more which can allow the opportunity of leaching by rainwater, or continued

biodegradation due to the long exposure time. In any event, the results obtained for the septage composting are among the most favourable observed for reducing pharmaceutical and fragrance compounds, and Bisphenol A, in septage or raw sludge.

Table 70. Mass Balance and Removal Calculations for Alkylphenolic and Fragrance Compounds in Septage Composting Process, Gatineau Valley, QC

Compound	Concentrations (median)			Mass of Contaminants (mg/d)			% Removal
	Dry Mud (ng/g TS dw)	Compost (ng/g TS dw)	Pad Leachate (ng/L)	Dry Mud	Compost	Pad Leachate	
Alkylphenolics							
Bisphenol A	150	70	200	371	102	5.0	71%
Fragrances							
DPMI	30	40	30	74.2	58.2	0.8	21%
AHDI	80	40	<80	198	58.2	<2.0	>70%
HHCB	6525	2215	75	16,150	3,220	1.9	80%
AHTN	2135	925	240	5,284	1,345	6.0	74%
ATII	190	120	15	470	174	0.4	63%

4.8.5 Section Summary

Interpretation of the nutrient data is difficult, because only the one set of nutrient data from grab samples of septage and finished compost was collected, and different concentration units were used for septage (mg/L) and compost (mg/kg TS dw). Some broad observations are possible, however. The effect of the aerobic composting of the dry mud is evident from the presence of nitrate-N in the compost but not in the septage. The proportion of ammonia-N in the septage is much higher than in the compost. The proportion of soluble ortho-phosphate, relative to total P, is also much higher in the septage than in the finished compost.

The first sampling round was used to provide the samples used for metal analysis. Due to a period of dry weather at the Gatineau Valley site when the first sampling campaign was completed, no sample of leachate was submitted with the raw septage and finished compost. All of the target metals were detected in the finished compost, while only half as many were observed above the detection limit in the raw septage. Zinc and copper were observed at the highest concentrations of any metals in both the raw septage and finished compost. Mercury was the metal with the lowest detected concentration. The mass balance for metals was based on a sample of the septage as received, and the final compost. Because no intermediate sample of the dewatered septage was analysed for metals, it was assumed for the mass balance that the metals in the septage are all retained in the dry mud. The mass closures for the metals range from a low of 46% for copper to a high of 230% for mercury. With a median value of 94% for the mass closures, it appears that the assumption that the mass of metals in the septage was carried through entirely in the dry mud is acceptable.

Only two pharmaceutical compounds are detected at concentrations above 1000 ng/g TS in the compost. The median concentration of the non-steroidal anti-inflammatory naproxen is 9890 ng/g TS, much higher than in the dry mud input to the composting process. The median concentration of the antibiotic ciprofloxacin is 1058 ng/g TS. Compounds such as triclosan, triclocarban and caffeine are greatly reduced in concentration in the finished compost compared to levels in the dry mud. There appears to be a significant shift in the distribution of detectable concentrations in the dry mud, compost and rain water composting pad samples. Most notably the number of compounds detected in all three campaigns declines from 18 in the dry mud to 6 in compost, and the number of compounds not detected in any of the 3 sampling campaigns increases from 29 in the dry mud to 45 in the finished compost. A total of 18 compounds were detected in both samples of the composting pad leachate, the same number as detected in all three samples of dry mud. The great majority of the removal efficiencies of the pharmaceutical compounds are high, i.e., greater than 90%. Lower removal efficiencies (less than 50% removal) were only identified for naproxen, carbamazepine and ofloxacin. In addition to these calculated removal efficiencies, an additional three compounds had calculated removal efficiency ranges that were potentially greater than 90%, but could not be determined due to their having non-detectable concentrations in the finished compost.

Bisphenol A was detected in both samples of dry mud and composting pad leachate, but in only one of the two compost samples. Octylphenol and nonylphenol were non-detected except for a low concentration of octylphenol in one leachate sample. The fragrance compounds HHCB and AHTN were found at levels approaching or above 1000 ng/g TS dw in the compost, and at higher levels in the dry mud prior to composting. The fragrance compounds DPMI, ADBI and AHDI were generally detected at low concentrations in only one of the two campaigns in the different matrices. Very little of the mass of the compounds from the dry mud is recovered in the pad leachate. Although most of the recovered mass of the compounds resides in the compost, for most of the fragrances (except DPMI) and Bisphenol A, the majority of the incoming contaminant masses in the dry mud have been removed, presumably by biodegradation.

The composting process as represented by the Gatineau Valley operation provided evidence of very high removal efficiencies of the pharmaceutical compounds. The results may be a result of prolonged storage periods which can allow the opportunity of leaching by rainwater, or continued biodegradation due to the long exposure time. In any event, the results obtained for the septage composting are among the most favourable observed for reducing pharmaceutical compounds in septage or raw sludge.

4.9 Filter Press Sludge Dewatering, Saguenay, QC

4.9.1 Site Description

The Saguenay Water Pollution Control Plant (WPCP) is an extended aeration treatment facility with the treated effluent discharged to the Saguenay River. The design capacity of the existing treatment plant is 72,000 m^3/d, while the average daily dry weather flow is 40,000 m^3/d.

4.9.2 Filter Press Dewatering Process Description

The waste activated sludge (WAS) from the secondary clarifiers is gravity thickened, stored in tanks and then sent to three filter presses for dewatering. The plant does not stabilise the WAS by either aerobic or anaerobic sludge digestion. The flow of feed sludge (thickened WAS) to the filter presses is 120 m^3/d (40 m^3/d per filter). During this study, the solids concentration in the feed sludge to the dewatering filter ranged from 4.1% to 8.1%, while the dewatered cake solids concentration ranged from 12% to 13.7%. The filtrate solids concentration ranged from non-detectable to 0.02%.

The dewatered cake is directly sent to agricultural lands without composting from March to October. Between November to February, it is taken to a site for composting by a private firm. Overall about 70% of the dewatered cake is land applied without composting and 30% are sent off-site for composting.

For this project assessment, the solids treatment process of interest is the filter press dewatering unit. The three sampling locations included the filter feed liquid sludge (i.e. thickened WAS), dewatered cake and press filtrate. A process schematic of the Saguenay dewatering process is shown in **Figure 15**.

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on July 8, July 22 and August 27, respectively.

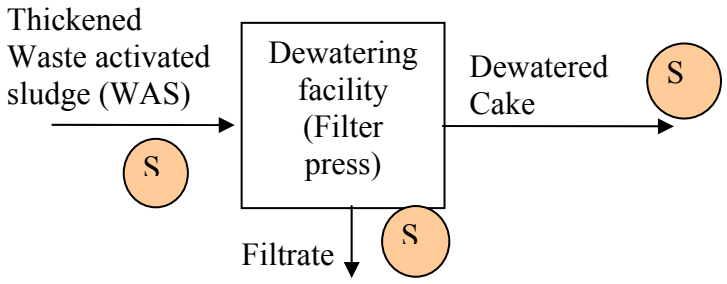

Figure 15. Schematic of Saguenay Biosolids Process and Sampling Locations

4.9.3 Sampling Results

4.9.3.1 Nutrients

Nitrogen was present in the thickened WAS feed mostly as the organic nitrogen component of TKN, as shown in **Table 71**. The ammonia concentration was substantially higher than the oxidised forms of nitrogen (nitrate and nitrite), which were less than the detection limit. Similar trends were observed in the dewatered cake and press filtrate. Soluble ortho-phosphate contributes only a very small amount to the total P concentration in the thickened WAS. With the exception of ortho-phosphate, other nutrient forms are significantly reduced in the press filtrate, leading to the conclusion that the nutrients are concentrated in the dewatered cake. Direct

comparison with the cake nutrient concentrations is not possible because they are measured differently and expressed on a dry weight basis.

Table 71. Nutrients in Filter Press Feed Sludge, Dewatered Cake and Filtrate, Saguenay, QC

Parameter	Concentration		
	Feed Sludge (mg/L)	Dewatered Cake (mg/kg TS dw)	Filtrate (mg/L)
Nitrate-N	<2.0	18.7	<0.30
Nitrite-N	<2.0	<1.0	<0.30
Total Kjeldahl Nitrogen	1300	59500	23.1
Ammonia as N	115	5060	6.09
Phosphorus, Total	449	17700	9.60
Phosphate-P (ortho)	0.268	11.3	0.169
Total Solids	22100	125000[a]	590

[a] The concentration unit of total solids is mg/L.

4.9.3.2 Metals

Concentrations of metals in the filter feed sludge and filtrate samples were analyzed as liquid matrices, with results reported in units of mg/L. Concentrations of metals in the dewatered cake were reported on a dry weight basis (mg/kg TS dw). As shown in **Table 72**, most target metals (except for arsenic, molybdenum and selenium) are detected in the thickened WAS feed to the filter press, and all target metals except for cadmium are detected in the dewatered cake. Few metals were identified above the detection limits in the press filtrate, except for chromium, copper, mercury and zinc. Zinc and copper were observed at the highest detected concentrations in the three sample matrices. Cadmium was the metal with the lowest detected concentration in the feed sludge, while in the dewatered cake and filtrate, mercury was the metal with the lowest detected concentrations. Additional discussion of the metals is found later in this section under Interpretation of data.

4.9.3.3 Pharmaceuticals

The frequency of detection and median detected concentrations of the pharmaceutical compounds in the thickened WAS sludge feed, the dewatered cake and filtrate samples at the Saguenay facility are presented in **Table 73**. Raw analytical data are provided in **Appendix Table A15**. In total, 18 pharmaceuticals were detected in the filter feed sludge from both sampling campaigns (there are only two instead of three rounds of sampling for this sample); 16 compounds were detected in the dewatered biosolids samples from all the three campaigns and 17 compounds were detected in the filtrate samples from all the three campaigns. Triclosan and triclocarban (antimicrobials), and the antibiotic ciprofloxacin were found at the highest concentrations in the dewatered cake. A number of compounds were detected in the press filtrate in all three sampling campaigns, but were not detected in the feed sludge or dewatered cake in any of the campaigns. These included gemfibrozil (blood lipid regulator), glyburide (anti-diabetic), hydrochlorothiazide (diuretic), dehydronifedipine (anti-anginal), and penicillin G (antibiotic).

Table 72 Metals in Filter Feed Sludge (Thickened WAS), Dewatered Cake and Filtrate, Saguenay, QC

Parameter	Concentration		
	Filter Feed Sludge (mg/L)	Dewatered Cake (mg/kg TS dw)	Filtrate (mg/L)
Arsenic (As)-Total	<0.10	**1.3**	<0.010
Cadmium (Cd)-Total	**0.014**	<1.0	<0.0010
Chromium (Cr)-Total	**1.24**	**24.5**	**0.010**
Cobalt (Co)-Total	**0.109**	**2.6**	<0.0080
Copper (Cu)-Total	**5.23**	**107**	**0.047**
Lead (Pb)-Total	**0.90**	**14.9**	<0.010
Mercury (Hg)-Total	**0.0234**	**0.677**	**0.00066**
Molybdenum (Mo)-Total	<0.10	**1.8**	<0.010
Nickel (Ni)-Total	**0.48**	**10.5**	<0.020
Selenium (Se)-Total	<0.50	**1.3**	<0.050
Zinc (Zn)-Total	**7.54**	**159**	**0.069**
Total Solids	22100	125000[a]	590

[a] The concentration unit of total solids is mg/L; Samples in **bold** font are above the detection limit

The distribution of detectable concentrations in the filter press feed sludge, dewatered cake and filtrate streams from the three sampling campaigns is found in **Table 74**. The distribution appears to be relatively stable in all three process streams, based on the number of compounds detected in all campaigns (2 for the feed sludge; 3 for the dewatered cake and filtrate), and the number of compounds never detected in any of the sampling campaign. For example the number of compounds detected in all sampling campaigns were 18 in the thickened WAS feed, 16 in the dewatered cake and 17 in the filtrate. The number of compounds never detected in any of the campaigns was 36 for the filter feed sludge, 37 for the dewatered cake and 33 for the filtrate. As indicated above, however, specific compounds within each process stream may vary, as a number of compounds were only detected in the filtrate and not in the feed sludge or dewatered cake in any of the campaigns.

Table 73. Frequency of Detection and Median and Range of Detected Concentrations of Pharmaceutical Compounds in Filter Feed Sludge (Thickened WAS), Dewatered Cake and Press Filtrate, Saguenay, QC

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Filter Feed Sludge [b]	Dewatered Cake	Filtrate	Filter Feed Sludge (ng/g TS dw)	Dewatered Cake (ng/g TS dw)	Filtrate (ng/L)	Filter Feed Sludge (ng/g TS dw)	Dewatered Cake (ng/g TS dw)	Filtrate (ng/L)
Furosemide	1	1	1	139 [a]	165 [a]	402 [a]	<109-139	<164-165	<113-402
Gemfibrozil	0	0	**3**	NA	NA	**5.85**	<7.18 [c]	<15.1 [c]	4.49-5.95
Glipizide	0	0	0	NA	NA	NA	<28.7 [c]	<60.3 [c]	<7.71 [c]
Glyburide	0	0	**3**	NA	NA	**12.5**	<14.4 [c]	<30.2 [c]	4.77-13.8
Hydrochlorothiazide	0	0	**3**	NA	NA	**461**	<95.8 [c]	<201 [c]	223-677
2-Hydroxy-ibuprofen	0	0	1	NA	NA	147 [a]	<529 [c]	<805 [c]	<103-147
Ibuprofen	1	0	**3**	47.8 [a]	NA	**190**	<40.8-47.8	<151 [c]	130-204
Naproxen	**2**	**3**	**3**	**18.4**	**83.8**	**132**	15.6-21.2	18.5-84.5	121-145
Triclocarban	**2**	**3**	**3**	**1875**	**1660**	**8.9**	1830-1920	1580-2030	8.41-11.1
Triclosan	**2**	**3**	1	**1852**	**1310**	84	963-2740	923-2820	<59.9-84
Warfarin	0	0	1	NA	NA	1.59	<7.18 [c]	<15.1 [c]	<1.5-1.59
Acetaminophen	0	0	0	NA	NA	NA	<287 [c]	<604 [c]	<77.1 [c]
Azithromycin	**2**	**3**	**3**	**334**	**262**	**162**	282-385	185-390	70.3-235
Caffeine	1	2	0	83 [a]	291	NA	<40.8-83	<61.5-387	<19.3 [c]
Carbadox	0	0	0	NA	NA	NA	<7.18 [c]	<15.1 [c]	<3.19 [c]
Carbamazepine	**2**	**3**	**3**	**50.5**	**51.6**	**345**	33.8-67.1	34.6-252	338-595
Cefotaxime	0	0	0	NA	NA	NA	<266 [c]	<183 [c]	<27.6 [c]
Ciprofloxacin	**2**	**3**	2	**7835**	**6440**	54.2	7000-8670	4840-7150	<12.3-83.1
Clarithromycin	**2**	**3**	**3**	**50.1**	**69.1**	**27.4**	43-57.1	47.3-72.2	23-70.1
Clinafloxacin	0	0	0	NA	NA	NA	<28.7 [c]	<60.4 [c]	<65.6 [c]
Cloxacillin	0	0	0	NA	NA	NA	<14.4 [c]	<30.2 [c]	<4.79 [c]
Dehydronifedipine	0	0	**3**	NA	NA	**2.93**	<2.87 [c]	<6.04 [c]	2.22-3.66
Diphenhydramine	**2**	**3**	**3**	**465**	**420**	**88.1**	443-487	349-607	72.6-150
Diltiazem	**2**	**3**	**3**	**40.9**	**69.3**	**17.5**	30.1-51.7	47-94.3	15.7-48.6
Digoxin	0	0	0	NA	NA	NA	<89.4 [c]	<151 [c]	<31.2 [c]
Digoxigenin	0	0	0	NA	NA	NA	<122 [c]	<94.2 [c]	<117 [c]
(continued)									

Table 73 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Filter Feed Sludge [b]	Dewatered Cake	Filtrate	Filter Feed Sludge (ng/g TS)	Dewatered Cake (ng/g TS)	Filtrate (ng/L)	Filter Feed Sludge (ng/g TS)	Dewatered Cake (ng/g TS)	Filtrate (ng/L)
Enrofloxacin	**2**	2	0	**19**	23.3	NA	10.9-27.1	<12.3-33	<8.47 [c]
Erythromycin-H_2O	**2**	1	**3**	**3.87**	5.69 [a]	**5.31**	2.08-5.66	<1.23-5.69	3.75-7.18
Flumequine	0	0	0	NA	NA	NA	<7.18 [c]	<15.1 [c]	<2.1 [c]
Fluoxetine	**2**	**3**	2	**24.9**	**36.5**	5.565	21.8-28	20.4-48.3	<1.5-6.89
Lincomycin	0	0	0	NA	NA	NA	<19 [c]	<30.2 [c]	<11.9 [c]
Lomefloxacin	0	0	0	NA	NA	NA	<14.4 [c]	<30.2 [c]	<9.53 [c]
Miconazole	**2**	**3**	0	**456**	**473**	NA	403-508	401-495	<1.93 [c]
Norfloxacin	**2**	**3**	2	**561**	**534**	272	480-642	305-586	<36.5-338
Norgestimate	0	0	0	NA	NA	NA	<14.9 [c]	<31.7 [c]	<6.42 [c]
Ofloxacin	**2**	**3**	0	**927**	**915**	NA	794-1060	483-1120	<19.3 [c]
Ormetoprim	0	0	0	NA	NA	NA	<2.87 [c]	<6.04 [c]	<0.77 [c]
Oxacillin	0	0	0	NA	NA	NA	<14.4 [c]	<30.2 [c]	<3.86 [c]
Oxolinic Acid	0	0	0	NA	NA	NA	<2.91 [c]	<6.04 [c]	<0.86 [c]
Penicillin G	0	0	**3**	NA	NA	**6.85**	<14.4 [c]	<30.2 [c]	5.76-15.2
Penicillin V	0	0	0	NA	NA	NA	<14.4 [c]	<30.2 [c]	<5.9 [c]
Roxithromycin	0	0	0	NA	NA	NA	<1.7 [c]	<3.02 [c]	<0.65 [c]
Sarafloxacin	0	0	0	NA	NA	NA	<125 [c]	<313 [c]	<122 [c]
Sulfachloropyridazine	0	0	0	NA	NA	NA	<7.18	<15.1	<4.42
Sulfadiazine	0	0	0	NA	NA	NA	<7.18 [c]	<15.1 [c]	<1.93 [c]
Sulfadimethoxine	0	0	0	NA	NA	NA	<1.96 [c]	<3.02 [c]	<0.841 [c]
Sulfamerazine	0	0	0	NA	NA	NA	<2.87 [c]	<6.04 [c]	<2.36 [c]
Sulfamethazine	0	0	0	NA	NA	NA	<2.87 [c]	<7.31 [c]	<3.08 [c]
Sulfamethizole	0	0	0	NA	NA	NA	<2.87 [c]	<6.04 [c]	<1.61 [c]
Sulfamethoxazole	**2**	**3**	**3**	**14.45**	**15.8**	**22.6**	10.8-18.1	8.25-15.9	8.47-67.8
Sulfanilamide	0	0	0	NA	NA	NA	<71.8 [c]	<151 [c]	<26.6 [c]
Sulfathiazole	0	0	0	NA	NA	NA	<7.18 [c]	<15.1 [c]	<2.27 [c]
Thiabendazole	**2**	**3**	**3**	**87.85**	**93.9**	**58.2**	65.7-110	62.7-116	16-74.3
Trimethoprim	**2**	**3**	**3**	**36.8**	**29.1**	**27.5**	26.9-46.7	24.3-29.6	12.8-31.3

(continued)

Table 73 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Filter Feed Sludge [b]	Dewatered Cake	Filtrate	Filter Feed Sludge (ng/g TS)	Dewatered Cake (ng/g TS)	Filtrate (ng/L)	Filter Feed Sludge (ng/g TS)	Dewatered Cake (ng/g TS)	Filtrate (ng/L)
Tylosin	0	0	0	NA	NA	NA	<54.3 [c]	<82 [c]	<25.7 [c]
Virginiamycin	0	0	0	NA	NA	NA	<73.1 [c]	<103 [c]	<16.4 [c]
1,7-Dimethylxanthine	0	0	0	NA	NA	NA	<718 [c]	<1510 [c]	<193 [c]

[a] indicates median value is from one detectable concentration only; [b] Only two sampling campaigns for filter feed sludge.
[c] indicates highest identified detection limit for compound
Data in **bold** font are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

Table 74. Summary of Pharmaceutical Compound Detections in Filter Feed Sludge, Dewatered Cake and Filtrate, Saguenay, QC

Frequency of detection in sampling campaigns (out of 3)	# Compounds in Process Streams		
	Filter Feed Sludge [a]	Dewatered Cake	Filtrate
3	0 [a]	16	17
2	18	2	3
1	3	2	4
0	36	37	33
Total	57	57	57

a: Only two rounds of sampling were done for the filter thickened WAS feed sludge.

4.9.3.4 Fragrance and Alkylphenolic Compounds

Analytical results from the two sampling campaigns are provided in **Table 75**. The raw analytical data are provided in **Appendix Table A16**. Bisphenol A was detected in both dewatered cake samples at concentrations ranging from 40 to 610 ng/g TS. BPA was only detected in the feed sludge and filtrate in one of the two campaigns. The polycyclic musk fragrances HHCB and AHTN were detected at the highest concentrations (i.e., greater than 1000 ng/g TS) in both the filter press feed sludge and dewatered cake solids. The polycyclic musk fragrances DPMI and AHDI were found in the filter press feed sludge and dewatered cake solids in both sampling campaigns; the two fragrances were not detected in the filtrates in either sampling campaign. None of the nitro musk compounds were observed above the limit of quantification in the sludge feed or dewatered cake. Most compounds were observed at higher concentrations in the dewatered cake than in the feed sludge. Mass balances are required to determine whether the concentration differences are significant.

4.9.4 Data Interpretation

4.9.4.1 Metals Mass Balances

The concentrations of metals in the liquid feed sludge and filtrate are reported in units of mg/L, while the concentrations in the dewatered cake are reported in units of mg/kg TS. The solids balance around the dewatering filter press is based on historical plant data. The information used for the solids balance was:

Thickened WAS flow to filter: 157.5 m^3/d
Dewatered cake flow out: 37.2 m^3/d
Filtrate return flow: 140 m^3/d
Total solids concentration in thickened WAS Feed: 29.3 kg/m^3 (2.9%)
Total solids concentration in dewatered cake: 138.0 kg/m^3 (13.4%)
Total solids concentration in filtrate return: 0.59 kg/m^3 (0.059%)
Pad leachate flow rate: 25 m^3/d

The data used result in solids mass flows of 4613 kg/d in the total WAS feed, 5134 kg/d in the dewatered cake solids and 82.6 kg/d in the press filtrate. Based on this balance, approximately

Table 75. Frequency of Detection and Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Filter Feed Sludge, Dewatered Cake and Filtrate, Saguenay, QC

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)			Median of Detected Concentration			Range of Detected Concentration		
	Filter Feed Sludge	Biosolids Cake	Press Filtrate	Filter Feed Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Press Filtrate (ng/L)	Filter Feed Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Press Filtrate (ng/L)
Alkylphenolics									
Bisphenol A	1	**2**	1	480	**325**	1080	<80-480	40-610	<50-1080
Octylphenol	0	0	**2**	NA	NA	**50**	<20	<20	30-70
Nonylphenol	0	0	0	NA	NA	NA	<140	<140	<90
Fragrances									
DPMI	**2**	**2**	1	**230**	**100**	30	50-410	70-130	<20-30
ADBI	0	0	0	NA	NA	NA	<20	<20	<10
AHDI	**2**	**2**	0	**100**	**105**	NA	90-110	50-160	<10
HHCB	**2**	**2**	**2**	**3630**	**3290**	**590**	3050-4210	1520-5060	290-890
AHTN	**2**	**2**	**2**	**1850**	**2685**	**315**	1200-2500	1130-4240	120-510
ATII	**2**	**2**	**2**	**180**	**280**	**95**	50-310	90-470	30-160
Musk Moskene	0	0	0	NA	NA	NA	<50	<50	<90
Musk Tibetene	0	0	0	NA	NA	NA	<80	<80	<50
Musk Ketone	0	0	0	NA	NA	NA	<120	<120	<90
Musk Ambrette	0	0	0	NA	NA	NA	<140	<140	<20
Musk Xylene	0	0	0	NA	NA	NA	<70	<70	<40

Data in **bold font** are detected in both sampling campaigns

13% more solids exit the filter press than arrive in the feed. Given the uncertainties in applying the historical flow and solids concentrations, the solids balance is deemed acceptable.

The mass balance closure for metals around the filter press at the Saguenay plant is provided in **Table 76**. The mass closures were consistently less than 100%, ranging from a low of approximately 50% for lead to a high of 91% for mercury. The balance indicates that a substantial mass of the metals is not accounted for in the dewatered cake or filtrate relative to the input mass in the thickened WAS. It is not clear from the data whether the discrepancy is systematic from uncertainties in the process stream flow and solids concentration data, or whether the liquid feed sample was not representative of typical operation, with higher than normal metal concentrations.

Table 76. Mass Balance Closure for Metals in Belt Filter Press Dewatering Process, Saguenay, QC

Metal	Concentration of contaminant			Mass of contaminant			Mass Closure (%)
	Liquid Sludge (mg/L)	Dewatered Cake (mg/kg TS dw)	Filtrate (mg/L)	Liquid Sludge (g/d)	Dewatered Cake (g/d)	Filtrate (g/d)	
Chromium (Cr)-Total	1.24	24.5	0.010	195	117	1.4	60.8
Cobalt (Co)-Total	0.109	2.6	<0.0080	17.2	12.5	<1.12	72.5-<79.1
Copper (Cu)-Total	5.23	107	0.047	824	512	6.6	63.0
Lead (Pb)-Total	0.90	14.9	<0.010	142	71.4	<1.40	50.3-<51.3
Mercury (Hg)-Total	0.0234	0.677	0.00066	3.69	3.24	0.092	90.5
Nickel (Ni)-Total	0.48	10.5	<0.020	75.6	50.3	<2.80	66.5-<70.2
Zinc (Zn)-Total	7.54	159	0.069	1188	762	9.66	64.9%

4.9.4.2 Pharmaceutical Compounds Mass Balances

The mass balance estimates for pharmaceutical compounds around the dewatering facility of the Saguenay plant appear in **Table 77**. Removal efficiencies were mostly negative, suggesting that little removal of the compounds occurs through the mechanical dewatering process, nor would any significant removal be expected from a solids separation device. The removal efficiencies ranged from a low of -429% for the non-steroidal anti-inflammatory naproxen to a high of 21% for the anti-microbial triclosan.

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 78**. The table reinforces the observation that most of the removals are negative. A total of 15 of the twenty pharmaceuticals for which mass balances were calculated exhibit negative removal efficiencies. Because the belt pressure filter at the Saguenay facility is a physical separation process for concentrating solids, little biodegradation of the pharmaceutical compounds would be expected.

Table 77. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Belt Filter Press Dewatering Process, Saguenay, QC

Pharmaceutical	Concentration (median)			Mass of Contaminants (mg/d)			% Removal
	Thickened WAS (ng/g TS dw)	Bio-solids Cake (ng/g TS dw)	Filtrate (ng/L)	Thickened WAS	Bio-solids Cake	Filtrate	
Furosemide	139	165	402	641	847	56.3	-40.9%
Ibuprofen	47.8	<145	190	221	<694	26.6	Not determined
Naproxen	18.4	83.8	132	84.9	430	18.5	-429%
Triclocarban	1875	1660	8.9	8,650	8,523	1.25	1.5%
Triclosan	1852	1310	84	8,541	6,726	11.8	21.1%
Azithromycin	334	262	162	1,538	1,345	22.7	11.1%
Caffeine	83	291	<17.2	383	1,492	<2.41	-290%
Carbamazepine	50.5	51.6	345	233	265	48.3	-34.6%
Ciprofloxacin	7835	6440	54.2	36,140	33,070	7.59	8.5%
Clarithromycin	50.1	69.1	27.4	231	355	3.84	-55.3%
Diphenhydramine	465	420	88.1	2,145	2,156	12.3	-1.1%
Diltiazem	40.9	69.3	17.5	189	356	2.45	-89.9%
Enrofloxacin	19	23.3	<8.37	87.7	119	<1.17	-36.2%
Erythromycin-H_2O	3.87	5.69	5.31	17.9	29.2	0.74	-67.8%
Fluoxetine	24.9	36.5	5.57	115	187	0.78	-63.8%
Miconazole	456	473	<1.72	2,101	2,429	<0.24	-15.6%
Norfloxacin	561	534	272	2,588	2,742	38.1	-7.4%
Ofloxacin	927	915	<17.2	4,276	4,698	<2.41	-9.9%
Sulfamethoxazole	14.5	15.8	22.6	66.7	81.1	3.16	-26.4%
Thiabendazole	87.9	93.9	58.2	405	482	8.15	-21.0%
Trimethoprim	36.8	29.1	27.5	170	149	3.85	9.7%

Table 78. Categorised Removal Efficiencies of Pharmaceutical Compounds by Belt Filter Press Dewatering Process, Saguenay, QC

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Naproxen	Furosemide	Triclocarban		
Caffeine	Carbamazepine	Triclosan		
Clarithromycin	Diphenhydramine	Azithromycin		
Diltiazem	Enrofloxacin	Ciprofloxacin		
Erythromycin-H_2O	Miconazole	Trimethoprim		
Fluoxetine	Norfloxacin			
	Ofloxacin			
	Sulfamethoxazole			
	Thiabendazole			
n=6	n=9	n=5	n=0	n=0

4.9.4.3 Fragrance and Alkylphenolic Compounds

The mass balance closures and calculated removal efficiencies for BPA and the fragrance compounds appear in **Table 79**. The mass of these compounds in the filtrate relative to the feed mass is slight, with nearly all of the compound mass in the feed sludge residing in the dewatered cake solids. As with the pharmaceutical compounds, the belt filter press dewatering process would not be expected to remove much of the incoming mass of alkylphenolic and fragrance compounds. The calculated reduction of Bisphenol A though the Saguenay dewatering process was 18%. With the exception of the polycyclic musk DPMI, the calculated removal efficiencies for the fragrance compounds were negative. The two feed sludge samples had a wide range of DPMI concentrations (50-410 ng/g TS dw), which was most likely the reason for the calculated removal efficiency of 51% for the compound.

Table 79. Mass Balance and Removal Calculations for Alkylphenolic and Fragrance Compounds in Belt Filter Press Dewatering Process, Saguenay, QC

Compound	Concentrations (median)			Mass of Contaminants			% Removal
	Filter Feed Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)	Filter Feed Sludge (mg/d)	Biosolids Cake (mg/d)	Filtrate (mg/d)	
Alkylphenolics							
Bisphenol A	480	325	1080	2,214	1,669	151	18%
Fragrances							
DPMI	230	100	30	1,061	513	4	51%
AHDI	100	105	<10	461	539	<1	-17%
HHCB	3630	3290	590	16,746	16,892	83	-1%
AHTN	1850	2685	315	8,534	13,786	44	-62%
ATII	180	280	95	830	1,438	13	-75%
Musk Xylene	30	40	<20	138	205	<3	-48%

4.9.4.4 Effectiveness of Process for ESOC Removal

The results of the sampling survey, as typified by the Saguenay data, indicate that the belt filter press used to dewater the thickened waste activated sludge provides a negligible barrier for reducing the mass of pharmaceutical and fragrance compounds in the dewatered cake.

4.9.5 Section Summary

Nitrogen was present in the thickened WAS feed mostly as the organic nitrogen component of TKN. The ammonia concentration was substantially higher than the oxidised forms of nitrogen (nitrate and nitrite), which were less than the detection limit. Similar trends were observed in the dewatered cake and press filtrate. Soluble ortho-phosphate contributes only a very small amount to the total P concentration in the thickened WAS. With the exception of ortho-phosphate, other nutrient forms are significantly reduced in the press filtrate, leading to the conclusion that the

nutrients are concentrated in the dewatered cake. Direct comparison with the cake nutrient concentrations is not possible because they are measured and expressed on a dry weight basis. Most target metals (except for arsenic, molybdenum and selenium) are detected in the thickened WAS feed to the filter press, and all target metals except for cadmium are detected in the dewatered cake. Few metals were identified above the detection limits in the press filtrate, except for chromium, copper, mercury and zinc. Zinc and copper were observed at the highest detected concentrations in the three sample matrices. Cadmium was the metal with the lowest detected concentration in the feed sludge, while in the dewatered cake and filtrate, mercury was the metal with the lowest detected concentrations. The mass closures were consistently less than 100%, ranging from a low of approximately 50% for lead to a high of 91% for mercury. The balance indicates that a substantial mass of the metals is not accounted for in the dewatered cake or filtrate relative to the input mass in the thickened WAS. It is not clear from the data whether the discrepancy is systematic from uncertainties in the process stream flow and solids concentration data, or whether the apparent loss is due to other process streams that were not accounted for in the mass balance.

A total of 18 pharmaceuticals were detected in the filter feed sludge from both sampling campaigns (note there are only two instead of three samplings for this feed stream); 16 pharmaceuticals were detected in the dewatered biosolids samples from all the three campaigns and 17 pharmaceuticals were detected in the filtrate samples from all the three campaigns. The antimicrobials triclosan and triclocarban, and the antibiotic ciprofloxacin were found at the highest concentrations in the dewatered cake. A number of compounds were detected in the press filtrate in all three sampling campaigns, but were not detected in the feed sludge or dewatered cake in any of the campaigns. These included gemfibrozil (blood lipid regulator), glyburide (anti-diabetic), hydrochlorothiazide (diuretic), dehydronifedipine (anti-anginal metabolite), and penicillin G (antibiotic). The distribution appears to be relatively stable in all three process streams, based on the number of compounds detected in all campaigns (2 for the feed sludge; 3 for the dewatered cake and filtrate), and the number of compounds never detected in any of the sampling campaign. For example the number of compounds detected in all sampling campaigns were 18 in the thickened WAS feed, 16 in the dewatered cake and 17 in the filtrate. The number of compounds never detected in any of the campaigns was 36 for the filter feed sludge, 37 for the dewatered cake and 33 for the filtrate. Removal efficiencies were mostly negative, suggesting that little removal of the compounds occurs. The removal efficiencies ranged from a low of -429% for the non-steroidal anti-inflammatory naproxen to a high of 21% for the anti-microbial triclosan. A total of 15 of the twenty pharmaceuticals with calculated mass balances exhibit negative removal efficiencies. Because the belt pressure filter at the Saguenay facility is a physical separation process for concentrating solids, little biodegradation of the pharmaceutical compounds would be expected.

Bisphenol A was detected in both dewatered cake samples at concentrations ranging from 40 to 610 ng/g TS dw. BPA was only detected in the feed sludge and filtrate in one of the two campaigns. The polycyclic musk fragrances HHCB and AHTN were detected at the highest concentrations (i.e., greater than 1000 ng/g TS dw) in both the filter press feed sludge and dewatered cake solids. The polycyclic musk fragrances DPMI and AHDI were found in the filter press feed sludge and dewatered cake solids in both sampling campaigns; these two fragrances were not detected in the filtrates in either sampling campaign. None of the nitro musk

compounds were observed above the limit of quantification in the sludge feed or dewatered cake. Most compounds were observed at higher concentrations in the dewatered cake than in the feed sludge. The mass of these compounds in the filtrate relative to the feed mass is slight, with nearly all of the compound mass in the feed sludge residing in the dewatered cake solids. The calculated reduction of Bisphenol A though the Saguenay dewatering process was 18%. With the exception of the polycyclic musk DPMI, the calculated removal efficiencies for the fragrance compounds were negative. The two feed sludge samples had a wide range of DPMI concentrations (50-410 ng/g TS), which was most likely the reason for the calculated removal efficiency of 51% for the compound.

The results of the sampling survey, as typified by the Saguenay data, indicate that the belt filter press used to dewater the thickened waste activated sludge provides a negligible barrier for reducing the mass of pharmaceutical and fragrance compounds in the dewatered cake.

4.10 Composting of Lime-Stabilised Biosolids, Moncton, NB

4.10.1 Site Description

The Moncton Wastewater Treatment Plant (WWTP) is a primary treatment facility, with unit treatment processes including bar screens, grit removal and primary clarification. The treated effluent is discharged to the Petitcodiac River. The design capacity of the existing treatment plant is 132,294 m^3/d, while the average daily dry weather flow is 62,621 m^3/d.

4.10.2 Biosolids Treatment Description

The primary sludge from the primary clarifiers is sent to two sludge holding tanks for three to four days storage. The purpose is to improve solids settling for more efficient dewatering. The settled primary sludge with an increased solids content (i.e. from about 1.5-2.5% increased to 3-5% solids) is dewatered by centrifuge and then is lime stabilised. The flow rate of the dewatered cake (i.e. abut 30% solids) to the lime stabilisation facility is 18.2 m^3/h on Mondays and Thursday each week for 7 hours per day, which is when the centrifuge usually operates. Dry lime is added at the lime stabilisation facility at an average flow rate of 164.4 kg/h to increase pH of the biosolids for pathogen reduction in the biosolids.

The lime stabilised biosolids is then transferred to the composting facility for composting. The compost bulking agents such as wood chips are added to the composting facility at a ratio of 2:1 (wood chips: lime stabilised biosolids, v/v) for dilution of the biosolids. After two months of active composting, the compost product is sent to a composting pad for curing for about twelve months. The finished compost product is applied to agricultural lands or for general public use (i.e. to lawns or gardens). During this study, the solids concentration in the feed sludge (i.e. lime stabilised biosolids) to the composting facility ranged from 34.7% to 37.9%; while the solids concentration in final finished compost product ranged from 40.8% to 46.9%.

For this project assessment, the biosolids treatment process of interest was composting. The two sampling locations included feed sludge (i.e. lime stabilised biosolids) to the composting facility

and finished compost (i.e. after two months of active composting and about twelve months of curing). No leachate generation from the composting process was identified by plant staff. A process schematic of the Moncton biosolids treatment process is shown in **Figure 16**.

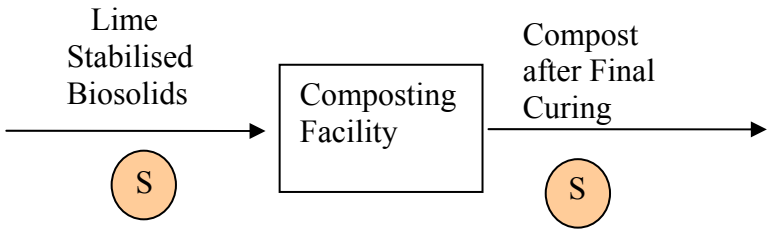

Figure 16. Schematic of Moncton Biosolids Process and Sampling Locations

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on July 30, August 10 and August 24, respectively.

4.10.3 Sampling Results

4.10.3.1 Nutrients

The concentration of nitrate-N is higher in compost product than in the process feed sludge (i.e. lime stabilised biosolids) (**Table 80**). At the same time, the concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) are much lower in the compost samples than in the feed biosolids. Biological conversion of ammonia-N to nitrate-N would be expected during composting, an aerobic biological process. The organic nitrogen component of the TKN appears to have undergone the greatest reduction in concentration, potentially due to biodegradation and stripping of volatile organic-N compounds, such as amines. The observed differences may also reflect the variations in the composition of the two process streams at the time of sampling.

Table 80. Nutrients in Lime Stabilised Biosolids and Compost from Moncton, NB

Parameter	Concentration (mg/kg TS dw)	
	Lime Stabilised Biosolids	Compost
Nitrate-N	4.2	260
Nitrite-N	<1.0	<1.0
Total Kjeldahl Nitrogen	19300	2390
Ammonia as N	953	701
Phosphorus, Total	5130	544
Phosphate-P (ortho)	1.36	35.6
Total Solids	291000	426000

The concentration of total phosphorus is substantially lower in the compost product than in the feed sludge (i.e. lime stabilised biosolids), while the concentration of ortho-phosphate is higher after composting. Loss of total phosphorus from the process is unexpected, as it is a conservative element. The increased concentration of ortho-phosphate may result from some increased solubilisation of organic P-bearing compounds, or of insoluble precipitates or polyphosphates. The observed difference may also reflect the variations in the composition of the two process streams at the time of sampling.

4.10.3.2 Metals

Most metals in the lime stabilised biosolids feed and final compost are identified above the detection limits, as shown in **Table 81**, with the exceptions of arsenic, cadmium and selenium. Concentrations of the metals are on the same order of magnitude in the feed and compost samples. Zinc and copper are the metals observed at the highest concentration in both the feed and compost streams, while mercury has the lowest detected concentration of the metals analysed. Additional discussion of the metals is found in the Data Interpretation section.

Table 81. Metals in Lime Stabilised Biosolids and Compost, Moncton, NB

Parameter	Concentration (mg/kg TS dw)	
	Lime Stabilised Biosolids	**Compost**
Arsenic (As)-Total	<1.0	**1.9**
Cadmium (Cd)-Total	<1.0	<1.0
Chromium (Cr)-Total	**9.1**	**15.0**
Cobalt (Co)-Total	**1.5**	**4.2**
Copper (Cu)-Total	**82.6**	**81.0**
Lead (Pb)-Total	**15.0**	**24.7**
Mercury (Hg)-Total	**0.690**	**0.493**
Molybdenum (Mo)-Total	**1.9**	**1.5**
Nickel (Ni)-Total	**3.7**	**8.7**
Selenium (Se)-Total	<1.0	<1.0
Zinc (Zn)-Total	**138**	**170**
Total Solids	291000	426000

Samples in **bold** font are above the detection limit

4.10.3.3 Pharmaceuticals

The frequency of detection and median and range of detected concentrations of the pharmaceutical compounds in the composting feed sludge (i.e. lime stabilised biosolids) and the finished compost at the Moncton facility are presented in **Table 82**. The raw concentration data for the three sampling campaigns are found in **Appendix Table A17**. A total of 20 pharmaceuticals were detected in the composting feed sludge (i.e. lime stabilised biosolids) from all the three sampling campaigns, while only six pharmaceuticals were detected in the finished compost samples from all the three campaigns. Median values of the detected concentrations of pharmaceuticals (except for Naproxen) are lower in the compost product than in the composting feed sludge (i.e. lime stabilised biosolids), which indicates that the composting process has the ability to remove many of the pharmaceuticals.

Table 82. Frequency of Detection, Median and Range of Detected Concentrations of Pharmaceutical Compounds in Lime Stabilised Biosolids and Compost from Moncton, NB

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Lime Stabilised Biosolids	Compost	Lime Stabilised Biosolids	Compost	Lime Stabilised Biosolids	Compost
Furosemide	0	1	NA	575 [a]	<165 [b]	<135-575
Gemfibrozil	1	0	17.5 [a]	NA	<5.51-17.5	<5.92 [b]
Glipizide	0	0	NA	NA	<24.7 [b]	<23.7 [b]
Glyburide	0	0	NA	NA	<12.3 [b]	<11.8 [b]
Hydrochlorothiazide	1	0	103 [a]	NA	<71.6-103	<78.9 [b]
2-Hydroxy-ibuprofen	0	1	<318	1050 [a]	<329 [b]	<271-1050
Ibuprofen	**3**	0	**147**	NA	105-180	<59.2 [b]
Naproxen	**3**	**3**	**78.2**	**3830**	48.2-106	2810-9640
Triclocarban	**3**	**3**	**1750**	**146**	1710-3070	64.4-166
Triclosan	**3**	**3**	**7020**	**634**	5910-7300	603-960
Warfarin	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Acetaminophen	1	0	222 [a]	NA	<215-222	<237 [b]
Azithromycin	**3**	0	**147**	NA	141-216	<5.92 [b]
Caffeine	**3**	0	**1090**	NA	1080-1110	<59.2 [b]
Carbadox	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Carbamazepine	**3**	**3**	**81.2**	**15.5**	69.4-142	5.27-18.8
Cefotaxime	0	0	NA	NA	<78.9 [b]	<36.3 [b]
Ciprofloxacin	**3**	2	**3770**	379	3060-4190	<113-433
Clarithromycin	**3**	0	**54.1**	NA	44.3-165	<5.92 [b]
Clinafloxacin	0	0	NA	NA	<26.4 [b]	<307 [b]
Cloxacillin	0	0	NA	NA	<12.3 [b]	<11.8 [b]
Dehydronifedipine	**3**	0	**2.98**	NA	2.85-4.93	<2.37 [b]
Diphenhydramine	**3**	**3**	**1090**	**13.5**	617-1270	6.58-57.7
Diltiazem	**3**	0	**121**	NA	58.1-140	<1.18 [b]
Digoxin	0	0	NA	NA	<61.7 [b]	<59.2 [b]
Digoxigenin	0	0	NA	NA	<34.7 [b]	<71.6 [b]
Enrofloxacin	**3**	0	**14.7**	NA	14.2-15.4	<30.7 [b]
Erythromycin-H_2O	**3**	0	**10.5**	NA	9.69-15.5	<1.18 [b]
Flumequine	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Fluoxetine	**3**	0	**18.7**	NA	10.3-62.7	<5.92 [b]
Lincomycin	0	0	NA	NA	<25.1 [b]	<26.8 [b]
Lomefloxacin	0	0	NA	NA	<12.3 [b]	<11.5 [b]
Miconazole	**3**	**3**	**312**	**26.4**	212-345	21.8-31.1
Norfloxacin	**3**	0	**793**	NA	793-877	<268 [b]
Norgestimate	0	0	NA	NA	<12.5 [b]	<11.8 [b]
Ofloxacin	**3**	2	**165**	121.95	148-392	<50.7-183

(continued)

Table 82 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Lime Stabilised Biosolids	Compost	Lime Stabilised Biosolids	Compost	Lime Stabilised Biosolids	Compost
Ormetoprim	0	0	NA	NA	<2.47 [b]	<2.37 [b]
Oxacillin	0	0	NA	NA	<12.3 [b]	<11.8 [b]
Oxolinic Acid	0	0	NA	NA	<2.87 [b]	<3.82 [b]
Penicillin G	0	0	NA	NA	<39.8 [b]	<33.8 [b]
Penicillin V	0	0	NA	NA	<12.3 [b]	<11.8 [b]
Roxithromycin	0	0	NA	NA	<1.94 [b]	<1.54 [b]
Sarafloxacin	0	0	NA	NA	<126 [b]	<1080 [b]
Sulfachloropyridazine	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Sulfadiazine	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Sulfadimethoxine	0	0	NA	NA	<2.05 [b]	<1.18 [b]
Sulfamerazine	0	0	NA	NA	<2.47 [b]	<3.04 [b]
Sulfamethazine	0	0	NA	NA	<2.78 [b]	<3.92 [b]
Sulfamethizole	0	0	NA	NA	<2.47 [b]	<2.37 [b]
Sulfamethoxazole	1	0	3.92 [a]	NA	<2.15-3.92	<2.51 [b]
Sulfanilamide	0	0	NA	NA	<61.7 [b]	<59.2 [b]
Sulfathiazole	0	0	NA	NA	<6.17 [b]	<5.92 [b]
Thiabendazole	**3**	0	**12.5**	NA	10.1-44.8	<5.92 [b]
Trimethoprim	**3**	0	**33.1**	NA	31.6-36.5	<8.13 [b]
Tylosin	0	0	NA	NA	<82.3 [b]	<78.9 [b]
Virginiamycin	0	0	NA	NA	<186 [b]	<66.1 [b]
1,7-Dimethylxanthine	0	0	NA	NA	<617 [b]	<592 [b]

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The distribution of detectable concentrations in the composting feed sludge (i.e. lime stabilised biosolids) and the finished compost from the three sampling campaigns is found in **Table 83**. There is a major shift in the distribution of detectable concentrations in the composting feed sludge (i.e. lime stabilised biosolids) and finished compost. Most notably the number of compounds detected in all three campaigns declines from 20 in the composting feed sludge to 6 in the finished compost, while the number of compounds never detected in any of the three campaigns increases from 33 in the composting feed sludge to 47 in the finished compost.

Table 83. Summary of Pharmaceutical Compound Detections in Lime Stabilised Biosolids and Compost from Moncton, NB

Frequency of detection in sampling campaigns (out of 3)	Number of Compounds in Process Streams	
	Lime Stabilised Biosolids	Compost
3	20	6
2	0	2
1	4	2
0	33	47
Total	57	57

4.10.3.4 Fragrance and Alkylphenolic Compounds

Analytical data for the alkylphenolic and fragrance compounds for the two sampling campaigns are available and are provided in **Table 84**. The raw analytical data are provided in **Appendix Table A18**. Bisphenol A was detected in both the lime stabilised feed solids and compost in the two campaigns. It was substantially reduced in concentration through the composting process, from a median concentration of 965 ng/g TS in the lime-stabilised feed sludge, to a median value of 125 ng/g TS in the finished compost. None of the polycyclic musks was detected at concentrations greater than 1000 ng/g TS in the finished compost, although the median concentration of HHCB and AHTN were both greater than 1,000 ng/g TS. None of the nitro musk compounds were observed above the limit of quantification. Many compounds in the compost were observed at lower concentrations than in the feed sludge, but mass balances are required to determine whether the concentration differences are significant (see the later section on Data Interpretation).

4.10.4 Data Interpretation

4.10.4.1 Total Solids Mass Balance Estimate

Concentrations of the metal and pharmaceutical contaminants in the dewatered lime-amended cake and final compost are expressed on a dry weight basis (i.e. mg/kg TS or ng/g TS). A solids balance around the composting process was developed using the following information:

Lime-stabilised dewatered cake feed mass feed rate = 15,500 kg/h
Dry lime feed rate = 164.4 kg/h
Measured total solids concentration in dewatered cake = 36.0%
Measured total solids concentration in compost = 43.5%

For the balance, it was assumed based on professional judgement that the volatile solids fraction of the dewatered cake was 0.75, and the volatile solids reduction achieved was 55%. It was further assumed that the mass of fixed solids in the dewatered cake was conserved in the compost following removal of the wood-based bulking agents. There was no report of leachate produced in the composting process and returned to the plant for treatment. The solids mass balance is presented in **Figure 17**.

Table 84. Frequency of Detection and Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Lime Stabilised Biosolids and Compost, Moncton, NB

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Lime stabilised sludge	Compost	Lime stabilised sludge	Compost	Lime stabilised sludge	Compost
Alkylphenolics						
Bisphenol A	**2**	**2**	**965**	**125**	770-1160	70-180
Octylphenol	1	1	20	40	<20-20	<20-40
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	**2**	**2**	**80**	**65**	60-100	40-90
ADBI	0	0	NA	NA	<20	<20
AHDI	**2**	**2**	**615**	**55**	228-900	40-70
HHCB	**2**	**2**	**3940**	**715**	3080-4800	530-900
AHTN	**2**	**2**	**1895**	**595**	1250-2540	230-960
ATII	**2**	1	**475**	90	370-580	<60-90
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	<120	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	0	0	NA	NA	<70	<70

Data in **bold font** are detected in both sampling campaigns

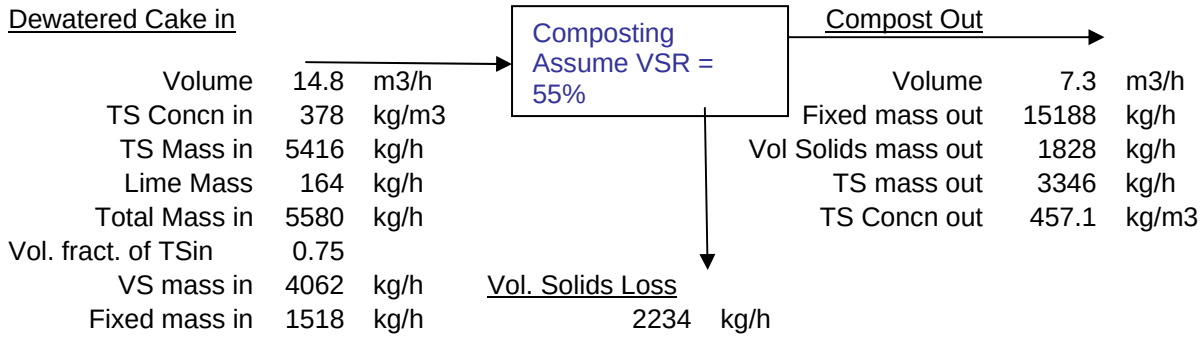

Figure 17. Solids Balance at Moncton, NB, Composting Process

4.10.4.2 Metals

Results of the metals mass balance and closure are presented in **Table 85**. Mass balances were not attempted for arsenic, cadmium and selenium because of non-detectable concentrations of these metals in the dewatered feed cake. Values of the mass closures range from a low of 43% for mercury to a high of 168 % for cobalt. The median mass closure was 86%, indicating that the mass of metals entering the process is mostly accounted for in the compost product. Mercury is a volatile metal, and some loss may be possible during aeration of the compost pile. Variations in the mass closures may be due to variations of the composition of the feed and product samples based on the one sampling campaign, due to some error derived from calculating the solids balance, and analytical error from determining concentrations close to the detection limit for compounds such as cobalt and molybdenum.

Table 85. Mass Balance of Metals through Composting Process, Moncton, NB

Metal	Concentration (mg/kg TS dw)		Mass of Contaminant (g/d)		Mass Closure (%)
	Lime stabilised biosolids	Compost	Lime stabilised biosolids	Compost	
Chromium (Cr)-Total	9.1	15.0	51	50	99
Cobalt (Co)-Total	1.5	4.2	8.4	14	168
Copper (Cu)-Total	82.6	81.0	461	271	59
Lead (Pb)-Total	15.0	24.7	84	83	99
Mercury (Hg)-Total	0.690	0.493	3.9	1.6	43
Molybdenum (Mo)-Total	1.9	1.5	11	5.0	47
Nickel (Ni)-Total	3.7	8.7	21	29	141
Zinc (Zn)-Total	138	170	770	569	74

4.10.4.3 Pharmaceuticals

The mass balances for the pharmaceutical compounds are presented in **Table 86**. Many of the calculated and estimated minimum removal efficiencies for the pharmaceuticals in the composting process were very high, above 90% of the mass in the feed lime-stabilised dewatered cake. The highest removals of 99% were noted for the stimulant diphenhydramine and the anti-angina drug diltiazem. Several more pharmaceuticals had removal efficiencies of 95% or higher. The non-steroidal anti-inflammatory naproxen exhibited substantial formation in the compost product with a negative removal of -2800% (i.e. the mass in the finished compost was 30 times higher than in the feed cake.

The distribution of removal efficiencies for the pharmaceutical compounds is presented in **Table 87.** The table clearly indicates that most of the compounds are removed effectively by the composting process. Twenty-one of the 24 compounds with calculated removal efficiencies were removed by more than 50%, and 11 of the 24 compounds were removed by greater than 90%.

Table 86. Mass Balance and Removal Calculations for Pharmaceutical Compounds in the Composting Process, Moncton, NB

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/h)		% Removal
	Lime stabilised sludge	Compost	Lime stabilised sludge	Compost	
Gemfibrozil	17.5	<5.89	97.7	<19.7	>80%
Hydrochlorothiazide	103	<76.6	575	<256	>55%
Ibuprofen	147	<57.4	820	<192	>77%
Naproxen	78.2	3830	436	128153	-2837%
Triclocarban	1750	146	9765	489	95%
Triclosan	7020	634	39170	2121	95%
Acetaminophen	222	<230	1239	<770	>38%
Azithromycin	147	<5.75	820	<19.2	>98%
Caffeine	1090	<57.5	6082	<192	>97%
Carbamazepine	81.2	15.5	453	51.9	89%
Ciprofloxacin	3770	379	21030	1268	94%
Clarithromycin	54.1	<5.75	302	<19.2	>94%
Dehydronifedipine	2.98	<2.3	16.6	<7.70	>54%
Diphenhydramine	1090	13.5	6082	45.2	99%
Diltiazem	121	<1.15	675	<3.85	>99%
Enrofloxacin	14.7	<27.75	82.0	<92.9	>-13%
Erythromycin-H_2O	10.5	<1.15	58.6	<3.85	>93%
Fluoxetine	18.7	<5.75	104	<19.2	>82%
Miconazole	312	26.4	1741	88.3	95%
Norfloxacin	793	<159.35	4425	<533	>88%
Ofloxacin	165	121.95	921	408	56%
Sulfamethoxazole	3.92	<2.3	21.9	<7.70	>65%
Thiabendazole	12.5	<5.75	69.8	<19.2	>72%
Trimethoprim	33.1	<5.75	185	<19.2	>90%

Table 87. Categorised Removal Efficiencies of Pharmaceutical Compounds by Composting Process, Moncton, NB

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Naproxen	Enrofloxacin	Acetaminophen	Gemfibrozil	Triclocarban
			Hydrochlorothiazide	Triclosan
			Ibuprofen	Azithromycin
			Carbamazepine	Caffeine
			Dehydronifedipine	Ciprofloxacin
			Fluoxetine	Clarithromycin
			Norfloxacin	Diphenhydramine

(continued)

Table 87 (continued)

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
			Ofloxacin	Diltiazem
			Sulfamethoxazole	Erythromycin-H_2O
			Thiabendazole	Miconazole
				Trimethoprim
n=1	n=1	n=1	n=10	n=11

Note: compounds listed in *italic font* are based on minimum estimated removal efficiencies

4.10.4.4 Fragrance and Alkylphenolic Compounds

Mass balances and removal efficiencies of the fragrance and alkylphenolic compounds are presented in **Table 88**. The assessment indicated a high removal efficiency (92%) of Bisphenol A through the composting process. The calculated negative removal efficiency for octylphenol was attributed to a single set of concentrations close to the limit of quantitation. Removal efficiencies for the polycyclic musk fragrances in general were high, ranging from 51% (DPMI) to 95% (AHDI).

Table 88. Mass Balance and Removal Calculations for Alkylphenolic and Fragrance Compounds by Composting Process, Moncton, NB

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/h)		% Removal
	Lime stabilised sludge	Compost	Lime stabilised sludge	Compost	
Alkylphenolics					
Bisphenol A	965	125	6797	526	92%
Octylphenol	20	40	141	168	-19%
Fragrances					
DPMI	80	65	564	273	51%
AHDI	615	55	4332	231	95%
HHCB	3940	715	27753	3007	89%
AHTN	1895	595	13348	2503	81%
ATII	475	90	3346	379	89%

4.10.4.5 Effectiveness of Process for ESOC Removal

The data for the composting process at Moncton indicate that high removal efficiencies are obtained for many pharmaceutical compounds, BPA and polycyclic fragrance compounds. Based on the concentration data for the non-steroidal anti-inflammatory compound naproxen, substantial formation of the compound occurs during composting. It may be possible that implementing lime stabilisation of the dewatered primary sludge cake prior to composting has a

beneficial effect on removal of pharmaceutical compounds in the composting process. Furthermore it is worth noting this system treated lime stabilised raw primary solids only, and none of the material had been previously exposed to a biological process that might have reduced masses entering the composting process. These potential effects could not be determined from the data collected.

4.10.5 Section Summary

The concentration of nitrate-N is higher in compost product than in the process feed sludge (i.e. lime stabilised biosolids), while at the same time, the concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) are much lower in the compost samples than in the feed biosolids. The organic nitrogen component of the TKN appears to have undergone the greatest reduction in concentration, potentially due to biodegradation and stripping of volatile organic-N compounds, such as amines. The concentration of total phosphorus is substantially lower in the compost product than in the feed sludge, while the concentration of ortho-phosphate is higher after composting. Loss of total phosphorus from the process is unexpected, as it is a conservative element. For both the nitrogen and phosphorus concentrations, observed difference may be a result of the variations in the composition of the two process streams at the time of sampling.

Most metals in the lime stabilised biosolids feed and final compost are identified above the detection limits, with the exceptions of arsenic, cadmium and selenium. Concentrations of the metals are on the same order of magnitude in the feed (lime-stabilised dewatered cake) and compost samples. Zinc and copper are the metals observed at the highest concentration in both the feed and compost streams, while mercury has the lowest detected concentration of the metals analysed. Mass balances were not attempted for arsenic, cadmium and selenium because of non-detectable concentrations of these metals in the dewatered feed cake. Values of the mass closures range from a low of 43% for mercury to a high of 168 % for cobalt. The median mass closure was 86%, indicating that the mass of metals entering the process is mostly accounted for in the compost product. Variations in the mass closures may be due to variations of the composition of the feed and product samples based on the one sampling campaign, due to some error derived from calculating the solids balance, and analytical error from determining concentrations close to the detection limit for compounds such as cobalt and molybdenum.

Many of the calculated and estimated minimum removal efficiencies for pharmaceutical compounds in the composting process were very high, above 90% of the mass in the feed lime-stabilised dewatered cake. The highest removals of 99% were noted for the stimulant diphenhydramine and the anti-angina drug diltiazem. Several more pharmaceuticals had removal efficiencies of 95% or higher. The non-steroidal anti-inflammatory naproxen exhibited substantial formation in the compost product with a negative removal of -2800% (i.e. the mass in the finished compost was 30 times higher than in the feed cake. Most of the pharmaceutical compounds are removed effectively by the composting process at Moncton. Of the 24 compounds with calculated removal efficiencies, 21 were removed by more than 50%, and 11 of the 24 compounds were removed by greater than 90%.

Bisphenol A was detected in both the lime stabilised feed solids and compost in the two campaigns. It was substantially reduced in concentration through the composting process, from a median concentration of 965 ng/g TS dw in the lime-stabilised feed sludge, to a median value of 125 ng/g TS dw in the finished compost. None of the polycyclic musks was detected at concentrations greater than 1000 ng/g TS in the finished compost, although the median concentration of HHCB and AHTN were both greater than 1,000 ng/g TS dw. None of the nitro musk compounds were observed above the limit of quantification. Many compounds in the compost were observed at lower concentrations than in the feed sludge. The mass balance assessment indicated a high removal efficiency (92%) of Bisphenol A through the composting process. The calculated negative removal efficiency for octylphenol was attributed to a single set of concentrations close to the limit of quantitation. Removal efficiencies for the polycyclic musk fragrances in general were high, ranging from 51% (DPMI) to 95% (AHDI).

The data for the composting process at Moncton indicate that high removal efficiencies are obtained for many pharmaceutical compounds, BPA and polycyclic fragrance compounds. It may be possible that implementing lime stabilisation of the dewatered primary sludge cake prior to composting has a beneficial effect on removal of pharmaceutical compounds in the composting process. Furthermore it is worth noting this system treated lime stabilised raw primary solids only, and none of the material had been previously exposed to a biological process that might have reduced masses entering the composting process. These potential effects could not be determined from the data collected.

4.11 N-Viro Alkaline Stabilisation Process, Halifax, NS

4.11.1 Site Description

The N-Viro process in Halifax is owned and operated by N-Viro Systems Canada and is designed to receive dewatered cake from five wastewater treatment facilities: Halifax, Aerotech, Herring Cove, Bedford and Dartmouth.

4.11.2 N-Viro Process Description

The Halifax wastewater treatment facility is currently not operating and so no sludge is being sent to the N-Viro Process. The Herring Cove facility will come on line later in 2009. The Dartmouth facility is an enhanced primary treatment plant utilizing alum and polymer. The Bedford facility is a pure oxygen system with mesophilic anaerobic digestion. After digestion, the biosolids are taken to the Aerotech facility for dewatering. The Aerotech facility is a SBR (sequencing batch reactor) sewage plant. It receives waste from the airport, an industrial park and also septage.

The N-Viro facility receives dewatered biosolids cake from the Aerotech and Dartmouth facilities. The system is designed to blend the incoming cake, but currently this is not practiced. It is currently done in batch mode for the cake of one facility and then the cake of another facility. Because of the mix of inputs sludges, it was not possible to determine which were being processed during the sampling periods. The average feed rate of the dewatered cake and alkaline

admixture to the N-Viro biosolids dryer is 9 wet tonnes/hour. The average N-Viro production rate is 7 wet tonnes/hour. During this study, the solids concentration in the feed sludge to the N-Viro dryer ranged from 44.7% to 50.4%, while the solids concentration in the final N-Viro product after curing and storage ranged from 67.4% to 69.6%. The processed biosolids are sent to agricultural lands.

For this project assessment, the biosolids treatment process of interest was the N-Viro system. The two sampling locations included the sludge feed (i.e. the dewatered cake and alkaline admixture) to dryer (i.e. the collection point was after mechanical mixing and before dryer) and final curing products (72 hours has elapsed after between when the product left the drier and when the sample was collected, which means the product is 72 hours old). A process schematic of the N-Viro treatment process is shown in **Figure 18**.

The plant was considered by plant staff to be in normal operation during the tree sampling campaigns. Samples were collected and shipped to the analytical laboratories on June 22, September 2 and October 7, 2009 respectively.

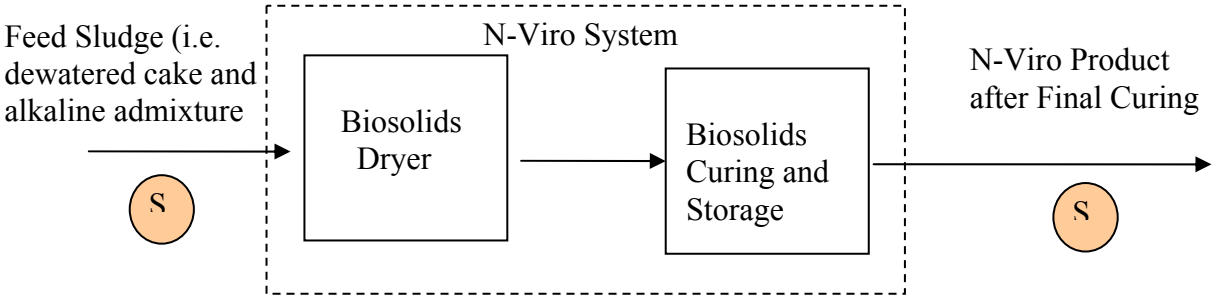

Figure 18. Schematic of Halifax N-Viro Biosolids Process and Sampling Locations

4.11.3 Sampling Results

4.11.3.1 Nutrients

A higher concentration of nitrate-N was observed in the N-Viro product than in feed sludge after admixture (**Table 89**). The observed differences may be due to variations in the composition of the two process streams at the time of sampling, considering the batch mode operation. The concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) were lower in the N-Viro products than in the feed sludge. The observed differences may be due to the high temperature during the biosolids drying process, which would drive off some ammonia and organic nitrogen containing compounds such as amines. The concentrations of total and ortho-phosphorus were observed to be similar, as expected during biosolids drying and curing processes.

Table 89. Nutrients in Feed Sludge after Admixture and N-Viro Products after Final Curing, Halifax, NS

Parameter	Concentration (mg/kg TS dw)	
	Feed Sludge after Admixture	N-Viro after Final Curing
Nitrate-N	<5.0	31.9
Nitrite-N	<5.0	<5.0
Total Kjeldahl Nitrogen	16500	12100
Ammonia as N	2360	1280
Phosphorus, Total	6570	5180
Phosphate-P (ortho)	0.626	0.795
Total Solids	437000	675000

4.11.3.2 Metals

All the metals except Cadmium were observed at detectable concentrations in both feed sludge to the N-Viro system and the finished N-Viro product, as shown in **Table 90**. Zinc and copper were detected at the highest concentrations in both feed sludge and finished product. Mercury had the lowest detectable concentration of the metals examined.

Table 90. Metals in Feed Sludge after Admixture and N-Viro Products after Final Curing, Halifax, NS

Metal	Concentration (mg/kg TS dw)	
	Feed Sludge after Admixture	N-Viro after Final Curing
Arsenic (As)-Total	**5.2**	**6.7**
Cadmium (Cd)-Total	<1.0	<1.0
Chromium (Cr)-Total	**9.0**	**11.3**
Cobalt (Co)-Total	**2.4**	**2.9**
Copper (Cu)-Total	**111**	**108**
Lead (Pb)-Total	**57.9**	**55.5**
Mercury (Hg)-Total	**0.150**	**0.279**
Molybdenum (Mo)-Total	**2.7**	**3.3**
Nickel (Ni)-Total	**8.4**	**8.3**
Selenium (Se)-Total	**5.9**	**3.0**
Zinc (Zn)-Total	**231**	**224**
Total Solids	437000	675000

Samples in **bold** font are above the detection limit

Note: The Halifax treatment plant was not operational at the time that samples were collected. At the time of sampling the process stream was primarily composed of septage solids, which typically have elevated selenium content.

4.11.3.3 Pharmaceuticals

The frequency of detection and median detected concentrations of the pharmaceutical compounds in the sludge feed after admixture and N-Viro product after final curing at the Halifax N-Viro

facility are presented in **Table 91**. The raw analytical data are provided in **Appendix Table A19**. A total of 19 pharmaceuticals were detected in the sludge feed samples in all three sampling campaigns; 14 pharmaceuticals were detected in N-Viro after final curing samples from the three campaigns. The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the N-Viro product were the anti-microbials triclosan and triclocarban. Slightly lower concentrations were found for the antibiotic ciprofloxacin and the non-steroidal anti-inflammatory ibuprofen.

Table 91. Frequency of Detection and Median Concentrations of Pharmaceutical Compounds in Feed Sludge after Admixture and N-Viro Product after Final Curing, Halifax, NS

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Sludge Feed After Admixture	N-Viro after Final Curing	Sludge Feed After Admixture	N-Viro after Final Curing	Sludge Feed After Admixture	N-viro after Final Curing
Furosemide	1	1	137 [a]	259 [a]	<89.4-137	<153-259
Gemfibrozil	**3**	**3**	**12.2**	**13.8**	10.1-12.4	9.86-21.9
Glipizide	0	0	NA	NA	<22.7 [b]	<23 [b]
Glyburide	1	0	12[a]	NA	<5.42-12	<11.5 [b]
Hydrochlorothiazide	1	1	166 [a]	91.4 [a]	<36.2-166	<40.5-91.4
2-Hydroxy-ibuprofen	2	1	200	189 [a]	<145-228	<162-189
Ibuprofen	**3**	**3**	**319**	**522**	315-623	369-528
Naproxen	**3**	**3**	**169**	**178**	155-169	126-212
Triclocarban	**3**	**3**	**3780**	**1590**	1540-9200	1260-1790
Triclosan	**3**	**3**	**7700**	**6120**	5730-11500	4780-6520
Warfarin	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Acetaminophen	0	0	NA	NA	<543 [b]	<230 [b]
Azithromycin	**3**	**3**	**349**	**36.8**	223-469	5.27-157
Caffeine	**3**	**3**	**355**	**240**	334-1120	143-386
Carbadox	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Carbamazepine	**3**	**3**	**137**	**79.4**	114-349	40.7-100
Cefotaxime	0	0	NA	NA	<129 [b]	<161 [b]
Ciprofloxacin	**3**	**3**	**1170**	**587**	724-1840	560-605
Clarithromycin	**3**	1	31.1	11.5 [a]	19.4-50.8	<3.05-11.5
Clinafloxacin	1	0	17 [a]	NA	<11.8-17	<67 [b]
Cloxacillin	0	0	NA	NA	<11.4 [b]	<11.5 [b]
Dehydronifedipine	**3**	2	2.4	2.36	1.29-3.01	<1.22-2.79
Diphenhydramine	**3**	**3**	**656**	**140**	298-900	87.4-216
Diltiazem	**3**	0	2.79	NA	0.66-3.86	<1.15 [b]
Digoxin	0	0	NA	NA	<56.8 [b]	<57.4 [b]
Digoxigenin	0	0	NA	NA	<94.4 [b]	<69.4 [b]
Enrofloxacin	1	0	12.6 [a]	NA	<5.44-12.6	<24.8
Erythromycin-H_2O	**3**	**3**	**22**	**8.88**	12.5-32.5	6.02-14.6
Flumequine	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Fluoxetine	**3**	2	23.3	9.23	23-48.3	<3.05-9.67

(continued)

Table 91 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Sludge Feed After Admixture	N-Viro after Final Curing	Sludge Feed After Admixture	N-Viro after Final Curing	Sludge Feed After Admixture	N-viro after Final Curing
Lincomycin	0	0	NA	NA	<25.3 [b]	<24.6 [b]
Lomefloxacin	0	0	NA	NA	<11.4 [b]	<13.7 [b]
Miconazole	**3**	**3**	**517**	**319**	448-664	230-400
Norfloxacin	**3**	2	105	99	84.9-218	<30.5-99.2
Norgestimate	0	0	NA	NA	<16.6 [b]	<15.3 [b]
Ofloxacin	**3**	**3**	**206**	**276**	121-399	125-325
Ormetoprim	0	0	NA	NA	<2.27 [b]	<2.27 [b]
Oxacillin	0	0	NA	NA	<11.4 [b]	<11.5 [b]
Oxolinic Acid	0	0	NA	NA	<2.8 [b]	<2.9 [b]
Penicillin G	0	0	NA	NA	<11.4 [b]	<11.5 [b]
Penicillin V	0	0	NA	NA	<11.4 [b]	<11.5 [b]
Roxithromycin	0	0	NA	NA	<1.30 [b]	<1.79 [b]
Sarafloxacin	0	0	NA	NA	<170 [b]	<279 [b]
Sulfachloropyridazine	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Sulfadiazine	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Sulfadimethoxine	0	0	NA	NA	<1.41 [b]	<6.64 [b]
Sulfamerazine	0	0	NA	NA	<3.08 [b]	<2.33 [b]
Sulfamethazine	0	0	NA	NA	<3.73 [b]	<4.72 [b]
Sulfamethizole	0	0	NA	NA	<2.84 [b]	<3.97 [b]
Sulfamethoxazole	2	1	2.07	2.22 [a]	<1.09-2.48	<1.22-2.22
Sulfanilamide	0	1	NA	49 [a]	<56.8 [b]	<30.5-49
Sulfathiazole	0	0	NA	NA	<5.68 [b]	<5.74 [b]
Thiabendazole	**3**	**3**	**6.67**	**7.7**	5.93-12.4	5.61-8.03
Trimethoprim	2	1	20.5	17.2 [a]	<3.71-33.1	<11.6-17.2
Tylosin	0	0	NA	NA	<127 [b]	<154 [b]
Virginiamycin	1	1	309 [a]	409 [a]	<54.4-309	<90.3-409
1,7-Dimethylxanthine	2	1	517	378 [a]	<272-727	<305-378

[a] indicates median value is from one detectable concentration only
[b] indicates highest identified detection limit for compound
Data in **bold font** are detected in all three sampling campaigns
NA = not applicable (no median for all non-detectable concentrations)

The distribution of detectable concentrations in the sludge feed after admixture and N-Viro after final curing samples from the three sampling campaigns is found in **Table 92**. The distribution of detectable concentrations in the digester feed is somewhat different compared to the finished stabilised product. The number of compounds detected in all three campaigns declines from 19 in the sludge feed samples to 14 in the N-Viro after final curing samples, while the number of compounds never detected in any of the three campaigns rises from 28 in sludge feed samples to 31 after final curing.

Table 92. Summary of Pharmaceutical Compound Detections Metals in Feed Sludge after Admixture and Alkaline Stabilised Product after Final Curing, Halifax, NS

# Detects in process stream for 3 sampling campaigns	# Compounds in Process Streams	
	Sludge Feed After Admixture	N-viro after Final Curing
3	19	14
2	4	3
1	6	9
0	28	31
Total	57	57

4.11.3.4 Fragrance and Alkylphenolic Compounds

Concentration data for the fragrance and alkylphenolic compounds are provided in **Table 93**. The raw analytical data are provided in **Appendix Table A20**. Bisphenol A was the only alkylphenolic compound detected at this site. BPA was detected in the feed sludge after admixture, and in the N-Viro product after curing, in both sampling campaigns. The median value of BPA in the product at 790 ng/g TS dw was substantially higher than was the

Table 93. Frequency of Detection and Median and Range of Detected Concentrations of Fragrance and Alkylphenolic Compounds in Feed Sludge after Admixture and Alkaline Stabilised Product after Final Curing, Halifax, NS

Compound	Frequency of Detection in Sampling Campaigns (out of 2)		Median of Detected Concentrations (ng/g TS dw)		Range of Detected Concentrations (ng/g TS dw)	
	Sludge Feed After Admixture	N-Viro Product after Final Curing	Sludge Feed After Admixture	N-Viro Product after Final Curing	Sludge Feed After Admixture	N-Viro Product after Final Curing
Alkylphenolics						
Bisphenol A	**2**	**2**	**200**	**790**	110-290	770-810
Octylphenol	0	0	NA	NA	<20	<20
Nonylphenol	0	0	NA	NA	<140	<140
Fragrances						
DPMI	**2**	1	**55**	50	50-60	<40-50
ADBI	0	0	NA	NA	<20	<20
AHDI	1	0	70	NA	<30-70	<30
HHCB	**2**	**2**	**3750**	**4115**	3090-4410	2880-5350
AHTN	**2**	**2**	**480**	**690**	300-660	620-760
ATII	**2**	**2**	**110**	**110**	90-130	70-150
Musk Moskene	0	0	NA	NA	<50	<50
Musk Tibetene	0	0	NA	NA	<80	<80
Musk Ketone	0	0	NA	NA	0.04-60	<120
Musk Ambrette	0	0	NA	NA	<140	<140
Musk Xylene	0	0	NA	NA	<70	<70

Data in **bold font** are detected in both sampling campaigns

median concentration in the sludge feed (200 ng/g TS dw). The difference is most likely due to the temporal difference in the composition of the various sludge streams that are combined as the process feed. No nitro musks were detected in any samples in either of the two sampling campaigns. The polycyclic musk HHCB was observed at the highest median concentration of 4115 ng/g TS dw in the finished biosolids product. The other synthetic musks detected in the finished product were observed at substantially lower median concentrations ranging from 50 to 690 ng/g TS dw. The concentrations of the fragrances in the finished biosolids product were similar but slightly elevated relative to the concentrations in the feed sludge samples.

4.11.4 Data Interpretation

4.11.4.1 Total Solids Mass Balance Estimate

Concentrations for metals and pharmaceutical compounds are expressed on a dry weight basis (i.e., mg/kg TS dw for metals, ng/g TS dw for pharmaceuticals), and so the mass balances for the both types of contaminants are based on a total solids balance around the alkaline stabilisation process. The solids balance around the N-Viro process is estimated using the mean values of the total solids concentrations in the feed dewatered sludge cake (following alkali amendment) and cured, stabilised biosolids out of the process from the three sampling campaigns. The pertinent solids concentration and flow data are:

Mass feed rate of dewatered cake = 9 wet tonnes/h
Mean measured total solids concentration in dewatered cake feed = 48.1% TS
Mass exit rate of cured, stabilised product = 7 wet tonnes/h
Mean measured total solids concentration in dried biosolids pellets = 68.6 % TS

In the balance, it was assumed the difference in the mass of dewatered cake solids entering and product leaving the process was the mass of water evaporated through the process. The total solids balance is depicted in **Figure 19**. The calculated masses of solids in and out of the process are approximately equal; the higher mass of solids leaving the drier than the mass entering the drier is considered an artifact of the variability associated with sampling sludge and biosolids, and estimating the flow rates.

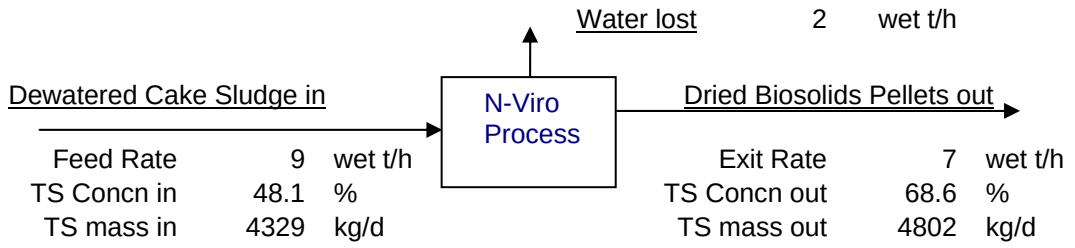

Figure 19. Total Solids Balance around N-Viro Alkaline Stabilisation Process, Halifax, NS

4.11.4.2 Metals

The mass balance closures for metals around the N-Viro process is provided in **Table 94**. The mass closures ranged from a low of 56% for selenium to a high value of 206% for mercury. With

the exception of selenium, the mass closure values were all greater than 100%, suggesting the more of the metal mass was measured in the final product than in the feed. Possible reasons for this observation include differences in composition between the dewatered sludge feed and final product, and possible inaccuracies in the estimated feed and product mass flow rates.

Table 94. Mass Balance Closures for Metals in N-Viro Alkaline Stabilisation Process, Halifax, NS

Metal	Concentration (mg/kg TS dw)		Mass of Contaminant (g/h)		Mass Closure (%)
	Sludge Feed after Admixture	N-Viro after Final Curing	Sludge Feed after Admixture	N-Viro after Final Curing	
Arsenic (As)-Total	5.2	6.7	22.5	32.2	143%
Cadmium (Cd)-Total	<1.0	<1.0			
Chromium (Cr)-Total	9.0	11.3	39.0	54.3	139%
Cobalt (Co)-Total	2.4	2.9	10.4	13.9	134%
Copper (Cu)-Total	111	108	480.5	518.6	108%
Lead (Pb)-Total	57.9	55.5	250.6	266.5	106%
Mercury (Hg)	0.150	0.279	0.6	1.3	206%
Molybdenum (Mo)-Total	2.7	3.3	11.7	15.8	136%
Nickel (Ni)-Total	8.4	8.3	36.4	39.9	110%
Selenium (Se)-Total	5.9	3.0	25.5	14.4	56%
Zinc (Zn)-Total	231	224	1,000.0	1,075.6	108%

4.11.4.3 Pharmaceuticals

Concentrations of the pharmaceutical compounds measured on a dry weight basis (i.e. ng/g TS dw) were converted to a mass flow rate (mg/d) for comparison of input and output masses. The results of the mass estimates are provided in **Table 95.** Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids were not included in **Table 95**.

Compounds that are removed to the greatest extent through the alkaline stabilisation process include the antibiotic azithromycin (88%), the stimulant diphenhydramine (76%) and the anti-angina medicine diltiazem (>76%). No compounds are removed in excess of 90% by the process. Among the poorest removal efficiencies (i.e., the mass out of the process is greater than the mass in) are the diuretic furosemide and the non-steroidal anti-inflammatory drug ibuprofen.

Table 95. Mass Balance and Removal Calculations for Pharmaceutical Compounds in N-Viro Alkaline Stabilisation Process, Halifax, NS

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/h)		% Removal
	Sludge Feed After Admixture	N-viro Product after Final Curing	Sludge Feed After Admixture	N-viro Product after Final Curing	
Furosemide	137	259	593	1244	-110%
Gemfibrozil	12.2	13.8	52.8	66.3	-25%
Glyburide	12	<6.15	51.95	<29.5	>43%
Hydrochlorothiazide	166	91.4	719	439	39%
2-Hydroxy-ibuprofen	200	189	866	908	-5%
Ibuprofen	319	522	1381	2507	-82%
Naproxen	169	178	732	855	-17%
Triclocarban	3780	1590	16364	7635	53%
Triclosan	7700	6120	33333	29388	12%
Azithromycin	349	36.8	1511	177	88%
Caffeine	355	240	1537	1152	25%
Carbamazepine	137	79.4	593	381	36%
Ciprofloxacin	1170	587	5065	2819	44%
Clarithromycin	31.1	11.5	135	55.2	59%
Clinafloxacin	17	<23	73.6	<110	>-50%
Dehydronifedipine	2.4	2.36	10.4	11.3	-9%
Diphenhydramine	656	140	2840	672	76%
Diltiazem	2.79	<0.615	12.1	<2.95	>76%
Enrofloxacin	12.6	<11.5	54.5	<55.2	>-1%
Erythromycin-H_2O	22	8.88	95.2	42.6	55%
Fluoxetine	23.3	9.23	101	44.3	56%
Miconazole	517	319	2238	1532	32%
Norfloxacin	105	99	455	475	-5%
Ofloxacin	206	276	892	1325	-49%
Sulfamethoxazole	2.07	2.22	9.0	10.7	-19%
Thiabendazole	6.67	7.7	28.9	37.0	-28%
Trimethoprim	20.495	17.2	88.7	82.6	7%
Virginiamycin	309	409	1338	1964	-47%
1,7-Dimethylxanthine	516.5	378	2236	1815	19%

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 96**. A total of seven compounds were reduced by between 50 to 89% by the process. As many pharmaceutical compounds were removed in the 0 to 49% efficiency range as there were compounds removed in the -49 to -1% range.

Table 96. Categorised Removal Efficiencies of Pharmaceutical Compounds by N-Viro Alkaline Stabilisation Process, Halifax, NS

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Furosemide	Gemfibrozil	Glyburide	Triclocarban	
Ibuprofen	2-Hydroxy-ibuprofen	Hydrochlorothiazide	Azithromycin	
	Naproxen	Triclosan	Clarithromycin	
	Clinafloxacin	Caffeine	Diphenhydramine	
	Dehydronifedipine	Carbamazepine	Diltiazem	
	Norfloxacin	Ciprofloxacin	Erythromycin-H_2O	
	Ofloxacin	Enrofloxacin	Fluoxetine	
	Sulfamethoxazole	Miconazole		
	Thiabendazole	Trimethoprim		
	Virginiamycin	1,7-Dimethylxanthine		
n=2	n=10	n=10	n=7	n=0

4.11.4.4 Fragrance and Alkylphenolic Compounds

The mass balance and removal calculations for BPA and the detected synthetic musk fragrances appear in **Table 97**. The observed higher concentration of Bisphenol A in the finished product relative to the feed results in a negative removal efficiency. As explained earlier in this Section, the difference between feed and product masses is likely due to the variability in the mixture of the different feed sludges. The polycyclic musk compounds also exhibited negative removal efficiencies, although to a lesser extent than for BPA. The probable reason for the negative removal efficiencies calculated for the fragrances is the same as that for BPA.

Table 97. Mass Balance and Removal Calculations for Alkylphenolic and Fragrance Compounds in N-Viro Alkaline Stabilisation Process, Halifax, NS

Pharmaceutical	Median of Detected Concentrations (ng/g TS dw)		Mass of Contaminants (mg/h)		% Removal
	Sludge Feed After Admixture	N-viro Product after Final Curing	Sludge Feed After Admixture	N-viro Product after Final Curing	
Alkylphenolics					
Bisphenol A	200	790	866	3794	-338%
Fragrances					
DPMI	55	50	238	240	-1%
AHDI	70	<30	303	<144	>52%
HHCB	3750	4115	16234	19760	-22%
AHTN	480	690	2078	3313	-59%
ATII	110	110	476	528	-11%

4.11.4.5 Effectiveness of Process for ESOC Removal

Based on the categorised removal efficiencies in **Table 96**, there appears to be a modest removal of the pharmaceutical compounds. There is a higher number of compounds (17) with positive removal efficiencies greater than 0 than the number (12) with removal efficiencies less than 0. Seven pharmaceuticals have removal efficiencies between 50% and 90%. The removal mechanism for pharmaceuticals cannot be determined from the collected data, but may include chemical reaction at the higher pH caused by the addition of alkaline material, temperature-mediated chemical reaction or breakdown, or volatilization from the mixture during the period of elevated temperatures in the process. There is no comparable positive removal of Bisphenol A or any of the polycyclic musk fragrances, which appear to be unaffected by the stabilisation process.

4.11.5 Section Summary

A higher concentration of nitrate-N was observed in the N-Viro product than in feed sludge after admixture. The concentrations of total Kjeldahl nitrogen (TKN) and ammonia-N (a component of TKN together with organic-N) were lower in the N-Viro products than in the feed sludge. The concentrations of total and ortho-phosphorus were observed similar or conservative, as expected during biosolids drying and curing processes.

All the metals except cadmium were observed at detectable concentrations in both feed sludge to the N-Viro system and the finished N-Viro product. Zinc and copper were detected at the highest concentrations in both feed sludge and finished product. Mercury had the lowest detectable concentration of the metals examined. The mass balance closures for the metals ranged from a low of 56% for selenium to a high value of 206% for mercury. With the exception of selenium, the mass closure values were all greater than 100%, suggesting the more of the metal mass was measured in the final product than in the feed. Possible reasons for this observation include differences in composition between the dewatered sludge feed and final product, and possible inaccuracies in the estimated feed and product mass flow rates.

A total of 19 pharmaceuticals were detected in the sludge feed samples in all three sampling campaigns; 14 pharmaceuticals were detected in N-Viro after final curing samples from the three campaigns. The compounds detected at the highest concentrations (above 1,000 ng/g TS) in the N-Viro product were the anti-microbials triclosan and triclocarban. Slightly lower concentrations were found for the antibiotic ciprofloxacin and the non-steroidal anti-inflammatory ibuprofen. Compounds that are removed to the greatest extent through the alkaline stabilisation process include the antibiotic azithromycin (88%), the stimulant diphenhydramine (76%) and the anti-angina medicine diltiazem (>76%). Among the poorest removal efficiencies (i.e., the mass out of the process is greater than the mass in) are the diuretic furosemide and the non-steroidal anti-inflammatory drug ibuprofen.

Bisphenol A was the only alkylphenolic compound detected at this site. BPA was detected in the feed sludge after admixture, and in the N-Viro product after curing, in both sampling campaigns. The median value of BPA in the product at 790 ng/g TS was substantially higher than was the median concentration in the sludge feed (200 ng/g TS). The difference is most likely due to the temporal difference in the composition of the various sludge streams that are combined as the process feed. The observed higher concentration of Bisphenol A in the finished product relative

to the feed results in a negative removal efficiency. No nitro musks were detected in any samples in either of the two sampling campaigns. The polycyclic musk HHCB was observed at the highest median concentration of 4115 ng/g TS in the finished biosolids product. The other synthetic musks detected in the finished product were observed at substantially lower median concentrations ranging from 50 to 690 ng/g TS. The concentrations of the fragrances in the finished biosolids product were similar but slightly elevated relative to the concentrations in the feed sludge samples. The polycyclic musk compounds also exhibited negative removal efficiencies, although to a lesser extent than for BPA. The probable reason for the negative removal efficiencies calculated for the fragrances is the same as that for BPA discussed above.

There appears to be a modest net benefit to the process for removal of the pharmaceutical compounds. There is a higher number of compounds (17) with positive removal efficiencies greater than 0 than the number (12) with removal efficiencies less than 0. Seven pharmaceuticals have removal efficiencies between 50% and 90%. No compounds are removed in excess of 90% by the process. There is no comparable positive removal of Bisphenol A or any of the polycyclic musk fragrances, which appear to be unaffected by the stabilisation process.

4.12 Filter Press Sludge Dewatering, Gander, NL

4.12.1 Site Description

The Gander facility (the Beaverwood Sewage Treatment Plant), is a hydrodynamic separator facility with chlorination disinfection of the treated effluent prior to discharge to the Gander Lake via Soulis Brook. The design capacity of the existing treatment plant is 80,000 m^3/d, while the average daily dry weather flow is 16,000 m^3/d.

4.12.2 Belt Filter Press Dewatering Process Description

The primary sludge from hydrodynamic separator is sent to the sludge thickening tank for thickening. The supernatant from the sludge thickening process is returned to the plant headwork for treatment. The flow rate of supernatant is not measured by the plant due to the fact that raw water is also introduced at this point for clearing processes making the calculations complex. The thickened primary sludge is pumped to the sludge holding tank, from where it is sent to the belt press for dewatering. The pumping rate of feed sludge to the belt press is about 21.6 $m^{3/}$d. During this study, the solids concentration in the belt press feed (i.e. thickened primary sludge) ranged from 0.5% to 3.3%, while the dewatered biosolids cake solids concentration ranged from 8.9% to 14.4%. The dewatered biosolids cake is sent to agricultural lands. The filtrate from the dewatering of primary sludge is directed back to the headworks of the treatment plant.
For this project assessment, the treatment process of interest was the belt press dewatering process. The three sampling locations included the belt press feed (i.e. thickened primary sludge), dewatered biosolids cake and filtrate. A process schematic of the Gander biosolids treatment process is shown in **Figure 20**.

The plant was considered by plant staff to be in normal operation during the three sampling campaigns. Samples were collected and shipped to the analytical laboratories on June 30, July 16 and August 13, respectively.

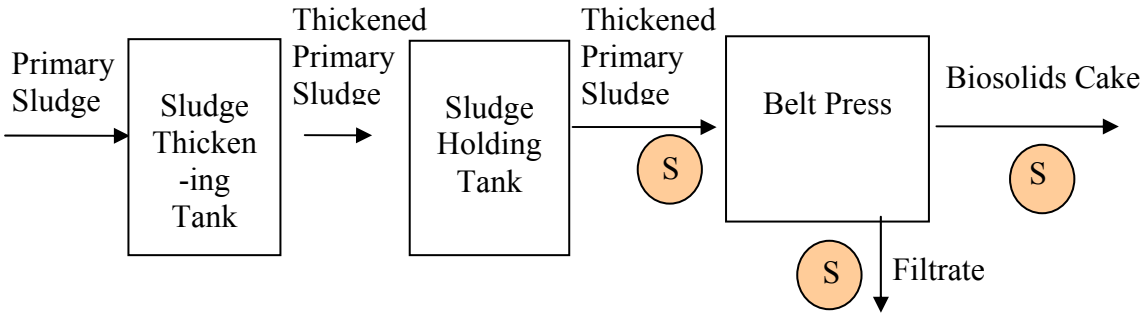

Figure 20. Schematic of Gander Biosolids Process and Sampling Locations

4.12.3 Sampling Results

4.12.3.1 Nutrients

The nutrient data as reported by the laboratory could not be directly compared because of the different concentration units used, for liquid samples (WAS and filtrate) and solids samples (dewatered biosolids cake). A comparison of the mostly soluble components of the thickened primary sludge and belt press filtrate is of interest for the observed changes. Both ammonia-N and ortho-phosphate are substantially reduced in the filtrate compared to the feed primary sludge (**Table 98**), suggesting that some physical or chemical processes may have occurred to reduce the concentrations through the dewatering process. Although the concentrations of TKN and total P also decrease substantially in the filtrate compared to the feed primary sludge, these components may be associated with the solid phase of the sludge and may thus have accumulated in the dewatered biosolids cake.

Table 98. Nutrients in Thickened Primary Sludge, Dewatered Biosolids Cake and Belt Press Filtrate, Gander, NL

Parameter	Concentration		
	Thickened primary sludge (mg/L)	Dewatered biosolids (mg/kg TS dw)	Belt Press Filtrate (mg/L)
Nitrate-N	<2.0	16.7	0.18
Nitrite-N	<2.0	<1.0	<0.10
Total Kjeldahl Nitrogen	174	20600	10.1
Ammonia as N	13.6	2830	4.04
Phosphorus, Total	29.5	3190	1.96
Phosphate-P (ortho)	7.08	195	0.417
Total Solids	7170	91000	160

4.12.3.2 Metals

Several metals are identified above the detection limits in thickened primary sludge, including chromium, copper, lead, mercury, and zinc (**Table 99**). All the metals (except cadmium) studied are detected in the dewatered biosolids. Copper is the only metal detected in the filtrate. Copper and zinc and are observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. Additional discussion of the metals is found later in this section under Data Interpretation.

Table 99. Metals in Thickened Primary Sludge, Dewatered Biosolids Cake and Filtrate, Gander, NL

Parameter	Concentration		
	Thickened primary sludge (mg/L)	Dewatered biosolids (mg/kg TS dw)	Filtrate (mg/L)
Arsenic (As)-Total	<0.10	**6.6**	<0.010
Cadmium (Cd)-Total	<0.010	<1.0	<0.0010
Chromium (Cr)-Total	**0.27**	**44.4**	<0.010
Cobalt (Co)-Total	<0.080	**1.7**	<0.0080
Copper (Cu)-Total	**5.92**	**890**	**0.093**
Lead (Pb)-Total	**0.19**	**37.5**	<0.010
Mercury (Hg)-Total	**0.00856**	**1.13**	<0.00010
Molybdenum (Mo)-Total	<0.10	**3.7**	<0.010
Nickel (Ni)-Total	<0.20	**9.9**	<0.020
Selenium (Se)-Total	<0.50	**1.4**	<0.050
Zinc (Zn)-Total	**1.38**	**331**	<0.030
Total Solids	7170	91000	160

Samples in **bold** font are above the detection limit

4.12.3.3 Pharmaceuticals

The frequency of detection and median and range of detected concentrations of the pharmaceutical compounds in the thickened primary sludge, dewatered cake and filtrate at the Gander facility are presented in **Table 100**. The raw concentration data from the three sampling campaigns are found in **Appendix Table A21**.

A total of 20 pharmaceuticals were detected in both the thickened primary sludge and the dewatered cake samples from all the three sampling campaigns; 19 pharmaceuticals were detected in the filtrate samples from all the three campaigns. Three pharmaceutical compounds or metabolites (hydrochlorothiazide, 2-hydroxy-ibuprofen and 1,7-dimethylxanthine) were detected in all three campaigns in the belt press filtrate, although they were never detected in either the primary sludge feed or the dewatered cake.

Table 100. Frequency of Detection and Median Concentrations of Pharmaceutical Compounds in Thickened Primary Sludge, Dewatered Biosolids Cake and Filtrate, Gander, NL

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Thickened Primary Sludge	Biosolids Cake	Filtrate	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)
Furosemide	0	2	1	<318	1630	270 [a]	<1770 [b]	<74.8-3030	<40.1-270
Gemfibrozil	1	0	0	13.1 [a]	NA	NA	<8.72-13.1	<17.3 [b]	<6.15 [b]
Glipizide	0	0	0	NA	NA	NA	<265 [b]	<51.3 [b]	<24 [b]
Glyburide	0	1	0	NA	16.3 [a]	NA	<133 [b]	<11.5-16.3	<12 [b]
Hydrochlorothiazide	0	0	**3**	NA	NA	**435**	<885 [b]	<230 [b]	410-474
2-Hydroxy-ibuprofen	0	0	**3**	NA	NA	**4660**	<3540 [b]	<921 [b]	4230-4880
Ibuprofen	**3**	**3**	**3**	**459**	**319**	**2130**	395-1630	304-380	1670-2320
Naproxen	**3**	**3**	**3**	**178**	**98.1**	**690**	79.7-405	55.8-111	608-695
Triclocarban	**3**	**3**	**3**	**2670**	**2470**	**15.3**	2280-3110	1880-2470	14.6-18.6
Triclosan	**3**	**3**	1	**11700**	**9560**	241 [a]	11400-13300	9240-20300	<234-241
Warfarin	0	0	0	NA	NA	NA	<66.3 [b]	<12.8 [b]	<5.99 [b]
Acetaminophen	**3**	0	**3**	**1170**	NA	**16600**	772-11300	<508 [b]	14900-19800
Azithromycin	**3**	**3**	**3**	**237**	**220**	**49.9**	95.7-252	146-248	47.5-61.3
Caffeine	**3**	**3**	**3**	**2590**	**1160**	**10200**	1990-7910	1130-1170	10100-12700
Carbadox	0	0	0	NA	NA	NA	<66.4 [b]	<12.7 [b]	<5.85 [b]
Carbamazepine	**3**	**3**	**3**	**275**	**214**	**1300**	122-858	111-403	835-1350
Cefotaxime	0	0	0	NA	NA	NA	<2610 [b]	<159 [b]	<48.2 [b]
Ciprofloxacin	**3**	**3**	**3**	**17200**	**16900**	**108**	16000-18100	13100-19100	91.4-109
Clarithromycin	**3**	**3**	**3**	**92.4**	**97.2**	**147**	69.1-95.6	30-147	61.4-194
Clinafloxacin	0	0	0	NA	NA	NA	<265 [b]	<50.8 [b]	<65.3 [b]
Cloxacillin	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<12.6 [b]
Dehydronifedipine	1	**3**	**3**	7.66 [a]	**8.42**	**82**	<3.49-7.66	7.09-12.6	55.1-132
Diphenhydramine	**3**	**3**	**3**	**222**	**205**	**145**	171-499	186-251	109-201
Diltiazem	**3**	**3**	**3**	**348**	**257**	**316**	145-563	254-600	294-337

(continued)

Table 100 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Thickened Primary Sludge	Biosolids Cake	Filtrate	Thickened Primary Sludge (ng/g TS)	Biosolids Cake (ng/g TS)	Filtrate (ng/L)	Thickened Primary Sludge (ng/g TS)	Biosolids Cake (ng/g TS)	Filtrate (ng/L)
Digoxin	0	0	0	NA	NA	NA	<664 [b]	<156 [b]	<58.5 [b]
Digoxigenin	0	0	0	NA	NA	NA	<265 [b]	<67.9 [b]	<83.3 [b]
Enrofloxacin	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<18.9 [b]
Erythromycin-H_2O	**3**	**3**	**3**	**28.7**	**32.9**	**53.3**	18.1-30.1	17.1-34.9	43.1-93.3
Flumequine	0	0	0	NA	NA	NA	<66.4 [b]	<12.7 [b]	<9.99 [b]
Fluoxetine	**3**	**3**	1	**36.1**	**51.9**	9.29 [a]	33-109	41-53	<5.37-9.29
Lincomycin	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<40.8 [b]
Lomefloxacin	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<11.7 [b]
Miconazole	**3**	**3**	0	**463**	**441**	NA	341-712	207-529	<5.85 [b]
Norfloxacin	**3**	**3**	2	**2010**	**2390**	379	1640-2520	2120-3700	<53.7-463
Norgestimate	0	0	0	NA	NA	NA	<135 [b]	<30.9 [b]	<12.6 [b]
Ofloxacin	**3**	**3**	0	**2590**	**2670**	<58.4	2080-5140	2260-2710	<58.5 [b]
Ormetoprim	0	0	0	NA	NA	NA	<26.5 [b]	<5.08 [b]	<2.34 [b]
Oxacillin	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<11.7 [b]
Oxolinic Acid	0	0	0	NA	NA	NA	<35.1 [b]	<5.08 [b]	<2.34 [b]
Penicillin G	0	0	0	NA	NA	NA	<133 [b]	<38.4 [b]	<11.7 [b]
Penicillin V	0	0	0	NA	NA	NA	<133 [b]	<25.4 [b]	<11.7 [b]
Roxithromycin	0	1	0	NA	0.76 [a]	NA	<13.3 [b]	<0.637-0.755	<1.17 [b]
Sarafloxacin	0	0	0	NA	NA	NA	<664 [b]	<127 [b]	<58.5 [b]
Sulfachloropyridazine	0	0	0	NA	NA	NA	<66.4 [b]	<12.7 [b]	<5.85 [b]
Sulfadiazine	0	0	0	NA	NA	NA	<66.4 [b]	<12.7 [b]	<5.85 [b]
Sulfadimethoxine	0	0	0	NA	NA	NA	<13.3 [b]	<2.54 [b]	<3.53 [b]
Sulfamerazine	0	0	2	NA	NA	13.1	<26.5 [b]	<5.08 [b]	<2.15-21.4
Sulfamethazine	0	0	1	NA	NA	8.3 [a]	<26.5 [b]	<5.08 [b]	<2.15-8.3
Sulfamethizole	0	0	0	NA	NA	NA	<26.5 [b]	<5.08 [b]	<2.34 [b]
Sulfamethoxazole	**3**	**3**	**3**	**17.6**	**5.17**	**80.5**	4.32-156	3.46-7.65	61.6-96.6

(continued)

Table 100 (continued)

Pharmaceutical	Frequency of Detection in Sampling Campaigns (out of 3)			Median of Detected Concentration			Range of Detected Concentration		
	Thickened Primary Sludge	Biosolids Cake	Filtrate	Thickened Primary Sludge (ng/g TS)	Biosolids Cake (ng/g TS)	Filtrate (ng/L)	Thickened Primary Sludge (ng/g TS)	Biosolids Cake (ng/g TS)	Filtrate (ng/L)
Sulfanilamide	0	0	0	NA	NA	NA	<664 [b]	<127 [b]	<58.5 [b]
Sulfathiazole	0	0	0	NA	NA	NA	<66.4 [b]	<12.7 [b]	<5.85 [b]
Thiabendazole	**3**	**3**	**3**	**44.1**	**50.9**	**16.9**	34.6-95.6	38.5-55.8	15.9-30.1
Trimethoprim	**3**	**3**	**3**	**92.9**	**76.7**	**149**	84.6-110	58-119	132-150
Tylosin	0	0	0	NA	NA	NA	<265 [b]	<50.8 [b]	<78 [b]
Virginiamycin	0	0	0	NA	NA	NA	<346 [b]	<89.6 [b]	<584 [b]
1,7-Dimethylxanthine	0	0	**3**	NA	NA	**2530**	<6640 [b]	<1270 [b]	2130-4470

[a] indicates median value is from one detectable concentration only; [b] indicates highest identified detection limit for compound

Data in **bold font** are detected in all three sampling campaigns; NA = not applicable (no median for all non-detectable concentrations)

The pharmaceutical compounds detected at the highest concentrations in the dewatered cake samples (i.e. at concentrations greater than 1,000 ng/g TS) included the antimicrobials triclosan and triclocarban, the antibiotics ciprofloxacin, norfloxacin and ofloxcacin, the stimulant caffeine and furosemide, a diuretic compound.

The distribution of detectable concentrations in the biosolids dewatering process feed and effluent streams from the three sampling campaigns is found in **Table 101**. The number of compounds detected in all three campaigns remains approximately the same (i.e., 19-20) in the dewatered primary sludge, dewatered biosolids cake and belt press filtrate. The same compounds are not consistently detected in these matrices however, as it was noted above that three pharmaceuticals or metabolites were observed in the press filtrate, but never in the primary sludge or dewatered cake. The number of non-detected compounds was similar in the thickened primary sludge and dewatered cake. The number of compounds never detected in the three press filtrate samples was lower, however, due to the compounds being detected in one or two of the sampling campaigns.

Table 101. Summary of Pharmaceutical Compound Detections in Aerobically Digested WAS, Dewatered Cake and Belt Press Filtrate in Gander, NL

Frequency of detection in sampling campaigns (out of 3)	# Compounds in Process Streams		
	Thickened Primary Sludge	Dewatered Cake	Belt Press Filtrate
3	20	20	19
2	0	1	2
1	2	2	4
0	35	34	32
Total	57	57	57

4.12.3.4 Fragrances and Alkylphenolic Compounds

The analytical results for fragrance and alkylphenolic compounds from the Gander facility are provided in **Table 102.** The raw analytical data for these compounds are provided in **Appendix Table A22**. The Bisphenol A concentration data exhibit substantial variability in the two sampling campaigns. Specifically, the data from the second sampling campaign (see **Table A22**) are much higher than the concentrations from the corresponding sample locations in Campaign 1, particularly the press filtrate collected during the second campaign. The variations in the BPA concentrations may be real resulting from an elevated industrial input to the wastewater treatment system. The possibility also exists, however, that the samples may have been inadvertently contaminated by the use of a sampling container, other than what was supplied for the study, which may have involved plastics manufactured with BPA. In any event, the reported BPA concentrations from the second sampling campaign are considered suspect.

Table 102. Concentrations of Fragrance and Alkylphenolic Compounds in Thickened Primary Sludge and Dewatered Cake (One Campaign), Gander, NL

Fragrance and Phenolic Compounds	Frequency of Detection in Sampling Campaigns (out of 2)			Median of Detected Concentration			Range of Detected Concentration		
	Thickened Primary Sludge	Biosolids Cake	Filtrate	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)
Alkylphenolics									
Bisphenol A	**2**	1	1	**375**	130	3650	130-620	<80-130	<50-3650
Octylphenol	0	0	0	NA	NA	NA	<20	<20	<10
Nonylphenol	0	0	0	NA	NA	NA	<140	<140	<90
Fragrances									
DPMI	**2**	1	0	**65**	50	NA	60-70	<40-50	<20
ADBI	0	0	1	NA	NA	20	<20	<20	<10-20
AHDI	1	1	0	730	330	NA	<30-730	<30-330	<10
HHCB	**2**	**2**	**2**	**1490**	**2800**	**460**	1040-1940	1780-3820	280-640
AHTN	**2**	**2**	**2**	**840**	**1340**	**255**	370-1310	630-2050	240-270
ATII	**2**	**2**	**2**	**220**	**255**	**115**	130-310	170-340	80-150
Musk Moskene	0	0	0	NA	NA	NA	<50	<50	<90
Musk Tibetene	0	0	0	NA	NA	NA	<80	<80	<50
Musk Ketone	0	0	1	NA	NA	90	<120	<120	<90-90
Musk Ambrette	0	0	0	NA	NA	NA	<140	<140	<20
Musk Xylene	0	0	0	NA	NA	NA	<70	<70	<20

Data in **bold font** are detected in both sampling campaigns

The polycyclic musk fragrances HHCB, AHTN and ATII were detected in all samples in both sampling campaigns. HHCB and AHTN were detected at the highest median concentrations (i.e., greater than 1000 ng/g TS dw) in the dewatered cake solids. None of the nitro musk compounds were observed above the limit of quantification in the primary sludge feed, dewatered cake or press filtrate. Some compounds were observed at higher concentrations in the dewatered cake than in the feed sludge, while the concentrations of other compounds in the dewatered cake were lower than in the feed sludge. Mass balances are required to determine whether the concentration differences are significant (see the Data Interpretation section for additional discussion).

4.12.4 Data Interpretation

4.12.4.1 Total Solids Mass Balance Estimate

The mass balance around the dewatering belt filter press is based on concentrations of metals and nutrients in the feed primary sludge expressed as a volumetric concentration, while pharmaceuticals in the feed sludge were expressed on a dry solids basis. Contaminant concentrations in dewatered cake samples were all expressed on a dry solids weight basis. Concentrations of contaminants in filter press filtrate were all expressed on a volumetric basis.

The volumetric flow rates of press filtrate and dewatered cake were not available from plant staff for the mass balance calculations. The volumetric flows of press filtrate and dewatered cake were developed from a solids balance around the filter press as shown in **Figure 21**.

Data used to develop the solids balance include:

Volumetric feed rate of primary sludge feed = 21.6 m^3/d
Total solids concentration in primary sludge feed = 7.17 kg/m^3
Total solids concentration in dewatered cake = 91 kg/m^3
Total solids concentration in press filtrate = 0.16 kg/m^3

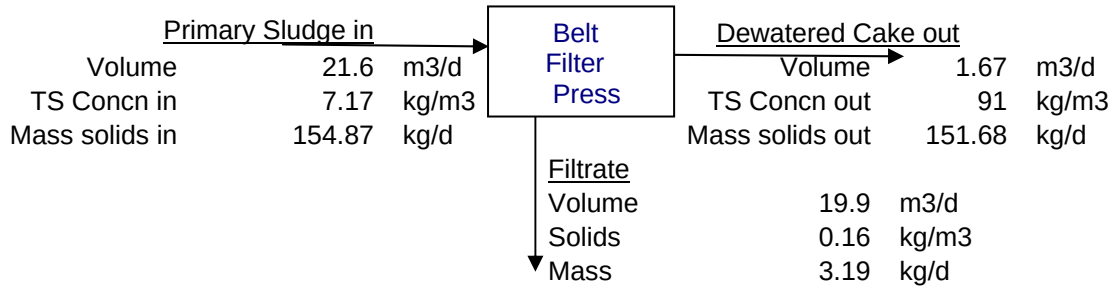

Figure 21. Solids Mass Balance around Belt Filter Press Dewatering Process, Gander, NL

4.12.4.2 Metals Mass Balances

Because copper was the only metal observed in the press filtrate at a detectable level, a full mass closure is only possible for that metals in **Table 103**. The estimated closure is 109% of the input mass. Four other metals are detected in the feed primary sludge and dewatered cake, but not the filtrate. Using the reported detection limit for each metal, it is possible to estimate a potential maximum mass in the filtrate, thereby establishing a potential mass closure range. This procedure reveals that the bulk of the feed metals resides in the dewatered cake, which would be as expected. The mass closures range from 96-97% for mercury to a high of 174-176% for zinc.

Table 103. Mass Balance Closure for Metals in Belt Filter Press Dewatering Process, Gander, NL

Nutrient	Concentration of contaminant			Mass of contaminant (g/d)			Mass Closure (%)
	Dewatered primary sludge (mg/L)	Dewatered biosolids (mg/kg TS dw)	Filtrate (mg/L)	Dewatered primary sludge	Dewatered biosolids	Filtrate	
Chromium (Cr)-Total	0.27	44.4	<0.010	5.83	6.95	<0.20	119%-<123%
Copper (Cu)-Total	5.92	890	0.093	127.9	139.3	1.9	109%
Lead (Pb)-Total	0.19	37.5	<0.010	4.10	5.87	<0.20	143%-<148%
Mercury (Hg)-Total	0.0086	1.13	<0.00010	0.185	0.177	<0.002	96%-<97%
Zinc (Zn)-Total	1.38	331	<0.030	29.8	51.8	<0.60	174%-<176%

4.12.4.3 Mass Balances for Pharmaceutical Compounds

Mass balances and removal efficiencies of the pharmaceutical compounds are presented in **Table 104**. Pharmaceutical compounds that were not detected in both the feed sludge and digested biosolids have not been included in **Table 104**.

Removal efficiencies through the belt filter press are low in comparison to many of the other biosolids treatment processes investigated in this survey. The belt filter press is a dewatering process, and is not intended to stabilise the organic matter or reduce pathogens or vector attraction. The calculated highest removal efficiency is 13.7% for the cardiac drug diltiazem. The lowest calculated removal efficiency of -185% is observed for the anti-anginal metabolite dehydronifedipine. The calculated masses of the pharmaceuticals in the primary sludge feed, dewatered cake and filtrate samples indicates that in most cases, the dewatered cake is the repository for the compounds entering the process in the feed sludge. For a few compounds, such as the non-steroidal anti-inflammatory compounds ibuprofen and naproxen, and the antibiotic sulfamethoxazole, as much of the feed mass exits in the press filtrate as it does in the dewatered cake.

Table 104. Mass Balance and Removal Calculations for Pharmaceutical Compounds in Belt Filter Press Dewatering Process, Gander, NL

Pharmaceutical	Concentration			Mass of Contaminants			% Removal
	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)	Thickened Primary Sludge (mg/d)	Biosolids Cake (mg/d)	Filtrate (mg/d)	
Ibuprofen	459	319	2130	71.1	49.9	42.5	-30.0%
Naproxen	178	98.1	690	27.6	15.4	13.8	-5.6%
Triclocarban	2670	2470	15.3	414	387	0.30	6.4%
Triclosan	11700	9560	241	1,812	1,496	4.8	17.2%
Acetaminophen	1170	<230	16600	181.2	<5	330.84	>-85.1%
Azithromycin	237	220	49.9	36.7	34.4	0.99	3.5%
Caffeine	2590	1160	10200	401	182	203	4.1%
Carbamazepine	275	214	1300	42.6	33.5	25.9	-39.5%
Ciprofloxacin	17200	16900	108	2,664	2,645	2.2	0.6%
Clarithromycin	92.4	97.2	147	14.3	15.2	2.9	-26.8%
Dehydronifedipine	7.66	8.42	82	1.19	1.32	1.63	-148.9%
Diphenhydramine	222	205	145	34.4	32.1	2.9	-1.7%
Diltiazem	348	257	316	53.9	40.2	6.3	13.7%
Erythromycin-H_2O	28.7	32.9	53.3	4.44	5.15	1.06	-39.8%
Fluoxetine	36.1	51.9	9.29	5.59	8.12	0.19	-48.6%
Miconazole	463	441	<5.84	71.7	69.0	<0.12	>3.6%
Norfloxacin	2010	2390	378.5	311	374	7.5	-22.6%
Ofloxacin	2590	2670	<58.4	401	418	<1.2	>-4.5%
Sulfamethoxazole	17.6	5.17	80.5	2.73	0.81	1.60	11.5%
Thiabendazole	44.1	50.9	16.9	6.83	7.97	0.34	-21.6%
Trimethoprim	92.9	76.7	149	14.4	12.0	3.0	-4.1%

Removal efficiencies of the pharmaceutical compounds are categorised in **Table 105**. A slightly higher number of compounds have negative removal efficiencies than those with positive removal efficiencies. The distribution suggests, given the uncertainty in the cake and filtrate volumetric flow rates developed from the solids balance, that there is no overall net removal of the pharmaceutical compounds by the belt filter press. In general, the mass entering the belt press in the primary sludge feed can be accounted for in the dewatered cake or filtrate. This conclusion is reasonable considering that the dewatering process is a physical separation that does not involve biological or chemical activity needed to reduce the mass of the pharmaceuticals.

4.12.4.4 Mass Balances for Fragrance and Alkylphenolic Compounds

The mass balance and removal efficiency results for BPA and the polycyclic musk fragrances are provided in **Table 106**. A negative removal efficiency was calculated for Bisphenol A at this site, due to the very high concentration of BPA in the press filtrate. As discussed earlier in this

Table 105. Categorised Removal Efficiencies of Pharmaceutical Compounds by Belt Filter Press Dewatering Process, Gander, NL

Estimated Removal Efficiency Range				
<-50%	**≥-49 to -1%**	**≥0 to 49%**	**≥50 to 89%**	**≥90%**
Acetaminophen	Ibuprofen	Triclocarban		
Dehydronifedipine	Naproxen	Triclosan		
	Carbamazepine	Azithromycin		
	Clarithromycin	Caffeine		
	Diphenhydramine	Ciprofloxacin		
	Erythromycin-H_2O	Diltiazem		
	Fluoxetine	Miconazole		
	Norfloxacin	Sulfamethoxazole		
	Ofloxacin			
	Thiabendazole			
	Trimethoprim			
n=2	n=11	n=8	n=0	n=0

section, the high concentration of BPA in the filtrate is suspect, and so the results of the mass balance and removal calculations are also in doubt. The removal efficiencies of the polycyclic musk fragrances spanned a wide range of values from a maximum positive removal of 54% for AHDI to the lowest negative removal of -94% for HHCB. Little removal of contaminants would be expected from this solids separation process.

Table 106. Mass Balance and Removal Calculations for Alkylphenolic and Fragrance Compounds in Belt Filter Press Dewatering Process, Gander, NL

Compound	Concentrations			Mass of Contaminants (mg/d)			% Removal
	Thickened Primary Sludge (ng/g TS dw)	Biosolids Cake (ng/g TS dw)	Filtrate (ng/L)	Thickened Primary Sludge	Biosolids Cake	Filtrate	
Alkylphenolics							
Bisphenol A	375	130	3650	58.1	20.3	72.74	-60%
Fragrances							
DPMI	65	50	<20	10.1	7.83	<0.40	22%
AHDI	730	330	<10	113	51.7	<0.20	54%
HHCB	1490	2800	460	231	438	9.17	-94%
AHTN	840	1340	255	130	210	5.08	-65%
ATII	220	255	115	34.1	39.9	2.29	-24%

4.12.4.5 Effectiveness of Process for ESOC Removal

The results of the sampling survey, as typified by the Gander data, indicate that the belt filter press used to dewater the primary sludge provides a negligible barrier for reducing the mass of metal or pharmaceutical contaminants in the dewatered cake. As a solids separation device, it is not designed to "remove" these contaminants. Both positive and negative removal efficiencies were observed for polycyclic musk fragrance compounds. The calculated negative removal efficiency for BPA was considered unreliable.

4.12.5 Section Summary

Ammonia-N and ortho-phosphate, both mostly soluble forms of the nutrients, are substantially reduced in the filtrate compared to the feed primary sludge, suggesting that some physical or chemical processes may have occurred to reduce the concentrations through the dewatering process. Although the concentrations of TKN and total P also decreased substantially in the filtrate compared to the feed primary sludge, these components may be associated with the solid phase of the sludge and may thus have accumulated in the dewatered biosolids cake. Mass balance closure for TKN and total P are slightly below the expected value of 100%. The apparent loss of TKN and total P may reflect uncertainty in the flow rates calculated using the solids balance. Substantially higher amounts of ammonia-N are identified in the dewatered cake compared to the feed sludge. The cause is uncertain but may possibly be due to breakdown of organic polymers used in the dewatering process. The apparent loss of ortho-phosphate likely reflects a shift from the soluble form to an insoluble precipitate.

Several metals are identified above the detection limits in thickened primary sludge, including chromium, copper, lead, mercury, and zinc. All the metals (except cadmium) studied are detected in the dewatered biosolids. Copper is the only metal detected in the filtrate. Copper and zinc and are observed at the highest concentrations in the samples, with mercury having the lowest detected concentrations. Because copper is the only metal observed in the press filtrate at a detectable level, a full mass closure is only possible for that metal, with an estimated closure of 109% of the input mass. Four other metals are detected in the feed primary sludge and dewatered cake, but not the filtrate. Using the reported detection limit for the additional four metals in the filtrate, it is possible to estimate a potential maximum mass in the filtrate, thereby establishing a potential mass closure range. This procedure reveals that the bulk of the feed metals resides in the dewatered cake, which would be as expected.

A total of 20 pharmaceuticals were detected in both the thickened primary sludge and the dewatered cake samples from all the three sampling campaigns; 19 pharmaceuticals were detected in the filtrate samples from all the three campaigns. Three pharmaceutical compounds or metabolites (hydrochlorothiazide, 2-hydroxy-ibuprofen and 1,7-dimethylxanthine were detected in all three campaigns in the belt press filtrate, although they were never detected in either the primary sludge feed or the dewatered cake. The pharmaceutical compounds detected at the highest concentrations in the dewatered cake samples (i.e. at concentrations greater than 1,000 ng/g TS) included the antimicrobials triclosan and triclocarban, the antibiotics ciprofloxacin, norfloxacin and ofloxcacin, the stimulant caffeine and furosemide, a diuretic compound.

The number of compounds detected in all three campaigns remains approximately the same (i.e., 19-20) in the dewatered primary sludge, dewatered biosolids cake and belt press filtrate. The same compounds are not consistently detected in these matrices however, as it was noted above that three pharmaceuticals or metabolites were observed in the press filtrate, but never in the primary sludge or dewatered cake. The number of non-detected compounds was similar in the thickened primary sludge and dewatered cake. The number of compounds never detected in the three press filtrate samples was lower, however, due to the compounds being detected in one or two of the sampling campaigns.

Removal efficiencies through the belt filter press are low in comparison to many of the other biosolids treatment processes investigated in this survey. The belt filter press is a dewatering process, and is not intended to stabilise the organic matter or reduce pathogens or vector attraction. The calculated highest removal efficiency is 13.7% for the cardiac drug diltiazem. The lowest calculated removal efficiency of -185% is observed for the anti-anginal compound dehydronifedipine. The calculated masses of the pharmaceuticals in the primary sludge feed, dewatered cake and filtrate samples indicate that in most cases, the dewatered cake is the repository for the compounds entering the process in the feed sludge. For a few compounds, such as the non-steroidal anti-inflammatory compounds ibuprofen and naproxen, and the antibiotic sulfamethoxazole, as much of the feed mass exits in the press filtrate as it does in the dewatered cake. A slightly higher number of compounds have negative removal efficiencies than those with positive removal efficiencies. The distribution suggests, given the uncertainty in the cake and filtrate volumetric flow rates developed from the solids balance, that there is no overall net removal of the pharmaceutical compounds by the belt filter press. In general, the mass entering the belt press in the primary sludge feed can be accounted for in the dewatered cake or filtrate. This conclusion is reasonable considering that the dewatering process is a physical separation that does not involve biological activity needed to reduce the concentrations of the pharmaceuticals.

The Bisphenol A concentration data exhibit substantial variability in the two sampling campaigns. Specifically, the data from the second sampling campaign (see Table A-15) were much higher than the concentrations from the corresponding sample locations in Campaign 1. The variations in the BPA concentrations may be real resulting from an elevated industrial input to the wastewater treatment system. The possibility exists, however, that the samples may have been inadvertently contaminated by the use of a sampling container involving plastics manufactured with BPA. In any event, the reported BPA concentrations are suspect. A negative removal efficiency was calculated for Bisphenol A at this site, due to the very high concentration of BPA in the press filtrate. Because the high concentration of BPA in the filtrate is suspect, the results of the mass balance and removal calculations are also in doubt.

The polycyclic musk fragrances HHCB, AHTN and ATII were detected in all samples in both sampling campaigns. HHCB and AHTN were detected at the highest median concentrations (i.e., greater than 1000 ng/g TS dw) in the dewatered cake solids. None of the nitro musk compounds were observed above the limit of quantification in the primary sludge feed, dewatered cake or press filtrate. Some compounds were observed at higher concentrations in the dewatered cake than in the feed sludge, while the concentrations of other compounds in the dewatered cake were lower than in the feed sludge. The removal efficiencies of the polycyclic musk fragrances spanned a wide range of values from a maximum positive removal of 54% for AHDI to the

lowest negative removal of -94% for HHCB. Little removal of contaminants would be expected from this solids separation process.

The results of the sampling survey, as typified by the Gander data, indicate that the belt filter press used to dewater the primary sludge provides a negligible barrier for reducing the mass of metal and pharmaceutical contaminants in the dewatered cake. As a solids separation device, it is not designed to "remove" these contaminants. Both positive and negative removal efficiencies were observed for polycyclic musk fragrance compounds. The calculated negative removal efficiency for BPA was considered unreliable.

5. INTEGRATED ASSESSMENT OF REVIEW OF BIOSOLIDS CONTAMINANTS

5.1 Occurrence of Emerging Substances of Concern in Processed Canadian Sludges and Biosolids

5.1.2 Metals

The metals analysed are not technically considered as ESOC since they have been widely documented in the literature and regulated by provincial standards for land application. Metals were analyzed only in the first round of biosolids collected at the different survey sites to provide a high-level comparison with historical data. The results are presented in **Table 107**. Cadmium was detected in only two of the biosolids samples from the eleven sites. Copper, mercury and zinc were found in biosolids samples from all eleven sites. The highest median detected concentrations were zinc and copper at 331 and 271 mg/kg TS dw, respectively. The lowest median detected concentration was mercury at 0.68 mg/kg TS dw. The medians concentrations in this study are consistent with mean values from recent surveys in Québec (Perron and Hébert, 2007). Median values of both detected and non-detected metal concentration in this study are all below limits used by jurisdictions in Canada for biosolids. Limits for metals in unrestricted use of compost (among the most stringent) are provided in the table for comparison. Although maximum concentration values of copper, mercury and molybdenum exceeded the most stringent limits for unrestricted use at 2, 4 and 1 sites, respectively, these maximum concentrations may still be acceptable for other beneficial uses for soil amendment.

Table 107. Metal Concentration Data in 11 Canadian Treated Sludge and Biosolids Samples

Metal (total)	No. of Detected Conc'ns (out of 11)	Concentration (mg/kg TS dw)			Conc'n Limit for Unrestricted Use (Compost) (CCME, 2005)
		Median of All Conc'ns	Median of Detected Conc'ns	Maximum Detected Conc'n	
Arsenic (As)	7	1.4	2.6	6.7	13
Cadmium (Cd)	2	<1.0	1.1	1.2	3
Chromium (Cr)	10	18.1	20.3	120	210
Cobalt (Co)	7	2.6	2.9	4.2	34
Copper (Cu)	**11**	**271**	**271**	890	400
Lead (Pb)	9	22.5	24.7	55.5	150
Mercury (Hg)	**11**	**0.68**	**0.68**	3.2	0.8
Molybdenum (Mo)	8	1.8	3.5	8.6	5
Nickel (Ni)	9	9.9	10.5	21.1	62
Selenium (Se)	6	1.3	2.2	3.2	2
Zinc (Zn)	**11**	**331**	**331**	647	700

Metals in **bold font** are detected in all samples of processed sludges or biosolids

When comparing metal concentrations in composted septage (Gatineau Valley) to median biosolids values, the metal concentrations are approximately the same, an observation also

reported by (Perron and Hébert, 2007), who evaluated a higher number of septage locations.. The data reinforce the success of source reduction of metals from industries, with metals contributed to biosolids now mainly originating from domestic rather than industrial sources.

The results of this survey are compared to other historical Canadian data in **Table 108**. It is evident that over the past three decades, very positive steps have been taken in Canada to reduce the concentrations of all metals in the biosolids. Current concentrations of cadmium, chromium, lead and nickel are reduced by greater than 90% compared to the 1981 levels. Only with arsenic was there no decrease between the historical 1995 data and the current survey results. While it is likely that the data in **Table 108** reflect a reduction of metal inputs to wastewater treatment systems, additional factors may affect the results including a substantial number of small municipalities in the 11 sites included in this survey (compared to the sites used in the previous surveys), and a difference in analytical procedures, with different limits of quantitation.

Table 108. Comparison of Current Metal Concentrations in Biosolids Compared to Historical Data

Metal	Concentration (mg/kg TS dw)			% Reduction (Current compared to 1981)
	1981[a]	1995[a]	Current Median (2009)	
Arsenic		2.3	2.6	-13.0%[b]
Cadmium	35	6.3	1.1	96.9%
Chromium	1040	319	20.3	98.0%
Cobalt	NA	NA	2.9	
Copper	870	638	271	68.9%
Lead	545	124	24.7	95.5%
Mercury	NA	3.5	0.677	80.9%[b]
Molybdenum	NA	22	3.5	84.1%[b]
Nickel	160	38	10.5	93.4%
Selenium	NA	3.3	2.15	34.8%[b]
Zinc	1390	823	331	76.2%

[a] from WEAO (2001) NA = not available

[b] Reduction based on current data (this report, 2009) compared to 1995 data

The literature review associated with this field survey (Hydromantis *et al*. 2009) noted that reductions of metal concentrations, such as nickel, chromium and cadmium, were effectively accomplished in the 1980s and 1990s by source control, pretreatment and sewer use limits.

Metals cannot be removed by the biosolids treatment processes. As a result, the only method to further reduce concentrations in the biosolids, if needed, is to restrict them at the source.

5.1.3 Pharmaceuticals

The pharmaceutical analyses included both acid positive and acid negative compounds; in total 57 compounds were included in the scans of the two lists. Of the 57 candidate pharmaceutical compounds, twenty were never above the detection level in the treated sludges and biosolids, as

indicated in **Table 109**. Sample detection limits were determined for each compound in each matrix, and as a result no single "representative" detection limit is provided.

Table 109. Pharmaceutical Compounds Never Detected in Treated Sludges and Biosolids in this Study

Non-Detected Pharmaceuticals	
Acetaminophen	Penicillin G
Carbadox	Sarafloxacin
Cefotaxime	Sulfachloropyridazine
Clinafloxacin	Sulfadiazine
Cloxacillin	Sulfadimethoxine
Flumequine	Sulfamethazine
Lomefloxacin	Sulfamethizole
Norgestimate	Sulfathiazole
Ormetoprim	Tylosin
Oxacillin	Warfarin

Only four of 57 pharmaceutical compounds (7 %) were found in detectable concentrations in all 31 samples of treated sludge or biosolids samples. These four compounds included:

- Triclocarban
- Carbamazepine
- Diphenhydramine and
- Miconazole.

The frequency of detection of the pharmaceutical, alkylphenolic and fragrance compounds in the feed sludges and treated biosolids is presented in **Table 110**. Although 25 pharmaceutical, alkylphenolic and fragrance compounds were found in detectable concentrations in more than 75% of the feed sludge samples, only 14 of these compounds were found in more than 75% of the treated biosolids samples. A greater proportion of pharmaceuticals were detected when septage was the feed sludge (49 % at Gatineau Valley) rather than from on-site wastewater processes. A shift in the frequency distribution occurred such that more of the pharmaceuticals were detected less frequently after the biosolids treatment, compared to frequency in the feed sludge samples, suggesting that on a broad overview, biosolids treatment processes reduce the number of detectable concentrations of ESOC in the feed sludge. As will be discussed below, the ability to reduce ESOC in biosolids is process dependent. **Table 111** provides a summary of the frequency of occurrence and median concentrations of the detectable concentrations of all organic target analytes.

Table 110. Frequency of Detection of Pharmaceuticals in Feed Sludges and Treated Sludges and Biosolids

Feed Sludge				Treated Sludge/Biosolids			
Frequency of Occurrence				Frequency of Occurrence			
>75%	25-75%	>0-<25%	0%	>75%	25-75%	>0-<25%	0%
Azithromycin	2-Hydroxy-ibuprofen	1,7-Dimethyl-xanthine	Carbadox	Azithromycin	2-Hydroxy-ibuprofen	1,7-Dimethyl-xanthine	Acetaminophen
Caffeine	Acetaminophen	Clinafloxacin	Cefotaxime	Carbamazepine	Caffeine	Digoxigenin	Carbadox
Carbamazepine	Enrofloxacin	Digoxigenin	Cloxacillin	Ciprofloxacin	Clarithromycin	Digoxin	Cefotaxime
Ciprofloxacin	Furosemide	Digoxin	Flumequine	Diphen-hydramine	Dehydro-nifedipine	Enrofloxacin	Clinafloxacin
Clarithromycin	Gemfibrozil	Glyburide	Glipizide	Fluoxetine	Diltiazem	Glipizide	Cloxacillin
Dehydro-nifedipine	Hydrochloro-thiazide	Lincomycin	Lomefloxacin	Miconazole	Erythromycin-H2O	Glyburide	Flumequine
Diltiazem		Oxolinic Acid	Norgestimate	Naproxen	Furosemide	Hydrochloro-thiazide	Lomefloxacin
Diphenhydramine		Sulfamerazine	Ormetoprim	Ofloxacin	Gemfibrozil	Lincomycin	Norgestimate
Erythromycin-H_2O		Sulfamethazine	Oxacillin	Triclocarban	Ibuprofen	Oxolinic Acid	Ormetoprim
Fluoxetine		Sulfathiazole	Penicillin G	Triclosan	Norfloxacin	Penicillin V	Oxacillin
Ibuprofen		Virginiamycin	Penicillin V		Sulfamethox-azole	Roxithromycin	Penicillin G
Miconazole			Roxithromycin		Thiabendazole	Sulfamerazine	Sarafloxacin
Naproxen			Sarafloxacin		Trimethoprim	Sulfanilamide	Sulfachloro-pyridazine
Norfloxacin			Sulfachloro-pyridazine			Virginiamycin	Sulfadiazine
Ofloxacin			Sulfadiazine				Sulfadimeth-oxine
Sulfamethoxazole			Sulfadimeth-oxine				Sulfamethazine
Thiabendazole			Sulfamethizole				Sulfamethizole
Triclocarban			Sulfanilamide				Sulfathiazole
Triclosan			Tylosin				Tylosin
Trimethoprim			Warfarin				Warfarin
20	6	11	20	10	13	14	20
			Gain/Loss	-10	7	3	0

Table 111. Occurrence and Median Concentrations of All ESOC in Treated Sludges and Biosolids in this Study

Compound	% occurrence	Median of detected conc'ns (ng/g TS dw)	Compound	% occurrence	Median of detected conc'ns (ng/g TS dw)
HHCB	100%	3470	Gemfibrozil	52%	56
Triclocarban	100%	1930	Trimethoprim	42%	31.2
AHTN	100%	1340	Dehydronifedipine	42%	7
Miconazole	100%	441	Sulfamethoxazole	39%	5.2
Diphenhydramine	100%	420	Furosemide	32%	543
Carbamazepine	100%	66.6	2-Hydroxy-ibuprofen	26%	497
Triclosan	97%	6085	Enrofloxacin	23%	22.2
ATII	96%	255	Octylphenol	18%	50
Ciprofloxacin	94%	3610	1,7-Dimethylxanthine	13%	378
Ofloxacin	87%	276	Sulfanilamide	13%	63.1
Bisphenol A	86%	325	Glyburide	13%	11.5
Azithromycin	84%	205	Hydrochlorothiazide	10%	143
Fluoxetine	84%	53.9	Sulfamerazine	10%	17.9
Naproxen	81%	98.1	Virginiamycin	6%	197
Clarithromycin	74%	41.8	Digoxin	6%	192
Thiabendazole	74%	17.9	Digoxigenin	6%	128
Erythromycin-H_2O	74%	12.5	Musk Xylene	5%	530
DPMI	73%	82.5	ADBI	5%	60
Ibuprofen	68%	522	Lincomycin	3%	71.1
Diltiazem	68%	29.8	Penicillin V	3%	59.3
AHDI	64%	158	Glipizide	3%	11.4
Caffeine	61%	266	Oxolinic Acid	3%	1.9
Norfloxacin	58%	558	Roxithromycin	3%	0.8

Elevated concentrations of ESOC such as triclosan, ciprofloxacin, BPA, HHCB and AHTN may be one criterion used for identifying ESOC that should be considered for detailed risk assessment. There are other criteria, however, such as persistence, potential for bioaccumulation, and toxicity, that are at least as important and also need to be considered for targeting the ESOC for priority risk assessment.

Many pharmaceuticals (nearly 30 % of those tested) were not detected in the final biosolids products. For those substances that were still detected after process treatments, the statistics provided in **Table 111** may help scientists to evaluate whether or not these concentrations may still pose risk with land application.

A small number of pharmaceutical compounds (12 of 57) were observed at concentrations exceeding 1,000 ng/g TS dw (1 mg/kg TS dw) in the final sludge or biosolids products at one or

more the test sites (**Table 112**). The identification of these compounds is a function of the concentration in the feed sludge material and the effectiveness of the treatment process. The antibacterial compounds triclosan and triclocarban, and the antibiotic ciprofloxacin were the compounds most frequently detected (9 of 11 sites) above 1000 ng/g TS dw. At a few sites, the concentrations of triclosan and ciprofloxacin exceeded 10,000 ng/g TS dw.

Biosolids or sludge treatment processes at four of the sampling locations involve the production of sidestreams (e.g. dewatering press filtrate, compost pad leachate) that contain may contain detectable concentrations of some of the hydrophilic pharmaceuticals (**Table 113**), which can represent a significant percentage of the input mass of the ESOC. In a few cases, the pharmaceutical mass calculated in the filtrate was greater than the input mass (e.g., ibuprofen and carbamazepine at Eganville, acetaminophen and dehydronifedipine at Gander). Because some compound mass in the feed sludge may be transferred to the aqueous sidestream, the change in frequency of occurrence of detectable concentrations from feed sludge to treated biosolids cannot be interpreted simplistically as a reduction or removal efficiency.

5.1.4 Fragrance and Alkylphenolic Compounds

Compounds that were never detected in any of the treated sludges or biosolids (**Table 114**) included the alkylphenolic compounds nonylphenol and the synthetic fragrances musk moskene, musk tibetene, musk ambrette and musk ketone. The fragrances ADBI and musk xylene were only detected once in the sampling program.

The non-detection of nonylphenol was surprising given that it has been identified in many other surveys of sludges and biosolids. Use of nonylphenol ethoxylates was restricted in Canada beginning in 2004 with a Notice regarding the implementation of pollution prevention plans for nonylphenol and its ethoxylates filed under the Canadian Environmental Protection Act in the Canada Gazette (Part I, Vol. 138, No. 49 December 4, 2004). The results observed in this study may reflect the restrictions of its use. The limit of quantitation at 140 ng/g TS dw was also higher than anticipated, which may also contribute to the observed non-detection.

Two polycyclic fragrance compounds, HHCB and AHTN were detected in all 22 samples of treated sludges and biosolids.

Table 112. Pharmaceutical Compounds exceeding 1000 ng/g TS dw in the Treated Sludges or Biosolids

Compound	Concentration (ng/g TS dw)											
	Salmon Arm	**Red Deer**	**Saska-toon**	**Prince Albert**	**Egan-ville WAS**	**Egan-ville Septage**	**Smiths Falls**	**Gatineau Valley**	**Saguenay**	**Moncton**	**Halifax**	**Gander**
Furosemide						1120						
2-Hydroxy-ibuprofen	1160											
Ibuprofen	1960											
Naproxen				2600				9890		3830		
Triclocarban	5010	4410	1930	1635	1845	6580	3960		1660		1590	2470
Triclosan	**21500**	**12700**	6050	3950	1826	**30600**	**11485**		1310		6120	9560
Caffeine	4110			1033		1070						1160
Ciprofloxacin	6900	6520	3610	2020	6135	**26800**	2530	1059	6440			**16900**
Diphenhydramine		2300		1220								
Miconazole	1350	1090										
Norfloxacin		3270				5590	1140					2390
1,7-Dimethylxanthine	1850											

Concentrations in **bold font** exceed 10,000 ng/g TS dw

Table 113. Concentrations of Pharmaceuticals in Aqueous Sidestreams of Biosolids Treatment Processes

	Concentration (ng/L)				Filtrate Mass (% of Input Mass)			
	Eganville	Gander	Gatineau Valley	Saguenay	Eganville	Gander	Gatineau Valley	Saguenay
Pharmaceutical	Geotextile Bag Filtrate	Press Filtrate	Compost Pad Leachate	Press Filtrate	Geotextile Bag Filtrate	Press Filtrate	Compost Pad Leachate	Press Filtrate
Furosemide	9800	270	<357	402	65.0%			8.8%
Gemfibrozil	<2.85	<6.01	41.3	5.85			1.2%	
Hydrochlorothiazide	1220	435	<21.1	461	3.3%			
2-Hydroxy-ibuprofen	113050	4660	388	147	19.0%		2.3%	
Ibuprofen	4870	2130	616	190	5.6%	59.7%	1.4%	12.1%
Naproxen	1090	690	126.6	132	429.7%	49.9%	1.0%	21.8%
Triclocarban	16.2	15.3	16.7	8.9	0.1%	0.1%	0.0%	0.0%
Triclosan	1490	241	129	84	10.8%	0.3%	0.0%	0.1%
Acetaminophen	115355	16600	<97.3	<77.1	6.2%	182.6%		
Azithromycin	236	49.9	10.3	162	12.3%	2.7%	0.0%	1.5%
Caffeine	49360	10200	431	<17.2	64.1%	50.7%	0.4%	
Carbamazepine	897	1300	561	345	135.9%	60.8%	10.7%	20.8%
Ciprofloxacin	118	108	75.9	54.2	0.1%	0.1%	0.0%	0.0%
Clarithromycin	195	147	11.6	27.4	39.5%	20.5%		1.7%
Dehydronifedipine	32.2	82	47.6	2.93	50.1%	137.8%	3.1%	
Diphenhydramine	213	145	32.7	88.1	6.3%	8.4%	0.0%	0.6%
Diltiazem	24.5	316	9.67	17.5	7.4%	11.7%	0.2%	1.3%
Erythromycin-H_2O	117	53.3	7.32	5.31	56.3%	23.9%	1.2%	4.2%
Fluoxetine	5.9	9.29	<1.58	5.57	1.3%	3.3%		0.7%
Miconazole	<2.24	<5.84	<6.62	<1.72				
Norfloxacin	<56.7	379	<15.8	272		2.4%		1.5%
Sulfamethoxazole	1100	80.5	<0.64	22.6	90.7%	58.9%		4.7%
Thiabendazole	29.2	16.9	5.96	58.2	9.5%	4.9%	0.1%	2.0%
Trimethoprim	507	149	<48.3	27.5	31.3%	20.6%		2.3%
1,7-Dimethylxanthine	38750	2530	550	<172	10.7%		0.9%	

Filtrate mass represents >20% of input mass

Table 114. Alkylphenolic and Fragrance Compounds Never Detected in Treated Sludge and Biosolids Samples

Compound	Category
Nonylphenol	Alkylphenol
Musk Moskene	Nitro musk fragrance
Musk Tibetene	Nitro musk fragrance
Musk Ketone	Nitro musk fragrance
Musk Ambrette	Nitro musk fragrance

The frequency of detection of the alkylphenolic and fragrance compounds in the feed sludges and treated sludges and biosolids from the test sites is presented in **Table 115.** The compounds within each range of frequency of occurrence remain mostly constant in the feed sludge and in the treated sludges or biosolids.

Table 115. Frequency of Detection of Alkylphenolic and Fragrance Compounds in Feed Sludges and Treated Sludges and Biosolids

Feed Solids				Treated Sludge/Biosolids			
Frequency of Occurrence				Frequency of Occurrence			
>75%	25-75%	>0-<25%	0%	>75%	25-75%	>0-<25%	0%
HHCB	DPMI	Musk Xylene	Nonylphenol	HHCB	DPMI	Octylphenol	Nonylphenol
AHTN	AHDI	Octylphenol	ADBI	AHTN	AHDI	ADBI	Musk Moskene
ATII		Musk Ketone	Musk Moskene	ATII		Musk Xylene	Musk Tibetene
Bisphenol A			Musk Tibetene	Bisphenol A			Musk Ketone
			Musk Ambrette				Musk Ambrette

As indicated by **Table 116**, the fragrance HHCB was observed above 1,000 ng/g TS dw in the solids from 10 of the 11 sites tested, while other fragrances AHTN and ATII were noted above 1,000 ng/g TS dw in the solids from six and one of the 11 sites sampled, respectively. Bisphenol A was observed at concentrations above 1,000 ng/g TS dw in the treated sludge or biosolids of three of the sites tested.

Table 116. Alkylphenolic and Fragrance Compounds exceeding 1,000 ng/g TS dw in the Final Sludge or Biosolids

Compound	Concentration (ng/g TS dw)										
	Salmon Arm	Red Deer	Saskatoon	Prince Albert	Eganville	Smiths Falls	Gatineau Valley	Saguenay	Moncton	Halifax	Gander
Bisphenol A	1220		1765		1375						
HHCB	8685	8975	5130	3470	5660	2795	2215	3290		4115	2800
AHTN	4440	4015	2225		1800			2685			1340
ATII	1025										

Four site locations were identified as producing a liquid process sidestream or leachate that may contain some of the target analytes. The estimated mass of Bisphenol A and synthetic musk fragrances associated with the sidestreams or leachate relative to the input mass in the feed sludge is provided in **Table 117**.

Table 117. Concentrations of Alkylphenolic and Fragrance Compounds in Aqueous Sidestreams of Biosolids Treatment Processes

Compound	Concentration (ng/L)				Filtrate Mass (% of Input Mass)			
	Eganville Geotextile Bag Filtrate	Gander Press Filtrate	Gatineau Valley Compost Pad Leachate	Saguenay Press Filtrate	Eganville Geotextile Bag Filtrate	Gander Press Filtrate	Gatineau Valley Compost Pad Leachate	Saguenay Press Filtrate
Bisphenol A	550	3650	200	1080	4.2%	125.0%	1.3%	6.83%
DPMI	<20	<20	30	30	<1.1%	<4.0%	1.0%	0.40%
AHDI	<10	<10	<10	<10	<0.3%	<0.2%	<1.0%	<0.2%
HHCB	570	280	75	590	0.8%	4.0%	0.0%	0.49%
AHTN	120	240	240	315	0.4%	3.9%	0.1%	0.52%
ATII	30	80	15	95	0.4%	6.7%	0.1%	1.60%

With the exception of the concentration of Bisphenol A in the Gander press filtrate, the mass of the BPA in the leachate represented between 1% and 7% of the mass in the feed sludge. The reported filtrate BPA concentration was considered suspect as discussed earlier in the report. With the exception of the musk fragrances in the Gander filtrate, the mass of the fragrances in the sidestreams or leachates represented less than 1% of the mass in the feed sludges. The mass of each fragrance compound in the Gander filtrate as a percentage of the input mass was somewhat higher, ranging between <0.2% to 6.7%. The Gander facility is the only one of these four sites without either some form of biological wastewater treatment (which would reduce the ESOC concentrations of the solids to be treated) or a solids treatmenr process involving biological treatment or an extended detention period prior to sampling. Either situation might lead to a lower observed concentration of ESOC in the treated sludge or biosolids. In general, there is a very minor loss of the fragrance compounds and BPA from the feed sludge to the leachate, as would be expected of hydrophobic compounds.

5.2 Removal of Emerging Substances of Concern by Canadian Sludge and Biosolids Treatment Processes

5.2.1 Removal of Pharmaceutical Compounds

Removal efficiencies of the various pharmaceutical ESOC by the biosolids treatment processes are summarized in **Table 118**. The removal efficiency ranges have been colour-coded in **Table 118** to provide a more visual depiction of the relative abilities different sludge or biosolids treatment processes for removing the ESOC. The table helps to differentiate the removal

Table 118. Calculated Removal Efficiencies of Pharmaceutical Compounds by Process

Pharmaceutical	% Removal										
	Biological-autothermal aerobic digestion	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-mesophilic anaerobic digestion	Biological-mesophilic anaerobic digestion	Physical-filter press dewatering	Physical-filter press dewatering	Physical-geotextile bag filter dewatering	Phys-chem (alkaline stabilis'n)	Thermal Drying
	Salmon Arm	Moncton	Gatineau Valley	Prince Albert	Saskatoon	Red Deer	Gander	Saguenay	Eganville (Septage)	Halifax N-Viro	Smiths Falls
Furosemide	-73%			-187%				-41%	-123%	-110%	20%
Gemfibrozil	-245%	>80%	>94.2%-98.8%	55%	-56%	-168%				-25%	-17%
Glyburide			>94.9%-99.5%							>43%	
Hydrochlorothiazide		>55%			36%	-79%			>75%- 80%	39%	
2-Hydroxy-ibuprofen	-73%		>46.7%-97.7%		3%	61%			>35%-37%	-5%	
Ibuprofen	-306%	>77%	95%	16%	-82%	-15%	-30%		13%	-82%	-8%
Naproxen	-141%	-2837%	-4270%	-1424%	62%	60%	-6%	-429%	23%	-17%	-11%
Triclocarban	-11%	95%	95%	49%	-20%	-39%	6%	2%	64%	53%	9%
Triclosan	-91%	95%	99%	68%	-55%	-17%	17%	21%	-123%	12%	1%
Acetaminophen		>38%			>55.2%		>-85.1%		78%		
Azithromycin	-6%	>98%	98%	93%	-12%	7%	4%	11%	14%	88%	-34%
Caffeine	-141%	>97%	>98.0%-99.6%	6%	92%	94%	4%	-290%	-26%	25%	-66%
Carbamazepine	-150%	89%	45%	50%	-112%	-219%	-40%	-35%	-183%	36%	-108%
Ciprofloxacin	61%	94%	96%	82%	-6%	-25%	1%	9%	-228%	44%	45%
Clarithromycin	-32%	>94%	>41.2%-99.9%	81%	43%	54%	-27%	-55%	-10%	59%	19%
Clinafloxacin										>-50%	
Dehydronifedipine	51%	>54%	67%	>79%	>13.3%	>19%	-149%			-9%	-121%

(continued)

Table 118 (continued)

Pharmaceutical	% Removal										
	Biological-autothermal aerobic digestion	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-mesophilic anaerobic digestion	Biological-mesophilic anaerobic digestion	Physical-filter press dewatering	Physical-filter press dewatering	Physical-geotextile bag filter dewatering	Phys-chem (alkaline stabilis'n)	Thermal Drying
	Salmon Arm	Moncton	Gatineau Valley	Prince Albert	Saskatoon	Red Deer	Gander	Saguenay	Eganville (Septage)	Halifax N-Viro	Smiths Falls
Diphenhydramine	-1%	99%	96%	74%	12%	-18%	-2%	-1%	36%	76%	-45%
Diltiazem	92%	>99%	>99.1-99.8%	93%	89%	86%	14%	-90%	61%	>76%	-94%
Digoxin						>64%					
Digoxigenin						37%					
Enrofloxacin		>-13%			-57%	49%		-36%		>-1%	22%
Erythromycin-H_2O	14%	>93%	>31.2%-98.8%	86%	41%	54%	-40%	-68%	-239%	55%	-1%
Fluoxetine	43%	>82%	91%	60%	40%	-39%	-49%	-64%		56%	-10%
Lincomycin						>13%					
Miconazole	-47%	95%	95%	64%	-97%	-113%	>3.6%	-16%	<-65%	32%	-45%
Norfloxacin	72%	>88%			71%	-31%	-23%	-7%	<-762%	-5%	46%
Ofloxacin	44%	56%	43%	73%	-6%	-44%	>-4.5%	-10%		-49%	39%
Oxolinic Acid				>-20%							
Sulfamerazine					-17%						
Sulfamethazine				>75%		>19%					
Sulfamethoxazole	90%	>65%		>98%	>90.4%	>77%	12%	-26%	77%	-19%	-282%
Sulfathiazole				>48%							
Thiabendazole	-1%	>72%	>97.2%-99.9%	70%	-40%	-69%	-22%	-21%		-28%	4%
Trimethoprim	55%	>90%		81%	91%	>79%	-4%	10%	-9%	7%	-3%
Virginiamycin										-47%	
1,7-Dimethylxanthine	-34%		>96.6%-99.1%						>63%-65%	19%	

(Continued)

Table 118 (continued)

Pharmaceutical	% Removal										
	Biological-autothermal aerobic digestion	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-aerobic (Compost)	Biological-mesophilic anaerobic digestion	Biological-mesophilic anaerobic digestion	Physical-filter press dewatering	Physical-filter press dewatering	Physical-geotextile bag filter dewatering	Phys-chem (alkaline stabilis'n)	Thermal Drying
	Salmon Arm	Moncton	Gatineau Valley	Prince Albert	Saskatoon	Red Deer	Gander	Saguenay	Eganville (Septage)	Halifax N-Viro	Smiths Falls
Range	Number										
<-50%	8	1	1	2	6	5	2	6	3	2	5
≥-50%-0%	7	1	0	1	6	8	11	9	3	9	9
≥0%-49%	3	1	5	4	7	6	8	5	4	10	9
50%-89%	4	10	1	14	4	8	0	0	11	8	0
≥90%	2	11	14	3	3	1	0	0	0	0	0
Total	24	24	21	24	26	28	21	20	21	29	23

efficiency of individual pharmaceuticals by the biosolids/sludge treatment processes, as well as the overall performance of the processes. Based on this table, it appears that the biological processes for biosolids treatment are more effective than the physical (including physical-chemical) processes for removing the pharmaceutical compounds.

A very few compounds appeared to be susceptible to removal by both aerobic and anaerobic biological treatment. These included sulfamethoxazole, trimethoprim, diltiazem and caffeine. It was observed that composting of sludges to produce biosolids generally resulted in the highest removal efficiencies of most ESOC.

Many other pharmaceuticals were effectively removed in the aerobic environment compared to the anaerobic environment. Compounds with this behaviour included azithromycin, ciprofloxacin, miconazole, triclosan, triclocarban, diphenhydramine, gemfibrozil, thiabendazole and carbamazepine. A limited number of pharmaceuticals, such as naproxen, survived and apparently increased through the composting process. Mesophilic anaerobic digestion of sludges, conversely, was found to substantially reduce concentrations of naproxen, as was noted in the project's literature review (Hydromantis *et al*., 2009). There was also limited evidence that anaerobic digestion may result in higher removal efficiencies of acetaminophen than composting, based on one location of each process type with quantifiable results. In general, however, anaerobic digestion was less successful in overall removal of ESOC than the composting process.

A limited number of pharmaceutical compounds appeared to be difficult to remove in almost all processes examined, when present at detectable concentrations. These included the diuretic furosemide, the anti-epileptic carbamazepine, and the antibiotic ofloxacin.

5.2.2 Removal of Alkylphenolic and Fragrance Compounds

Removal efficiencies of the various alkylphenolic and fragrance ESOC by the biosolids treatment processes are summarized in **Table 119**. The removal efficiency ranges have been colour-coded in the Table to provide a more visual depiction of the relative abilities different sludge or biosolids treatment processes for removing the ESOC. Inspection of this table clearly indicates that two sites, namely Gatineau Valley and Moncton, are associated with the best removal of the alkylphenolic and fragrance compounds.

Table 119. Calculated Removal Efficiencies of Alkylphenolic and Fragrance Compounds by Process

Pharmaceutical	% Removal										
	Biological-autothermal aerobic digestion	Biological-mesophilic anaerobic digestion)	Biological-mesophilic anaerobic digestion	Biological-aerobic (Compost)	Physical-geotextile bag filter dewatering	Thermal Drying	Biological-aerobic (Compost)	Physical-filter press dewatering	Biological-aerobic (Compost	Phys-chem (alkaline stabilis'n)	Physical-filter press dewatering
	Salmon Arm	Red Deer	Saskatoon	Prince Albert	Eganville (Septage)	Smiths Falls	Gatineau Valley	Saguenay	Moncton	Halifax N-Viro	Gander
Bisphenol A	-16%	-47%	78%	36%	-71%	-12%	71%	18%	92%	-338%	-60%
Octylphenol	13%		-103%						-19%		
DPMI	-16%	5%	-52%	66%	13%		21%	51%	51%	-1%	22%
AHDI	51%	89%	-14%		21%		>70%	-17%	95%	>52%	54%
HHCB	7%	-108%	-30%	-31%	-10%	28%	80%	-1%	89%	-22%	-94%
AHTN	11%	-61%	-32%	45%	46%	45%	74%	-62%	81%	-59%	-65%
ATII	0%	-30%	-61%	24%	43%	3%	63%	-75%	89%	-11%	-24%
Musk Xylene	>79%		>19%		28%			-48%			
Range											
<-50%	0	2	3	0	1	0	0	2	0	1	3
≥-50%-0%	2	2	3	1	1	1	0	3	1	4	1
≥0%-49%	4	1	1	3	5	3	1	1	0	0	1
50%-89%	2	1	1	1	0	0	5	1	4	1	1
≥90%	0	0	0	0	0	0	0	0	2	0	0
Total	8	6	8	5	7	4	6	7	7	6	6

5.3 Effectiveness of Different Biosolids Treatment Technologies

5.3.1 Study Evaluation

Most metals are conservative materials through biosolids treatment processes, i.e. the processes cannot specifically reduce the mass of metals in the feed sludge. Metals may be lost from the biosolids treatment process in aqueous sidestreams such as leachates, filtrates or supernatants. One metal that may be an exception is mercury. Under anaerobic conditions, mercury can be biotransformed to a methylated species, which can then be stripped from the process. This is not truly a removal, but a loss from the system to the atmosphere.

As a test of the different process capabilities for removing pharmaceuticals, alkylphenolic and fragrance compounds, the different removal efficiency ranges were assigned a numerical score, ranging from 1 for compounds which were removed by over 90%, to a value of 5 for compounds with calculated removal efficiencies that had a magnitude greater than -50%. (A negative removal means that the total mass leaving the biosolids treatment unit is greater than the mass entering the unit.) By summing the points assigned to each process for each compound, and dividing by the number of detections of the compound per site (i.e., counts) included in the assessment, a mean score for each process was calculated. The interpretation of this procedure considers that the lower the mean score (i.e. closer to unity), the more effective the process is at removing the pharmaceuticals. The results of this process comparison are presented in **Table 120**.

Table 120. Ranking of Sludge and Biosolids Treatment Processes for Removal of Pharmaceutical Compounds

Location	Process	score total	count	average score
Gatineau Valley	Biological – aerobic (Compost)	36	21	1.7
Moncton	Biological – aerobic (Compost)	43	24	1.8
Prince Albert	Biological – aerobic (Compost)	57	24	2.4
Eganville (Septage)	Physical – geotextile bag dewatering	61	21	2.9
Halifax N-Viro	Physical-chemical (alkaline stabilisation)	92	29	3.2
Red Deer	Biological – mesophilic anaerobic digestion	92	28	3.3
Saskatoon	Biological – mesophilic anaerobic digestion	86	26	3.3
Salmon Arm	Biological – autothermal aerobic digestion	87	24	3.6
Gander	Physical – filter press dewatering	78	21	3.7
Smiths Falls	Physical – thermal drying	88	23	3.8
Saguenay	Physical – filter press dewatering	81	20	4.1

A similar rating was applied to the alkylphenolic and fragrance compounds, with the results appearing in **Table 121**. The scoring confirms that the Moncton and Gatineau Valley composting processes provide the best removal efficiencies for the alkylphenolics. Furthermore, the four processes with the best overall ratings are aerobic biological treatment processes.

Table 121. Ranking of Sludge and Biosolids Treatment Processes for Removal of Alkylphenolic and Fragrance Compounds

Location	Process	score total	count	average score
Moncton	Biological – aerobic (Compost)	14	7	2.0
Gatineau Valley	Biological – aerobic (Compost)	13	6	2.2
Salmon Arm	Biological – autothermal aerobic digestion	24	8	3.0
Prince Albert	Biological – aerobic (Compost))	15	5	3.0
Smiths Falls	Physical – thermal drying	13	4	3.3
Eganville	Physical – geotextile bag dewatering	24	7	3.4
Red Deer	Biological – mesophilic anaerobic digestion	23	6	3.8
Halifax	Physical-chemical (alkaline stabilisation)	23	6	3.8
Saguenay	Physical – filter press dewatering	27	7	3.9
Saskatoon	Biological – mesophilic anaerobic digestion	32	8	4.0
Gander	Physical – filter press dewatering	24	6	4.0

The results of **Tables 120** and **121** were combined to determine the processes which are most efficient at reducing all of the target organic analytes. Note that the target analytes represent a small portion of the overall ESOC and so the interpretation is applicable only to this assessment. **Table 122** provides the summary ratings for the processes with respect to all target organic analytes. Composting was the most effective treatment for reducing loadings of the target analytes in the feed sludges. Anaerobic digestion was less successful than the aerobic composting processes. One of the more surprising results from this assessment was the lower removal efficiencies of pharmaceutical compounds than might have been expected in the autothermal aerobic digestion process, considering it is an aerobic process that operates at an elevated temperature, which should result in faster removal rates. The reasons for this observed performance are not clear and have been identified below as a knowledge gap.

Table 122. Ranking of Sludge and Biosolids Treatment Processes for Removal of All of the Study Target Organic Analytes

Location	Process	score total	count	average score
Gatineau Valley	Biological – aerobic (Compost)	49	27	1.81
Moncton	Biological – aerobic (Compost)	57	31	1.84
Prince Albert	Biological – aerobic (Compost)	72	29	2.48
Eganville (Septage)	Physical – geotextile bag dewatering	85	28	3.04
Halifax N-Viro	Physical-chemical (alkaline stabilisation)	115	35	3.29
Red Deer	Biological – mesophilic anaerobic digestion	115	34	3.38
Salmon Arm	Biological – autothermal aerobic digestion	111	32	3.47
Saskatoon	Biological – mesophilic anaerobic digestion	118	34	3.47
Smiths Falls	Physical – thermal drying	101	27	3.74
Gander	Physical – filter press dewatering	102	27	3.78
Saguenay	Physical – filter press dewatering	108	27	4.00

The geotextile bag filtration and alkaline stabilisation processes were the most effective of the non-biological processes. The processes involving solids dewatering alone were the least effective; however, because they operate only as physical separation devices, they are not designed for removal of ESOC. The low rating for theses processes is therefore not surprising. Dewatering may remain helpful in a series of biosolids treatment processes, however, to reduce concentration of water-soluble compounds.

Tables 120 through 122 identify in general terms the ability of a treatment process to reduce ESOC loading from the feed sludge to the treated sludge or biosolids. A higher score is not a criticism of the process because none of the treatment processes was either designed or implemented specifically for removal of these contaminants. The removal efficiencies are also not a reflection on the overall operation of all processes at a WWTP.

5.3.2 Comparison of Field Sampling Results with Literature Review Results

The literature review associated with the field study examined the occurrence and concentrations of ESOC in sludges and biosolids. The data obtained in this study can be compared to results with corresponding pharmaceutical compounds from the U.S. EPA's Targeted National Sewage Sludge Survey (TNSSS) (U.S. EPA, 2009) (**Table 123**).

Table 123. Comparison of Pharmaceutical Compound Occurrence and Concentration Data from This Study with the TNSSS Data

Pharmaceutical	This Study		EPA TNSSS		Ratio of median conc'ns TNSSS/ This Study
	Occurrence	Median Conc'n (ng/g TS dw)	Occurrence	Median Conc'n (ng/g TS dw)	
Triclocarban	100%	1930	100%	21050	10.9
Triclosan	97%	6085	94%	7615	1.3
Carbamazepine	100%	66.6	95%	37.4	0.6
Ciprofloxacin	94%	3610	100%	5910	1.6
Diphenhydramine	100%	420	100%	593	1.4
Erythromycin-H_2O	74%	12.5	92%	19.7[a]	1.6
Fluoxetine	84%	53.9	94%	146	2.7
Miconazole	100%	441	95%	472	1.1
Ofloxacin	87%	276	99%	4020	14.6

[a] Listed as total Erythromycin in U.S. EPA (2009)

For the most part, the corresponding compounds in this study and the TNSSS are comparable in frequency of occurrence and concentrations, as indicated by the final column of the table, which is the ratio of the median concentration of the TNSSS to the median concentration in final sludge or treated biosolids from this study. Of the nine compounds that can be compared, the ratio of the median concentrations for seven of the compounds falls between 0.6 and 2.7. For these six compounds, the ratio is greater than unity for 5 compounds, indicating that median levels in U.S. sludges are slightly higher than in the Canadian sludges and biosolids examined in this study.

For the other two pharmaceutical compounds (triclocarban and ofloxacin), median concentrations in the U.S. sludges were an order of magnitude higher than observed in the sludges and biosolids tested in this study. The higher median concentrations in the U.S. TNSSS than in this study may be reflective of a greater proportion of untreated sludges included in the U.S. study compared to this, as the extent of sludge treatment was not of primary consideration in the U.S. study. The results of this analysis suggest that data from the U.S. TNSSS can be used as a general indicator of compounds found in Canadian sludges and biosolids, with some compounds in Canadian samples being substantially lower than the levels in the U.S. TNSSS.

When the results of this field study were compared to the observations documented in the accompanying literature review, similar trends were noted. In both the literature and this field study, anaerobic digestion readily removed the antibiotic sulfamethoxazole and the non-steroidal anti-inflammatory drug naproxen, with ibuprofen removed to a lesser extent. Compounds such as the anti-epileptic carbamazepine, the anti-microbials triclosan and triclocarban, Bisphenol A, and the polycyclic musk fragrances HHCB and AHTN either remained unaffected by anaerobic digestion or increased in concentration through the process.

Few studies were identified that documented removal efficiencies of different biosolids treatment processes as this study did. Consequently, the technical literature is not able to provide much in the way of benchmarks for the field study results.

In the literature review, data provided by Kinney *et al*. (2006), suggested that there might be evidence of some reduction in ESOC concentrations resulting from certain biosolids treatment processes such as composting or drying. This indication was based only on differences in concentrations between the treated biosolids samples, however, and without accompanying raw sludge data, no firm conclusions can be drawn from the data of Kinney *et al*. (2006).

Sulfamethoxazole was found to be highly amenable to mesophilic anaerobic digestion in laboratory-scale studies by Carballa et al. (2006, 2007a). The sulfa drug was so readily degradable in anaerobic digestion (99% removal) that no difference in removal efficiency due to solids retention time could be discerned. This field study likewise identified that biological processes, whether aerobic or anaerobic tended to remove sulfamethoxazole to a high degree.

Studies by Carballa *et al*. (2006, 2007a) indicated that roxithromycin was highly degradable (85-99%) in laboratory-scale mesophilic anaerobic digesters. There were insufficient detectable concentrations of roxithromycin in the field survey to confirm the laboratory-scale data.

Trials with anaerobic digestion documented by Carballa *et al*. (2007a) at laboratory scale indicated that carbamazepine was not reduced by the treatment at time up to 30 days at mesophilic conditions. Ternes *et al*. (2005) also reported that batch anaerobic digestion tests resulted in no removal of carbamazepine. In a full-scale study in Canada, concentrations of carbamazepine and two metabolites were higher in anaerobically digested biosolids than in the feed sludge (Miao *et al*., 2005). This anti-epileptic compound was one of the more difficult compounds to remove by the different processes investigated in this study. Anaerobic digestion at Saskatoon and Red Deer resulted in negative removal efficiencies of the compound. Only composting appeared to have some success in reducing carbamazepine loads in feed sludges.

With non-steroidal anti-inflammatory drugs (NSAIDs), Carballa *et al*. (2007a) observed that naproxen was readily removed by mesophilic anaerobic digestion, even at the shortest retention times tested. Ibuprofen was more resistant to removal during anaerobic digestion, while diclofenac was relatively more resistant to removal in anaerobic digestion than either naproxen or ibuprofen. The relative removal efficiencies of the three NSAIDs were confirmed in laboratory batch anaerobic digestion tests completed by Ternes *et al*. (2005). Data from this field survey suggest that anaerobic digestion and geotextile bag filter dewatering are the only processes examined that are capable of reducing loads of naproxen in feed sludges. Similar to the studies of Carballa *et al*. (2007a) and Ternes *et al.* (2005), ibuprofen was less readily degradable in the mesophilic digestion processes of Saskatoon and Red Deer than was the naproxen.

The concentrations of the pharmaceuticals glibenclamide and famotidine were higher following anaerobic digestion than in the raw primary sludge, indicating that the drugs are not amenable to reduction by anaerobic digestion (Radjenović *et al*., 2009). These pharmaceuticals were not included in the target list of the field sampling study.

Lee and Peart (2002) assessed the concentrations of triclosan in both Canadian raw sludge and anaerobically digested biosolids. The median values of triclosan in raw sludge and digested biosolids were 10,600 and 14,450 ng/g TS, respectively, suggesting that there is no reduction of triclosan as a result of digestion. In this field study, the removal efficiencies of triclosan were negative, indicating a higher mass of the compound leaving the processes than entering them. Only the aerobic composting processes appeared successful in reducing the input mass of triclosan in the feed sludges.

Concentrations of the polycyclic musks increased from raw sludge to anaerobically digested biosolids, suggesting that no reduction was occurring to due anaerobic biodegradation (Lee *et al*., 2003; Yang at Metcalfe, 2005; Smyth *et al*., 2007). Carballa et al. (2007a), conversely, in laboratory-scale studies of mesophilic anaerobic digestion reported that the concentrations of polycyclic musks HHCB and AHTN were removed by approximately 50-70%.

Anaerobic digestion had an apparent beneficial reduction of the nitro musk compounds (Lee et al., 2003; Smyth et al., 2007). Data from Smyth *et al*. (2007) indicate aerobic digestion resulted in a decrease in the concentrations of the polycyclic musks, whereas increased concentrations of the polycyclic musks were observed following anaerobic digestion, in agreement with the data of Lee *et al*. (2003). Smyth *et al*. (2007 found that the nitro musk xylene concentrations in raw sludge were reduced by aerobic digestion, but were essentially unchanged by anaerobic digestion. Conversely, it appeared that musk ketone would be reduced in concentration by anaerobic digestion of the raw sludge (in agreement with the data from Lee *et al*. (2003)), whereas aerobic treatment would result in a slight increase in concentration.

Xia *et al*. (2005) and Das and Xia (2008). observed that composting periods of between 40 and 70 days reduce the starting level of 4-NP by over 90%. A higher proportion of wood shavings mixed with the biosolids results in a lower initial concentration of the 4-NP and a faster rate of reduction of the 4-NP. Gibson *et al*. (2007) found that composting resulted in a higher removal efficiency (88%) of 4-NP than did heat drying (39%). Conversely, Ghanem *et al*. (2007) noted

only an 18% reduction in 4-NP with composting, compared to removal efficiency of 72% for drying by pelletization. Removals of 4-NP by lime treatment were generally low (19-31%) (Gibson *et al*., 2007; Ghanem *et al*., 2007).

Concentrations of Bisphenol A in the raw sludge and anaerobically digested sludges from a number of Canadian municipalities were reported by Lee and Peart (2002). The median concentration of BPA in the raw sludge increased from 280 ng/g TS dw to 555 ng/g TS dw following anaerobic digestion. Two sites in this field study use mesophilic anaerobic digestion. At the Red Deer site, BPA increased from 295 to 515 ng/g TS dw following anaerobic digestion, a calculated negative increase of -47%. At the Saskatoon site however, the BPA concentrations in the feed sludge and digested biosolids were 6495 and 1765 ng/g TS dw, resulting in a calculated removal efficiency of 78%.

Lee *et al*. (2003) examined a number of fragrance compounds in raw sludges and anaerobically digested sludges from Canadian municipalities. Concentrations of the fragrance compounds were higher after anaerobic digestion. Similar results were obtained in this field study, as indicated in **Table 124**. The one exception was a decline in the concentration of AHDI at Red Deer following anaerobic digestion. The concentrations of HHCB, AHTN and ATII appear to be lower in this study than in the samples reported by Lee *et al*. (2003).

Table 124. Comparison of Literature and Study Fragrance Concentrations Before and After Anaerobic Digestion

	Fragrance Concentration (ng/g TS dw)					
Biosolids Source	**Galaxolide (HHCB)**	**Tonalide (AHTN)**	**Celestolide (ADBI)**	**Phantolide (AHDI or HMI)**	**Traesolide (ATII)**	**DPMI**
Median concentration raw sludge (Lee *et al*., 2003)	11850	8005	175	110	1345	
Median concentration anaerobic digested biosolids (Lee *et al*., 2003)	14500	12300	320	120	1870	
Red Deer Feed (this study)	3615	2090		780	335	190
Red Deer Digested (this study)	8975	4015		100	520	215
Saskatoon Feed (this study)	3205	1365		230	305	245
Saskatoon Digested (this study)	5130	2225		325	605	460

5.4 Triple Bottom Line Considerations in Biosolids Treatment Technologies

Based on Section 5.3, it appears that biological processes, specifically aerobic processes as represented by composting, offer the greatest probability of success in reducing the pharmaceuticals tested. [Note however there is a vast array of other ESOC that were not tested, including other pharmaceuticals, industrial chemicals, human hormones, and personal care products.] Because the aerobic processes appear most likely to succeed, methods that make the ESOC more readily available to the microbes involved in the biotransformation should be

considered. The organic compounds must be made more water-soluble so that they pass through the microbe's cell membrane to the cell interior, where biodegradation occurs. Essentially then, these processes would be intended to make the compounds less hydrophobic, or to eliminate the solid surfaces on which they accumulate.

With respect to changing the hydrophobicity of the compounds, procedures that change the environmental conditions of the sludge and biosolids, such as pH, ionic balances or redox conditions may be helpful. Such processes may transform a neutrally-charged compound to an ionic form, thus increasing the water solubility. Use of these processes would typically involve addition of chemicals. The overall environmental and social benefits of adding more bulk chemicals to sludges and biosolids to improve reductions of ESOC are not clear.

Other processes may disrupt the solids with which the ESOC are associated, reducing the surface areas for sorption sites. A number of processes are commercially available, for example, that are intended to disrupt the microbial cells in waste activated sludge (WAS), releasing their cell contents and making the treated WAS more amenable to anaerobic or aerobic digestion. Such processes typically involve the use of combinations of chemicals (acids or bases), heat or pressure. To the best of the authors' knowledge, such studies have not been published. Again, the overall environmental and social benefits of adding high temperature and pressure vessels at wastewater treatment plants specifically for improving reductions of ESOC in sludges and biosolids are not clear.

The Triple Bottom Line (TBL) approach or "People, Planet, Profit" captures an expanded spectrum of values and criteria for measuring organizational success. Triple bottom line assessments are very site-specific, and depend on local social and environmental values. In **Table 125**, a prototype TBL assessment is provided, highlighting some of the considerations of each of the processes employed in the site survey.

5.5 Best Management Practices for Emerging Contaminants in Biosolids

The sampling survey reported here provided an interesting look at different biosolids and sludge treatment processes, and their ability to remove metals, pharmaceutical, fragrance and alkylphenolic compounds present in the process feed sludge streams. The different treatment processes examined, however, are not replicated sufficiently to draw statistical inferences. Some of the processes in fact were represented by only one site. It is therefore difficult to state definitively from this initial survey which processes should be categorized as "Best Management Practices".

The data presented in Section 5.1 for metal concentrations in biosolids revealed very substantial reductions in most of the metals of industrial significance (e.g., electro-plating and surface finishing) over the past two to three decades. The reductions of these metals are almost entirely due to source control measures or substitution (e.g. substituting cadmium-plating with other metals). Such measures should continue to be implemented and enforced. It is interesting to note that the two metals that were regularly observed at the highest concentrations in the biosolids and

sludges are copper and zinc, which are commonly used in residential, commercial and institutional plumbing. Further reductions of these two metals in biosolids can be accomplished

Table 125. Prototype Triple Bottom Line Assessment for Biosolids Treatment Processes

Process	People		Planet					Profit		
	Worker safety	Pathogen & Vector Reduction	Use of Chemicals	Land footprint	Energy Consumption & Carbon footprint	ESOC Air Emissions	Aqueous Sidestream	Initial Cost	Operating Cost	Saleable Product
Biological – aerobic (Compost)	possible fungus or mould spores	can produce Class A or B biosolids	None	large areas for windrows	compost aeration consumes energy	possible minor stripping due to process aeration	leachate produced needs treatment	-	-	++
Biological – autothermal aerobic digestion	minimal	can produce Class A or B biosolids	None	minor area for digester	process produces its own thermal energy	possible minor stripping due to process aeration	no supernatant reported	--	0	0
Biological – mesophilic anaerobic digestion	minimal	can produce Class B biosolids	None	substantial for large plants	digester gas off-sets plant energy use	none	no supernatant reported	0	+	0
Physical – geotextile bag dewatering	minimal	Class B biosolids not achieved	flocculants and dewatering aids	minimal pad area for bags	low energy	none	filtrate needs treatment	++	+	-
Physical-chemical (alkaline stabilisation)	minimal	can produce Class B biosolids	alkaline materials (cement kiln dust)	larger processing facility	thermal energy required for curing	possible minor volatilization due to process heat	no sidestream	--	-	++
Physical – filter press dewatering	minimal	Class B biosolids not achieved	flocculants and dewatering aids	dewatering building required	low energy requirements for belt press	minimal	press filtrate needs treatment	--	0	-
Physical – thermal drying	possible fire/explosive hazard	can produce Class A or B biosolids	None	drying building required	thermal drying consumes energy	possible minor volatilization due to process heat	no sidestream	--	--	++

Cost Legend: + = benefit; 0 = neutral; - = disadvantage

by a substitution of plumbing pipes and appurtenances with other materials such as polyvinyl chloride (PVC) or high density polyethylene (HDPE) if the concentrations in the biosolids warrant this expenditure.

With respect to removal of pharmaceutical compounds by biosolids or sludge treatment, the technology that appears to be more effective than others is composting, an aerobic biological process that operates at thermophilic (e.g. approximately 55^{o}C) temperatures for an extended period of time. Anaerobic digestion removes a limited number of different pharmaceutical compounds, presumably because of the different microbial consortia present in the two environments with and without oxygen. If greater reduction of ESOC in biosolids is determined by risk assessment to be necessary, a combination of treatments may act as a multi-barrier approach for reducing concentrations in treated biosolids. For smaller municipalities, the geotextile bag filter dewatering process may offer some reduction in pharmaceuticals at low cost. It is possible that the ability of geotextile bag filters to reduce levels of some contaminants in sludges is due to a long holding time in the geotextile bag, potentially including a freeze-thaw cycle, which provides time and opportunity for some microbes to degrade the compounds.

If some pharmaceutical compounds of potential concern are difficult to remove by the biosolids treatment processes examined herein, consideration may be given to preventing their deposition in the sludge feed streams for the biosolids processes. This prevention concept may be implemented in several ways. First, many of the pharmaceutical compounds are hydrophobic, and are thus removed to a great extent with the underflow from primary clarifiers. Because of this early removal from the liquid treatment train, they are not subject to aerobic biological treatment, which could enhance their overall removal from the incoming wastewater. It may be possible that overall reduction in pharmaceutical compounds could be improved without a primary clarification step, as is often practiced with the extended aeration process and aerated lagoons used at smaller municipalities. Implementation of such a practice in conventional activated sludge processes would represent a radical departure from existing design and operating philosophies, however. Alternatively, preliminary separate treatment of the primary sludge, either by aerobic or other treatment, prior to mixing with secondary sludge, may provide reduced concentrations of pharmaceuticals entering the biosolids or sludge treatment processes.

Pre-ozonation of the feed sludge may also provide some beneficial effect on removal in the biosolids treatment processes. Carballa *et al*. (2007b) evaluated the potential beneficial effect of pre-ozonating feed sludge prior to anaerobic digestion at laboratory scale. Other than the poorer results obtained with the non-steroidal anti-inflammatory drugs (naproxen, ibuprofen and diclofenac), pre-ozonation of the raw feed sludge appeared to have a generally beneficial effect on removal of a variety of contaminants (sulfamethoxazole, carbamazepine, two musk fragrances) by anaerobic digestion.

Source control of pharmaceutical compounds may be accomplished to some extent through pharmaceutical take-back programs and education of the public that they should not flush unused medications via toilets to the sanitary sewer system. Product substitution is likely difficult to implement, as the public needs their medications. Other ESOC, such as fragrances, surfactants and anti-microbials could be candidates for product substitution, however.

Depending on the mode of action, some pharmaceuticals can be metabolized in the body and excreted in urine. Others are excreted in feces. For those pharmaceuticals that are excreted in urine, use of toilets equipped with urine traps may help to remove the compounds from entering the wastewater stream. Such a shift in technology substitution would require a long period to implement across the country.

5.6 Knowledge Gaps and Research Needs

This study afforded an opportunity to investigate in detail the potential removal of ESOC by sludge and biosolids treatment processes commonly used in Canada. The study produced much valuable information on the fate of the ESOC selected for investigation, but as is often the case the acquisition of new knowledge leads to additional questions. Below are listed some of the knowledge gaps and research needs arising from this survey and from the literature review (Hydromantis, 2009) in no particular order of importance.

This study looked at a select group of pharmaceuticals, fragrance and alkylphenolic compounds. Due to budgetary limitations, it did not look at myriad other ESOC, including many other pharmaceutical compounds, natural and synthetic human hormones, industrial chemicals (e.g. phthalate esters, polybrominated diphenyl ethers and other flame retardants, perfluorinated organic substances, alkylphenol ethoxylates, quaternary ammonium compounds), and personal care products (insect repellents, sunscreens, parabens, organic siloxanes, fabric softeners, fluorescent whitening agents, etc.). Research at full-scale similar to this study for these many types of ESOC is encouraged to round out the knowledge of ESOC behaviour in biosolids treatment processes. [At the time of this report preparation, another field study was being conducted by Environment Canada under the Chemical Management Plan to analyse sample of wastewater liquid and solids process streams for a range of substances including selected pharmaceutical and personal care products, brominated flame retardants, perfluorinated organic compounds, volatile methyl siloxanes nonylphenol ethoxylates and a suite of 18 metals (Smythe, 2010).]

Some unexpected results were obtained in this study, both positive and otherwise. An unexpected result was the reduction of a number of organic ESOC by the geotextile bag filter dewatering process at the Eganville, ON treatment plant. Only one of this type of dewatering process was included in this sampling survey. Additional sites using this technology should be tested in a similar manner to determine if the process does offer a low-cost means of dewatering wastewater sludge with better removal efficiencies of more ESOC than other processes examined herein. Factors to consider in additional testing should include the type of feed solids (primary sludge, septage, waste activated sludge) to the process, loss of ESOC in bag filtrate, possible effect of freezing and thawing, retention time and microbial aerobic/anaerobic activity in the geotextile bags.

The autothermal aerobic digestion process exhibited lower removal efficiencies of pharmaceutical, fragrance and alkylphenolic compounds than might have been expected considering it is an aerobic process that operates at an elevated temperature, which should result in faster removal rates. The possible reason may be that the relatively short detention time at the elevated temperature of thermophilic operation (e.g. approximately 55 ^{o}C) reduces the number

and types of microbes that can biodegrade the ESOC. Composting is an aerobic process in which temperatures reach thermophilic conditions, which is similar to those experienced in the ATAD process. Additional studies with this type of process should be undertaken to determine this discrepancy.

It was observed that composting of sludges to produce biosolids generally resulted in the highest removal efficiencies of most ESOC. A limited number of pharmaceuticals, such as naproxen, however, survived and apparently increased through the composting process. Mesophilic anaerobic digestion of sludges was found to substantially reduce concentrations of naproxen, but was less successful in overall removal of ESOC. The ability of a combination of anaerobic digestion, followed by dewatering and composting, or lime/alkaline stabilisation followed by composting, for example, might provide a means of reducing more of the ESOC, including other that were not tested in this program. Such a study, either at pilot-scale or at existing full-scale facilities with this treatment combination would be helpful in determining the possible benefits of different redox environments for ESOC removal.

The biological treatment processes for biosolids in general were able to reduce ESOC in the feed sludge more efficiently than were the physical (including physical-chemical) processes. Of the physical-chemical processes, the N-Viro alkaline stabilisation process appeared to offer the best performance for ESOC removal. Only one example of this process was included in this survey (i.e. the Halifax site). Moncton, NB uses a partially lime-stabilised biosolids as the feed material for the composting operation, but the focus there was on the composting process, rather than on lime stabilisation. Additional testing of lime- and alkaline-stabilisation processes for reduction of ESOC should be undertaken.

The thermal drying process (pelletisation) was not efficient in the reduction of ESOC, with the knowledge that it was not intended for that purpose. A study should be undertaken to assess whether tt may be possible to accomplish greater reduction of ESOC to take advantage of thermal or chemical decomposition by a change in process operating conditions.

It is of high importance to evaluate whether the detected concentrations of pharmaceuticals and other ESOC in land applied biosolids could are of concern for either human health or environmental risk. The U.S. EPA is currently conducting such risk assessments (Hebert, 2010). The results of such studies will help to determine if further reductions in concentration of specific compounds may be needed. Studies by Carballa *et al*. (2007b) indicated that pre-ozonation of the feed sludge to the anaerobic digestion process generally resulted in improved removal of several classes of ESOC. Because it is unlikely that source control can restrict inputs of pharmaceuticals to wastewater treatment plants, the cost-effectiveness of improving the removal of the compounds by biosolids treatment processes by pre-ozonation or other processes should be investigated, including sludge feeds to all the different biosolids treatment processes (i.e., not just anaerobic digestion).

6. CONCLUSIONS

The conclusions that follow relate to the suite of target ESOC evaluated in this study.

1. Metal contaminants in biosolids are in general unaffected by the biosolids stabilisation process employed, as compared to organic constituents. A potential exception may be mercury, which can be biologically activated in anaerobic environments, and also undergo transfer from biosolids to the gas phase by stripping or volatilisation.
2. All median metal concentration in sludge and biosolids, with the exception of selenium, met the current most stringent quality criteria for land application, although a limited number of exceedances were observed for copper, mercury and molybdenum on a site-specific basis.
3. Metal concentration of biosolids and septage were quite similar, indicating that metals in biosolids now mainly originate from domestic rather than industrial sources.
4. Although 24 pharmaceutical, alkylphenolic and fragrance compounds were found in detectable concentrations in more than 75% of the feed sludge samples, only 14 of 71 pharmaceutical, alkylphenolic and fragrance compounds (20%) were found in more than 75% of the treated biosolids samples likely to be land applied.
5. The antibacterial compounds triclosan and triclocarban, the antibiotic ciprofloxacin, the fragrance compound HHCB were the compounds most frequently detected (9 or more of 11 sites) above 1000 ng/g TS dw.
6. For the most part, the corresponding compounds in this study and the U.S. EPA's Targeted National Sewage Sludge Survey (TNSSS) are comparable in frequency of occurrence and concentrations.
7. Biosolids stabilisation processes using some form of biological treatment are more efficient at reducing the organic ESOC concentrations than are non-biological processes.
8. Of the biological treatment processes, the composting process (aerobic) appears to be more effective in overall reduction (in number and degradation) of ESOC than does mesophilic anaerobic digestion.
9. ESOC removed efficiently by composting, but not well reduced by anaerobic digestion include compounds such as ciprofloxacin, miconazole, triclosan, gemfibrozil, thiabendazole, carbamazepine, Bisphenol A, HHCB, AHTN, AHDI, and ATII.
10. The autothermal aerobic digestion process was much less effective in reducing ESOC than was either composting or mesophilic anaerobic digestion.
11. The geotextile bag filter used for dewatering sludge and septage was capable of reducing a number of ESOC, although the exact mechanism is unclear at this time.
12. Of the physical processes (including physical-chemical) processes, the N-Viro alkaline stabilisation process appeared to offer the best performance for ESOC removal
13. The thermal drying process (pelletisation) alone was not efficient in the reduction of ESOC, acknowledging that it was not intended for that purpose.
14. Mechanical sludge dewatering processes alone are among the least effective for reducing concentrations of ESOC in the feed sludge.
15. A few pharmaceutical compounds appear to be removed readily by either aerobic or anaerobic biological treatment, including sulfamethoxazole, trimethoprim, caffeine and diltiazem.

16. A limited number of pharmaceutical compounds appeared to be difficult to remove in almost all processes examined, when present at detectable concentrations. These included the diuretic furosemide, the anti-epileptic carbamazepine, and the antibiotic ofloxacin.
17. Naproxen appears to increase substantially through aerobic composting, possibly due to biotransformation from other compounds, but it appears to be more efficiently removed by anaerobic digestion.
18. While many of the ESOC remain associated with the solid phase of the sludges or biosolids, a number of compounds can be lost in any aqueous process sidestream (e.g., dewatering filtrate, leachate, digester supernatant), including furosemide, ibuprofen and 2-hydroxy-ibuprofen, naproxen, acetaminophen, caffeine, carbamazepine, clarithromycin, dehydronifedipine, erythromycin-H_2O, sulfamethoxazole and trimethoprim.
19. Less than 1% of the mass of fragrance compounds in feed sludge resides in the process sidestreams or leachates from the treatment processes, while between 1% and 6% of the mass of Bisphenol A in the feed sludges was transferred to the process sidestreams or leachates.
20. A combination of processes (e.g. anaerobic digestion plus dewatering plus composting as at Prince Albert; lime stabilisation plus composting as at Moncton) result in the highest reductions of many ESOC.
21. The treatment efficiencies of ESOC by anaerobic digestion observed in this field study are comparable to results reported in the technical literature; published removal efficiencies of ESOC in other biosolids treatment processes are sparse.
22. The ESOC concentration data in sludges and biosolids produced in this sampling program are insufficient alone, without applying formal risk assessment methods, to determine human health or environmental risks of managed biosolids land application, land reclamation, and production of commercial and soil amendments.

7. RECOMMENDATIONS

1. Risk assessments should be conducted with ESOC to evaluate if they may pose risk to human health or the environment when applied to land amended with biosolids. Based on frequency and concentrations observed in the treated sludges and biosolids, candidate compounds for initial risk assessment may include triclosan and triclocarban, ciprofloxacin, the fragrances HHCB and AHTN, and BPA, although other factors such as persistence, bioaccumulation potential and toxicity also need to be considered.
2. Research at full-scale, similar to this study, for many other types of ESOC (other classes of pharmaceutical compounds, natural and synthetic human hormones, industrial chemicals (e.g. phthalate esters, brominated flame retardants, perfluorinated organic substances, alkylphenol ethoxylates, quaternary ammonium compounds), and personal care products (insect repellents, sunscreens, parabens, organic siloxanes, fabric softeners, fluorescent whitening agents, etc.) is encouraged to round out the knowledge of ESOC behaviour in biosolids treatment processes.
3. Additional sites using the geotextile bag filtration technology should be tested in a manner similar to this survey to determine if the process offers a low-cost means of dewatering wastewater sludge with substantial removal efficiencies of certain ESOC. Factors to consider in additional testing should include the type of feed solids (primary sludge, septage, waste activated sludge) to the process, loss of ESOC in bag filtrate, possible effect of freezing and thawing, and retention time in the geotextile bags.
4. Additional sampling of the autothermal aerobic digestion process at other locations should be undertaken to determine if the lower removal efficiencies of pharmaceutical, fragrance and alkylphenolic compounds observed at the one site tested (compared to other aerobic processes such as composting), was an isolated event, or is representative of the process behaviour with respect to ESOC.
5. Because only one example of lime- or alkaline-stabilisation processes was included in this survey, and because the alkaline stabilisation process appeared to offer the best performance for ESOC removal of any of the physical (including physical-chemical) processes, additional testing should be undertaken for confirmation and optimization of ESOC reduction.
6. A study examining the ability of a combination of processes (e.g. anaerobic digestion, followed by dewatering and composting; alkaline/lime stabilization followed by composting), either at pilot- or full-scale, is recommended for determining the possible benefits of different redox environments for reducing ESOC, including others that were not tested in this program.
7. Because only one example of lime- or alkaline-stabilisation processes was included in this survey, and because the alkaline stabilisation process appeared to offer the best performance for ESOC removal of any of the physical (including physical-chemical) processes, additional testing to document reduction of ESOC by this type of process should be undertaken.
8. Studies of pre-treatment of feed sludges, such as by ozonation, prior to the biosolids treatment processes should be investigated to determined the potential beneficial effects and cost-effectiveness for overall improvement in ESOC removal efficiencies.
9. Data produced by this and similar investigations need to be transferred out to appropriate departments and agencies, federal and provincial regulators, municipalities and academic researchers for risk assessment purposes.

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APPENDIX A. RAW ANALYTICAL DATA FOR PHARMACEUTICAL, FRAGRANCE AND ALKYLPHENOLIC COMPOUNDS

Table A-1. Concentrations and Detection Limits of Pharmaceutical Compounds for Salmon Arm, BC, Samples

Item	First Sampling Round				Second Round Sampling				Third Round Sampling			
CCME Sample ID #	ATAD Feed Sludge		ATAD Digested Sludge		ATAD Feed Sludge		ATAD Digested Sludge		ATAD Feed Sludge		ATAD Digested Sludge	
Lab Work Order #	L12952-1		L12952-2		L13283-3		L13283-4		L13380-1		L13380-2	
Sampled Date	29-Jun-09		29-Jun-09		11-Aug-09		11-Aug-09		19-Aug-09		19-Aug-09	
Sample Rec'vd Date	30-Jun-09		30-Jun-09		12-Aug-09		12-Aug-09		25-Aug-09		25-Aug-09	
Result Rept Date (AN)	13-Jul-09		13-Jul-09						3-Oct-09		3-Oct-09	
Result Rept Date (AP)	6-Aug-09		6-Aug-09		16-Sep-09		16-Sep-09					
Parameters	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)
Furosemide	ND	130	ND	287	233	111	543	109	ND	162	ND	108
Gemfibrozil	49.9	4.89	219	10.7	47.2	4.16	245	17.1	42.2	6.23	177	4.14
Glipizide	ND	19.5	ND	43	ND	16.7	ND D	68.4	ND	24.3	ND	16.1
Glyburide	ND	9.77	ND	21.5	ND	8.33	ND D	34.2	ND	12.1	ND	8.07
Hydrochlorothiazide	ND	65.1	ND	143	106	55.5	ND D	228	ND	80.9	ND	53.8
2-Hydroxy-ibuprofen	609	261	1570	573	387	222	1160	912	ND	324	571	215
Ibuprofen	196	48.9	3010	119	359	41.6	1130	171	466	60.7	1960	40.3
Naproxen	85.8	9.77	431	42.2	82.7	8.33	247	37.2	127	12.6	278	8.07
Triclocarban	3360	9.77	6700	21.5	3700	22.2	4900	34.2	3080	12.1	5010	8.07
Triclosan	6670	195	21300	430	8390	167	24000	684	9640	243	21500	161
Warfarin	ND	4.89	ND	10.7	ND	4.16	ND D	17.1	ND	6.07	ND	4.03
Acetaminophen	367	195	ND	430	299	167	ND	228	ND	243	ND	161
Azithromycin	267	4.89	385	10.7	154	4.16	219	5.7	112	6.07	220	4.03
Caffeine	1360	154	4550	144	1260	41.6	2960	57	1270	60.7	4110	40.3
Carbadox	ND	4.89	ND	10.7	ND	4.16	ND	5.7	ND	6.07	ND	4.03
Carbamazepine	423	4.89	2360	10.7	168	4.16	579	5.7	213	6.07	717	4.03
Cefotaxime	ND	218	ND	1260	ND	32.3	ND	37.5	ND	26.4	ND	55.9
Ciprofloxacin	9620	19.5	6900	56.2	13000	16.7	8210	40.8	14400	24.3	4220	63.1
Clarithromycin	344	4.89	249	10.7	50.4	4.16	126	5.7	71.1	6.07	73.4	4.03
Clinafloxacin	ND	36	ND	91.2	ND	16.7	ND	22.8	ND	27.8	ND	55.4

Cloxacillin	ND	13.6	ND	44.5	ND	8.33	ND	11.4	ND	12.1	ND	8.06
Dehydronifedipine	9.94	1.95	7.36	6.04	9.19	1.67	ND	3.19	6.28	2.43	4.71	2.51
Diphenhydramine	559	3.09	807	9.82	451	1.67	612	2.28	424	2.43	514	1.61
Diltiazem	480	0.977	27.7	3.01	162	0.862	21.7	1.14	192	1.21	5.94	0.806
Digoxin	ND	48.9	ND	107	ND	41.6	ND	57	ND	138	ND	40.3
Digoxigenin	ND	19.5	ND	130	ND	16.7	ND	22.8	ND	42.9	ND	116
Enrofloxacin	12.5	9.77	ND	21.5	14.1	10.1	ND	12.9	20.4	14.3	ND	20.1
Erythromycin-H2O	33.4	0.977	95.3	2.15	27.5	0.833	29.8	1.14	18.8	1.21	31.9	0.806
Flumequine	ND	4.89	ND	14	ND	4.16	ND	5.7	ND	6.07	ND	4.03
Fluoxetine	153	6.94	278	16.8	122	4.16	96.7	5.7	127	6.07	80.1	4.03
Lincomycin	ND	9.77	71	21.5	ND	8.33	ND	12.3	ND	28.3	ND	54.4
Lomefloxacin	ND	9.77	ND	21.5	ND	8.33	ND	11.4	ND	12.1	ND	8.06
Miconazole	683	4.89	1710	10.7	901	4.16	1160	5.7	533	6.07	1350	4.47
Norfloxacin	99.2	48.9	154	134	434	41.6	ND	57	410	60.7	ND	82.3
Norgestimate	ND	10.4	ND	37	ND	11.3	ND	14.2	ND	12.1	ND	24.7
Ofloxacin	300	48.9	279	107	326	41.6	245	57	394	60.7	187	40.3
Ormetoprim	ND	1.95	ND	4.3	ND	1.67	ND	2.28	ND	2.43	ND	1.61
Oxacillin	ND	9.77	ND	21.5	ND	8.33	ND	11.4	ND	12.1	ND	8.06
Oxolinic Acid	ND	2.68	ND	6.15	ND	1.67	ND	2.45	ND	2.43	ND	1.62
Penicillin G	ND	9.77	ND	21.5	ND	27.8	ND	38	ND	12.1	ND	8.06
Penicillin V	ND	10.3	59.3	38.1	ND	8.33	ND	11.4	ND	12.1	ND	8.06
Roxithromycin	ND	0.977	ND	2.15	ND	1.08	ND	1.32	ND	1.21	ND	0.806
Sarafloxacin	ND	48.9	ND	107	ND	97.5	ND	135	ND	276	ND	128
Sulfachloropyridazine	ND	4.89	ND	10.7	ND	4.16	ND	5.7	ND	6.07	ND	4.03
Sulfadiazine	ND	4.89	ND	10.7	ND	4.16	ND	5.7	ND	6.07	ND	4.03
Sulfadimethoxine	ND	0.977	ND	14.6	ND	0.833	ND	1.14	ND	1.21	ND	66.2
Sulfamerazine	ND	1.95	36.3	4.3	ND	1.67	19.2	2.73	ND	2.43	ND	1.61
Sulfamethazine	ND	1.95	ND	4.3	ND	2.87	ND	5.45	ND	3.44	ND	4.9
Sulfamethizole	ND	2.75	ND	7.19	ND	1.67	ND	2.4	ND	2.43	ND	3.3
Sulfamethoxazole	43	2.01	ND	4.3	20.3	2.58	3.41	2.74	25.8	2.43	ND	1.61
Sulfanilamide	ND	48.9	ND	107	ND	41.6	92.5	57	ND	60.7	164	40.3
Sulfathiazole	ND	4.89	ND	10.7	ND	4.16	ND	5.7	ND	6.07	ND	4.03
Thiabendazole	18	4.89	27.5	10.7	16	4.16	21.7	5.7	14.1	6.07	13.4	4.91
Trimethoprim	75.3	4.89	ND	41.5	60.2	4.16	36.4	21.3	56.5	12.9	ND	4.03
Tylosin	ND	19.5	ND	43	ND	16.7	ND	22.8	ND	81	ND	53.8

Virginiamycin	ND	46.7	ND	157	ND	115	ND	127	ND	90.3	197	138
1,7-Dimethylxanthine	1030	489	2800	1070	ND	416	1030	570	ND	607	1850	403
% Moisture	94.3		97.4		92.9		94.8		89		92.7	

ND = not detected

Detected concentration

Table A-2. Concentrations and Detection Limits of Fragrance and Alkylphenolic Compounds for Salmon Arm, BC, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	ATAD Feed Sludge		Digested Sludge		ATAD Feed Sludge		Digested Sludge	
Lab Work Order #	L13206-5		L13206-6		L13206-13		L13206-14	
Sampled Date	11-Aug-09		11-Aug-09		19-Aug-09		19-Aug-09	
Sample Received Date	12-Aug-09		12-Aug-09		25-Aug-09		25-Aug-09	
Result Report Date	28-Jan-10		28-Jan-10		28-Jan-10		28-Jan-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	520	80	700	80	1050	80	1740	80
Octylphenol	ND	20	ND	20	60	20	70	20
Nonylphenol	NA	140	NA	140	NA	140	NA	140
DPMI	130	40	180	40	120	40	210	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	230	30	210	30	900	30	530	30
HHCB	7520	90	8800	90	6430	90	8570	90
AHTN	3510	70	4230	70	3870	70	4650	70
ATII	740	60	1140	60	780	60	910	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	<LOQ	120	ND	120	ND	120	ND	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	410	70	<LOQ	70	80	70	<LOQ	70
Total Musks	12530		14560		12190		14890	
% Moisture	92.90		94.80		89.00		92.70	
% TSS	7.10		5.20		11.00		7.30	

ND = not detected; LOQ = Limit of Quantitation

Detected concentration

Table A-3. Concentrations and Detection Limits of Pharmaceutical Data for Red Deer, AB, Samples

Item	First Round Sampling				Second Round Sampling				Third Round Sampling			
CCME Sample ID #	Digester Feed Sludge		Digested Sludge #1 Grab		Digester Feed Sludge		Digested Sludge #1 Grab		Digester Feed Sludge		Digested Sludge #1 Grab	
Lab Work Order #	L12972-6		L12972-3		L13139-6		L13139-3		L13435-3		L13435-4	
Sampled Date	29-Jun-09		2-Jul-09		23-Jul-09		23-Jul-09		27-Aug-09		27-Aug-09	
Sample Rec'vd Date	3-Jul-09		3-Jul-09		24-Jul-09		24-Jul-09		28-Aug-09		28-Aug-09	
Result Report Date (AN)	14-Jul-09		14-Jul-09		12-Aug-09		12-Aug-09		3-Oct-09		3-Oct-09	
Result Report Date (AP)	6-Aug-09		6-Aug-09		20-Aug-09		20-Aug-09		6-Oct-09		6-Oct-09	
Parameters	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)	Conc. (ng/g)	Detect'n Limit (ng/g)
Furosemide	ND	236	ND	692	ND	732	ND	398	ND	83.6	93.9	88.8
Gemfibrozil	17.8	8.85	75.7	25.9	136	27.4	57.5	14.9	18	3.22	22.4	3.42
Glipizide	ND	35.4	ND	104	ND	110	ND	59.6	ND	12.5	ND	13.3
Glyburide	ND	17.7	ND	51.9	ND	54.9	ND	29.8	ND	6.27	ND	6.66
Hydrochlorothiazide	164	118	349	346	ND	366	ND	199	ND	41.8	ND	44.4
2-Hydroxy-ibuprofen	755	472	ND	1380	1540	1460	ND	795	638	167	348	178
Ibuprofen	502	106	1910	259	1330	274	686	149	257	31.4	350	33.3
Naproxen	73.6	17.7	ND	51.9	270	54.9	35.3	29.8	66.4	6.27	ND	6.66
Triclocarban	2440	17.7	4710	51.9	10800	54.9	4410	29.8	2660	49.2	3560	6.66
Triclosan	9130	354	13900	1040	36800	1100	11700	596	5340	125	12700	133
Warfarin	ND	8.85	ND	25.9	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Acetaminophen	551	354	ND	1040	ND	1100	ND	594	181	126	ND	133
Azithromycin	616	8.85	793	26	1830	27.4	679	14.9	459	3.14	419	3.33
Caffeine	2530	88.5	No data		8970	284	175	149	1700	31.4	ND	33.3
Carbadox	ND	8.85	ND	26	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Carbamazepine	230	8.85	1060	26	1070	27.4	987	14.9	260	3.14	430	3.33
Cefotaxime	ND	505	ND	1320	ND	567	ND	173	ND	20.6	ND	38.7
Ciprofloxacin	3690	35.4	8450	104	17000	110	6520	59.4	4390	12.6	4140	23.7

Clarithromycin	142	8.85	77.4	26	442	27.4	111	14.9	62.3	3.14	20.2	3.33
Clinafloxacin	ND	41.6	ND	120	ND	116	ND	59.4	ND	16.7	ND	33
Cloxacillin	ND	20.5	ND	67.6	ND	54.8	ND	29.7	ND	6.28	ND	6.65
Dehydronifedipine	6.17	3.54	ND	10.4	14.3	11	ND	5.94	3.75	1.26	ND	1.33
Diphenhydramine	1510	5.21	2300	16.1	4380	11	2360	5.94	1640	1.26	1980	1.33
Diltiazem	209	1.77	35.2	9.83	947	5.48	41.2	2.97	129	0.628	17.2	0.665
Digoxin	ND	88.5	ND	260	560	495	ND	242	ND	31.4	ND	33.3
Digoxigenin	ND	35.4	ND	104	257	248	193	119	ND	40.5	ND	27.8
Enrofloxacin	ND	17.7	ND	51.9	82.9	54.8	35.5	29.7	6.78	6.28	19.1	13.1
Erythromycin-H2O	24.1	1.77	26.9	5.19	171	5.48	20	2.97	36.7	0.628	5.25	0.665
Flumequine	ND	8.85	ND	26	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Fluoxetine	126	10.4	255	26	373	27.4	297	14.9	154	3.14	120	3.33
Lincomycin	ND	17.7	ND	51.9	ND	54.8	ND	29.7	28.8	14.6	ND	15.5
Lomefloxacin	ND	17.7	ND	51.9	ND	54.8	ND	29.7	ND	6.28	ND	6.65
Miconazole	429	8.85	1220	26	1720	27.4	1090	14.9	225	3.14	518	3.33
Norfloxacin	2100	88.5	4380	260	10200	274	3270	149	1910	31.4	1810	33.3
Norgestimate	ND	17.7	ND	51.9	ND	57.8	ND	35.6	ND	6.28	ND	10.5
Ofloxacin	416	88.5	1290	260	1790	274	712	149	263	31.4	649	33.3
Ormetoprim	ND	3.54	ND	10.4	ND	11	ND	5.94	ND	1.26	ND	1.33
Oxacillin	ND	17.7	ND	51.9	ND	61.5	ND	29.7	ND	6.28	ND	6.65
Oxolinic Acid	ND	3.54	ND	12.5	ND	11	ND	5.94	ND	1.26	ND	1.55
Penicillin G	ND	17.7	ND	51.9	ND	54.8	ND	29.7	ND	6.28	ND	6.65
Penicillin V	ND	17.7	ND	51.9	ND	54.8	ND	29.7	ND	6.28	ND	6.65
Roxithromycin	ND	1.77	ND	5.19	ND	7.89	ND	2.97	ND	1.25	ND	0.872
Sarafloxacin	ND	88.5	ND	260	ND	408	ND	202	ND	130	ND	346
Sulfachloropyridazine	ND	8.85	ND	26	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Sulfadiazine	ND	8.85	ND	26	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Sulfadimethoxine	ND	1.77	ND	5.19	ND	5.48	ND	2.97	ND	1.2	ND	0.665
Sulfamerazine	ND	3.54	ND	10.4	ND	12.9	ND	7.61	ND	1.26	ND	1.33
Sulfamethazine	ND	3.54	ND	10.4	ND	11	ND	5.94	6.13	1.49	ND	1.39
Sulfamethizole	ND	3.54	ND	10.4	ND	11	ND	5.94	ND	1.37	ND	1.52
Sulfamethoxazole	22	3.54	ND	10.4	43.6	11	ND	5.94	16.7	1.26	ND	1.33
Sulfanilamide	ND	88.5	ND	260	ND	274	ND	149	ND	31.4	ND	33.3
Sulfathiazole	ND	8.85	ND	26	ND	27.4	ND	14.9	ND	3.14	ND	3.33
Thiabendazole	9.66	8.85	ND	26	35	27.4	25.4	14.9	10	3.14	14.9	3.33

Trimethoprim	58.7	8.85	ND	26	262	39.8	ND	14.9	54.8	6.5	ND	3.33
Tylosin	ND	35.4	ND	104	ND	110	ND	59.4	ND	71.7	ND	44.3
Virginiamycin	ND	156	ND	249	ND	373	ND	182	ND	131	ND	139
1,7-Dimethylxanthine	ND	885	ND	2600	ND	2740	ND	1490	475	314	ND	333
% Moisture	96.6		99		98.9		98.1		90.6		91.1	

ND = not detected

Detected concentration (on dry weight basis)

Table A-4. Concentrations and Detection Limits of Fragrance and Alkylphenolic Compounds for Red Deer, AB, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Digester Feed Sludge		Digested Sluge #1 Grab		Digester Feed Sludge		Digested Sluge #1 Grab	
Lab Work Order #	L13139-6		L13139-3		L13206-19		L13206-20	
Sampled Date	23-Jul-09		23-Jul-09		27-Aug-09		27-Aug-09	
Sample Received Date	24-Jul-09		24-Jul-09		28-Aug-09		28-Aug-09	
Result Report Date	17-Mar-10		17-Mar-10		28-Jan-10		28-Jan-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	140	80	280	80	450	80	750	80
Octylphenol	<LOQ	20	<LOQ	20	ND	20	ND	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	<LOQ	140
DPMI	<LOQ	40	30	40	190	40	400	40
ADBI	<LOQ	20	60	20	ND	20	ND	20
AHDI	<LOQ	30	50	30	780	30	150	30
HHCB	2640	90	6830	90	4590	90	11120	90
AHTN	1350	70	2850	70	2830	70	5180	70
ATII	120	60	210	60	550	60	830	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	50	120	<LOQ	120	ND	120	ND	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	<LOQ	70	<LOQ	70	<LOQ	70	<LOQ	70
Total Musks	4160		10070		8950		17700	
% Moisture	98.9		98.1		90.6		91.1	
% TSS	1.1		1.9		9.4		8.9	

ND = not detected; LOQ = Limit of Quantitation

Detected concentration

Table A-5. Concentrations and Detection Limits of Pharmaceutical Data for Saskatoon, SK, Samples

Item	First Round Sampling				Second Round Sampling				Third Round Sampling			
CCME Sample ID #	Digester Feed Sludge		Digested Sludge		Digester Feed Sludge		Digested Sludge		Digester Feed Sludge		Digested Sludge	
Lab Work Order #	L13054-2		L13054-1		L13174-2		L13174-1		L13283-7		L13283-8	
Sampled Date	13-Jul-09		13-Jul-09		28-Jul-09		28-Jul-09		11-Aug-09		11-Aug-09	
Sample Received Date	16-Jul-09		16-Jul-09		29-Jul-09		29-Jul-09		12-Aug-09		12-Aug-09	
Result Report Date (AN)	12-Aug-09		12-Aug-09		31-Aug-09		31-Aug-09		9/182009		18-Sep-09	
Result Report Date (AP)	20-Aug-09		20-Aug-09		4-Sep-09		4-Sep-09		16-Sep-09		16-Sep-09	
Parameters	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)	Conc. (ng/g TS)	Detect'n Limit (ng/g TS)
Furosemide	ND	165	ND	138	ND	256	ND	203	ND	326	ND	617
Gemfibrozil	50.4	6.17	55.7	5.9	34.3	9.86	75.2	7.81	52	12.2	110	23.1
Glipizide	ND	24.7	ND	20.7	ND	38.4	ND	30.4	ND	48.9	ND	92.5
Glyburide	ND	12.3	ND	10.3	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Hydrochlorothiazide	ND	82.3	ND	68.9	234	133	143	101	ND	163	ND	308
2-Hydroxy-ibuprofen	535	329	561	276	ND	512	432	406	ND	653	ND	1230
Ibuprofen	308	61.7	365	51.7	362	96	561	76	323	122	1160	231
Naproxen	97.5	12.3	ND	10.3	165	19.2	ND	15.2	155	24.5	56.5	46.2
Triclocarban	1760	12.3	1930	10.3	1680	19.2	1850	15.2	1550	24.5	3130	56.5
Triclosan	4150	247	5590	207	3540	384	6270	304	4090	489	6050	925
Warfarin	ND	6.17	ND	5.17	ND	9.6	ND	7.6	ND	12.2	ND	23.1
Acetaminophen	ND	247	ND	207	1110	384	ND	475	ND	489	ND	925
Azithromycin	621	6.17	480	5.17	399	9.6	330	7.61	164	12.2	426	23.1
Caffeine	1740	61.7	136	71.1	2130	96	ND	76.1	1670	122	ND	231
Carbadox	ND	6.17	ND	5.17	ND	9.6	ND	7.61	ND	12.2	ND	23.1
Carbamazepine	79.2	6.17	131	5.17	48.1	9.6	115	7.61	64.7	12.2	133	23.1
Cefotaxime	ND	162	ND	295	ND	98	ND	63.2	ND	48.9	ND	92.5
Ciprofloxacin	3390	24.7	3100	20.7	3570	38.4	3610	30.5	3580	48.9	6900	92.5
Clarithromycin	141	6.17	84.3	5.17	71	9.6	38.6	7.61	23.4	12.2	31.2	23.1

Clinafloxacin	ND	41.1	ND	21.1	ND	38.4	ND	30.5	ND	48.9	ND	92.5
Cloxacillin	ND	13.4	ND	11.1	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Dehydronifedipine	3.45	2.47	ND	2.07	3.92	3.84	ND	3.05	ND	4.89	ND	9.25
Diphenhydramine	1310	3.23	994	2.07	1050	3.84	984	3.05	1180	4.89	2290	9.25
Diltiazem	186	1.23	29.9	1.03	201	1.92	11.5	1.52	61	2.45	18.7	4.62
Digoxin	ND	327	287	154	ND	96	ND	76.1	ND	122	ND	231
Digoxigenin	ND	96.4	63.1	35.9	ND	84	ND	44.3	ND	48.9	ND	92.5
Enrofloxacin	14.1	12.3	21.1	10.3	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Erythromycin-H2O	62.2	1.23	312	1.03	46.1	1.92	13.7	1.52	58.7	2.45	33.1	4.62
Flumequine	ND	6.17	ND	5.17	ND	9.6	ND	7.61	ND	12.2	ND	23.1
Fluoxetine	126	7.61	62.6	5.17	109	9.6	55.8	7.61	49.6	12.2	130	23.1
Lincomycin	ND	12.3	ND	10.3	ND	37.9	ND	22.3	ND	24.5	ND	46.2
Lomefloxacin	ND	12.3	ND	10.3	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Miconazole	418	6.17	517	5.17	226	9.6	375	7.61	259	12.2	488	23.1
Norfloxacin	ND	61.7	87.1	51.7	312	96	ND	76.1	ND	122	ND	231
Norgestimate	ND	20.5	ND	12.5	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Ofloxacin	108	61.7	109	51.7	ND	96	90	76.1	ND	122	232	231
Ormetoprim	ND	2.47	ND	2.07	ND	3.84	ND	3.05	ND	4.89	ND	9.25
Oxacillin	ND	13	ND	11	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Oxolinic Acid	ND	2.73	ND	2.07	ND	3.84	ND	3.05	ND	4.89	ND	9.25
Penicillin G	ND	12.3	ND	10.3	ND	19.2	ND	15.2	ND	81.6	ND	154
Penicillin V	ND	12.3	ND	10.3	ND	19.2	ND	15.2	ND	24.5	ND	46.2
Roxithromycin	ND	2.66	ND	1.05	ND	2.04	ND	1.52	ND	2.87	ND	4.62
Sarafloxacin	ND	144	ND	210	ND	116	ND	90.3	ND	122	ND	231
Sulfachloropyridazine	ND	6.17	ND	5.17	ND	9.6	ND	7.61	ND	12.2	ND	23.1
Sulfadiazine	ND	6.17	ND	5.17	ND	9.6	ND	7.61	ND	12.2	ND	23.1
Sulfadimethoxine	ND	1.23	ND	1.03	ND	1.92	ND	1.52	ND	2.45	ND	4.62
Sulfamerazine	ND	5.08	ND	4.42	7.21	3.84	8.03	3.05	ND	4.89	ND	9.25
Sulfamethazine	ND	4.43	ND	3.14	ND	3.84	ND	3.05	ND	4.89	ND	9.25
Sulfamethizole	ND	2.47	ND	2.07	ND	3.84	ND	3.05	ND	4.89	ND	9.25
Sulfamethoxazole	35.8	2.47	ND	2.07	33.3	3.84	ND	3.05	10.3	4.91	ND	9.25
Sulfanilamide	ND	61.7	ND	51.7	ND	96	ND	76.1	ND	122	ND	231
Sulfathiazole	ND	6.17	ND	5.17	ND	9.6	ND	7.61	ND	12.2	ND	23.1
Thiabendazole	13.4	6.17	18.8	5.17	12.4	9.6	16.9	7.61	19.2	12.2	ND	23.1
Trimethoprim	144	6.17	ND	5.17	147	9.6	12.5	7.61	74	12.2	ND	23.1

Tylosin	ND	24.7	ND	20.7	ND	128	ND	102	ND	48.9	ND	92.5
Virginiamycin	ND	202	ND	53.4	ND	158	ND	128	ND	100	ND	276
1,7-Dimethylxanthine	ND	617	ND	517	ND	960	ND	761	ND	1220	ND	2310
% Moisture	95.1		94.4		96.9		96		97.6		98.7	

ND = not detected

Detected concentration (on dry weight basis)

Table A-6. Concentrations and Detection Limits of Fragrance and Alkylphenolic Compound for Saskatoon, SK, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Digester Feed Sludge		Digested Sludge		Digester Feed Sludge		Digested Sludge	
Lab Work Order #	L13174-2		L13174-1		L13206-9		L13206-10	
Sampled Date	28-Jul-09		28-Jul-09		11-Aug-09		11-Aug-09	
Sample Received Date	29-Jul-09		29-Jul-09		12-Aug-09		12-Aug-09	
Result Report Date	28-Jan-10		28-Jan-10		28-Jan-10		28-Jan-10	
Parameters	Conc. (ng/g TS)	LOQ (ng/g TS)	Conc. (ng/g TS)	LOQ (ng/g TS)	Conc. (ng/g TS)	LOQ (ng/g TS)	Conc. (ng/g TS)	LOQ (ng/g TS)
Bisphenol A	790	80	970	80	12200	80	2560	80
Octylphenol	20	20	40	20	ND	20	60	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	<LOQ	140
DPMI	420	40	770	40	70	40	150	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	230	30	150	30	230	30	500	30
HHCB	4160	90	5470	90	2250	90	4790	90
AHTN	1630	70	2260	70	1100	70	2190	70
ATII	300	60	690	60	310	60	520	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	ND	120	ND	120	ND	120	ND	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	70	70	<LOQ	70	<LOQ	70	<LOQ	70
Total Musks	6780		9350		3970		8140	
% Moisture	96.9		96		97.6		98.7	
% TSS	3.1		4		2.4		1.3	

ND = not detected LOQ = Limit of Quantitation

Detected concentration (on dry weight basis)

Table A-7. Concentrations and Detection Limits of Pharmaceutical Data in Dewatered Biosolids Cake and Compost for Prince Albert, SK, Samples

Item	First Round Sampling				Second Round Sampling			
	Dewatered Cake		Compost		Dewatered Cake		Compost	
Sampled Date	29-Sep-09		29-Sep-09		15-Oct-09		15-Oct-09	
Sample Received Date	1-Oct-09		1-Oct-09		16-Oct-09		16-Oct-09	
Result Report Date (AN)	23-Oct-09		25-Nov-09		1-Dec-09		1-Dec-09	
Result Report Date (AP)	22-Oct-09		4-Dec-09		1-Dec-09		1-Dec-09	
Parameters	Conc. (ng/g TS)	Detection Limit (ng/g TS)	Conc. (ng/g TS)	Detection Limit (ng/g TS)	Conc. (ng/g TS)	Detection Limit (ng/g TS)	Conc. (ng/g TS)	Detection Limit (ng/g TS)
Furosemide	167	103	ND	153	ND	124	817	247
Gemfibrozil	80	2.96	12	2.77	63.9	2.76	97	2.48
Glipizide	ND	11.8	ND	11.1	ND	11.1	11.4	9.9
Glyburide	ND	5.92	ND	5.53	ND	5.53	6.63	4.95
Hydrochlorothiazide	ND	39.5	ND	36.9	ND	36.8	ND	33
2-Hydroxy-ibuprofen	ND	158	ND	148	ND	147	ND	132
Ibuprofen	254	29.6	ND	27.7	179	27.6	310	24.8
Naproxen	94.5	5.92	4170	6.43	106	5.53	1030	11.4
Triclocarban	1490	5.92	1660	5.53	2270	5.53	1610	4.95
Triclosan	6300	118	2320	111	8300	111	5580	99
Warfarin	ND	2.96	ND	2.77	ND	2.76	ND	2.48
Acetaminophen	ND	118	ND	110	ND	111	ND	99
Azithromycin	1460	2.96	24.4	2.75	1680	2.76	356	2.48
Caffeine	846	29.6	596	27.5	446	27.6	1470	24.8
Carbadox	ND	2.96	ND	2.75	ND	2.76	ND	2.48
Carbamazepine	58.2	2.96	43.6	2.75	67.9	2.76	63.7	2.48
Cefotaxime	ND	38.1	ND	116	ND	63.3	ND	74.7
Ciprofloxacin	5990	15.4	860	138	7200	26	3180	9.9
Clarithromycin	42.6	2.96	4.49	2.75	39.7	2.76	22	2.48
Clinafloxacin	ND	11.8	ND	238	ND	20	ND	39.6

Cloxacillin	ND	3.91	ND	4.35	ND	3.92	ND	6
Dehydronifedipine	4.19	1.53	ND	1.53	3.51	1.11	ND	1.23
Diphenhydramine	2860	1.18	680	1.17	2610	1.79	1760	1.07
Diltiazem	254	0.592	ND	0.549	175	0.553	24.3	0.495
Digoxin	ND	29.6	ND	27.5	ND	27.6	ND	24.8
Digoxigenin	ND	41.4	ND	14.3	ND	22.7	ND	59.3
Enrofloxacin	ND	7.64	ND	64.3	ND	11.9	ND	9.19
Erythromycin-H2O	46.6	0.592	7.06	0.549	55.9	0.553	18	0.495
Flumequine	ND	2.96	ND	2.75	ND	2.76	ND	2.48
Fluoxetine	65.9	2.96	38.3	2.75	100	2.76	73.3	2.48
Lincomycin	ND	13.8	ND	12.8	ND	12.9	ND	11.6
Lomefloxacin	ND	5.92	ND	21.6	ND	5.53	ND	4.95
Miconazole	591	2.96	205	2.75	293	2.76	330	2.48
Norfloxacin	ND	29.6	ND	27.5	ND	27.6	ND	62.8
Norgestimate	ND	8.81	ND	7.51	ND	8.13	ND	9.84
Ofloxacin	92.9	29.6	ND	55.9	137	27.6	53.1	24.8
Ormetoprim	ND	1.18	ND	1.1	ND	1.11	ND	0.99
Oxacillin	ND	5.92	ND	5.49	ND	5.53	ND	4.95
Oxolinic Acid	1.35	1.18	ND	2.42	ND	3.93	ND	3.08
Penicillin G	ND	2.37	ND	2.2	ND	2.21	ND	1.98
Penicillin V	ND	5.92	ND	5.49	ND	5.53	ND	6.19
Roxithromycin	ND	0.592	ND	0.655	ND	2.28	ND	1.53
Sarafloxacin	ND	146	ND	498	ND	164	ND	107
Sulfachloropyridazine	ND	2.96	ND	2.75	ND	2.76	ND	2.48
Sulfadiazine	ND	2.96	ND	2.75	ND	2.76	ND	2.48
Sulfadimethoxine	ND	0.592	ND	0.549	ND	5.94	ND	1.83
Sulfamerazine	ND	2.05	ND	1.1	ND	2.57	ND	1.71
Sulfamethazine	ND	2.64	ND	1.1	7	3.57	ND	4.95
Sulfamethizole	ND	1.52	ND	1.1	ND	1.11	ND	1.62
Sulfamethoxazole	23.5	1.18	ND	1.1	34.2	1.18	ND	0.99
Sulfanilamide	ND	29.6	ND	27.5	ND	27.6	ND	24.8
Sulfathiazole	ND	2.96	ND	2.75	2.93	2.76	ND	2.48
Thiabendazole	23.6	2.96	7.96	2.75	21.4	2.76	14.8	2.48
Trimethoprim	261	5.75	ND	2.75	273	3.01	85.9	11.8
Tylosin	ND	39.5	ND	77.8	ND	11.1	ND	9.9

Virginiamycin	ND	201	ND	54.9	ND	5.53	ND	16.5
1,7-Dimethylxanthine	ND	296	ND	275	ND	276	ND	248
% Moisture	81.7		32.6		77		56.4	

ND = not detected

Detected concentration (on dry weight basis)

Table A-8. Concentrations and Detection Limits of Fragrance and Alkylphenolic Compound for Prince Albert, SK, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Dewatered Cake		Compost		Dewatered Cake		Compost	
Lab Work Order #	L13206-29		L13206-28		L13206-33		L13206-32	
Sampled Date	29-Sep-09		29-Sep-09		15-Oct-09		15-Oct-09	
Sample Received Date	1-Oct-09		1-Oct-09		16-Oct-09		16-Oct-09	
Result Report Date	17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	120	80	100	80	90	80	130	80
Octylphenol	<LOQ	20	<LOQ	20	<LOQ	20	<LOQ	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	<LOQ	140
DPMI	70	40	40	40	<LOQ	40	ND	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	<LOQ	30	<LOQ	30	<LOQ	30	ND	30
HHCB	1240	90	3920	90	1870	90	3020	90
AHTN	470	70	730	70	690	70	360	70
ATII	100	60	130	60	70	60	90	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	40	120	40	120	<LOQ	120	<LOQ	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	<LOQ	70	<LOQ	70	<LOQ	70	<LOQ	70
Total Musks	1910		4860		2670		3810	

ND = not detected LOQ = Limit of Quantitation

Detected concentration (on dry weight basis

Table A-9. Concentrations and Detection Limits of Pharmaceutical Data for Eganville, ON, Samples

Item	First Sampling Round						Second Round Sampling						Third Round Sampling					
CCME Sample ID #	WAS		Dewatered Biosolids		Filtrate		Raw Septage		Dewatered Biosolids		Filtrate		WAS		Dewatered Biosolids		Filtrate	
Lab Work Order #	L13068-2		L13072-1		L13068-1		L13283-5		L13283-6		L13285-1		L13380-6		L13380-5		L13285-5	
Sampled Date	14-Jul-09		14-Jul-09		14-Jul-09		11-Aug-09		11-Aug-09		11-Aug-09		25-Aug-09		25-Aug-09		25-Aug-09	
Sample Received Date	17-Jul-09		17-Jul-09		17-Jul-09		13-Aug-09		13-Aug-09		13-Aug-09		26-Aug-09		26-Aug-09		26-Aug-09	
Result Report Date (AN)	12-Aug-09		12-Aug-09		14-Aug-09		18-Sep-09		18-Sep-09		15-Oct-09		3-Oct-09		3-Oct-09		2-Oct-09	
Result Report Date (AP)	13-Aug-09		13-Aug-09		18-Aug-09		16-Sep-09		16-Sep-09		8-Oct-09		6-Oct-09		6-Oct-09		2-Oct-09	
Parameters	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/L)**	**Detect-ion Limit (ng/L)**	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/L)**	**Detect-ion Limit (ng/L)**	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/g TS)**	**Detect-ion Limit (ng/g TS)**	**Conc. (ng/L)**	**Detect-ion Limit (ng/L)**
Furosemide	ND	144	ND	371	ND	180	987	955	1120	56.7	9800	360	ND	156	ND	148	ND	553
Gemfibrozil	ND	5.4	ND	13.9	ND	1.71	ND	35.8	ND	16.8	ND	4.77	ND	6.02	ND	5.68	ND	2.85
Glipizide	ND	21.6	ND	55.6	ND	6.81	ND	143	ND	22.5	ND	19.1	ND	23.4	ND	22.1	ND	11.1
Glyburide	ND	10.8	ND	27.8	6.95	3.41	ND	71.6	16.5	11.2	89.6	9.54	ND	11.7	ND	11.1	14.1	5.54
Hydrochlorothia-zide	117	72	ND	185	1090	22.7	4700	477	ND	225	8030	63.6	ND	78.1	ND	73.8	1220	36.9
2-Hydroxy-ibuprofen	ND	288	ND	741	ND	90.8	39000	1910	ND	898	208000	373	ND	313	ND	295	18100	148
Ibuprofen	ND	54	ND	139	174	17	11300	358	931	168	75700	142	62.8	58.6	176	55.3	4870	27.7
Naproxen	13.7	10.8	ND	27.8	7.68	3.41	3740	71.6	41.7	33.7	23900	35.4	16.6	11.7	ND	11.1	1090	6.62
Triclocarban	1410	10.8	1140	27.8	4.08	3.41	16900	94.7	6580	42.7	321	9.54	1360	11.7	2550	11.1	16.2	5.54
Triclosan	901	216	602	556	ND	68.1	12700	1430	30600	674	1490	191	817	234	3050	221	ND	111
Warfarin	ND	5.4	ND	13.9	ND	1.7	ND	35.8	ND	5.61	20.5	4.77	ND	5.86	ND	5.53	ND	2.77
Acetaminophen	ND	216	ND	556	ND	68.1	121000	1750	ND	225	226000	3000	ND	234	ND	221	4710	249
Azithromycin	126	5.4	88.8	13.9	135	1.7	230	35.8	58.9	5.61	1200	1.85	94.9	5.86	111	5.54	236	2.77
Caffeine	80.5	54	148	139	ND	17	10000	358	1070	56.1	97400	26.3	ND	58.6	ND	55.4	1320	27.7
Carbadox	ND	5.4	ND	13.9	ND	1.7	ND	35.8	ND	5.61	NQ		ND	5.86	ND	5.54	ND	2.77

Carbamazepine	43.2	5.4	40.2	13.9	738	1.7	94	35.8	172	5.61	897	4.63	34.5	5.86	31.9	5.54	991	6.7
Cefotaxime	ND	122	ND	77.7	ND	6.81	ND	143	ND	22.5	NQ		ND	25.3	ND	31.9	ND	64.9
Ciprofloxacin	5330	21.6	5800	55.6	118	40.2	7570	143	26800	39.2	112	89.9	7440	23.4	6470	22.1	196	143
Clarithromycin	32.3	5.4	27.5	13.9	195	1.7	225	35.8	40.3	5.61	1770	1.59	17.2	5.86	32.3	5.54	145	2.77
Clinafloxacin	ND	21.6	ND	55.6	ND	134	ND	143	ND	27.4	ND	144	ND	31.5	ND	55	ND	83.1
Cloxacillin	ND	10.8	ND	27.8	ND	3.41	ND	71.6	ND	11.2	NQ		ND	11.7	ND	11.1	ND	13.1
Dehydronifedi-pine	2.86	2.16	ND	5.56	32.2	0.681	ND	14.3	ND	2.25	28.1	4.21	5.56	2.34	2.88	2.21	57.2	3.03
Diphenhydramine	223	2.16	203	5.56	141	1.02	900	14.3	525	2.25	798	1.8	223	2.34	286	2.21	213	1.11
Diltiazem	19.4	1.08	6.24	2.78	1.63	0.341	96.4	7.16	4.95	1.12	278	0.675	21.6	1.17	5.4	1.11	24.5	0.555
Digoxin	ND	55.4	ND	139	ND	17	ND	358	96.4	56.1	NQ		ND	58.6	ND	118	ND	27.7
Digoxigenin	ND	45.9	ND	81.2	ND	115	ND	143	ND	22.5	NQ		ND	32.2	ND	67.9	ND	11.1
Enrofloxacin	11.2	10.8	ND	27.8	ND	9.61	ND	71.6	ND	11.2	NQ		ND	11.7	ND	23	ND	14.5
Erythromycin-H2O	13.6	1.08	10.1	2.78	133	0.341	16.9	7.16	46.9	1.12	117	0.318	9.94	1.17	10.7	1.11	28.5	0.555
Flumequine	ND	5.4	ND	13.9	ND	1.7	ND	35.8	ND	5.61	NQ		ND	5.86	ND	5.54	ND	2.77
Fluoxetine	38	5.4	45.9	13.9	5.9	1.7	ND	35.8	101	5.61	ND	7.02	19.3	5.86	28.7	5.54	ND	2.77
Lincomycin	ND	10.8	ND	27.8	ND	3.41	ND	71.6	ND	11.2	NQ		ND	27.3	ND	25.8	ND	18.1
Lomefloxacin	ND	10.8	ND	27.8	ND	8.62	ND	71.6	ND	11.2	NQ		ND	11.7	ND	11.1	ND	10.1
Miconazole	328	5.4	350	13.9	ND	1.7	211	35.8	376	5.61	NQ		221	5.86	291	5.54	ND	2.77
Norfloxacin	458	54	581	139	ND	85.7	600	358	5590	56.1	NQ		1090	58.6	451	59.8	ND	27.7
Norgestimate	ND	12	ND	27.8	ND	3.81	ND	71.6	ND	11.2	NQ		ND	11.7	ND	19.1	ND	13.2
Ofloxacin	433	54	351	139	ND	17	ND	358	975	56.1	NQ		763	58.6	754	55.4	ND	27.7
Ormetoprim	ND	2.16	ND	5.56	ND	0.681	ND	14.3	ND	2.25	NQ		ND	2.34	ND	2.21	ND	1.11
Oxacillin	ND	10.8	ND	27.8	ND	3.41	ND	71.6	ND	11.2	NQ		ND	11.7	ND	11.1	ND	5.55
Oxolinic Acid	ND	2.16	ND	5.56	ND	1.73	ND	14.3	ND	3.43	NQ		ND	2.34	ND	2.62	ND	5.7
Penicillin G	ND	10.8	ND	27.8	4.73	3.83	ND	239	ND	37.4	NQ		ND	11.7	ND	11.1	ND	5.55
Penicillin V	ND	10.8	ND	27.8	ND	4.51	ND	71.6	ND	11.2	NQ		ND	11.7	ND	11.1	ND	5.83
Roxithromycin	ND	1.08	ND	2.78	ND	0.341	ND	7.16	ND	2.05	ND	3.64	ND	1.17	ND	1.19	ND	0.555
Sarafloxacin	ND	140	ND	165	ND	94.4	ND	358	ND	209	NQ		ND	372	ND	306	ND	27.7
Sulfachloropyrida-zine	ND	5.4	ND	13.9	NQ		ND	35.8	ND	5.61	ND	27.2	ND	5.86	ND	5.54	NQ	
Sulfadiazine	ND	5.4	ND	13.9	NQ		ND	35.8	ND	5.61	ND	7	ND	5.86	ND	5.54	NQ	
Sulfadimethoxine	ND	1.08	ND	2.78	NQ		ND	7.16	ND	1.12	ND	5.92	ND	1.17	ND	1.11	NQ	
Sulfamerazine	ND	2.16	ND	5.56	NQ		ND	14.3	ND	2.25	126	26.5	ND	2.34	ND	2.21	NQ	
Sulfamethazine	ND	2.16	ND	5.56	NQ		ND	14.3	ND	3.5	ND	0.636	ND	2.36	ND	4.07	NQ	

Sulfamethizole	ND	2.16	ND	5.56	NQ		ND	14.3	ND	2.25	22.7	4.47	ND	2.34	ND	2.21	NQ	
Sulfamethoxazole	41	2.16	17.4	5.56	NQ		577	14.3	ND	2.25	1100	1.83	79.4	2.34	4.26	2.21	NQ	
Sulfanilamide	ND	54	ND	139	NQ		ND	358	ND	56.1	ND	15.9	ND	58.6	ND	55.4	NQ	
Sulfathiazole	ND	5.4	ND	13.9	NQ		ND	35.8	ND	5.61	71.7	5.76	ND	5.86	ND	5.54	NQ	
Thiabendazole	18.2	5.4	14.4	13.9	29.2	1.7	ND	35.8	12.1	5.61	39.9	4.26	22.1	5.86	16.2	5.54	24.1	2.77
Trimethoprim	ND	5.4	ND	13.9	ND	21	106	35.8	59.5	19.3	507	38	ND	5.86	ND	5.54	ND	31.2
Tylosin	ND	21.6	ND	55.6	NQ		ND	143	ND	22.5	ND	21.2	ND	78.1	ND	73.8	ND	37
Virginiamycin	ND	40	ND	67.5	ND	3.41	ND	349	ND	87.2	NQ		ND	77.3	ND	110	ND	278
1,7-Dimethylxanthine	ND	540	ND	1390	ND	170	23700	3580	ND	561	69200	4550	ND	586	ND	554	8300	277
% Moisture	94.9		84.6				99.2		85.6		99.94		95		86.4		99.98	

ND = not detected NQ = not quantified

Detected concentration

Table A-10. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Eganville, ON, Samples

Item	First Round Sampling						Second Round Sampling					
	WAS		Dewatered Biosolids		Filtrate		Raw Septage		Dewatered Biosolids		Filtrate	
Lab Work Order #	L13068-2		L13072-1		L13068-1		L13206-7		L13206-8		L13286-1	
Sampled Date	14-Jul-09		14-Jul-09		14-Jul-09		11-Aug-09		11-Aug-09		11-Aug-09	
Sample Received Date	17-Jul-09		17-Jul-09		17-Jul-09		13-Aug-09		13-Aug-09		13-Aug-09	
Result Report Date	28-Jan-10		28-Jan-10		28-Jan-10		28-Jan-10		28-Jan-10		not received	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)
Bisphenol A	290	80	160	80	<LOQ	50	1550	80	2590	80	550	50
Octylphenol	<LOQ	20	ND	20	20	10	ND	20	ND	20	<LOQ	10
Nonylphenol	NA	140	NA	140	NA	90	NA	140	NA	140	<LOQ	90
DPMI	140	40	70	40	ND	20	210	40	180	40	ND	20
ADBI	ND	20	ND	20	ND	10	ND	20	ND	20	<LOQ	10
AHDI	40	30	50	30	ND	10	470	30	370	30	ND	10
HHCB	1890	90	2330	90	830	30	8220	90	8990	90	570	30
AHTN	1110	70	1510	70	590	40	3930	70	2090	70	120	40
ATII	270	60	250	60	<LOQ	10	960	60	550	60	30	10
M MOSKENE	ND	50	ND	50	ND	90	ND	50	ND	50	ND	90
M TIBETENE	ND	80	ND	80	ND	50	ND	80	ND	80	ND	50
M KETONE	ND	120	ND	120	ND	90	<LOQ	120	<LOQ	120	40	90
M AMBRETTE	ND	140	ND	140	ND	20	ND	140	ND	140	ND	20
M XYLENE	<LOQ	70	<LOQ	70	ND	20	730	70	530	70	<LOQ	20
Total Musks	3460		4220		1420		14520		12700		780	
% Moisture	94.9		84.6		100		99.2		85.6		99.94	
% TSS	5.1		15.4		0		0.8		14.4		0.06	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-11. Concentrations and Detection Limits of Pharmaceutical Compounds in Smiths Falls, ON, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Belt Press Cake		Heat Dried Pellets		Belt Press Cake		Heat Dried Pellets	
Lab Work Order #	L13435-6		L13435-7		L13672-12		L13672-13	
Sampled Date	2-Sep-09		2-Sep-09		19-Oct-09		19-Oct-09	
Sample Received Date	3-Sep-09		3-Sep-09		20-Oct-09		20-Oct-09	
Result Report Date (AN)	23-Oct-09		23-Oct-09		1-Dec-09		1-Dec-09	
Result Report Date (AP)	22-Oct-09		22-Oct-09		1-Dec-09		1-Dec-09	
Parameters	Conc. (ng/g)	Detection Limit (ng/g)	Conc. (ng/g)	Detection Limit (ng/g)	Conc. (ng/g)	Detection Limit (ng/g)	Conc. (ng/g)	Detection Limit (ng/g)
Furosemide	441	83.1	238	123	169	142	ND	107
Gemfibrozil	11.3	2.9	8.69	2.59	7.54	2.68	12.9	2.68
Glipizide	ND	11.6	ND	10.3	ND	10.7	ND	10.7
Glyburide	ND	5.8	ND	5.17	ND	5.37	6.39	5.35
Hydrochlorothiazide	ND	38.7	ND	34.5	ND	35.8	ND	35.7
2-Hydroxy-ibuprofen	ND	155	ND	138	ND	143	ND	143
Ibuprofen	183	29	204	25.9	210	26.8	211	26.8
Naproxen	69.8	5.8	48.2	5.17	53.9	5.37	86.2	5.35
Triclocarban	4470	9.73	3830	5.17	4490	5.44	4090	8.41
Triclosan	11900	116	14300	103	11800	107	8670	107
Warfarin	ND	2.9	ND	2.59	ND	2.68	ND	2.68
Acetaminophen	ND	116	ND	103	ND	107	ND	107
Azithromycin	95.3	2.9	138	2.81	127	2.68	153	2.68
Caffeine	98.3	29	118	25.9	82.7	26.8	175	26.8
Carbadox	ND	2.9	ND	2.59	ND	2.68	ND	2.68
Carbamazepine	40.8	2.9	91.3	2.59	57.6	2.68	108	2.68
Cefotaxime	ND	17.2	ND	25.5	ND	39.4	ND	52.6

Ciprofloxacin	3380	11.6	2160	10.3	6130	10.7	2900	10.7
Clarithromycin	30.6	2.9	31.8	2.59	83.1	2.68	58	2.68
Clinafloxacin	ND	16.8	ND	14.3	ND	17.2	ND	22.8
Cloxacillin	ND	2.79	ND	3.25	ND	2.3	ND	4.17
Dehydronifedipine	4.63	1.18	9.02	1.28	4.3	1.07	10.2	1.07
Diphenhydramine	571	1.16	895	1.03	609	1.07	770	1.28
Diltiazem	143	0.596	284	0.517	177	0.537	321	0.535
Digoxin	ND	29	ND	25.9	ND	26.8	ND	26.8
Digoxigenin	ND	24.4	ND	69.9	ND	28.4	ND	26.4
Enrofloxacin	10.2	6.36	8.05	7.26	16.4	5.6	12.2	6.02
Erythromycin-H2O	8.9	0.58	11	0.517	9.45	0.537	7.15	0.535
Flumequine	ND	2.9	ND	2.59	ND	2.68	ND	2.68
Fluoxetine	97	2.9	96.4	2.59	70.4	2.68	83.6	2.68
Lincomycin	ND	13.5	ND	12	ND	12.5	ND	12.5
Lomefloxacin	ND	5.8	ND	5.17	ND	5.37	ND	5.35
Miconazole	408	2.9	609	2.59	360	2.68	474	3.17
Norfloxacin	1800	29	1090	25.9	2490	26.8	1190	26.8
Norgestimate	ND	6.16	ND	9.85	ND	6.71	ND	9.62
Ofloxacin	149	29	101	25.9	415	26.8	233	26.8
Ormetoprim	ND	1.16	ND	1.03	ND	1.07	ND	1.07
Oxacillin	ND	5.8	ND	5.17	ND	5.37	ND	5.35
Oxolinic Acid	ND	1.16	1.04	1.03	ND	1.07	ND	1.12
Penicillin G	ND	2.32	ND	2.07	ND	2.15	ND	2.14
Penicillin V	ND	5.8	ND	5.17	ND	5.37	ND	5.35
Roxithromycin	ND	0.58	ND	0.517	ND	2.41	ND	2.95
Sarafloxacin	ND	41.7	ND	61.2	ND	85.4	ND	159
Sulfachloropyridazine	ND	2.9	ND	2.59	ND	2.68	ND	2.68
Sulfadiazine	ND	2.9	ND	2.59	ND	2.68	ND	2.68
Sulfadimethoxine	ND	0.58	ND	0.517	ND	1.49	ND	1.48
Sulfamerazine	ND	1.16	ND	4.59	ND	2.09	ND	2.98
Sulfamethazine	ND	2.23	ND	3.01	ND	4.94	ND	6.32
Sulfamethizole	ND	1.77	ND	2.02	ND	1.51	ND	2.21
Sulfamethoxazole	3.67	1.16	18.6	1.48	11.6	2.32	38.3	1.08
Sulfanilamide	ND	29	63.1	25.9	ND	26.8	ND	26.8
Sulfathiazole	ND	2.9	ND	2.59	ND	3.02	ND	2.68

Thiabendazole	7.82	2.9	7.88	2.59	6.6	2.68	5.62	2.68
Trimethoprim	30	8.87	30.6	9.08	31.9	4.41	31.8	9.42
Tylosin	ND	38.7	ND	34.5	ND	10.7	ND	10.7
Virginiamycin	ND	145	ND	157	ND	17.9	ND	17.8
1,7-Dimethylxanthine	ND	290	ND	259	ND	268	ND	268

ND = not detected

Detected concentration (on dry weight basis)

Table A-12. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Smiths Falls, ON, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Belt Press Cake		Heat Dried Pellets		Belt Press Cake		Heat Dried Pellets	
Lab Work Order #	L13206-22		L13206-23		L13206-35		L13206-36	
Sampled Date	2-Sep-09		2-Sep-09		19-Oct-09		19-Oct-09	
Sample Received Date	3-Sep-09		3-Sep-09		20-Oct-09		20-Oct-09	
Result Report Date	17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	<LOQ	80	<LOQ	80	110	80	120	80
Octylphenol	<LOQ	20	<LOQ	20	<LOQ	20	<LOQ	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	<LOQ	140
DPMI	ND	40	ND	40	<LOQ	40	<LOQ	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	<LOQ	30	<LOQ	30	<LOQ	30	<LOQ	30
HHCB	3810	90	2850	90	4170	90	2740	90
AHTN	1740	70	550	70	1340	70	1110	70
ATII	70	60	100	60	130	60	90	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	<LOQ	120	<LOQ	120	<LOQ	120	<LOQ	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	<LOQ	70	<LOQ	70	<LOQ	70	<LOQ	70
Total Musks	5650		3520		5780		4030	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-13 Concentrations and Detection Limits of Pharmaceutical Data for Gatineau Valley, QC, Samples

Item	First Round Sampling				Second Round Sampling						Third Round Sampling					
CCME Sample ID #	Dry Mud		Compost		Dry Mud		Compost		Leachate from Composting		Dry Mud		Compost		Leachate from Composting	
Lab Work Order #	L13108-1		L13108-2		L13672-3		L13672-1		L13732-3		L13672-10		L13672-14		L13732-9	
Sampled Date	21-Jul-09		21-Jul-09		29-Sep-09		29-Sep-09		29-Sep-09		13-Oct-09		27-Oct-09		13-Oct-09	
Sample Received Date	22-Jul-09		22-Jul-09		30-Sep-09		30-Sep-09		30-Sep-09		14-Oct-09		28-Oct-09		14-Oct-09	
Result Report Date (AN)	12-Aug-09		12-Aug-09		25-Nov-09		25-Nov-09		25-Nov-09		1-Dec-09		30-Nov-09		25-Nov-09	
Result Report Date (AP)	20-Aug-09		20-Aug-09		4-Dec-09		4-Dec-09		26-Nov-09		1-Dec-09		3-Dec-09		26-Nov-09	
Parameters	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/L)	Detect-ion Limit (ng/L)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/L)	Detect-ion Limit (ng/L)
Furosemide	760	349	ND	425	ND	238	ND	390	ND	379	318	77.9	ND	80.4	ND	335
Gemfibrozil	66.2	13.1	ND	15.9	ND	8.91	ND	2.54	37.2	1.56	3.98	2.83	ND	3.01	45.3	1.6
Glipizide	ND	52.4	ND	63.7	ND	35.6	ND	10.2	ND	6.25	ND	11.3	ND	12.1	ND	6.42
Glyburide	27.4	26.2	ND	31.9	801	17.8	ND	5.08	30.8	3.13	70.9	5.66	ND	6.03	32.4	3.21
Hydrochlorothia-zide	532	175	ND	212	235	39.6	ND	33.9	ND	20.8	133	37.8	ND	40.2	ND	21.4
2-Hydroxy-ibuprofen	ND	699	ND	849	ND	475	ND	136	339	83.4	171	151	ND	161	437	202
Ibuprofen	432	131	ND	159	433	89.1	29.2	25.4	590	15.6	266	28.3	ND	30.1	642	19.3
Naproxen	665	26.2	2360	31.9	133	17.8	10800	44.4	76.2	7.22	50.7	5.66	9890	6.2	177	16.7
Triclocarban	2450	26.2	2120	31.9	20700	46.6	529	5.08	16.5	3.13	10000	8.04	784	6.03	16.8	3.59
Triclosan	27600	524	ND	637	38600	356	645	102	121	62.5	46400	113	918	121	136	64.2
Warfarin	ND	13.1	ND	15.9	ND	8.91	ND	2.54	ND	1.56	ND	2.83	ND	3.01	ND	1.6
Acetaminophen	ND	524	ND	637	ND	115	ND	110	ND	118	ND	113	ND	121	ND	76.5
Azithromycin	694	13.1	ND	15.9	315	2.87	ND	2.75	9.11	1.56	250	2.83	10.8	3.01	11.4	1.69
Caffeine	1240	131	ND	159	910	28.7	ND	27.5	529	16.4	1090	28.3	ND	30.1	332	16
Carbadox	ND	13.1	ND	15.9	ND	2.87	ND	2.75	ND	1.56	ND	2.83	ND	3.01	ND	1.6
Carbamazepine	291	13.1	45.9	15.9	12.1	2.87	40.2	2.75	727	2	53	2.83	31	3.01	395	2.77

Cefotaxime	ND	341	ND	142	ND	127	ND	62.5	ND	137	ND	47.7	ND	52.4	ND	114
Ciprofloxacin	13900	52.4	928	63.7	18100	17.4	NQ		58.1	38.2	11800	11.3	1190	151	93.7	84.4
Clarithromycin	353	13.1	ND	15.9	135	2.87	ND	2.75	5.34	1.56	146	2.83	ND	3.01	17.8	1.6
Clinafloxacin	ND	52.4	ND	80.5	ND	23.6	NQ		ND	52.5	ND	19	ND	135	ND	138
Cloxacillin	ND	26.2	ND	31.8	ND	3.78	ND	3.2	ND	5.18	ND	2.4	ND	2.41	ND	6.16
Dehydronifedipine	7.33	5.24	18.5	6.37	17.1	1.15	8.01	1.33	45.2	2.08	15.6	1.13	6.09	1.21	49.9	4.42
Diphenhydramine	1070	5.24	80	6.37	778	1.15	47.7	1.1	38	1.13	652	1.13	42.3	1.21	27.3	0.938
Diltiazem	47.6	2.62	ND	3.18	29.2	0.574	ND	0.551	11.7	0.782	81.9	0.566	ND	0.603	7.63	0.4
Digoxin	ND	149	ND	159	ND	28.7	ND	27.5	ND	15.6	ND	28.3	ND	30.1	ND	16
Digoxigenin	ND	92.3	ND	87	15.6	13.9	ND	45.5	ND	335	ND	30.5	ND	28.1	ND	330
Enrofloxacin	35.3	26.2	ND	31.8	ND	7.43	NQ		ND	5.65	ND	5.71	ND	27.5	ND	8.28
Erythromycin-H_2O	6.35	2.62	ND	3.18	2.75	0.574	ND	0.551	7.03	0.313	14.3	0.566	ND	0.603	7.6	0.321
Flumequine	ND	13.1	ND	15.9	ND	2.87	ND	2.75	ND	4.06	ND	2.83	ND	3.01	ND	7.22
Fluoxetine	39.1	13.1	ND	15.9	95	5.21	8.63	2.75	ND	1.56	ND	2.83	11.2	3.01	ND	1.6
Lincomycin	ND	26.2	ND	31.8	ND	13.4	ND	12.9	ND	10.2	ND	13.2	ND	14.1	ND	16.8
Lomefloxacin	ND	26.2	ND	31.8	ND	5.74	NQ		ND	3.13	ND	5.66	ND	9.73	ND	3.21
Miconazole	1040	20.8	109	15.9	ND	2.87	62.6	2.75	ND	6.66	531	3.4	54.2	3.01	ND	6.58
Norfloxacin	212	131	ND	159	119	28.7	NQ		ND	15.6	53.7	28.3	ND	30.1	ND	16
Norgestimate	ND	33	ND	31.8	ND	11.3	ND	7.48	ND	3.13	ND	8.72	ND	6.03	ND	3.21
Ofloxacin	402	131	ND	159	299	28.7	NQ		ND	15.6	185	28.3	288	30.1	ND	16
Ormetoprim	ND	5.24	ND	6.37	ND	1.15	ND	1.1	ND	0.625	ND	1.13	ND	1.21	ND	0.642
Oxacillin	ND	26.2	ND	31.8	ND	5.74	ND	5.51	8.84	3.29	ND	5.66	ND	6.03	ND	3.21
Oxolinic Acid	ND	5.24	ND	6.37	ND	2.31	ND	5.83	ND	8.46	ND	1.13	ND	5.57	ND	10.6
Penicillin G	ND	26.2	ND	31.8	ND	2.3	ND	2.2	ND	1.25	ND	2.27	ND	2.41	ND	1.28
Penicillin V	ND	26.2	ND	31.8	ND	5.74	ND	5.51	ND	7.41	ND	5.66	ND	6.03	ND	13.2
Roxithromycin	ND	2.62	ND	3.18	ND	2.28	ND	0.685	ND	0.313	ND	2.48	ND	1.28	ND	0.321
Sarafloxacin	ND	131	ND	182	ND	142	NQ		ND	15.6	ND	98.3	ND	311	ND	16
Sulfachloropyrida-zine	ND	13.1	ND	15.9	ND	2.87	ND	2.75	31.1	24.7	ND	2.83	ND	3.01	ND	1.6
Sulfadiazine	ND	13.1	ND	15.9	ND	2.87	ND	2.75	NQ		ND	2.83	ND	3.01	ND	1.6
Sulfadimethoxine	ND	2.62	ND	3.18	ND	1.27	ND	1.08	NQ		ND	2.03	ND	1.22	ND	0.321
Sulfamerazine	ND	5.24	ND	6.37	ND	1.6	ND	1.29	NQ		ND	4.7	ND	1.59	ND	5.05
Sulfamethazine	ND	7.25	ND	6.37	ND	1.15	ND	1.1	NQ		ND	5.96	ND	1.21	ND	17.9
Sulfamethizole	ND	5.24	ND	6.37	ND	1.39	ND	1.39	NQ		ND	8.42	ND	1.21	ND	3.67
Sulfamethoxazole	ND	5.24	ND	6.37	7.75	1.15	ND	1.1	NQ		ND	1.13	ND	1.21	ND	0.642

Sulfanilamide	ND	131	ND	159	ND	28.7	ND	27.5	NQ		ND	28.3	ND	30.1	ND	16
Sulfathiazole	ND	13.1	ND	15.9	ND	2.87	ND	2.75	NQ		ND	4.56	ND	3.01	ND	4.58
Thiabendazole	29.7	13.1	ND	15.9	62.7	2.87	ND	2.75	7.66	1.56	65.2	2.83	ND	3.01	4.25	1.64
Trimethoprim	ND	13.1	ND	15.9	78.4	8.05	ND	11.7	ND	25.9	63.9	2.83	ND	3.01	ND	70.6
Tylosin	ND	52.4	ND	63.7	ND	80.4	ND	82.3	ND	20.8	ND	11.3	ND	49.8	ND	21.4
Virginiamycin	ND	67.8	ND	65.3	ND	57.4	ND	55.1	ND	81.9	ND	18.9	ND	60.3	ND	64
1,7-Dimethylxanthine	ND	1310	ND	1590	645	287	ND	275	508	156	ND	283	ND	301	591	160
% Moisture	60.5		52.9		63.9		58.7		100		64.7		55.2		100	

ND = not detected NQ = not quantified

Detected concentration

Table A-14. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Gatineau Valley, QC, Samples

Item	First Round Sampling						Second Round Sampling					
	Dewatered Cake (Dry Mud)		Compost		Leachate Composting Pad		Dewatered Cake (Dry Mud)		Compost		Leachate Composting Pad	
Lab Work Order #	L13206-27		L13206-26		L13286-8		L13206-34		L13672-14		L13286-11	
Sampled Date	29-Sep-09		29-Sep-09		29-Sep-09		13-Oct-09		27-Oct-09		13-Oct-09	
Sample Received Date	30-Sep-09		30-Sep-09		30-Sep-09		14-Aug-09		28-Oct-09		14-Oct-09	
Result Report Date	17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)
Bisphenol A	120	80	<LOQ	80	270	50	180	80	70	80	130	50
Octylphenol	<LOQ	20	ND	20	<LOQ	10	<LOQ	20	<LOQ	20	30	10
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	90	<LOQ	140	<LOQ	140	<LOQ	90
DPMI	30	40	40	40	30	20	<LOQ	40	<LOQ	40	ND	20
ADBI	90	20	<LOQ	20	<LOQ	10	ND	20	ND	20	<LOQ	10
AHDI	80	30	40	30	ND	10	<LOQ	30	<LOQ	30	ND	10
HHCB	10520	90	3160	90	80	30	2530	90	1270	90	70	30
AHTN	3700	70	1050	70	250	40	570	70	800	70	230	40
ATII	340	60	120	60	10	10	40	60	120	60	20	10
M MOSKENE	ND	50	ND	50	ND	90	ND	50	ND	50	ND	90
M TIBETENE	ND	80	ND	80	ND	50	ND	80	ND	80	ND	50
M KETONE	110	120	<LOQ	120	ND	90	0.04	120	<LOQ	120	<LOQ	90
M AMBRETTE	ND	140	ND	140	ND	20	ND	140	ND	140	ND	20
M XYLENE	<LOQ	70	<LOQ	70	ND	20	<LOQ	70	<LOQ	70	40	20
Total Musks	14910		4440		340		3170		2230		360	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-15. Concentrations and Detection Limits of Pharmaceutical Compounds in Saguenay, QC, Samples

Item	First Round Sampling				Second Round Sampling						Third Round Sampling					
	Cake		Filtrate		Filter Feed Sludge		Cake		Filtrate		Filter Feed Sludge		Cake		Filtrate	
Lab Work Order #	L13037-1		L13036-1		L13129-1		L13129-2		L13128-1		L13435-1		L13435-2		L13439-1	
Sampled Date	8-Jul-09		8-Jul-09		22-Jul-09		22-Jul-09		22-Jul-09		27-Aug-09		27-Aug-09		27-Aug-09	
Sample Received Date	9-Jul-09		9-Jul-09		23-Jul-09		23-Jul-09		23-Jul-09		28-Aug-09		28-Aug-09		28-Aug-09	
Result Report Date (AN)	12-Aug-09		14-Aug-09		12-Aug-09		12-Aug-09		14-Aug-09		3-Oct-09		3-Oct-09		15-Oct-09	
Result Report Date (AP)	20-Aug-09		18-Aug-09		20-Aug-09		20-Aug-09		18-Aug-09		6-Oct-09		6-Oct-09		15-Oct-09	
Parameters	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/L)	Detect-ion Limit (ng/L)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/L)	Detect-ion Limit (ng/L)	Conc. (ng/g)	Detect-ion Limit (ng/L)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/L)	Detect-ion Limit (ng/L)
Furosemide	ND	386	ND	360	ND	192	ND	402	ND	113	139	109	165	164	402	140
Gemfibrozil	ND	14.5	5.95	1.72	ND	7.18	ND	15.1	4.49	1.93	ND	4.19	ND	6.32	5.85	1.5
Glipizide	ND	57.9	ND	6.89	ND	28.7	ND	60.3	ND	7.71	ND	16.3	ND	24.6	ND	5.99
Glyburide	ND	29	12.5	3.44	ND	14.4	ND	30.2	13.8	3.86	ND	8.16	ND	12.3	4.77	2.99
Hydrochlorothiazide	ND	193	677	23	ND	95.8	ND	201	461	25.7	ND	54.4	ND	82.1	223	29.8
2-Hydroxy-ibuprofen	ND	772	ND	151	ND	383	ND	805	147	103	ND	529	ND	329	ND	153
Ibuprofen	ND	145	204	17.2	ND	71.8	ND	151	190	19.3	47.8	40.8	ND	61.6	130	15
Naproxen	84.5	29	121	3.44	21.2	14.4	83.8	30.2	132	3.86	15.6	8.16	18.5	12.3	145	3.54
Triclocarban	1580	29	8.41	3.44	1830	14.4	1660	30.2	11.1	3.86	1920	8.16	2030	12.3	8.9	2.99
Triclosan	923	579	ND	68.9	963	287	1310	603	ND	77.1	2740	163	2820	246	84	59.9
Warfarin	ND	14.5	ND	1.72	ND	7.18	ND	15.1	ND	1.93	ND	4.08	ND	6.16	1.59	1.5
Acetaminophen	ND	577	ND	68.9	ND	287	ND	604	ND	77.1	ND	163	ND	246	ND	59.9
Azithromycin	390	14.4	235	1.72	385	7.18	262	15.1	162	1.93	282	4.08	185	6.15	70.3	1.5
Caffeine	387	144	ND	17.2	83	71.8	194	162	ND	19.3	ND	40.8	ND	61.5	ND	15
Carbadox	ND	14.4	ND	3.19	ND	7.18	ND	15.1	ND	1.93	ND	4.08	ND	6.15	ND	1.5
Carbamazepine	252	14.4	595	1.72	67.1	7.18	51.6	15.1	345	1.93	33.8	4.08	34.6	6.15	338	1.5
Cefotaxime	ND	164	ND	6.89	ND	266	ND	183	ND	7.71	ND	24.4	ND	24.6	ND	27.6
Ciprofloxacin	7150	57.7	ND	45.4	8670	28.7	6440	60.4	83.1	16	7000	16.3	4840	24.6	25.3	12.3
Clarithromycin	72.2	14.4	70.1	1.72	57.1	7.18	69.1	15.1	27.4	1.93	43	4.08	47.3	6.15	23	1.5
Clinafloxacin	ND	57.7	ND	65.6	ND	28.7	ND	60.4	ND	44.6	ND	24.2	ND	24.6	ND	31.7
Cloxacillin	ND	28.8	ND	4.79	ND	14.4	ND	30.2	ND	3.86	ND	8.15	ND	12.3	ND	2.99
Dehydronifedipine	ND	5.77	3.66	0.689	ND	2.87	ND	6.04	2.93	0.771	ND	1.63	ND	2.46	2.22	1.12
Diphenhydramine	349	5.77	150	0.689	443	2.87	420	6.04	88.1	0.771	487	1.63	607	2.46	72.6	0.599

Diltiazem	94.3	2.88	48.6	0.344	51.7	1.44	47	3.02	17.5	0.386	30.1	0.815	69.3	1.23	15.7	0.299
Digoxin	ND	144	ND	31.2	ND	89.4	ND	151	ND	21.2	ND	40.8	ND	61.5	ND	15
Digoxigenin	ND	64.5	ND	117	ND	122	ND	94.2	ND	86	ND	41.6	ND	32.2	ND	69.4
Enrofloxacin	ND	28.8	ND	8.47	27.1	14.4	33	30.2	ND	8.37	10.9	8.15	13.5	12.3	ND	5.94
Erythromycin-H_2O	ND	2.88	3.75	0.344	2.08	1.44	ND	3.02	5.31	0.386	5.66	0.815	5.69	1.23	7.18	0.299
Flumequine	ND	14.4	ND	1.72	ND	7.18	ND	15.1	ND	1.93	ND	4.08	ND	6.15	ND	2.1
Fluoxetine	48.3	14.4	4.24	1.72	28	7.18	36.5	16.4	6.89	2.11	21.8	4.08	20.4	6.15	ND	1.5
Lincomycin	ND	28.8	ND	3.44	ND	14.4	ND	30.2	ND	4.28	ND	19	ND	28.6	ND	11.9
Lomefloxacin	ND	28.8	ND	7.1	ND	14.4	ND	30.2	ND	9.53	ND	8.15	ND	12.3	ND	2.99
Miconazole	401	14.4	ND	1.72	508	7.18	495	15.1	ND	1.93	403	4.08	473	6.15	ND	1.5
Norfloxacin	586	144	206	36.5	642	71.8	534	151	ND	55.6	480	40.8	305	61.5	338	55.9
Norgestimate	ND	28.8	ND	6.42	ND	14.9	ND	31.7	ND	4.7	ND	8.15	ND	12.3	ND	3.72
Ofloxacin	1120	144	ND	17.2	1060	71.8	915	151	ND	19.3	794	40.8	483	61.5	ND	15
Ormetoprim	ND	5.77	ND	0.689	ND	2.87	ND	6.04	ND	0.771	ND	1.63	ND	2.46	ND	0.599
Oxacillin	ND	28.8	ND	3.44	ND	14.4	ND	30.2	ND	3.86	ND	8.15	ND	12.3	ND	2.99
Oxolinic Acid	ND	5.77	ND	0.766	ND	2.91	ND	6.04	ND	0.863	ND	1.78	ND	2.71	ND	0.653
Penicillin G	ND	28.8	6.85	3.44	ND	14.4	ND	30.2	5.76	4.28	ND	8.15	ND	12.3	15.2	6.44
Penicillin V	ND	28.8	ND	3.55	ND	14.4	ND	30.2	ND	5.9	ND	8.15	ND	12.3	ND	2.99
Roxithromycin	ND	2.88	ND	0.652	ND	1.7	ND	3.02	ND	0.479	ND	0.834	ND	1.23	ND	0.299
Sarafloxacin	ND	144	ND	122	ND	73	ND	313	ND	72.9	ND	125	ND	102	ND	15
Sulfachloropyridazine	ND	14.4	ND	4.42	ND	7.18	ND	15.1	ND	2.56	ND	4.08	ND	6.15	ND	2.13
Sulfadiazine	ND	14.4	ND	1.83	ND	7.18	ND	15.1	ND	1.93	ND	4.08	ND	6.15	ND	1.5
Sulfadimethoxine	ND	2.88	ND	0.824	ND	1.44	ND	3.02	ND	0.841	ND	1.96	ND	1.23	ND	0.591
Sulfamerazine	ND	5.77	ND	2.36	ND	2.87	ND	6.04	ND	0.962	ND	1.63	ND	2.46	ND	0.963
Sulfamethazine	ND	5.77	ND	2.3	ND	2.87	ND	7.31	ND	3.08	ND	1.63	ND	2.46	ND	1.24
Sulfamethizole	ND	5.77	ND	1.61	ND	2.87	ND	6.04	ND	1.33	ND	1.63	ND	2.46	ND	0.83
Sulfamethoxazole	15.8	5.77	67.8	1.65	18.1	2.92	15.9	6.04	22.6	1.69	10.8	1.63	8.25	2.46	8.47	0.599
Sulfanilamide	ND	144	ND	26.6	ND	71.8	ND	151	ND	19.3	ND	40.8	ND	61.5	ND	15
Sulfathiazole	ND	14.4	ND	2.27	ND	7.18	ND	15.1	ND	1.93	ND	4.08	ND	6.15	ND	1.5
Thiabendazole	93.9	14.4	74.3	1.72	110	7.18	116	15.1	58.2	1.93	65.7	4.08	62.7	6.15	16	1.5
Trimethoprim	24.3	14.4	31.3	8.36	46.7	10.1	29.1	15.1	27.5	11.2	26.9	4.08	29.6	6.15	12.8	6.67
Tylosin	ND	57.7	ND	23	ND	28.7	ND	60.4	ND	25.7	ND	54.3	ND	82	ND	5.99
Virginiamycin	ND	79.6	ND	3.44	ND	73.1	ND	103	ND	3.86	ND	60	ND	83.4	ND	16.4
1,7-Dimethylxanthine	ND	1440	ND	172	ND	718	ND	1510	ND	193	ND	408	ND	615	ND	150
% Moisture	87.4		99.96		95.9		88		100		91.9		86.3		99.98	

ND = not detected

Detected concentration (on dry weight basis)

Table A-16. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Saguenay, QC, Samples

Item	First Round Sampling						Second Round Sampling					
	Filter Feed Sludge		Cake		Filtrate		Filter Feed Sludge		Cake		Filtrate	
Lab Work Order #	L13129-1		L13129-2		L13128-1		L13206-17		L13206-18		L13286-5	
Sampled Date	22-Jul-10		22-Jul-10		22-Jul-09		27-Aug-09		27-Aug-09		27-Aug-09	
Sample Received Date	23-Jul-10		23-Jul-10		23-Jul-09		28-Aug-09		28-Aug-09		28-Aug-09	
Result Report Date	17-Mar-10		17-Mar-10		28-Jan-10		28-Jan-10		28-Jan-10		17-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)
Bisphenol A	ND	80	<LOQ	80	<LOQ	50	480	80	610	80	1080	50
Octylphenol	ND	20	ND	20	30	10	ND	20	ND	20	70	10
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	90	<LOQ	140	<LOQ	140	<LOQ	90
DPMI	50	40	70	40	ND	20	410	40	130	40	30	20
ADBI	<LOQ	20	<LOQ	20	ND	10	ND	20	ND	20	ND	10
AHDI	90	30	50	30	ND	10	110	30	160	30	ND	10
HHCB	4210	90	1520	90	890	30	3050	90	5060	90	290	30
AHTN	1200	70	1130	70	510	40	2500	70	4240	70	120	40
ATII	50	60	90	60	160	10	310	60	470	60	30	10
M MOSKENE	ND	50	ND	50	ND	90	ND	50	ND	50	ND	90
M TIBETENE	ND	80	ND	80	ND	50	ND	80	ND	80	ND	50
M KETONE	<LOQ	120	<LOQ	120	ND	90	ND	120	ND	120	<LOQ	90
M AMBRETTE	ND	140	ND	140	ND	20	ND	140	ND	140	ND	20
M XYLENE	<LOQ	70	<LOQ	70	ND	20	<LOQ	70	ND	70	<LOQ	20
Total Musks	5650		2930		1600		6390		10050		510	
% Moisture	95.9		88								99.98	
% TSS	4.1		12		0		8.1		100		0.02	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-17. Concentrations and Detection Limits of Pharmaceutical Compounds in Moncton, NB, Samples

Item	First Sampling Round				Second-Round Sampling				Third-Round Sampling			
CCME Sample ID #	Lime Stabilised Biosolids		Compost		Lime Stabilised Biosolids		Compost		Lime Stabilised Biosolids		Compost	
Lab Work Order #	L13207-1		L13207-2		L13283-1		L13283-2		L13380-3		L13380-4	
Sampled Date	30-Jul-09		29-Jul-09		10-Aug-09		10-Aug-09		24-Aug-09		24-Aug-09	
Sample Received Date	31-Jul-09		31-Jul-09		12-Aug-09		12-Aug-09		25-Aug-09		25-Aug-09	
Result Report Date (AN)	31-Aug-09		31-Aug-09		18-Sep-09		18-Sep-09		3-Oct-09		3-Oct-09	
Result Report Date (AP)	4-Sep-09		4-Sep-09		16-Sep-09		16-Sep-09		6-Oct-09		6-Oct-09	
Parameters	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**	**Conc. (ng/g)**	**Detect'n Limit (ng/g)**
Furosemide	ND	165	ND	158	ND	159	ND	135	ND	143	575	426
Gemfibrozil	17.5	6.33	ND	5.92	ND	5.97	ND	5.07	ND	5.51	ND	5.89
Glipizide	ND	24.7	ND	23.7	ND	23.9	ND	20.3	ND	21.5	ND	23
Glyburide	ND	12.3	ND	11.8	ND	11.9	ND	10.1	ND	10.7	ND	11.5
Hydrochlorothiazide	103	82.3	ND	78.9	ND	79.6	ND	67.7	ND	71.6	ND	76.6
2-Hydroxy-ibuprofen	ND	329	ND	316	ND	318	1050	271	ND	286	ND	306
Ibuprofen	180	61.7	ND	59.2	105	59.7	ND	50.7	147	53.7	ND	57.4
Naproxen	106	12.3	3830	60.7	48.2	11.9	9640	10.1	78.2	10.7	2810	11.5
Triclocarban	3070	12.3	166	11.8	1710	15.4	64.4	10.1	1750	10.7	146	11.5
Triclosan	7300	247	603	237	5910	239	960	203	7020	215	634	230
Warfarin	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.37	ND	5.74
Acetaminophen	ND	247	ND	237	ND	239	ND	203	222	215	ND	230
Azithromycin	216	6.17	ND	5.92	141	5.97	ND	5.07	147	5.38	ND	5.75
Caffeine	1110	61.7	ND	59.2	1090	59.7	ND	50.7	1080	53.8	ND	57.5
Carbadox	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.38	ND	5.75
Carbamazepine	142	6.17	15.5	5.92	69.4	5.97	5.27	5.07	81.2	5.38	18.8	5.75
Cefotaxime	ND	78.9	ND	36.3	ND	25.9	ND	21.5	ND	27.9	ND	23
Ciprofloxacin	3770	24.7	NQ		3060	23.9	433	185	4190	21.5	325	113
Clarithromycin	54.1	6.17	ND	5.92	165	5.97	ND	5.07	44.3	5.38	ND	5.75

Clinafloxacin	ND	24.7	NQ		ND	26.4	ND	307	ND	24.8	ND	97.9
Cloxacillin	ND	12.3	ND	11.8	ND	11.9	ND	10.1	ND	10.8	ND	11.5
Dehydronifedipine	4.93	2.47	ND	2.37	2.85	2.39	ND	2.03	2.98	2.15	ND	2.3
Diphenhydramine	1090	2.47	13.5	2.37	617	2.39	6.58	2.03	1270	2.15	57.7	2.3
Diltiazem	121	1.23	ND	1.18	58.1	1.19	ND	1.01	140	1.08	ND	1.15
Digoxin	ND	61.7	ND	59.2	ND	59.7	ND	50.7	ND	53.8	ND	57.5
Digoxigenin	ND	26.7	ND	71.6	ND	23.9	ND	20.3	ND	34.7	ND	36.9
Enrofloxacin	14.2	12.3	NQ		14.7	11.9	ND	30.7	15.4	10.8	ND	24.8
Erythromycin-H2O	15.5	1.23	ND	1.18	9.69	1.19	ND	1.01	10.5	1.08	ND	1.15
Flumequine	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.38	ND	5.75
Fluoxetine	10.3	6.17	ND	5.92	18.7	5.97	ND	5.07	62.7	5.38	ND	5.75
Lincomycin	ND	25.1	ND	17.3	ND	11.9	ND	10.1	ND	25.1	ND	26.8
Lomefloxacin	ND	12.3	NQ		ND	11.9	ND	10.1	ND	10.8	ND	11.5
Miconazole	312	6.17	26.4	5.92	345	5.97	21.8	5.07	212	5.38	31.1	5.75
Norfloxacin	793	61.7	NQ		877	59.7	ND	50.7	793	53.8	ND	268
Norgestimate	ND	12.5	ND	11.8	ND	11.9	ND	10.1	ND	10.9	ND	11.5
Ofloxacin	392	61.7	NQ		165	59.7	60.9	50.7	148	53.8	183	57.5
Ormetoprim	ND	2.47	ND	2.37	ND	2.39	ND	2.03	ND	2.15	ND	2.3
Oxacillin	ND	12.3	ND	11.8	ND	11.9	ND	10.1	ND	10.8	ND	11.5
Oxolinic Acid	ND	2.47	ND	2.37	ND	2.39	ND	2.03	ND	2.87	ND	3.82
Penicillin G	ND	12.3	ND	11.8	ND	39.8	ND	33.8	ND	10.8	ND	11.5
Penicillin V	ND	12.3	ND	11.8	ND	11.9	ND	10.1	ND	10.8	ND	11.5
Roxithromycin	ND	1.23	ND	1.53	ND	1.94	ND	1.54	ND	1.15	ND	1.15
Sarafloxacin	ND	89	NQ		ND	126	ND	1080	ND	89	ND	417
Sulfachloropyridazine	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.38	ND	5.75
Sulfadiazine	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.38	ND	5.75
Sulfadimethoxine	ND	2.05	ND	1.18	ND	1.19	ND	1.01	ND	1.08	ND	1.15
Sulfamerazine	ND	2.47	ND	3.04	ND	2.39	ND	2.03	ND	2.24	ND	2.3
Sulfamethazine	ND	2.47	ND	3.92	ND	2.78	ND	3.06	ND	2.52	ND	2.93
Sulfamethizole	ND	2.47	ND	2.37	ND	2.39	ND	2.03	ND	2.47	ND	2.3
Sulfamethoxazole	3.92	2.47	ND	2.51	ND	2.9	ND	2.25	ND	2.15	ND	2.3
Sulfanilamide	ND	61.7	ND	59.2	ND	59.7	ND	50.7	ND	53.8	ND	57.5
Sulfathiazole	ND	6.17	ND	5.92	ND	5.97	ND	5.07	ND	5.38	ND	5.75
Thiabendazole	12.5	6.17	ND	5.92	44.8	5.97	ND	5.07	10.1	5.38	ND	5.75
Trimethoprim	31.6	6.17	ND	8.13	36.5	5.97	ND	5.07	33.1	7.6	ND	5.75

Tylosin	ND	82.3	ND	78.9	ND	23.9	ND	20.3	ND	71.7	ND	76.6
Virginiamycin	ND	157	ND	59.3	ND	142	ND	46.4	ND	186	ND	66.1
1,7-Dimethylxanthine	ND	617	ND	592	ND	597	ND	507	ND	538	ND	575
% Moisture	65.3		57.1		64.6		53.1		61.2		59.2	

ND = not detected NQ = not quantified

Detected concentration (on dry weight basis)

Table A-18. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Moncton, NB, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Lime Stabilized Sludge		Compost		Lime Stabilized Sludge		Compost	
Lab Work Order #	L132063-3		L132063-4		L13206-15		L13206-16	
Sampled Date	10-Aug-09		10-Aug-09		24-Aug-09		24-Aug-09	
Sample Received Date	12-Aug-09		12-Aug-09		25-Aug-09		25-Aug-09	
Result Report Date	28-Jan-10		28-Jan-10		17-Mar-10		28-Jan-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	770	80	180	80	1160	80	70	80
Octylphenol	20	20	40	20	ND	20	ND	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	NA	140
DPMI	60	40	40	40	100	40	90	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	300	30	70	30	930	30	40	30
HHCB	3080	90	530	90	4800	90	900	90
AHTN	1250	70	960	70	2540	70	230	70
ATII	370	60	90	60	580	60	<LOQ	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	ND	120	ND	120	ND	120	ND	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	<LOQ	70	<LOQ	70	<LOQ	70	ND	70
Total Musks	5060		1700		8960		1260	
% Moisture	64.6		53.1		61.2		59.2	
% TSS	35.4		46.9		37.9		40.8	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-19. Concentrations and Detection Limits of Pharmaceutical Compounds in N-Viro Halifax, NS, Samples

CCME Sample ID #	Sludge Feed after Admixture		N-viro after Final Curing		Sludge Feed After Admixture		N-viro after Final Curing		Sludge Feed After Admixture		N-viro after Final Curing	
Lab Work Order #	L13130-2		L13130-1		L13503-1		L13503-2		L13672-6		L13672-7	
Sampled Date	22-Jul-09		22-Jul-09		2-Sep-09		2-Sep-09		7-Oct-09		7-Oct-09	
Sample Received Date	23-Jul-09		23-Jul-09		9-Sep-09		9-Sep-09		9-Oct-09		9-Oct-09	
Result Report Date (AN)	31-Aug-09		31-Aug-09		23-Oct-09		23-Oct-09		25-Nov-09		25-Nov-09	
Result Report Date (AP)	4-Sep-09		4-Sep-09		22-Oct-09		22-Oct-09		4-Dec-09		4-Dec-09	
Parameters	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)	Conc. (ng/g)	Detect-ion Limit (ng/g)
Furosemide	ND	152	ND	153	137	93.7	259	164	ND	89.4	ND	354
Gemfibrozil	12.4	5.83	21.9	5.89	12.2	2.94	13.8	3.08	10.1	2.71	9.86	3.04
Glipizide	ND	22.7	ND	23	ND	11.8	ND	12.3	ND	10.8	ND	12.1
Glyburide	ND	11.4	ND	11.5	ND	5.89	ND	6.15	12	5.42	ND	6.07
Hydrochlorothiazide	166	88.7	91.4	78.6	ND	39.3	ND	41	ND	36.2	ND	40.5
2-Hydroxy-ibuprofen	ND	303	ND	306	228	157	ND	164	172	145	189	162
Ibuprofen	623	56.8	528	57.4	315	29.4	369	30.8	319	27.1	522	30.4
Naproxen	169	11.4	212	11.5	169	5.89	178	8.97	155	5.42	126	6.07
Triclocarban	9200	25.7	1260	11.5	1540	5.89	1590	6.15	3780	5.42	1790	10.7
Triclosan	11500	227	6120	230	5730	118	6520	123	7700	108	4780	682
Warfarin	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.71	ND	3.04
Acetaminophen	ND	543	ND	230	ND	118	ND	123	ND	109	ND	122
Azithromycin	469	7.78	36.8	5.74	223	3.13	157	3.08	349	2.72	5.27	3.05
Caffeine	1120	56.8	386	57.4	355	29.4	240	30.8	334	27.2	143	30.5
Carbadox	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.72	ND	3.05
Carbamazepine	349	5.68	100	5.74	137	2.94	79.4	3.08	114	2.72	40.7	3.05
Cefotaxime	ND	58.4	ND	98.9	ND	55.3	ND	55.6	ND	129	ND	161

Ciprofloxacin	1840	22.7	605	23	724	19.7	560	12.3	1170	10.9	587	35.6
Clarithromycin	50.8	5.68	ND	5.74	19.4	2.94	11.5	3.08	31.1	2.72	ND	3.05
Clinafloxacin	ND	22.7	ND	23	17	11.8	ND	20.9	ND	19.2	ND	67
Cloxacillin	ND	11.4	ND	11.5	ND	2.73	ND	3.29	ND	3.61	ND	5.12
Dehydronifedipine	3.01	2.27	ND	2.3	2.4	1.62	2.79	1.23	1.29	1.09	1.93	1.22
Diphenhydramine	900	2.27	87.4	2.3	298	1.18	216	1.23	656	1.09	140	1.22
Diltiazem	3.86	1.14	ND	1.15	0.66	0.589	ND	0.615	2.79	0.544	ND	0.61
Digoxin	ND	56.8	ND	57.4	ND	29.4	ND	30.8	ND	27.2	ND	30.5
Digoxigenin	ND	94.4	ND	46.8	ND	36.1	ND	53.6	ND	63.8	ND	69.4
Enrofloxacin	ND	11.4	ND	11.5	ND	6.23	ND	9.75	12.6	5.44	ND	24.8
Erythromycin-H2O	32.5	1.14	8.88	1.15	12.5	0.589	6.02	0.615	22	0.544	14.6	0.61
Flumequine	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.72	ND	3.05
Fluoxetine	48.3	5.68	8.79	5.74	23.3	2.94	9.67	3.08	23	3.28	ND	3.05
Lincomycin	ND	25.3	ND	24.6	ND	13.7	ND	14.3	ND	12.7	ND	14.2
Lomefloxacin	ND	11.4	ND	11.5	ND	5.89	ND	6.15	ND	5.44	ND	13.7
Miconazole	664	5.68	230	5.74	448	2.94	319	3.08	517	2.72	400	7.67
Norfloxacin	218	56.8	98.8	57.4	84.9	38.5	99.2	79	105	27.2	ND	30.5
Norgestimate	ND	15.8	ND	15.2	ND	16.6	ND	10.8	ND	6.09	ND	15.3
Ofloxacin	399	56.8	325	57.4	121	29.4	125	30.8	206	27.2	276	30.5
Ormetoprim	ND	2.27	ND	2.3	ND	1.18	ND	1.23	ND	1.09	ND	1.22
Oxacillin	ND	11.4	ND	11.5	ND	5.89	ND	6.15	ND	5.44	ND	6.1
Oxolinic Acid	ND	2.8	ND	2.9	ND	1.18	ND	1.23	ND	2.24	ND	1.45
Penicillin G	ND	11.4	ND	11.5	ND	2.36	ND	2.46	ND	2.17	ND	2.44
Penicillin V	ND	11.4	ND	11.5	ND	5.89	ND	6.15	ND	5.44	ND	7.78
Roxithromycin	ND	1.17	ND	1.79	ND	0.589	ND	0.615	ND	1.3	ND	1.3
Sarafloxacin	ND	133	ND	165	ND	112	ND	99.8	ND	170	ND	279
Sulfachloropyridazine	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.72	ND	3.05
Sulfadiazine	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.72	ND	3.05
Sulfadimethoxine	ND	1.41	ND	2.22	ND	0.589	ND	6.64	ND	0.844	ND	2.39
Sulfamerazine	ND	2.27	ND	2.33	ND	2.98	ND	1.23	ND	3.08	ND	2.03
Sulfamethazine	ND	3.07	ND	4.72	ND	3.73	ND	4.32	ND	1.09	ND	1.22
Sulfamethizole	ND	2.27	ND	3.97	ND	2.33	ND	2.2	ND	2.84	ND	3.67
Sulfamethoxazole	ND	2.27	ND	3.96	2.48	1.18	2.22	1.23	1.66	1.09	ND	1.22
Sulfanilamide	ND	56.8	ND	57.4	ND	29.4	ND	30.8	ND	27.2	49	30.5
Sulfathiazole	ND	5.68	ND	5.74	ND	2.94	ND	3.08	ND	2.72	ND	3.05

Thiabendazole	12.4	5.68	7.7	5.74	5.93	2.94	5.61	3.08	6.67	2.72	8.03	3.05
Trimethoprim	33.1	17.7	ND	11.6	7.89	3.71	17.2	14.1	ND	15.1	ND	20.4
Tylosin	ND	75.8	ND	76.5	ND	39.3	ND	41	ND	127	ND	154
Virginiamycin	309	209	ND	98	ND	108	ND	90.3	ND	54.4	409	342
1,7-Dimethylxanthine	ND	568	ND	574	306	294	378	308	727	272	ND	305
% Moisture	55.3		31.2		49.6		30.4		50.7		32.6	

ND = not detected

Detected concentration

Table A-20. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in N-Viro Halifax, NS, Samples

Item	First Round Sampling				Second Round Sampling			
CCME Sample ID #	Dryer Infeed		Final Product		Dryer Infeed		Final Product	
Lab Work Order #	L13206-24		L13206-25		L13206-30		L13206-31	
Sampled Date	2-Sep-09		2-Sep-09		7-Oct-09		7-Oct-09	
Sample Received Date	9-Sep-09		9-Sep-09		9-Oct-09		9-Oct-09	
Result Report Date	17-Mar-10		17-Mar-10		17-Mar-10		17-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)
Bisphenol A	110	80	770	80	290	80	810	80
Octylphenol	<LOQ	20	<LOQ	20	<LOQ	20	<LOQ	20
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	140	<LOQ	140
DPMI	50	40	50	40	60	40	ND	40
ADBI	ND	20	ND	20	ND	20	ND	20
AHDI	70	30	<LOQ	30	<LOQ	30	<LOQ	30
HHCB	3090	90	2880	90	4410	90	5350	90
AHTN	660	70	760	70	300	70	620	70
ATII	90	60	70	60	130	60	150	60
M MOSKENE	ND	50	ND	50	ND	50	ND	50
M TIBETENE	ND	80	ND	80	ND	80	ND	80
M KETONE	<LOQ	120	<LOQ	120	<LOQ	120	<LOQ	120
M AMBRETTE	ND	140	ND	140	ND	140	ND	140
M XYLENE	<LOQ	70	<LOQ	70	<LOQ	70	<LOQ	70
Total Musks	4050		3820		4940		5890	

ND = not detected LOQ = limit of quantitation

Detected concentration

Table A-21. Concentrations and Detection Limits of Pharmaceutical Compounds in Gander, NL, Samples

Item	First Round Sampling						Second Round Sampling						Third Round Sampling					
CCME Sample ID #	Thickened Primary Sludge		Cake Solids		Filtrate		Thickened Primary Sludge		Cake Solids		Filtrate		Thickened Primary Sludge		Cake Solids		Filtrate	
Lab Work Order #	L12974-1		L12974-2		L12973-1		L13084-2		L13084-1		L13085-1		L13283-10		L13283-9		L13285-4	
Sampled Date	30-Jun-09		30-Jun-09		30-Jun-09		16-Jul-09		16-Jul-09		16-Jul-09		13-Aug-09		16-Jul-09		13-Aug-09	
Sample Received Date	3-Jul-09		3-Jul-09		3-Jul-09		20-Jul-09		20-Jul-09		20-Jul-09		17-Aug-09		20-Jul-09		17-Aug-09	
Result Report Date (AN)	20-Jul-09		31-Jul-09		4-Aug-09		12-Aug-09		12-Aug-09		24-Aug-09		18-Sep-09		18-Sep-09		15-Oct-09	
Result Report Date (AP)	6-Aug-09		6-Aug-09		23-Aug-09		20-Aug-09		20-Aug-09		15-Sep-09		16-Sep-09		16-Sep-09		8-Oct-09	
Parameters	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/L)	Detect-ion Limit (ng/L)	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/L)	Detect-ion Limit (ng/L)	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/g TS)	Detect-ion Limit (ng/g TS)	Conc. (ng/L)	Detect-ion Limit (ng/L)
Furosemide	ND	1770	ND	342	ND	296	ND	318	3030	338	ND	156	ND	232	229	74.8	270	40.1
Gemfibrozil	ND	66.3	ND	13.2	ND	6.15	13.1	11.9	ND	12.7	ND	6.01	ND	8.72	ND	17.3	ND	5.85
Glipizide	ND	265	ND	51.3	ND	24	ND	47.7	ND	50.7	ND	23.4	ND	34.9	ND	23	ND	23.4
Glyburide	ND	133	ND	25.6	ND	12	ND	23.8	ND	25.4	ND	11.7	ND	17.4	16.3	11.5	ND	11.7
Hydrochlorothia-zide	ND	885	ND	171	410	79.9	ND	159	ND	169	435	78	ND	116	ND	230	474	77.9
2-Hydroxy-ibuprofen	ND	3540	ND	684	4660	320	ND	636	ND	677	4880	312	ND	465	ND	921	4230	312
Ibuprofen	1630	766	304	128	2320	59.9	459	119	319	127	2130	58.5	395	87.2	380	173	1670	58.5
Naproxen	405	133	55.8	25.6	695	27.2	178	23.8	111	25.4	690	11.7	79.7	17.4	98.1	34.6	608	27.9
Triclocarban	3110	133	1880	25.6	15.3	12	2670	23.8	2470	25.4	14.6	11.7	2280	17.4	2470	34.6	18.6	11.7
Triclosan	13300	2650	20300	513	241	240	11400	477	9560	507	ND	234	11700	349	9240	691	ND	234
Warfarin	ND	66.3	ND	12.8	ND	5.99	ND	11.9	ND	12.7	ND	5.85	ND	8.72	ND	5.76	ND	5.85

Acetaminophen	11300	2650	ND	127	19800	215	772	687	ND	508	14900	234	1170	349	ND	230	16600	332
Azithromycin	237	66.4	248	3.18	61.3	5.37	252	11.9	220	12.7	47.5	5.85	95.7	8.72	146	5.76	49.9	5.84
Caffeine	7910	911	1130	34.6	10200	53.7	2590	119	1170	176	12700	58.5	1990	87.2	1160	57.6	10100	58.4
Carbadox	ND	66.4	ND	3.18	ND	5.37	ND	11.9	ND	12.7	ND	5.85	ND	8.72	ND	5.76	ND	5.84
Carbamazepine	275	66.4	111	3.18	835	5.37	858	11.9	403	12.7	1300	5.85	122	8.72	214	5.76	1350	5.84
Cefotaxime	ND	2610	ND	143	ND	29	ND	129	ND	159	ND	48.2	ND	34.9	ND	23	ND	43.7
Ciprofloxacin	18100	265	13100	12.7	91.4	21.5	17200	47.7	19100	50.8	109	23.4	16000	34.9	16900	23	108	23.3
Clarithromycin	95.6	66.4	30	3.18	147	5.37	69.1	11.9	97.2	12.7	61.4	5.85	92.4	8.72	147	5.76	194	5.84
Clinafloxacin	ND	265	ND	31.1	ND	35.2	ND	47.7	ND	50.8	ND	65.3	ND	34.9	ND	24	ND	23.3
Cloxacillin	ND	133	ND	7.69	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	12.6
Dehydronifedi-pine	ND	26.5	7.09	1.27	55.1	2.49	7.66	4.77	8.42	5.08	82	3.27	ND	3.49	12.6	2.3	132	2.33
Diphenhydra-mine	499	33.8	186	2.81	109	2.15	222	4.77	251	5.08	145	2.34	171	3.49	205	2.3	201	2.33
Diltiazem	348	13.3	257	0.66	294	1.07	563	2.38	600	2.54	337	1.31	145	1.74	254	1.15	316	1.17
Digoxin	ND	664	ND	31.8	ND	53.7	ND	135	ND	156	ND	58.5	ND	87.2	ND	57.6	ND	58.4
Digoxigenin	ND	265	ND	12.7	ND	21.5	ND	65.7	ND	67.9	ND	83.3	ND	34.9	ND	23	ND	23.3
Enrofloxacin	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	18.9
Erythromycin-H2O	18.1	13.3	17.1	0.637	53.3	1.07	28.7	2.38	34.9	2.54	43.1	1.17	30.1	1.74	32.9	1.15	93.3	1.17
Flumequine	ND	66.4	ND	3.18	ND	5.37	ND	11.9	ND	12.7	ND	9.99	ND	8.72	ND	5.76	ND	5.84
Fluoxetine	109	68.5	51.9	16.3	ND	5.37	33	11.9	53	15.4	ND	5.85	36.1	8.72	41	5.76	9.29	5.84
Lincomycin	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	40.8
Lomefloxacin	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	11.7
Miconazole	712	66.4	207	3.18	ND	5.37	463	11.9	529	12.7	ND	5.85	341	8.72	441	5.76	ND	5.84
Norfloxacin	1640	664	3700	52.2	ND	53.7	2520	119	2120	127	463	58.5	2010	87.2	2390	57.6	294	58.4
Norgestimate	ND	135	ND	6.37	ND	11.9	ND	24.5	ND	30.9	ND	11.7	ND	17.4	ND	11.5	ND	12.6
Ofloxacin	5140	664	2260	31.8	ND	53.7	2080	119	2710	127	ND	58.5	2590	87.2	2670	57.6	ND	58.4
Ormetoprim	ND	26.5	ND	1.27	ND	2.15	ND	4.77	ND	5.08	ND	2.34	ND	3.49	ND	2.3	ND	2.33
Oxacillin	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	11.7
Oxolinic Acid	ND	35.1	ND	1.27	ND	2.15	ND	4.77	ND	5.08	ND	2.34	ND	3.49	ND	2.3	ND	2.33
Penicillin G	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	58.1	ND	38.4	ND	11.7
Penicillin V	ND	133	ND	6.37	ND	10.7	ND	23.8	ND	25.4	ND	11.7	ND	17.4	ND	11.5	ND	11.7

Roxithromycin	ND	13.3	0.755	0.637	ND	1.07	ND	2.38	ND	2.54	ND	1.17	ND	1.74	ND	1.26	ND	1.17
Sarafloxacin	ND	664	ND	31.8	ND	53.7	ND	119	ND	127	ND	58.5	ND	90.3	ND	103	ND	58.4
Sulfachloropyrida-zine	ND	66.4	ND	3.18	ND	5.37	ND	11.9	ND	12.7	ND	5.85	ND	8.72	ND	5.76	ND	5.84
Sulfadiazine	ND	66.4	ND	3.18	ND	5.37	ND	11.9	ND	12.7	ND	5.85	ND	8.72	ND	5.76	ND	5.84
Sulfadimethoxine	ND	13.3	ND	0.637	ND	1.07	ND	2.38	ND	2.54	ND	3.53	ND	1.74	ND	1.15	ND	2.24
Sulfamerazine	ND	26.5	ND	1.27	ND	2.15	ND	4.77	ND	5.08	21.4	2.34	ND	3.49	ND	2.3	4.83	2.81
Sulfamethazine	ND	26.5	ND	1.27	ND	2.15	ND	4.77	ND	5.08	8.3	3.72	ND	3.49	ND	3.08	ND	2.33
Sulfamethizole	ND	26.5	ND	1.27	ND	2.15	ND	4.77	ND	5.08	ND	2.34	ND	3.49	ND	2.3	ND	2.33
Sulfamethoxazole	156	26.5	5.17	1.27	96.6	2.15	17.6	4.77	7.65	5.08	61.6	2.34	4.32	3.49	3.46	2.76	80.5	2.33
Sulfanilamide	ND	664	ND	31.8	ND	53.7	ND	119	ND	127	ND	58.5	ND	87.2	ND	57.6	ND	58.4
Sulfathiazole	ND	66.4	ND	3.18	ND	5.37	ND	11.9	ND	12.7	ND	5.85	ND	8.72	ND	5.76	ND	5.84
Thiabendazole	95.6	66.4	55.8	3.18	30.1	5.37	44.1	11.9	50.9	12.7	16.9	5.85	34.6	8.72	38.5	5.76	15.9	5.84
Trimethoprim	92.9	66.4	58	3.18	150	6.95	110	11.9	119	12.7	132	6.55	84.6	8.72	76.7	5.76	149	8.37
Tylosin	ND	265	ND	12.7	ND	21.5	ND	47.7	ND	50.8	ND	78	ND	34.9	ND	23	ND	77.8
Virginiamycin	ND	346	ND	39.8	ND	156	ND	75.2	ND	68.1	ND	17.5	ND	70.2	ND	89.6	ND	584
1,7-Dimethylxanthine	ND	6640	ND	318	2130	537	ND	1190	ND	1270	4470	585	ND	872	ND	576	2530	584
% Moisture	99.5		91.1		99.92		97.6		83.1		100		96.7		85.6		99.98	

ND = not detected

Detected concentration (on dry weight basis)

Table A-22. Concentrations and Detection Limits of Fragrance and Alkylphenol Compounds in Gander, NL, Samples

Item	First Round Sampling						Second Round Sampling					
	Thickened Primary Sludge		Cake Solids		Filtrate		Thickened Primary Sludge		Cake Solids		Filtrate	
Lab Work Order #	L13084-2		L13084-1		L13085-1		L13206-12		L13206-11		L13286-4	
Sampled Date	16-Jul-09		16-Jul-09		16-Jul-09		13-Aug-09		13-Aug-09		13-Aug-09	
Sample Received Date	20-Jul-09		20-Jul-09		20-Jul-09		17-Aug-09		17-Aug-09		17-Aug-09	
Result Report Date	13-Mar-10		13-Mar-10		28-Jan-10		28-Jan-10		28-Jan-10		13-Mar-10	
Parameters	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/g)	LOQ (ng/g)	Conc. (ng/L)	LOQ (ng/L)
Bisphenol A	130	80	ND	80	<LOQ	50	620	80	130	80	3650	50
Octylphenol	ND	20	ND	20	ND	10	ND	20	ND	20	<LOQ	10
Nonylphenol	<LOQ	140	<LOQ	140	<LOQ	90	<LOQ	140	NA	140	<LOQ	90
DPMI	60	40	50	40	ND	20	70	40	ND	40	ND	20
ADBI	ND	20	ND	20	ND	10	ND	20	ND	20	20	10
AHDI	<LOQ	30	<LOQ	30	ND	10	730	30	330	30	ND	10
HHCB	1040	90	3820	90	640	30	1940	90	1780	90	280	30
AHTN	370	70	630	70	270	40	1310	70	2050	70	240	40
ATII	130	60	170	60	150	10	310	60	340	60	80	10
M MOSKENE	ND	50	ND	50	ND	90	ND	50	ND	50	ND	90
M TIBETENE	ND	80	ND	80	ND	50	ND	80	ND	80	ND	50
M KETONE	<LOQ	120	<LOQ	120	ND	90	ND	120	ND	120	90	90
M AMBRETTE	ND	140	ND	140	ND	20	ND	140	ND	140	ND	20
M XYLENE	<LOQ	70	<LOQ	70	ND	20	<LOQ	70	ND	70	<LOQ	20
Total Musks	1650		4730		1050		4360		4500		700	
% Moisture	97.6		83.1		100		96.7		85.6		99.98	
% TSS	2.4		16.9		0		3.3		14.4		0.02	

ND = not detected LOQ = limit of quantitation

Detected concentration