Control of Toxic Chemicals in Puget Sound

Phase 3: Pharmaceuticals and Personal Care Products in Municipal Wastewater and Their Removal by Nutrient Treatment Technologies

DEPARTMENT OF ECOLOGY
State of Washington

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

PUGET SOUND PARTNERSHIP
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Contact Information

For more information contact:

Publications Coordinator
Environmental Assessment Program
P.O. Box 47600, Olympia, WA 98504-7600
Phone: (360) 407-6764

Washington State Department of Ecology – www.ecy.wa.gov

- Headquarters, Olympia (360) 407-6000
- Northwest Regional Office, Bellevue (425) 649-7000
- Southwest Regional Office, Olympia (360) 407-6300
- Central Regional Office, Yakima (509) 575-2490
- Eastern Regional Office, Spokane (509) 329-3400

This report was prepared by a licensed engineer and a licensed hydrogeologist. A signed and stamped copy of the report is available upon request.

Pharmaceuticals and Personal Care Products in Municipal Wastewater and their Removal by Nutrient Treatment Technologies

Prepared by:

Brandi Lubliner, Professional Engineer
and
Melanie Redding, Licensed Hydrogeologist
Washington State Department of Ecology
Environmental Assessment Program
P.O. Box 47600
Olympia, WA 98504-7710

and

David Ragsdale, Environmental Engineer
United States Environmental Protection Agency
Region 10
Office of Water and Watersheds

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Abstract

In August 2008, the Washington State Department of Ecology (Ecology) and the U.S. Environmental Protection Agency (EPA) conducted a one-day screening study to characterize pharmaceuticals and personal care products (PPCPs) at five municipal wastewater treatment plants (WWTPs) in the Pacific Northwest. Wastewater influent, secondary effluent, tertiary effluent, and biosolids were sampled.

Four of the five WWTPs discharge within the Puget Sound watershed. Two of the plants provide secondary treatment, and three employ advanced (tertiary) treatment for nitrogen and phosphorus removal. Two of the plants produce tertiary-treated reclaimed water.

Target analytes included 172 organic compounds (PPCPs, hormones, steroids, semi-volatile organics). In addition, nutrients and total suspended solids were sampled. Newly approved EPA methods were used to measure PPCPs, hormones, and steroids at low concentrations. Removal efficiencies were evaluated for each analyte at the five WWTPs.

In the study, PPCPs were found in all samples at concentrations comparable to those found in the literature. Secondary treatment alone achieved high removals for hormones and steroids. Approximately 21% of the 172 analytes were reduced to below reporting limits by conventional secondary treatment, whereas 53% were reduced to below reporting limits by at least one advanced nutrient-removal technology.

Roughly 20% of the 172 analytes (mainly polycyclic aromatic hydrocarbons) were found only in the biosolids and not the wastewater samples. Some analytes were clearly concentrating in the biosolids.

Three PPCPs (carbamazepine, fluoxetine, and thiabendazole) were relatively untreated by the surveyed WWTP technologies. These three PPCPs may serve well as human-influence tracer compounds in the environment.

Overall, this screening study indicates that (1) there are differences in PPCP removal between the WWTP processes and (2) advanced nutrient reduction and tertiary filtration may provide additional PPCP removal.

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Executive Summary

On August 19, 2008, five wastewater treatment plants (WWTPs) were sampled to characterize pharmaceuticals and personal care products (PPCPs) in the influent, effluent, and biosolids. These WWTPs had well-operated but different secondary and/or tertiary treatment processes.

Seven effluents were characterized at the five WWTPs. Of these seven, five employed nutrient removal beyond the standard secondary treatment process. Two of the three tertiary technologies were reclaimed water processes.

Target analytes included 172 organic compounds: 72 pharmaceuticals and personal care products, 27 hormones and steroids, and 73 semi-volatile organic compounds. For purposes of this report, the 172 organic compounds are collectively referred to as PPCPs, and do not include the seven nutrient or total suspended solids parameters.

The results of this sampling were compared to determine if removal of PPCPs differed between WWTPs that provide only secondary treatment and WWTPs that provide advanced treatment for removal of nutrients. The treatment processes employed at the five WWTPs are described in Table ES-1. Each WWTP is given a treatment identifier code that is used throughout the report.

Table ES-1. Wastewater treatment plant descriptions, processes, and treatment codes.

	Wastewater Treatment Plant	Treatment Code	Process of Treatment
1	LOTT, Budd Inlet WWTP Olympia, WA	EBNR*	Enhanced biological nitrogen removal (EBNR) incorporated into the secondary treatment process via a modified four-stage process.
	LOTT, Budd Inlet Reclaimed Water Plant Olympia, WA.	EBNR+F*	A portion of the secondary effluent from the EBNR process is treated by chemical addition and sand filtration.
2	LOTT, Martin Way Reclaimed Water Plant Lacey, WA	EBNR+MF*	Enhanced biological nitrogen removal with membrane filtration.
3	Pierce County, Chambers Creek WWTP Tacoma, WA	AS	Activated sludge.
4	Puyallup WWTP Puyallup, WA	AS+N*	Activated sludge with partial nitrification and denitrification.
5	City of Hayden WWTP Hayden, Idaho	AD	Secondary treatment by aeration ditch.
	Hayden Wastewater Research Facility Operated by Blue Water Inc. Hayden, Idaho	CA+F*	A portion of the Hayden WWTP secondary effluent receives tertiary treatment by chemical addition and tertiary two-stage sand filtration for phosphorus removal.

LOTT - Lacey, Olympia, Tumwater, and Thurston County Alliance.
*Five of seven effluents sampled provide some degree of nutrient removal.

Two recently developed analytical methods were used:

1. EPA Method 1694 for a specific list of pharmaceuticals and personal care products ($PPCPs^{1694}$).
2. EPA Method 1698 for hormone and steroid compounds.

These new methods performed adequately in this study.

PPCPs were routinely detected in municipal wastewater. Of the 172 analytes monitored in this study, 96 (56%) were detected in at least one sample. Every sample had detectable concentrations of multiple PPCPs. The concentrations of the majority of the PPCP compounds were reduced to varying degrees by the sampled wastewater technologies.

Approximately 21% of the analytes detected in influents were reduced to concentrations below the laboratory reporting limit by conventional secondary treatment. Approximately 32% more of the analytes were brought below the laboratory reporting limits by at least one of the advanced nutrient-removal technologies.

Some of the analytes removed from wastewater were found in the biosolids. Biosolids were found to have PPCPs in a wide range of concentrations. Roughly 20% of the 172 analytes were found only in the biosolids and not the wastewater samples. The fate of the compounds in biosolids is unknown.

Contaminant removals are categorized and presented by treatment technologies in Table ES-2.

Table ES-2. Categorical removal of contaminants in wastewater effluent by treatment type.

Category	$PPCPs^{1694}$	Hormones/ Steroids	Semi-volatile Organics
High = >80% of analytes had at least 80% reduction in concentration	EBNR+F * EBNR+MF	EBNR+F * EBNR+MF * CA+F * AS+N * EBNR AD AS	EBNR * EBNR+F * CA+F AD
Moderate = 60-80% of analytes had at least 80% reduction in concentration	CA+F	--	AS+N AS
Low = <60% of the analytes had at least 80% reduction in concentration	EBNR AS+N AS AD	--	EBNR+MF

* = The treatment technologies that produced a 1-log reduction (90%) for 80% of the detected influent analytes. See Table ES-1 for treatment code definitions.

The advanced treatment technologies studied included (1) enhanced biological nutrient removal for phosphorus and nitrogen, and (2) chemical addition with filtration for phosphorus removal. These technologies appeared to remove 31 more PPCP analytes from the wastewater, primarily by extended biological contact time, nutrient reduction, and/or tertiary filtration.

Three PPCPs (carbamazepine, fluoxetine, and thiabendazole) stood out as relatively untreated by these treatment technologies. These may be useful as effluent tracer compounds in the environment.

Results of this sampling study are consistent with findings of published studies which reported that additional WWTP nutrient removal provides better removal of PPCPs than is achieved by secondary treatment technologies alone.

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Introduction

Pharmaceuticals and personal care products (PPCPs) are used daily. In 2005, Washington State residents filled an average of 8.5 prescriptions per person (PH:ARM Pilot Team, 2007). Chemical residuals from these PPCPs enter the environment from municipal wastewater treatment plants (WWTPs)(Ternes, 1998; Ternes and Joss, 2004) or populated urban areas (Kolpin et al., 2004; Glassmeyer et al., 2005; and Rounds et al., 2009).

The term "PPCPs" describes a wide array of prescription and over-the-counter drugs for humans and animals (Daughton and Ternes, 1998). These include illicit drugs and diagnostic agents such as x-ray contrast media, nutraceuticals (bioactive chemicals in nutritional supplements), and inert ingredients such as pill coatings (Motzer, 2006). Personal care products include items for personal care such as shampoo, soap, fragrances, and lotions.

PPCPs are found in wastewater because the human body does not completely metabolize all the compounds (Heberer, 2002). Additionally, PPCPs wash off of the human body or are improperly disposed of in toilets, sinks, or trash. Other sources to the environment may include PPCPs in landfills, as well as drugs used for livestock, pets, and aquaculture.

Thousands of pharmaceutical chemicals are in use today, particularly in developed countries (Rounds et al., 2009), and their environmental fate has been studied by only a few researchers. Of note was a national reconnaissance study by the U.S. Geological Survey (USGS) that assessed the presence and occurrence of PPCP detections in 139 streams across the U.S. (Kolpin et al., 2002).

The presence of PPCPs in the environment results from their universal, frequent, and cumulative usage. This continual introduction into the environment causes a pseudo-persistence that might not otherwise exist (Halling-Sorensen et al., 1998). Low concentrations of PPCPs have been detected in a wide array of environmental media including surface water, groundwater, marine waters, soils, sediments, and drinking water. The impact of continuous low-level PPCP exposure on human health and wildlife is unknown.

The relative lack of information on the environmental concentration or potential subsequent ecological effects of these chemicals has led to debate about the specific need to remove them from wastewater. The Washington State Department of Ecology (Ecology) prepared a literature review of sources, ecological effects, and removal efficiency of PPCPs by WWTPs (Appendix A).

Scientists are beginning to understand the treatability of PPCPs, and there appears to be a relationship between nutrient removal and PPCP removal. The volume of wastewater and the pollutant load of nutrients and PPCPs increase with population growth. The population growth rate in the Puget Sound/Georgia Basin region exceeds the average global growth rate (EPA, 2009a). Each year several areas of Puget Sound experience high nutrient concentrations that exceed water quality standards. A recent nutrient loading study found loads from WWTPs to be greater than loads from rivers in the South Puget Sound (Roberts et al., 2008). Currently

the concentrations of PPCPs in municipal wastewater discharged from WWTPs within the Puget Sound watershed are unknown.

Problem Statement

Local information on sources of PPCPs, as well as their fate, transport, and impacts, is needed.

There are numerous studies which document the presence of PPCPs in the environment. A few of these have been conducted or had sites in the Pacific Northwest (Johnson et al., 2004; Rounds et al., 2009; Lower Columbia River Estuary Partnership, 2007; Kinney et al., 2008; and Kolpin et al., 2002). The occurrence of several PPCPs at low concentrations has been documented in surface water, groundwater, marine waters, drinking water, soils, and sediments.

Three studies to date have sampled for PPCPs in the Pacific Northwest environment.

1. A 2004 screening study in Sequim, Washington sampled discharges from Sequim and Sunland WWTPs, as well as several local creeks and wells (Johnson et al., 2004). The researchers found PPCPs occurred in all locations; however, only three compounds (caffeine, metformin, and nicotine) were found in groundwater or surface water. The methods used for the analyses have since been improved.

2. USGS published a recent study of PPCPs in surface waters in the Tualatin River basin, Oregon (Rounds et al., 2009). The occurrence of 21 pharmaceutical compounds was surveyed from five streams and the Tualatin River, as well as at one WWTP. The samples were field filtered, a requirement of the USGS study methods; the results therefore only reflect the dissolved fraction of the targeted compounds.

   Six of the 21 targeted analytes (cotinine, caffeine, acetaminophen, carbamazepine, codeine, and sulfamethoxazole) were found in the stream samples. Five (carbamazepine, cotinine, ibuprofen, metformin, and sulfamethoxazole) were detected in the Durham WWTP effluent. The authors reported wastewater effluents were the primary sources of low concentrations of carbamazepine and cotinine measured in the Tualatin River.

3. The Lower Columbia River Estuary Partnership (2007) measured the concentrations of 33 organic compounds including PPCPs in salmon and the Columbia River. Caffeine was detected in water samples at every site. Bisphenol A, HHCB[1], trimethoprim, and anhydroerythromycin were also frequently detected. PPCPs were more commonly detected during the low-flow sampling event in August than the high-flow sampling event in April. Concentrations were measured in the microgram per liter range (ppb).

As a first step, this 2008 reconnaissance study was proposed to quantify the concentrations of PPCPs in municipal effluents, reclaimed water, and biosolids from five Northwest WWTPs. The primary objectives were to characterize PPCP concentrations and assess PPCP removals by different wastewater treatment processes.

[1] HHCB (1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta-gamma-2-benzopyran and related isomers) belongs to a group of polycyclic musk fragrances and is known as Galaxolide®.

Municipal Wastewater and Treatment - Background

Wastewater treatment has several stages which may include the following processes: primary clarification, biological or secondary treatment, secondary clarification, tertiary treatment, disinfection, and solids (sludge) treatment. Conventional secondary wastewater treatment typically involves primary solids removal, biological treatment, secondary clarification, and disinfection. Tertiary treatment is generally considered any additional treatment beyond the secondary process, such as nutrient removal, chemical addition, or filtration. Solids removed during wastewater treatment undergo treatment to stabilize the solids organic content and reduce pathogens. The product, termed *biosolids*, is usually supplied as a soil amendment or infrequently disposed of at a landfill.

Wastewater treatment traditionally focused on the reduction of solids, organic material, and pathogens. Federal requirements during the 1970s mandated secondary treatment as the baseline treatment in the U.S. due to degraded water quality conditions. Population growth since secondary treatment was installed has significantly increased the volume of wastewater to be treated. Therefore, although the quality of wastewater discharged by most WWTPs is much better than in decades past, the increased volume of these discharges has offset some of those benefits. Secondary treatment requirements do not specify performance standards for nutrient removal. Exceptions are case specific where excess nutrients indirectly cause oxygen depletion in the water column through algae growth and decomposition.

Emerging Contaminants

In the last decade, organic wastewater compounds have become a concern. Research on the treatability of various organic compounds by wastewater treatment technologies is beginning to be available.

The science for detecting PPCPs at very low levels has recently been developed. For example, the concentration of a common PPCP, such as ibuprofen, is between 1-40 ug/L in the influent and is reduced to 0.026 ug/L or below in the effluent. Compared to nutrients, these concentrations do not appear to be large. However, because pharmaceuticals were designed with a physiologically effective purpose, the low concentrations in wastewater effluent should not be dismissed as trivial. However, an assessment of risk, which is not based on environmental concentrations but on dose and response, was beyond the scope of this study.

The growing volume of wastewater carries a wide array of chemicals, including PPCPs, which are used in ever increasing quantities by our society. The Associated Press reported that as of 2008, over 50% of Americans with health care insurance are using prescription drugs on a daily basis.

The fate of PPCPs in the environment is a complex issue. First, there are thousands of chemicals used in the manufacture of PPCPs. Second, the different types of chemicals react differently in the wastewater treatment processes. Third, there are many different wastewater treatment processes employed to reduce nutrients, solids, and chemicals.

Research

Several studies have evaluated removal efficiency of PPCPs by different treatment processes (Snyder et al., 2007; Miege et al., 2008). These include reverse osmosis, ozonation, membrane bioreactors, constructed wetlands, and riverbank filtration (Snyder et al., 2007; Drury et al., 2006; Kimura et al., 2005; Barber et al., 2006; Heberer et al., 2004, respectively).

None of the processes evaluated has been found to remove 100% of all PPCPs. Some treatment processes effectively reduce some pharmaceuticals down to very low levels, while other pharmaceuticals remain resilient to removal by conventional secondary or tertiary wastewater treatment. PPCPs resistant to treatment include, but are not limited to, carbamazepine, fluoxetine, clofibric acid, mefenamic acid, phenazone, diclofenac, and dimethylaminophenazone (Kinney et al., 2006a; Kimura et al., 2005; Miege et al., 2008; Rounds et al., 2009; Ternes, 1998).

Researchers at the Cemagref Water Quality and Pollution Control Research Unit in France have compiled a database from 113 international research papers on the occurrence and removal of PPCPs from WWTPs (Miège et al., 2008). Data collection included types of processes, operating conditions, influent and effluent data, mixed liquor in the biological reactor, volume of the reactor, retention times, and other physical characteristics. The Cemagref database found that only 32 PPCP chemicals comprise 80% of the data in their database.

Some studies have shown that operating the WWTP with a longer solids retention time (SRT), which allows for a longer biological contact time, will increase PPCP removal rates. Retention time is often longer for WWTPs that operate biological nutrient removal. Also, pH changes within the treatment system may increase the rate of antibiotic removals (Holtz, 2006).

The Cemagref database allowed researchers to calculate removal efficiencies based on data from 24-hour flow proportional composites samples. Operating conditions were cited as playing a large role in PPCP removal. In fact, processes with nitrogen treatment and high hydraulic retention time (HRT) of >12 hours, and high SRT of >10 days, were found to be more efficient in removing PPCPs than processes without nitrogen treatment. The most effective processes were biological treatment (50-90%) such as conventional activated sludge with nitrogen treatment and with membrane bioreactors combined with nitrogen treatment (Miège et al., 2008).

Reclaimed Water

Reclaimed water has been used in the U.S. for more than 40 years. The level of treatment of reclaimed water varies depending on the intended use. Tertiary filtration and additional disinfection of secondary effluent produce high quality reclaimed wastewater which is most often used for irrigation. A reclaimed water facility in San Diego was tested for 138 organic compounds plus other inorganic chemicals. Researchers found no significant non-carcinogenic health risks, and the carcinogenic risks were 1000 times less than the public water supply (Olivieri, 2008).

Advanced treatment processes used to reclaim water – such as reverse osmosis, multiple barriers (Olivieri, 2008), or ozonation (Drury et al., 2006) – are effective at reducing PPCP concentrations. Levels of PPCPs detected in reclaimed water discharges (Kinney et al., 2006a)

and receiving waters (Kolpin et al., 2002) are much lower than in conventional WWTP effluents (Cooperative Research Centre, 2007).

Washington State currently has 321 municipal WWTPs (Jones, 2008). There are also 20 reclaimed water treatment facilities, which differ from WWTPs in that they achieve a higher level of treatment than secondary wastewater treatment. These additional treatments are to protect the beneficial uses of the water and the potential for human contact (Cupps, 2003). Only a few WWTPs in Washington State provide reclaimed water treatments because they are expensive treatment options (Jones, 2008).

Kinney et al. (2006a) determined that reclaimed water used for irrigation resulted in low soil concentrations for some pharmaceuticals. Most of the pharmaceuticals did not show a net accumulation over the six-month study, with the exception of carbamazepine that consistently increased in concentration at the three field sites. Carbamazepine, fluoxetine, and diphenhydramine concentrations persisted for six months post irrigation. The low water solubility for these compounds was believed to be an important factor.

Biosolids

Biosolids are the organic, nutrient-rich by-products of wastewater treatment. They can be beneficially used as soil amendments. The definition of biosolids and the treatment requirements by Washington State are further described in Chapter 70.95 RCW, Chapter 173-308 WAC, 40 CFR Part 503, and the "Biosolids Management Guidelines for Washington State".

As part of the wastewater treatment process, the solid material removed from the clarifiers, called sludge, is stabilized. Two common methods to stabilize sludge are heat and lime stabilization or heat and biological digesters. The stabilized sludge is subsequently dewatered and is called biosolids.

In the U.S., WWTPs generate approximately 7 million dry tons of biosolids per year. The per capita volume of wastewater is 450 liters, which contains approximately 240 mg/L of suspended solids (80% organic matter) (Kinney et al., 2006b). This equates to 86 grams of biosolids produced per person per day. Approximately 50% of the biosolids generated in the U.S. are land-applied, with less than 1% being applied to the nation's agricultural lands (Kinney et al., 2006b). The other 50% is either sent to the landfill or incinerated.

Some WWTPs further treat their biosolids to yield a market product. There may be further reduction to the PPCP contaminant levels in the marketed biosolids products, but there is a lack of information on this topic.

The individual chemical structure dictates whether PPCPs will biodegrade, volatilize, or degrade into metabolites, and whether they will concentrate and persist in the environment (Holtz, 2006). Biosolids are rich in organic matter, meaning they have a large capacity to bind to organic compounds.

There is debate about the importance of the physiochemical property called the octanol-water partition coefficient (K_{ow}) in predicting the fate of PPCPs in biosolids. The K_{ow} of a chemical

can be used to predict whether it will move out of wastewater and into biosolids. Chiou and Kile (2000) stated the most influential factor in determining the fate of organic chemicals in the environment was K_{ow}. On the other hand, Oppenheimer and Stephenson (2006) found no correlation between K_{ow} and frequency of occurrence of PPCPs in secondary effluents.

Organic compounds with low water solubility and large log K_{ow} are generally expected to be found in the biosolids. However, Kinney et al. (2006b) found chemicals with a wide range of log K_{ow} values (1.50 to 9.65) in biosolids, leading them to conclude that partitioning of PPCPs from wastewater to biosolids is variable and not well-correlated to K_{ow}. Kinney et al. (2006b) also found that the composition and concentration of PPCPs in biosolids varied little with WWTP operations or size.

Kinney et al. (2006b) sampled the biosolids products at nine municipal WWTPs (two in Washington) and found 55 of 87 analytes were detected in at least one biosolids product. The most commonly detected compounds in biosolids were pharmaceuticals, detergent metabolites, steroids, fragrances, and polycyclic aromatic hydrocarbons (PAHs).

Table 1 presents the mass loading rate for a single application determined by Kinney (2006b) based on the average detected concentration and an EPA agricultural application rate of 10 dry tons per acre. This was considered a representative application rate for many crops.

Table 1. PPCP loading estimates from Kinney et al. (2006b).

Parameter	Load
Organic wastewater contaminants	3.4 kg/acre
Carbamazepine (pharmaceutical)	0.2 g/acre
Triclosan (disinfectant)	20 g/acre
Tonalide AHTN (synthetic musk)	26 g/acre
Para-nonylphenol (detergent metabolite)	760 g/acre

Little is known about the environmental fate of organic wastewater contaminants from land application of biosolids. However, Kinney (2006b) concludes there is considerable contaminant loading to the terrestrial environmental from biosolids and reclaimed water. The persistence of organic wastewater contaminants in the soil warrants concern, and further research is needed.

Eastern Washington University scientists, in conjunction with USGS, conducted a study investigating anthropogenic organic contaminants in biosolids, manure, and unimpacted fields (Kinney et al., 2008). They evaluated contaminants in the soils and earthworms. The study found that organic chemicals, including some PPCPs, were introduced into the environment through land application of manure or biosolids. Earthworms continually ingest soils for nourishment and were found to accumulate PPCPs contained in those soils, therefore indicating bioaccumulation of some PPCPs and an introduction to the food chain. There is an additional level of uncertainty, however, surrounding whether or not these concentrations are capable of causing a risk to the environment or human health.

Methods

Study Design

Ecology and EPA collaboratively developed a project to characterize the concentrations and removal efficiencies of PPCPs by municipal WWTP processes in the Pacific Northwest. On August 19, 2008, influent, effluent, and biosolids were sampled for 172 organic compounds: 72 pharmaceuticals and personal care products; 27 hormones and steroids; and 73 semi-volatile organics. We also tested for phosphorus, nitrogen, and total suspended solids. Appendix B lists the 172 organic compounds analyzed during this study.

Five WWTPs providing conventional secondary treatment, advanced nutrient removal, reclaimed water, and/or filtration were selected for sampling to determine if different treatment processes displayed differences in removal of PPCPs.

Sampling Locations

In accordance with the Quality Assurance (QA) Project Plan for this project (Lubliner et al., 2008), composite samples for influent as well as secondary and tertiary effluent(s) were collected from seven wastewater streams at four WWTPs located near Puget Sound. Puget Sound area WWTPs do not currently provide low-level phosphorous treatment. The nearest WWTP providing this treatment was the Hayden, Idaho, Reclaimed Water Plant. Therefore, the fifth WWTP sampled is located in Hayden, Idaho. Class B biosolids were sampled from three of the five WWTPs.

The WWTPs are listed in Table 2, shown in Figure 1, and described in detail in Appendix C. LOTT Alliance contributed to the study by conducting sampling of the influent and effluent from their reclaimed water facility on Martin Way in Lacey. These five facilities were selected for sampling because each is well-operated and employs a different treatment process.

The goals of the monitoring project are to (1) better understand the range of PPCP concentrations in different effluents and (2) gain some insight on PPCP removal by different treatment processes.

Domestic WWTPs and industrial discharges operate under individual National Pollutant Discharge Elimination System (NPDES) permits and Reclaimed Water permits. Reclaimed Water permits are administered by Ecology and the state Department of Health. Ecology administers NPDES permits for discharges in Washington, except for federal and tribal facilities which are regulated by EPA. EPA also administers the NPDES program in Idaho. Currently there are neither NPDES nor Reclaimed Water monitoring requirements established for PPCPs in the permits.

Table 2. Sampling locations.

Wastewater Treatment Plants		Location of WWTP	Sampling Location at WWTP	Receiving Water For Effluent
1	LOTT Budd Inlet WWTP and LOTT Budd Inlet Reclaimed Water Plant (RWP)	Olympia, WA	• Raw influent • Secondary treatment process • Tertiary effluent (reclaimed water) • Biosolids	Budd Inlet in Puget Sound
2	LOTT Martin Way Reclaimed Water Plant (RWP)	Olympia, WA	• Raw influent • Tertiary effluent (reclaimed water)	Groundwater recharge
3	Pierce County, Chambers Creek WWTP	Tacoma, WA	• Raw influent • Secondary treatment process • Final effluent • Biosolids	Puget Sound
4	City of Puyallup WWTP	Puyallup, WA	• Raw influent • Final effluent • Biosolids	Puyallup River which flows into Puget Sound
5	City of Hayden WWTP and Hayden Wastewater Research Facility (WRP)	Hayden, ID	• Raw influent • Secondary treatment process • Tertiary effluent (reclaimed water)	Land application or Spokane River

Figure 1. Sampling locations.

Table 3 lists the treatment technology employed by each plant and the code used to describe that technology throughout this report.

Table 3. WWTP descriptions and treatment codes.

	Wastewater Treatment Plant	Abbreviation for Treatment	Process of Treatment
1	LOTT, Budd Inlet WWTP Olympia, WA	EBNR*	Enhanced biological nitrogen removal (EBNR) incorporated into the secondary treatment process via a modified four-stage process.
	LOTT, Budd Inlet Reclaimed Water Plant Olympia, WA.	EBNR+F*	A portion of the secondary effluent from the EBNR process is treated by chemical addition and sand filtration.
2	LOTT, Martin Way Reclaimed Water Plant Lacey, WA	EBNR+MF*	Enhanced biological nitrogen removal with membrane filtration.
3	Pierce County, Chambers Creek WWTP Tacoma, WA	AS	Activated sludge.
4	Puyallup WWTP Puyallup, WA	AS+N*	Activated sludge with partial nitrification and denitrification.
5	Hayden WWTP. Hayden, Idaho	AD	Secondary treatment by aeration ditch.
	Hayden Wastewater Research Facility Operated by Blue Water Inc. Hayden, Idaho	CA+F*	A portion of the Hayden WWTP secondary effluent receives tertiary treatment by chemical addition and tertiary two-stage sand filtration for phosphorus removal.

*Effluents sampled that had some degree of additional nutrient removal.
EBNR = secondary effluent with enhanced biological nutrient removal.
EBNR+F = enhanced biological nutrient removal and tertiary filtration.
EBNR+MF = enhanced biological nutrient removal and tertiary membrane filtration.
CA+F = chemical addition and filtration applied to secondary effluent.
AS+N = final effluent from activated sludge treatment operated to provide nitrification.
AS = secondary effluent from activated sludge treatment.
AD = secondary effluent from aeration ditch treatment.

All of the EBNR and CA+F treatments were considered to be advanced treatment for nutrient removal. The EBNR treatment process provides biological removal of nitrogen. Nitrogen removal is accomplished by recycling of wastewater where nitrification occurs in aerated zones and denitrification occurs in anoxic zones maintained within the biological treatment process.

LOTT has implemented design changes to the Budd Inlet WWTP to improve treatment efficiency and reduce energy consumption. At the Budd Inlet WWTP, a portion of the secondary effluent is routed through additional treatment to meet Washington State Class A reclaimed wastewater treatment standards. This treatment includes coagulant addition and filtration through single-stage, continuous-backwashing, upflow sand filters (from Parkson Corporation), and additional disinfection with chlorine. The reclaimed water is used for irrigation at various locations in the Olympia area, and the secondary effluent is discharged to Puget Sound.

The Martin Way Reclaimed Water Plant (RWP) diverts a portion of the wastewater flowing to the Budd Inlet WWTP from the collection system in Lacey and Olympia. The Martin Way RWP treats the wastewater to Washington State Class A reclaimed water standards using enhanced biological nutrient removal followed by membrane filtration and disinfection. The reclaimed water is used at the Hawks Prairie Reclaimed Water Ponds and Recharge Basins. Planned uses include toilet flushing, decorative fountains and ponds, and dust suppression.

The third plant with advanced nutrient-removal technological processes is the Hayden Wastewater Research Facility (WRF), which treats approximately 0.25 MGD of the Hayden secondary effluent using Blue PRO (registered trademark name). This treatment process uses chemical addition (ferric sulfate) and two-stage filtration through the company's continuous-backwashing, upflow sand filters. The low phosphorus effluent is seasonally land-applied or discharged to the Spokane River.

The Puyallup WWTP provides some nitrification in the activated sludge process that typically reduces ammonia concentrations in the final effluent. The anoxic and aerobic zones in the activated sludge process also provide incidental biological removal of phosphorus. For purposes of this study, this WWTP provides an intermediate level of nutrient removal between secondary and tertiary treatment. The plant's effluent is discharged to the Puyallup River which is a tributary to Puget Sound.

Two of the studied facilities provide only secondary treatment to the wastewater. Chambers Creek WWTP employs activated sludge for secondary treatment and discharges directly to Puget Sound. The Hayden WWTP uses aeration ditches to oxidize the wastewater followed by clarifiers to settle the solids. At the time of sampling, two of the three oxidation ditches and two clarifiers were in service.

Parameters Sampled at Each Location

Table 4 lists nutrient characteristics to indicate operation efficiency of the WWTP at the time of sampling. These data come from either the monthly discharge monitoring reports (DMRs) or are sample results of this project.

Nutrient results from this 2008 study may be different from the WWTPs' own sampling results. This difference may be due to different sampling methods. For example, the Puyallup WWTP measured effluent ammonia values of 0.51 mg/L on August 17 and 0.8 mg/L on August 20, an order of magnitude below this study result of 5.17 on August 19. Their method for measurement uses a 24-hour composite (as the EPA permit requires); whereas our 2008 study sampled by 8-hour composite by hand grabs. This anomaly was not explored.

Table 4. WWTP effluent nutrient characteristics at time of sampling, August 19, 2008, mg/L.

Parameter (One sampler per result)	Budd Inlet (EBNR)	Budd Inlet RWP (EBNR+F)	Martin Way RWP (EBNR+MF)	Chambers Creek (AS)	Puyallup (AS+N)	Hayden (AD)	Hayden WRF (CA+F)
Total Suspended Solids	5	1	2 U[1]	5	2	2	1 U
Ammonia (NH_4)	0.04	0.01	--	41.1	5.17[3]	5.87	5.93
Nitrite-Nitrate (NO_3-NO_4)	1.2	1.4	--	4.1	5.0	2.3	2.3
Total Persulfate Nitrogen (TPN)	2.0	2.1	3.4[2]	37.6	11.9	8.6	9.3
Organic Nitrogen (TPN-NH_4-(NO_3-NO_4))	0.8	0.7	--	<0.01	1.7	0.4	1.1
Total Inorganic Nitrogen (TIN) = (NO_3+NO_2)+NH_4	1.2	1.4	--	45.2	10.2	8.2	8.2
Orthophosphate (OP)	3.44	3.42	--	1.53	3.25	4.27[4]	0.004[4]
Total Phosphorus (TP)	3.24	2.85	--	1.56	2.79	4.22[4]	0.02[4]
Organic Phosphorus (TP-OP)	<0.005	<0.005	--	0.03	<0.005	<0.005[4]	0.016[4]

-- No data.
U = non-detect at the given laboratory reporting limit.
TSS = total suspended solids.
[1] Daily value from the LOTT Discharge Monitoring Report for the Martin Way RWP.
[2] Monthly mean for total nitrogen from the LOTT Discharge Monitoring Report for the Martin Way RWP.
[3] Routine monitoring by WWTP operators typically measures ammonia concentrations to be approximately 0.5 mg/L.
[4]Results from resample date, November 19, 2008.

WWTP Operating Conditions

Operating conditions, such as retention time for water and solids, are summarized in Table 5. These are used to gain an understanding of the WWTP secondary process capacity and flow design.

The hydraulic retention time (HRT) for the entire plant is the time calculated by dividing the entire plant volume by the discharge rate. The secondary process HRT is the whole secondary process volume divided by the discharge rate. This would be inclusive of aerobic and anoxic zones and is the same value the plant lists on their DMR, but does not include the secondary clarifier(s).

The SRT is the average time of retention of suspended solids in a biological waste treatment system, equal to the total weight of suspended solids leaving the system, per unit time. The SRT value is larger if the plant recycles the wastewater back to the start of the secondary process. SRT is often used synonymously with mean cell residence time.

Table 5 lists the operating conditions at the time of sampling, as described by each plant or from DMRs.

Table 5. WWTP operating conditions at time of sampling, August 19, 2008.

Operating Conditions	Budd Inlet (EBNR)	Budd Inlet RWP (EBNR+F)	Martin Way RWP (EBNR+MF)	Chambers Creek (AS)	Puyallup (AS+N)	Hayden (AD)	Hayden WRF (CA+F)
August average plant discharge, MGD	8.6 [a]	0.4 [ab]	0.6[a]	16[a]	3[a]	2	0.5 [b]
Total Plant Hydraulic Retention Time (HRT), hours	24.5 [b]		24.5	10.7	42	28 [b]	
Mixed Liquor Total Suspended Solids, mg/L	1984 [b]		7000	1460	2,000	2,876[b]	
Solids Retention[c] Time (SRT), days	18.6 [b]		25	2.5	18	12[b]	
Total Secondary Process HRT, hours	22.8[b]		24	4.7	26	26[b]	

[a] = Value from DMR accessed through Ecology's files.
[b] = A portion of the secondary effluent is treated by the tertiary process, which does not add to the residence time.
[c] = Synonymous with mean cell residence time (MCRT).

Sample Collection

Ecology, EPA, and LOTT staff sampled on Tuesday, August 19, 2008. LOTT and the Puyallup operators have reported that weekday flow rates include commercial and manufacturing flows and tend to be higher than weekend flow rates. Also, samples were collected on a work day for convenience with plant operators.

Influent and Effluent

Sampling was conducted to collect the most representative sample of raw influent, secondary effluent, and tertiary effluent (if a tertiary treatment process was employed).

Individual grab samples were collected (morning, noon, and afternoon) and hand composited by equal volume into clean[2] glass jars with Teflon lids. Sample bottles were provided by MEL. Samples were kept on ice in coolers between sampling times. After the third sub-sampling, the composite was shaken to mix, and a small portion of the mixture was transferred into the nutrient bottles. Nutrient samples were preserved, and the orthophosphate sample was field-filtered using a 0.45 um syringe filter and preserved.

[2] Priority-pollutant cleaned according to EPA QA/quality control specifications (EPA, 1990).

A re-sampling at the Hayden WWTP for total phosphorus and orthophosphate occurred on November 19, 2008, due to field and laboratory errors of the original total-phosphorus sample. The re-sampling was coordinated with facility staff in an effort to match influent phosphorus concentrations to the August 19 sampling date.

Biosolids

Grab samples of Class B biosolids were collected from Budd Inlet, Chambers Creek, and Puyallup WWTPs on August 19, 2008. These three WWTPs use digesters to stabilize the biosolids on site. Biosolids samples were not collected from the Hayden WWTP or Water Research Facility because they were not processing solids at that time. Biosolids were also not collected from the Martin Way RWP because the sludge is re-introduced to the sewer system and sent to Budd Inlet WWTP for processing.

Class B biosolids were scooped from the truck, piles, or belt press at each site. Five sub-grabs were combined into clean[2] glass jars and kept on ice in coolers. The biosolids samples were intentionally taken post digester and dewatering, and before any further treatment, to be comparable across the three WWTPs. Chambers Creek also produces Class A biosolids, but they were not sampled.

For all sampling, field personnel wore powder-free nitrile gloves at all times during sample collection, and they followed standard health and safety procedures. The samples were maintained on ice in coolers and transported to Ecology's Manchester Environmental Laboratory (MEL) or sent by Fed-Ex directly to Axys Analytical Laboratory, British Columbia, Canada. Chain-of-custody was maintained.

Laboratory Analysis

Target Analytes

The term PPCP does not independently define a list of analytes. At the onset of this 2008 study, a list of 24 targeted analytes was compiled from national and international studies based on their reported traceability, bioaccumulation, and endocrine-disruption potential. Table 6 lists these 24 targeted analytes.

These analytes are reported on throughout the document; however, a total of 172 organic compounds were evaluated (see Appendix B) for this study.

Table 6. Target PPCP analytes for this 2008 study.

Analyte	Chemical Type	Rationale	Reference
17α-ethinyl-estradiol	reproductive hormone	synthetic hormone in pharmaceuticals	--
17β-Estradiol	reproductive hormone	synthetic hormone in pharmaceuticals	--
4-nonylphenol	non-ionic detergent metabolite	recommended indicator parameter	Zdwadzkas, 2005
Acetaminophen	analgesic	detected 83% in surface waters	Boyd and Fulong, 2002
		present in groundwater	Benotti et al., 2006
		detected in drinking water	Zimmerman, 2005
Bis(2-ethylhexyl) phthalate (DEHP)	plasticizer	emerging contaminant	--
Bisphenol A	plasticizer	recommended indicator parameter	Zdwadzkas, 2005
Caffeine	stimulant	common indicator, only human source	Buerge et al., 2003
		recommended indicator parameter	Zdwadzkas, 2005
		present in groundwater	Benotti et al., 2006
Carbamazepine	antiepileptic	high detection frequency in environment	Glassmeyer et al., 2005
		only human source	Glassmeyer et al., 2005
		detected 83% in surface waters	Boyd and Fulong, 2002
		present in surface water	Ternes et al., 2002
		most commonly detected PPCP	Benotti et al., 2006
		present in groundwater	Heberer et al., 2004
		persistent in soils	Kinney et al., 2006a
		detected in drinking water	Zimmerman, 2005
Coprostanol	fecal sterol	recommended indicator parameter	Zdwadzkas, 2005
		dramatic differences in up/down stream	Glassmeyer et al., 2005
Cotinine	nicotine metabolite	high detection frequency in environment	Glassmeyer et al., 2005
		present in groundwater	Benotti et al., 2006
Di-n-butylphthalate (DBP)	plasticizer	ingredient in nail polish and hair spray	--
Diphenhydramine	antihistamine	only human source	Glassmeyer et al., 2005
		persistent in soils	Kinney et al., 2006a
Erythromycin	antibiotic	persistent in soils	Kinney et al., 2006a
Fluoxetine	antidepressant	persistent in soils	Kinney et al., 2006a
Gemfibrozil	lipid regulator	present in surface water	Ternes et al., 2002
		present in groundwater	Benotti et al., 2006
Ibuprofen	anti-inflammatory	present in surface water	Ternes et al., 2002
Metformin	anti-diabetic	commonly used pharmaceutical	--
Naproxen	anti-inflammatory	present in surface water	Ternes et al., 2002
Sulfamethoxazole	antibiotic	high detection frequency in environment	Glassmeyer et al., 2005
		present in groundwater	Benotti et al., 2006
		most commonly detected PPCP	Benotti et al., 2006
		detected in drinking water	Zimmerman, 2005

Analyte	Chemical Type	Rationale	Reference
Tetracycline	antibiotic	commonly used pharmaceutical	--
TCEP tri(chloroethyl) phosphate	fire retardant	high detection frequency in environment	Glassmeyer et al., 2005
		recommended indicator parameter	Zdwadzkas, 2005
		persistent	Stephenson and Oppenheimer, 2007
		resistant to treatment	Snyder et al., 2007
Triclocarban	anti-microbial disinfectant	high detection frequency in environment	Glassmeyer et al., 2005
Triclosan	anti-microbial disinfectant	high detection frequency in environment	Glassmeyer et al., 2005
Trimethoprim	antibiotic	present in groundwater	Benotti et al., 2006

An international effort to prioritize the list of PPCPs was published in 2009 by the Global Water Research Coalition. Their goal was to consolidate PPCP prioritization activities in North America, Europe, Australia, and East Asia, based on seven criteria (including use, toxicity, consumption, properties, and persistence) (de Voogt et al., 2009). The Coalition developed three classes of pharmaceuticals: (1) high priority, (2) priority, and (3) lower priority. The high priority Class I chemicals are listed below and should constitute the *minimum* for any PPCP study consideration:

- Carbamazepine*
- Sulfamethoxazole*
- Diclofenac
- Ibuprofen*
- Erythromycin*
- Bezafibrate
- Ciprofloxacin
- Atenolol
- Naproxen*
- Gemfibrozil*

Their lists purposefully excluded veterinary medications and were based only on published studies (de Voogt et al., 2009). Seven of the ten compounds (noted with asterisks *) were a part of our 2008 study; the remaining three were not available in the EPA analytical methods used by this study.

Analytical Methods

Methods development for PPCPs has been highly active in the last decade with techniques that have converged on tandem mass spectrometry (Terns and Joss, 2007).

Unfortunately, most researchers have developed their own list of analytes within the technological capacity of the laboratory equipment, even within federal agencies. For example, USGS and EPA each have laboratory methods with different chemicals on each list. As a result,

comparisons between studies are difficult. The recent Water Environment Research Foundation publication on trace organic compounds indicated a need for consistency in analytes and methods for chemical analyses (Anderson, 2008).

Fortunately, two new EPA methods were released in December 2007 (EPA, 2007a; 2007b). These methods were chosen for this 2008 study because they (1) provide a predetermined list of analytes which included most of our 24 target analytes, (2) provide low laboratory reporting limits with the highest degree of quality assurance, and (3) are capable of dealing with strong matrix interferences from wastewaters and solids. The two new methods are single-lab validated methods for pharmaceuticals (EPA Method 1694) and steroids and hormones (EPA Method 1698).

In addition an older EPA method, Method 625:8270d for semi-volatile organics extractable compounds, was used to capture additional consumer products, including personal care products of interest. Many of the compounds on this list are PAHs and were not the focus of our study.

A synopsis of the analytical methods used in this 2008 study to quantify the organic compounds is provided below. An important distinction between the EPA methods and USGS methods is that the EPA methods used in this study do not require field filtration and therefore analyze the whole sample. Using these three analytical methods, all 24 target analytes were measured.

- **EPA Method 1694 for Pharmaceuticals and Personal Care Products "PPCPs[1694]"**. A total of 72 PPCPs were analyzed for by high performance liquid chromatography combined with tandem mass spectrometry (HPLC/MS/MS) using isotope dilution and internal standard quantitation techniques. This specific list of 72 PPCPs will be noted herein as PPCPs[1694]. Axys Method MLA-052 is functionally equivalent to EPA Method 1694 (EPA, 2007a).

- **EPA Method 1698 for Hormones and Steroids "Hormones/Steroids"**. A total of 27 hormones and steroids were analyzed by this method as follows: solvents are used to extract the sample, followed by cleanup with a layered alumina/Florisil column, and an option to remove sulfur using copper. Following cleanup, the target analytes are derivatized to make them sufficiently volatile for analysis by Gas Chromatography/High Resolution Mass Spectrometry (GC/HRMS). Quantitation is performed by isotope dilution and internal standard techniques (EPA, 2007b).

- **EPA SW-846 Method 8270d for Base-Neutral and Acid Extractable Compounds "Semi-Volatile Organics"**. A total of 73 semi-volatile organics were extracted by Method 846 and analyzed within the guidelines of Method 8270d. A standard operating procedure was followed to document any modifications for the particular compounds. Some semi-volatile organic compounds are commonly used in PPCPs. Approximately 15 of the analytes available using this method were of interest to this project.

  Appendix B lists all the semi-volatile compounds tested. Of particular interest were chemicals used in consumer products that have estrogenic properties (EPA, 1984). These include bis-phenol A, 4-nonylphenol, multiple phthalates, and tri(2-chloroethyl) phosphate (TCEP).

All analytical methods employed by this project are listed in Table 7.

Table 7. Laboratory methods, number of samples, and reporting limits.

Parameter	Total Number of Samples	Analytical Method	Practical Quantitation Limit	Laboratory
Pharmaceuticals and Personal Care Products[1]	18 (water)	EPA Method 1694	2-10 ng/L	Axys
	4 (solids)		01-100 ug/Kg	
Hormones/Steroids[1]	18 (water)	EPA Method 1698	2-10 ng/L	Axys
	4 (solids)		01-100 ug/Kg	
Semi-Volatile Base/Neutral Acid/Extractables[1]	16 (water)	EPA SW-846 Method 8270	2-10 ng/L	MEL
	4 (solids)		01-100 ug/Kg	
Ammonia (NH4)	14	SM 4500-NH_3 H	10 ug/L	MEL
Nitrate+Nitrite (NO_3+NO_2)	14	SM 4500-NO_3 I	10 ug/L	MEL
Total Persulfate Nitrogen (TN)	14	SM 4500-NO_3 B	25 ug/L	MEL
Orthophosphate (OP)	16[2]	SM 4500-P G	3 ug/L	MEL
Total Phosphorus (TP)	16[2]	SM 4500-P I	1 ug/L	MEL
Total Suspended Solids (TSS)	14	SM 2540D	1 mg/L	MEL
Percent Solids (% solids)	4	SM 2540G	1% wet weight	MEL

SM = Standard Methods for the Examination of Water and Wastewater, 21st Edition (APHA, 2005).
[1] A range of values is presented due to the large list of analytes for each method.
[2] A resample of orthophosphate and total phosphorus was made at the Hayden WWTP for the secondary effluent and Water Research Facility.

Sample containers, preservations, and holding times were listed in the QA Project Plan for this project (Lubliner et al., 2008).

Two laboratories were used during this project:

1. Ecology's Manchester Environmental Laboratory (MEL) in Manchester, Washington, analyzed for semi-volatile organics, nutrients, and solids.
2. Axys Analytical Laboratory Inc. (Axys) in Sydney, B.C., Canada, analyzed for PPCPs and hormones/steroids using Methods 1694 and 1698. Axys was the only laboratory in North America at the time of this study (August 2008) that had the commercial capability for using these newly released methods.

Data Analysis

The values of all reported concentrations are deemed acceptable for the purposes of the study. The data are presented primarily as ranges in this report; calculations of central tendency (i.e., mean) were not performed. The goal is to characterize the concentrations from the various technologies. Therefore, there was no need to perform substitutions for non-detected compounds.

Non-detected compounds are represented as "nd" in the report; however, the actual reporting limit and "U" qualifier is provided in Appendices D and E.

Data Quality

Data from MEL was reviewed according to laboratory protocol (MEL, 2006) and by the Ecology project lead. All analytical results were subjected to thorough data review procedures. In addition, data from Axys were third-party reviewed by EPA's Quality Assurance Officer. Ecology and EPA reviewed the qualitative and quantitative precision and bias in methods, protocols, and results from both laboratories. These procedures used Ecology or EPA's protocols to ensure that the results met the measurement quality objectives (Lubliner et al., 2008).

The data verification process includes checking that:

1. Holding times, blanks, instrument calibration, laboratory control sample analyses, and appropriateness of data qualifiers assigned were acceptable and appropriate.
2. Calibrations, checks on quality control (QC), and intermediate calculations were performed for all samples.
3. Data were consistent, correct, and complete, with no errors or omissions.
4. Targets for laboratory reporting limits were met.

Laboratory, field, and data management controls met expectations set forth in the QA Project Plan for this project (Lubliner et al., 2008). A complete QA discussion, case narratives, and performance data are provided in Appendix D of this report.

Quality Control

All samples were sent to MEL and Axys in coolers at 4ºC. Coolers and samples arrived intact by August 21, 2008. Preparation, storage, and handling were deemed acceptable by each laboratory. Sample analyte concentrations in blanks were not subtracted from sample results. A summary of codes used to qualify the data in this report is shown in Table 8.

Table 8. Data qualifier codes.

Code	Description
D	The sample was diluted. The reported value is dilution corrected.
U	The analyte was not detected at or above the reported result
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in the sample.
NJ	There is evidence that the analyte is present. The associated numerical result is an estimate.

All data are available in Ecology's EIM database available on the Internet at www.ecy.wa.gov/eim/. Search the User Study ID, BRWA0005.

Laboratory

PPCPs (Method 1694) and Hormones/Steroids (Method 1698)

The new methods for PPCPs[1694] and hormones/steroids from wastewater samples use a suite of analytical controls to ensure precision by instrument calibration, linearity checks, ongoing precision and recovery (OPR), and surrogates or spiked labeled analogs in the samples. Full details on initial calibration, continuing calibration, OPRs, and matrix spikes are provided in the case narratives (Appendix D). Many of the slight variations in the QC data were not deemed by Axys to have a significant effect on the data.

Qualification flags on data are common for low-level analyses. The EPA independent review of the data from Methods 1694 and 1698 (also in Appendix D) considered the data to be of high quality and acceptable for all purposes. The number and percent of the data qualified with the "J" flag is presented in Table 9. Data with "J" flags deemed as positively identified analytes are used in discussing the results for this study.

Organics - Semi-Volatile Organics

For all parameters, the calibrations, recoveries, and ongoing precision were performed in accordance with the appropriate method. Laboratory control samples, method blanks, standards/labeled compounds, and laboratory duplicates for this study are acceptable. Data are accepted with the appropriate qualifications, and the data are considered usable for making calculations, determinations, and decisions for which the project was conducted.

Nutrients/Solids

The August 19, 2008 Hayden secondary effluent and Hayden Water Research Facility samples for orthophosphate and total phosphorus data were rejected due to field and laboratory error. The total phosphorus and orthophosphate data from November 19, 2008 were used for this report.

Qualified Data

Data qualified by the "J" flag ranged from 20% to 96%. Method 1694 for PPCPs accounted for the least number of qualifications. It is important to note that the values of 96% and 90% were for biosolids data with high concentrations. Therefore the bulk of the qualifications were below 60%. The EPA study recently published, that spurred the development of Methods 1694 and 1698, indicated that 46% of the PPCPs[1694] data and 42% of the hormones and steroid data were qualified in their study (EPA, 2009b). These new EPA methods performed better in this 2008 study, and may be a reflection of a honing of the methods by Axys. Axys was contracted for both studies.

Analyses for caffeine and triclosan were performed by both Method 1694 and 8270. Results from Method 1694 were deemed more appropriate by MEL staff and are included in this report. The reasoning was that Method 1694 is an isotopic dilution method and has inherently more QA.

Table 9. The number and percent of detected and "J"-flagged data by method and sample type.

Parameter	Influents	Effluents/ Discharges	Biosolids
Method 1694 - PPCPs[1694]			
Number Detected	174	166	88
Percent Detected of Total	48%	33%	40%
Number "J" Flagged	42	38	18
Percent "J" Flagged of Detected	24%	23%	20%
Method 1698 - Hormones/Steroids			
Number Detected	87	57	49
Percent Detected of Total	64%	31%	22%
Number "J" Flagged	51	18	47
Percent "J" Flagged of Detected	60%	32%	96%
Method 8270d - Semi-Volatile Organics			
Number Detected	71	54	49
Percent Detected of Total	19%	10%	22%
Number "J" Flagged	27	34	44
Percent "J" Flagged of Detected	38%	63%	90%

Field Replicates

Field replicates were taken side-by-side from all Budd Inlet WWTP samples (influent, effluents, and biosolids). Replicates provide estimates of field and laboratory variability. Variability can be expressed as the relative percent difference (RPD) between a sample and its duplicate. The complete set of replicate data is attached in Appendix D. Field replicate RPDs for water samples were below 15% for nutrients and 40% for organics, with only two exceptions in the tertiary effluent samples for ammonia and benzoic acid. These exceptions are due to the difference between very small numbers. Biosolids RPDs for organics were below 20%. For the remainder of this document, the calculated mean of the original sample and replicate sample value is used.

Field Transfer Blank

A field transfer blank was analyzed to detect contamination arising from sample containers or sample handling. The blank was prepared by transferring organic-free water supplied by Axys from one bottle to another in the field, which mimicked the grab sampling procedure. A field transfer blank was poured onsite at the Budd Inlet WWTP.

The field transfer blank had very little contamination, with only three analytes (bisphenol A, phenol, and naproxen) detected above the laboratory reporting limit. Data affected by this contamination is limited to three "J" qualified data points, including bisphenol A in the Chambers Creek effluent. The field transfer blank values were generally lower than the laboratory method blank which indicates there is little concern of field contamination in the sample.

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Results

Wastewater Treatment Plant Conditions

Discharge

The WWTPs provided wastewater flow rates for August 19, 2008 when wastewater samples were collected. The operating conditions of the plant during the day of sampling, as previously discussed, were considered to be normal. As a means for comparing the WWTPs' operating conditions, the computed parameter of SRT is discussed.

Solids Retention Time (SRT)

The SRT for each WWTP sampled in this study is listed in Table 10. These SRT values were provided by the facility operators and follow the simplified SRT Equation 1, as described by the *Water Environment Federation Manual, Operation of Municipal Wastewater Treatment Plants.*

Equation 1. Solids Retention Time (d) = $$\frac{\textbf{\textit{Secondary system SS (lb)}}}{\left(\textbf{\textit{WASS}}\ (\frac{lb}{d}) + \textbf{\textit{Effluent SS}}\ (\frac{lb}{d})\right)}$$

Where:

SS = suspended solids
WASS = Waste activated suspended solids

Table 10. Solids retention time (SRT) of the WWTPs included in this study.

	WWTP	Treatment	SRT
1	Budd Inlet	EBNR	18.6 days
	Budd Inlet RWP [a]	EBNR+F	
2	Martin Way RWP	EBNR+MF	25 days
3	Chambers Creek	AS	2.5 days
4	Puyallup	AS+N	18 days
5	Hayden	AD	13 days
	Hayden WRF [a]	CA+F	

[a] = Further treats a portion of the effluent from the secondary process.

At the time of sampling, SRTs ranged from 2.5 to 25 days. The LOTT (Budd Inlet and Martin Way) facilities had the longest SRTs, and Chambers Creek had the shortest.

Nutrients in Influent and Effluent

Table 11 shows the concentrations of total suspended solids (TSS) and nutrient parameters from each WWTP. Organic nitrogen, organic phosphorus, and total inorganic nitrogen (TIN) are calculated by the laboratory based on individual nutrient values. The equations are shown in the header of Table 11.

Sampling for nutrients was not conducted at the Martin Way RWP when the organic samples were collected. Instead, values were obtained from LOTT's August 2008 Discharge Monitoring Report.

Table 11. Solids and nutrient results for all wastewater samples, mg/L.

	WWTP	TSS	Ortho-phosphate (OP)	Total Phosphorus (TP)	Organic Phosphorus (TP-OP)	Ammonia (NH_4)	Nitrite-Nitrate (NO_3-NO_4)	Total Persulfate Nitrogen (TPN)	Organic Nitrogen (TPN-NH_4)-(NO_3-NO_4)	Total Inorganic Nitrogen (NO_3+NO_2 +NH_4)
1	Budd Inlet WWTP Influent (mean)	258	4.87	7.00	2.13	34.6	0.1	40.5	5.8	34.7
	Budd Inlet WWTP Secondary Effluent (mean)	5	3.44	3.24	< 0.1	0.04	1.2	2	0.8	1.2
	Budd Inlet RWP Discharge (mean)	1	3.42	2.85	< 0.1	0.01	1.4	2.1	0.7	1.4
2	Martin Way RWP Influent	371[a]	--	--	--	--	--	55[b]	--	--
	Martin Way RWP Discharge	2U[a]	--	--	--	--	--	3.4[b]	--	--
3	Chambers Creek Influent	183	11.3	13.7	2.40	61.6	0.05	64.3	2.7	61.7
	Chambers Creek Effluent	5	1.53	1.56	0.03	41.1	4.1	37.6	< 0.1	45.2
4	Puyallup Influent	240	3.79	6.92	3.13	29.5	0.03	34.6	5.1	29.5
	Puyallup Effluent	2	3.25	2.79	< 0.1	5.17[c]	5.0	11.9	1.7	10.2
5	Hayden WRF Influent	202	7.04	5.57	< 0.1	47.8	0.02	48.2	0.4	47.8
	Hayden WRF Secondary Effluent	2	4.27[d]	4.22[d]	< 0.1[d]	5.87	2.3	8.6	< 0.1	8.2
	Hayden WRF Tertiary Effluent	1 U	0.004[d]	0.02[d]	0.016[d]	5.93	2.3	9.3	< 0.1	8.2

Results are from a single sample.
-- No data.
TSS = total suspended solids.
U = non-detect at the given laboratory reporting limit.
[a] Daily value from the LOTT Discharge Monitoring Report for the Martin Way RWP.
[b] Monthly mean for total nitrogen from the LOTT Discharge Monitoring Report for the Martin Way RWP.
[c] Routine monitoring by facility operators typically measures ammonia concentrations to be approximately 0.5 mg/L.
[d] Results from resample date: November 19, 2008.
< 0.1 = Reported values for organic phosphorus or organic nitrogen that are calculated as negative numbers.

Influent

In general, influent concentrations of total phosphorus and total nitrogen in raw municipal wastewater range from 6 to 8 mg/L and 40 to 60 mg/L, respectively. It is not uncommon for the nutrient concentrations to fluctuate from these ranges occasionally or seasonally. Nitrogen in the raw municipal influent is predominantly in the form of ammonia. During the biological process of activated sludge, some ammonia-nitrogen is consumed and a variable portion may also be converted to nitrate-nitrogen. About 60% of the phosphorus in raw municipal influent is typically removed during most secondary treatment processes, yielding an average effluent concentration of 2 mg/L in the final effluent.

Influent nutrient concentrations sampled in this study are considered typical for WWTPs, with the exception of the elevated phosphorus concentration (13.7 mg/L) at the Chambers Creek WWTP. This is twice the typical concentration measured in raw domestic wastewater. Despite the higher influent concentration, the Chambers Creek effluent concentration (1.56 mg/L) is within the average range for a secondary treatment process.

Effluent

Nutrient concentrations in the discharges of the three state-of-the-science facilities (LOTT Budd Inlet, LOTT Martin Way, and Hayden Water Research Facility) were very low. The Puyallup WWTP demonstrated a greater nutrient reduction capability, due to the anoxic and aerobic zones, than the typical secondary process. The Hayden aeration ditch and the Chambers Creek activated sludge processes produced typical secondary effluent concentrations for nutrients.

The Hayden Water Research Facility provides tertiary treatment to remove phosphorus from a portion of the Hayden secondary effluent (up to about 1 MGD). The treatment at Hayden Water Research Facility consists of chemical addition (ferric chloride or ferric sulfate) and filtration through a two-stage, continuous-backwashing, up-flow sand filter. The long-term average total phosphorus concentrations produced through Hayden Water Research Facility are reported as routinely less than 0.02 mg/L, which is the same total phosphorus concentration measured in this study. This performance represents state-of-the-science treatment for removal of phosphorus from municipal wastewater.

Treatment for reclaimed water is employed at the Budd Inlet RWP and Martin Way RWP. Although the Reclaimed Water Law establishes a permit limitation of 10 mg/L total nitrogen in the effluent, the Budd Inlet RWP and Martin Way RWP total nitrogen levels are routinely much lower. Reclaimed water concentrations of 2 to 3 mg/L total inorganic nitrogen are considered to represent the current state-of-the-science treatment for nitrogen removal.

PPCPs, Hormones, Steroids, and Semi-Volatile Organics

Non-Detected Chemicals

Table 12 lists 83 of the total 172 targeted PPCPs[1694], hormones/steroids, and semi-volatile organics that were not detected in any samples. Most of the non-detected compounds were semi-volatile organic compounds which were not the main focus of the study. The lack of detection for the compounds is likely a result of multiple factors. These may include, but are not limited to, the chemical nature of each compound, very low concentrations in the samples, high interfering matrices, or an absence of these compounds in the wastewater.

Table 12. Lists of the 83 chemicals not detected, by laboratory method.

Method 1694 for PPCPs: 22 of 72 analytes

Clinafloxacin	Cloxacillin
Digoxin	Digoxigenin
Flumequine	Lincomycin
Ormetoprim	Oxacillin
Penicillin G	Sarafloxacin
Sulfachloropyridazine	Sulfamethizole
Sulfanilamide	Sulfathiazole
Tylosin	Virginiamycin
Anhydrochlortetracycline (ACTC)	Demeclocycline
4-Epianhydrochlortetracycline (EACTC)	4-Epichlortetracycline (ECTC)
4-Epioxytetracycline (EOTC)	Isochlortetracycline (ICTC)

Method 1698 for Hormones and Steroids: 5 of 27 analytes

17a-Ethinyl-Estradiol	17a-Dihydroequilin
Equilenin	Mestranol
Progesterone	

Method 8270d for Semi-Volatile Organics: 56 of 73 analytes

1,2,4-Trichlorobenzene	1,2-Diphenylhydrazine	1,3-Dichlorobenzene
1-Methylnaphthalene	2,2'-Oxybis[1-chloropropane]	2,4,5-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene	2,6-Dinitrotoluene	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol
2-Nitroaniline	2-Nitrophenol	3-Nitroaniline
4,6-Dinitro-2-Methylphenol	4-Bromophenyl-Phenylether	4-Chloro-3-Methylphenol
4-Chloroaniline	4-Chlorophenyl-Phenylether	4-Nitroaniline
4-Nitrophenol	Acenaphthene	Acenaphthylene
Anthracene	Benzo(a)anthracene	Benzo(a)pyrene
Benzo(b)fluoranthene	Benzo(ghi)perylene	Benzo(k)fluoranthene
Bis(2-Chloroethoxy)Methane	Carbazole	Chrysene
Dibenzo(a,h)anthracene	Dibenzofuran	Dimethylphthalate
Di-N-Octyl Phthalate	Fluoranthene	Fluorene
Hexachlorobenzene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexachloroethane	Indeno(1,2,3-cd)pyrene	Isophorone
Naphthalene	Nitrobenzene	N-Nitrosodimethylamine
N-Nitroso-Di-N-Propylamine	N-Nitrosodiphenylamine	Pentachlorophenol
Pyrene	Retene	

Detected Chemicals

The results of this study confirm that PPCPs can be routinely found in municipal wastewater. Table 13 lists concentration ranges for the 24 target analytes. All sample results are available in Appendix E.

Table 13. Concentration ranges of 24 target compounds in WWTP influents and effluents.

Chemical Class	Analyte	Wastewater Influent Concentrations (ng/L)	Secondary Effluent [A] Concentrations (ng/L)	Tertiary Effluent[B] or Reclaimed Water[C] Concentrations (ng/L)
PPCPs[1694]	Acetaminophen	182,000-233,000	nd	nd
	Caffeine	69,000-168,000	nd-747	nd
	Carbamazepine	536-1,330	608-785	917-1,600
	Cotinine	3,390-4,600	39-113	nd-40
	Diphenhydramine	1,360-3,810	255-924	nd-343
	Erythromycin	255-556	154-327	nd-168
	Fluoxetine	38-178	43-75	42-58
	Gemfibrozil	4,400-21,900	251-3,880	nd-1,230
	Ibuprofen	28,900-38,600	28-170	30-158
	Metformin	98,900-126,000	4,385-43,800	542-1,760
	Naproxen	25,100-53,600	19-340	nd-251
	Sulfamethoxazole	2,770-4,010	2-1830	2-104
	Tetracycline	13-186	10-40	nd
	Triclosan	1,480-2,770	nd-805	nd-77
	Triclocarban	289-541	31-78	3-103
	Trimethoprim	611-1,400	308-791	nd-294
H/S	17α-ethinyl-estradiol	nd-8	nd-2	nd
	17β-Estradiol	nd	nd-12	nd
	Coprostanol	1,910,000-2,760,000	1,170-28,200	7-148
SVOC	4-nonylphenol	nd-400	nd-200	nd
	Bis(2-ethylhexyl) phthalate (DEHP)	7800-33000	nd-1600	nd-28000
	Bis-phenol A	nd-44000	nd-1900	nd-6000
	Di-n-butylphthalate (DBP)	nd-3200	nd	nd-900
	tri(chloroethyl) phosphate (TCEP)	nd-3600	900-1000	900-1400

[A] = Results represent four WWTPs (EBNR, AS, AD, and AS+N).
[B] = Result represent one WWTP (CA+F).
[C] = Results represent two RWPs (EBNR+MF and EBNR+F).
PPCPs[1694] = pharmaceutical or personal care product compound, measured by EPA Method 1694.
H/S = hormone or steroid, measured by EPA Method 1698.
SVOC = semi-volatile organic compound, measured by EPA Method 8270d.
nd = not detected. Laboratory reporting limits for non-detects are listed in Appendix E.

All 24 target analytes were detected in at least one site, with the exception of 17β-estradiol in the influent samples. 17α-ethinyl-estradiol is a synthetic estrogen, and 17β-estradiol is the natural form made by male and female mammals. Both estradiols are readily bio-transformed by standard secondary WWTPs (Servos et al., 2005). The lack of incoming estradiol concentrations is not well understood and may be due to conditions or biodegradation in the pipeline or sampling containers.

Analytes found to have the highest concentrations in at least one secondary or tertiary effluent, and not already listed in Table 13, are listed in Table 14.

Table 14. Additional analytes detected at relatively high concentrations.

Chemical Class	Analyte	Secondary Effluent [A] Concentrations (ng/L)	Tertiary Effluent[B] or Reclaimed Water[C] Concentrations (ng/L)
PPCPs[1694]	Azithromycin	150-700	10-380
	Cimetidine	140-610	nd-310
	Ofloxacin	110-640	nd
	Ranitidine	280-1630	nd-740
H/S	b-Sitosterol	nd-6110	nd
	Campesterol	280-2050	nd-4
	Cholestanol	600-1890	nd-50
	Cholesterol	3250-28200	nd
	Ergosterol	170-2680	nd-120
	Estrone	nd-1000	nd-39.2
	Stigmasterol	1320-25700	nd
SVOC	4-Methylphenol	nd-320	nd-26000
	Diethylphthalate	nd	nd-5200
	Phenol	nd-1600	nd-24000

[A] = Results represent four WWTP codes (EBNR, AS, AD, and AS+N).
[B] = Result represent one WWTP code (CA+F).
[C] = Results represent two RWP codes (EBNR+MF and EBNR+F).
H/S = hormone or steroid, measured by EPA Method 1698.
SVOC = semi-volatile organic compound, measured by EPA Method 8270d.
nd = not detected.

These compounds were not included in the list of targeted analytes due to a lack of literature references at the onset of this 2008 study. These analytes were found in relatively large concentrations in the secondary or tertiary effluents. These analytes should be considered for future studies if references indicate they are toxic, persistent, or bioaccumulative.

Overall, the concentrations of PPCPs detected in the effluents are similar to, or are above, concentrations observed in other studies. Secondary effluent concentrations which were higher in this study include carbamazepine, gemfibrozil, sulfamethoxazole, triclosan, trimethoprim, and TCEP. Tertiary effluent concentrations which were higher in this study include carbamazepine, erythromycin, fluoxetine, ibuprofen, triclosan, trimethoprim, and TCEP.

Reasons for greater effluent concentrations were not explored but may be attributed to the use of more sensitive methods by this study, regional consumption patterns, the differences in population size, the increased use of a particular PPCP over time, or the WWTP technologies sampled in this study.

Table 15 compares effluent concentrations of the 24 target analytes from this 2008 study to values reported in other studies.

Table 15. Comparison of 24 analytes with literature values, ng/L (pptr).

Analyte	Secondary Effluent[A] Concentrations in This Study	Secondary Effluent Literature Values[1,2]	Tertiary Effluent[B] or Reclaimed Water[C] Concentrations in This Study	Tertiary Effluent Literature Values[1]
Acetaminophen	nd	nd - <20	nd	2.5
Caffeine	nd-747	<20 - 51	nd	<10
Carbamazepine	608-785	nd - 272	917-1,600	19
Cotinine	39-113	--	nd-40	--
Diphenhydramine	255-924	--	nd-343	--
Erythromycin	154-327	133 - 336	nd-168	<1.0
Fluoxetine	43-75	18 - 24	42-58	8.5
Gemfibrozil	251-3,880	nd - 24	nd-1,230	<1.0
Ibuprofen	28-170	19	30-158	6.0
Metformin	4,385-43,800	--	542-1,760	--
Naproxen	19-340	<20 - 25	nd-251	<1.0
Sulfamethoxazole	2-1830	90 - 841	2-104	<1.0
Tetracycline	10-40	--	nd	--
Triclosan	nd-805	29 - 85	nd-77	1.2
Triclocarban	31-78	--	3-103	--
Trimethoprim	308-791	35 - 186	nd-294	<1.0
17α-ethinyl-estradiol	nd	nd	nd	--
17β-Estradiol	nd-12	nd	nd	<1.0
Coprostanol	1,170-28,200	--	7-148	--
4-nonylphenol	nd-200	--	nd	--
Bis(2-ethylhexyl) phthalate (DEHP)	nd-1600	--	nd-28000	--
Bis-phenol A	nd-1900	23	nd-600	--
Di-n-butylphthalate (DBP)	nd	--	nd-900	--
Tri(chloroethyl) phosphate (TCEP)	900-1000	189 - 373	900-1400	133

[A] = Results represent four WWTP codes (EBNR, AS, AD, and AS+N).
[B] = Result represent one WWTP code (CA+F).
[C] = Results represent two RWP codes (EBNR+MF and EBNR+F).
[1]Snyder et al., 2007.
[2]Drury et al., 2006; or Heberer et al., 2004.
nd = not detected. Laboratory reporting limits for non-detects are listed in Appendix E.
-- = not found in the literature.

Biosolids Data

Biosolids samples were collected from three of the five WWTPs: LOTT Budd Inlet, Chambers Creek, and Puyallup. Table 16 lists analyte concentrations for the 24 target analytes and a few additional analytes present in relatively high concentrations. Previously mentioned biosolids data (Kinney et al., 2006b and 2008) are compared to this study's results in Table 16.

EPA conducted a national biosolids quality survey, sampling at 74 WWTPs for 145 contaminants, including 72 pharmaceuticals and 25 hormones and steroids (EPA, 2009c). The goal of the study was to characterize the mean concentration levels and develop statistically sound national estimates for selected contaminants. EPA (2009c) "national estimates" represent analyte concentrations from WWTPs nationwide. EPA national estimates are compared in Table 16 to the biosolids results of this 2008 monitoring study.

Kinney et al. (2006b and 2008) did not use the same methods as this 2008 study; however, the results are comparable. The EPA (2009c) reference does not report on the methods used; however, the assumption in presenting the data here is that the EPA results were analyzed by Methods 1694 and 1698, which are directly comparable with the current study. With such a lack of data on biosolids concentrations, these "rough" agreements are considered adequate to gain a sense of occurrence and relative concentrations of PPCPs in biosolids.

Kinney et al. (2006b) noted in his survey that, regardless of production method, demographics, or size, the most common organic compound concentrations did not vary greatly. This appears to be the case for this current 2008 study as well. The biosolids data presented in Table 16 come from three WWTPs each with different treatment processes and levels of treatment; however, the results are roughly similar. Particularly, the magnitude of the compounds concentrations are comparable across the three WWTPs sampled, and to available literature values. There does not appear to be a consistent pattern for those analytes with the greatest differences in concentration.

Table 16. Selected biosolids analyte concentrations and summary statistics, ug/Kg dw (ppb).

Analyte	Chambers Creek[1]	LOTT Budd Inlet[1]	Puyallup[1]	This 2008 study	Kinney et al., 2008	Kinney et al., 2006b	EPA, 2009c.
	µg/kg (dw)	µg/kg (dw)	µg/kg (dw)	mean µg/kg (dw)	mean µg/kg (dw)	median µg/kg (OC)	mean µg/kg
PPCPs[1694]							
Carbamazepine	265	358	376	333	390	68	135
Ciprofloxacin	10,800	12,350	11,000	11,380	--	--	10,501
Diphenhydramine	2,190	2,525	2,340	2,352	7000	340	871
Erythromycin-H2O	15	11	8	11	--	--	36
Fluoxetine	522	653	459	545		370	245
Gemfibrozil	211	250	14500	4990	--	--	--
Ibuprofen	458	438	499	465	--	--	--
Metformin	116	error*	nd	58	--	--	--
Miconazole	1,560	1,595	1,710	1,622	--	--	1,239
Naproxen	nd	10	nd	3	--	--	--
Ofloxacin	6,830	5,785	6,090	6,235	--	--	8,573
Sulfamethoxazole	nd	1	1	1	--	--	--
Tetracycline (TC)	3,200	3,290	1,220	2,570	--	--	1,278
Triclocarban	12,900	17,700	nd	10,200	--	--	39,433
Triclosan	36,600	7,985	19,800	21,462	10,500	10,200	16,097
Hormones/ Steroids							
Coprostanol	4,030,000	3,730,000	1,620,000	3,127,000	--	--	4,366,714
Epicoprostanol	3,280,000	2,630,000	985,000	2,298,000	--	--	1,702,708
Norgestrel	195	1,900	500	865	--	--	--
Stigmastanol	240,000	170,500	110,000	173,500	--	17,400	321,199
Semi-Volatile Organics							
Benzoic Acid	nd	8335	13,400	7,245	--	--	--
Bis(2-Ethylhexyl) Phthalate	nd	15,950	43,900	19,950	--	--	--
Bisphenol A	6,850	58,700	32,100	32,550	4,600	4,690	
Indeno(1,2,3-cd)pyrene	2,450	678	nd	1,043	--	--	--
Phenol	24,200	2,745	5,890	10,945	6,270	2,180	
Tri(2-chloroethyl) phosphate	1,480	nd	nd	493	--	--	--
Triethyl citrate	4,800	293	6,330	3,808	--	--	--

[1] Results based on single sample for this study.
nd = not detected, laboratory reporting limit for non-detects available in Appendix E.
* Result rejected based on analytical error.
(OC) = organic carbon normalized.
Dw = dry weight.

Nineteen analytes, shown in Table 17, were detected only in biosolids samples. It is believed that the concentrations were not high enough to detect in the wastewater using current analytical technology.

Table 17. Analytes detected only in the biosolids samples by all three methods.

PPCPs[1694]	Hormones/steroids	Semi-volatile organics (all are PAHs)
Enrofloxacin	Androstenedione	Anthracene
Lomefloxacin	b-Estradiol 3-benzoate	Benzo(a)anthracene
Norfloxacin	Mestranol	Benzo(a)pyrene
Anhydrotetracycline (ATC)		Benzo(b)fluoranthene
Chlortetracycline (CTC)		Benzo(k)fluoranthene
Minocycline		Bis(2-chloroethyl)ether
Oxytetracycline (OTC)		Chrysene
		Indeno(1,2,3-cd)pyrene
		Pyrene

The semi-volatile organics list of compounds in Table 17 is comprised of only polycyclic aromatic hydrocarbons (PAHs). This is not surprising given PAHs have low water solubility and larger log K_{ow}. In other words, PAHs have an affinity for soil, sediment, or organic matter.

Discussion of Contaminant Reduction

Nutrients

Table 18 shows the percent reduction of nutrients achieved by each WWTP. These results were calculated from values reported in Table 11 using the following equation:

Equation 2: Percent Reduction $\left[\frac{(influent\ concentration - effluent\ concentration)}{influent\ concentration}\right] \times 100\%$

Table 18. Nutrient-removal efficiency by WWTP and treatment level.

	Wastewater Plant	Treatment Code	Total Nitrogen Removal	TIN[1] Removal	Organic Phosphorus Removal	Total Phosphorus Removal[2]	TSS Removal
1	Budd Inlet	EBNR	95%	96%	99%	54%	98%
	Budd Inlet RWP	EBNR+F	95%	96%	99%	59%	99%
2	Martin Way RWP	(Nutrient samples were not collected)					
3	Chambers Creek	AS	42%	27%	99%	89%	97%
4	Puyallup	AS+N	66%	66%	99%	60%	99%
5	Hayden	AD	82%	83%	--	24%	99%
	Hayden WRF	CA+F	81%	83%	99%	99%	99%

[1] Total Inorganic Nitrogen (TIN) = (NO3+NO2)+NH4.
[2]The total phosphorus values for the Hayden tertiary treatment are based on resample data.

The Martin Way RWP nutrient samples were not collected due to oversight. The WWTPs sampled were well operated at the time of sampling based on the nutrient treatment efficiencies.

The Budd Inlet WWTP achieved the highest removal of total nitrogen and TIN during the secondary treatment process. Organic phosphorus removal was consistently high across the treatment technologies. The Hayden Water Research Facility achieved the highest total phosphorus removal efficiency using their proprietary tertiary treatment process. The Chambers Creek WWTP had only a 27% decrease in TIN, but removed 89% of the total phosphorus. All WWTPs removed more than 97% of the TSS from the influent.

PPCPs, Hormones, Steroids, and Semi-Volatile Organic Compounds

Analytes with the highest concentrations were primarily those on the list of the 24 target analytes. The compounds of a relatively high concentration are not necessarily the compounds that pose the largest potential threat to the environment. Assessing threat or risk to Puget Sound is beyond the scope of this study. However, the analytes detected in multiple effluents and their respective concentration ranges may be useful to others who are assessing risk.

The focus of this 2008 screening study was to gain an overall understanding of occurrence and reductions of PPCPs by different treatment technologies.

Tables 19-21 were created to separate the tertiary treatment technologies from the secondary technologies. The columns represent the sampled wastewater treatment technologies and biosolids. The secondary treatment column reflects the four secondary treatment effluents sampled: EBNR, AS, AS+N, and AD. The numerical concentration or laboratory reporting limit for every sample is available in Appendix E.

Tables 19-21 visually present the occurrence of an analyte by displaying the name when detected. Italicized (blue) text was used if the analyte was detected in only one of the five possible WWTPs or one of the three WWTPs where biosolids were collected. For example, in Table 19, of the 72 PPCPs[1694] analytes, 46 were found in at least one influent sample.

If the name of a compound is absent, that analyte was not detected.

Several large-scale patterns were observed in Table 19.

1. Twelve analytes were removed by all secondary treatment technologies and *were not* present in the biosolids (e.g., acetaminophen).
2. Eight analytes were removed by all secondary treatment technologies and *were* present in the biosolids (e.g., 17a-Estradiol).
3. Eight analytes were present in the secondary effluent, but were removed by at least one of the tertiary technologies, and *were not* present in the biosolids (e.g., albuterol).
4. Thirty-one analytes were present in the secondary effluent, but were removed by at least one of the tertiary technologies, and *were* detected in the biosolids (e.g., ciprofloxacin, triclosan).

The tertiary effluent from the EBNR+F process had the fewest PPCPs[1694] and semi-volatile compounds detected. In general, it appears that most analytes were removed by secondary treatment, and even more analytes were removed by the combination of advanced (tertiary) nutrient reduction and filtration treatments.

Table 19. PPCPs[1694] presence/absence data by wastewater treatment technology.

Influents (5 WWTPs Sampled)	Secondary Treatment (4 WWTPs Sampled)	Biological Nutrient Removal and Tertiary Treatment			Biosolids (3 WWTPs Sampled)
At least 3 of 5 *Italics = 1 of 5*	At least 3 of 4	CA+F	EBNR+MF	EBNR+F	At least 2 of 3 *Italics = 1 of 3*
Acetaminophen	--	--	--	--	--
Albuterol	Albuterol	Albuterol	--	--	--
Azithromycin	Azithromycin	Azithromycin	--	Azithromycin	Azithromycin
Caffeine	--	--	--	--	--
--	Carbadox	--	Carbadox	--	--
Carbamazepine	Carbamazepine	Carbamazepine	Carbamazepine	Carbamazepine	Carbamazepine
Ciprofloxacin	Ciprofloxacin	--	--	--	Ciprofloxacin
Cimetidine	Cimetidine	Cimetidine	--	--	Cimetidine
Clarithromycin	Clarithromycin	Clarithromycin	--	--	Clarithromycin
Codeine	Codeine	Codeine	--	--	*Codeine*
Cotinine	Cotinine	Cotinine	Cotinine	--	Cotinine
Dehydronifedipine	Dehydronifedipine	Dehydronifedipine	Dehydronifedipine	--	--
Diphenhydramine	Diphenhydramine	Diphenhydramine	Diphenhydramine	--	Diphenhydramine
Diltiazem	Diltiazem	Diltiazem	--	--	Diltiazem
Digoxigenin (1/5)	--	--	--	--	--
Doxycycline	Doxycycline	--	--	--	Doxycycline
--	--	--	--	--	Enrofloxacin
Erythromycin-H2O	Erythromycin-H2O	Erythromycin-H2O	--	Erythromycin-H2O	Erythromycin-H2O
Fluoxetine	Fluoxetine	Fluoxetine	Fluoxetine	Fluoxetine	Fluoxetine
Gemfibrozil	Gemfibrozil	Gemfibrozil	Gemfibrozil	--	Gemfibrozil
Ibuprofen	Ibuprofen	Ibuprofen	Ibuprofen	Ibuprofen	Ibuprofen
--	--	--	--	--	*Lomefloxacin*
Metformin	Metformin	(Rejected sample)	Metformin	Metformin	*Metformin*
Miconazole	--	--	--	--	Miconazole
--	--	--	--	--	Norfloxacin
Norgestimate (1/5)	--	--	--	--	--
Ofloxacin	Ofloxacin	Ofloxacin	--	--	Ofloxacin
--	--	--	--	Oxolinic Acid	Oxolinic Acid
--	--	--	--	Penicillin G	--
Penicillin V	--	--	--	--	--
Ranitidine	Ranitidine	Ranitidine		--	Ranitidine
Sulfadiazine	--	--	--	--	--
Sulfadimethoxine (1/5)	--	--	--	--	--
Sulfamerazine	--	--	--	--	--
Sulfamethazine (1/5)	--	--	--	--	--
Sulfamethoxazole	Sulfamethoxazole	Sulfamethoxazole	Sulfamethoxazole	Sulfamethoxazole	*Sulfamethoxazole*
Thiabendazole	Thiabendazole	Thiabendazole	Thiabendazole	Thiabendazole	Thiabendazole
Trimethoprim	Trimethoprim	Trimethoprim	--	Trimethoprim	--
1,7-Dimethylxanthine	--	--	--	--	--
Naproxen	Naproxen	Naproxen	--	Naproxen	*Naproxen*
Triclocarban	Triclocarban	Triclocarban	Triclocarban	--	Triclocarban
Triclosan	--	Triclosan	--	--	Triclosan
Warfarin	Warfarin	Warfarin	--	--	*Warfarin*
Tetracycline (TC)	Tetracycline (TC)	--	--	--	Tetracycline (TC)
--	--	--	--	--	ATC
--	--	--	--	--	*CTC*
EATC (1/5)	--	EATC	--	--	EATC
ETC	ETC	--	--	--	ETC
--	--	--	--	--	Minocycline
--	--	--	--	--	Oxytetracycline

ATC= Anhydrotetracycline. EATC= 4-Epianhydrotetracycline.
CTC = Chlortetracycline. ETC= 4-Epitetracycline.
See Appendix F for definitions of treatment codes: CA+F, EBNR+MF, and EBNR+F.

Table 20. Hormones and steroids presence/absence data by level of treatment.

Influents (5 WWTPs Sampled)	Secondary Treatment (4 WWTPs Sampled)	Biological Nutrient Removal and Tertiary Treatment			Biosolids (3 WWTPs Sampled)
At least 3 of 5 *Italics = # of 5*	At least 3 of 4	CA+F	EBNR+MF	EBNR+F	At least 2 of 3 *Italics = 1 of 3*
17a-Dihydroequilin (1/5)	--	--	--	--	--
17a-Estradiol	--	--	--	--	*17a-Estradiol*
--	--	--	--	--	Androstenedione
Androsterone	--	--	--	--	Androsterone
--	--	--	--	--	*b-Estradiol 3-benzoate*
b-Sitosterol	b-Sitosterol	--	--	--	b-Sitosterol
b-Stigmastanol	b-Stigmastanol	--	--	--	b-Stigmastanol
Campesterol	Campesterol	--	--	Campesterol	Campesterol
Cholestanol	Cholestanol	--	--	Cholestanol	Cholestanol
Cholesterol	Cholesterol	--	--		Cholesterol
Coprostanol	Coprostanol	Coprostanol	Coprostanol	Coprostanol	Coprostanol
Desmosterol	Desmosterol	--	Desmosterol	Desmosterol	Desmosterol
Desogestrel	--	--	--	--	*Desogestrel*
Epicoprostanol	Epicoprostanol	Epicoprostanol	--	Epicoprostanol	Epicoprostanol
Equilin (2/5)	--	--	--	--	*Equilin*
Ergosterol	Ergosterol	Ergosterol		Ergosterol	Ergosterol
Estriol	--	--	--	--	*Estriol*
Estrone	Estrone	Estrone	--	--	Estrone
--	--	--	--	--	*Mestranol*
Norethindrone (1/5)	--	--	--	--	Norethindrone
Norgestrel (2/5)	--	--	--	--	Norgestrel
Stigmasterol	Stigmasterol	--	--	--	Stigmasterol
Testosterone	--	--	--	--	--

See Appendix F for definitions of treatment codes: CA+F, EBNR+MF, and EBNR+F.

Table 21. Semi-volatile organics presence/absence data by level of treatment.

Influents (5 WWTPs Sampled)	Secondary Treatment (4 WWTPs Sampled)	Biological Nutrient Removal and Tertiary Treatment			Biosolids (3 WWTPs Sampled)
At least 3 of 5 *Italics = # of 5*	At least 3 of 4 *Italics = # of 4*	CA+F	EBNR+MF	EBNR+F	At least 2 of 3 *Italics = 1 of 3*
1,4-Dichlorobenzene	--	--	1,4-Dichlorobenzene	--	1,4-Dichlorobenzene
--	2,4,6-Trichlorophenol	--	--	--	--
4-Methylphenol	4-Methylphenol	--	4-Methylphenol	4-Methylphenol	4-Methylphenol
--	--	--	--	--	*Anthracene*
--	--	--	--	--	Benzo(a)anthracene
--	--	--	--	--	Benzo(a)pyrene
--	--	--	--	--	Benzo(b)fluoranthene
--	--	--	--	--	*Benzo(k)fluoranthene*
Benzoic Acid	Benzoic Acid	--	--	Benzoic Acid	Benzoic Acid
Benzyl Alcohol	--	--	Benzyl Alcohol	--	--
--	--	--	--	--	*Bis(2-Chloroethyl)Ether*
BEP	*BEP (1)*	--	BEP	--	BEP
Bisphenol A	Bisphenol A	--	Bisphenol A	--	Bisphenol A
Butylbenzylphthalate	*Butylbenzylphthalate (1)*	--	Butylbenzylphthalate	--	*Butylbenzylphthalate*
--	--	--	--	--	Chrysene
Diethylphthalate	--	--	Diethylphthalate	--	--
Di-N-Butylphthalate	--	--	Di-N-Butylphthalate	--	--
--	--	--	--	--	Indeno(1,2,3-cd)pyrene
Naphthalene	--	--	--	--	Naphthalene
--	--	--	Phenanthrene	--	Phenanthrene
Phenol	*Phenol (2)*	Phenol	Phenol	--	Phenol
--	--	--	--	--	Pyrene
--	*4-Nonylphenol (1)*	--	--	--	--
TCEP	TCEP	TCEP	TCEP	TCEP	*TCEP*
Triethyl citrate	Triethyl citrate	Triethyl citrate	Triethyl citrate	Triethyl citrate	Triethyl citrate

BEP = Bis(2-Ethylhexyl) Phthalate.
TCEP = Tri(2-chloroethyl) phosphate.
See Appendix F for definitions of treatment codes: CA+F, EBNR+MF, and EBNR+F.

Removal

Removal or treatment for the purposes of this report is defined as *a reduction in concentration to below the laboratory reporting limit for each analytical method.* The methods chosen for this project have very low limits, ranging roughly from parts per billion to parts per million, depending on the compound.

Influent concentrations of the following 12 analytes appeared to be treated in the water column by all treatment technologies and were not detected in the biosolids:

1,7-Dimethylxanthine	Caffeine
Acetaminophen	Penicillin V
Sulfadiazine	Sulfadimethoxine
Sulfamethazine	Sulfamerazine
Digoxigenin	Testosterone
Norgestimate	17a-Dihydroequilin

The following seven compounds were treated in the wastewater column but were detected in at least one of three biosolids samples:

Miconazole	Estriol
17a-Estradiol	Norethindrone
Desogestrel	Norgestrel
Equilin	Naphthalene

Other studies have found excellent removal for most of the analytes listed above, particularly caffeine, ibuprofen, and acetaminophen. Caffeine is often used by engineers to trace leaks in faulty sewage treatment plants. Effective treatment of caffeine by wastewater treatment was previously known; therefore, detections in the receiving water would indicate raw wastewater or stormwater sources.

Partial Removal

To assess the removal efficiency of each wastewater treatment technology, the reduction fraction (not percent reduction) was calculated for each sample using the following equation:

Equation 3: Reduction Fraction = $\left[\frac{(influent\ concentration - effluent\ concentration)}{influent\ concentration}\right]$

Tables 22 - 24 show the reduction fraction for PPCPs[1694], hormones/steroids, and semi-volatile organics at each WWTP. For example, the first entry in Table 22 is sulfamethazine, which was found only in the influent for the Puyallup WWTP; therefore, that is the only treatment showing a reduction fraction (in this case 1, which represents a greater than 99.5% reduction).

The data have been sorted to enhance the visual representation for reduction fractions. A double dash (--) indicates the analyte was not detected in either the influent or effluent.

Secondary treatment with nutrient reduction appears to result in higher rates of removal than secondary treatment alone. Enhanced biological nutrient removal and tertiary treatment appear to reduce concentrations of PPCPs and hormones/steroids to a higher degree than the secondary process with some nutrient removal. While each of the WWTPs reduced PPCPs to varying degrees, nearly all of the PPCPs measured in this study were below detectable concentrations in the tertiary filtration effluent of the Budd Inlet RWP (EBNR+MF).

The overall pattern in removal factors observed for PPCPs[1694] and hormones/steroids seemed to hold for the semi-volatile organic removal factors, with one exception. The EBNR+MF treatment had lower removal efficiencies for the semi-volatile organics than for the PPCPs and hormones/steroids.

There was an apparent increase in concentration from influent to effluent for 11 analytes, indicated by a negative removal rate (e.g., dehydronifedipine) in Tables 22-24. The mechanism for increasing concentrations through the wastewater treatment system is unclear and was not explored as part of this project. However, this phenomenon was noted by other researchers.

Oppenheimer and Stephenson (2006) explained the increase as desorption from the reactor biosolids. A second possibility is that metabolic breakdown of the parent compound would yield a higher concentration in degradates. For example, 4 EATC is a degradate of tetracycline. Microorganisms break down tetracycline, and therefore 4 EATC concentrations increase. This may explain why 4 EATC showed up in only the effluent. A third possibility is metabolite reconjugation back to the parent compound (Axys, personal communication, 2008).

Poor Removal

Several analytes had particularly low removals and may be appropriate tracers of wastewater through the environment. Carbamazepine, fluoxetine, and thiabendazole were detected in every sample and had some of the lowest removal factors across the different treatment types.

Removal efficiency of carbamazepine, fluoxetine, and thiabendazole was irregular, showing both positive and negative removal efficiencies. These compounds have been identified in other studies as being difficult to treat. Lubick (2009) found the anticonvulsant carbamazepine to be a good tracer in groundwater; however, low consumption rates may limit its usefulness. Chenxi et al. (2008) found carbamazepine, triclosan, and ciprofloxacin to be resistant to biological wastewater treatment. The chemical structures of these compounds do not appear to be susceptible to the biologically oxidative processes employed at WWTPs. In addition, Chenxi et al. (2008) found their concentrations to persist in biosolids during a 77-day biosolids storage experiment.

Table 22. PPCPs[1694] removal factors by wastewater treatment technology.

Method 1694: Analytes	Secondary Treatment		Secondary Treatment with Nutrient Removal		Enhanced Nutrient Removal and Tertiary Treatment		
	AS	AD	EBNR	AS+N	CA+F	EBNR+MF	EBNR+F
Sulfamethazine	--	--	--	1	--	--	--
Norgestimate	--	--	--	--	--	1	--
4-Epianhydrotetracycline (EATC)	--	--	--	--	--	1	--
Sulfadiazine	1	1	1	-0.10	1	--	1
Sulfadimethoxine	--	--	0.59	--	--	--	1
Sulfamerazine	1	1	1	--	1	--	1
Acetaminophen	1	1	1	1	1	1	1
Miconazole	1	1	1	1	1	1	1
Penicillin V	1	1	1		1	1	1
1,7-Dimethylxanthine	1	1	1	1	1	1	1
Caffeine	0.99	1	1	1	1	1	1
Ibuprofen	0.99	0.99	1	1	1	1	1
Naproxen	0.99	1	1	0.99	1	1	1
Cotinine	0.97	0.99	0.99	0.99	0.99	0.99	1
Ciprofloxacin	0.44	0.78	0.69	0.78	1	1	1
Ranitidine	0.65	0.73	0.83	0.93	0.77	1	1
Triclosan	0.60	1	0.93	1	0.95	1	1
Doxycycline	0.64	0.78	0.61	0.51	1	1	1
4-Epitetracycline (ETC)	0.43	0.55	0.57		1	1	1
Tetracycline (TC)	-2.03	0.78	0.62	0.57	1	1	1
Ofloxacin	-0.26	0.09	0.49	-0.21	1	1	1
Codeine	0.50	0.88	0.92	0.75	0.77	1	1
Triclocarban	0.86	0.83	0.90	0.85	0.83	0.80	1
Diltiazem	0.74	0.85	0.79	0.79	0.73	1	1
Clarithromycin	0.32	0.73	0.06	0.32	0.81	1	1
Erythromycin-H2O	0.09	0.40	0.38	0.56	0.34	1	1
Warfarin	0.20	0.09	0.09	0.27	0.05	1	1
Albuterol	0.17	-0.03	0.47	0.33	0.03	1	1
Cimetidine	-0.02	0.85	0.57	0.81	0.81	1	1
Diphenhydramine	0.71	0.91	0.85	0.79	0.88	1	1
Trimethoprim	0.44	0.50	0.52	0.68	0.52	1	1
Gemfibrozil	0.31	0.94	0.95	0.87	0.94	0.99	1
Metformin	0.64	--	0.96	0.65	--	0.99	1
Sulfamethoxazole	0.62	0.34	0.64	0.49	1	0.96	0.98
Azithromycin	-0.07	0.72	0.74	0.31	0.83	1	0.98
Dehydronifedipine	-0.74	-0.59	-0.09	--	-1.13	-0.13	1
Fluoxetine	-0.34	0.23	0.60	-0.15	0.14	0.09	0.76
Thiabendazole	-0.36	-0.59	-0.16	-0.50	-0.38	0.15	-0.04
Carbamazepine	0.51	-0.41	0.41	0.13	-0.71	0.29	-0.20

See Appendix F for definitions of treatment codes: AS, AD, EBNR, AS+N, CA+F, EBNR+MF, EBNR+F.

Table 23. Hormone and steroid removal factors by wastewater treatment technology.

Method 1698: Analytes	Secondary Treatment		Secondary Treatment with Nutrient Removal		Enhanced Nutrient Removal and Tertiary Treatment		
	AS	AD	EBNR	AS+N	CA+F	EBNR+MF	EBNR+F
b-Estradiol 3-benzoate	--	1	--	--	1	--	--
Norethindrone	--	--	--	--	--	1	--
Equilin	--	--	1	1	--	--	1
Norgestrel	--	--	1	1	--	--	1
Cholesterol	1	1	1	1	1	1	1
Estriol	1	1	1	1	1	1	1
Testosterone	1	1	1	1	1	1	1
Campesterol	1	1	1	1	1	1	1
Coprostanol	1	1	1	1	1	1	1
Androsterone	1	1	1	1	1	1	1
b-Sitosterol	1	1	1	1	1	1	1
b-Stigmastanol	0.98	1	1	1	1	1	1
Epicoprostanol	0.98	1	1	1	1	1	1
Cholestanol	0.98	1	0.97	1	1	1	1
17a-Estradiol	0.75	1	1	1	1	1	1
Desogestrel	0.77	1	1	1	1	1	1
Desmosterol	0.92	0.98	0.92	1	1	1	1
Ergosterol	0.91	0.84	0.89	1	1	1	1
Stigmasterol	0.92	0.76	0.89	0.98	1	1	1
Estrone	-8.43	0.66	1	0.90	0.45	1	1

See Appendix F for definitions of treatment codes: AS, AD, EBNR, AS+N, CA+F, EBNR+MF, EBNR+F.

Table 24. Semi-volatile organics removal factors by wastewater treatment technology.

Method 8270d: Analytes	Secondary Treatment		Secondary Treatment with Nutrient Removal		Enhanced Nutrient Removal and Tertiary Treatment		
	AS	AD	EBNR	AS+N	CA+F	EBNR+MF	EBNR+F
2,4-Dichlorophenol	1	--	--	--	--	--	--
Phenol, 4-Nonyl-	1	--	--	1	--	--	--
Naphthalene	1	--	1	--	--	--	1
Benzoic Acid	0.99	0.99	1	0.99	1	1	0.99
4-Methylphenol	0.99	1	1	1	1	0.72	1
Diethylphthalate	1	1	1	1	1	0.53	1
Benzyl Alcohol	1	1	1	1	1	0.50	1
Butylbenzylphthalate	1	1	1	1	1	0.78	1
Phenol	0.96	0.99	1	1	0.99	0.51	1
Bis(2-Ethylhexyl) Phthalate	0.95	1	1	1	1	0.15	1
Bisphenol A	-0.46	0.97	--	0.38	1	0.86	--
Tri(2-chloroethyl) phosphate	-0.22	0.72	--	-1.34	0.73	0.61	--
1,4-Dichlorobenzene	0.65	1	1	nd	1	0.13	1
Triethyl citrate	0.28	0.59	0.49	0	0.61	0.33	0.43

See Appendix F for definitions of treatment codes: AS, AD, EBNR, AS+N, CA+F, EBNR+MF, EBNR+F.

Categorical Summary

The 1-log (10^{-1} or 90%) removal threshold is a common statistic to rate wastewater treatment removal efficiencies. The number of analytes that achieved at least a 1-log reduction across all technologies studied (shown in Tables 22-24 with a reduction fraction of ≥ 0.9), are listed in Table 25.

Table 25. Analytes with at least a 1-log removal reduction fraction.

PPCPs[1694]	Hormones/Steroids	Semi-Volatile Organics
1,7-Dimethylxanthine Acetaminophen Caffeine Cotinine Ibuprofen Miconazole Naproxen	Androsterone b-Sitosterol b-Stigmastanol Campesterol Cholestanol Cholesterol Coprostanol Epicoprostanol Estriol Testosterone	Benzoic Acid

A categorical summary of removal efficiencies was developed based on the number of analytes that achieved an 80% concentration reduction from Tables 22-24. Three categories (high, moderate, and low) represent the performance of the treatment technologies (Table 26).

Table 26. Categorical removal efficiencies in wastewater effluent by treatment type.

Category	PPCPs[1694]	Hormones/ Steroids	Semi-volatile Organics
High = >80% of analytes had at least 80% reduction in concentration	EBNR+F * EBNR+MF	EBNR+F * EBNR+MF * CA+F * AS+N * EBNR AD AS	EBNR * EBNR+F * CA+F AD
Moderate = 60-80% of analytes had at least 80% reduction in concentration	CA+F	--	AS+N AS
Low = <60% of the analytes had at least 80% reduction in concentration	EBNR AS+N AS AD	--	EBNR+MF

* = The treatment technologies that produced a 1-log reduction for at least 80% of the detected influent analytes.

The enhanced biological nutrient removal plus tertiary filtration was the only treatment technology that achieved a 1-log removal for more than 80% of the analytes using each of the three analytical methods.

The largest PPCPs[1694] removal was achieved by the two WWTPs with the combination of enhanced biological nutrient removal and filtration. Hormones and steroids were reduced well by all technologies.

For semi-volatile organics, the high reduction technologies were slightly different. The aeration ditch achieved overall a higher removal rate than EBNR+MF, which is used to produce reclaimed water. Although semi-volatiles were not the focus of the study, this result is interesting and may be a result of the higher mixed liquor suspended solids maintained by the Martin Way RWP.

It is possible that the EBNR removal process associates more PPCPs with particulates, which makes the PPCPs more amenable to subsequent removal via filtration. Biological nutrient removal exposes wastewater to aerobic and anoxic zones in which different bacteria have an opportunity to consume nutrients and break down pollutants. The higher wastewater recycling rate required to operate a biological nutrient-removal system also results in a longer time of exposure to treatment.

Although this study did not evaluate the specific processes by which PPCP reduction is achieved within the respective treatment processes, the correlation between solids retention time (SRT) and PPCP removal was observed in the results of the study. Several studies have concluded there is a strong correlation between better PPCP removal and the longer SRT routinely employed in biological nutrient processes (Snyder et al., 2006; Miege, 2008; Clara et al., 2004; Stephenson and Oppenheimer, 2007; MWH, 2008).

Stephenson and Oppenheimer (2007) concluded that half of the 20 PPCPs they studied in secondary effluent were reduced by at least 80% with an SRT of five days. Triclosan, benzophenone, DEET, BHA, musk ketone, and galaxolide all required longer SRTs, up to 25 days, for 80% removal.

Figure 2 presents percent removal for six analytes across the various treatment technologies studied. Cotinine is effectively removed by all the WWTP technologies. On the other hand, the removal efficiency for albuterol and erythromycin was low for five of the treatments but improved with the combination of EBNR and tertiary filtration. These treatment types also had the longest SRTs in the study.

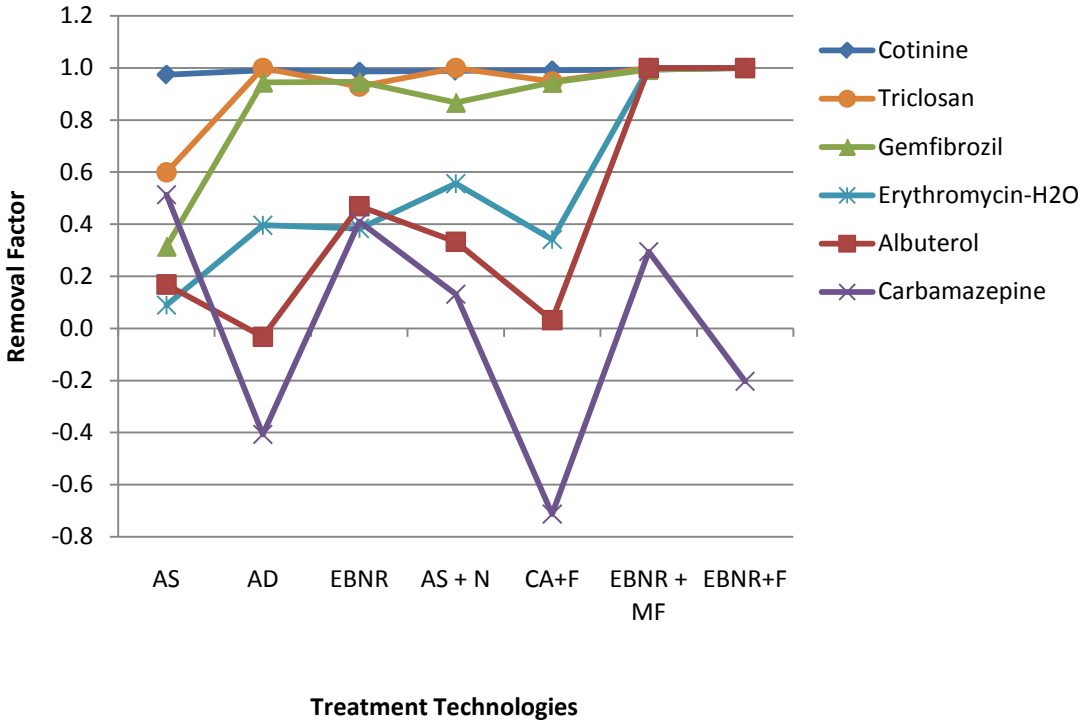

Figure 2. Percent removal of select analytes as measured by EPA Method 1694.

Conclusions

This 2008 screening study detected pharmaceuticals and personal care products (PPCPs), hormones, steroids, semi-volatile organic compounds, and nutrients in every influent, effluent, and biosolids sample analyzed from five Pacific Northwest wastewater treatment plants (WWTPs).

Three analytical methods were used to evaluate 172 organic compounds: 72 PPCPs, 27 hormones/ steroids, and 73 semi-volatile organics. Two methods recently published by EPA for the detection of PPCPs, hormones, and steroids performed well in the complex matrix of the wastewater and biosolids. The semi-volatile organics method detected a few PPCPs and also analyzed for a large number of non-target analytes, primarily PAHs.

Results

This study helped to demonstrate how different wastewater treatment processes affect removal of PPCPs. Of the 172 organic compounds monitored in this study, 96 (56%) were detected in at least one sample. Every sample in this study had detectable concentrations of multiple PPCPs.

Detections of the 172 organic compounds (analytes) among the five WWTP technologies revealed the following patterns:

1. Twelve analytes were removed by all secondary treatment technologies and *were not* present in the biosolids (e.g., acetaminophen).
2. Eight analytes were removed by all secondary treatment technologies and *were* present in the biosolids (e.g., 17a-Estradiol).
3. Eight analytes were present in the secondary effluent, but were removed by at least one of the tertiary technologies, and *were not* present in the biosolids (e.g., albuterol).
4. Thirty-one analytes were present in the secondary effluent, but were removed by at least one of the tertiary technologies, and *were* detected in the biosolids (e.g., ciprofloxacin, triclosan).
5. Nineteen analytes were detected only in the biosolids, not in the influent or effluent (e.g., enrofloxacin).
6. Eleven analytes apparently increased in concentration from influent to effluent in one or more of the wastewater processes, as indicated by a negative removal rate (e.g., carbamazepine).

The results of this study confirm findings from published studies that (1) PPCPs are routinely found in municipal wastewater, (2) treatment of PPCPs varies by chemical and treatment process, and (3) PPCP concentrations in influents, effluents, and biosolids are comparable to those found in the literature.

Class B biosolids were found to have a wide range of PPCP concentrations. Roughly 20% (mainly PAHs) of the 172 analytes were found only in the biosolids and not the wastewater samples. The PPCP concentrations found in the biosolids were from three WWTPs each with a different treatment process and different levels of treatment; however the results are roughly similar. Particularly, the magnitude of the PPCP concentrations is comparable across the three WWTPs sampled, and to available literature values. Some analytes were clearly concentrating in the biosolids, whereas other analytes were not. This may be due to affinity for soils, solids, or sediments.

In wastewater, approximately 21% of the 172 chemicals were reduced in effluents to below reporting limits by conventional secondary treatment; whereas 53% were removed by at least one advanced nutrient-removal technology. Secondary treatment alone achieved high removals for hormones and steroids. PPCP concentrations were reduced most effectively by the advanced biological nutrient removal with tertiary treatment technologies.

Three PPCP compounds stood out as relatively untreatable by the treatment technologies studied: carbamazepine, fluoxetine, and thiabendazole. These compounds may serve well as human-influence tracer compounds in the environment.

Results of this screening study indicate that the combination of enhanced biological nutrient removal and filtration processes provides the greatest PPCP removal. Although very few WWTPs discharging to Puget Sound have advanced nutrient-removal designs, other options – such as increasing biological contact times or adding tertiary filtration – may further reduce PPCP and nutrient concentrations in municipal discharges.

Recommendations

This 2008 screening study analyzed 172 organic compounds using three EPA analytical methods. Except for estradiol, the 24 PPCP target analytes for this study were detected in all the samples. In addition to the 24 analytes shown in Table 6, we recommend adding bezafibrate, diclofenac, atenolol, and ciprofloxacin as target analytes for future studies.

The two new EPA methods (1694 and 1698) performed well in this reconnaissance study and are recommended for use in other studies for comparability with this effort. Fewer compounds can be tested for using Method 1694, if necessary, to reduce project costs. Details can be found at www.epa.gov/waterscience/methods/method/ppcp/method1694-qna.html.

There is little research on the environmental transport, fate, and impacts of PPCPs in Pacific Northwest watersheds. A statistically robust study effort is recommended to assess the loading of PPCPs to Puget Sound. Sediment assessments should be incorporated into future study designs. Results may be useful to the modeling efforts currently underway to ascertain if chemical concentrations are building up in different areas of Puget Sound. Determining appropriate and protective levels for wastewater effluents and biosolids is needed.

The shortage of studies on the treatment, fate, and transport of PPCPs from biosolids to final biosolids product warrants further study, considering some biosolids are land-applied and the potential for their transport to the environmental is plausible. It is unclear at this time if PPCPs found in the biosolids are further reduced or destroyed by treatment before the biosolids are land-applied.

More research is needed to (1) better quantify wastewater treatment of PPCPs, (2) understand the fate of PPCPs in biosolids, and (3) determine whether discharged or land-applied PPCPs pose a concern for the environment.

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Appendices

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Appendix A. Pharmaceuticals and Personal Care Products in the Environment: A Literature Review

by

Melanie Redding
Licensed Geologist, Licensed Hydrogeologist

Summary

Pharmaceuticals and personal care products (PPCPs) are present at low concentrations in surface water, groundwater, soils, sediments, marine waters, and drinking water. Researchers monitoring the environment find PPCPs nearly everywhere domestic wastewater is discharged. PPCPs enter the environment as they pass through the human body or when unwanted PPCPs are disposed in the trash or down the drain. The human health effects resulting from daily exposure to low concentrations of PPCPs are unclear, but there are some documented impacts to wildlife from PPCPs in the environment.

Conventional wastewater treatment systems are not effective at removing some PPCPs from effluent. Some advanced wastewater treatment processes are more effective in removing these contaminants; however, these treatment processes are less commonly used. No single treatment process will completely remove all PPCPs from wastewater to non-detectable concentrations.

Prevention strategies, such as pharmaceutical take-back programs, are excellent tools but will only address a fraction of the issue. This PPCP source of contamination cannot be eliminated; it must be managed. The literature reviewed suggests that a combination of prevention strategies combined with advanced wastewater treatment could reduce PPCP loads into the environment.

Introduction

PPCPs are widely present in the environment. PPCPs are anthropogenic contaminants; their presence in the environment results from the universal, frequent, and cumulative usage by multitudes of individuals. This impact illustrates the inter-connectedness and the influence that humans have with their environment.

Low concentrations of PPCPs have been detected in surface water, groundwater, marine waters, soils, sediments, and drinking water. Large quantities of pharmaceuticals are used to treat and cure diseases and other medical conditions. PPCPs enter the environment primarily as they pass through the body or are improperly disposed of in toilets, sinks, and garbage. Generally, conventional wastewater treatment plants do not effectively remove PPCPs. These chemicals migrate into groundwater and surface water; scientists have also detected them in drinking water systems.

Since many of these chemicals are endocrine-disrupting compounds, carcinogens, or toxic chemicals, there is concern about the potential effects of these chemicals at low concentrations in the environment. It is unclear how the unintended exposure to low concentrations of multiple

chemicals may affect an organism or an individual. Scientists and policy makers do not yet know the full effects on wildlife and human health.

Purpose

This literature review focuses on the state of knowledge for PPCPs in several key areas of concern:

- The presence and occurrence of PPCPs in the environment.
- The removal efficiency of PPCPs by wastewater treatment plants (WWTPs).
- The effectiveness of source reduction efforts, such as drug take-back programs.

A limited discussion is provided on sources, fate and transport, and impacts, but a detailed review of the literature in these areas was deemed beyond the scope of this effort.

Definition

PPCPs include drugs made for humans and animals; they include prescription and over-the-counter drugs. They also include diagnostic agents such as x-ray contrast media, nutraceuticals (bioactive chemicals in nutritional supplements), and excipients (inert ingredients such as pill coatings) (Motzer, 2006). The PPCP definition also includes illicit drugs, personal care products (chemicals in consumer products), and veterinary medicines (Daughton and Ternes, 1999).

Personal care products are items that individuals use every day to take care of themselves. They include a wide variety of products: shampoo, deodorant, toothpaste, lotions, make-up, after-shave lotions, hair dyes, anti-dandruff shampoos, teeth whiteners, sunless tanning products, colognes, and fragrances. There are over 10,500 different chemicals used in personal care products. Only 11% of these chemicals have been tested for human health safety in the United States.

Recent Development of Analytical Methods

The U.S. Geological Survey (USGS) has developed analytical methods to detect PPCPs at very low concentrations in water (Kolpin et al., 2002). Concentrations of these chemicals can be detected at micrograms per liter (µg/l) and sometimes nanograms per liter (ng/l). The advent of these new methods has allowed researchers to detect pharmaceuticals and ingredients in personal care products at concentrations that were not previously detectable. As these new methods have been applied in recent environmental investigations, researchers are finding PPCPs in water, soil, and wildlife.

Sources of PPCPs in the Environment

PPCPs enter the environment from several different sources. Humans are a predominant source of PPCPs. PPCPs enter the environment by being washed off the body, excreted, or disposed down the drain or in the garbage. These PPCPs enter the environment either through wastewater treatment systems or landfills. Other sources of PPCPs include livestock, agriculture, pets, and aquaculture.

Humans typically excrete 50% to 90% of the active ingredients in ingested drugs, either as unmetabolized pharmaceuticals or as metabolites (McGovern and McDonald, 2003). When these excreted chemicals leave the body, they typically enter a municipal WWTP, an on-site sewage system, or a reclaimed water treatment facility. Different treatment processes vary in their treatment efficiency for PPCPs. Typically wastewater from the treatment system is discharged into the environment.

Nationally approximately 50% of all biosolids from wastewater treatment are applied to land, with the remainder disposed of in landfills.

Consumers dispose of an estimated 25% to 33% of pharmaceuticals sold, either to a landfill or WWTP. This rate was extrapolated from data generated in Germany and Australia (Heberer, 2006). A consumer survey conducted in King County in 2005 also supports this disposal rate. The King County study found 36.5% of residents stated that they typically dispose of pharmaceuticals in the garbage, and 29.4% typically dispose of pharmaceuticals in the sink or toilet (PH:ARM Pilot Team, 2007). Ultimately these disposed PPCPs enter a municipal WWTP, an on-site sewage system, or a reclaimed water treatment facility. Unused or expired PPCPs thrown away in the trash, and disposed of at a landfill, can be mobilized in the environment via landfill leachate.

Unlike human sources of PPCPs, animal excretions of pharmaceuticals at a confined animal feeding operation do not pass through a treatment system prior to entering the environment.

Fish farms add pharmaceuticals to the environment in the form of feed additives (Halling-Sorensen et al., 1998).

Pets also use prescription drugs as well as other products similar to personal care products marketed for humans.

Fate and Transport

The fate of PPCPs in the environment is complex for a number of reasons. First, there are thousands of chemicals used in the manufacture of a wide variety of PPCPs. Not all PPCPs are similar chemically, and the different types of chemicals react differently to different treatment processes. The individual chemical structure dictates whether PPCPs will biodegrade, volatilize, degrade into metabolites, or whether they will concentrate and persist in the environment. Some PPCPs are water soluble, and some are fat soluble. A chemical's solubility determines how a chemical will be transported in water (Chiou and Kile, 2000). Some PPCPs are affected by pH, turbidity, sunlight, and oxidation (Holtz, 2006). How a chemical partitions between sediment and water is the most influential factor in determining the fate of organic chemicals in the environment.

A review of environmental studies indicates that PPCPs are present in surface water, groundwater, drinking water, and sediments (Halling-Sorensen et al., 1998). Figure A-1 illustrates how PPCPs enter the environment.

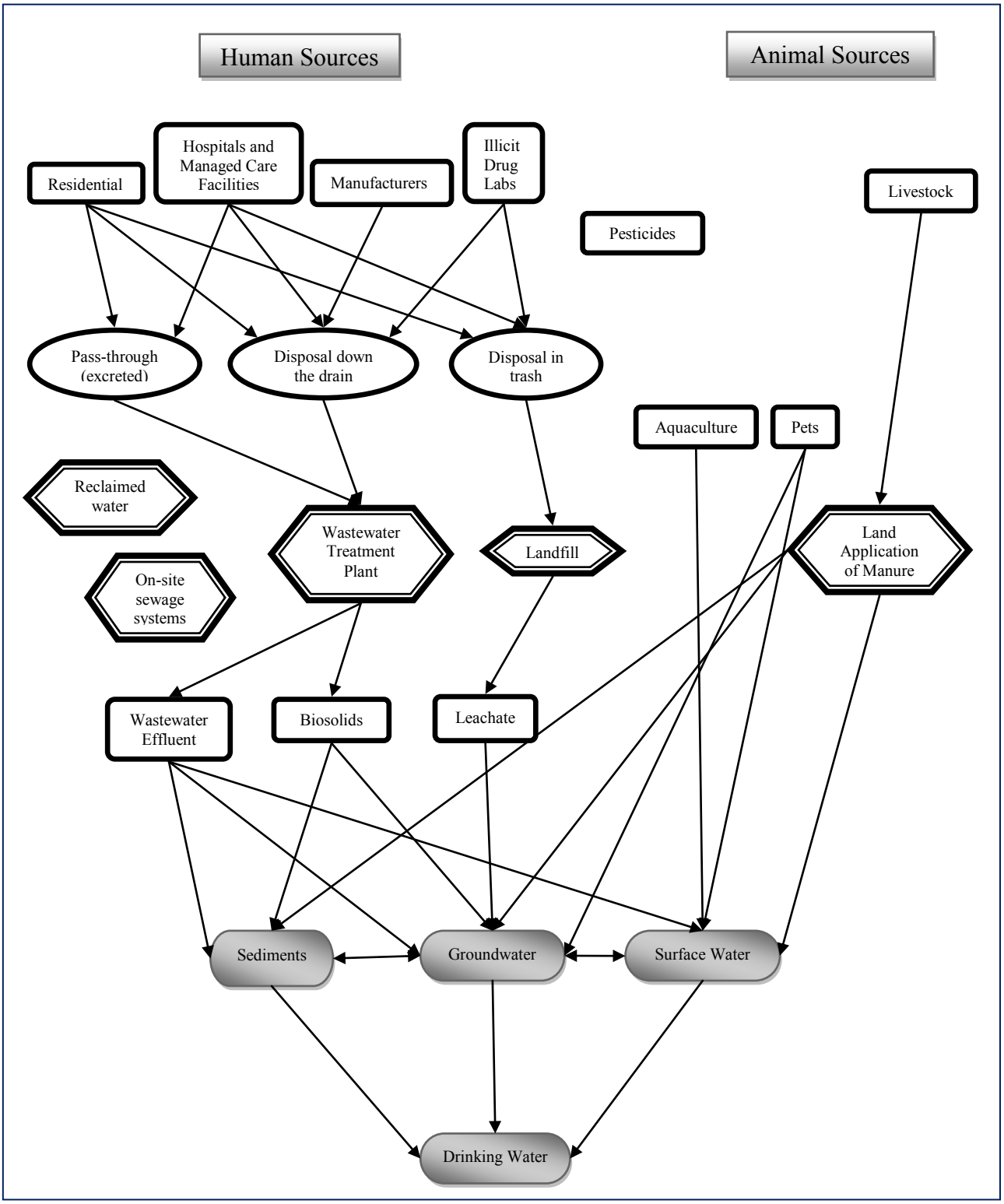

Figure A-1. Environmental Fate of PPCPs.

Presence in the Environment

There are numerous environmental studies which document the presence of PPCPs in surface water, groundwater, and sediments.

Nationwide Surface Water Monitoring Study

From 1999 to 2000, the USGS (Kolpin et al., 2002; Barnes et al., 2002) conducted the first national assessment of pharmaceuticals in U.S. streams. This study sampled 139 streams in 30 states. This study also captured a variety of hydrogeologic, climatic, and land-use settings. Ninety-five PPCPs were analyzed, and 82 (86%) were detected in the aquatic environment. All of the 95 chemicals tested are used extensively by the general public. Eighty percent of the sites had at least 1 PPCP detected, and 75% of the sites had multiple PPCPs detected. Concentrations were low, generally in the µg/l range. Standards have been established for only 14 of the compounds, and rarely were any of these standards exceeded. The lack of standards is due to the limited information about potential human and aquatic health effects.

Certain types of organic chemicals were detected more frequently than others. Steroids, non-prescription drugs, and insect repellent were the three groups most frequently detected during this study. Detergent metabolites, plasticizers, steroids, and non-prescription drugs were found at the highest concentrations.

The organic chemicals chosen for monitoring in this study were aimed at pharmaceuticals, personal care products, biogenic hormones, and other household chemicals released directly into the environment after wastewater treatment processes. The high level of occurrence indicates that many compounds are not sufficiently removed by the wastewater treatment processes. The presence of PPCPs in sediments is an area which was determined to need more attention.

The most frequently detected compounds are shown in Figure A-2. These include coprostanol, cholesterol, N,N-diethyltoluamide, caffeine, triclosan, tri(2-chloroethyl)phosphate, and 4-nonylphenol. Thirty-three of the 95 compounds monitored are endocrine-disrupting compounds, and all 33 were detected during the study.

Groundwater Monitoring

Benotti et al. (2006) sampled 61 groundwater wells located in the shallow glacial aquifer in Suffolk County, New York for 24 pharmaceuticals. Pharmaceuticals were detected in 28 wells (46%) at concentrations ranging from 0.001 to 0.1 µg/l. The study found that many of the pharmaceuticals were detected at similar concentrations as were detected in streams (Kolpin et al., 2002), but at lower frequencies. The median concentration of detected pharmaceuticals in groundwater was similar to the median detected concentration in surface water. Ten pharmaceuticals had median concentrations that were similar in groundwater and surface water. These included acetaminophen, caffeine, codeine, cotinine, gemfibrozil, dehydronifedipine, diltiazem, paraxanthine, sulfamethoxazole, and trimethoprim.

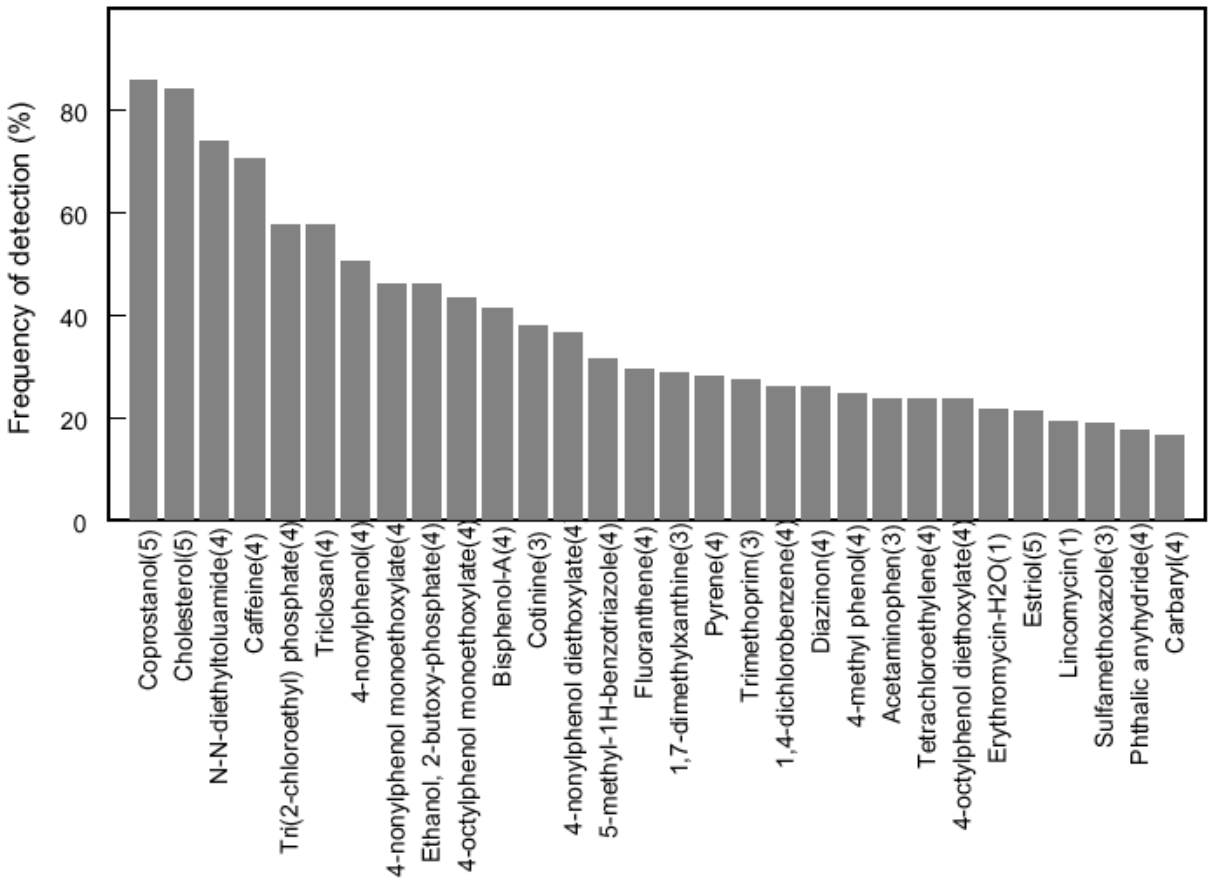

Figure A-2. Most frequently detected compounds in the National Reconnaissance Study of PPCPs in surface water conducted by the USGS (Kolpin et al., 2002; Barnes et al., 2002).

The presence of these compounds in groundwater indicates that current wastewater practices generally do not remove pharmaceuticals and may act as a loading source to the aquifer. These compounds are discharged to the environment in the effluent from on-site sewage systems and municipal WWTPs.

Fifty-four percent of the pharmaceuticals tested were detected in groundwater. Carbamazepine was detected in 26% of the samples. Sulfamethoxazole was detected in 13% of the samples. These two compounds were also found in other studies to be among the most commonly detected pharmaceuticals.

This study concluded that shallow groundwater downgradient of municipal wastewater discharges can contain low concentrations of pharmaceuticals, and aquifers which receive wastewater have the potential for contamination with pharmaceutical compounds.

Drinking Water and Wastewater Monitoring

Zimmerman (2005) sampled PPCPs and other organic chemicals from both wastewater sources and drinking water supplies in Cape Cod, Massachusetts in 2004. Eighty-five chemical

compounds were analyzed and 43 were detected. Thirteen of the compounds detected were in drinking water sources. The PPCPs detected in drinking water include acetaminophen, sulfamethoxazole, and carbamazepine. Nine of the chemicals detected are endocrine-disrupting compounds. Concentrations in the water supplies ranged from 0.0037 to 0.0576 µg/l. Concentrations from the wastewater sources ranged from 0.0036 to 6.4 µg/l.

Surface Water Monitoring

Boyd and Furlong (2002) analyzed 33 pharmaceutical compounds in (1) Las Vegas Wash, Nevada, the primary channel through which the valley's excess water returns to Lake Mead, and (2) Lake Mead, Nevada/Arizona. Carbamazepine, dehydronifedipine, acetaminophen, cimetidine, codeine, and diltiazem were detected in 83% of the Las Vegas Wash samples. Thirteen compounds were detected in one sample.

Galloway et al. (2005) sampled upstream and downstream sites from WWTP effluent discharges on 7 streams in northern Arkansas for 108 pharmaceutical and other organic compounds. At least one of the 108 compounds was detected at all but one site. Figure A-3 illustrates the relationship of upstream and downstream effects. The number of pharmaceutical detections was greater at downstream sites (median = 14) than upstream sites (median =3).

Forty-two of the 108 compounds were detected during this study. The most frequently detected constituents include caffeine, phenol, para-cresol, and acetyl hexamethyl tetrahydronaphthalene.

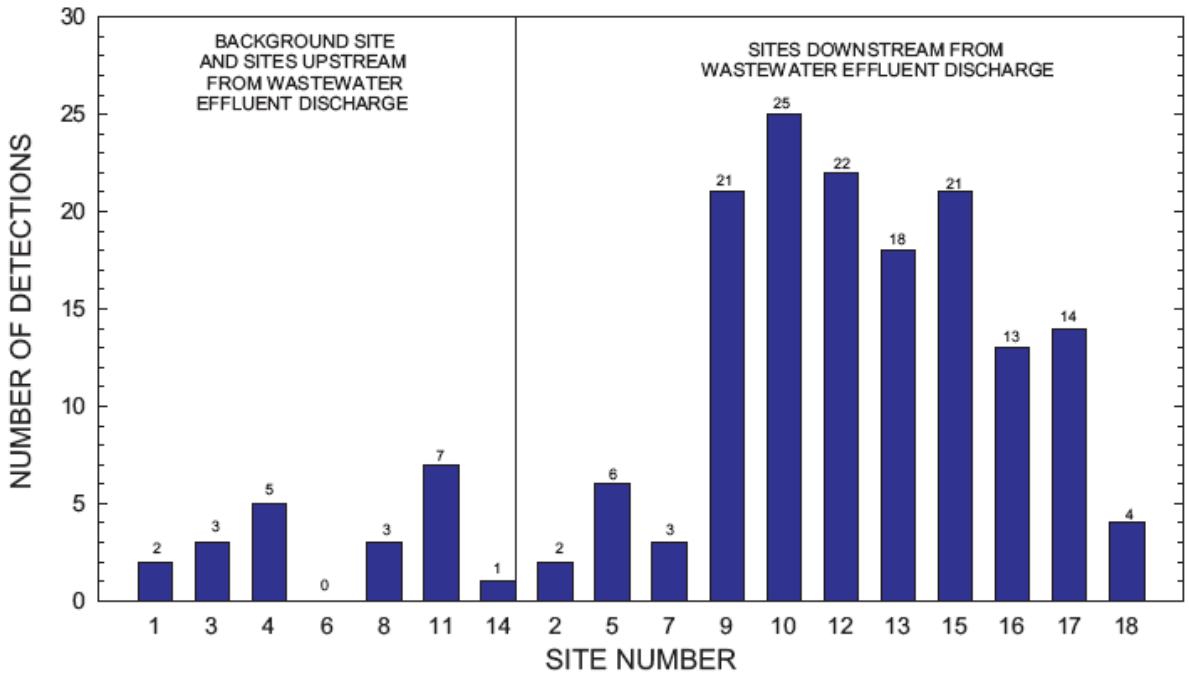

Figure A-3. Summary of pharmaceuticals and other organic wastewater constituents detected from selected sites in northern Arkansas, March and April 2004 (Galloway et al., 2005).

Surface Water, Groundwater, and Wastewater Monitoring in Sequim, Washington

The Washington State Department of Ecology **(**Johnson et al., 2004**)** conducted a PPCP product study in the Sequim-Dungeness area. This one-time collection of water quality samples from WWTP effluent, surface water, and groundwater tested for 24 chemicals. Seventeen PPCPs were detected in the effluent, 4 were detected in surface water, and 3 were detected in groundwater. All of the concentrations were low (µg/l).

Impacts on Wildlife

The literature also documents impacts to wildlife from the direct exposure to PPCPs. This includes impacts to vultures, fish, and alligators.

Vultures

In 1990 the population of vultures in India and Pakistan numbered in the millions. Vultures served a natural role in the management of livestock carcasses. When cattle died, they were left for vultures to pick the bones clean within a few days**.** In the late 1990s the vulture population began declining. By the year 2000, 95% of the population had died. Three species of vultures nearly became extinct.

Oakes et al. (2004) determined that diclofenac, given to sick cattle, caused the vulture population decline. Diclofenac is an anti-inflammatory drug that doctors prescribe for arthritis and pain in humans. Additionally, veterinarians commonly prescribe this medicine to treat fever and lameness in cattle. It is largely available and inexpensive, which has led to widespread use in India and Pakistan. In vultures, diclofenac causes acute kidney failure. Diclofenac does not appear to be toxic to other predatory birds or mammals.

The use of diclofenac not only led to the near extinction of three species of vultures, but it also resulted in indirect health effects on humans and other species. Vultures served as the predominant natural scavengers for dead livestock. With less than 5% of the vulture population remaining, the feral dog population increased, replacing vultures in the ecological niche. Feral dogs have a high incidence of rabies. Additionally, the uneaten carcasses are left to rot in the sun, and create a human health hazard through exposure to decaying remains.

Fish

Vajda et al. (2003) studied white sucker fish in Boulder Creek, Colorado. These fish have limited territories and do not migrate up and downstream. They collected fish upstream and downstream of a WWTP outfall. They analyzed effluent quality and found a number of endocrine-disrupting compounds including alkylphenols, bisphenol A, and reproductive steroids. They noted a number of effects in the downstream fish that were not present upstream. The male to female ratio upstream was roughly equal, but downstream of the WWTP, the ratio was 90% female and 10% male. The remaining downstream males all showed significant signs of abnormal reproductive organs. Additionally, the downstream female population also exhibited reproductive abnormalities. Intersex fish and elevated vitellogenin in juvenile fish were only detected downstream of the WWTP outfall.

This study concluded that the reproductive potential of native fishes may be compromised in small effluent-dominated streams.

Orlando et al. (2004) determined that fathead minnows exposed to effluent from confined animal feeding operations (CAFOs) showed significant impacts on reproduction. CAFOs are sources of contaminants that can run off into surface waters and in some cases into groundwater. These contaminants can also enter the environment when animal manure is spread onto the land surface.

The Lower Columbia River Estuary Partnership (2007) measured toxic contaminants, including PPCPs, in the Lower Columbia River and its tributaries to determine the effects on water quality, fish, and wildlife. Thirty-three PPCPs were monitored in both salmon and water. Caffeine was detected at every site. Bisphenol A, HHCB, trimethoprim, and anhydroerythromycin were also frequently detected. PPCPs were more commonly detected during the low-flow sampling event in August than the high-flow sampling event in April. This is probably the result of dilution associated with higher river flows. The study reported concentrations in the microgram-per-liter range in the water column. Researchers also detected PPCPs in all sediment samples, with the highest concentrations near urban and industrial areas.

Jobling et al. (1998) documented widespread sexual disruption to wildlife from exposure to ambient (background) levels of "estrogenic constituents of sewage effluents" in rivers. The study correlated reproductive and developmental effects from exposure to hormonally active substances discharged from WWTPs. Intersex fish were found at all sites including the control site, suggesting that a low incidence of intersexuality may be natural. A much higher incidence of intersex fish was detected at sites impacted by sewage effluent, indicating that the effluent may be causing sexual disruption to wild fish.

Alligators

Guillette et al. (2000) attributed reproductive and developmental impairment in the American alligator population in Florida to the presence of endocrine-disrupting compounds (EDCs) in the environment. These disorders include altered fertility, reduced viability of offspring, impaired hormone activity, and modified reproductive anatomy. This study compared behavior and population statistics for the American Alligator from a contaminated lake to a clean reference lake in Central Florida. The alligators living in the lake contaminated with dicofol showed altered hormone concentrations and exhibited modified reproductive anatomy and function.

Wastewater Treatment

The treatability of PPCPs depends on the physicochemical properties of each compound of interest and the specific set of treatment processes. Some WWTP processes efficiently remove some chemicals, but are ineffective at treating others. Some treatment processes merely remove the chemical from one media (water) and transfer it to another media (biosolids) without destroying it. For example, nonylphenol is removed from water through settling, but subsequently partitions to the solids. Once land-applied it remains in the environment, available for transport to surface or groundwater. Natural processes include adsorption, filtration,

volatilization, photodegradation, biodegradation, chemical alteration, and plant or animal utilization.

Primary treatment at WWTPs is the process of separating the solid phase from the liquid phase. Primary treatment can include screens, sand and grit chambers, and primary clarifiers. The chemical and physical properties of a particular organic compound influence the degree of removal during primary treatment. Solubility, volatility, and lipophilicity are all properties which affect removal rates. Generally, primary treatment does not do an adequate job of removing PPCPs from wastewater.

Secondary treatment degrades the biochemical organic compounds of sewage using biological processes. Secondary treatment provides a biologically oxidative environment, which converts the dissolved biological matter into sludge by settling or filtering the microorganisms from the effluent. Secondary treatment can include anaerobic treatment methods as well as oxidative biological treatment methods such as trickling filters, oxidation ditches, activated sludge, aerated lagoons, or high rate reactors. A compound's susceptibility to microbial degradation will affect whether the compound degrades or persists in the effluent.

Tertiary, or advanced treatment, is the polishing stage designed to raise effluent quality prior to being discharged to the environment through a variety of biological, chemical, or physical treatment processes. Tertiary treatment may include chemical addition for coagulation, conventional filtration, membrane filtration, reverse osmosis, nutrient removal, electrodialysis, advanced oxidation processes, activated carbon, or natural processes such as constructed wetlands. Membrane bioreactors combine secondary treatment with tertiary membrane filtration processes.

Disinfection substantially reduces the number of microorganisms in the water prior to being released into the environment. This is important to prevent the transmission of disease. The most common types of disinfection include chlorine, ultraviolet light, and ozone.

Sludge treatment is necessary to further stabilize the organic matter in the solids. Treatment methods include anaerobic digestion, and aerobic digestion or composting, to produce a usable soil amendment called biosolids.

Reclaimed water, also known as water reuse or water recycling, is a highly engineered, multi-step process which includes primary, secondary, and advanced treatment techniques and disinfection to produce reliable, high quality water before it leaves the WWTP.

Wastewater Treatment Plant Studies

Ternes (1998) monitored 32 pharmaceutical drugs and 5 metabolites in municipal WWTP influent and effluent, and in the receiving surface waters. The WWTP monitored in Frankfurt/Main Germany had three principle treatment steps: preliminary clarification, secondary aeration with Fe(II) chloride for phosphate elimination, and clarification.

Generally, the WWTP removed greater than 60% of the pharmaceuticals. Only carbamazepine, clofibric acid, phenazone, and dimethylaminophenazone had lower than average removal rates. The study did not differentiate whether removal occurred by sorption or biodegradation.

Over 80% of the pharmaceuticals were detectable in at least one effluent sample. Twenty pharmaceuticals and 4 metabolites were detected in the receiving surface water. Ternes found mainly the acidic drugs ubiquitously in surface waters in the nanogram-per-liter range. Acidic drugs include lipid regulators (bezafibrate, gemfibrozil), the antiphlogistics (diclofenac, ibuprofen, indomethacine, naproxen, phenazone, and the metabolites clofibric acid, fenofibric acid and salicylic acid) as well as neutral or weakly basic drugs such as the beta blockers (metoprolol, propranolol), and the antiepileptic drug carbamazepine.

Flocculation with iron (III) chloride showed no significant removal of any of the five pharmaceuticals tested (Ternes et al., 2002).

- Diclofenac 4%
- Clofibric acid 13%
- Bezafibrate 11%
- Carbamazepine 13%
- Primidone 10%

Khan and Ongerth (2004) developed a conceptual model for determining which pharmaceutical compounds would most likely be found in municipal sewage, as well as their concentrations. They choose 50 pharmaceuticals based on their prescribing volumes, their excretion rates, and the type of drug. The model predicted that 29 (58%) of the pharmaceuticals would be present in the influent at concentrations of greater than or equal to 1 µg/l, and 20 (40%) of the pharmaceuticals would still be present in the wastewater at concentrations greater than or equal to 1 µg/l after secondary treatment. Table A-1 summarizes their statistical projections of pharmaceutical removal rates.

Table A-1. Statistical summary of 50 pharmaceutical removal rates.
(Modified from Khan and Ongerth, 2004).

Statistic	Percent Removal to Sludge	Percent Biodegradation	Percent Removal by Secondary Treatment
Mean	6	37	44
Median	4	39	42
Range	1 - 50	4 - 80	14 - 99

This model assumes that wastewater undergoes primary settling, secondary aeration, and clarification in an activated sludge WWTP. The authors determined that the majority of pharmaceutical removal occurs in the aeration tank. Additionally, they noted that pharmaceuticals were removed more efficiently during secondary clarification by biodegradation, rather than during primary settling.

The penicillin-based antibiotics degrade rapidly in wastewater treatment systems and aquatic systems because of their susceptibility to hydrolysis (Daughton and Ternes, 1999).

The antiviral agent oseltamivir, or Tamiflu by its brand name, does not readily degrade with normal WWTP processes. Additionally, it is not substantially degraded by ultraviolet disinfection. A study conducted by Fick et al. (2007) analyzed concentrations of oseltamivir in influent to a WWTP as well as water from primary mechanical treatment, chemical treatment with the addition of $FeCl_3$ or $FeSO_4$ to reduce nutrients, and activated sludge treatment. At the end of the treatment process, scientists recovered nearly 100% of the oseltamivir they added to the sewage; conventional secondary sewage treatment did not remove or treat oseltamivir.

Solids Retention Time

Higher solids retention times (SRTs) can increase removal of some PPCPs from the influent. Depending on the length of time and the contaminant, the average removal efficiency is approximately 60% with a variance of 10% to 100%.

Strenn et al. (2003) found higher SRTs resulted in greater percent removals of hydrophobic compounds. Joss et al. (2004) and Cleary (2007) determined that activated sludge with increased SRT increases the removal rates of PPCPs from wastewater. Phillips et al. (2005) reported that SRTs of 5 to 10 days were effective, while Siegrist et al. (2005) and Ternes et al. (2005) found that SRTs of 10 to 15 days were optimal. Stephenson and Oppenheimer (2007) determined the critical SRT to consistently remove 80% of the compounds of concern is chemical specific; however, 5 to 15 days seemed to remove the majority of contaminants. Some persistent PPCPs include galaxolide, musk ketone and tri(chloroethyl) phosphate, which required SRTs of greater than 30 days to remove.

Secondary treatment plus nutrient removal also provides good removal of steroids, antibiotics, pain relievers, and other PPCPs. Stephenson and Oppenheimer (2007) found that activated sludge combined with a longer SRT, which is typically required for nutrient removal, is responsible for PPCP removal. These researchers determined that media filtration, including membrane bioreactors (MBRs), added no substantial benefit beyond the increased SRT. Reverse osmosis was determined to reduce concentrations in the sludge. Stephenson and Oppenheimer (2007) found that hydraulic retention time (HRT) does not have a significant impact on PPCP removal, but Drewes et al. (2006) reported that sorption to biosolids is an important PPCP removal mechanism.

Filtration

Snyder et al. (2007a) performed a comprehensive analysis of the use of various membrane and activated carbon technologies on the removal of pharmaceuticals, endocrine-disrupting compounds, and personal care products. The wastewater treatment technologies analyzed included microfiltration, ultrafiltration, nanofiltration, granular activated carbon, powdered activated carbon, reverse osmosis, electrodialysis reversal, membrane bioreactors, and combinations of these technologies in series.

Microfiltration was not shown to be effective at removing the majority of organic compounds tested. However, microfiltration did effectively remove steroids, especially when coupled with a membrane bioreactor.

Ultrafiltration reduced concentrations but was not shown effective at removing the majority of organic compounds tested. However, ultrafiltration effectively removed steroids, especially when coupled with a membrane bioreactor. Snyder et al. (2006a) determined that ultrafiltration provided an average removal rate of 59%, and ranged from 1% to 100% depending on the chemical.

Nanofiltration was shown to be capable of removing almost all the pharmaceuticals tested, although a few pharmaceuticals were present in the permeate.

Granular activated carbon (GAC) was shown to be highly effective at removing trace levels of many pharmaceuticals. Since water soluble contaminants can break through the filter, GAC is more efficient when the media is regenerated on a regular basis. Organic compounds that have a greater hydrophilicity pass through the GAC unit faster than hydrophobic compounds. In treatment systems with high levels of total organic carbon where the GAC is not regenerated, very little removal occurred. Figure A-4 illustrates the importance of regenerating or replacing the media in order for this treatment technology to achieve high rates of removal. To prevent transfer to another environmental medium, the regeneration process and the contaminated media must be controlled (Snyder et al., 2006a).

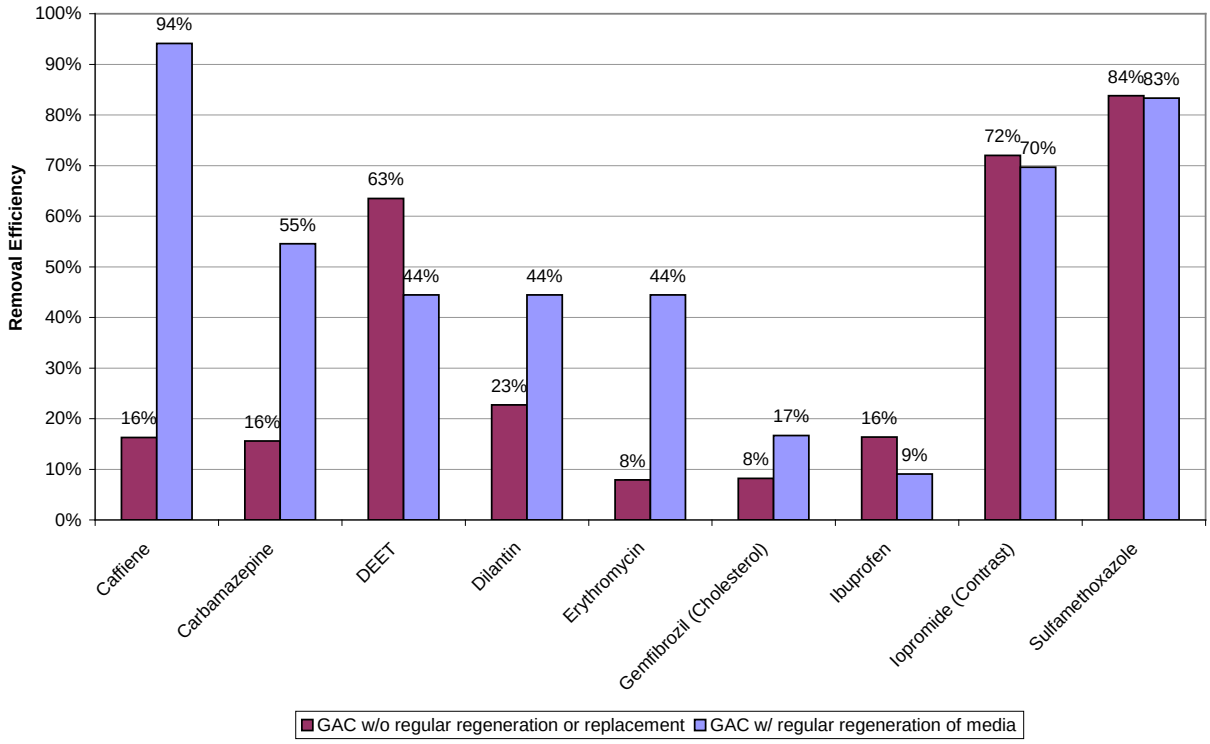

Figure A-4. Comparison of PPCP removal rates with granular activated carbon based on the regeneration of environmental media (modified from Snyder et al., 2006a).

GAC efficiently removed 4 of the 5 pharmaceuticals tested at WWTPs. Sorption efficiencies depend on the competition of adsorption sites with other organic compounds. The adsorption capacity for pharmaceuticals is lower if other organic compounds are present in the water. Clofibric acid had the lowest sorption capacity on granular activated carbon, and carbamazepine had the highest adsorption capacity (Ternes et al., 2002).

Powdered activated carbon (PAC): Snyder et al. (2006a) found PAC effectively removed more than 90% of nearly all compounds tested. The presence of natural organic matter, which competes for sorption sites, affects the removal efficiency. The treatment efficiency depends most importantly on the amount of PAC used in the system, which is illustrated in Figure A-5. Additionally, the contact time and the characteristics of the specific contaminant are also important. Increased contact time provided a greater removal rate. When the media is regenerated, the contaminated media must be disposed of in such a manner that the concentrated organic chemicals do not re-enter the environment (Snyder et al., 2006a).

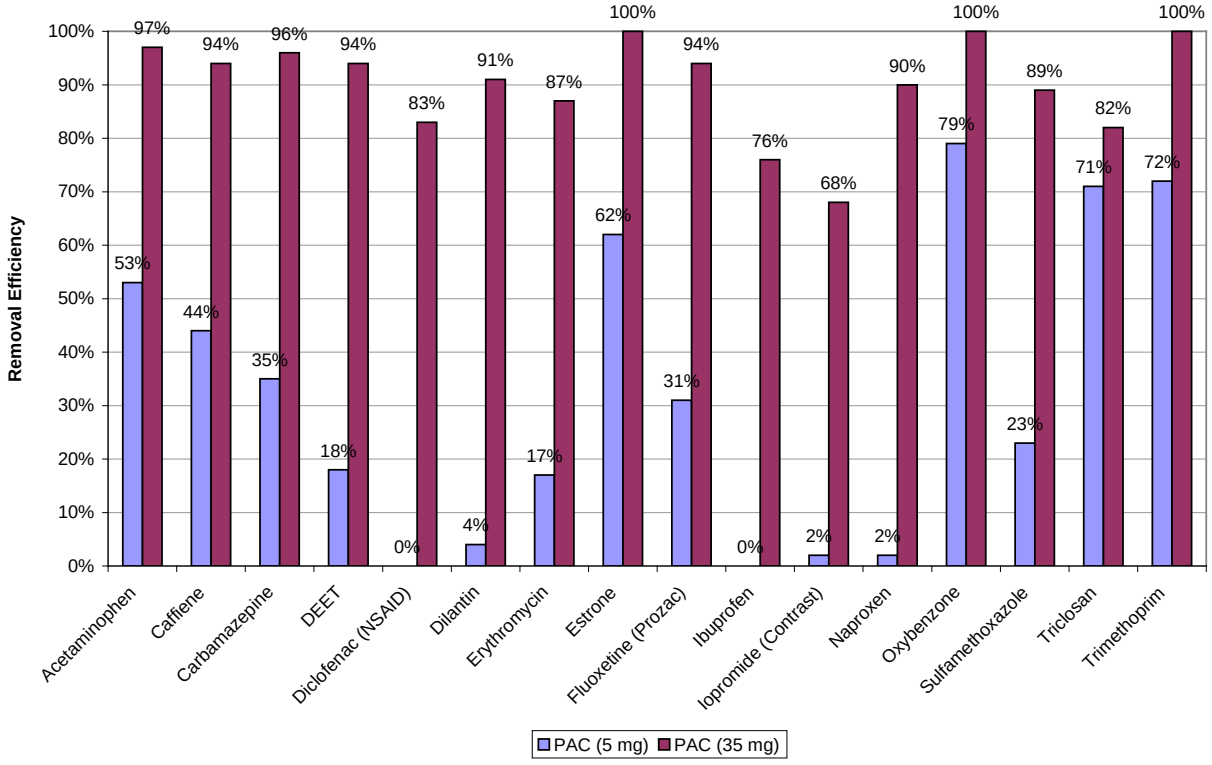

Figure A-5. Comparison of PPCP removal rates with powdered activated carbon based on the dose (modified from Snyder et al., 2006a).

The process of *reverse osmosis* (RO) generates a brine solution which contains all the rejected compounds. The RO process does not destroy pharmaceuticals; it only removes them from the filtered water and concentrates them in the brine solution. Disposal of the brine must be addressed to assure that the pharmaceuticals do not cycle back into the environment (Snyder et al., 2006a).

RO membranes are capable of removing almost all compounds to concentrations below reporting limits. However, this does not universally apply to pharmaceuticals; detectable levels of some pharmaceuticals remain in the RO permeate. The compounds that breached the RO membrane showed no consistent patterns in concentration or molecular structure. Snyder et al. (2006a) determined that a double-pass RO system was the more effective treatment technology, removing almost all pharmaceuticals to less than reporting limits. They further determined that a multi-barrier approach proved most successful in removing trace organic contaminants, as illustrated in Figure A-6. Note that negative treatment removal efficiencies are recorded as metabolites, which return back to the parent compound during different phases of treatment.

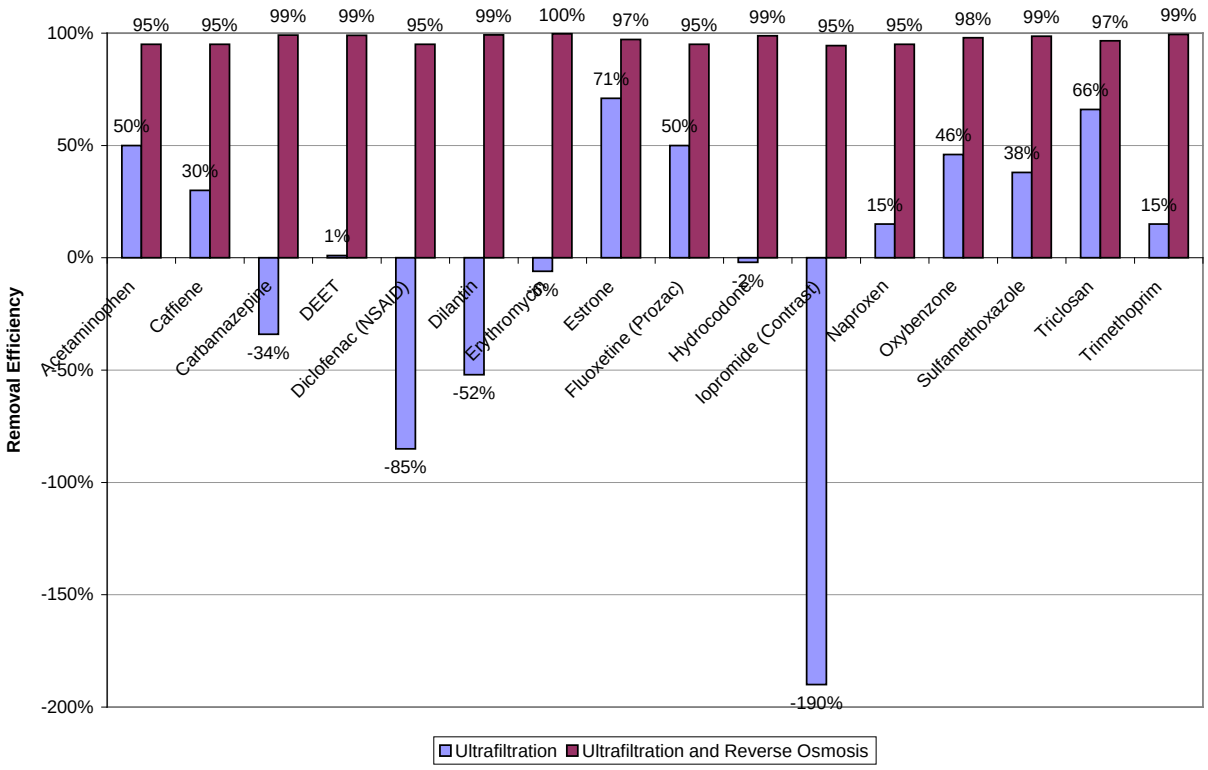

Figure A-6. Comparison of PPCP removal using single treatment (ultrafiltration) and multiple levels of treatment (ultrafiltration plus reverse osmosis) (modified from Snyder et al., 2006a).

Figure A-7 also illustrates this by comparing removal efficiencies of primary treatment, MBR treatment, and MBR plus RO treatment.

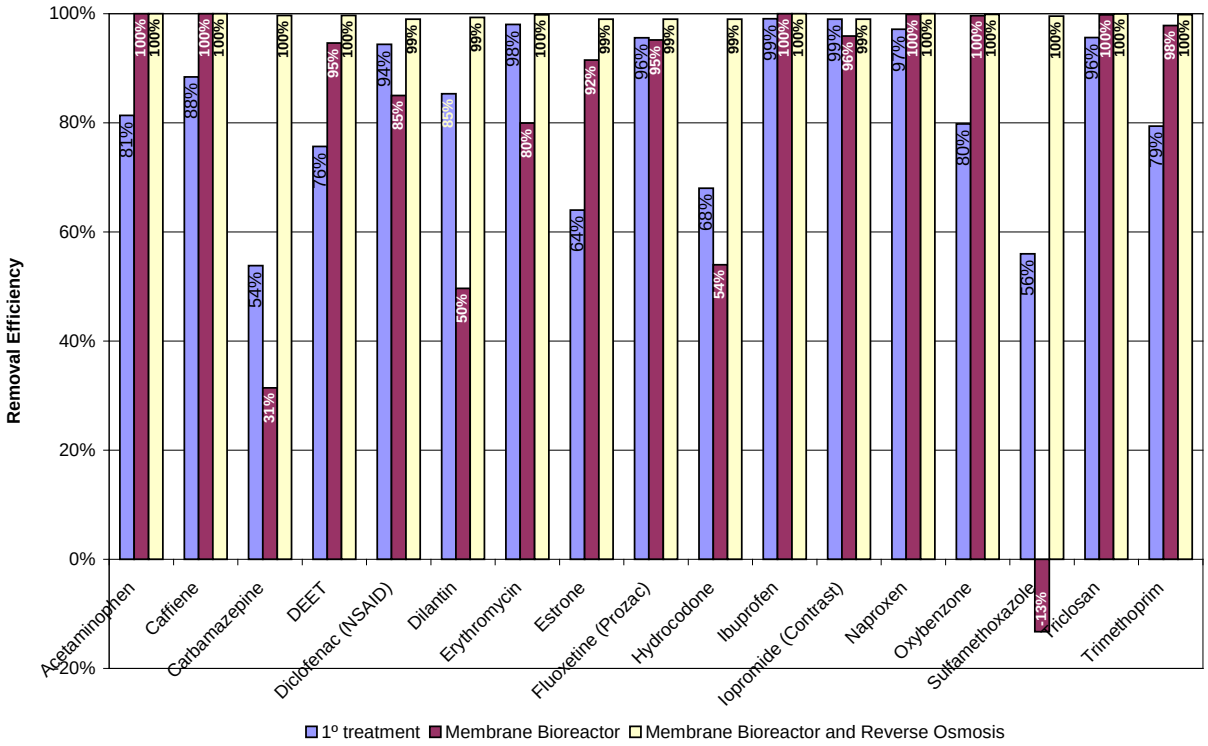

Figure A-7. Treatment removal of PPCPs comparing primary treatment, membrane bioreactor, and membrane bioreactor plus reverse osmosis treatment (modified from Snyder et al., 2006a).

Riverbank Filtration

Heberer et al. (2004) conducted a riverbank filtration and artificial groundwater recharge project in Berlin, Germany to determine the fate and transport of pharmaceuticals. Europeans use natural riverbank filtration as a common method to treat surface water prior to using it for drinking water. They have historically considered it an efficient means of treating waters through natural attenuation processes. One hundred percent of the drinking water in Berlin originates from groundwater; riverbank filtration and artificial groundwater recharge account for approximately 70% of the treatment. The Germans have used this approach to treatment for over 100 years. They typically drill drinking-water-supply wells as close as 600 meters from surface water. However, water quality degradation can arise when municipal WWTPs discharge wastewater upstream of the public drinking water supply wells.

Heberer et al. (2004) analyzed surface water and groundwater samples for more than 60 organic compounds. They detected diclofenac, propyphenazone, carbamazepine, primidone, clofibric acid, and 1-acetyl-1-methyl-2-dimethyl-oxamoyl-2-phenylhydrazide in groundwater downgradient of a riverbank filtration site. They also detected these compounds in low concentrations in public drinking-water-supply wells. The concentrations in groundwater were

lower than in surface water, indicating either dilution or partial or total removal is occurring. Riverbank filtration efficiently removed bezafibrate, indomethacine, some antibiotics, and some estrogenic steroids.

Ternes et al. (2002) studied the treatment efficiencies of various drinking water treatment systems on five pharmaceuticals. They conducted studies under laboratory, pilot, and real-world conditions, and analyzed for bezafibrate, clofibric acid, diclofenac, carbamazepine, and primidone. The treatment systems include natural biodegradation, flocculation, granular activated carbon, ozonation, and a combination of treatment trains. These tests did not take into account the complex environments and the diverse bioactivity that could enhance removals in the environment. The authors noted that diclofenac appears to be removed by riverbank filtration; they did not identify the mechanism for this removal.

Membrane Bioreactors

Kimura et al. (2005) investigated the ability of submerged MBRs at a municipal WWTP to remove six pharmaceuticals and one herbicide (dichlorprop). They compared this treatment to the removal efficiency of an activated sludge process.

As shown in Table A-2, MBRs demonstrated a better removal rate for ketoprofen and naproxen. For the other compounds, the removal rate was comparable with activated sludge. The authors attributed the poor removal of some compounds in both treatment processes to either the inclusion of chlorine within their chemical structure, or a double aromatic ring structure. Ibuprofen has a relatively simple chemical structure with no chlorine molecules, and both treatment systems efficiently removed it.

Table A-2. Relative removal efficiencies of membrane bioreactors and activated sludge. *(Modified from Kimura et al., 2005.)*

Pharmaceutical	Membrane Bioreactor	Activated Sludge
Clofibric acid	poor	poor
Diclofenac	poor	poor
Ketoprofen	excellent	poor
Naproxen	excellent	moderate
Dichlorprop	moderate	poor
Ibuprofen	excellent	excellent
Mefenamic acid	good	moderate

Ozone Disinfection

Drury et al. (2006) investigated the use of ozone as a means to oxidize organic contaminants while also providing disinfection to the filtered secondary treated effluent. This study investigated concentrations of ozone from 3 to 8 mg/l under both summer and winter conditions to determine optimal organic removal in conjunction with the process of disinfecting wastewater.

Previous studies had demonstrated that ozone is much more effective than hypochlorite in the oxidation of organic chemicals, including steroids, and PPCPs (Westerhoff et al., 2005). They showed complete ozone decay in 10 to 20 minutes, based on the concentration of ozone. Drury et al. (2006) found ozone achieved a 90% reduction for 90% of the contaminants. Compounds still present in the wastewater after the highest exposure to ozone included estrone, dilantin, iopromide, meprobamate, triclosan, TCEP, DEET, and oxybenzone.

Snyder et al. (2006b) investigated the removal of 36 organic compounds with the use of ozone and ozone combined with hydrogen peroxide. They conducted laboratory, pilot-scale, and full- scale wastewater testing to analyze wastewater and surface water concentrations of pharmaceuticals, steroids, and other organic compounds. They determined that the addition of hydrogen peroxide for advanced oxidation provided little benefit as compared to ozone alone.

The researchers concluded that ozone is a highly effective oxidant for removing the majority of organic contaminants from wastewater. Of the 36 compounds tested, they demonstrated removal of 22 from surface water by ozone concentrations of 1.25 mg/l or more. Only 6 compounds had removal rates less than 50%. As shown in Table A-3, musk ketone, lindane, and TCEP were the most resilient compounds with removal rates less than 20%. Snyder et al. (2006b) also noted that if dissolved organic carbon is not significantly reduced, treatment by-products will be formed.

Table A-3. Percent PPCP removal with ozone treatment (Snyder et al., 2006b).

>80% removal	80-50% removal	50-20% removal	<20% removal
Acetaminophen	Benzo(a)pyrene	Atrazine	TCEP
Androstenedione	DDT	Iopromide	Lindane
Caffeine	DEET	Meprobamate	Musk ketone
Carbamazepine	Diazepam		
Diclofenac	Dilantin		
Erythromycin	Fluorine		
Estradiol	Ibuprofen		
Estriol	Metolachlor		
Estrone			
Ethinylestradiol			
Fluoxetine			
Galaxolide			
Gemfibrozil			
Hydrocodone			
Naproxen			
Oxybenzone			
Pentoxifylline			
Progesterone			
Sulfamethoxazole			
Testosterone			
Triclosan			
Trimethoprim			

Ozonation removal rates depend on the specific pharmaceutical (Figure A-8). For example, ozonation at 0.5 mg/l will reduce diclofenac and carbamazepine concentrations by 97%, while reducing clofibric acid by only 10-15%. Extremely high doses of ozone (2.5-3.0 mg/l) resulted in less than a 40% reduction of clofibric acid. An ozone concentration of 1.0 to 1.5 mg/l reduced primidone and bezafibrate by 50%, but ozonation never achieved complete removal even at ozone concentrations of 3.0 mg/l. The reactivity of organic compounds with ozone depends on the reactivity of the benzene rings (Ternes et al., 2002).

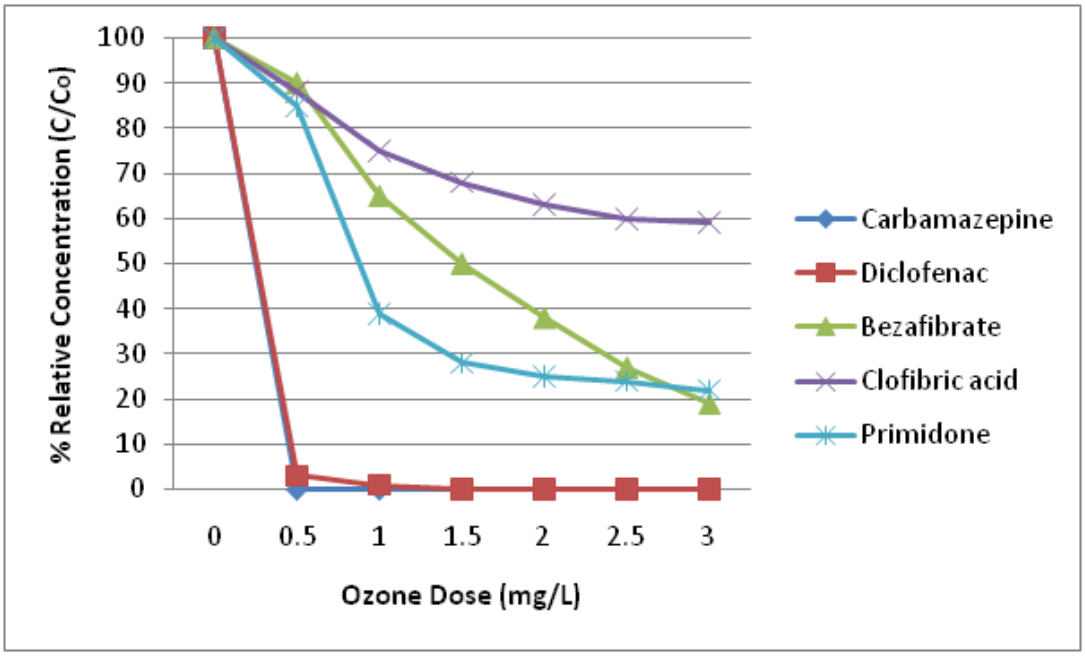

Figure A-8. Removal of target pharmaceuticals using varied ozone concentrations (Ternes et al., 2002).

Biosolids

The solid byproducts produced during wastewater treatment are commonly called sewage sludge. After additional treatment to remove pathogens, nutrients, and metals, they are classified as biosolids. Biosolids may be land-applied for beneficial use. Approximately 50% of the biosolids generated in the U.S. are land-applied; the other half are either sent to the landfill or incinerated. Researchers have documented the presence of PPCPs in biosolids (Kinney et al., 2006b).

On-Site Sewage Systems

Conn et al. (2006) conducted a study to characterize the occurrence and removal efficiencies of PPCPs in on-site sewage systems wastewater. They sampled 30 on-site wastewater treatment systems in Colorado, including both residential and nonresidential systems. They analyzed on-site sewage system effluent for 24 organic contaminants including pharmaceuticals and endocrine-disrupting compounds. The researchers detected 88% percent of the 24 organic

compounds during the course of the study, and detected several compounds in every effluent sample analyzed. They reported that six compounds (caffeine, coprostanol, cholesterol, EDTA, 4-methylphenol and sigmaNPEC) were detected in all of the anaerobic effluent samples. Additionally, five other compounds (4-thylphenol, NTA, 4-nonylphenol, sigmaNPEO, and triclosan) had median concentrations greater than the reporting limit. On-site sewage systems with additional aerobic treatment had lower median concentrations compared to the anaerobic tank treatment systems.

Reclaimed Water

Reclaimed water is treated wastewater that can be used for irrigation and other non-potable uses to extend water supplies. Tertiary-treated reclaimed water includes a set of treatment processes that provides a higher level of treatment and a higher degree of reliability than conventional WWTPs. The water released from a reclaimed water facility is designed to meet the quality standards for its intended use. Analysis of tertiary-treated reclaimed water indicate that these facilities can consistently produce water that is of a chemical quality comparable to that of drinking water for most parameters, including heavy metals, organic chemicals, pesticides, and disinfection by-products (Crook, 1998; EPA, 2004).

Reclaimed water is an important water resource with over 3,300 reclaimed water projects worldwide (Rodriguez et al., 2009). Endocrine-disrupting chemicals have been detected in reclaimed waters and in receiving waters where reclaimed water is released (Kolpin et al., 2002). Studies also find PPCPs in reclaimed water and at sites where reclaimed water is used (Kinney et al, 2006a); however, these concentrations are much lower than at conventional WWTP outfalls.

Kinney et al. (2006a) found some pharmaceuticals persist in the soil for several months after irrigation. Erythromycin, carbamazepine, fluoxetine, and diphenhydramine were present in the soils prior to irrigation. The researchers concluded that these chemicals persisted in the soils through adsorption from the previous irrigation season.

Rodriguez et al. (2009) evaluated the available epidemiological and toxicological studies involving potable reuse. Table A-4 summarizes their findings from the literature. PPCPs were included in the set of contaminants evaluated. The conclusion drawn is that the risk from reclaimed water projects is similar, or less than the risks from conventional drinking water sources. Additionally, no significant health risks were identified from these studies. In Denver, a treatment efficiency study of a reclaimed water project dosed the water with 15 organic compounds at a strength 100 times the normal treatment plant influent, and demonstrated that contaminants were removed to non-detectable levels.

Olivieri et al. (1998) investigated water from a reclaimed water facility in San Diego, in which 138 organic compounds plus other inorganic chemicals were analyzed. These researchers found no significant non-carcinogenic health risks, and the carcinogenic risks were 1,000 times less than the public water supply.

Table A-4. Epidemiological and toxicological findings from reclaimed water use.

Project	Findings
Orange County Water District, California	• Carcinogenic risk associated with consumption of recycled water was lower than other drinking water sources. • Water treated with microfiltration and reverse osmosis was safe for consumption and improved the groundwater quality.
Denver Potable Water Demonstration Project, Colorado	• No adverse toxicological health effects were detected. • Recycled water quality was better than the Denver drinking water quality for organic compounds. • Multi-barrier process removed the most number of contaminants to non-detectable levels.
Montebello Forebay Groundwater Recharge Project, California	• Industrial organic contaminants were higher in the recycled water, but below EPA standards. • Recycled water had no measureable impact on groundwater quality or human health.
Tampa Water Resource Recovery Project, Florida	• Recycled water did not present significant toxicological risks. • Panel of water quality and health experts concluded that recycled water is safe for human consumption.
San Diego Water Repurification Project, California	• Tests showed some mutagenic activity, but less than drinking water. • No significant health risk from non-carcinogenic chemicals. • Risk from human consumption of recycled water for bis(ethylhexyl)phthalate is 40 times lower than drinking water.

Modified from Rodriguez et al., 2009.

The level of treatment of reclaimed water varies depending on the intended use. In the U.S., there has been more than 40 years of experience with using reclaimed water with no known deleterious health effects. During a study investigating six large-scale reclamation projects, it was found that indirect potable reclaimed water met drinking water standards and was of a better quality and lower risk than potable water supplies (Olivieri, 2008). It was determined that advance treatment processes such as reverse osmosis or multiple barriers are the key to public health protection.

Reclaimed water facilities do a far superior job of removing contaminants, including PPCPs, than conventional WWTPs (Cooperative Research Centre, 2007). A review of the literature indicates that reclaimed water is safe for public contact and almost any use, except direct potable use. Additionally, there is no evidence to show that endocrine-disrupting compounds and PPCPs present a health risk from non-potable reclaimed water applications (Rock, 2008).

Daughton (2004) contends that the controversy over the use of reclaimed water stems from fundamental inaccuracies, misrepresentation, or oversimplification of the water cycle and its importance. Unplanned or incidental use of reclaimed water for beneficial uses, including drinking water, has occurred long before the idea of reclaiming wastewater. Indirect reuse already occurs when wastewater is discharged to land or streams as part of the traditional treatment process. However, this indirect use of reclaimed water receives less treatment and undergoes less redundancy, making it a lower-quality and less-reliable product.

Wetland Treatment

Constructed wetlands are a common treatment process used in the reclaimed water process. The USGS investigated contaminant attenuation in wetland treatment, including bisphenol A, caffeine, 4-nonylphenol, and triclosan (Table A-5). The researchers found high levels of contaminant removal in the wetlands with the average hydraulic retention time of 3.5 days (Barber et al., 2006).

Table A-5. Concentrations and removal of contaminants by wetland treatment.

Contaminant	Summer			Winter		
	Inlet Conc.	Outlet Conc.	% Removal	Inlet Conc.	Outlet Conc.	% Removal
BPA	120 ng/l	25 ng/l	79%	120 ng/l	104 ng/l	13%
Caffeine	490 ng/l	181 ng/l	63%	650 ng/l	87 ng/l	87%
4-nonylphenol	0.66 µg/l	0.42 µg/l	37%	0.64 µg/l	0.37 µg/l	42%
Triclosan	81 ng/l	86 ng/l	-6%	130 ng/l	92 ng/l	29%

(Barber et al., 2006.)

Treatment Summary

In summary, no single treatment process effectively removes 100% of the PPCPs. Some treatment processes effectively reduce some pharmaceuticals down to very low levels, while other pharmaceuticals remain resilient. A comparison of wastewater treatment removal efficiencies is presented in Table A-6.

Table A-6. Comparison of wastewater treatment concentrations (ng/l) for a select set of pharmaceuticals.
(Modified from Snyder et al., 2006a; Drury et al., 2006; Ternes et al., 2002; Heberer et al., 2004.)

Chemical	Influent	Primary	Secondary	Micro-filtra-tion	Ultra-filtration	PAC	EDR	MBR	RO	Double pass RO	Ozone
Acetaminophen	21,950	4095	<20	10	<10	53	3.4	<1.0	<1.0	<1.0	nd
Caffeine	58,550	6775	<20	6125	14	44	<10	<1.0	16	1.2	nd
Carbamazepine	299	138	110	271	147	35	18	205	<1.0	<1.0	<0.5
DEET	690	168	104	3365	103	18	112	37	3.4	<1.0	10
Erythromycin	479	9.4	336	507	357	17	<1.0	96	<1.0	<1.0	nd
Estradiol	<100	<1.0		<1.0		42	<1.0	<1.0	<1.0		nd
Estriol	226	67		<5.0		40	<5.0	<1.0	<5.0		nd
Ibuprofen	70,350	641	19	422		nd	5.4	4	<1.0	<1.0	nd
Meprobamate	520	92	693	341	715	19	71	236	<1.0	<1.0	97
Naproxen	21,000	599	<20	1205	17	2	<1.0	26	2	<1.0	<0.5
Oxybenzone	896	181	48	60	26	79	3.8	3.1	1.9	<1.0	nd
Sulfamethoxazole	234	103	90	805	56	23	<1.0	265	2	<1.0	3.2
TCEP	464	151	189	467	219	15	127	186	1.9	1.3	352
Triclosan	4,030	176	29	424	<10	71	<1.0	7.6	<1.0	<1.0	<1.0
Trimethoprim	699	144	186	409	158	72	<1.0	15	<1.0	<1.0	4.4

EDR - Electrodialysis reversal.

Table A-7 summarizes the relative cost of different treatment systems and their relative use in Washington State.

Table A-7. Wastewater treatment cost effectiveness summary for Washington State (Jones, 2008).

Treatment	Number of Facilities in Washington State	Relative Cost	Relative Effectiveness
Primary	100%	--	--
Secondary	100%	--	--
Filtration	20%	--	--
Activated Sludge	--	--	--
Microfiltration	0%	very expensive	poor
Ultrafiltration	0%	very expensive	poor
Nanofiltration	0%	very expensive	excellent
Granular Activated Carbon (GAC)	0%	--	excellent
Powdered Activated Carbon (PAC)	0%	--	excellent
Reverse osmosis (RO)	--	very expensive	excellent
Riverbank filtration	--	--	poor
Membrane bioreactor (MBR)	15%	very expensive	--
Electrodialysis reversal (EDR)	0%		--
Ozonation	few	expensive	excellent
Flocculation	--	--	poor

Source Reduction

A few mechanisms for preventing the release of PPCPs in the environment are reviewed below: pharmaceutical take-back programs, controlled disposal, and education.

Pharmaceutical Take-Back Programs

A Washington State coalition created PH:ARM to provide a simple, low-cost, and secure pharmaceutical take-back system for unwanted medications from household sources. The system is operating as a pilot program, year-round at local pharmacies and nursing homes. The designers hope to expand the pilot program throughout the state and nationwide. The program focuses on managing waste medications to prevent pharmaceutical pollution while improving public safety.

The coalition includes the Washington Board of Pharmacy, King County Local Hazardous Waste Program, Snohomish County Solid Waste Management Division, Northwest Product Stewardship Council, Seattle-King County Public Health, and the Washington State Department of Ecology.

The coalition recognized the need for a practical solution that would not involve hazardous waste facilities for common household items. The take-back model is an approach which relies on the interactions between consumers, retailers, and wholesalers of pharmaceuticals. The goal is to include over-the-counter, prescription, and controlled substances in the collection scheme

because source-separation poses problems for consumers and collection locations (PH:ARM, 2007).

This program began in 2005, with two Washington state businesses (Group Health Cooperative and Bartell Drug Company) participating in the pilot development. Additionally two leaders for producer responsibility and pollution prevention participated in the pilot development: Washington Citizens for Resource Conservation and the Northwest Pollution Prevention Resource Center. The state Department of Social and Health Services, another partner, helped the team with the challenges at nursing homes, adult family homes, and boarding homes. In the first year, over 3,300 lbs of unwanted medicines were safely collected (PH:ARM, 2007).

It is estimated that once a statewide pharmaceutical take-back program is fully implemented, 66,000 pounds of drugs could be collected every year from Washington's 6.2 million citizens (PH:ARM, 2007). A King County survey found that 74% of the respondents would be willing to properly dispose of their unused and unwanted medicines by using a take-back-program at a local pharmacy, which was determined as the most convenient location (PH:ARM, 2007). Take-back programs provide the optimal disposal option for residential drugs. Pharmaceuticals are also sent to hazardous waste incinerators where complete destruction occurs. Disposal to sewers or landfills does not remove the drugs from the environment.

Disposal Options

Disposal options for PPCPs are listed in the following order of preference (PH:ARM, 2007):

1. Take advantage of community pharmaceutical take-back programs.
2. Take prescription drugs out of their original container, mix them with an undesirable substance such as kitty litter or coffee grounds, and put them in an impermeable (watertight) container, then throw the container in the garbage.
3. Do not flush drugs down the toilet or drain.

Education

Educational programs have also been established worldwide to minimize the volume of unwanted pharmaceuticals. These include general public education, developing options for smart consumer choices, educating doctors and pharmacists, and reducing prescription fraud and illegal pharmaceutical use.

Indicator Parameters

Indicator parameters are useful tools for identifying locations which are susceptible to pharmaceutical contamination. They are a convenient, economical, and efficient means of quickly determining where potential contamination exists. The key with a good indicator parameter is constant and reliable use by the general population, its presence in wastewater, and the analytical capability to detect the indicator parameter in wastewater and the environment. The indicator must also have conservative transport characteristics and be relatively persistent in the environment.

The characteristics of the individual chemicals determine whether they would be suitable as indicator parameters. PPCPs which are relatively insoluble, do not readily degrade in the environment, and are present at detectable concentrations should be considered as indicator parameters (Motzer, 2006).

Literature Recommendations

Barnes et al. (2002) conducted the first comprehensive national reconnaissance testing of pharmaceuticals in the United States. This study provides a statistical basis for determining indicator parameters.

Zdwadzkas (2006) developed an abbreviated list of indicator parameters based on the data collected in Barnes et al. (2002). He determined that monitoring for the four parameters listed in Table A-8 would result in capturing 91% of the sites where pharmaceuticals were detected. Additionally, if the last two parameters are added, the results would capture 96% of the sites where pharmaceuticals were detected in surface water across the United States.

Table A-8. Indicator parameters based on surface water occurrence (Zdwadzkas, 2006).

Captures 91% of detected sites:
Coprostanol
Tri(2-chloroethyl)phosphate (TCEP)
4-nonylphenol
Bis-phenol A
Captures 96% of detected sites if these are added:
Cholesterol
Caffeine

EPA and USGS scientists designed a national study to determine potential indicator parameters of human waste. The goal of the project was to sample upstream and downstream locations near ten WWTPs across the U.S. for 110 organic chemicals to determine if a correlation exists between the presence of these chemicals and known human waste sources. The number of compounds at the detected sites ranged from 3 in a background location, to 50 in a WWTP effluent sample (Glassmeyer et al., 2005).

At almost every location, the downstream concentrations were higher than the upstream concentrations. Additionally, the concentration and presence of chemicals decreased downstream as the distance from the WWTP effluent outfall increased. The study concluded that the chemicals listed in Table A-9 would make useful indicator parameters.

Table A-10 lists the most frequently detected compounds during this national study (Glassmeyer et al., 2005). These are all considered candidate indicators.

Table A-9. Indicator parameters based on upstream and downstream concentrations near 10 wastewater treatment plants across the U.S. (Glassmeyer et al., 2005).

Indicators	Reason
ethyl citrate	Dramatic change in concentrations between the upstream site, the wastewater effluent, and the downstream site.
galaxolide	Dramatic change in concentrations between the upstream site, the wastewater effluent, and the downstream site.
tonalide	Dramatic change in concentrations between the upstream site, the wastewater effluent, and the downstream site.
carbamazepine	Pharmaceuticals used extensively only by humans.
diphenhydramine	Pharmaceuticals used extensively only by humans.
caffeine	Drug used extensively only by humans.
coprostanol	Fecal sterol from human sources. Exhibited the most changes between the upstream site, the wastewater effluent, and the downstream site.

Table A-10. Detection frequency near 10 wastewater treatment plants across the U.S. *(Modified from Glassmeyer et al., 2005).*

Chemical Name	Detection Frequency (%)
cotinine	92.5
cholesterol	90
carbamazepine	82.5
tonalide (AHTN)	80
tri(2-chloroethyl) phosphate (TCEP)	75
codeine	72.5
ethyl citrate	72.5
sitosterol	72.5
sulfamethoxazole	72.5
caffeine	70
ethanol, 2-butoxy-phosphate	70
N-N-diethyltoluamide (DEET)	70
tributylphosphate	70
benzophenone	67.5
diltiazem	67.5
4-nonylphenol diethoxylate	62.5
4-nonylphenol monoethoxylate	62.5
triclosan	62.5
coprostanol	60
trimethoprim	60
dehydronifedipine	57.5
galaxolide (HHCB)	57.5
diphenhydramine	55
acetaminophen	50
diazinon	47.5
5-methyl-1 H-benzotriazole	45
phenol	40
triphenyl phosphate	37.5
1,7-Dimethylxanthine	35
4-octylphenol diethoxylate	32.5
bisphenol A	30
1,4-dichlorobenzene	27.5

Caffeine as an Indicator

Caffeine is an anthropogenic chemical only used by humans, and therefore, is a good indicator of human waste. Caffeine is also contained in numerous PPCPs.

Buerge et al. (2003) conducted an in-depth study of the appropriateness of using caffeine as an indicator parameter in surface waters. Table A-11 describes the characteristics which make caffeine a useful indicator parameter for the presence of other anthropogenic contaminants. This study evaluated caffeine concentrations in WWTP influents and effluents, receiving surface waters, pristine mountain lakes, and moderately polluted lakes and rivers in the Swiss midland region. Mass balances and a quantitative correlation between caffeine concentrations and the anthropogenic burden were determined to definitely conclude that caffeine is a suitable indicator.

Table A-11. Caffeine facts (Buerge et al., 2003).

Caffeine Facts	Concentration
Wastewater influent concentration	7 µg/l to 73 µg/l
Wastewater effluent concentration	0.03 µg/l to 9.5 µg/l
Removal rates in treatment process	81% to 99.9%
Global average consumption	70 mg/person/day
U.S. average consumption	210 mg/person/day
Average person discharges	15.8 mg/day (+/-3.8 mg)
Coffee caffeine content	100 mg
Tea caffeine content	50 mg
Cacao caffeine content	10 mg
Cola caffeine content	40 mg
Swiss lakes and rivers concentration	6 ng/l to 250 ng/l
Pristine mountain lakes concentration	<2 ng/l

Summary of Indicator Parameters Recommended in the Literature

Many of the environmental monitoring studies cited in this literature review focused on pharmaceuticals which are most prevalent or which have been detected most frequently in the environment. Table A-12 lists chemicals which were most frequently cited in the literature as suitable indicator parameters.

Table A-12. Recommended pharmaceutical indicator parameters from reviewed literature.

Chemical Name	References which Identified Chemicals as Indicator Parameters
1,4-dichlorobenzene	Barnes et al., 2002; Glassmeyer et al., 2005.
1,7-dimethylxanthine	Kinney et al., 2006a; Barnes et al., 2002; Glassmeyer et al., 2005.
2,6-dimethylnaphthalene	Motzer, 2006.
3,4-dichlorophenyl isocyanate	Glassmeyer et al., 2005.
4-methyl phenol	Barnes et al., 2002.
4-nonylphenol	Motzer, 2006; Barnes et al., 2002.
4-nonylphenol diethoxylate	Barnes et al., 2002; Glassmeyer et al., 2005.
4-nonylphenol monoethoxylate	Barnes et al., 2002; Glassmeyer et al., 2005.
4-octylphenol diethoxylate	Barnes et al., 2002; Glassmeyer et al., 2005.
4-octylphenol monoethoxylate	Barnes et al., 2002.
5-methyl-1 H-benzotriazole	Barnes et al., 2002; Motzer, 2006; Glassmeyer et al., 2005.
acetaminophen	Kinney et al., 2006a; Barnes et al., 2002; Glassmeyer et al., 2005.
benzophenone	Motzer, 2006; Glassmeyer et al., 2005.
bis-phenol A	Barnes et al., 2002; Glassmeyer et al., 2005.
caffeine	Kinney et al., 2006a; Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
carbamazepine	Kinney et al., 2006a; Motzer, 2006; Glassmeyer et al., 2005.
carbaryl	Motzer, 2006.
cholesterol	Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
cimetidine	Kinney et al., 2006a.
codeine	Kinney et al., 2006a; Glassmeyer et al., 2005.
coprostanol	Barnes et al., 2002; Glassmeyer et al., 2005.
cotinine	Kinney et al., 2006a; Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
dehydronifedipine	Kinney et al., 2006a; Glassmeyer et al., 2005.
diazinon	Barnes et al., 2002; Glassmeyer et al., 2005.
diltiazem	Kinney et al., 2006a; Glassmeyer et al., 2005.
diphenhydramine	Kinney et al., 2006a; Glassmeyer et al., 2005.
erythromycin	Kinney et al., 2006a; Barnes et al., 2002.
estriol	Barnes et al., 2002.

Chemical Name	References which Identified Chemicals as Indicator Parameters
ethanol, 2-butoxy-phosphate	Barnes et al., 2002; Glassmeyer et al., 2005.
ethyl citrate	Glassmeyer et al., 2005.
fluoranthene	Barnes et al., 2002.
fluoxetine	Kinney et al., 2006a.
galaxolide (HHCB)	Glassmeyer et al., 2005.
gemfibrozil	Kinney et al., 2006a.
isophorone	Motzer, 2006.
lincomycin	Barnes et al., 2002.
miconazole	Kinney et al., 2006a.
N-N-diethyltoluamide (DEET)	Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
pentachlorophenol	Glassmeyer et al., 2005.
phenol	Barnes et al., 2002; Glassmeyer et al., 2005.
phthalic anhydride	Barnes et al., 2002.
pyrene	Barnes et al., 2002.
ranitidine	Glassmeyer et al., 2005.
salbutamol (albuterol)	Kinney et al., 2006a.
sitosterol	Glassmeyer et al., 2005.
sulfamethoxazole	Kinney et al., 2006a; Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
tetrachloroethylene	Barnes et al., 2002.
thiabendazole	Kinney et al., 2006a.
tonalide (AHTN)	Glassmeyer et al., 2005.
tri(2-chloroethyl) phosphate (TCEP)	Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
tri(dichloroisopropyl)phosphate	Glassmeyer et al., 2005.
tributylphosphate	Motzer, 2006; Glassmeyer et al., 2005.
triclosan	Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005.
trimethoprim	Kinney et al., 2006a; Barnes et al., 2002; Glassmeyer et al., 2005.
triphenyl phosphate	Glassmeyer et al., 2005.
warfarin	Kinney et al., 2006a.

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Appendix B. Organic Compounds Analyzed During This Study

A total of 172 organic compounds were analyzed using three EPA methods during this 2008 Ecology/ EPA study. These compounds are listed in Tables B-1 – B-3.

Table B-1. Method 1694: 72 Pharmaceuticals or Personal Care Products in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS. (Note: two antibiotics were added to this list in 2009: ampicillin and erythromycin).

PPCP Analyte	CAS #	Classification
1,7-Dimethylxanthine	611-59-6	Antispasmodic, caffeine metabolite
4-Epianhydrochlortetracycline (EACTC)	158018-53-2	Chlorotetracycline degradate
4-Epianhydrotetracycline (EATC)	4465-65-0	Chlorotetracycline degradate
4-Epichlortetracycline (ECTC)	14297-93-9	Chlorotetracycline degradate
4-Epioxytetracycline (EOTC)	14206-58-7	Oxytetracycline degradate
4-Epitetracycline (ETC)	23313-80-6	Tetracycline degradate
Acetaminophen	103-90-2	Antipyretic, Analgesic
Albuterol	18559-94-9	Antiasthmatic
Anhydrochlortetracycline (ACTC)	13803-65-1	Chlorotetracycline degradate
Anhydrotetracycline (ATC)	4496-85-9	Chlorotetracycline degradate
Azithromycin	83905-01-5	Macrolide antibiotic
Caffeine	58-08-2	Stimulant
Carbadox	6804 07 05	Quinoxaline antibiotic
Carbamazepine	298-46-4	Anticonvulsant
Cefotaxime	63527-52-6	Cephalosporin antibiotic
Chlortetracycline (CTC)	57-62-5	Tetracycline antibiotic
Cimetidine	51481-61-9	Anti-acid reflux
Ciprofloxacin	85721-33-1	Quinoline antibiotic
Clarithromycin	81103-11-9	Macrolide antibiotic
Clinafloxacin	105956-97-6	Quinoline antibiotic
Cloxacillin	61-72-3	β-lactam antibiotic
Codeine	76-57-3	Opiate
Cotinine	486-56-6	Nicotine metabolite
Dehydronifedipine	67035-22-7	Nifedipine metabolite
Demeclocycline	127-33-3	Tetracycline antibiotic
Digoxigenin	1672-46-4	Immunohistochemical Marker Steroid
Digoxin	20830-75-5	Cardiac glycoside
Diltiazem	42399-41-7	Antihypertensive
Diphenhydramine	58-73-1	Antihistamine
Doxycycline	564-25-0	Tetracycline antibiotic
Enrofloxacin	93106-60-6	Tetracycline antibiotic
Erythromycin anhydrate	59319-72-1	Macrolide antibiotic

PPCP Analyte	CAS #	Classification
Flumequine	42835-25-6	Quinolone antibiotic
Fluoxetine	54910-89-3	SSRI antidepressant
Gemfibrozil	25812-30-0	Antilipemic
Ibuprofen	15687-27-1	Analgesic
Isochlortetracycline (ICTC)	514-53-4	Chlorotetracycline degradate
Lincomycin	154-21-2	Lincosamide antibiotic
Lomefloxacin	98079-51-7	Quinoline antibiotic
Metformin	657-24-9	Anti-diabetic drug
Miconazole	22916-47-8	Antifungal agent
Minocycline	10118-91-8	Tetracycline antibiotic
Naproxen	22204-53-1	Non-steroidal anti-inflammatory drug
Norfloxacin	70458-96-7	Quinoline antibiotic
Norgestimate	35189-28-7	Hormonal contraceptives
Ofloxacin	82419-36-1	Quinoline antibiotic
Ormetoprim	6981-18-6	Macrolide antibiotic
Oxacillin	66-79-5	β-lactam antibiotics
Oxolinic acid	14698-29-4	Quinolone antibiotic
Oxytetracycline (OTC)	79-57-2	Tetracycline antibiotic
Penicillin G	61-33-6	β-lactam antibiotics
Penicillin V	87-08-1	β-lactam antibiotics
Ranitidine	66357-35-5	Anti-acid reflux
Roxithromycin	80214-83-1	Macrolide antibiotic
Sarafloxacin	98105-99-8	Fluoroquinolone antibiotic
Sulfachloropyridazine	80-32-0	Sulfonamide antibiotic
Sulfadiazine	68-35-9	Sulfonamide antibiotic
Sulfadimethoxine	122-11-2	Sulfonamide antibiotic
Sulfamerazine	127-79-7	Sulfonamide antibiotic
Sulfamethazine	57-68-1	Sulfonamide antibiotic
Sulfamethizole	144-82-1	Sulfonamide antibiotic
Sulfamethoxazole	723-46-6	Sulfonamide antibiotic
Sulfanilamide	63-74-1	Sulfonamide antibiotic
Sulfathiazole	72-14-0	Sulfonamide antibiotic
Tetracycline (TC)	60-54-8	Tetracycline antibiotic
Thiabendazole	148-79-8	Fungicide and parasiticide
Triclocarban	101-20-2	Antimicrobial, disinfectant
Triclosan	3380-34-5	Antimicrobial, disinfectant
Trimethoprim	738-70-5	Pyrimidine antibiotic
Tylosin	1401-69-0	Macrolide antibiotic
Virginiamycin	11006-76-1	Macrolide antibiotic
Warfarin	81-81-2	Anticoagulant

Table B-2. Method 1698: 27 Steroids and Hormones in Water, Soil, Sediment, and Biosolids by HRGC/HRMS.

Analyte	CAS #	Classification
Androstenedione	63-05-8	Anabolic agent
Androsterone	53-41-8	Hormone metabolite
Equilenin	517-09-9	Hormone replacement
Equilin	474-86-2	Hormone replacement
17a-Ethynyl Estradiol (EE2)	57-63-6	Ovulation inhibitor
Desogestrel	54024-22-5	Ovulation inhibitor
Mestranol	72-33-3	Ovulation inhibitor
Norethindrone	68-22-4	Ovulation inhibitor
Norgestrel	6533-00-2	Ovulation inhibitor
Campesterol	474-62-4	Phytosterol (plant sterol)
beta-Sitosterol	83-46-5	Phytosterol (plant sterol)
Stigmasterol	83-48-7	Phytosterol (plant sterol)
Beta-Stigmastanol	83-45-4	Phytosterol (plant sterol)
17a-Estradiol	57-91-0	Sex hormone
17b-Estradiol (E2)	50-28-2	Sex hormone
Estriol (E3)	50-27-1	Sex hormone
Estrone (E1)	53-16-7	Sex hormone
Progesterone	57-83-0	Sex hormone
Testosterone	58-22-0	Sex hormone
17a-Dihydroequilin	651-55-8	Sterol
Cholestanol	80-97-7	Sterol
Cholesterol	57-88-5	Sterol
Desmosterol	313-04-2	Sterol
Ergosterol	57-87-4	Sterol
b-Estradiol-3-benzoate	50-50-0	Sterol
Coprostanol	360-68-9	Sterol
Epi-Coprostanol	516-92-7	Sterol

Table B-3. Method 8270d: 73 Semi-Volatile Organics.

Analyte	CAS #	Classification
1,2,4-Trichlorobenzene	120-82-1	SVOC
1,2-Dichlorobenzene	95-50-1	SVOC
1,2-Diphenylhydrazine	122-66-7	Dye intermediate
1,3-Dichlorobenzene	541-73-1	SVOC
1,4-Dichlorobenzene	106-46-7	SVOC
1-Methylnaphthalene	90-12-0	PAH, dye, plastic, pesticide
2,2'-Oxybis[1-chloropropane]	108-60-1	Fumigant
2,4,5-Trichlorophenol	95-95-4	SVOC
2,4,6-Trichlorophenol	88-06-2	SVOC
2,4-Dichlorophenol	120-83-2	SVOC, herbicide intermediate
2,4-Dimethylphenol	105-67-9	Pesticide, antioxidant
2,4-Dinitrophenol	51-28-5	Dyestuff, pharmaceutical, pesticide, explosive
2,4-Dinitrotoluene	121-14-2	Explosive, dye, plastic
2,6-Dinitrotoluene	606-20-2	Explosive, dye, plastic
2-Chloronaphthalene	91-58-7	SVOC, electrical industry
2-Chlorophenol	95-57-8	SVOC, disinfectant
2-Methylnaphthalene	91-57-6	PAH, dye, plastic, pesticide
2-Methylphenol	95-48-7	Cresol; disinfectant, deodorizer, antiseptic, wood preservative
2-Nitroaniline	88-74-4	Dyestuff, pharmaceutical
2-Nitrophenol	88-75-5	Dye, rubber, fungicide
3-Nitroaniline	99-09-2	Dyestuff, pharmaceutical
4,6-Dinitro-2-Methylphenol	534-52-1	Pesticide, herbicide, insecticide
4-Bromophenyl-Phenylether	101-55-3	Research purposes, flame retardant
4-Chloro-3-Methylphenol	59-50-7	Cresol; disinfectant, deodorizer, antiseptic, preservative
4-Chloroaniline	106-47-8	Antimicrobial
4-Chlorophenyl-Phenylether	7005-72-3	Dielectric fluid
4-Methylphenol	106-44-5	Cresol; disinfectant, deodorizer, antiseptic, wood preservative
4-Nitroaniline	100-01-6	Dyestuff, pharmaceutical
4-Nitrophenol	100-02-7	Drug, fungicide, dye
Acenaphthene	83-32-9	PAH
Acenaphthylene	208-96-8	PAH
Anthracene	120-12-7	PAH
Benzo(a)anthracene	56-55-3	PAH
Benzo(a)pyrene	50-32-8	PAH
Benzo(b)fluoranthene	205-99-2	PAH
Benzo(ghi)perylene	191-24-2	PAH
Benzo(k)fluoranthene	207-08-9	PAH
Benzoic Acid	65-85-0	Food preservative

Analyte	CAS #	Classification
Benzyl Alcohol	100-51-6	Solvent, bacterial inhibitor, antipruritic, personal care products
Bis(2-Chloroethoxy)Methane	111-91-1	Industrial precursor for polysulfide polymers
Bis(2-Chloroethyl)Ether	111-44-4	Air pollutant likely from combustion
Bis(2-Ethylhexyl) Phthalate	117-81-7	Plasticizer, hydraulic fluid, dielectric fluid
Bis-phenol A	80-05-7	Plastic products, epoxy resign, flame retardant, fungicide
Butylbenzylphthalate	85-68-7	Dyestuff, pharmaceutical
Carbazole	86-74-8	Synthesis of dyes, pharmaceuticals, plasticizers
Chrysene	218-01-9	PAH
Dibenzo(a,h)anthracene	53-70-3	PAH
Dibenzofuran	132-64-9	Dioxin, insecticide, plastic manufacture
Diethylphthalate	84-66-2	Plasticizer, personal care products
Dimethyl phthalate	131-11-3	Plasticizer, personal care products
Di-N-Butylphthalate	84-74-2	Plasticizer, personal care products
Di-N-Octyl Phthalate	117-84-0	Plasticizer, personal care products
Fluoranthene	206-44-0	PAH
Fluorene	86-73-7	PAH, dye, plastic, pesticide
Hexachlorobenzene	118-74-1	Fungicide, pesticide, explosives, rubber
Hexachlorobutadiene	87-68-3	Chlorine solvent
Hexachlorocyclopentadiene	77-47-4	Pesticide, flame retardant, plastic, dye
Hexachloroethane	67-72-1	Smoke devices, aluminum manufacture, biocide, plastic
Indeno(1,2,3-cd)pyrene	193-39-5	PAH
Isophorone	78-59-1	Household and industrial chemical
Naphthalene	91-20-3	PAH, dye, plastic, pesticide
Nitrobenzene	98-95-3	Rubber, pesticide, dye, pharmaceutical, explosive, solvents, perfume
N-Nitrosodimethylamine	62-75-9	Rocket fuel, manufacturing byproduct
Nitrosodi-n-propylamine	621-64-7	Rubber byproduct, herbicide contaminant, smoke byproduct
N-Nitrosodiphenylamine	86-30-6	Industrial rubber compound
Pentachlorophenol	87-86-5	Biocide, disinfectant, wood preservative
Phenanthrene	85-01-8	PAH
Phenol	108-95-2	Antiseptic
Phenol, 4-Nonyl-	104-40-5	Surfactant
Pyrene	129-00-0	PAH
Retene	483-65-8	PAH
Tri(2-chloroethyl) phosphate	115-96-8	Fire retardant
Triethyl citrate	77-93-0	Food additive, plasticizer, pill coating

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Appendix C. Descriptions of Wastewater Treatment Plants

1. LOTT Alliance, Budd Inlet WWTP

LOTT Alliance
500 Adams St. NE
Olympia, WA 98501

The Lacey, Olympia, Tumwater, Thurston County (LOTT) Alliance provides wastewater treatment and reclaimed water production services for over 50,000 equivalent residential units, with a residential population of over 90,000 people (Figure C-1). Connections include homes, apartments, and commercial/industrial facilities served by the sewer utilities of Lacey, Olympia, and Tumwater. The vast majority of connections are residential. Commercial and industrial connections include colleges, hospitals, medical treatment facilities, and nursing homes.

The majority of wastewater flows through the LOTT system are treated at the central Budd Inlet WWTP. About 11 million gallons of wastewater flow through the Budd Inlet WWTP on an average day. During the wettest months, flows have averaged as high as 23.2 MGD.

The quality of the water LOTT facilities discharge is regulated by Ecology under an NPDES permit. This permit requires the Budd Inlet WWTP to achieve a seasonal monthly average total inorganic nitrogen limitation of 3 mg/L.

Figure C-1. Aerial photo of the Budd Inlet Wastewater Treatment Plant.

The treatment process at the Budd Inlet WWTP includes biological removal of nitrogen from the water (April to October) to prevent the nitrogen from feeding excessive algae growth after the treated water is discharged into marine waters at the southern end of Puget Sound. Nitrogen removal is accomplished by internal recycling of wastewater where nitrification and denitrification occurs in aerated and anaerobic zones within the four-stage biological treatment process shown in Figures C-2 and C-3.

As the wastewater is cleaned, remaining solid material is removed.

- *Thickening:* The material removed in the primary and secondary treatment processes is sent to the solids handling building to a Dissolved Air Flotation Thickener. The Thickener concentrates the solids and separates it from the liquid before it goes to the digesters.
- *Digestion:* The thickened solids are fed to the two primary digesters. The solids are heated, mixed, and held for at least 15 days to further reduce pathogens. This process also produces methane gas for beneficial reuse within the WWTP. The methane is used as fuel for boilers within the WWTP. The boilers produce hot water for the digesters and the high-voltage alternating current (HVAC) system.
- *Dewatering:* The digested biosolids are sent to a centrifuge for dewatering after their pathogens have been sufficiently reduced. This machine spins to create centrifugal force, which further separates liquids from the biosolids. As the biosolids leave the machine, they are carried via screw conveyor to a biosolids hauling truck.
- *Hauling and Beneficial Use:* The resulting biosolids are trucked to locations in Eastern Washington and Lewis County where they are used to fertilize pastureland, forests, and dry-land wheat. A small portion of Budd Inlet WWTP biosolids are used to produce compost.

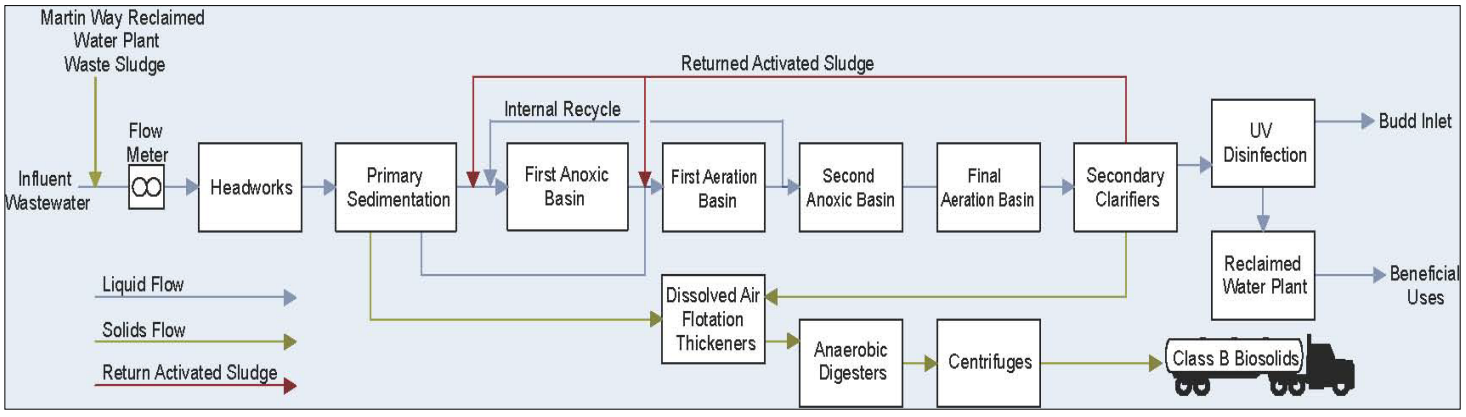

Figure C-2. Budd Inlet Treatment Plant and Reclaimed Water Plant Overall Process Schematic.

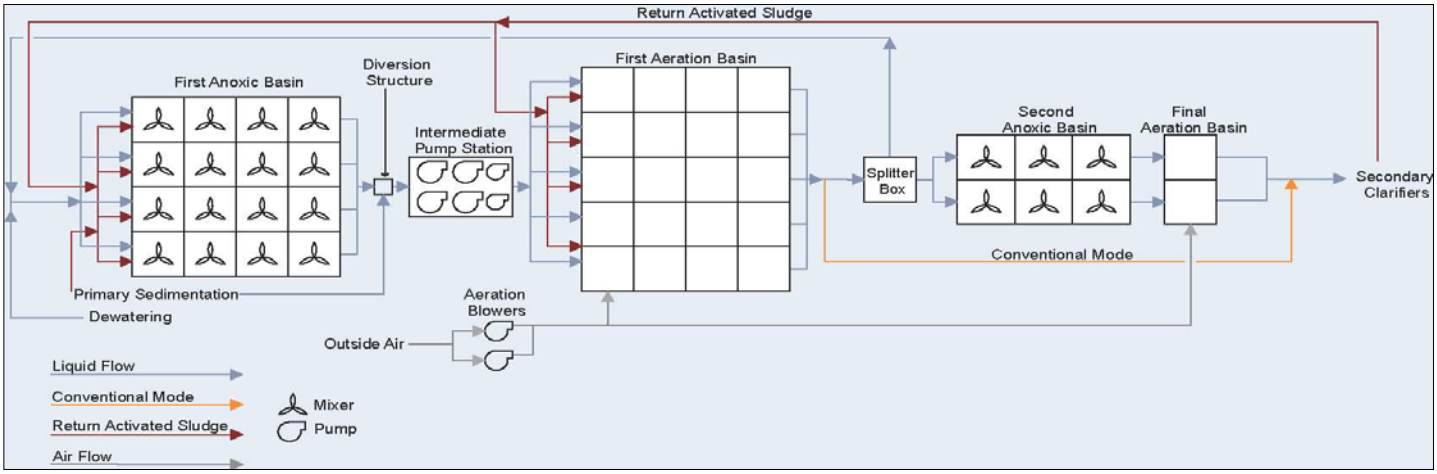

Figure C-3. Budd Inlet WWTP Anoxic and Aeration Basin Recycle Schematic.

Budd Inlet WWTP's biological nutrient-removal system is operated to optimize total inorganic nitrogen and biochemical oxygen demand (BOD) removal from the primary effluent. Primary effluent is combined with other recycle flows through a series of anoxic (low dissolved oxygen) basins and aeration (higher dissolved oxygen) basins. These basins are identified as the first anoxic, first aeration, second anoxic, and final aeration basins. To achieve the required nitrogen limits, flows are recycled inside the aeration basin system from the first aeration basin back to the first anoxic basin at a rate that is typically four times the WWTP's influent flow.

The second anoxic and final aeration basins (stages 3 and 4) provide the final biological denitrification and nitrification steps prior to settling and disinfection. Stages 3 and 4 consist of two trains, each with four cells. The first three cells of each train serve as the second anoxic zone, and the fourth cell as the final aeration zone. In the anoxic cells, additional nitrate removal is achieved. In the final aeration cells, the mixed liquor is aerated to further freshen the mixed liquor prior to the secondary clarifiers.

Reclaimed Water

A portion of the final effluent from the Budd Inlet WWTP is routed through additional treatment to meet Washington State Class A reclaimed water treatment standards. The reclaimed water plant is given a separate name for clarification, the Budd Inlet Reclaimed Water Plant (RWP). This treatment includes chemical addition and filtration through single-stage, continuous backwashing, upflow sand filters (from Parkson Corporation), and additional disinfection with chlorine. The reclaimed water is used for irrigation at various locations in the Olympia area.

2. LOTT Alliance, Martin Way Reclaimed Water Plant (RWP)

LOTT Alliance
111 Market Street
Olympia, WA 98501

A portion of the wastewater flowing to the Budd Inlet WWTP is diverted from the collection system and treated at the Martin Way "satellite" plant. Construction and operational startup of the Martin Way RWP was completed in 2006. The initial treatment capacity is 2 MGD with future capacity planned that may reach 5 MGD.

Treatment to meet the Washington State Class A reclaimed wastewater standard is accomplished using two-stage biological nutrient removal in bioreactors. After biological treatment, the wastewater is filtered through membranes in tanks that are separate from the bioreactors, followed by disinfection. Nitrification and denitrification are provided in the treatment system to meet a State Reclaimed Water permit monthly average limitation of 10 mg/L for total nitrogen, but the effluent routinely contains less than 5 mg/L total nitrogen. Solids removed during wastewater treatment at the Martin Way RWP are routed back into the sewer main where the solids then flow to the Budd Inlet WWTP. The process flow diagram is shown in Figure C-4.

The Class A reclaimed water produced at the Martin Way RWP is sent through three miles of purple pipe to the Hawks Prairie Reclaimed Water Ponds and Recharge Basins. The ponds consist of a series of five constructed wetland ponds, containing about 225,000 wetland plants. The ponds provide opportunities for public education, recognition, and acceptance of reclaimed water. Water from the constructed wetland ponds flows to groundwater recharge basins. From there, the water infiltrates through the soils to a shallow underground aquifer.

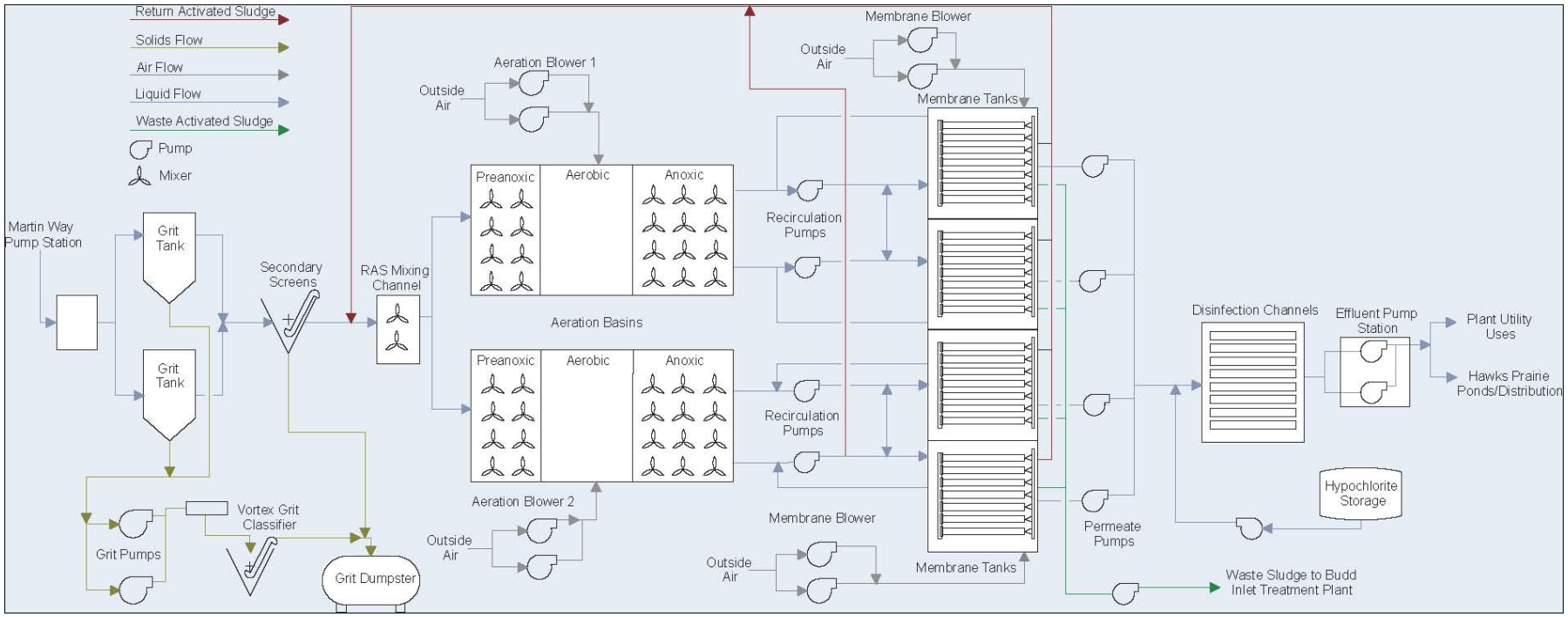

Figure C-4. Process flow diagram of the Martin Way Reclaimed Water Plant.

3. Chambers Creek WWTP

Public Works and Utilities
9850 Chambers Creek Road
University Place, WA 98467-1040

The Pierce County Chambers Creek WWTP (Figure C-5) employs activated sludge for secondary treatment and discharges treated effluent into the marine waters of Puget Sound via a diffused 760-foot-long outfall. The original facility began operation in 1984 to treat municipal wastewater. Since then, major upgrades to the WWTP have been installed to the disinfection process, headwords, biosolids processing, and aeration basins. The plant is currently designed to treat 28.7 MGD average monthly influent flow.

Figure C-5. Aerial photo of the Chambers Creek Wastewater Treatment Plant.

The wastewater treatment process is described below:

Step Screens

There are three step screens in the head works section of the plant. These screens build a mat of rags and solids. The screens move vertically in discrete steps according to computer control to remove trapped solids. The trapped solids go to a washing system to remove organics, then to a press to remove water. The washed and pressed solids are transported to a dumpster for disposal in a sanitary landfill.

Grit Tanks

There are three grit tanks in the head works. Grit tanks use air diffusers to keep most of the solids suspended in the wastewater while allowing sand and grit to settle to the bottom of the tank. The sand and grit is conveyed to a dumpster.

Primary Clarifiers

There are six primary clarifiers that are placed into service by the operation staff as needed based on flow. The primary influent channel is controlled by five motorized gate valves. Primary sludge pumping is controlled by blanket levels and density. The primary sludge solids are pumped by six piston pumps to the gravity belt thickeners. There are automatic skimmers installed on the clarifiers for skimming of grease. The grease is either pumped to the digesters or to a rotostrainer located in the head works.

Aeration Basins

There are five operational aeration basins: 1&3 and 2&4 have been combined and function as two aeration basins instead of the original design of four separate complete mix basins. Aeration basins 1&3 and 2&4 are designed for 6 MGD average flow each. All basins have been modified and divided into zones for Bioselection Nutrient Removal (BNR). The primary effluent flow has been modified to direct all the flow into AB1 and AB2 zone 1. All return activated sludge flows are also directed into zone 1 of these basins. Each basin has six sections or zones; three are anoxic, two are oxic, and one is polishing.

Aeration basins 5-6&7 have been divided into six zones and function in the same fashion as 1&3 and 2&4, with all primary effluent and return activated sludge being directed to zone one. The air system for all the aeration basins has been modified for the bioselector process.

Each aeration basin has an internal recycle pump installed that returns flows from zone 5 back to zones 1&3. The rate of return for the aeration basins is based on the design primary effluent flows to the aeration basin.

Secondary Clarifiers

After the mixed liquor leaves the aeration basins, it flows to the final clarifiers in two modes. Mixed liquor flows to final clarifiers 1&2 through an open channel and is delivered to the periphery of the clarifiers. Mixed liquor to final clarifiers 3&5 uses an open channel that routes flow into pipes that deliver it to the center well of the clarifiers. The flows to the final clarifiers can be divided evenly between the clarifiers in service or can be split unevenly as determined by the operator. When divided unevenly, the sum of the division must equal 100%.

The valves controlling the flow to the final clarifiers in service are controlled by the liquid level in the channel. As the liquid level increases, the valves open more but still maintain the desired split set by the operator. If the flow is more than the clarifiers can handle, the liquid level in the channel will rise, setting off a high channel alarm.

Digesters

The digestion system consists of three anaerobic digesters. Presently two digesters are operated in parallel as primary digesters. The third digester provides storage and balancing for feed to the dewatering system. Thickened solids from the gravity belts are pumped into the primary digesters. The digester content is heated and continuously mixed to create a homogeneous mixture of new solids and actively digesting solids. This process decreases the volume of solids and increases the amount of methane gas produced. Mixing and heating also accelerate the digestion process, prevent stratification, and bring the raw solids into contact with the microorganisms.

The methane gas generated during anaerobic digestion is collected and used to fuel the plant boilers and biosolids dryer. Paddle stirrers are used to mix the contents in the digester. The digesters are heated by a hot-water heat exchanger fed from the boilers.

Biosolids

Two high-speed centrifuges are used to dewater the digested biosolids. Polymer is injected just ahead of the centrifuges to enhance the separation of the biosolids from the water. The excess water is returned to the head works. Dewatered solids coming off the centrifuges are pumped by screw conveyor to the direct drum dryer.

Fertilizer Manufacturing

The Fertilizer Manufacturing facility opened in 2006. The anaerobic digesters produce methane gas and stabilized biosolids. Methane from the digesters supplements the natural gas used in a drum dryer, reducing natural gas purchase by about 50%. Dewatered biosolids are mixed with dry pellets from earlier processing. The coated pellets are heated to 200°F in the drum dryer. This produces a pelletized, 90% dry product. This product is registered as a commercial fertilizer and meets State Class A Exceptional Quality biosolids criteria. The dried product is sold in bulk and in bags and is used in county projects.

Ultraviolet Disinfection

After biological secondary treatment, the treated effluent flow enters the ultraviolet (UV) disinfection system. The UV system is comprised of four individual channels which hold a combined total of 720 lamps. Flow passes across the UV lamps for a brief period. The lamps and channel operation are controlled through a remote programmable logic controller which varies lamp intensity and brings on additional channels as needed.

4. Puyallup WWTP

City of Puyallup Wastewater Treatment Plant
1602 18th St NW
Puyallup, WA 98371

The City of Puyallup WWTP (Figure C-6) provides service for approximately 37,000 residences as well as local businesses, a hospital, and health care facilities. There are also some light industrial activities connected to the wastewater collection system. The annual average discharge flow is 4 to 5 MGD, while the maximum month design capacity is 13.98 MGD. A chemical precipitant (sodium trithiocarbonate) is added to the mixed liquor to meet a monthly average permit limit of 0.0085 mg/L for copper. The WWTP provides nitrification in the activated sludge process that reduces ammonia concentrations in the final effluent to below 1 mg/L. Typical biochemical oxygen demand (BOD) and TSS concentrations in the final effluent are between 2 to 4 mg/L.

Over 100% of the mixed liquor is recycled back to the head of the aeration basins where denitrification occurs in the initial anoxic zones. This provides molecular oxygen, the reduction of nitrite and nitrate, and the creation of alkalinity that augments that which is consumed in the subsequent nitrification process. The anoxic and aerobic zones in the activated sludge basins provide additional biological removal of phosphorus. Disinfection of the final effluent before discharge into the Puyallup River is accomplished by ultraviolet light. Solids removed during treatment are stabilized by anaerobic digestion, dewatered in centrifuges, and land-applied.

Figure C-6. Aerial photo of the Puyallup Wastewater Treatment Plant.

The wastewater treatment process, shown in Figure C-6, is as follows:

Influent → Primary Clarification → Activated Sludge* Basins → Secondary Clarification → UV Disinfection → Discharge

*The activated sludge basins include anoxic and aerobic zones with internal recycle to accomplish biological nutrient removal. See Figure C-7.

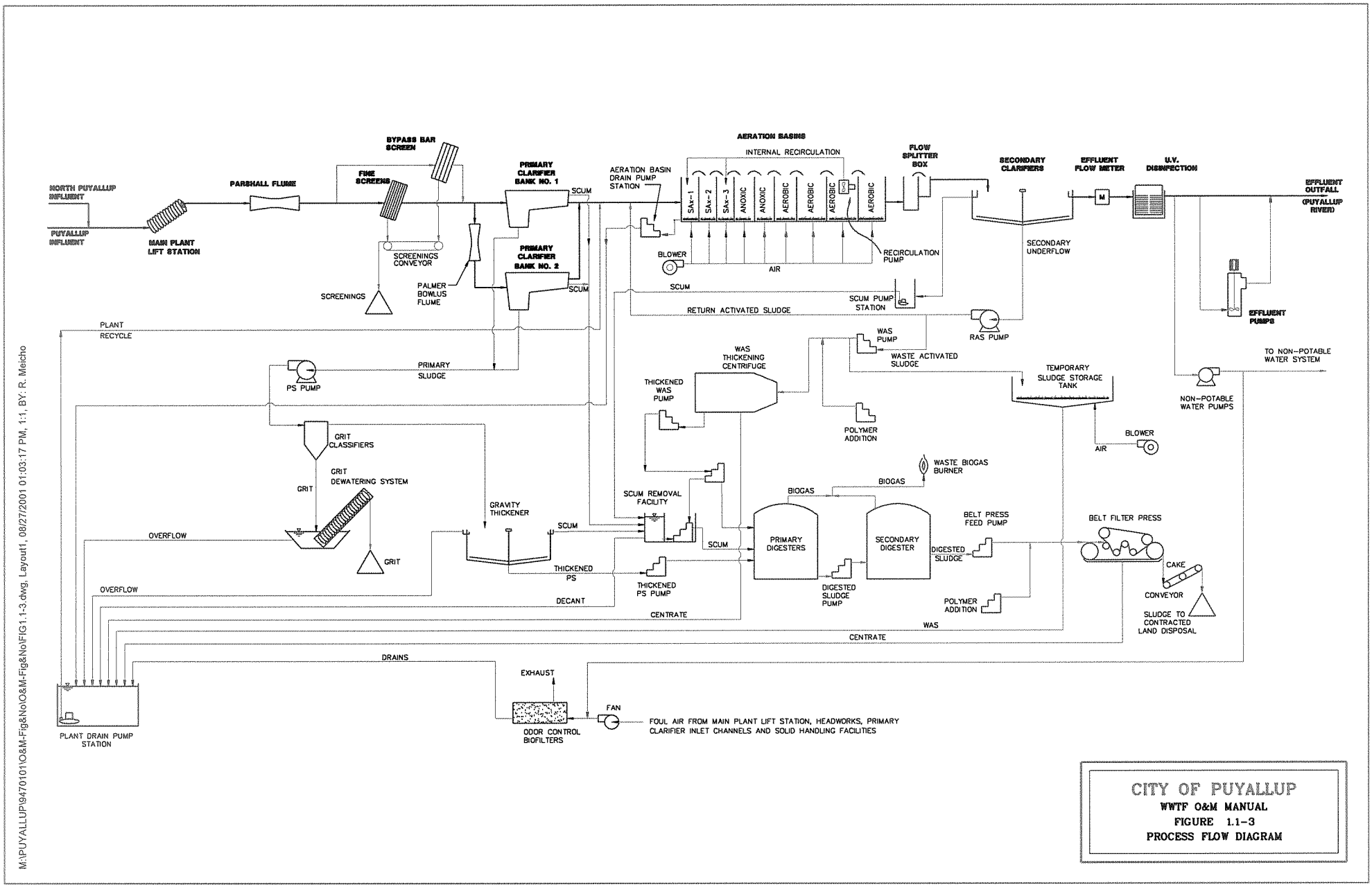

Figure C-7. Schematic of Treatment Processes at the City of Puyallup Wastewater Treatment Plant.

5. City of Hayden WWTP and Hayden Wastewater Research Facility (WRF)

The Hayden WWTP provides service to the City of Hayden, Hayden Lake, and the surrounding area, roughly 10,000 people or about 3500 homes. Also included in the Hayden sewer service area are some light industries, hospitals, manufacturing, 17 dental offices, 3 veterinarian offices, 2 nursing homes, and 1 urgent care center.

The WWTP's design capacity is 2.0 MGD, and the average dry-weather discharge flow is approximately 1.2 MGD. Peak design flow treatment capacity is reported to be 4.2 MGD.

Treated effluent is land-applied for irrigation during the warm, dry months. Discharge at other times of the year is into the Spokane River. Removed solids are aerobically digested and dewatered by belt press.

The Hayden WWTP provides biological treatment via three IEMCO carousel ditches operated to nitrify in an extended aeration mode. There is no primary treatment. At the time of sampling, two of the three oxidation ditches were in service; the ditches are 0.6 million gallon capacity each. There are four secondary clarifiers available; two were in service during this study.

A flow diagram for the Hayden WWTP is as follows:

Influent → Headworks → Aeration Ditches (3) → Secondary Clarifiers (4) → Tertiary Filtration → Disinfection → Discharge

Tertiary treatment of about 0.25 MGD of the Hayden WWTP secondary effluent was being provided by the Hayden Water Research Facility which is operated by Blue Water Technologies, Inc. The Blue PRO (registered trademark name) treatment process includes chemical addition (ferric sulfate) and two-stage filtration through the company's continuous backwashing, upflow sand filters (Figure C-7). "Two-stage" means that wastewater is treated sequentially, through a first-stage filter and then through a secondary-stage filter of the same design.

Long-term operation of the Blue PRO treatment system has demonstrated this technology is effective at producing very low concentrations of phosphorus. Solids removed by the tertiary filters at Hayden Water Research Facility are recycled to influent wastewater upstream of the Hayden aeration ditches.

Figure C-8 was taken from a report for a recent treatment pilot study conducted with the Blue PRO process at the Town of Innisfil, Ontario, Canada.

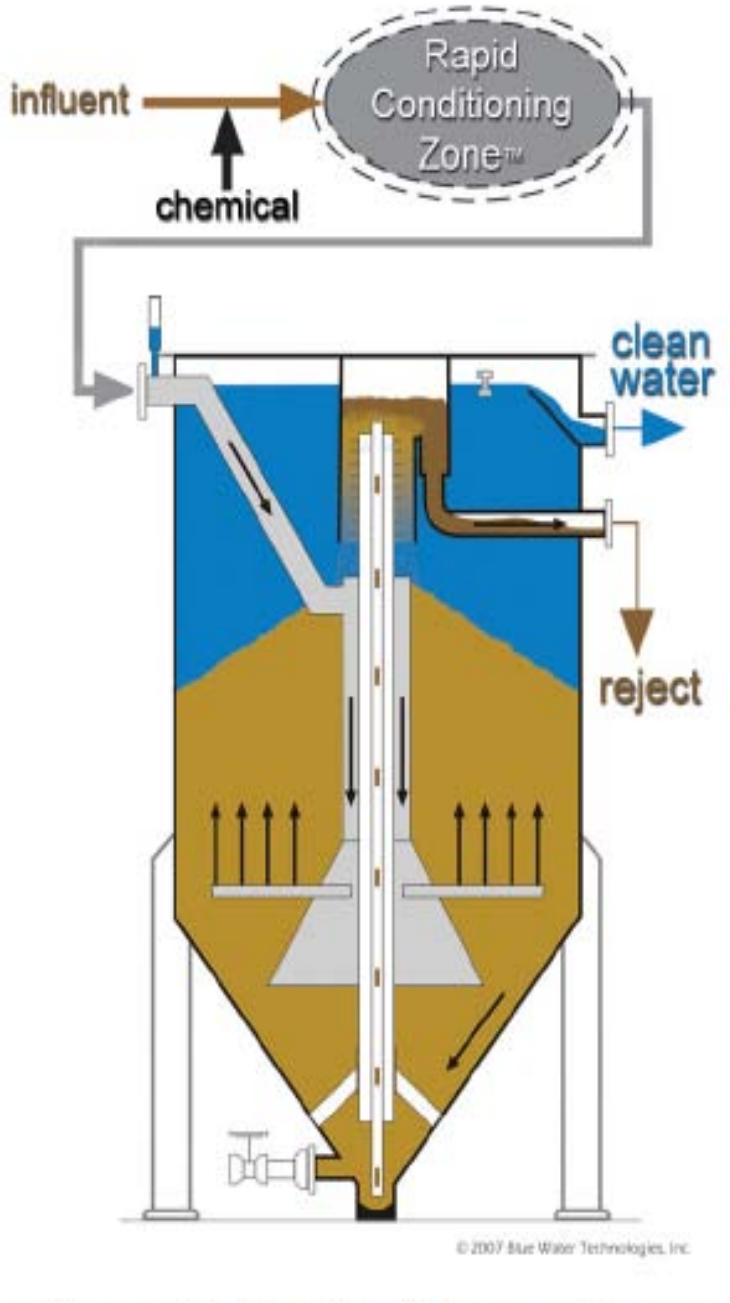

Figure C-8. Blue PRO® Process Diagram.

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Appendix D. Quality Assurance Information

Data Quality Discussion

Sample Collection

All samples were sent to Ecology's Manchester Environmental Laboratory (MEL) and Axys Analytical Laboratory Inc. (Axys) in coolers at 4ºC. Coolers and samples arrived intact by August 21, 2008, with two exceptions. The August 19, 2008 Hayden secondary and tertiary effluent orthophosphate and total phosphorus data was rejected due to field and laboratory error. The total phosphorus and orthophosphate data from November 19, 2008 were used for this report.

Field transfer blank

A field transfer blank was analyzed to detect contamination arising from sample containers or sample handling. The blank was prepared by transferring organic-free water supplied by Axys from one bottle to another in the field, which mimicked the grab sampling procedure. A field transfer blank was poured onsite at the Budd Inlet WWTP.

Results for the field blank used in the study are presented in Table D-1.

Table D-1. Select Results for the Field Transfer Blank (ug/L).

Analyte	Sample Number	Field Transfer Blank 1		5x Conc.	Affected Results	Action Taken
Bisphenol A	8344186	1		5	Yes	Qualified data below 5 as J (Chambers Creek effluent sample #8344184 value as 1.9J)
Naproxen	8344186	6.85		34.3	Yes	Qualified results below 34.3 as J (BITP secondary effluent sample #0834193 and #0834194 replicate values as 19.6J and 18.2J)
Phenol	8344186	0.12	J	0.19	No	--

J - Analyte positively identified; numerical value is the approximate concentration.
BITP= Budd Inlet Treatment Plant.

The field transfer blank had very little contamination with only three analytes (bisphenol A, phenol, and naproxen) detected above the laboratory reporting limit. The results for all analytes are provided in Appendix E.

Data affected by the bisphenol A and naproxen contamination was limited to three data points carrying the J qualifier, including Chambers Creek effluent bisphenol A. The field transfer blank values were generally lower than the laboratory method blank values (below) which indicate there was no container or sample handling contamination.

Laboratory Quality Control

Sample Preparation

Preparation, storage, and handling are deemed acceptable by both laboratories, MEL and Axys. The details of sample handling and preparation are contained in the case narratives, which are provided in this appendix.

MEL or EPA and the project lead reviewed the laboratory data packages, verified the case summaries, and assessed the usability of the data. Based on these assessments, the data are accepted with the appropriate qualifications, and the data are considered usable for making calculations, determinations, and decisions for which the project was conducted.

Provided in the case narratives are performance of matrix spikes, surrogates, laboratory blanks, and calibrations. A procedural blank and a lab-generated reference sample are called ongoing precision and recovery. By virtue of the isotope dilution/internal standard quantification procedures, data for Methods 1694 and 1698 are recovery corrected for possible losses during extraction and cleanup. All three EPA methods are performance based. This means that Axys and MEL may modify the method to improve recovery performance of the instrumentation, provided they meet requirements of the published method. The following provides a summary from the case narratives for results interpretation.

Sample Analyte Concentrations Are Not Blank Corrected.

Samples may contain high levels of the targeted analyte or sometimes high levels of an interfering matrix. This is common for wastewater samples and biosolids. To bring the concentrations down to fit within the calibration range of the instrument, samples are diluted with water. The dilution factor is used to calculate the original concentrations once detection has been made.

A single letter code was used to indicate analytical actions such as dilution, non-detections, or estimated values. Table D-2 lists the letter codes used for this report. The case narratives also contain the codes specific to each laboratory.

Table D-2. Data Qualifier Codes.

Code	Description
D	The sample was diluted, reported value is dilution corrected.
U	The analyte was not detected at or above the reported result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in the sample.
NJ	There is evidence that the analyte is present. The associated numerical result is an estimate.

Semi-Volatile Organics

Laboratory control samples, method blanks, standards/labeled compounds, and laboratory duplicates for this study are acceptable. Results for check standards/laboratory control samples, duplicate samples, and labeled compounds were compared to quality control (QC) limits. The results for field and method blanks were examined for significant contamination of the samples. Non-detects are reported at the laboratory reporting limit and flagged with either a U or UJ.

For all parameters, the calibrations, recoveries, and ongoing precision were performed in accordance with the appropriate method. A summary of calibration, ongoing precision, and internal standards recovery is provided later in this appendix.

PPCPs– Method 1694 and Hormones/ Steroids- Method 1698

The EPA methods to assess PPCPs and hormones/steroids from wastewater samples use a suite of analytical controls to ensure precision by instrument calibration, linearity checks, ongoing precision and recovery (OPR), and surrogates or spiked labeled analogs in the samples. The case narratives discuss the outcome of some of the QC data points. For example, the upper or lower point on the calibration curve may have been outside the range for the method, but the remainder of the data points fell within the method range. Full details on initial calibration, continuing calibration, OPRs, and matrix spikes are provided in the case narratives. Many of the slight variations in the QC data were not deemed by Axys to have a significant effect on the data.

The EPA QA case narrative (also in this appendix) considers the data to be of high quality and, with the exception of a few data points being qualified, the data are acceptable and can be used for all purposes.

The following statements discuss the few exceptions for the qualified data.

- Some target compounds were reported at elevated laboratory reporting limits due to interferences and/or contamination in the associated blank. To avoid potential false positives due to blank contamination, results in the associated samples at concentrations of <5x the value in the method blank were qualified as non-detects, "U". Concentrations >5x were not qualified.
- Some of the internal standard labeled compounds did not meet the recovery control limits. The affected target compounds were qualified accordingly.
- The MS/MSD for sample BITP-secondary effluent (sample number 0834194) had a few instances of native concentrations overwhelming the spike. The associated sample results were qualified accordingly.

Compounds or procedures that were analyzed at both laboratories were compared. The percent moisture of the biosolids had very good agreement for all samples with the relative percent difference ranging from 0.5 to 1.5 %. Caffeine was analyzed by different methods at the two labs; however, both procedures reached the same non-detect conclusion for the effluent samples.

Laboratory Blanks, Laboratory Spikes, and Laboratory Replicates

Samples sent to MEL were analyzed using standard protocols (MEL, 2005). Samples sent to Axys were analyzed by standard protocols. Measurement quality objectives (MQOs) were presented in Table 10 of the QA Project Plan (Lubliner et al., 2008). All samples were received and processed within established holding times, within the proper temperature range, and in good condition.

Some laboratory calibration checks were not within acceptance limits, and these results are reported as estimates. Some matrix spikes for influent samples were not high enough to be seen above the native sample noise, and data were qualified. Few of the Axys and MEL qualified data actually affected results.

PPCPs – wastewater

- Caffeine was detected in some the QC controls. The initial and continuing calibration concentrations may be slightly over-reported. OPRs for the following compounds were outside the method control limits.
    - Poor recovery includes: cefotaxime, miconazole, norgestimate, minocycline, cimetidine, and ranitidine.
    - Over reported includes: norfloxacin.

PPCPs – biosolids

- Laboratory QC checks were within acceptable limits, and data were not qualified.

Hormones/Steroids – wastewater

- Several batches of samples were prepared and run through the instrumentation. Each batch has a QC comment that may have impacted the results.
    - Batch WG27292
        - Low recovery of norethindrone and norgestrel in the OPR.
        - Low recovery of labeled norethindrone in one sample.
        - Low recovery of labeled ethinylestradiol in the blank.
        - Over reporting of androsterone, desogestrel 17b-estradiol, and progesterone in the OPR.
    - Batch WG26896
        - Some analytes detected in the laboratory blank.
        - Data were not blank corrected by Axys.
        - Blank effects were considered at less than 5 times the blank value by the EPA QA reviewer.

Hormones/Steroids – biosolids

- Laboratory QC checks were within acceptable limits, and data were not qualified.

EPA performed a third-party QA on the Axys results because of the newness of the methods and the sole source contract to Axys. Rejecting or qualifying data based on the field transfer blank was not part of their contract; therefore Axys was in complete compliance with the duties as prescribed. MQOs were met for laboratory QC samples.

Qualification flags on data are common for low level analyses. The independent EPA review of the data from Methods 1694 and 1698 considered the data to be of high quality and acceptable for all purposes. The number and percent of the data qualified with the "J" flag is presented in Table D-3. Data with "J" flags are considered to be positively identified and are used in discussing the results for this study.

Table D-3. The number and percent of detected and "J" flagged data by method and sample type.

Parameter	Influents	Effluents/ Discharges	Biosolids
Method 1694 for PPCPs[1694]			
Number Detected	174	166	88
Percent Detected of Total	48%	33%	40%
Number "J" Flagged	42	38	18
Percent "J" Flagged of Detected	24%	23%	20%
Method 1698 for Hormones/Steroids			
Number Detected	87	57	49
Detected of Total	64%	31%	22%
Number "J" Flagged	51	18	47
Percent "J" Flagged of Detected	60%	32%	96%
Method 8270d for Semi-Volatile Organics			
Number Detected	71	54	49
Detected of Total	19%	10%	22%
Number "J" Flagged	27	34	44
Percent "J" Flagged of Detected	38%	63%	90%

Data qualified by the "J" flag ranged from 20% to 96%. Method 1694 for PPCPs accounted for the least number of qualifications. The values of 96% and 90% were for biosolids data with high concentrations. Therefore the bulk of the qualifications were below 60%. The recently published EPA study that spurred the development of Methods 1694 and 1698 indicated that 46% of the PPCPs[1694] data and 42% of the hormones/steroids data were qualified in their study (EPA, 2009b).

These new EPA methods performed better in this 2008 PPCP study. This may be a reflection of a honing of the methods by Axys. (Axys was contracted for both studies.)

Analyses for caffeine and triclosan were performed by both Method 1694 and 8270. Results from the EPA Method 1694 were deemed more appropriate by MEL staff and are included this report. The reasoning was that Method 1694 is an isotopic dilution method and has inherently more QA. Additionally the 1694 and 1698 data results were independently QA'd by EPA as mentioned previously.

Laboratory blanks were below the laboratory reporting limit. Mean laboratory control samples (spikes) were within the acceptance criteria for the datasets for both laboratories. Individual sample pairs fell outside the acceptance criteria for a few samples, but these did not occur on the same date for all parameters.

Biosolids Data

Table D-4 lists the percent moisture of the biosolids that were analyzed by both Axys and MEL which had very good agreement with the relative percent difference (RPD) ranging from 0.5 to 1.5 %. To ensure comparability of results, all sample results are reported on a dry-weight basis.

Table D-4. Percent Moisture in Sludge.

Biosolids Sample	Percent Moisture		RPD
	Axys	MEL	
Budd Inlet WWTP	80.3	79.4	1.1
Budd Inlet (field duplicate)	80	79.6	0.5
Chambers Creek WWTP	79.2	80.3	1.4
Puyallup WWTP	85.4	86.3	1.0

Field Replicate Data

Field replicates were taken side-by-side from the Budd Inlet WWTP influent, effluents, and biosolids. Replicates provide estimates of field and laboratory variability. Variability can be expressed as the RPD between a sample and its duplicate, Equation 1.

Equation 4

$$\text{RPD} = \left(\frac{\text{difference of 2 results}}{\text{mean}} \right) \times 100$$

The reclaimed water sample was collected from the discharge at the Budd Inlet Reclaimed Water holding tank. Replicate data results for wastewater samples are shown in Tables D5 - D7.

Field replicate RPDs for water samples were below 15% for nutrients and 40% for PPCPs[1694], hormones/steroids and semi-volatile organics. The only two exceptions were in the tertiary effluent samples for ammonia and benzoic acid. These exceptions are due to the difference between very small numbers.

Biosolids RPDs for the PPCPs, hormones/steroids, and semi-volatile organics were below 20%. For the remainder of this document, the mean of the original sample and replicate sample value is used.

Replicate data for nutrients, total suspended solids, and Method 8270d semi-volatile organics are shown in Table D-5.

Table D-5. Relative Percent Difference (RPD) between Field Replicates for Budd Inlet WWTP (BITP) and RWP (BIRWP).

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep	Mean	RPD	Orig.	Rep.	Mean	RPD
Nutrients/Solids, mg/L												
Ammonia	34.5	34.6	34.6	0.3	0.044	0.042	0.043	3.5	0.012	0.014	0.013	15.4
Nitrite-Nitrate	0.1	0.1	0.1	0.9	1.2	1.2	1.2	4.2	1.3	1.5	1.4	10.7
Orthophosphate	4.7	5.1	4.9	7.0	3.2	3.6	3.4	11.6	3.2	3.7	3.4	13.5
Total Persulfate Nitrogen	39.9	41	40.5	2.7	2.1	1.9	2	7	2.1	2.1	2.1	2.4
Total Phosphorus	6.5	7.5	7	14.6	3.2	3.3	3.2	0.6	2.8	2.9	2.9	1.4
Total Suspended Solids, mg/L	275	240	257.5	13.6	5	5	5	0	1	1	1	0
Semi-Volatile Organics, ug/L												
1,2,4-Trichlorobenzene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
1,2-Dichlorobenzene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
1,2-Diphenylhydrazine	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
1,3-Dichlorobenzene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
1,4-Dichlorobenzene	0.7	0.6	0.61	13.1	0.27U	0.28U	--	--	0.28U	0.28U	--	--
1-Methylnaphthalene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
2,2'-Oxybis[1-chloropropane]	0.27UJ	0.29U	--	--	0.27UJ	0.28UJ	--	--	0.28UJ	0.28UJ	--	--
2,4,5-Trichlorophenol	1.1U	1.2U	--	--	1.1U	1.1U	--	--	1.1U	1.1U	--	--
2,4,6-Trichlorophenol	1.1U	1.2U	--	--	1.1U	0.8	0.8*	--	1.1U	1.1U	--	--
2,4-Dichlorophenol	2.7U	2.9U	--	--	2.7U	2.8U	--	--	2.8U	2.8U	--	--
2,4-Dimethylphenol	2.7U	2.9U	--	--	2.7U	2.8U	--	--	2.8U	2.8U	--	--
2,4-Dinitrophenol	0	2.9U	--	--	0	0	--	--	0	0	--	--
2,4-Dinitrotoluene	1.1U	1.2U	--	--	1.1U	1.1U	--	--	1.1U	1.1U	--	--
2,6-Dinitrotoluene	1.1U	1.2U	--	--	1.1U	1.1U	--	--	1.1U	1.1U	--	--
2-Chloronaphthalene	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
2-Chlorophenol	1.1U	1.2U	--	--	1.1U	1.1U	--	--	1.1U	1.1U	--	--

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep	Mean	RPD	Orig.	Rep.	Mean	RPD
2-Methylnaphthalene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
2-Methylphenol	2.7U	2.9U	--	--	2.7U	2.8U	--	--	2.8U	2.8U	--	--
2-Nitroaniline	5.4U	5.8U	--	--	5.3U	5.5U	--	--	5.6U	5.6U	--	--
2-Nitrophenol	0.54U	0.58U	--	--	0.53UJ	0.55U	--	--	0.56U	0.56U	--	--
3-Nitroaniline	1.1U	1.2U	--	--	1.1U	1.1U	--	--	1.1U	1.1U	--	--
4,6-Dinitro-2-Methylphenol	0	1.2U	--	--	0	0	--	--	0	0	--	--
4-Bromophenyl-Phenylether	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
4-Chloro-3-Methylphenol	2.9U	2.9U	--	--	2.7U	2.8U	--	--	2.8U	2.8U	--	--
4-Chloroaniline	0	12U	--	--	0	0	--	--	0	0	--	--
4-Chlorophenyl-Phenylether	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
4-Methylphenol	46	41	43.5	11.5	0.2	0.2	0.2	23	0.2	0.2	0.2	0
4-Nitroaniline	0	1.2U	--	--	0.0	0.0	--	--	0	0	--	--
4-Nitrophenol	2.7U	2.9U	--	--	2.7U	2.8U	--	--	2.8U	2.8U	--	--
Acenaphthene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Acenaphthylene	0.27UJ	0.29U	--	--	0.27UJ	0.28UJ	--	--	0.28UJ	0.28UJ	--	--
Anthracene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Benzo(a)anthracene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Benzo(a)pyrene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Benzo(b)fluoranthene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Benzo(ghi)perylene	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Benzo(k)fluoranthene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Benzoic Acid	270	181	225.5	39.4	2.3	2.8UJ	2.3*	--	3.7	2.3	3.7	38
Benzyl Alcohol	49	48	48.5	2.1	2.7U	2.8U	--	--	2.8U	2.8U	--	--
Bis(2-Chloroethoxy)Methane	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Bis(2-Chloroethyl)Ether	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Bis(2-Ethylhexyl) Phthalate	28	24	26	15.4	1.6	0.28U	1.6*	--	0.28U	0.28U	--	--
Bisphenol A	0.54U	0.58U	--	7.1	1.3UJ	2.8U	--	--	1.1UJ	1.1UJ	--	--
Butylbenzylphthalate	16	15	15.5	6.5	0.53U	0.28U	--	--	0.56U	0.56U	--	--
Carbazole	0.54UJ	0.58U	--	--	0.53UJ	0.55UJ	--	--	0.56UJ	0.56UJ	--	--
Chrysene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep	Mean	RPD	Orig.	Rep.	Mean	RPD
Dibenzo(a,h)anthracene	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Dibenzofuran	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Diethylphthalate	6.8	6.5	6.65	4.5	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Dimethylphthalate	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Di-N-Butylphthalate	3.3	3.1	3.20	6.2	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Di-N-Octyl Phthalate	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Fluoranthene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Fluorene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Hexachlorobenzene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Hexachlorobutadiene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Hexachlorocyclopentadiene	1.1UJ	1.2U	--	--	0	1.1UJ	--	--	1.1UJ	1.1UJ	--	--
Hexachloroethane	1.1UJ	1.2U	--	--	0.27UJ	1.1UJ	--	--	1.1UJ	1.1UJ	--	--
Indeno(1,2,3-cd)pyrene	5.4U	5.8U	--	--	5.3U	5.5U	--	--	5.6U	5.6U	--	--
Isophorone	0.54U	0.58U	--	--	0.53U	0.55U	--	--	0.56U	0.56U	--	--
Naphthalene	0.2	0.1	0.16	19.4	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Nitrobenzene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
N-Nitrosodimethylamine	1.1U	1.2UJ	--	--	1.1UJ	1.1UJ	--	--	1.1UJ	1.1UJ	--	--
N-Nitroso-Di-N-Propylamine	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
N-Nitrosodiphenylamine	0.54UJ	0.58U	--	--	0.53UJ	0.55UJ	--	--	0.56UJ	0.56UJ	--	--
Pentachlorophenol	2.7U	2.9U	--	--	2.7UJ	2.8UJ	--	--	2.8UJ	2.8UJ	--	--
Phenanthrene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Phenol	42	44	43	4.7	1.1U	1.1U	--	--	1.1U	1.1U	--	--
Phenol, 4-Nonyl-	0.54U	0.58U	--	--	0.2	0.55U	0.2*	--	0.56U	0.56U	--	--
Pyrene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Retene	0.27U	0.29U	--	--	0.27U	0.28U	--	--	0.28U	0.28U	--	--
Tri(2-chloroethyl) phosphate	0.27U	0.29U	--	--	0.9	0.8	0.9	5	0.9	0.8	0.9	5
Triethyl citrate	3.9	4.1	4	5	1.9	2.2	2.0	15	2.3	2.3	2.3	0

-- Not an appropriate calculation due to non-detect
* Not a mean because one replicate was undetected.

Field replicate RPDs for water samples were below 15% for nutrients and below 40% for semi-volatile organics.

Table D-6. Relative Percent Difference (RPD) for MEL replicate biosolids data collected at the Budd Inlet WWTP (BITP).

Analyte	BITP-Biosolids, ug/Kg (dw), ppb			
	Orig.	Rep.	Mean	RPD
1,2,4-Trichlorobenzene	604U	604U	--	--
1,2-Dichlorobenzene	604U	604U	--	--
1,2-Diphenylhydrazine	604U	604U	--	--
1,3-Dichlorobenzene	604U	604U	--	--
1,4-Dichlorobenzene	419.0	481.0	450	13.8
1-Methylnaphthalene	604U	604U	--	--
2,2'-Oxybis[1-chloropropane]	604UJ	604UJ	--	--
2,4,5-Trichlorophenol	2420UJ	2420UJ	--	--
2,4,6-Trichlorophenol	2420UJ	2420UJ	--	--
2,4-Dichlorophenol	6040U	6040U	--	--
2,4-Dimethylphenol	6040U	6040U	--	--
2,4-Dinitrophenol	0	0	--	--
2,4-Dinitrotoluene	2420U	2420U	--	--
2,6-Dinitrotoluene	2420U	2420U	--	--
2-Chloronaphthalene	1210UJ	1210U	--	--
2-Chlorophenol	2420U	2420U	--	--
2-Methylnaphthalene	604U	604U	--	--
2-Methylphenol	6040U	6040U	--	--
2-Nitroaniline	12100U	12100U	--	--
2-Nitrophenol	1210U	1210U	--	--
3-Nitroaniline	2420U	2420U	--	--
4,6-Dinitro-2-Methylphenol	0	0	--	--
4-Bromophenyl-Phenylether	1210U	1210U	--	--
4-Chloro-3-Methylphenol	6040U	6040U	--	--
4-Chloroaniline	0	0	--	--
4-Chlorophenyl-Phenylether	604U	604U	--	--
4-Methylphenol	609.0	607.0	608*	0.3
4-Nitroaniline	0	0	--	--
4-Nitrophenol	6040U	6040U	--	--
Acenaphthene	604U	604U	--	--
Acenaphthylene	604UJ	604UJ	--	--
Anthracene	170	168	169	1.2
Benzo(a)anthracene	450	520	485	14.4
Benzo(a)pyrene	375	364	370	3.0
Benzo(b)fluoranthene	835	865	850	3.5
Benzo(ghi)perylene	1210U	1210U	1210	--
Benzo(k)fluoranthene	260	309	285	17.2
Benzoic Acid	8390	8280	8335	1.3

Analyte	BITP-Biosolids, ug/Kg (dw), ppb			
	Orig.	Rep.	Mean	RPD
Benzyl Alcohol	6040U	604U	3322	--
Bis(2-Chloroethoxy)Methane	604U	604U	--	--
Bis(2-Chloroethyl)Ether	1210U	308	308	--
Bis(2-Ethylhexyl) Phthalate	14800	17100	15950	14.4
Bisphenol A	55700	61700	58700	10.2
Butylbenzylphthalate	1210U	1210U	--	--
Carbazole	1210UJ	1210UJ	--	--
Chrysene	594	636	615	6.8
Dibenzo(a,h)anthracene	1210U	1210U	--	--
Dibenzofuran	1210U	1210U	--	--
Diethylphthalate	1210U	1210U	--	--
Dimethylphthalate	604U	604U	--	--
Di-N-Butylphthalate	604U	604U	--	--
Di-N-Octyl Phthalate	1210U	1210U	--	--
Fluoranthene	604U	604U	--	--
Fluorene	604U	604U	--	--
Hexachlorobenzene	604U	604U	--	--
Hexachlorobutadiene	604U	604U	--	--
Hexachlorocyclopentadiene	2420U	2420U	--	--
Hexachloroethane	2420U	2420U	--	--
Indeno(1,2,3-cd)pyrene	612	743	678	19.3
Isophorone	1210U	1210U	--	--
Naphthalene	499	598	549	18.0
Nitrobenzene	604U	604U	--	--
N-Nitrosodimethylamine	2420UJ	242UJ	--	--
N-Nitroso-Di-N-Propylamine	604U	604U	--	--
N-Nitrosodiphenylamine	1210UJ	1210UJ	--	--
Pentachlorophenol	6040UJ	6040UJ	--	--
Phenanthrene	655	766	711	15.6
Phenol	2500	2990	2745	17.9
Phenol, 4-Nonyl-	1210U	1210U	--	--
Pyrene	860.0	1040.0	950	18.9
Retene	604U	604U	--	--
Tri(2-chloroethyl) phosphate	974U	604U	--	--

-- Not detected.
* Not a mean because one replicate was undetected.

Biosolids RPDs for the semi-volatile organics were below 20%.

Replicate data for PPCPs[1694] and hormones/steroids are shown in Table D-7 to D-8.

Table D-7. Relative Percent Difference (RPD) between Field Replicates for Budd Inlet WWTP (BITP) and RWP (BIRWP), ng/L (pptr).

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD
PPCPs, ng/L												
Acetaminophen	201000	208000	204500	3.4	173U D	173U D	--	--	179U D	160U D	--	--
Azithromycin	451	690	571	41.9	186	115	150.5	47	6.28UJ	11.8	11.8*	--
Caffeine	80700	108000	94350	28.9	43.4U D	105	--	--	53U D	40U D	--	--
Carbadox	61.9U D	49.4UJ	--	--	11.4	15	13.2	27	108UJ	65.3UJ	--	--
Carbamazepine	1100	1560	1330	34.6	672.0	897	784.5	29	1710	1490	1600	14
Cefotaxime	79.1U D	131UJ	--	--	38.6U D	43.6	43.6*	--	369UJ	95UJ	--	--
Ciprofloxacin	701	632	667	10.4	205	211	208	3	15.7U D	14U D	--	--
Clarithromycin	116	150	133	25.6	142	108	125	27	4.48U D	4.6	--	--
Clinafloxacin	42.5U D	48.2U D	--	--	17.6U D	25.1U D	--	--	59U D	16U D	--	--
Cloxacillin	33.6U D	39.1UJ	--	--	8.67U D	8.84U D	--	--	39.9U D	21.8U D	--	--
Codeine	464	884	674	62.3	30.2	73.3	51.8	83	8.97UJ	8UJ	--	--
Cotinine	3420	3360	3390	1.8	36.6	58.3	47.5	46	22.2UJ	77.3	--	--
Dehydronifedipine	12	21	16	58.5	16.1	19.2	17.7	18	95.8UJ	83.8UJ	--	--
Diphenhydramine	1750	3120	2435	56.3	291	448	369.5	42	17.3UJ	43.5UJ	--	--
Diltiazem	751	1080	916	35.9	155	221	188	35	3.19UJ	21.8UJ	--	--
Digoxin	338UJ	325UJ	--	--	45.3UJ	144UJ	--	--	565UJ	137UJ	--	--
Digoxigenin	40.6U D	64	64	--	19.6U D	35.1UJ	--	--	145UJ	66.1UJ	--	--
Enrofloxacin	20.3U D	19.5U D	--	--	8.67U D	8.67U D	--	--	8.97U D	8U D	--	--
Erythromycin-H2O	238	333	286	33.3	169	183	176	8	3.29U D	9.1	9.1*	--
Flumequine	10.2U D	14.8UJ	--	--	4.34U D	4.33UJ	--	--	11.4UJ	6.57UJ	--	--
Fluoxetine	131	224	178	52.4	59.4	82.7	71.1	33	45.4	39.3	42.4	14
Lincomycin	20.3U D	36UJ	--	--	12.8	8.67UJ	--	--	8.97UJ	8UJ	--	--

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD
Lomefloxacin	20.3U D	19.5U D	--	--	8.67U D	8.67U D	--	--	8.97U D	8U D	--	--
Miconazole	32	30	31	7.1	4.34U D	4.33UJ	--	--	4.48UJ	4UJ	--	--
Norfloxacin	102U D	97.5U D	--	--	43.4U D	43.3U D	--	--	44.8U D	40U D	--	--
Norgestimate	20.3UJ	33.8UJ	--	--	8.67UJ	10.5UJ	--	--	22.4UJ	14.8UJ	--	--
Ofloxacin	212	211	212	0.5	86.7	130	108.4	40	44.8U D	40U D	--	--
Ormetoprim	4.06U D	3.9UJ	--	--	1.73U D	1.73UJ	--	--	1.79UJ	1.6UJ	--	--
Oxacillin	20.3U D	19.5UJ	--	--	8.67U D	8.67UJ	--	--	14.1UJ	8UJ	--	--
Oxolinic Acid	4.06U D	4.9UJ	--	--	1.73U D	1.73UJ	--	--	4.48UJ	10	10.0*	--
Penicillin G	20.3U D	19.5UJ	--	--	8.67U D	8.67UJ	--	--	28.3UJ	30.7	30.7*	--
Penicillin V	50	74	62	39.2	8.67U D	8.67UJ	--	--	34.8UJ	9.16UJ	--	--
Roxithromycin	2.03U D	1.95U D	--	--	6.4	3.9	5.2	49	0.897U D	0.8U D	--	--
Sarafloxacin	92.6U D	88.9U D	--	--	39.5U D	39.5U D	--	--	40.9U D	36.5UJ	--	--
Sulfachloropyridazine	10.2U D	9.75U D	--	--	4.34U D	4.33U D	--	--	4.48U D	16.6UJ	--	--
Sulfadiazine	16	20	18	23.6	4.34U D	4.33U D	--	--	4.48U D	4UJ	--	--
Sulfadimethoxine	7	9	8	23.0	3.7	2.9	3.3	24	11.5UJ	4.9UJ	--	--
Sulfamerazine	20	13	17	44.3	3.1	1.73U D	--	--	1.79U D	9.83UJ	--	--
Sulfamethazine	13.5U D	U D	--	--	4.43U D	5.77U D	--	--	5.97U D	16.7UJ	--	--
Sulfamethizole	4.06U D	U D	--	--	1.73U D	1.73U D	--	--	8.21UJ	3.9U D	--	--
Sulfamethoxazole	3820	4200	4010	9.5	1390	1490	1440	7	72.7	192UJ	72.7*	--
Sulfanilamide	338U D	13U D	--	--	144U D	144U D	--	--	149U D	225UJ	--	--
Sulfathiazole	10.2U D	4.76U D	--	--	4.34U D	4.33U D	--	--	9.38UJ	5.72UJ	--	--
Thiabendazole	21	21	21	1.4	24.4	24.4	24.4	0	22.7	20.9	21.8	8
Trimethoprim	998	1530	1264	42.1	682	542	612	23	35.5UJ	73.3	73.3*	--
Tylosin	54.5U D	67.8U D	--	--	26.1U D	17.3U D	--	--	40.5U D	10.7U D	--	--
Virginiamycin	63.5U D	93.1UJ	--	--	11.4U D	16.9UJ	--	--	73.3UJ	46.7U D	--	--
1,7-Dimethylxanthine	55600	55900	55750	0.5	434U D	433U D	--	--	448U D	46.9U D	--	--
Gemfibrozil	4840	4580	4710	5.5	261	241	251	8	14.9UJ	5.68UJ	--	--
Ibuprofen	27800	31600	29700	12.8	29.8	26.9	28.4	10	33	26.6	29.8	21
Naproxen	22400	21000	21700	6.5	19.6	18.2	18.9	7	127	38.5	38.5*	--

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD
Triclocarban	308	334	321	8.1	30.5	32.3	31.4	6	2.99U	2.67U	--	--
Triclosan	1580	1600	1590	1.3	125	102	113.5	20	59.8U	53.3U	--	--
Warfarin	11	11	11	7.3	10.3	9.7	10	6	8.5	3.14UJ	--	--
Anhydrochlortetracycline (ACTC)	87.1U	75.4U	--	--	35.8U	41.2UJ	--	--	23.3U	23.5U	--	--
Anhydrotetracycline (ATC)	83U	86.3U	--	--	14.5U	14.4UJ	--	--	14.9U	13.3U	--	--
Chlortetracycline (CTC)	13.5U	13U	--	--	8.24U	6.46UJ	--	--	8.85U	8.83U	--	--
Demeclocycline	54.2U	35.1U	--	--	15.4U	14.4UJ	--	--	14.9U	13.3U	--	--
Doxycycline	82	77	80	6.0	33.8	28.5	31.2	17	5.98U	5.32U	--	--
4-Epianhydrochlortetracycline (EACTC)	135U	130U	--	--	88.5U	107UJ	--	--	59.8U	53.2U	--	--
4-Epianhydrotetracycline (EATC)	84.7U	113U	--	--	28.9U	30.8UJ	--	--	17.1U	23.1U	--	--
4-Epichlortetracycline (ECTC)	72.6U	42.9U	--	--	24.3U	19.8UJ	--	--	25.9U	25.5U	--	--
4-Epioxytetracycline (EOTC)	36.9U	65.4U	--	--	23.8U	8.66UJ	--	--	7.8U	19U	--	--
4-Epitetracycline (ETC)	92	29	60	103.5	26.0	25.6	25.8	2	8.81U	7.02U	--	--
Isochlortetracycline (ICTC)	13.5U	14U	--	--	5.78U	5.78UJ	--	--	5.98U	5.32U	--	--
Minocycline	135U	130U	--	--	64.4U	57.8UJ	--	--	65.6U	57.7U	--	--
Oxytetracycline (OTC)	24.2U	41.1U	--	--	13.8U	5.9UJ	--	--	5.98U	11.2U	--	--
Tetracycline (TC)	142	18	80	154.8	29.2	32	30.6	9	6.29U	5.32U	--	--
Albuterol	30	27	28	8.8	15.3	14.8	15.1	3	0.861U D	0.897U D	--	--
Cimetidine	482	642	562	28.5	241	240	240.5	0	1.72U D	1.79U D	--	--
Metformin	107000	115000	111000	7.2	4720	4050	4385	15	627	457	542	31
Ranitidine	4790	3950	4370	19.2	700	777	738.5	10	1.72U D	1.79U D	--	--
Hormones/ Steroids												
17a-Ethinyl-Estradiol	14.3U	11.5U	--	--	2.02U	1.75U	--	--	1.43U	1.47U	--	--
17a-Dihydroequilin	12.5	10U	13	--	1.05U	1.04U	--	--	1.54U	0.456U	--	--
17a-Estradiol	5.3	7	6	26.8	0.075U	0.0273U	--	--	0.033U	0.054U	--	--
17b-Estradiol	25.5U	20.4U	--	--	1.77U	1.38U	--	--	0.699U	0.705U	--	--
Androstenedione	767	519U	--	--	13.4U	9.95U	--	--	21.2U	19.3U	--	--
Androsterone	1410	1510	1460	6.8	0.0057U	0.0064U	--	--	0.222U	0.0927U	--	--

Analyte	BITP-Influent				BITP-EBNR-Effluent				BIRWP-EBNR+F-Effluent			
	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD	Orig.	Rep.	Mean	RPD
b-Estradiol 3-benzoate	15.5U	21.8U	--	--	1.08U	2.05U	--	--	1.47U	1.2U	--	--
b-Sitosterol	508000	470000	489000	7.8	5670	3330	4500	52	13.2U	10.2U	--	--
b-Stigmastanol	40300	38600	39450	4.3	450	404	427	10.8	11.5U	6.35U	--	--
Campesterol	150000	150000	150000	0.0	694	698	696	0.6	5.3	3.1	4.2	52.1
Cholestanol	64900	62900	63900	3.1	1940	1830	1885	5.8	46.4	44.1U	46.4*	--
Cholesterol	2540000	2600000	2570000	2.3	7410	7080	7245	4.6	108U	101U	--	--
Coprostanol	1990000	2100000	2045000	5.4	6700	6340	6520	5.5	151	145	148	4.1
Desmosterol	10500	11000	10750	4.7	823	808	815.5	1.8	35.6	32.9	34.3	7.9
Desogestrel	18.1	18.9	19	4.3	1	0.808U	--	--	1.86U	1.43U	--	--
Epicoprostanol	23800	23600	23700	0.8	337	287	312	16	15	13.6	14.3	9.8
Equilenin	9.95U	8.06U	--	--	1.45U	1.6U	--	--	1.31U	1.27U	--	--
Equilin	31.8	31.5	32	0.9	0.758U	0.876U	--	--	1.43U	1.26U	--	--
Ergosterol	16400	15100	15750	8.3	1700	1610	1655	5.4	1.7	2.05U	1.7*	--
Estriol	144	133	139	7.9	0.676U	1.55U	--	--	0.825U	0.545U	--	--
Estrone	98.7	111	105	11.7	2.19U	1.88U	--	--	2.02U	0.828U	--	--
Mestranol	11.1U	9.76U	--	--	0.934U	1.02U	--	--	1.5U	1.52U	--	--
Norethindrone	18.4U	12.8U	--	--	1.83U	2.77U	--	--	4.05U	1.67U	--	--
Norgestrel	43.4	48.1	46	10.3	3.48U	8U	--	--	6.32U	6.46U	--	--
Progesterone	244U	421U	--	--	5.65U	9.42U	--	--	20.2U	10.1U	--	--
Stigmasterol	76500	70600	73550	8	8860	7960	8410	10.7	8.23U	6.37U	--	--
Testosterone	2900	3040	2970	4.7	8.72U	10.9U	--	--	31.8U	9.87U	--	--

-- Not detected.
* Not a mean because one replicate was undetected.
D = dilution; and the concentration was corrected at the laboratory. The D qualifier was not entered into EIM by protocol.

Overall, PPCPs ranged from 0 to 155%, with the 80th percentile mark at 42.1% for the influents, 35.1% for the secondary effluent, and 23.1 for the tertiary effluent.

Table D-8. Relative Percent Difference (RPD) for Axys replicate biosolids data collected at the Budd Inlet WWTP (BITP).

Analyte	BITP-Biosolids, ug/Kg (dw), ppb			
	Orig.	Rep.	Mean	RPD
PPCPs				
Acetaminophen	111U	105U	--	--
Azithromycin	145.0	142.0	144	2.1
Caffeine	7.43U	26.3U	--	--
Carbadox	2.78U	16.8U	--	--
Carbamazepine	393.0	323.0	358	19.6
Cefotaxime	33.6U	36.8U	--	--
Ciprofloxacin	11900.0	12800.0	12350	7.3
Clarithromycin	7.0	7.2	7	3.2
Clinafloxacin	33.3U	17.3U	--	--
Cloxacillin	9.94U	9.21U	--	--
Codeine	5.57U	5.27U	--	--
Cotinine	9.28UJ	8.78UJ	--	--
Dehydronifedipine	1.37U	1.27U	--	--
Diphenhydramine	2600.0	2450.0	2525	5.9
Diltiazem	7.7	6.7	7	14.1
Digoxin	92.8U	87.8U	--	--
Digoxigenin	20U	18.9U	--	--
Enrofloxacin	13.4	15.2	14	12.6
Erythromycin-H2O	13.8	7.6	11	58.1
Flumequine	5.16U	3.48U	--	--
Fluoxetine	675.0	630.0	653	6.9
Lincomycin	7.02U	5.27U	--	--
Lomefloxacin	9.6	7.5	9	24.4
Miconazole	1660.0	1530.0	1595	8.2
Norfloxacin	89.1	84.5	87	5.3
Norgestimate	9.47U	9.21U	--	--
Ofloxacin	6070.0	5500.0	5785	9.9
Ormetoprim	1.11U	1.05U	--	--
Oxacillin	5.57U	5.27U	--	--
Oxolinic Acid	2.7	2.1	2	25.9
Penicillin G	5.57U	5.27U	--	--
Penicillin V	11.1U	10.5U	--	--
Roxithromycin	1.77U	2.26U	--	--
Sarafloxacin	25.4U	24U	--	--
Sulfachloropyridazine	2.78U	2.63U	--	--
Sulfadiazine	2.78U	2.63U	--	--

Analyte	BITP-Biosolids, ug/Kg (dw), ppb			
	Orig.	Rep.	Mean	RPD
Sulfadimethoxine	0.617U	0.527U	--	--
Sulfamerazine	1.11U	1.05U	--	--
Sulfamethazine	3.71U	3.51U	--	--
Sulfamethizole	1.6U	1.54U	--	--
Sulfamethoxazole	1.4	1.14U	--	--
Sulfanilamide	92.8UJ	87.8UJ	--	--
Sulfathiazole	2.78U	2.63U	--	--
Thiabendazole	32.1	31.3	32	2.5
Trimethoprim	4.39U	5.57U	--	--
Tylosin	120U	245U	--	--
Virginiamycin	47.5U	61.9U	--	--
1,7-Dimethylxanthine	278U	263U	--	--
Gemfibrozil	277.0	223.0	250	21.6
Ibuprofen	460.0	415.0	438	10.3
Naproxen	13.6	6.9	10	65.6
Triclocarban	18400.0	17000.0	17700	7.9
Triclosan	8210.0	7760.0	7985	5.6
Warfarin	2.78U	2.63U	--	--
Anhydrochlortetracycline (ACTC)	27.8U	26.3U	--	--
Anhydrotetracycline (ATC)	291.0	301.0	296	3.4
Chlortetracycline (CTC)	11.1U	10.5U	--	--
Demeclocycline	27.8U	26.3U	--	--
Doxycycline	2370.0	2370.0	2370	0.0
4-Epianhydrochlortetracycline (EACTC)	111UJ	105UJ	--	--
4-Epianhydrotetracycline (EATC)	471.0	415.0	443	12.6
4-Epichlortetracycline (ECTC)	27.8U	26.3U	--	--
4-Epioxytetracycline (EOTC)	11.1U	10.5U	--	--
4-Epitetracycline (ETC)	3640.0	3400.0	3520	6.8
Isochlortetracycline (ICTC)	11.1U	10.5U	--	--
Minocycline	378.0	429.0	404	12.6
Oxytetracycline (OTC)	45.0	45.7	45	1.5
Tetracycline (TC)	3300.0	3280.0	3290	0.6
Albuterol	0.969U	0.567U	--	--
Cimetidine	55.1	35.7	45	42.7
Metformin	86.3U	63U	--	--
Ranitidine	5.0	6.4	6	24.6
Hormones/ Steroids				
17a-Ethinyl-Estradiol	40.1U	16.9U	--	--
17a-Dihydroequilin	18.2U D	21U D	--	--
17a-Estradiol	8.65U	4.64U	--	--

Analyte	BITP-Biosolids, ug/Kg (dw), ppb			
	Orig.	Rep.	Mean	RPD
17b-Estradiol	40.2U	46.1U	--	--
Androstenedione	263U D	190.0	190*	--
Androsterone	11.9	0.0962U D	12*	--
b-Estradiol 3-benzoate	74U D	46.3U D	--	--
b-Sitosterol	675000	737000	706000	8.8
b-Stigmastanol	366000	387000	376500	5.6
Campesterol	388000	377000	382500	2.9
Cholestanol	1590000	1720000	1655000	7.9
Cholesterol	756000	830000	793000	9.3
Coprostanol	3610000	3850000	3730000	6.4
Desmosterol	43100.0	47800.0	45450	10.3
Desogestrel	13.5U D	18.6U D	--	--
Epicoprostanol	2540000	2720000	2630000	6.8
Equilenin	14.5U D	20.1U D	--	--
Equilin	45.8	51.4	49	11.5
Ergosterol	37300	119000	78150	104.5
Estriol	20.6U D	3.01U	--	--
Estrone	58.2	53.2	56	9.0
Mestranol	30U	68.8	69*	--
Norethindrone	1590.0	101U D	1590*	--
Norgestrel	1900.0	119U D	1900*	--
Progesterone	652U	218U D	--	--
Stigmasterol	163000	178000	170500	8.8
Testosterone	134U D	216U D	--	--

-- Not detected.
* Not a mean because one replicate was undetected.
D = dilution; and the concentration was corrected at the laboratory. The D qualifier was not entered into EIM by protocol.

Budd Inlet biosolids RPDs for PPCPs[1694] and hormones/steroids were below 40% except for erythromycin-H20, cimetidine, naproxen, and ergosterol.

EPA Independent Review of the Axys Data Packages.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 Sixth Avenue
Seattle, Washington 98101

March 31, 2009

Reply to: OEA-095
Attn of: Ginna Grepo-Grove

MEMORANDUM

Subject: Data Validation Report for the Pharmaceuticals and Personal Care Products Analyses of Effluent, Influent and Bio-solid Samples Collected from the Puget Sound Outfalls

From: Ginna Grepo-Grove, R10 QA Manager
Office of Environmental Assessment, USEPA

To: Dave Ragsdale, Project Manager
Office of Water and Watersheds, USEPA

CC: Martha Turvey, Project Manager
Office of Water and Watersheds, USEPA

The quality assurance (QA) review of the analytical data generated from the analysis of 4 bio-solids and 16 aqueous samples collected from the above referenced site has been completed. These samples were analyzed for Pharmaceuticals, Personal Care (PPCP) Products, Steroids and Hormones (S&H) in accordance with the USEPA Method 1694, *"Pharmaceuticals and Personal Care Products in Water, soil, Sediment and Bio-solids by HPLC/MS/MS, December 2007"and* Method 1698 *"Steroids and Hormones Water, soil, Sediment and Bio-solids by HPGC/HR/MS, December 2007"*. The analyses were performed by Axys Laboratory located in Sudney, BC Canada. All the sample analyses were validated following the specification of the methods cited, the Lab's SOPs and the USEPA National Guidelines for Organic Data Review and the USEPA's *Guidance for Labeling Externally Validated Laboratory Analytical data for Superfund Use, EPA 540-R-08-05, January 2009*.

The following samples were evaluated in this validation report:

Table 1 – Sample Index, Dates of Collection, Extraction and Analysis.

EPA Sample No.	Lab Sample No.	Collection Dates	VTSR *	Extraction Dates PPCP	Extraction Dates H& S **	Analysis Date PPCP **	Analysis Date H&S
PUY Bio-Solid 4182	L11618-1	08/19/08	08/21/08	09/11 -17/08	11/25/08	09/19-28/08	12/2-16/08
CC Bio-Solid 4185	L11618-2	08/19/08	08/21/08	09/11 -17/08	11/25/08	09/19-28/08	12/2-16/08
LOTT Bio-Solid 4199	L11618-3	08/20/08	08/21/08	09/11 -17/08	11/25/08	09/19-28/08	12/2-16/08
LOTT Bio-Solid D 4200	L11619-4	08/20/08	08/21/08	09/11 -17/08	11/25/08	09/19-28/08	12/2-16/08
PUY- INF 4180	L11626 -4	08/19/08	08/21/08	09/11 -17/08	12/4/08	Sept-Dec 2008	
PUY-EFF 4181	L11626-5	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	12/11-13/08
CC- INF 4183	L11626-2	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	11/20/08
CC-EFF 4184	L11626-3	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08
CC-BLANK 4186	L11626-1	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	11/21/08
HAY INF 4187	L11626-6	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08
HAY SEFF 4188	L11626-7	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	11/20/08
HAY TEFF 4189	L11626-8	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	11/20/08
LOTT INF 4191	L11626-9	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08
LOTT INFD 4192	L11626-10	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08
LOTT SEFF 4193	L11626-11	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	11/20/08
LOTT SEFFD 4194	L11626-12	08/19/08	08/21/08	09/11 -17/08	10/31/08	Sept-Dec 2008	11/20/08
LOTT TEFF 4197	L11626-13	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11/08
LOTT TEFFD 4198	L11626-14	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11/08
MWPS MWRWP Influent	L11626-15	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08
MWRWP Effluent	L11626-16	08/19/08	08/21/08	09/11 -17/08	12/04/08	Sept-Dec 2008	12/11-13/08

- VTSR – Verified Time of Sample Receipt at the laboratory
- ** - Different Groups of Target Analytes were analyzed on different dates. Sample Results were not qualified based on Holding Times.

[*Ecology Insertion to this letter]*

The following EPA Sample Numbers correspond to the sampling location codes used in this report.

EPA Sample No.	Sample Code used in report and EIM
PUY Bio-Solid 4182	Puy- Biosolids
CC Bio-Solid 4185	CC-Biosolids
LOTT Bio-Solid 4199	BITP-Biosolids
LOTT Bio-Solid D 4200	BITP-Biosolids; flagged as duplicate in EIM
PUY- INF 4180	Puy-Influent
PUY-EFF 4181	Puy-AS+N-eff
CC- INF 4183	CC-Influent
CC-EFF 4184	CC-AS-eff
CC-BLANK 4186	CC-blank; (not in EIM)
HAY INF 4187	Hayden-Influent
HAY SEFF 4188	Hayden-AD-eff
HAY TEFF 4189	Hayden-CA+F-eff
LOTT INF 4191	BITP-Influent
LOTT INFD 4192	BITP-Influent; flagged as duplicate in EIM
LOTT SEFF 4193	BITP-EBNR-eff
LOTT SEFFD 4194	BITP-EBNR-eff; flagged as duplicate in EIM
LOTT TEFF 4197	BIRWP-EBNR+F-dis
LOTT TEFFD 4198	BIRWP-EBNR+F-dis; flagged as duplicate in EIM
MWPS MWRWP Influent	MWRWP-Influent
MWRWP Effluent	MWRWP-dis

DATA QUALIFICATIONS

The following comments refer to the laboratory performance in meeting the Quality Control Specifications outlined in the technical specifications of the USEPA Methods 1694 and 1698, *"Pharmaceuticals and Personal Care Products in Water, soil, Sediment and Bio-solids by HPLC/MS/MS, December 2007"and "Steroids and Hormones Water, soil, Sediment and Bio-solids by HPGC/HR/MS, December 2007"*, respectively and the *Guidance for Labeling Externally Validated Laboratory Analytical data for Superfund Use, EPA 540-R-08-05, January 2009*. Some of the data quality elements were qualified using the reviewer's professional judgment.

The conclusions presented herein are based on the information provided for the review.

Samples' Condition upon Receipt - Acceptable

All of the samples were received intact and were stored frozen by the laboratory at -20 ^{o}C while waiting for extraction. The integrity of the samples were maintained while on storage and waiting for analysis. None of the data were qualified on the basis of sample preservation.

Holding Time

USEPA has no established holding times for the PPCP, hormones and steroids target analytes. The analytical methods recommend the extraction of stored frozen samples within 7 days of sample collection to avoid the potential of losses and all of the samples were extracted on different dates for each of the target analyte group. The 7 day method recommended holding time was exceeded by all analyses, however, none of the data were qualified based on holding time exceedances.

Sample Preparation and Analysis – Acceptable

Samples were prepared and extracted in accordance with the USEPA Methods 1694 and 1698 and the lab Standard Operating Procedure Axys Method MLA-052 and MLA-068. Acid and basic PPCP extracts were cleaned through SPE cartridges as specified by the method. Each of the 4 group of PPCP target compounds were analyzed separately. Samples for hormones and steroids analyses were extracted using a continuous solvent extraction (18 hours). Primary extract were split and cleaned through chromatographic columns prior to separate analysis.

The sample matrices were so complex thus requiring the lab to perform multiple extractions and analyses for each of the 4 PPCP and 2 hormones and steroids groups. Most of the sample results were taken from several sample runs that were performed within a span of more than 30 days from extraction dates.

Instrument Performance - Acceptable

The frequency of system performance checks were met for all instruments used for the PPCP, hormones and steroids analyses. For hormones and steroids, the analyses were conducted using a HRGC/HRMS equipped with RTX-5 capillary column (30 m 0.25 mm id, 0.25 film thickness) and was operated in the electron ionization (EI) mode at a static power mass resolution of >5000. The appropriate switching times for the Selected Ion Monitoring (SIM) descriptors, the chromatographic resolutions and retention times were checked prior to each analytical sequence. All of the hormones and steroids target and labeled compounds met the signal to noise ratio (S/N) 3:1, the ion abundance criteria specified by the method and the retention times criteria.

The frequency of system performance check for each group of PPCP target and labeled compounds were met. The MS instrument was calibrated and optimized prior to the analysis of the 4 groups of PPCP target and labeled compounds. Each target and labeled compound peaks met the S/N criterion of >10 and the retention time limits. The chromatographic resolutions of each target compound were resolved >75% of each other.

Initial Calibrations

Several initial calibrations (ICAL) were performed for each group of target parameters. The frequency of analysis, percent relative standard deviations (%RSDs), percent recoveries, the correlation coefficient (r) of >0.995, the ion abundance, S/N ratios, retention times and chromatographic resolution criteria were met for most of the target compounds. Some of the target compounds did not meet the correlation coefficient (r). However, since quantifications used isotope dilution techniques, none of the reported results were qualified on this basis.

Continuing Calibrations Verification Standards (VERS)

The frequency of analysis, the percent recoveries, retention times, chromatographic resolution, ion abundance and S/N ratio criteria were met by all VERS with a few exceptions. Some of target and labeled compounds did not meet recovery control limits, however, since quantifications used isotope dilution techniques, none of the reported results were qualified on this basis.

Ongoing Precision and Recovery (OPR) - Acceptable

The frequency of analysis and recovery criteria were met by all OPRs extracted and analyzed with the samples. A few target and labeled compounds did not meet the recovery control limits; however, since quantifications used isotope dilution techniques, none of the reported results were qualified on this basis.

Compound Quantitation and Detection Limits

All of the samples were analyzed at the project required concentration levels. Multiple analyses needed to be performed for almost all samples to get the detected analytes within instrument's linear calibration range. Some target compounds laboratory reporting limits were raised due to the elevated detection limits from interferences and/or contamination in the associated blank.

Compound Identification

All of the detected compounds met the technical acceptance criteria for identification, e.g., S/N ratios greater than 2.5, ion abundance ratios, RRTs within established limits. None of the data were qualified on this basis.

Method Blanks

The frequency of analysis of laboratory blank was met. Some of the target compounds were detected in the method blanks at concentration levels that were acceptable as specified by the methods. To avoid potential false positives due to blank contamination, results in the associated samples at concentrations <5x the value in the method blank were qualified non-detects, "U". Concentrations >5x were not qualified.

Analytical Sequence - Acceptable

All of the standards, blanks, samples, and QC samples were analyzed in accordance with the method specified analytical sequence. Mass ion locks and resolution and window defining mix were analyzed and checked at the beginning and end of each analytical sequence. All of the analytical sequences were also bracketed by the continuing calibration check standards. None of the data were qualified on this basis.

Internal Standards Recoveries

Some of the labeled compounds did not meet the recovery control limits. The affected target compounds were qualified accordingly.

Clean-up and Recovery Standard - Acceptable

Surrogate was not required for this method. However, clean-up and recovery standard were added to all samples and QC samples to monitor losses and clean-up efficiency. The clean-up standard recoveries were acceptable for all analyses. None of the data were qualified on this basis.

Matrix Spike and Matrix Spike Duplicate

Sample LOTT SEFF 4193 was designated as the QC sample for MS/MSD analysis. The frequency of analysis, recovery, and relative percent difference (RPD) were met with a few exceptions. The associated sample results were qualified accordingly.

Laboratory Contact

The laboratory was not contacted for this review.

Overall Assessment

All of the samples were analyzed in accordance with the methods specifications. With the exception of a couple of data points that were flagged unusable due to extremely low IS recoveries (<10%), the rest of the data, as qualified, are acceptable and can be used for all purposes.

Data Qualifiers	
U	The analyte was not detected at or above the reported result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result. The associated numerical value is an estimate of the quantitation limit of the analyte in this sample.
R	The data are unusable for all purposes.
N	There is evidence the analyte is present in this sample.
JN	There is evidence that the analyte is present. The associated numerical result is an estimate.

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PHARMACEUTICALS ANALYSIS

INFLUENT/EFFLUENT/PROCESS SAMPLES

AXYS METHOD: MLA-052

PROJECT NUMBER: PF324169

PROJECT NAME: DST27

Contract: 4404
Data Package Identification: DPWG27192
Analysis WG26496 and WG26497

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

26 November 2008

WASHINGTON STATE DEPT OF ECOLOGY
SLUDGE SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-052
4499: L11618-1 to -4

Project Name: DST27

27 November 2008

NARRATIVE

This narrative describes the analysis of four sludge samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC-MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at -20°C prior to extraction and analysis.

SAMPLE PREPARATION AND ANALYSIS

The samples were pre-treated prior to analysis, as documented on the Solid Preparation Record forms included in this data package.

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG26496 and WG26497 for acid- and base-extracted pharmaceutical compounds, respectively. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-052: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Compounds in Solid and Aqueous Samples by LC-MS/MS***. A method summary of AXYS Method MLA-052 is included in the data package.

Two aliquots of accurately weighed sub-sample for each sample (approximately 0.5 gram dry weight) were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH2 and pH 10, respectively, in two separate analysis batches WG26496 and WG26497. The resulted extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4

WASHINGTON STATE DEPT OF ECOLOGY
SLUDGE SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-052
4499: L11618-1 to -4

Project Name: DST27

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List 3	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.0 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

For all target compounds, linear equations were determined from a multi-point calibration series with 1/X weighting fit and expressed as below:

$$Y = \text{slope} \times X + \text{intercept}$$

Where: $Y = \text{response ratio} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} \right)$

$X = \text{weight of target (ng)}$

The slope and intercept were used to convert raw peak areas in sample chromatograms to final concentrations as follows:

Sample Conc. =

$$\left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} - \text{intercept} \right) \times \left(\frac{1}{\text{slope}} \right) \times \left(\frac{1}{\text{sample size(g)}} \right)$$

The recovery of the surrogate standard was calculated and monitored as an indication of overall method performance.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.0 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L11618-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

- i = instrumental re-analysis was performed on the sample extract
- N = dilution of the sample extract in a new micro vial, followed by instrumental reanalysis.

The following laboratory qualifier flags were used in this data package:

- D = dilution data
- E = exceeds calibration range

N = authentic recovery is not within method/contract control limits
U = identifies a compound that was not detected
V = surrogate recovery is not within method/contract control limit.

Results are reported in concentration units of nanograms per gram (ng/g), dry weight basis. Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

Due to Ion suppression that caused significant drop of responses, the highest-level calibration standards in the initial calibrations were excluded for Cimetidine, Ranitidine, d6-Metformin (data filename PP8K_171 S:20), Caffeine, Carbadox, Cefotaxime, Clinafloxacin, Digoxin, Digoxigenin, Norgestimate, Ofloxacin, Ormetoprim, Sarafloxacin, Sulfachloropyridazine, Sulfanilamide, Sulfathiazole, 1,7-Dimethylxanthine (data filename PP8J_156 S:23), EACTC, EATC and Minocycline (PP8K_221 S:10). As low responses were observed, the lowest-level calibration standards were excluded for Cotinine, Digoxin, Sulfamethazine and Sulfanilamide (data filename PP8J_156 S:11). However, a minimum of 5 (for list 1 and list 3 analytes) or 6 (for list 2 and list 4 analytes) calibration standard points was used to construct the linear equations for quantification of target analytes or to calculate response factor (RF) for quantification of labeled surrogates except for the following compounds. 4 calibration points were used to construct linear calibration equations for Digoxin and Sulfanilamide. Digoxin and Sulfanilamide were not detected in all client samples; therefore, sample data were not affected.

Percent recoveries of target analyte Digoxin in initial CS3 level calibration standard PP8J_156 S:19 and 1,7-Dimethylxanthine in initial CS0 level calibration standard PP8J_156 S:11 were above the method upper control limit 140%. Correlation coefficients for Digoxin and 1,7-Dimethylxanthine in this initial calibration were slightly below the method control limit 0.985. As multi-point calibrations were used, slight variations in recovery and correlation coefficient are deemed not to significantly affect sample data. Percent recoveries of labeled surrogate $^{13}C_3$-Caffeine for CS0 and CS3 (data filename PP8J_156 S:11 and S:19, respectively) in this initial calibration were in the range of 60 to 140%. Given that recoveries of all target analytes quantified using this labeled surrogate in the calibration standards were within the method control limits, sample analytes data were not affected by the variances. In addition, Caffeine, Digoxin and 1,7-Dimethylxanthine were not detected in all client samples; sample analytes data were not impacted by the variances.

The correlation coefficient for Demeclocycline in initial calibration PP8K_221 S:4-10 was slightly below the method control limit 0.985. As multi-point calibrations were used, slight variation in correlation coefficient is deemed not to significantly affect sample data.

Percent recoveries of labeled surrogates d_6-Metformin and $^{13}C_{12}$-Triclosan in initial CS5 level calibration standards (data filename PP8K_171 S:19 and PP8K_175 S:23, respectively) were below the method lower control limits. Given that recoveries of all target analytes quantified using these labeled surrogates in the calibration standards were within the method control limits, sample analytes data were not affected by the variances.

Percent recoveries of target analytes Digoxin and Sulfanilamide in calibration verification PP8J_156 S:55 were outside the method nominal control limits. Given that Digoxin and Sulfanilamide were not detected in all client samples, sample analytes data were not affected by the variances.

Percent recoveries of labeled surrogates $^{13}C_6$-Triclocarban and d_5-Warfarin in calibration verification PP8K_175 S:50 were above the method nominal upper control limit 130%. Given that recoveries of all target analytes quantified using these labeled surrogates in the calibration verification were within the method control limits, sample analytes data were not affected by the variances.

The recovery of target analyte Caffeine in the OPR (AXYS ID WG26496-102) was above the AXYS method upper control limits while recoveries of Sulfanilamide, Anhydrotetracycline, 4-Epianhydrochlortetracycline and Minocycline in the OPR (AXYS ID WG26496-102) and Cimetidine in the OPR (AXYS ID WG26497-102) were below the AXYS method lower control limits; these compounds are flagged with an 'N' on the report from. Given that Caffeine was not detected in all client samples, sample Caffeine data were not impacted by the variances. Sample data for the rest analytes might be similarly under-estimated as the OPR. However, percent recoveries of Sulfanilamide, Anhydrotetracycline, 4-Epianhydrochlortetracycline, Minocycline and Cimetidine in all OPRs were all within the USEPA Method 1694 acceptance criteria.

The percent recovery of $^{13}C_2$-^{15}N-Acetaminophen in sample LOTT-Biosolid D 4200 (AXYS ID L11618-4) were above the method upper control limits. The percent recovery of $^{13}C_3$-N_{15}-Ciprofloxacin in the Lab Blank (AXYS ID WG26496-101) were below the method lower control limits These labeled surrogates are flagged with a 'V' on the report form. Since the isotope dilution method of quantification produces data that are recovery corrected, the variances of surrogate recoveries from the method acceptance criteria are deemed not to affect the quantification of the analytes. Percent recoveries of labeled quantification standards are used as general method performance indicator only. In addition, as Acetaminophen was not detected in the sample, sample data were not impacted by the variance.

ANALYTICAL DISCUSSION

List 1 Compounds

To bring the area response of target analyte Ciprofloxacin and/or Diphenhydramine to within the calibrated linear range of the instrument, all client sample extracts were diluted and instrumentally re-analyzed. Data obtained from the analysis of the diluted extracts are reported for Ciprofloxacin and/or Diphenhydramine (indicated by suffix 'N' on the AXYS ID). Dilution factors are noted on the report form.

Extracts for the Lab Blank and the OPR (AXYS ID WG26496-101 and –102 respectively) were routinely re-analyzed for a second time on instrument for confirmative purposes; results obtained in the initial analysis are reported.

List 2 Compounds

Due to not all method control limits were met in initial analysis, extracts for all client samples and QC samples were re-analyzed on instrument for List 2 compounds. To confirm results, extracts for all client samples and QC samples were re-analyzed for a second time on instrument with a new initial calibration standard series. Data obtained from the second re-analysis are reported as indicated by the suffix 'i2' on the AXYS ID. Extracts for the Lab Blank and the OPR (AXYS ID WG26496-101 and –102 respectively) were routinely re-analyzed on instrument for an additional time for confirmative purposes; results obtained in the third or fourth re-analyses are reported for the Lab Blank and the OPR, respectively (indicated by the suffix 'i3' or 'i4' on the AXYS ID).

In initial analysis, area responses of some target analytes in all client samples were observed above the calibrated linear range of the instrument; all sample extracts were diluted and re-analyzed on instrument. However, when sample extracts were re-analyzed with the new initial calibration standard series, all area

responses were within the calibrated linear range. Therefore, data obtained in analysis of the diluted extracts are not required.

List 3 Compounds

To lessen suppressions or to bring area responses of some target analytes to within the calibrated linear range of the instrument, all client sample extracts were diluted and instrumentally re-analyzed. Data obtained from the analysis of the diluted extracts are reported for the affected analytes (indicated by suffix 'N' on the AXYS ID). Dilution factors are noted on the report form.

Extracts for the Lab Blank and the OPR (AXYS ID WG26496-101 and –102 respectively) were routinely re-analyzed for a second time on instrument for confirmative purposes; results obtained in the initial analysis are reported.

List 4 Compounds

Due to not all method control limits were met in initial analysis, extracts for all client samples and QC samples were re-analyzed on instrument for List 4 compounds. Data obtained from the re-analysis are reported as indicated by the suffix 'i' on the AXYS ID. Extracts for the Lab Blank and the OPR (AXYS ID WG26497-101 and –102 respectively) were routinely re-analyzed for a second time on instrument for confirmative purposes; results obtained in the first re-analysis are reported.

Suppressions affecting the response of recovery (internal) standard $^{13}C_3$-Atrazine were observed in all client samples. All sample extracts were diluted and re-analyzed on instrument. Data obtained from the analysis of the diluted extracts indicated that the percent recovery of $^{13}C_3$-Atrazine was within method control limits while concentrations of target analytes were in a good agreement with original data. Therefore, original data were not impacted by the variances; original data are reported.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG27192, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: (Matthew) Ziqing Ou, PhD, QA Chemist

November 27, 2008
Date Signed

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PHARMACEUTICALS ANALYSIS

EFFLUENT and INFLUENT SAMPLES

AXYS METHOD: MLA-052

PROJECT NAME: LOTT ALLIANCE

Contract: 4404
Data Package Identification: DPWG27438
Analysis WG26425 and WG26426

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

17 December 2008

WASHINGTON STATE DEPT OF ECOLOGY
AQUEOUS SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-052
4499: L11626-15 and -16

Project Name: LOTT ALLIANCE

18 December 2008

NARRATIVE

This narrative describes the analysis of two aqueous samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC-MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at 4°C prior to extraction and analysis. Some discrepancies between the sample labeling and client COC were noted on receiving records and resolved through communicating to the client.

SAMPLE PREPARATION AND ANALYSIS

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG26425 and WG26426. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-052: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Compounds in Solid and Aqueous Samples by LC-MS/MS***. A method summary of AXYS Method MLA-052 is included in the data package following this narrative.

Two aliquots of accurately weighed sub-sample for each sample were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH2 and pH 10, respectively. The resulted extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
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List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.0 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

For all target compounds, linear equations were determined from a multi-point calibration series with 1/X weighting fit and expressed as below:

$$Y = \text{slope} \times X + \text{intercept}$$

Where: $Y = \text{response ratio} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} \right)$

$X = \text{weight of target (ng)}$

The slope and intercept were used to convert raw peak areas in sample chromatograms to final concentrations as follows:

Sample Conc. =

$$\left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} - \text{intercept} \right) x \left(\frac{1}{\text{slope}} \right) x \left(\frac{1}{\text{sample size(L)}} \right)$$

The recovery of the surrogate standard was calculated and monitored as an indication of overall method performance.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.0 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L11626-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

- I = instrumental re-analysis was performed on the sample extract
- N = dilution of the sample extract in a new micro vial, followed by instrumental reanalysis.

The following laboratory qualifier flags were used in this data package:

- D = dilution data
- E = exceeds calibration range
- N = authentic recovery is not within method/contract control limits

U = identifies a compound that was not detected
V = surrogate recovery is not within method/contract control limit.
X = results reported separately.

Results are reported in concentration units of nanograms per liter (ng/L). Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and would not affect the data.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

Initial calibrations:

In order to meet the initial calibration requirements, the lower or higher end calibration standards in the initial calibrations may be excluded for some compounds.

Initial calibration series PP8J_156 S: 11, 15, 17, 19, 21, 23: Four calibration points (S: 15, 17, 19, 21) were used to construct linear calibration equations for Digoxin. The percent recovery for Digoxin is slightly above the method upper limit of 140% in the calibration point (CS3, filename PP8J_156 S:19) but meet method criteria for all the other calibration points. The percent recovery for 1,7-Dimethylxanthine is slightly above the method upper limit of 140% in the calibration point (CS1, filename PP8J_156 S:11) but meet method criteria for all the other calibration points. Regression co-efficient values for Caffeine (0.979), Digoxin (0.934) and 1,7-Dimethylxanthine (0.964) are slightly below the method limit of 0.985. Percent recoveries for Caffeine (141%) and Digoxin (133%) in the associated Calibration verification (filename PP8J_156 S: 30) are slightly above the method upper limit of 130%. Caffeine was detected in the associated sample MWPS-MWRWP Influent (AXYS ID L11626-15), and the concentration may be slightly over-reported. Digoxin was not detected in the associated sample L11626-15, data were not affected. Percent recovery of 1,7-Dimethylxanthine in the associated Calibration verification (filename PP8J_156 S: 30) meets the method criteria, the results for this compound in the associated sample L11626-15 are not considered affected.

Initial calibration series PP8J_165 S: 7 to 12: Four calibration points (S: 7 to 11) were used to construct linear calibration equations for Digoxin. Regression co-efficient value (0.982) for Sulfanilamide is slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the linearity, the associated calibration verification (filename PP8J_165 S: 36) and the OPR (AXYS ID WG26425-102) meet the method criteria, and these compounds were not detected in the associated sample and the lab blank (AXYS ID L11626-16 and WG26425-101 respectively), the data are not affected by the variances.

Initial calibration series PP8K_182 S: 9 to 15: The percent recovery of the surrogate d6-Metformin in the calibration point (filename PP8K_182 S: 15) is below the method lower limit of 60% but meets the method requirements for other calibration points and in the associated calibration verification (filename PP8K_182 S: 43). Data are not considered affected.

Initial calibration series PP8K_210 S: 9 to 15: Regression co-efficient values for Anhydrochlortetracycline (ACTC: 0.952), Chlortetracycline (CTC: 0.979) and Oxytetracyclin (OTC: 0.981) are slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the initial calibration and the associated calibration verification (filename PP8K_210 S: 19) meet the method criteria, data are not considered affected by this variance.

Initial calibration series PP8K_211 S: 5 to 11: Four calibration points (S: 5 to 8) were used to construct linear calibration equations for Anhydrochlortetracycline (ACTC). Regression co-efficient value (0.979) for minocycline is slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the initial calibration and the associated calibration verification (filename PP8K_211 S: 15) meet the method criteria, data are not considered affected by this variance.

Calibration verifications:

Calibration verification PP8K_165 S: 39: the percent recovery (148%) for the labeled surrogate d5-Warfarin is above the method upper limit of 130% but the percent recovery for the authentic compound Warfarin is within acceptance range. The percent recovery for the labeled surrogate may be similarly biased high in the associated sample MWPS-MWRWP Influent (AXYS ID L11626-15) but the target Warfarin concentration would not be affected by this variance.

Calibration verification PP8K_168 S: 4: the percent recovery (60.7%) for the labeled surrogate d5-Warfarin is below the method lower limit of 70% but the percent recovery for the authentic compound Warfarin is within acceptance range. The percent recovery for the labeled surrogate may be similarly biased low in the associated sample MWRWP-Effluent (AXYS ID L11626 -16). The target Warfarin was not detected in the sample, data were not affected.

Calibration verification PP8J_156 S: 30: percent recoveries for Caffeine (141%), Digoxin (133%), and Sulfanilamide (65.9%) are slightly outside of the method limits of 70-130%. Caffeine was detected in the associated sample MWPS-MWRWP Influent (AXYS ID L11626-15), and the concentration may be slightly over-reported. Both the analytes Digoxin and Sulfanilamide were not detected in the associated sample L11626-15, data were not affected.

Calibration verification PP8J_165 S: 36: percent recoveries for Diphenhydramine (62.6%) and Virginiamycin (62.3%) are slightly below the method lower limits of 70%. However, the percent recoveries in the associated OPR (AXYS ID WG26425-102) meet the method criteria, indicating that the data in the associated sample MWRWP-Effluent and the lab blank (AXYS ID L11626-16 and WG26425-101, respectively) would not be affected by this variance.

OPRs:

Percent recoveries of the compounds listed in the following table in the OPR (AXYS ID WG26425-102) are outside of the method control limits and these compounds have been flagged with an 'N' on report forms. Results for these compounds may be similarly impacted for the samples.

Compounds	Authentic (%rec)	Lower Limit (%rec)	Upper Limit (%rec)
CEFOTAXIME	29.7	70	200
MICONAZOLE	25.1	50	200
NORFLOXACIN	153	50	150
NORGESTIMATE	30.6	40	150
MINOCYCLINE	41.7	50	150

Percent recoveries of Cimetidine and Ranitidine in the OPR (AXYS ID WG26426-102) are slightly below the method lower control limits and these two compounds have been flagged with an 'N' on report from. Results in the samples may be similarly affected.

Labeled Surrogates:

Percent recoveries for some labeled surrogates in samples and the lab blank (listed in the following table) fall outside of the control limits and these surrogates have been flagged with a 'V' on report forms. Given that the data were quantified using isotope dilution/internal standard method, slight variation in surrogate recovery is not deemed to have significantly affected the data.

CLIENT ID	AXYS ID	V flagged surrogate
MWPS-MWRWP Influent	L11626-15	D3-Cotinine, D5-Warfarin
MWRWP-Effluent	L11626-16	13C2-Erythromycin-H2O
Lab Blank	WG26425-101	D3-Cotinine, 13C6-Triclocarban

Percent recovery of the labeled surrogate d6-Metformin is slightly below the method lower control limit for the QC samples (AXYS ID WG26426-101 and -102, respectively) and the client samples, and the surrogate has been flagged with a 'V' on report forms. However, the percent recovery for the associated target, Metformin, falls within the acceptance range for the OPR, indicating that the data would not be affected by this variance.

ANALYTICAL DISCUSSION

List 1 Compounds

To improve surrogate recoveries, all QC samples and client samples were diluted and instrumentally re-analyzed. Dilution data are reported (indicated by suffix 'N' on the AXYS ID). Dilution factors are reported on the report forms.

List 2 Compounds

To confirm results, extracts for all client samples and QC samples were instrumentally re-analyzed. The re-analysis data are reported (indicated by the suffix 'i' on the sample AXYS IDs). Extracts for the Lab Blank and the OPR (AXYS ID WG26425-101 and –102, respectively) were routinely re-analyzed on instrument for an additional time for confirmative purposes; the secondary re-analysis data are reported for the Lab Blank and the OPR (indicated by the suffix 'i2' on the AXYS IDs).

List 3 Compounds

To bring area responses of some target analytes to within the calibrated linear range of the instrument, all client sample extracts were diluted and instrumentally re-analyzed. The results for the affected analytes are reported from the dilution data (indicated by suffix 'N' on the AXYS ID). Dilution factors are noted on the report form.

List 4 Compounds

To improve surrogate recoveries, the client samples were diluted and instrumentally re-analyzed. Dilution data are reported (indicated by suffix 'N' on the AXYS ID). Dilution factors are reported on the report forms.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG27438, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Henry Huang, PhD, QA Chemist

19 Dec 2008
Date Signed

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PHARMACEUTICALS ANALYSIS

INFLUENT and EFFLUENT SAMPLES

AXYS METHOD: MLA-052

PROJECT NUMBER: PF324169

PROJECT NAME: DST27

Contract: 4404
Data Package Identification: DPWG27439
Analysis WG26425 and WG26426

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

18 December 2008

WASHINGTON STATE DEPT OF ECOLOGY
AQUEOUS SAMPLES

PHARMACEUTICALS ANALYSIS
AXYS METHOD: MLA-052
4499: L11626-1 to -14

Project Name: DST27

21 December 2008

NARRATIVE

This narrative describes the analysis of fourteen aqueous samples for the determination of pharmaceutical products using High Performance Liquid Chromatography coupled with tandem Mass Spectrometry (LC- MS/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions upon receipt are provided on the Sample Receiving Record form included in this data package. The samples were stored at 4°C prior to extraction and analysis. Some discrepancies between the sample labeling and client COC were noted on receiving records and resolved through communicating to the client.

SAMPLE PREPARATION AND ANALYSIS

Samples and QC samples (a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were analyzed in two analysis batches named WG26425 and WG26426. Composition of each analysis batch is shown on the Cover Page and Correlation Table, and on the Batch List that accompanies the extraction workup sheets.

Extraction and analysis procedures were in accordance with AXYS Method MLA-052: ***Analytical Procedure for the Analysis of Pharmaceutical and Personal Care Compounds in Solid and Aqueous Samples by LC-MS/MS***. A method summary of AXYS Method MLA-052 is included in the data package following this narrative.

Two aliquots of accurately weighed sub-sample for each sample were spiked with labeled quantification standards and extracted with acetonitrile using sonication at pH2 and pH 10, respectively. The resulted extracts were reduced in volume, reconstituted in water and cleaned up on Waters Oasis HLB SPE cartridges. The final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

Analysis was performed on Waters 2690 or 2795 HPLC equipped with Micromass Quattro Ultima MS/MS using four instrument and LC conditions as shown in table below.

Target Group	LC Column	Ionization	Acquisition	LC Conditions
List 1	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	1
List 2	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	2
List 3	Waters Xtera C18 (10.0 cm, 2.1 mm i.d., 3.5 µm particle size)	Negative Ion Electrospray	MRM mode, unit resolution	3
List 4	Waters Atlantis HILIC (10.0 cm, 2.1 mm i.d., 3.0 µm particle size)	Positive Ion Electrospray	MRM mode, unit resolution	4

CALCULATION

Target analyte concentrations were determined by isotope dilution or internal standard quantification procedures using MassLynx 4.0 software. Quantification was conducted by comparing the area of the quantification ion to that of the quantification standard (surrogate) and correcting for response factors.

For all target compounds, linear equations were determined from a multi-point calibration series with 1/X weighting fit and expressed as below:

$$Y = \text{slope} \times X + \text{intercept}$$

Where: $Y = \text{response ratio} = \left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} \right)$

X = weight of target (ng)

The slope and intercept were used to convert raw peak areas in sample chromatograms to final concentrations as follows:

Sample Conc. =

$$\left(\frac{\text{area of Target}}{\text{area of Surrogate}} \times \text{weight of Surrogate (ng)} - \text{intercept} \right) \times \left(\frac{1}{\text{slope}} \right) \times \left(\frac{1}{\text{sample size(L)}} \right)$$

The recovery of the surrogate standard was calculated and monitored as an indication of overall method performance.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. If the MassLynx 4.0 software selected an unrepresentative area for the detection limit calculation, the data interpretation chemist or the QA chemist made corrections. These corrections are hand noted on the quantification report pages attached to the chromatograms.

The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed, prorated for the extract volume and sample size, or the SDL, whichever is greater.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4499. The samples were assigned a unique laboratory identifier L11626-X, where X is a numeral. All data reports reference the unique AXYS IDs plus the client sample identifiers.

Any extra work required and performed after the initial instrumental analysis of the sample's extract is given an extra "test suffix" code. The single letter code per extra work performed is added to the AXYS sample ID as a suffix, and is combined with any other applicable test suffix codes. The extra work codes used to report data in this package include:

- i = instrumental re-analysis was performed on the sample extract
- N = dilution of the sample extract in a new micro vial, followed by instrumental reanalysis.

The following laboratory qualifier flags were used in this data package:

- D = dilution data.
- E = exceeds calibration range.
- N = authentic recovery is not within method/contract control limits.

NQ = not quantifiable.
U = identifies a compound that was not detected.
V = surrogate recovery is not within method/contract control limit.
X = results reported separately.

Results are reported in concentration units of nanograms per liter (ng/L). Concentration and reporting limits are provided to three significant figures.

QA/QC NOTES

Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. The data should be evaluated with consideration of the procedural blank results.
- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and cleanup.
- Due to the limitation of the software, signal to noise ratio (S/N) was measured as '0' in some cases where even a large peak was present. This has been visually inspected and would not affect the data.
- All linearity, calibration verification, OPR, MS/MSD and labeled compound recovery specifications were met with the following exceptions:

Initial calibrations:

In order to meet the initial calibration requirements, the lower or higher end calibration standards in the initial calibrations may be excluded for some compounds.

Initial calibration series PP8J_156 S: 11, 15, 17, 19, 21, 23: Four calibration points (S: 15, 17, 19, 21) were used to construct linear calibration equations for Digoxin. The percent recovery for Digoxin is slightly above the method upper limit of 140% in the calibration point (CS3, filename PP8J_156 S:19) but meet method criteria for all the other calibration points. The percent recovery for 1,7-Dimethylxanthine is slightly above the method upper limit of 140% in the calibration point (CS1, filename PP8J_156 S:11) but meet method criteria for all the other calibration points. Regression co-efficient values for Caffeine (0.979), Digoxin (0.934) and 1,7-Dimethylxanthine (0.964) are slightly below the method limit of 0.985. Percent recoveries for Caffeine (141%) and Digoxin (133%) in the associated Calibration verification (filename PP8J_156 S: 30) are slightly above the method upper limit of 130%. Caffeine was detected in some of the associated samples, and the concentration may be slightly over-reported. Digoxin was not detected in any of the associated samples, data were not affected. Percent recovery of 1,7-Dimethylxanthine in the associated Calibration verification (filename PP8J_156 S: 30) meets the method criteria, the results for this compound in the associated samples are not considered affected.

Initial calibration series PP8J_163 S: 13 to 18: Four calibration points (S: 13 to 16) were used to construct linear calibration equations for Digoxin. However, the percent recoveries for the compound in the linearity and the associated calibration verification (filename PP8J_163 S: 66), the data are not considered affected by the variance.

Initial calibration series PP8J_165 S: 7 to 12: Four calibration points (S: 7 to 11) were used to construct linear calibration equations for Digoxin. Regression co-efficient value (0.982) for Sulfanilamide is slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the linearity, the associated calibration verification (filename PP8J_165 S: 36) and the OPR (AXYS ID WG26425-102) meet the method criteria, and these compounds were not detected in the associated samples and the lab blank, the data are not affected by the variances.

Initial calibration series PP8K_182 S: 9 to 15: The percent recovery of the surrogate d6-Metformin in the calibration point (filename PP8K_182 S: 15) is below the method lower limit of 60% but meets the method requirements for other calibration points and the associated calibration verification (filename PP8K_182 S: 43). Data are not considered affected.

Initial calibration series PP8K_210 S: 9 to 15: Regression co-efficient values for Anhydrochlortetracycline (ACTC: 0.952), Chlortetracycline (CTC: 0.979) and Oxytetracyclin (OTC: 0.981) are slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the initial calibration and the associated calibration verification (filename PP8K_210 S: 19) meet the method criteria, data are not considered affected by this variance.

Initial calibration series PP8K_211 S: 5 to 11: Four calibration points (S: 5 to 8) were used to construct linear calibration equations for Anhydrochlortetracycline (ACTC). Regression co-efficient value (0.979) for minocycline is slightly below the method limit of 0.985. However, the percent recoveries for these compounds in the initial calibration and the associated calibration verification (filename PP8K_211 S: 15) meet the method criteria, data are not considered affected by this variance.

Calibration verifications:

Calibration verification PP8K_165 S: 39: the percent recovery (148%) for the labeled surrogate d5-Warfarin is above the method upper limit of 130% but the percent recovery for the authentic compound Warfarin is within acceptance range. The percent recovery for the labeled surrogate may be similarly biased high in the associated samples but the target Warfarin concentration would not be affected by this variance.

Calibration verification PP8K_168 S: 4: the percent recovery (60.7%) for the labeled surrogate d5-Warfarin is below the method lower limit of 70% but the percent recovery for the authentic compound Warfarin is within acceptance range. The percent recovery for the labeled surrogate may be similarly biased low in the associated samples. The target Warfarin was not detected in the sample, data were not affected.

Calibration verification PP8J_156 S: 30: percent recoveries for Caffeine (141%), Digoxin (133%), and Sulfanilamide (65.9%) are slightly outside of the method limits of 70-130%. Caffeine was detected in some of the associated samples, and the concentration may be slightly over-reported. Both the analytes Digoxin and Sulfanilamide were not detected in the associated samples, data were not affected.

Calibration verification PP8J_163 S: 66: percent recoveries for Diphenhydramine (59.6%) and Virginiamycin (63.9%) are slightly below the method lower limits of 70%. However, the percent recoveries in the associated OPR (AXYS ID WG26425-102) meet the method criteria, indicating that the data in the associated samples would not be affected by this variance. The percent recovery (148%) of the surrogate 13C2-15N-Acetaminophen is slightly above the method upper limit of 130%, but the percent for the target, Acetaminophen, falls within acceptance range. The recovery for the surrogate in the associated samples may be similarly high biased but the target results would not be affected by this variance.

Calibration verification PP8J_165 S: 36: percent recoveries for Diphenhydramine (62.6%) and Virginiamycin (62.3%) are slightly below the method lower limits of 70%. However, the percent recoveries in the associated OPR (AXYS ID WG26425-102) meet the method criteria, indicating that the data in the associated samples and the lab blank would not be affected by this variance.

OPRs:

Percent recoveries of the compounds listed in the following table in the OPR (AXYS ID WG26425-102) are outside of the method control limits and these compounds have been flagged with an 'N' on report forms. Results for these compounds may be similarly impacted for the samples.

Compounds	Authentic (%rec)	Lower Limit (%rec)	Upper Limit (%rec)
CEFOTAXIME	29.7	70	200
MICONAZOLE	25.1	50	200
NORFLOXACIN	153	50	150
NORGESTIMATE	30.6	40	150
MINOCYCLINE	41.7	50	150

Percent recoveries of Cimetidine and Ranitidine in the OPR (AXYS ID WG26426-102) are slightly below the method lower control limits and these two compounds have been flagged with an 'N' on report from. Results in the samples may be similarly affected.

MS/MSD:

Percent recoveries for some compounds did not meet the method internal criteria of 50-150%. Since the OPR meets the method criteria, the issues with MS/MSD may be due to the spiked amount of the compound being too small compared to the original sample background and/or matrix impact, sample data may similarly impacted.

Labeled Surrogates:

Percent recoveries for some labeled surrogates in samples and the lab blank (listed in the following table) fall outside of the control limits and these surrogates have been flagged with a 'V' on report forms. Given that the data were quantified using isotope dilution/internal standard method, slight variation in surrogate recovery is not deemed to have significantly affected the data.

CLIENT ID	AXYS ID	V flagged surrogate
CC-Blank 4186	L11626-1	D3-Cotinine, 13C6-Triclocarban
CC-Inf 4183	L11626-2	d6-Metformin
CC-Eff 4184	L11626-3	13C2-15N-Acetaminophen, 13C3-Caffeine, 13C6-Sulfamethazine, 13C6-Sulfamethoxazole, D5-Warfarin, d6-Metformin
Puyinf-4180	L11626-4	D6-Thiabendazole, d6-Metformin
Puyeff-4181	L11626-5	d6-Metformin
HAY-INF 4187	L11626-6	D5-Warfarin
HAY-SEFF 4188	L11626-7	13C2-Erythromycin-H2O,13C6-Sulfamethazine, 13C6-Sulfamethoxazole
LOTT-INF 4191	L11626-9	D3-Cotinine, D5-Warfarin, d6-Metformin
LOTT-INFD 4192	L11626-10	13C3-Trimethoprim, D5-Warfarin, d6-Metformin
LOTT-SEFF 4193	L11626-11 (A)	13C2-15N-Acetaminophen,D3-Cotinine, d6-Metformin
LOTT-SEFFD 4194	L11626-12	D3-Cotinine, D6-Thiabendazole,13C3-Trimethoprim,13C6-Sulfamethoxazole, D5-Warfarin, d6-Metformin
LOTT-TEFF 4197	L11626-13	13C2-15N-Acetaminophen, D3-Cotinine, 13C3-Trimethoprim,13C6-Sulfamethoxazole, 13C-D3-Naproxen, D5-Warfarin, D6-Gemfibrozil, d6-Metformin
LOTT-TEFFD 4198	L11626-14	D3-Cotinine, 13C6-Sulfamethazine, 13C3-Trimethoprim,13C6-Sulfamethoxazole, D6-Gemfibrozil, 13C-D3-Naproxen, D5-Warfarin, d6-Metformin
LOTT-SEFF 4193 (MS)	WG26425-103 (MS)	13C2-15N-Acetaminophen, d6-Metformin

LOTT-SEFF 4193 (MSD)	WG26425-104 (MSD)	13C2-15N-Acetaminophen, D3-Cotinine, d6-Metformin
Lab Blank	WG26425-101	D3-Cotinine, 13C6-Triclocarban

Percent recovery of the labeled surrogate d6-Metformin is slightly below the method lower control limit for the QC samples (AXYS ID WG26426-101 and -102, respectively) and the client samples, and the surrogate has been flagged with a 'V' on report forms. However, the percent recovery for the associated target, Metformin, falls within the acceptance range for the OPR, indicating that the data would not be affected by this variance.

ANALYTICAL DISCUSSION

List 1 Compounds

To improve surrogate recoveries, all QC samples and the client samples were diluted and instrumentally re-analyzed. Dilution data are reported (indicated by suffix 'N' on the AXYS ID). The samples listed in the following table required further dilution to bring the area counts of some targets to within the calibrated linear ranges. The affected compounds are reported from the second dilution data (indicated by the suffix 'N2' on report forms. Dilution factors are reported on the report forms.

CLIENT ID	AXYS ID
LOTT-SEFF 4193	L11626-11 (A)
LOTT-SEFFD 4194	L11626-12
Puyinf-4180	L11626-4
LOTT-SEFF 4193 (MS)	WG26425-103 (MS)

List 2 Compounds

To confirm results, extracts for all client samples and QC samples were instrumentally re-analyzed. The re-analysis data are reported (indicated by the suffix 'i' on the sample AXYS IDs). Extracts for the Lab Blank and the OPR (AXYS ID WG26425-101 and –102, respectively) were routinely re-analyzed on instrument for an additional time for confirmative purposes; the secondary re-analysis data are reported for these QC samples (indicated by the suffix 'I2' on the AXYS IDs). Extracts for the MS/MSD (AXYS ID WG26425-103 and -104, respectively) were re-analyzed on instrument for multiple times, and the third re-analysis data are reported (indicated by the suffix 'I3' on the AXYS IDs).

List 3 Compounds

To bring area responses of some target analytes to within the calibrated linear range of the instrument, extracts for samples listed in the following table were diluted and instrumentally re-analyzed. The results for the affected analytes are reported from the dilution data (indicated by suffix 'N' on the AXYS ID) for all the samples except for the sample L11626-3, which required a further dilution followed by instrumental re-analysis, and the affected compounds were reported from second dilution data (indicated by suffix ' on the AXYS ID). Dilution factors are noted on the report form.

CLIENT ID	AXYS ID
CC-Inf 4183	L11626-2
CC-Eff 4184	L11626-3
Puyinf-4180	L11626-4
HAY-INF 4187	L11626-6
LOTT-INF 4191	L11626-9
LOTT-INFD 4192	L11626-10

Samples LOTT-TEFF 4197 and LOTT-TEFF 4198 (AXYS ID L11626-13 and -14, respectively) were diluted and instrumentally re-analyzed due to low surrogate recoveries. Dilution did not improve the surrogate recoveries, the original data are reported.

Extracts for the Lab Blank and the OPR (AXYS ID WG26425-101 and –102, respectively) were routinely re-analyzed on instrument for an additional time for confirmative purposes; the original data are reported for the Lab Blank and the OPR.

List 4 Compounds

To improve surrogate recoveries, all the client samples and QC samples were diluted and instrumentally re-analyzed. Dilution data are reported (indicated by suffix 'N' on the AXYS ID). Dilution factors are reported on the report forms.

Metformin and its surrogate d6-Metformin were flagged with 'NQ' on report forms for the samples HAY-SEFF 4188 and HAY-SEFF 4189 (AXYS ID L11626-7 and -8, respectively) as the labeled surrogate was not quantifiable. Data are not available for the affected compounds.

DATA PACKAGE

This data package has been assigned a unique identifier, DPWG27439, shown on the cover page. Included in this data package following the narrative is the following documentation:

- Method summary
- Sample 'Cover Page' and 'Correlation Table'
- Sample Receiving Documentation
- Laboratory extraction worksheets
- Sample data reports (in order of AXYS Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of AXYS Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Henry Huang, PhD, QA Chemist

21 Dec. 2008
Date Signed

HORMONE AND STEROL ANALYSIS

SLUDGE SAMPLES

AXYS METHOD: MLA-068

PROJECT NUMBER: PF324169

PROJECT NAME: DST27

Contract: 4499
Data Package Identification: DPWG27495
Analysis WG26105

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

29 December 2008

WASHINGTON STATE DEPT OF ECOLOGY
SLUDGE

STEROLS ANALYSIS
METHOD: MLA-068
4499: L11618-1 to -4

30 December 2008

NARRATIVE

This narrative describes the analysis of four sludge samples for the determination of sterols and hormones using high-resolution gas chromatography / high-resolution mass spectrometry (HR-GC/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions on receipt are provided on the Sample Receiving Record forms. The samples were stored at -20°C prior to extraction and analysis.

The sample receiving chemist noted discrepancies between the sample labels and the Chain of Custody on the sample receiving log. The Chain of Custody Ids were used as the sample identifiers.

SAMPLE PREPARATION AND ANALYSIS

The samples were homogenized as documented on the Sample Preparation Records.

Samples and QC samples (a procedural blank, and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR)) were extracted and analyzed in one analysis batch, STWG26105. The composition of the batch is shown on the Cover Page and Correlation Table.

Extraction and analysis procedures were in general accordance with Axys Method MLA-068, Analytical Method for the Determination of Sterols and Hormones with BSTFA Derivatization by GC/MS and GC/HRMS. A summary of the method is supplied.

The samples were extracted according to the routine procedure used for samples anticipated to have high levels of sterols:

An accurately weighed subsample, equivalent to approximately 0.25 gram dry, was spiked with the routine suite of labeled quantification standards, plus an additional aliquot of deuterated cholesterol standard. The subsample was extracted by Soxhlet using 60:40 acetone/hexane, and the raw extract split into a (1/5th) portion for hormone analysis, and a (1/100th) portion for sterol analysis. Both portions were derivatized, and then cleaned up using the chromatography columns listed in the extraction workup sheets. The final extract was reduced in volume and spiked with labeled internal standards (referred to as the "recovery standard" in the method summary) before being submitted for instrumental analysis

CALCULATION

Target analyte concentrations were determined by isotope dilution quantification procedures using Micromass OPUSQuan software. Formulae used in the conversion of the raw chromatograms to concentration are provided in the method summary document.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. SDLs were determined from the analysis data by converting three times the height of the average noise signal to a response, using the area/height ratio of the labeled standard, and then to a concentration following the same procedures used to convert target peak responses to concentrations

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4499. Samples were logged under unique laboratory identifier L11618-1 to -4. All data reports reference both the Axys ID and the client sample identifier.

Suffixes are added to the Axys IDs such that each GC-MS acquisition is uniquely identified. The suffixes appearing in this data package are:

N = dilution of the extract in a new microvial
W = dilution of the extract in the same microvial
i = instrumental reanalysis of the extract

The laboratory qualifiers used are as follows:

D = dilution data
K = a GC peak was detected that did not meet the criteria for identification as the target analyte; the reported value represents the estimated maximum possible concentration of analyte present.
N = the recovery of the target analyte in the OPR fell outside the method control limits
U = identifies a compound that was not detected
V = the recovery of the labeled compound fell outside the method control limits
J = indicates an estimated value where the concentration of the analyte is less than the LMCL but greater than the SDL

Final analysis results are reported in concentration units of nanograms per gram (ng/g) on a dry weight basis. Concentration and detection limits are provided to three significant figures. Data are rounded up in the event the value ends in a 5.

ANALYTICAL DISCUSSION

A method detection limit (MDL) study is not available for the solid analysis of sterols and hormones using high-resolution gas chromatography / high-resolution mass spectrometry (HR-GC/MS).

Some area responses for sterols exceeded the concentration range of the instrumental calibration and AXYS diluted and re-injected selected sample extracts to bring responses within the calibrated range. Replication between the original the dilution re-injection results were within the normal precision range of the method, demonstrating the validity of the original analysis results, and the results from the original injections were reported as final.

The hormone portions for samples listed in the following table were diluted and reanalyzed to confirm the labeled compound recoveries. The diluted extract required instrumental reanalysis as not all instrumental method acceptance criteria were met during the initial analysis. The hormone data are reported from this reanalyzed dilution analysis (indicated by the suffix 'Ni' added to the AXYS ID).

CLIENT ID	AXYS ID
PUY biosolids 4182	L11618-1
CC-Biosolid 4185	L11618-2
LOTT-Biosolid 4199	L11618-3

The hormone portion for sample LOTT-Biosolid D 4200 (AXYS ID L11618-4) was diluted and reanalyzed to confirm the labeled compound recoveries. A further dilution was conducted to confirm data results. As the data was confirmed, all data are reported from the first dilution analysis (indicated by the suffix 'N' added to the AXYS ID).

The sterol portion of the Lab Blank was instrumentally reanalyzed to confirm results. The OPR extract was instrumentally reanalyzed because not all method acceptance criteria were met during the initial analysis. It was further reanalyzed to confirm results. Data are reported from the reanalyses (indicated by the suffix 'i' and 'i2' added to the AXYS ID).

QA/QC NOTES AND DISCUSSION

QC samples were analyzed in one analysis batch, carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to its batch QC samples.

- Sample analyte concentrations are not blank corrected and should be compared to the blank levels for significance.
- Analysis results are recovery corrected for possible losses through the extraction and clean up steps of the analytical procedure.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

The recoveries of Androsterone (151%), Desogestrel (142%), 17 alpha-Dihydroequilin (11%) and Testosterone (138%) for the Ongoing Precision and Recovery (OPR) sample (AXYS ID WG26105-102) were outside the currently documented method MLA068 acceptance criteria but the results were judged by AXYS to demonstrate acceptable method performance. MLA068 is a recently developed method, the acceptance criteria are based on limited performance data and as such are used by AXYS as general guidelines for evaluation of acceptability.

The concentration of Progesterone in the Lab Blank (AXYS ID WG26105-101) was above the MLA068 reporting limit for biosolids (373 n/g vs reporting limit of 75 ng/g) and similar to concentrations in samples. Concentrations of other target compounds detected in the Lab Blank were below method reporting limits. The sample results have not been blank corrected and should be evaluated for significance against the Lab Blank results.

The recoveries of D6-Norethindrone and D7-Cholesterol in sample LOTT-Biosolid D 4200 (AXYS ID L11618-4), D4-17 alpha-Ethinyl-Estradiol in the Lab Blank and D7-Cholesterol in samples CC-Biosolid 4185 and LOTT-Biosolid 4199 (AXYS Ids L11618-2 and –3) are outside method acceptance criteria and these compounds were flagged with a 'V' on reports. The results are recovery corrected by the analytical method and these variances were deemed to have no significant impact on data accuracy. The percent surrogate recoveries are reported as general method performance indicators only.

The OPR results for Ergosterol, Stigmasterol and beta-Sitosterol were flagged with 'K' since the confirming ion relative abundances were slightly outside the method prescribed range. This is attributable to the fact that the OPR sterols spiking level for this batch was close to the method detection limit. Sample data accuracy or reliability are not affected by this OPR variance. All sterols OPR recoveries were within method specified ranges.

Beta-sitosterol in CS1 for linearity file ST83_274A is flagged with 'K' since the confirming ion relative abundances was slightly outside the method prescribed range. Sample data are not significantly affected by this variance.

DATA PACKAGE

This data package is assigned a unique data package identification workgroup, DPWG27495. This ID is shown on the front page of the data package.

Included in the paper data package following the narrative is the following documentation:

- Method summary
- Sample Cover Page and Correlation Table
- Sample Receiving Documentation
- Sample homogenization and pretreatment records
- Laboratory extraction logs for each sample
- Sample data reports (in order of Axys Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of Axys Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: Teresa Rawsthorne, B.Sc., QC Chemist

30DEC08
Date Signed

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HORMONE AND STEROL ANALYSIS

EFFLUENT and INFLUENT SAMPLES

AXYS METHOD: MLA-068

PROJECT NAME: LOTT ALLIANCE

Contract: 4499
Data Package Identification: DPWG27499
Analysis WG26360, WG27291 and WG27292

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

29 December 2008

WASHINGTON STATE DEPT OF ECOLOGY
AQUEOUS SAMPLES

STEROLS ANALYSIS
METHOD: MLA-068
4499: L11626-15 & -16

Project Name: LOTT ALLIANCE

30 December 2008

NARRATIVE

This narrative describes the analysis of two aqueous samples for the determination of sterols and hormones using high-resolution gas chromatography / high-resolution mass spectrometry (HR-GC/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions on receipt are provided on the Sample Receiving Record forms. The samples were stored at -20°C prior to extraction and analysis.

The sample receiving chemist noted discrepancies between the sample labels and the Chain of Custody on the sample receiving log. The Chain of Custody IDs were used as the sample identifiers.

SAMPLE PREPARATION AND ANALYSIS

The two samples were analyzed among three batches -- WG26360, WG27291, and WG27292 -- the compositions of which are shown on the Cover Page and Correlation Table, and on the Batch Lists accompanying the extraction workup sheets. Each batch contained a procedural blank, and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR).

Extraction and analysis procedures were in general accordance with Axys Method MLA-068, Analytical Method for the Determination of Sterols and Hormones with BSTFA Derivatization by GC/MS and GC/HRMS. A summary of the method is supplied.

The influent sample was spiked and extracted in batch WG26291 according to the routine procedure used for samples anticipated to have high levels of sterols:

> An accurately-weighed subsample of approximately 500mL was spiked with the routine suite of labeled quantification standards, plus an additional aliquot of deuterated cholesterol standard, and then liquid-liquid extracted using dichloromethane. The raw extract split into a (1/5)th portion for hormone analysis, and a (1/100)th portion for sterol analysis. Both portions were derivatized, and then cleaned up using the chromatography columns listed in the extraction workup sheets. The final extract was reduced in volume and spiked with labeled internal standards (referred to as the "recovery standard" in the method summary) before being submitted for instrumental analysis.

The effluent sample was analyzed in batch WG26360 for sterols and batch WG27292 for hormones, according to the routine procedure for sample anticipated to have low levels of sterols:

> An accurately-weighed subsample of approximately 1L was spiked with the routine suite of labeled quantification standards, and then liquid-liquid extracted using dichloromethane. The raw was derivatized, and then cleaned up using the chromatography columns listed in the extraction workup sheets, and finally prepared for instrumental analysis as describe above.

CALCULATIONS

Target analyte concentrations were determined by either isotope dilution or internal standard quantification procedures, using Micromass OPUSQuan software. Formulae used in the conversion of the raw chromatograms to concentration are provided in the method summary document.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. SDLs were determined from the analysis data by converting three times the height of the average noise signal to a response, using the area/height ratio of the labeled standard, and then to a concentration following the same procedures used to convert target peak responses to concentrations.

LMCLs are calculated from the concentration in the lowest calibration standard, prorated to the sample size and final extract volume, and accounting for any splitting of the extract. Concentrations above the SDL but below the LMCL are flagged "J".

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4499. Samples were logged under unique laboratory identifiers L11626-15 & -16. All data reports reference both the Axys ID and the client sample identifier.

Suffixes are added to the Axys IDs such that each GC-MS acquisition is uniquely identified. The suffixes appearing in this data package are:

R = the analysis was repeated starting from a fresh subsample

The laboratory qualifiers used are as follows:

B = the flagged analyte is detected in the associated blank, and the concentration in the sample is less than ten times the blank concentration

J = indicates an estimated value where the concentration of the analyte is less than the LMCL but greater than the SDL

K = a GC peak was detected that did not meet the criteria for identification as the target analyte; the reported value represents the estimated maximum possible concentration of analyte present.

U = identifies a compound that was not detected

V = the recovery of the labeled compound fell outside the method control limits

Final results are reported to three significant figures, in units of nanograms per Liter (ng/L). (The rounding procedure was to round up for digits 5 and above, down for digits less than 5).

ANALYTICAL DISCUSSION

A method detection limit (MDL) study is not available for sterols and hormones in effluents by HR-GC/MS.

Sample MWRWP-Effluent was first extracted and analyzed in batch WG26360. Because of QC issues affecting the hormone analytes, the analysis was repeated in batch WG27292. The analysis in batch WG27292 had some QC issues affecting the sterol analytes. Therefore data were combined from both batches, reporting hormones from the repeat analysis WG28292, and sterols from the original batch WG26360.

QA/QC NOTES AND DISCUSSION

QC samples (a procedural blank and an OPR in each batch) were prepared alongside the client samples and carried through the entire analytical procedures. The sample data were evaluated in relation to its corresponding batch QC samples.

- Sample analyte concentrations are not blank-corrected. Data should be compared to the corresponding blank. Analyte concentrations that are less than ten times the concentration in the corresponding blank are flagged "B".
- Analysis results are recovery-corrected for possible losses through the extraction and clean up steps of the analytical procedure.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

WG26360, MWRWP-Effluent, sterol analysis

All QC criteria were met

WG27292, MWRWP-Effluent, hormone analysis

1. The recoveries of deuterated norethindrone and norgestrel in OPR WG27292-102, and the recovery d4-17α-ethinylestradiol in blank WG27292-101, fell below the lower method control limit. The recovery of d6-norethindrone in MWRWP-Effluent exceeded the upper method control limit. The affected surrogates are flagged "V".
2. The recoveries of native androsterone, desogestrel, 17β-estradiol, and progesterone in the OPR exceeded the upper method control limit and are flagged "N" accordingly. These analytes may be over-reported in MWRWP-Effluent to a similar degree.

WG27291, MWPS-MWRWP Influent

1. The recoveries of all six deuterated hormone surrogates exceeded the upper method control limit. The cause of this variance, and its effect on the hormone data, are uncertain.
2. In the OPR WG27291-102, the ion abundance ration of cholestanol fell outside the method control limits, and the analyte is flagged "K" on the report Form 8A accordingly. Only a hundredth portion of the OPR extract is analyzed sterols, matching the extract split performed on the sample, which is split in anticipation of high levels. The OPR extract is therefore very dilute, and at low concentrations the ion abundance ratios are less precise. This variance has no impact on the data.

DATA PACKAGE

This data package is assigned a unique data package identification workgroup, DPWG27499, shown on the front page. The following documents are included:

- Method summary
- Sample Cover Page and Correlation Table
- Sample Receiving Documentation
- Sample homogenization and pretreatment records
- Laboratory extraction logs for each sample
- Sample data reports (in order of Axys Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of Axys Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Brian Watson
Signed: Brian Watson, B.Sc., QC Chemist

30 December 2008
Date Signed

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HORMONE AND STEROL ANALYSIS

INFLUENT and EFFLUENT SAMPLES

AXYS METHOD: MLA-068

PROJECT NUMBER: PF324169

PROJECT NAME: DST27

Contract: 4499
Data Package Identification: DPWG27500
Analysis WG26360, WG26896, WG27291 and WG27292

Prepared for:
Washington State Dept of Ecology

Prepared by:
AXYS Analytical Services Ltd.
2045 Mills Rd
Sidney, British Columbia V8L 5X2
CANADA

Contact: Cynthia Tomey
Project Manager

29 December 2008

WASHINGTON STATE DEPT OF ECOLOGY
AQUEOUS SAMPLES

STEROLS ANALYSIS
METHOD: MLA-068
4499: L11626-1 to -14

Project Name: DST27

31 December 2008

NARRATIVE

This narrative describes the analysis of fourteen aqueous samples for the determination of sterols and hormones using high-resolution gas chromatography / high-resolution mass spectrometry (HR-GC/MS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 21st of August 2008. Details of sample conditions on receipt are provided on the Sample Receiving Record forms. The samples were stored at -20°C prior to extraction and analysis.

The sample receiving chemist noted discrepancies between the sample labels and the Chain of Custody on the sample receiving log. The Chain of Custody IDs were used as the sample identifiers.

SAMPLE PREPARATION AND ANALYSIS

The fourteen samples were analyzed among four batches, the five influent samples in batch WG27291, and the eight effluent samples in batches WG26360, WG26896, and WG27292. The compositions of these batches are shown on the Cover Page and Correlation Table, and on the Batch Lists accompanying the extraction workup sheets. Each batch contained a procedural blank and a lab-generated reference sample known as the Ongoing Precision and Recovery (OPR), and batch WG26896, in addition, contained a matrix spike (MS) and matrix spike duplicate (MSD). The procedural blanks and OPRs were prepared using ultra pure Seastar water as the matrix, and the MS/MSD was prepared using sample LOTT-SEFF 4193.

Extraction and analysis procedures were in general accordance with Axys Method MLA-068, Analytical Method for the Determination of Sterols and Hormones with BSTFA Derivatization by GC/MS and GC/HRMS. A summary of the method is supplied.

The influent samples were spiked and extracted in batch WG27291 according to the routine procedure used for samples anticipated to have high levels of sterols:

> An accurately-weighed subsample of approximately 500mL was spiked with the routine suite of labeled quantification standards, plus an additional aliquot of deuterated cholesterol standard, and then liquid-liquid extracted using dichloromethane. The raw extract split into a $(1/5)^{th}$ portion for hormone analysis, and a $(1/100)^{th}$ portion for sterol analysis. Both portions were derivatized, and then cleaned up using the chromatography columns listed in the extraction workup sheets. The final extract was reduced in volume and spiked with labeled internal standards (referred to as the "recovery standard" in the method summary) before being submitted for instrumental analysis.

The effluent samples were analyzed in batches WG26360, WG26896, and WG27292, according to the routine procedure for sample anticipated to have low levels of sterols:

> An accurately-weighed subsample of approximately 1L was spiked with the routine suite of labeled quantification standards, and then liquid-liquid extracted using dichloromethane. The raw was derivatized, and then cleaned up using the chromatography columns listed in the extraction workup sheets, and finally prepared for instrumental analysis as describe above.

CALCULATIONS

Target analyte concentrations were determined by either isotope dilution or internal standard quantification procedures, using Micromass OPUSQuan software. Formulae used in the conversion of the raw chromatograms to concentration are provided in the method summary document.

Sample specific detection limits (SDLs) were calculated for each target analyte and used as the detection qualifier. SDLs were determined from the analysis data by converting three times the height of the average noise signal to a response, using the area/height ratio of the labeled standard, and then to a concentration following the same procedures used to convert target peak responses to concentrations.

LMCLs are calculated from the concentration in the lowest calibration standard, prorated to the sample size and final extract volume, and accounting for any splitting of the extract. Concentrations above the SDL but below the LMCL are flagged "J".

REPORTING CONVENTIONS

For internal tracking, Axys assigned the Washington State Dept of Ecology a contract number 4499. Samples were logged under unique laboratory identifiers L11626-1 to -14. All data reports reference both the Axys ID and the client sample identifier.

Suffixes are added to the Axys IDs such that each GC-MS acquisition is uniquely identified. The suffixes appearing in this data package are:

- i = the extract was re-acquired on the GC-MS
- N = the extract was diluted, with transfer to a fresh gc-vial
- R = the analysis was repeated starting from a fresh subsample

The laboratory qualifiers used are as follows:

- B = the flagged analyte is detected in the associated blank, and the concentration in the sample is less than ten times the blank concentration
- D = dilution data
- E = the extract concentration of the flagged analyte exceeds the calibrated range of the GC-MS.
- J = indicates an estimated value where the concentration of the analyte is less than the LMCL but greater than the SDL
- K = a GC peak was detected that did not meet the criteria for identification as the target analyte; the reported value represents the estimated maximum possible concentration of analyte present.
- N = the recovery of the flagged native analyte in the OPR fell outside the method control limit
- U = identifies a compound that was not detected
- V = the recovery of the labeled compound fell outside the method control limits
- X = the flagged analyted is reported on another report form

Final results are reported to three significant figures, in units of nanograms per Liter (ng/L). (The rounding procedure was to round up for digits 5 and above, down for digits less than 5).

ANALYTICAL DISCUSSION

A method detection limit (MDL) study is not available for sterols and hormones in effluents by HR-GC/MS.

WG26360 and WG27292

Samples CC-Eff 4184, LOTT-TEFF 4197, and LOTT-TEFFD 4198 were first extracted and analyzed in batch WG26360. Because of QC issues affecting the hormone analytes, the analysis was repeated in batch WG27292. The analysis in batch WG27292 had some QC issues affecting the sterol analytes. Therefore data were combined from both batches, reporting hormones from the repeat analysis WG27292, and sterols from the original batch WG26360. The repeat hormone analyses in WG27292 are indicated by the suffix "R" added to the Axys IDs.

The sterol portion of extract for sample CC-Eff 4184, in batch WG26360, required a dilution for coprostanol and cholesterol. The dilution is indicated by the suffix "N". The diluted extract then required a further acquisition on the GC-MS, indicated by the suffix "Ni". Coprostanol and cholesterol are reported from the acquisition suffix "Ni", while the remaining analytes are reported from the initial acquisition.

WG26896

Some sample required an additional acquisition on the GC-MS before all instrumental QC criteria were met. These are indicated by the suffix "i" added to the Axys IDs.

QA/QC NOTES AND DISCUSSION

QC samples (a procedural blank and an OPR in each batch, and an MS/MSD in batch WG26896) were prepared alongside the client samples and carried through the entire analytical procedures. The sample data were evaluated in relation to its corresponding batch QC samples.

- Sample analyte concentrations are not blank-corrected. Data should be compared to the corresponding blank. Analyte concentrations that are less than ten times the concentration in the corresponding blank are flagged "B".
- Analysis results are recovery-corrected for possible losses through the extraction and clean up steps of the analytical procedure.
- All linearity, calibration verification, OPR and labeled compound recovery specifications were met with the following exceptions:

WG27292

1. The recoveries of deuterated norethindrone & norgestrel in OPR WG27292-102, d6-norethidrone in sample LOTT-TEFFD 4198, and d4-17α-ethinylestradiol in blank WG27292-101 fell below the lower method control limit, and are flagged "V" accordingly.
2. The recoveries of native androsterone, desogestrel, 17β-estradiol, and progesterone in the OPR exceeded the upper method control limit and are flagged "N" accordingly. These analytes may be over-reported in the samples to a similar degree.
3. In sample CC-Eff 4184, the recoveries of all the deuterated surrogates except 17-β-estradiol exceeded the upper method control limit, and are flagged "V" accordingly. The cause of the high surrogate recoveries, and their impact on the data, are uncertain.

WG26896

4. Slightly elevated background concentrations of some target analytes were observed in the Lab Blank (AXYS ID WG26896-101); and the concentrations of the samples are at levels similar to the blank. As noted above, sample analyte concentrations are not blank corrected and blank levels should be considered during sample data review.

5. Concentrations of some target analytes were above the method upper control limits for the OPR (AXYS ID WG26896-102); these compounds are flagged with an 'N' on the report forms. The concentration detected in samples may be similarly affected. In cases where the analyte was not detected, the data are not considered affected; In cases where the analyte was detected, the concentration should be considered as maximum value.

6. The recovery of some labeled surrogates for the samples in the following table did not meet the method criteria; these compounds are flagged with a 'V'. As the isotope dilution method of quantification produces data that are recovery corrected, the slight variances from the method acceptance criteria are deemed not to affect the quantification of these analytes. Percent surrogate recoveries are used as general method performance indicator only.

CLIENT ID	AXYS ID
LOTT-SEFF 4193	L11626-11
LOTT-SEFF 4193 (MSD)	WG26896-106
HAY-SEFF 4188	L11626-7
LOTT-SEFFD 4194	L11626-12
SPIKED MATRIX	WG26896-102

The native spiking concentration for sterols between the Matrix Spike /Matrix Spike Duplicate (MS/MSD) pair was very small relative the sample concentration. Consequently, the MS /MSD test cannot be used for quantification of spike recovery

WG26360, MWRWP-Effluent, sterol analysis

All QC criteria were met

WG27291

The surrogate recoveries in the hormone analysis were all elevated, and flagged "V" accordingly. The impact on the data is not certain.

DATA PACKAGE

This data package is assigned a unique data package identification workgroup, DPWG27500, shown on the front page. The following documents are included:

- Method summary
- Sample Cover Page and Correlation Table
- Sample Receiving Documentation
- Sample homogenization and pretreatment records
- Laboratory extraction logs for each sample
- Sample data reports (in order of Axys Sample ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Sample raw data (in order of Axys Sample ID)
- Laboratory QC raw data
- Instrumental QC raw data (organized by analysis date)

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Brian Watson — Signed: Brian Watson, B.Sc., QC Chemist

31 December 2008 — Date Signed

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Data Qualifier Codes for MEL Case Narratives

U	The analyte was not detected at or above the reported result.
J	The analyte was positively identified. The associated numerical result is an estimate.
UJ	The analyte was not detected at or above the reported estimated result.
REJ	The data are unusable for all purposes.
NAF	Not analyzed for.
N	For organic analytes there is evidence the analyte is present in this sample.
NJ	There is evidence that the analyte is present. The associated numerical result is an estimate.
NC	Not Calculated
E	The concentration exceeds the known calibration range.
bold	The analyte was present in the sample. (visual aid to locate detected compounds on report sheet.)

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Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative
September 5, 2008

Subject: General Chemistry PPCPs in WWTPs - 34

Project No: 161108

Officer: Brandi Lubliner

By: Dean Momohara

Summary

The results for orthophosphate in samples 344187 and 344198 were greater the associated total phosphorous. The laboratory will analyze sample 344182 for percent solids when the organic analysis is performed. The laboratory did not encounter any other problems in the analyses of these samples. All sample results were reported without qualification.

All analyses requested were evaluated by established regulatory quality assurance guidelines.

Methods

The laboratory analyzed the samples by the following methods: Standard Methods (SM) 4500PG for orthophosphate, SM4500PF total phosphorous (TP), SM4500NB for total persulfate nitrogen (TPN), SM4500NO3I for nitrate-nitrite, SM4500NH3H for ammonia (NH_3), SM2540E for total suspended solids, and SM2540G for percent solids.

Sample Information

The laboratory received the samples on 08/20/08. The temperature(s) of the coolers received were within the proper range of 0°C - 6°C. All samples were received in good condition and where applicable, properly preserved. Eighteen samples were received and assigned laboratory identification numbers 344180 – 344189, 344191 – 344194, and 344197 – 344200.

Holding Times

The laboratory performed all analyses within established EPA holding times.

Calibration

Instrument calibrations and calibration checks were performed in accordance with the appropriate method. All initial and continuing calibration checks were within control limits. Calibration correlation coefficients for TPN, OP, nitrate-nitrite, NH_3, and TP were within the

acceptance range of 1.000 - 0.995. The instruments were calibrated with NIST traceable standards and verified to be in calibration with a second source NIST traceable standard. Oven and incubator temperatures were recorded before and after each analysis batch and were within acceptable limits.

Method Blanks

No analytically significant levels of analyte were detected in the method blanks associated with these samples.

Matrix Spikes

All associated matrix spike recoveries were within the acceptance limits of 75% - 125% for all other analyses.

Replicates

All associated duplicate relative percent differences of samples with concentrations greater than 5 times the reporting limit were within the acceptance range of 0% - 20%.

Laboratory Control Samples

All laboratory control sample recoveries were within the acceptance limits of 80% - 120%.

Other Quality Assurance Measures and Issues

U The analyte was not detected at or above the reported result.

bold The analyte was present in the sample.
(visual aid to locate detected compounds on report sheet.)

Please call Dean Momohara at (360) 871-8808 to further discuss this project.

cc: Project File

Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative - Addendum
October 1, 2008

Subject: General Chemistry PPCPs in WWTPs - 34

Project No: 161108

Officer: Brandi Lubliner

By: Dean Momohara

Summary

The results for orthophosphate (OP) in samples 344187 and 344198 were greater the associated total phosphorous. Sample 344187 was reanalyzed for total phosphorous (TP) at a dilution similar to the OP analysis. The TP reanalysis compared favorably with the OP result and was reported. The laboratory will analyze sample 344182 for percent solids when the organic analysis is performed. The laboratory did not encounter any other problems in the analyses of these samples. All sample results were reported without qualification.

All analyses requested were evaluated by established regulatory quality assurance guidelines.

Methods

The laboratory analyzed the samples by the following methods: Standard Methods (SM) 4500PG for orthophosphate, SM4500PF total phosphorous (TP), SM4500NB for total persulfate nitrogen (TPN), SM4500NO3I for nitrate-nitrite, SM4500NH3H for ammonia (NH_3), SM2540E for total suspended solids, and SM2540G for percent solids.

Sample Information

The laboratory received the samples on 08/20/08. The temperature(s) of the coolers received were within the proper range of 0°C - 6°C. All samples were received in good condition and where applicable, properly preserved. Eighteen samples were received and assigned laboratory identification numbers 344180 – 344189, 344191 – 344194, and 344197 – 344200.

Holding Times

The laboratory performed all analyses within established EPA holding times.

Calibration

Instrument calibrations and calibration checks were performed in accordance with the appropriate method. All initial and continuing calibration checks were within control limits. Calibration correlation coefficients for TPN, OP, nitrate-nitrite, NH_3, and TP were within the acceptance range of 1.000 - 0.995. The instruments were calibrated with NIST traceable standards and verified to be in calibration with a second source NIST traceable standard. Oven and incubator temperatures were recorded before and after each analysis batch and were within acceptable limits.

Method Blanks

No analytically significant levels of analyte were detected in the method blanks associated with these samples.

Matrix Spikes

All associated matrix spike recoveries were within the acceptance limits of 75% - 125% for all other analyses.

Replicates

All associated duplicate relative percent differences of samples with concentrations greater than 5 times the reporting limit were within the acceptance range of 0% - 20%.

Laboratory Control Samples

All laboratory control sample recoveries were within the acceptance limits of 80% - 120%.

Other Quality Assurance Measures and Issues

U The analyte was not detected at or above the reported result.

bold The analyte was present in the sample.
(visual aid to locate detected compounds on report sheet.)

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cc: Project File

Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative
November 3, 2008

Subject: PPCPs in WWTPs - 34

Sample(s): 08 – 344180 to -344189, -344191 to -344194, and -344197 to -344202.

Project: 1611-08

Officer(s): Brandi Lubliner

By: Dickey Huntamer

Semivolatiles
PAHNOAA

Analytical Method(s)

The semivolatile biosolid samples were Soxtherm extracted with methylene chloride and the water samples were extracted with methylene chloride following the Manchester modification of the EPA SW 846 8270 with capillary GC/MS analysis of the sample extracts. The samples were analyzed as is with no cleanup. The water samples were taken to 1.0 mL final volume and the biosolids were taken to 10.0 mL final volume.

Holding Times

All samples were prepared and analyzed within the method holding times.

Instrument Tuning

Calibration against DFTPP is acceptable for the initial calibration, continuing calibration and all associated sample analyses.

Calibration

Instrument calibrations and calibration checks were performed in accordance with the appropriate method. The September 24th initial calibration correlation coefficients were within the acceptance range of 1.000 - 0.995 for linear curve, 0.99 for quadratic fit or an average response of $\leq$15%.

N-nitrosodiphenylamine, and carbazole fell outside these criteria and all results were qualified, J. Three compounds, 4-chloroaniline, 4-nitroaniline, and 4, 6-dinitro-2-methylphenol had poor responses and all results were rejected, REJ.

The Initial Calibration Verification (ICV) was within the acceptable limits of ±30% for the September 24th analysis.

Back calculations for the initial calibration were all within acceptance limits except coprostanol and cholesterol which were high in the 0.25 through 1.0 ug/mL level standards. If they were not detected or are outside the range no qualifiers were added.

Several others were affected but were already qualified for other reasons.

QC Limits for the Continuing Calibration (CC) are ±20%.

All target analytes were within the maximum of 20% for the October 1st continuing calibration except 2, 2' oxybis(1-chloropropane), acenaphthylene, benzoic acid and carbazole which were low. All results for these compounds were qualified as estimates, J. Bisphenol A, pentachlorophenol, and nonylphenol were biased high. No qualification was added unless the compounds were detected.

All target analytes were within the maximum of 20% for the October 2nd continuing calibration except n-nitrosodimethylamine, pentachlorophenol, n-nitrosodiphenylamine, 2, 2' oxybis(1-chloropropane), acenaphthylene, and carbazole which were low. All results for these compounds were qualified as estimates. Nonylphenol, and 3-nitroaniline were biased high. No qualification was added unless the compound was detected

Blanks

In the water blank, OB08239H1only benzoic acid was detected.

In the biosolids blank, OB08266S1 no target compounds were detected.

Compounds that were found in the sample and blank were considered native to the sample if the area counts in the sample are greater than or equal to 5 times the area counts in the associated method blank.

Surrogates (Isotopes)

The surrogate recoveries were reasonable, acceptable, and within QC limits of 25%-121% for 2-fluorophenol, 24%-113% for d5-phenol, 20%-130% for d4-2-chlorophenol, and d4-1,2-dichlorobenzene, 23%-120% for d5-nitrobenzene, 18%-137% for d14-terphenyl, 50%-150% for d10-pyrene and 30%- 115% for 2-fluorobiphenyl.

Surrogates were missing in blank OB08239H1 and all results were qualified, J.

One surrogate 2-fluorophenol was low in blank OB08266S1 (13%), and LCS OL08266S1 (24%). Since the other surrogates were acceptable no qualifiers were added.

One surrogate d5-phenol was high in samples: -344186 to -344189, -344191, -344192, -344197, -434198, and -344201. In sample -344199 and -344200 2-fluorobiphenyl was high. Since the other surrogates were acceptable no qualifiers were added.

All surrogates except d5-nitrobenzene (27%) were low in sample -344180. All results were qualified, J.

Matrix Spikes

Matrix spike recoveries for water sample -344193 were within the acceptance limits of 50% to 150% except for: n-nitrosodimethylamine (43%, 45%), hexachloroethane (47%, 37%), 2-nitrophenol (33%, 44%), and pentachlorophenol (0%, 38%).

Isophorone (52%, 48%), benzoic acid (32%, 55%), and carbazole (158%, 146%) had one acceptable recovery and no qualifiers were added.

Five compounds: 4-chloraniline, hexachlorocyclopentadiene, 2, 4 dinitrophenol, 4, 6-dinitro-2-methylphenol, and pentachlorophenol were not detected partly due to the low spiking level. All results for these compounds were rejected, REJ in the matrix spike source sample-344193.

The Relative Percent Differences (RPD) for those compounds detected was within the acceptable limit of 40% except for benzoic acid (52%).

The biosolids matrix spike using sample -344185, were within the acceptance limits of 50% to 150% except for n-nitrosodimethylamine (48%, 48%), n-nitrosodipropylamine (49%, 0%) and caffeine (19%, 15%). Results for these compounds were qualified, J in the matrix sample -344185. One recovery was acceptable for n-nitrosodiphenylamine (46%, 69%), 2-nitrophenol (134%, 163%), acenaphthylene (113%, 155%), and dibenzofuran (102%, 255%). No qualifiers were added for these compounds

Both recoveries were high for benzoic acid (232%, 207%), 2, 4, 6 trichlorophenol (186%, 222%), 2, 4, 5-trichlorophenol (196%, 207%), carbazole (176%, 197%), nonylphenol (195%, 226%) and di-n-octylphthalate (229%, 257%). These compounds were only qualified if detected.

A number of other compounds are marked not calculated, NC due to the high amounts of native present or interferences, the 1 to 10 dilution and the low spiking amount 10 ug added.

The RPD for those compounds detected was within the acceptable limit of 40% except for dimethylphthalate (48%). Results for this compound were qualified, J in the matrix spike source sample -344185.

Duplicates

A duplicate was analyzed using biosolid sample -344182. The RPD for those compounds detected was within the acceptable limit of 40% except for phenol (102%). Phenol was qualified, J in samples -344182 and -344182 LDP1.

Laboratory Control Samples

The laboratory control samples were spiked at a level of 10 ug, equivalent to 20 ug/Kg or 10 ug/L.

One laboratory control sample (LCS) was analyzed with the water samples, OL08239H1. All recoveries were within acceptable limits 50% to 150% except for hexachloroethane (32%) benzoic acid (8.3%), hexachlorocyclopentadiene (14%) which were low. All results for these compounds were qualified as estimates, J, in the associated samples.

Two compounds, 2, 4-dinitrophenol and 4, 6-dinitro-2-methylphenol were not recovered. Results for these compounds were rejected, REJ in the associated samples.

One laboratory control sample (LCS) was analyzed with the biosolid samples, OL08266H1. All recoveries were within acceptable limits 50% to 150% except for 2, 4, 6-trichlorophenol (47%), and 2, 4, 5-trichlorophenol (31%). All biosolid sample results for these compounds were qualified J.

Benzoic acid, pentachlorophenol, and bisphenol A were not detected due to the low spiking level used. All results for these compounds were qualified, J. Both 2, 4-dinitrophenol and 4-nitroaniline were not detected and all results for these compounds were rejected, REJ in the associated samples.

4 Chloroaniline was detected in OL08239H1 (129%) and OL08266S1 (178%) but all results were REJ due to calibration difficulties.

SIM Analysis

Some of the samples in which Bisphenol A, triethylcitrate, Triclosan and tri(2-chloroethyl) phosphate were not detected were analyzed using selected ion monitoring (SIM) to see if lower detection limits could be achieved. This was done on samples -344180, -181, -183, -186, -187, -191, -192, -199, and -200. Generally the SIM result was the same as the full scan result so the full scan result was reported. Unfortunately the interference's present in the samples affect both the scan and SIM analysis particularly in the biosolids samples.

The following table has the exceptions where it was not detected in scan mode but was in SIM. The SIM value is next to the scan result in parenthesis. These results were added to the final report sheets.

Sample	Bisphenol A	Triclosan	Triethyl citrate
-344183	1.3 ug/L J (0.63 U)	-	-
-344199	-	-	283 J ug/Kg (6040 U)
-344200	-	-	302 J ug/Kg (604 U)

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Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative
December 08, 2008

Subject: General Chemistry PPCP in WWTPs - 47

Project No: 184908

Officer: Brandi Lubliner

By: Dean Momohara

Summary

Sample 474026 for total phosphorous by Standard Method 4500PF was qualified as an estimate. The laboratory did not encounter any other problems in the analyses of these samples. All other sample results were reported without qualification.

All analyses requested were evaluated by established regulatory quality assurance guidelines.

Methods

The laboratory analyzed the samples by the following methods: Standard Methods (SM) 4500PG for orthophosphate, SM4500PF for total phosphorous (TP), and EPA 200.8M for TP.

Sample Information

The laboratory received the samples on 11/20/08. The temperature(s) of the coolers received were within the proper range of 0°C - 6°C. All samples were received in good condition and where applicable, properly preserved. Two samples were received and assigned laboratory identification numbers 474025 and 474026.

Holding Times

The laboratory performed all analyses within established EPA holding times.

Calibration

Instrument calibrations and calibration checks were performed in accordance with the appropriate method. All initial and continuing calibration checks were within control limits. The calibration correlation coefficients were within the acceptance range of 1.000 - 0.995. The instruments were calibrated with NIST traceable standards and verified to be in calibration with a second source NIST traceable standard.

Method Blanks

The method blank (MB) for sample 474026 for TP by SM4500PF was greater than the method detection limit. Since the concentration in the sample was less than 10 times the contamination in the MB, the result was qualified as an estimate. No other analytically significant levels of analyte were detected in the method blanks associated with these samples.

Matrix Spikes

All associated matrix spike recoveries were within the acceptance limits of 75% - 125%.

Replicates

All associated duplicate relative percent differences of samples with concentrations greater than 5 times the reporting limit were within the acceptance range of 0% - 20%.

Laboratory Control Samples

All laboratory control sample recoveries were within the acceptance limits of 85% - 115% for TP analysis by EPA 200.8M and 80% - 120% for all other analyses.

Internal Standards

All internal standard recoveries for TP analysis by EPA 200.8M were within acceptance limits of 60% - 125%.

Other Quality Assurance Measures and Issues

U The analyte was not detected at or above the reported result.

J The analyte was positively identified. The associated numerical result is an estimate.

bold The analyte was present in the sample.
(visual aid to locate detected compounds on report sheet.)

Please call Dean Momohara at (360) 871-8808 to further discuss this project.

cc: Project File

Appendix E. Analytical Results

The following acronyms are used in Tables E-1 – E-9:

- CC Chambers Creek Wastewater Treatment Plant.
- Puy Puyallup Wastewater Treatment Plant.
- BITP Budd Inlet Treatment Plant.
- BIRWP Budd Inlet Reclaimed Water Plant.
- MWRWP Martin Way Reclaimed Water Plant.
- Hayden Hayden Wastewater Treatment Plant.
- Hayden WRF Hayden Wastewater Research Facility.

Table E-1. Method 1694 PPCP concentrations in wastewater influents, ng/L, pptr.

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (Replicate)		MWRWP-Influent		Hayden-Influent	
Acetaminophen	337	U D	182000	D	212000	D	201000	D	208000	D	182000	D	233000	D
Azithromycin	11.8	U D	655	D	247	D	451	D	690	***J***	734	D	2210	D
Caffeine	84.1	U D	69900	D	118000	D	80700	D	108000	D	138000	D	168000	D
Carbadox	8.41	U D	72.2	U D	81.8	U D	61.9	U D	49.4	***UJ***	73.7	U D	39.6	U D
Carbamazepine	8.41	U D	1250	D	807	D	1100	D	1560	***J***	1300	D	536	D
Cefotaxime	33.7	U D	63.9	U D	184	U D	79.1	U D	131	***UJ***	157	***UJ***	114	U D
Ciprofloxacin	29.4	U D	748	D	449	D	701	D	632	D	1350	D	706	D
Clarithromycin	8.41	U D	251	D	379	D	116	D	150	D	190	D	170	D
Clinafloxacin	33.7	U D	39.9	U D	55.2	U D	42.5	U D	48.2	U D	54.3	U D	43.6	U D
Cloxacillin	16.8	U D	34.9	U D	27.9	U D	33.6	U D	39.1	***UJ***	33.8	U D	26.8	U D
Codeine	16.8	U D	827	D	231	D	464	D	884	D	566	D	852	D
Cotinine	12.3	***UJ***	4380	D	3610	D	3420	***J***	3360	***J***	4600	***J***	4360	***J***
Dehydronifedipine	3.37	U D	12.5	D	5.52	U D	11.5	D	21	***J***	12.4	D	9.16	D
Diphenhydramine	3.37	U D	3240	D	1360	D	1750	D	3120	***J***	3810	D	2780	D
Diltiazem	1.68	U D	1160	D	419	D	751	D	1080	***J***	927	D	545	D
Digoxin	84.1	***UJ***	373	***UJ***	199	***UJ***	338	***UJ***	325	***UJ***	418	U D	363	***UJ***
Digoxigenin	33.7	U D	95.8	U D	55.2	U D	40.6	U D	63.8	***J***	194	U D	72.1	U D
Enrofloxacin	16.8	U D	20	U D	27.6	U D	20.3	U D	19.5	U D	25.1	U D	21.8	U D
Erythromycin-H2O	6.17	U D	359	D	556	***J***	238	D	333	D	390	D	255	D

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (Replicate)		MWRWP-Influent		Hayden-Influent	
Flumequine	8.41	U D	10.5	U D	13.8	U D	10.2	U D	14.8	***UJ***	12.6	U D	10.9	U D
Fluoxetine	8.41	U D	56.2	D	37.9	D	131	D	224	D	68.6	D	67.6	D
Lincomycin	16.8	U D	58.2	U D	33.7	U D	20.3	U D	36	***UJ***	29.9	U D	21.8	U D
Lomefloxacin	16.8	U D	20	U D	27.6	U D	20.3	U D	19.5	U D	25.1	U D	21.8	U D
Miconazole	8.41	U D	36.8	D	13.9	D	32.1	D	29.9	***J***	33.6	***J***	13.7	D
Norfloxacin	84.1	U D	99.8	U D	138	***UJ***	102	U D	97.5	U D	126	U D	109	U D
Norgestimate	16.8	U D	24.3	***UJ***	27.6	U D	20.3	***UJ***	33.8	***UJ***	32.9	***J***	23.4	***UJ***
Ofloxacin	84.1	U D	509	D	174	D	212	D	211	D	259	D	342	D
Ormetoprim	3.37	U D	3.99	U D	5.52	U D	4.06	U D	3.9	***UJ***	5.03	U D	4.36	U D
Oxacillin	16.8	U D	20	U D	27.6	U D	20.3	U D	19.5	***UJ***	25.1	U D	21.8	U D
Oxolinic Acid	3.37	U D	3.99	U D	5.52	U D	4.06	U D	4.9	***UJ***	5.03	U D	5.14	U D
Penicillin G	16.8	U D	20	U D	27.6	U D	20.3	U D	19.5	***UJ***	25.1	U D	21.8	U D
Penicillin V	16.8	U D	72.6	D	27.6	U D	49.8	D	74.1	***J***	***51.7***	D	39.6	D
Roxithromycin	1.68	U D	2	U D	2.76	U D	2.03	U D	1.95	U D	2.51	U D	2.18	U D
Sarafloxacin	76.7	U D	91	U D	224	U D	92.6	U D	88.9	U D	115	U D	99.4	U D
Sulfachloropyridazine	8.41	U D	9.98	U D	13.8	U D	10.2	U D	9.75	U D	12.6	U D	10.9	U D
Sulfadiazine	8.41	U D	27.2	D	17.5	D	15.7	D	19.9	D	12.6	U D	17.3	D
Sulfadimethoxine	1.68	U D	7.03	U D	10.1	U D	7.25	D	9.13	D	2.51	U D	4.25	U D
Sulfamerazine	3.37	U D	22.8	D	5.67	U D	20.4	D	13	D	5.03	U D	26.1	D
Sulfamethazine	3.37	U D	13.3	U D	9.04	D	13.5	U D		U D	16.7	U D	14.5	U D
Sulfamethizole	3.37	U D	11.5	U D	8.18	U D	4.06	U D		U D	5.9	U D	5.57	U D
Sulfamethoxazole	3.37	U D	3420	D	2770	D	3820	D	4200	D	2880	D	2780	D
Sulfanilamide	280	U D	332	U D	460	U D	338	U D	13	U D	418	U D	363	U D
Sulfathiazole	8.41	U D	9.98	U D	13.8	U D	10.2	U D	4.76	U D	12.6	U D	10.9	U D
Thiabendazole	8.41	U D	22.8	D	18.1	***J***	20.8	D	21.1	D	28.5	D	14.3	D
Trimethoprim	8.41	U D	1400	D	1030	D	998	D	1530	***J***	979	D	611	D
Tylosin	112	U D	127	U D	372	U D	54.5	U D	67.8	U D	91.9	U D	105	U D
Virginiamycin	16.8	U D	75.2	U D	41.8	U D	63.5	U D	93.1	***UJ***	89.6	U D	65.7	U D
1,7-Dimethylxanthine	841	U D	36200	D	33100	D	55600	D	55900	D	64100	D	46300	D
Gemfibrozil	2.8	U	5660		4400		4840		4580		6920		21900	D
Ibuprofen	28	U	28900		29400		27800		31600		38600		33000	

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (Replicate)		MWRWP-Influent		Hayden-Influent	
Naproxen	6.85		28400	***J***	21800	D	22400	D	21000	D	25100	D	53600	D
Triclocarban	5.61	U	541	***J***	289	***J***	308	***J***	334	***J***	518	***J***	301	
Triclosan	112	U	2010		1860	***J***	1580	***J***	1600	***J***	2770	***J***	1480	***J***
Warfarin	2.8	U	10.4	D	14.1		11.4	***J***	10.6	***J***	10.8	***J***	11.7	***J***
Anhydrochlortetracycline (ACTC)	32.9	***U***	66.5	***U***	57.8	***UJ***	87.1	***U***	75.4	***U***	96.6	U	140	***U***
Anhydrotetracycline (ATC)	28	***U***	13.3	***U***	94.6	***UJ***	83	***U***	86.3	***U***	89.9	U	88.9	***U***
Chlortetracycline (CTC)	11.2	***U***	33.3	***U***	68.7	***UJ***	13.5	***U***	13	***U***	16.8	U	14.5	***U***
Demeclocycline	28	***U***	17.7	***U***	48.7	***UJ***	54.2	***U***	35.1	***U***	65.8	U	45.4	***U***
Doxycycline	11.2	***U***	***135***		32.9	***J***	82.1		77.3		155		54.3	
4-Epianhydrochlortetracycline (EACTC)	112	***U***	133	***U***	184	***UJ***	135	***U***	130	***U***	168	U	145	***U***
4-Epianhydrotetracycline (EATC)	28	***U***	47	***U***	46	***UJ***	84.7	***U***	113	***U***	66.7		48.4	***U***
4-Epichlortetracycline (ECTC)	28	***U***	34.5	***U***	48.2	***UJ***	72.6	***U***	42.9	***U***	80.1	U	70.9	***U***
4-Epioxytetracycline (EOTC)	11.2	***U***	23.5	***U***	27.6	***UJ***	36.9	***U***	65.4	***U***	63.2	U	38	***U***
4-Epitetracycline (ETC)	11.2	***U***	57.6		35.8	***UJ***	91.5		29.1		140		32.2	
Isochlortetracycline (ICTC)	11.2	***U***	13.3	***U***	18.4	***UJ***	13.5	***U***	14	***U***	16.9	U	15.3	***U***
Minocycline	112	***U***	143	***U***	189	***UJ***	135	***U***	130	***U***	168	***UJ***	145	***U***
Oxytetracycline (OTC)	11.2	***U***	16.2	***U***	19.5	***UJ***	24.2	***U***	41.1	***U***	40.4	U	25.1	***U***
Tetracycline (TC)	11.2	***U***	13.3		22.6	***J***	142		18.1		186		72.5	
Albuterol	0.693	U	26.1	D	32.5	D	29.6	D	27.1	D	28	D	25	D
Cimetidine	1.39	U	594	D	755	D	482	D	642	D	1480	***J***	1640	D
Metformin	73	U	123000	***J***	98900	***J***	107000	***J***	115000	***J***	126000	***J***	111000	***J***
Ranitidine	1.39	U	4620	D	4260	D	4790	D	3950	D	4840	***J***	3150	D

[1]= Blank data is not entered into EIM by protocol.

D = dilution; and the concentration was corrected at the laboratory. The D qualifier was not entered into EIM by protocol.

Table E-2. Method 1694 PPCP concentrations in wastewater effluents and reclaimed water discharges, ng/L, pptr.

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Dissolved		Hayden-AD Effluent		Hayden-WRF Effluent	
Acetaminophen	189	U D	184	U D	173	U D	173	U D	179	U D	160	U D	198	U D	171	U D	172	U D
Azithromycin	698	D	170	D	186	D	115	***J***	6.28	***UJ***	11.8	***J***	6.92	U D	608	D	376	D
Caffeine	747	D	45.9	U D	43.4	U D	105	D	53	U D	40	U D	49.5	U D	42.7	U D	43	U D
Carbadox	19	D	9.01	U D	11.4	D	15	***J***	108	***UJ***	65.3	***UJ***	26.5	D	16.1	D	22.8	U D
Carbamazepine	608	D	701	D	672	D	897	D	1710	D	1490	D	917	D	754	D	918	D
Cefotaxime	28.3	U D	46.2	U D	38.6	U D	43.6	***J***	369	***UJ***	95	***UJ***	53.1	***UJ***	39	U D	51.5	U D
Ciprofloxacin	419	D	96.8	D	205	D	211	D	15.7	U D	14	U D	17.3	U D	158	D	15.1	U D
Clarithromycin	170	D	257	D	142	D	108	D	4.48	U D	4.58	D	4.95	***UJ***	46.2	***J***	32.7	D
Clinafloxacin	41	U D	22.8	U D	17.6	U D	25.1	U D	59	U D	16	U D	23.7	U D	21.4	U D	17.2	***UJ***
Cloxacillin	12.2	U D	9.19	U D	8.67	U D	8.84	U D	39.9	U D	21.8	U D	9.89	U D	8.55	U D	9.5	U D
Codeine	410	D	57.4	D	30.2	D	73.3	***J***	8.97	***UJ***	8	***UJ***	9.89	U D	98.3	D	194	D
Cotinine	113	D	39.5	D	36.6	***J***	58.3	***J***	22.2	***UJ***	77.3	***R***	28.6	D	38.7	D	40	D
Dehydronifedipine	21.7	D	5.86	D	16.1	D	19.2	***J***	95.8	***UJ***	83.8	***UJ***	14	D	14.6	D	19.5	D
Diphenhydramine	924	D	286	D	291	D	448	***J***	17.3	***UJ***	43.5	***UJ***	4.13	D	255	D	343	D
Diltiazem	300	D	88.3	D	155	D	221	***J***	3.19	***UJ***	21.8	***UJ***	0.989	U D	84.3	D	145	D
Digoxin	55.3	***UJ***	45.9	***UJ***	45.3	***UJ***	144	***UJ***	565	***UJ***	137	***UJ***	49.5	U D	42.7	***UJ***	143	***UJ***
Digoxigenin	32	U D	18.4	U D	19.6	U D	35.1	***UJ***	145	***UJ***	66.1	***UJ***	19.8	U D	28.2	U D	33.4	U D
Enrofloxacin	10.2	U D	9.19	U D	8.67	U D	8.67	U D	8.97	U D	8	U D	9.89	U D	9.64	U D	8.6	U D
Erythromycin-H2O	327	D	247	D	169	D	183	D	3.29	U D	9.11	D	3.63	***UJ***	154	***J***	168	D
Flumequine	4.73	U D	4.59	U D	4.34	U D	4.33	***UJ***	11.4	***UJ***	6.57	***UJ***	4.95	U D	4.27	U D	4.3	U D
Fluoxetine	75.2	D	43.7	D	59.4	D	82.7	D	45.4	D	39.3	D	62.4	D	51.8	D	58.2	D
Lincomycin	9.8	U D	9.19	U D	12.8	D	8.67	***UJ***	8.97	***UJ***	8	***UJ***	9.89	U D	8.55	U D	4.3	U D
Lomefloxacin	9.46	U D	9.19	U D	8.67	U D	8.67	U D	8.97	U D	8	U D	9.89	U D	8.55	U D	8.6	U D
Miconazole	4.73	U D	4.59	U D	4.34	U D	4.33	***UJ***	4.48	***UJ***	4	***UJ***	4.95	***UJ***	4.27	U D	8.6	U D
Norfloxacin	47.3	U D	54.9	U D	43.4	U D	43.3	U D	44.8	U D	40	U D	49.5	U D	42.7	U D	4.3	U D
Norgestimate	9.46	***UJ***	9.19	***UJ***	8.67	***UJ***	10.5	***UJ***	22.4	***UJ***	14.8	***UJ***	9.89	***UJ***	8.55	***UJ***	43	***UJ***
Ofloxacin	639	D	210	D	86.7	D	130	D	44.8	U D	40	U D	49.5	U D	312	D	8.6	U D
Ormetoprim	1.89	U D	1.84	U D	1.73	U D	1.73	***UJ***	1.79	***UJ***	1.6	***UJ***	1.98	U D	1.71	U D	43	U D

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Dissolved		Hayden-AD Effluent		Hayden-WRF Effluent	
Oxacillin	9.46	U D	9.19	U D	8.67	U D	8.67	***UJ***	14.1	***UJ***	8	***UJ***	9.89	U D	8.55	U D	1.72	U D
Oxolinic Acid	1.89	U D	1.84	U D	1.73	U D	1.73	***UJ***	4.48	***UJ***	9.99	***J***	18.5	U D	1.71	U D	8.6	U D
Penicillin G	9.46	U D	9.19	U D	8.67	U D	8.67	***UJ***	28.3	***UJ***	30.7	***J***	9.89	U D	8.55	U D	2.61	U D
Penicillin V	9.46	U D	9.19	U D	8.67	U D	8.67	***UJ***	34.8	***UJ***	9.16	***UJ***	9.89	U D	8.55	U D	8.6	U D
Roxithromycin	1.37	U D	0.919	U D	6.44	D	3.92	D	0.897	U D	0.8	U D	0.989	***UJ***	0.855	***UJ***	8.6	U D
Sarafloxacin	83.5	***UJ***	41.9	U D	39.5	U D	39.5	U D	40.9	U D	36.5	***UJ***	45.1	U D	44.8	U D	0.86	U D
Sulfachloropyridazine	9.32	***UJ***	4.59	U D	4.34	U D	4.33	U D	4.48	U D	16.6	***UJ***	4.95	U D	4.27	***UJ***	39.2	U D
Sulfadiazine	4.73	***UJ***	19.2	D	4.34	U D	4.33	U D	4.48	U D	4	***UJ***	4.95	U D	4.27	***UJ***	4.3	U D
Sulfadimethoxine	2.08	***UJ***	0.919	U D	3.73	D	2.92	D	11.5	***UJ***	4.9	***UJ***	1.39	U D	3	***UJ***	4.3	U D
Sulfamerazine	11.4	***UJ***	1.84	U D	3.09	D	1.73	U D	1.79	U D	9.83	***UJ***	1.98	U D	4.11	***UJ***	1.65	U D
Sulfamethazine	13.5	***UJ***	6.26	U D	4.43	U D	5.77	U D	5.97	U D	16.7	***UJ***	1.98	U D	8.79	***J***	1.84	U D
Sulfamethizole	3.1	***UJ***	1.84	U D	1.73	U D	1.73	U D	8.21	***UJ***	3.9	U D	1.98	U D	2.15	***UJ***	5.73	U D
Sulfamethoxazole	1300	***J***	1420	D	1390	D	1490	D	72.7	***J***	192	***UJ***	104	D	1830	***J***	1.76	D
Sulfanilamide	252	***UJ***	153	U D	144	U D	144	U D	149	U D	225	***UJ***	165	U D	142	***UJ***	143	U D
Sulfathiazole	4.73	***UJ***	4.59	U D	4.34	U D	4.33	U D	9.38	***UJ***	5.72	***UJ***	4.95	U D	4.27	***UJ***	4.3	U D
Thiabendazole	31.1	D	27.1	D	24.4	D	24.4	***J***	22.7	D	20.9	D	24.1	D	22.7	D	19.7	D
Trimethoprim	791	D	334	D	682	D	542	***J***	35.5	***UJ***	73.3	***J***	11.5	U D	308	D	294	D
Tylosin	196	U D	110	U D	26.1	U D	17.3	U D	40.5	U D	10.7	U D	174	***UJ***	83.6	***UJ***	35	U D
Virginiamycin	18.9	U D	9.19	U D	11.4	U D	16.9	***UJ***	73.3	***UJ***	46.7	U D	9.89	U D	12	U D	18.5	U D
1,7-Dimethylxanthine	473	U D	459	U D	434	U D	433	U D	448	U D	46.9	U D	495	U D	427	U D	430	U D
Gemfibrozil	3880	D	585		261		241		14.9	***UJ***	5.68	***UJ***	46.5		1210		1230	
Ibuprofen	147		99.1		29.8		26.9		33		26.6		74.2		170		158	
Naproxen	340		113		19.6	***J***	18.2	***J***	127	***R***	38.5	***J***	3.3	U	242		251	
Triclocarban	78.4	***J***	42.7		30.5		32.3	***J***	2.99	U	2.67	U	103		51.3		52.4	***J***
Triclosan	805		61.2	U	125		102		59.8	U	53.3	U	65.9	U	57	U	76.8	
Warfarin	8.35	***J***	10.3		10.3		9.73	***J***	8.53	***R***	3.14	***UJ***	1.65	U	10.6		11.1	
Anhydrochlortetracycline (ACTC)	28.5	***U***	36	***U***	35.8	***U***	41.2	***UJ***	23.3	***U***	23.5	***U***	23.9	U	22.6	***U***	23.4	***U***
Anhydrotetracycline (ATC)	15.8	***U***	15.3	***U***	14.5	***U***	14.4	***UJ***	14.9	***U***	13.3	***U***	16.5	U	14.2	***U***	14.3	***U***
Chlortetracycline	6.31	***U***	7.06	***U***	8.24	***U***	6.46	***UJ***	8.85	***U***	8.83	***U***	6.59	U	7.78	***U***	10.2	***U***

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Dissolved		Hayden-AD Effluent		Hayden-WRF Effluent	
(CTC)																		
Demeclocycline	15.8	***U***	15.3	***U***	15.4	***U***	14.4	***UJ***	14.9	***U***	13.3	***U***	16.5	U	14.2	***U***	16.8	***U***
Doxycycline	48		16		33.8		***28.5***	***J***	5.98	***U***	5.32	***U***	6.59	U	11.8		5.74	***U***
4-Epi-anhydrochlortetracycline (EACTC)	63.1	***U***	87.2	***U***	88.5	***U***	107	***UJ***	59.8	***U***	53.2	***U***	65.9	U	57	***U***	57.4	***U***
4-Epi-anhydrotetracycline (EATC)	27.9	***U***	15.3	***U***	28.9	***U***	30.8	***UJ***	17.1	***U***	23.1	***U***	16.5	U	16.4		15.8	
4-Epichlortetracycline (ECTC)	15.8	***U***	21.5	***U***	24.3	***U***	19.8	***UJ***	25.9	***U***	25.5	***U***	18.7	U	23.1	***U***	29.2	***U***
4-Epioxytetracycline (EOTC)	6.42	***U***	6.59	***U***	23.8	***U***	8.66	***UJ***	7.8	***U***	19	***U***	15	U	7.88	***U***	8.77	***U***
4-Epitetracycline (ETC)	33		8.37		26		***25.6***	***J***	8.81	***U***	7.02	***U***	8.47	U	14.4		18	***U***
Isochlortetracycline (ICTC)	6.31	***U***	6.12	***U***	5.78	***U***	5.78	***UJ***	5.98	***U***	5.32	***U***	6.59	U	5.7	***U***	5.74	***U***
Minocycline	63.1	***U***	64.6	***U***	64.4	***U***	57.8	***UJ***	65.6	***U***	57.7	***U***	65.9	***UJ***	67.6	***U***	83.7	***U***
Oxytetracycline (OTC)	6.31	***U***	6.12	***U***	13.8	***U***	5.9	***UJ***	5.98	***U***	11.2	***U***	9.39	U	5.7	***U***	5.95	***U***
Tetracycline (TC)	40.3		9.73		29.2		***32***		6.29	***U***	5.32	***U***	6.59	U	15.9		12.5	***U***
Albuterol	21.7	D	21.7	D	15.3	D	14.8	D	0.861	U D	0.897	U D	0.972	U D	25.8	D	24.2	D
Cimetidine	607	D	140	D	241	D	240	D	1.72	U D	1.79	U D	1.94	***UJ***	245	D	309	D
Metformin	43800	***J***	34900	***J***	4720	***J***	4050	***J***	627	***J***	457	***J***	1760	***J***	nq	***R***	nq	***R***
Ranitidine	1630	D	283	D	700	D	777	D	1.72	U D	1.79	U D	1.94	***UJ***	863	D	735	D

nq = Not quantified due to analytical instrument error.

* Not a mean because one replicate was undetected.

D = dilution; and the concentration was corrected at the laboratory. The D qualifier was not entered into EIM by protocol.

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Table E-3. Method 1694 PPCP concentrations in biosolids, ug/Kg (dw), ppb.

Analyte	CC-Biosolids		BITP-Biosolids		BITP-Biosolids (replicate)		Puy-Biosolids	
Acetaminophen	96.4	U	111	U	105	U	109	U
Azithromycin	159		145		142		289	
Caffeine	24.1	U	7.43	U	26.3	U	27.1	U
Carbadox	6.34	U	2.78	U	16.8	U	8.47	U
Carbamazepine	265		393		323		376	
Cefotaxime	31.3	U	33.6	U	36.8	U	29.2	U
Ciprofloxacin	10800		11900	D	12800	D	11000	
Clarithromycin	3.43		7		7.23		4.51	
Clinafloxacin	40.7	U	33.3	U	17.3	U	35.4	U
Cloxacillin	7.49	U	9.94	U	9.21	U	14.9	U
Codeine	31.8		5.57	U	5.27	U	5.43	U
Cotinine	9.57	***J***	9.28	***UJ***	8.78	***UJ***	21.1	***J***
Dehydronifedipine	1.37	U	1.37	U	1.27	U	1.09	U
Diphenhydramine	2190	D	2600	D	2450	D	2340	D
Diltiazem	11.9		7.74		6.72		5.61	
Digoxin	80.3	U	92.8	U	87.8	U	90.5	U
Digoxigenin	10.8	U	20	U	18.9	U	11.9	U
Enrofloxacin	9.79		13.4		15.2		14.6	
Erythromycin-H2O	15.3		13.8		7.59		8.33	
Flumequine	4.45	U	5.16	U	3.48	U	2.97	U
Fluoxetine	522	***J***	675		630		459	
Lincomycin	5.09	U	7.02	U	5.27	U	10.1	U
Lomefloxacin	4.82	U	9.61		7.52		5.43	U
Miconazole	1560		1660		1530		1710	
Norfloxacin	108		89.1		84.5		261	
Norgestimate	8.04	U	9.47	U	9.21	U	12.6	U
Ofloxacin	6830		6070		5500		6090	
Ormetoprim	0.964	U	1.11	U	1.05	U	1.09	U
Oxacillin	4.82	U	5.57	U	5.27	U	5.43	U
Oxolinic Acid	2.21		2.66		2.05		2.18	U
Penicillin G	4.82	U	5.57	U	5.27	U	5.43	U
Penicillin V	9.64	U	11.1	U	10.5	U	10.9	U
Roxithromycin	3.02	U	1.77	U	2.26	U	2	U
Sarafloxacin	22	U	25.4	U	24	U	52.7	U
Sulfachloropyridazine	2.41	U	2.78	U	2.63	U	2.71	U
Sulfadiazine	2.41	U	2.78	U	2.63	U	2.71	U
Sulfadimethoxine	0.771	U	0.617	U	0.527	U	0.601	U
Sulfamerazine	0.964	U	1.11	U	1.05	U	1.09	U
Sulfamethazine	3.21	U	3.71	U	3.51	U	3.62	U
Sulfamethizole	1.27	U	1.6	U	1.54	U	2.14	U

Analyte	CC-Biosolids		BITP-Biosolids		BITP-Biosolids (replicate)		Puy-Biosolids	
Sulfamethoxazole	1.75	U	1.43		1.14	U	1.43	
Sulfanilamide	80.3	***UJ***	92.8	***UJ***	87.8	***UJ***	90.5	***UJ***
Sulfathiazole	2.41	U	2.78	U	2.63	U	2.71	U
Thiabendazole	16.8		32.1		31.3		54	
Trimethoprim	2.41	U	4.39	U	5.57	U	2.71	U
Tylosin	110	U	120	U	245	U	261	U
Virginiamycin	48.7	U	47.5	U	61.9	U	68.2	U
1,7-Dimethylxanthine	241	U	278	U	263	U	271	U
Gemfibrozil	211	D	277	D	223	D	14500	D
Ibuprofen	458		460		415		499	
Naproxen	8.91	***UJ***	13.6		6.88		5.45	U
Triclocarban	12900	***J***	18400	***J***	17000	***J***	2.71	U
Triclosan	36600	D	8210	D	7760	D	19800	D
Warfarin	2.41	U	2.78	U	2.63	U	134	D
Anhydrochlortetracycline (ACTC)	24.1	U	27.8	U	26.3	U	10.9	U
Anhydrotetracycline (ATC)	122	***J***	291	***J***	301	***J***	56.6	
Chlortetracycline (CTC)	9.64	U	11.1	U	10.5	U	1450	
Demeclocycline	24.1	U	27.8	U	26.3	U	27.1	U
Doxycycline	3150		2370		2370		45.1	***J***
4-Epianhydrochlortetracycline (EACTC)	96.4	***UJ***	111	***UJ***	105	***UJ***	109	***UJ***
4-Epianhydrotetracycline (EATC)	146		471		415		74.3	
4-Epichlortetracycline (ECTC)	24.1	U	27.8	U	26.3	U	27.1	U
4-Epioxytetracycline (EOTC)	14.2	U	11.1	U	10.5	U	10.9	U
4-Epitetracycline (ETC)	2820		3640		3400		1530	
Isochlortetracycline (ICTC)	9.64	U	11.1	U	10.5	U	10.9	U
Minocycline	433	***J***	378	***J***	429	***J***	272	***J***
Oxytetracycline (OTC)	65.8		45		45.7		20.5	
Tetracycline (TC)	3200		3300		3280		1220	
Albuterol	0.715	U	0.969	U	0.567	U	0.945	U
Cimetidine	13.4	***J***	55.1	***J***	35.7	***J***	24	***J***
Metformin	116		86.3	U	63	U	149	U
Ranitidine	7.32		4.98		6.38		4.32	

D = dilution; and the concentration was corrected at the laboratory. The D qualifier was not entered into EIM by protocol.

Table E-4. Method 1698 hormones and steroid concentrations in wastewater influents, ng/L, pptr.

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (replicate)		MWRWP-Influent		Hayden-Influent	
17a-Ethinyl-Estradiol	2.12	***U***	15.1	***U***	12.5	***U***	14.3	***U***	11.5	***U***	13.7	***U***	16.8	***U***
17a-Dihydroequilin	0.584	***U***	10	U	8.82	U	12.5	***J***	10	U	13.7	U	14.7	U
17a-Estradiol	0.016	***U***	7.22	***J***	6.18	***J***	5.31	***J***	6.95	***J***	7.75	***J***	5.17	***J***
17b-Estradiol	2.22	***U***	25.1	***U***	25.1	***U***	25.5	***U***	20.4	***U***	33.6	***UJ***	32.1	***U***
Androstenedione	2.43	U	620	U	466	U	767	***J***	519	U	761	U	469	U
Androsterone	0.0308	U	1240	***J***	1520	***J***	1410	***J***	1510	***J***	2060		1480	***J***
b-Estradiol 3-benzoate	5.96	U	7.87	U	11.2	U	15.5	U	21.8	U	21.6	U	25.4	***J***
b-Sitosterol	283	***U***	514000		519000		508000		470000		617000		643000	
b-Stigmastanol	3.47	***U***	37500		36900		40300		38600		50100		48300	
Campesterol	6.72	***U***	154000		157000		150000		150000		176000		194000	
Cholestanol	1.33	***U***	72800		51800		64900		62900		90700		76400	
Cholesterol	97.5	***U***	2440000		2560000		2540000		2600000		3410000		3020000	
Coprostanol	0.0133	U	1920000		1930000		1990000		2100000		2760000		2480000	
Desmosterol	2.02	U	10300	***J***	13500	***J***	10500	***J***	11000	***J***	13800	***J***	13300	***J***
Desogestrel	0.026	U	30.2	***J***	22.1	***J***	18.1	J	18.9	***J***	45.9	***J***	20.7	***J***
Epicoprostanol	0.137	***U***	31700		15000		23800		23600		34600		19600	
Equilenin	0.996	***U***	7.33	U	8.8	U	9.95	U	8.06	U	5.57	U	8.47	U
Equilin	0.0194	U	12	***U***	141	***J***	31.8	***J***	31.5	***J***	12	***U***	4.66	***U***
Ergosterol	1.56	U	17500		19500		16400		15100		24100		16300	
Estriol	1.15	U	162	***J***	175	***J***	144	***J***	133	***J***	264	***J***	317	***J***
Estrone	0.094	***U***	106	***J***	196	***J***	98.7	***J***	111	***J***	87.8	***J***	71.4	***J***
Mestranol	1.81	***U***	9.9	U	14.8	U	11.1	U	9.76	U	10.1	***U***	10.6	***U***
Norethindrone	4.01	***UJ***	25.6	U	16.7	U	18.4	U	12.8	U	9.22	***J***	17.6	U
Norgestrel	4.44	***UJ***	45.7	U	70.9	***J***	43.4	***J***	48.1	***J***	50.4	U	39.9	U
Progesterone	14.4	***UJ***	180	***U***	320	U	244	U	421	U	367	U	314	U
Stigmasterol	62.8	***U***	80600		82200		76500		70600		91800		105000	
Testosterone	3.46	U	3180	***J***	2690	***J***	2900	***J***	3040	***J***	3730	***J***	3170	***J***

[1]= Blank data is not entered into EIM by protocol.

Table E-5. Method 1698 hormones and steroid concentrations in wastewater effluents ng/L, pptr.

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Discharge		Hayden-AD Effluent		Hayden-WRF Effluent	
17a-Ethinyl-Estradiol	3.16	***U***	1.92	***U***	2.02	***U***	1.75	***U***	1.43	***U***	1.47	***U***	1.27	***U***	2.02	***UJ***	2.15	***U***
17a-Dihydroequilin	1.85	U	1.02	U	1.05	U	1.04	U	1.54	U	0.456	U	0.851	U	0.591	U	0.764	U
17a-Estradiol	1.78	***J***	0.289	***U***	0.075	***U***	0.0273	U	0.033	***U***	0.054	***U***	0.041	***U***	0.206	***U***	0.091	***U***
17b-Estradiol	***11.9***	***J***	1.79	***U***	1.77	***U***	1.38	***U***	0.699	***U***	0.705	***U***	0.935	***U***	1.99	***U***	2.33	***U***
Androstenedione	36.4	U	9.36	U	13.4	***U***	9.95	U	21.2	U	19.3	U	10.9	U	17	U	11.9	U
Androsterone	0.451	***J***	0.0102	U	0.0057	U	0.0064	U	0.222	U	0.0927	U	0.0025	U	0.0066	U	0.0078	U
b-Estradiol 3-benzoate	0.998	U	2.14	U	1.08	U	2.05	U	1.47	U	1.2	U	1.3	U	1.6	U	1.7	U
b-Sitosterol	6110		925	***U***	5670		3330		13.2	***U***	10.2	***U***	11.1	***U***	6720		370	***U***
b-Stigmastanol	567		76.3		450		404		11.5	***U***	6.35	***U***	3	***U***	176		6.61	***U***
Campesterol	2050	***J***	283		694		698	***J***	5.3	***J***	3.11	***J***	2.01	U	797		23.3	***U***
Cholestanol	1370		602		1940		1830		***46.4***		44.1	***U***	3.7	***U***	712		19.8	***U***
Cholesterol	25700	D	3250		7410		7080		108	***U***	101	***U***	38.3	***U***	5500		297	***U***
Coprostanol	28200	D	1170		6700		6340		151		145		7.13	***J***	2590		81.3	
Desmosterol	862		159	***U***	823	***J***	808	***J***	***35.6***		32.9		7.13	***J***	315	***J***	10.2	***U***
Desogestrel	7.04	***J***	0.738	U	1.01	***J***	0.808	U	1.86	U	1.43	U	1.77	U	1.15	U	1.01	U
Epicoprostanol	716		29.5	J	337		287		15		13.6	***J***	1.96	U	92.8		3.65	***J***
Equilenin	3.43	U	1.21	U	1.45	***U***	1.6	***U***	1.31	U	1.27	U	1.3	U	1.46	***U***	1.49	***U***
Equilin	1.98	U	0.698	U	0.758	U	0.876	U	1.43	U	1.26	U	1.17	U	1.07	U	0.745	U
Ergosterol	1650		170		1700		1610		1.67		2.05	U	1.52	U	2680		116	
Estriol	0.848	U	1.63	U	0.676	U	1.55	U	0.825	U	0.545	U	0.317	U	0.947	U	0.827	U
Estrone	1000	***J***	20.2		2.19	***U***	1.88	***U***	2.02	***U***	0.828	U	0.53	U	24.1		39.2	
Mestranol	1.59	U	1.19	U	0.934	U	1.02	U	1.5	U	1.52	U	1.48	U	1.02	U	0.882	U
Norethindrone	3.52	U	3.57	U	1.83	U	2.77	U	4.05	U	1.67	U	2.22	U	2.79	U	3.08	U
Norgestrel	8.26	U	7.38	U	3.48	U	8	U	6.32	U	6.46	U	4.15	U	4.78	U	5.62	U
Progesterone	11.6	U	12.1	U	5.65	U	9.42	U	20.2	U	10.1	U	7.76	U	6.96	U	5.77	U
Stigmasterol	6610		1320		8860		7960		8.23	***U***	6.37	***U***	7.84	***U***	25700		231	***U***
Testosterone	25.9	U	6.39	U	8.72	U	10.9	U	31.8	U	9.87	U	16.7	U	10.2	U	9.6	U

Table E-6. Method 1698 hormones and steroid concentrations in biosolids, ug/Kg (dw), ppb.

Analyte	CC-Biosolids		BITP-Biosolids		BITP-Biosolids (replicate)		Puy-Biosolids	
17a-Ethinyl-Estradiol	24.4	***U***	40.1	***U***	16.9	***U***	23.2	***U***
17a-Dihydroequilin	10.3	U D	18.2	U D	21	U D	7.92	U D
17a-Estradiol	14	D J	8.65	***U***	4.64	***U***	6.8	***U***
17b-Estradiol	20	***U***	40.2	***U***	46.1	***U***	22.5	***U***
Androstenedione	257	***J***	263	U D	190	***J***	191	U D
Androsterone	61	D J	11.9	***J***	0.0962	U D	6.41	***U***
b-Estradiol 3-benzoate	70.4	U D	74	U D	46.3	U D	56.7	D J
b-Sitosterol	729000	***J***	675000	***J***	737000	***J***	440000	
b-Stigmastanol	269000	***J***	366000	***J***	387000	***J***	102000	
Campesterol	524000	***J***	388000	***J***	377000	***J***	136000	***J***
Cholestanol	1420000	***J***	1590000	***J***	1720000	***J***	601000	
Cholesterol	832000	***J***	756000	***J***	830000	***J***	478000	
Coprostanol	4030000	***J***	3610000	***J***	3850000	***J***	1620000	
Desmosterol	41100	***J***	43100	***J***	47800	***J***	18500	***J***
Desogestrel	11.3	***J***	13.5	U D	18.6	U D	2.56	U D
Epicoprostanol	3280000	***J***	2540000	***J***	2720000	***J***	985000	
Equilenin	10.3	U D	14.5	U D	20.1	U D	15.1	***U***
Equilin	8.63	U D	45.8	D J	51.4	D	6.03	U D
Ergosterol	123000	***J***	37300	***J***	119000	***J***	48800	
Estriol	22.4	***J***	20.6	U D	3.01	***U***	10.9	***U***
Estrone	228	D	58.2	***J***	53.2	***J***	38.6	***J***
Mestranol	27.2	U D	30	***U***	68.8	***J***	16.9	***U***
Norethindrone	124	***J***	1590	***J***	101	U D	393	***J***
Norgestrel	195	***J***	1900	***J***	119	U D	500	***J***
Progesterone	854	***U***	652	***U***	218	U D	484	***U***
Stigmasterol	240000	***J***	163000	***J***	178000	***J***	110000	
Testosterone	82.9	U D	134	U D	216	U D	95.3	U D

Table E-7. Method 8270 semi-volatile organics in wastewater influents, ug/L, ppb.

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (replicate)		MWRWP-Influent		Hayden-Influent	
1,2,4-Trichlorobenzene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
1,2-Dichlorobenzene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
1,2-Diphenylhydrazine	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
1,3-Dichlorobenzene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
1,4-Dichlorobenzene	0.29	U	0.91		0.3	UJ	0.65		0.57		0.78		0.65	
1-Methylnaphthalene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
2,2'-Oxybis[1-chloropropane]	0.29	UJ	0.31	UJ	0.3	UJ	0.27	UJ	0.29	U	0.31	UJ	0.28	UJ
2,4,5-Trichlorophenol	1.2	U	1.2	U	1.2	U	1.1	U	1.2	U	1.2	U	1.1	U
2,4,6-Trichlorophenol	1.2	U	1.2	U	1.2	U	1.1	U	1.2	U	1.2	U	1.1	U
2,4-Dichlorophenol	2.9	U	0.47	J	3	UJ	2.7	U	2.9	U	3.1	U	0.41	J
2,4-Dimethylphenol	2.9	U	3.1	U	3	UJ	2.7	U	2.9	U	3	U	2.8	U
2,4-Dinitrophenol		REJ		REJ		REJ		REJ	2.9	U		REJ		REJ
2,4-Dinitrotoluene	1.2	U	1.2	U	1.2	UJ	1.1	U	1.2	U	1.2	U	1.1	U
2,6-Dinitrotoluene	1.2	U	1.2	U	1.2	UJ	1.1	U	1.2	U	1.2	U	1.1	U
2-Chloronaphthalene	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
2-Chlorophenol	1.2	U	1.2	U	1.2	UJ	1.1	U	1.2	U	1.2	U	1.1	U
2-Methylnaphthalene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
2-Methylphenol	2.9	U	3.1	U	3	UJ	2.7	U	2.9	U	3	U	2.8	U
2-Nitroaniline	5.9	U	6.2	U	5.9	UJ	5.4	U	5.8	U	6.1	U	5.5	U
2-Nitrophenol	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
3-Nitroaniline	1.2	U	1.2	U	1.2	UJ	1.1	U	1.2	U	1.2	U	1.1	U
4,6-Dinitro-2-Methylphenol		REJ		REJ		REJ		REJ	1.2	U		REJ		REJ
4-Bromophenyl-Phenylether	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
4-Chloro-3-Methylphenol	2.9	U	3.1	U	3	UJ	2.9	U	2.9	U	3	U	2.8	U
4-Chloroaniline		REJ		REJ		REJ		REJ	12	U		REJ		REJ
4-Chlorophenyl-Phenylether	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
4-Methylphenol	0.29	U	37		5.1	J	46		41		93		154	
4-Nitroaniline		REJ		REJ		REJ		REJ	1.2	U		REJ		REJ
4-Nitrophenol	2.9	U	3.1	U	3	UJ	2.7	U	2.9	U	3	U	2.8	U

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (replicate)		MWRWP-Influent		Hayden-Influent	
Acenaphthene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Acenaphthylene	0.29	UJ	0.31	UJ	0.3	UJ	0.27	UJ	0.29	U	0.31	UJ	0.28	UJ
Anthracene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Benzo(a)anthracene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Benzo(a)pyrene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Benzo(b)fluoranthene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Benzo(ghi)perylene	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
Benzo(k)fluoranthene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Benzoic Acid	2.9	UJ	330	J	365	J	270	J	181	J	223	J	835	J
Benzyl Alcohol	2.9	U	20		8.9	J	49		48		24		14	
Bis(2-Chloroethoxy)Methane	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Bis(2-Chloroethyl)Ether	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
Bis(2-Ethylhexyl) Phthalate	0.29	U	20		7.8	J	28		24		33		23	
Bisphenol A	1		1.3	J	2.1	J	0.54	U	0.58	U	44		24	J
Butylbenzylphthalate	0.59	U	2		0.62	J	16		15		7.6		0.55	U
Carbazole	0.29	U	0.62	UJ	0.6	UJ	0.54	UJ	0.58	U	0.61	UJ	0.55	UJ
Chrysene	0.59	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Dibenzo(a,h)anthracene	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
Dibenzofuran	0.59	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
Diethylphthalate	0.29	U	7.5		0.95	J	6.8		6.5		11		10	
Dimethylphthalate	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Di-N-Butylphthalate	0.59	U	0.9		0.3	UJ	3.3		3.1		1.7		0.28	U
Di-N-Octyl Phthalate	0.29	U	0.62	U	0.6	UJ	0.54	U	0.58	U	0.61	U	0.55	U
Fluoranthene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Fluorene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Hexachlorobenzene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Hexachlorobutadiene	1.2	UJ	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	UJ	0.28	U
Hexachlorocyclopentadiene	1.2	UJ	1.2	UJ	1.2	UJ	1.1	UJ	1.2	U	1.2	UJ	1.1	UJ
Hexachloroethane	5.9	U	1.2	UJ	1.2	UJ	1.1	UJ	1.2	U	1.2	U	1.1	UJ
Indeno(1,2,3-cd)pyrene	0.59	U	6.2	U	5.9	UJ	5.4	U	5.8	U	6.1	U	5.5	U

Analyte	CC-Blank[1]		CC-Influent		Puy-Influent		BITP-Influent		BITP-Influent (replicate)		MWRWP-Influent		Hayden-Influent	
Isophorone	0.29	U	0.62	U		UJ	0.54	U	0.58	U	0.61	U	0.55	U
Naphthalene	0.29	U	0.15	J	0.3	UJ	0.17	J	0.14	J	0.31	U	0.16	J
Nitrobenzene	1.2	UJ	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	UJ	0.28	U
N-Nitrosodimethylamine	0.29	U	1.2	UJ	1.2	UJ	1.1	U	1.2	UJ	1.2	UJ	1.1	U
N-Nitroso-Di-N-Propylamine	0.59	UJ	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	UJ	0.28	U
N-Nitrosodiphenylamine	2.9	U	0.62	UJ	0.6	UJ	0.54	UJ	0.58	U	0.61	UJ	0.55	UJ
Pentachlorophenol	0.29	U	3.1	UJ	6.6	J	2.7	U	2.9	U	3	U	2.8	U
Phenanthrene	0.12	J	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Phenol	0.59	U	45		22	J	42		44		49		64	
Phenol, 4-Nonyl-	0.29	U	0.34	J	0.4		0.54	U	0.58	U	0.61	U		U
Pyrene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Retene	0.29	U	0.31	U	0.3	UJ	0.27	U	0.29	U	0.31	U	0.28	U
Tri(2-chloroethyl) phosphate	2.9	U	0.82		0.47	J	0.27	U	0.29	U	3.6		0.28	U
Triethyl citrate			4		2.4	J	3.9		4.1		5.4		4.6	

Table E-8. Method 8270 semi-volatile organics in wastewater effluents and reclaimed water discharges, ug/L, ppb.

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Discharge		Hayden-AD Effluent		Hayden-WRF Effluent	
1,2,4-Trichlorobenzene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
1,2-Dichlorobenzene	0.11	J	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
1,2-Diphenylhydrazine	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
1,3-Dichlorobenzene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
1,4-Dichlorobenzene	0.32		0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.68		0.28	U	0.26	U
1-Methylnaphthalene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
2,2'-Oxybis[1-chloropropane]	0.28	UJ	0.31	UJ	0.27	UJ	0.28	UJ	0.28	UJ	0.28	UJ	0.29	UJ	0.28	UJ	0.26	UJ
2,4,5-Trichlorophenol	1.1	U	1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	1.2	U	1.1	U	1	U
2,4,6-Trichlorophenol	0.89	J	0.92	J	1.1	U	0.81	J	1.1	U	1.1	U	1	J	1.1	U	1	U
2,4-Dichlorophenol	2.8	U	3.1	U	2.7	U	2.8	U	2.8	U	2.8	U	2.9	U	2.8	U	2.6	U
2,4-Dimethylphenol	2.8	U	3	U	2.7	U	2.8	U	2.8	U	2.8	U	2.9	U	2.8	U	2.6	U
2,4-Dinitrophenol		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ
2,4-Dinitrotoluene	1.1	U	1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	1.2	U	1.1	U	1	U
2,6-Dinitrotoluene	1.1	U	1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	1.2	U	1.1	U	1	U
2-Chloronaphthalene	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
2-Chlorophenol	1.1	U	1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	1.2	U	1.1	U	1	U
2-Methylnaphthalene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
2-Methylphenol	2.8	U	3	U	2.7	U	2.8	U	2.8	U	2.8	U	2.9	U	2.8	U	2.6	U
2-Nitroaniline	5.7	U	6.1	U	5.3	U	5.5	U	5.6	U	5.6	U	5.8	U	5.6	U	5.2	U
2-Nitrophenol	0.57	U	0.61	U	0.53	UJ	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
3-Nitroaniline	1.1	U	1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	1.2	U	1.1	U	1	U
4,6-Dinitro-2-Methylphenol		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ
4-Bromophenyl-Phenylether	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
4-Chloro-3-Methylphenol	2.8	U	3	U	2.7	U	2.8	U	2.8	U	2.8	U	2.9	U	2.8	U	2.6	U
4-Chloroaniline		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ
4-Chlorophenyl-Phenylether	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
4-Methylphenol	0.32	J	0.31	U	0.24	J	0.19	J	0.18	J	0.18	J	26		0.15	J	0.26	U
4-Nitroaniline		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ		REJ

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Discharge		Hayden-AD Effluent		Hayden-WRF Effluent	
4-Nitrophenol	2.8	U	3	U	2.7	U	2.8	U	2.8	U	2.8	U	2.9	U	2.8	U	2.6	U
Acenaphthene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Acenaphthylene	0.28	UJ	0.31	UJ	0.27	UJ	0.28	UJ	0.28	UJ	0.28	UJ	0.29	UJ	0.28	UJ	0.26	UJ
Anthracene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Benzo(a)anthracene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Benzo(a)pyrene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Benzo(b)fluoranthene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Benzo(ghi)perylene	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Benzo(k)fluoranthene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Benzoic Acid	3.6	J	2.6	J	2.3	J	2.8	UJ	3.7	J	2.3	J	2.9	UJ	1.8	J	2.5	UJ
Benzyl Alcohol	2.8	U	3	U	2.7	U	2.8	U	2.8	U	2.8	U	12		2.8	U	0.26	U
Bis(2-Chloroethoxy)Methane	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Bis(2-Chloroethyl)Ether	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Bis(2-Ethylhexyl) Phthalate	0.96		0.61	U	1.6		0.28	U	0.28	U	0.28	U	28	J	0.28	U	0.26	U
Bisphenol A	1.9	J	1.3	NJ	1.3	UJ	2.8	U	1.1	UJ	1.1	UJ	6.2		1.2	NJ	0.52	U
Butylbenzylphthalate	0.57	U	0.61	U	0.53	U	0.28	U	0.56	U	0.56	U	1.7	J	0.56	U	0.52	U
Carbazole	0.57	UJ	0.61	UJ	0.53	UJ	0.55	UJ	0.56	UJ	0.56	UJ	0.58	UJ	0.56	UJ	0.52	UJ
Chrysene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Dibenzo(a,h)anthracene	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Dibenzofuran	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Diethylphthalate	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	5.2	J	0.56	U	0.52	U
Dimethylphthalate	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Di-N-Butylphthalate	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.92	J	0.28	U	0.26	U
Di-N-Octyl Phthalate	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Fluoranthene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Fluorene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Hexachlorobenzene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	UJ	0.28	U	0.26	U
Hexachlorobutadiene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Hexachlorocyclopentadiene	1.1	UJ	1.2	UJ		REJ	1.1	UJ	1.1	UJ	1.1	UJ	1.2	UJ	1.1	UJ	1	UJ

Analyte	CC-AS Effluent		Puy-AS+N Effluent		BITP-EBNR Effluent		BITP-EBNR Effluent (replicate)		BIRWP-EBNR+F Discharge		BIRWP-EBNR+F Discharge (replicate)		MWRWP Discharge		Hayden-AD Effluent		Hayden-WRF Effluent	
Hexachloroethane	1.1	UJ	1.2	UJ	0.27	UJ	1.1	UJ	1.1	UJ	1.1	UJ	1.2	UJ	1.1	UJ	1	UJ
Indeno(1,2,3-cd)pyrene	5.7	U	6.1	U	5.3	U	5.5	U	5.6	U	5.6	U	5.8	U	5.6	U	5.2	U
Isophorone	0.57	U	0.61	U	0.53	U	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Naphthalene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Nitrobenzene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
N-Nitrosodimethylamine	1.1	U	1.2	U	1.1	UJ	1.1	UJ	1.1	UJ	1.1	UJ	1.2	UJ	1.1	U	1	U
N-Nitroso-Di-N-Propylamine	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
N-Nitrosodiphenylamine	0.57	UJ	0.61	UJ	0.53	UJ	0.55	UJ	0.56	UJ	0.56	UJ	0.58	UJ	0.56	UJ	0.52	UJ
Pentachlorophenol	2.8	U	3	U	2.7	UJ	2.8	UJ	2.8	UJ	2.8	UJ	2.9	UJ	2.8	U	2.6	U
Phenanthrene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.12	J	0.28	U	0.26	U
Phenol	1.6		1.2	U	1.1	U	1.1	U	1.1	U	1.1	U	24		0.45	J	0.35	J
Phenol, 4-Nonyl-	0.57	U	0.61	U	0.18	J	0.55	U	0.56	U	0.56	U	0.58	U	0.56	U	0.52	U
Pyrene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Retene	0.28	U	0.31	U	0.27	U	0.28	U	0.28	U	0.28	U	0.29	U	0.28	U	0.26	U
Tri(2-chloroethyl) phosphate	1		1.1		0.88		0.84		0.88		0.84		1.4		1		0.99	
Triethyl citrate	2.9		2.4	J	1.9	J	2.2	J	2.3	J	2.3	J	3.6		2.2	J	2.1	J

Table E-9. Method 8270 semi-volatile organics in biosolids, ug/Kg (dw), ppb.

Analyte	CC-Biosolids		Puy-Biosolids		BITP-Biosolids		BITP-Biosolids (replicate)	
1,2,4-Trichlorobenzene	627	U	890	U	604	U	604	U
1,2-Dichlorobenzene	627	U	890	U	604	U	604	U
1,2-Diphenylhydrazine	627	U	890	U	604	U	604	U
1,3-Dichlorobenzene	627	U	890	U	604	U	604	U
1,4-Dichlorobenzene	565	J	246	J	419	J	481	J
1-Methylnaphthalene	627	U	890	U	604	U	604	U
2,2'-Oxybis[1-chloropropane]	627	UJ	890	UJ	604	UJ	604	UJ
2,4,5-Trichlorophenol	2510	UJ	3560	UJ	2420	UJ	2420	UJ
2,4,6-Trichlorophenol	2510	UJ	3560	UJ	2420	UJ	2420	UJ
2,4-Dichlorophenol	6270	U	8900	U	6040	U	6040	U
2,4-Dimethylphenol	6270	U	8900	U	6040	U	6040	U
2,4-Dinitrophenol		REJ		REJ		REJ		REJ
2,4-Dinitrotoluene	2510	U	3560	U	2420	U	2420	U
2,6-Dinitrotoluene	2510	U	3560	U	2420	U	2420	U
2-Chloronaphthalene	1250	U	1780	U	1210	UJ	1210	U
2-Chlorophenol	2510	U	3560	U	2420	U	2420	U
2-Methylnaphthalene	627	U	890	U	604	U	604	U
2-Methylphenol	6270	U	8900	U	6040	U	6040	U
2-Nitroaniline	12500	U	17800	U	12100	U	12100	U
2-Nitrophenol	1250	U	1780	U	1210	U	1210	U
3-Nitroaniline	2510	U	3560	U	2420	U	2420	U
4,6-Dinitro-2-Methylphenol		REJ		REJ		REJ		REJ
4-Bromophenyl-Phenylether	1250	U	1780	U	1210	U	1210	U
4-Chloro-3-Methylphenol	6270	U	8900	U	6040	U	6040	U
4-Chloroaniline		REJ		REJ		REJ		REJ
4-Chlorophenyl-Phenylether	627	U	890	U	604	U	604	U
4-Methylphenol	6270	U	604	J	609	J	607	J
4-Nitroaniline		REJ		REJ		REJ		REJ
4-Nitrophenol	6270	U	8900	U	6040	U	6040	U
Acenaphthene	627	UJ	890	U	604	U	604	U
Acenaphthylene	627	UJ	890	UJ	604	UJ	604	UJ
Anthracene	627	U	890	U	170	J	168	J
Benzo(a)anthracene	376	J	435	J	450	J	520	J
Benzo(a)pyrene	627	U	371	J	375	J	364	J
Benzo(b)fluoranthene	627	U	618	J	835		865	
Benzo(ghi)perylene	1250	U	1780	U	1210	U	1210	U
Benzo(k)fluoranthene	627	U	890	U	260	J	309	J
Benzoic Acid	6270	UJ	13400	J	8390	J	8280	J
Benzyl Alcohol	6270	U	8900	U	6040	U	604	U
Bis(2-Chloroethoxy)Methane	627	U	890	U	604	U	604	U

Analyte	CC-Biosolids		Puy-Biosolids		BITP-Biosolids		BITP-Biosolids (replicate)	
Bis(2-Chloroethyl)Ether	1250	U	1780	U	1210	U	308	J
Bis(2-Ethylhexyl) Phthalate		E	43900		14800		17100	
Bisphenol A	6850	J	32100	J	55700	J	61700	
Butylbenzylphthalate	631	J	1780	U	1210	U	1210	U
Carbazole	1250	UJ	1780	UJ	1210	UJ	1210	UJ
Chrysene	416	J	404	J	594	J	636	
Dibenzo(a,h)anthracene	1250	U	1780	U	1210	U	1210	U
Dibenzofuran	1250	U	1780	U	1210	U	1210	U
Diethylphthalate	1250	U	1780	U	1210	U	1210	U
Dimethylphthalate	627	UJ	890	U	604	U	604	U
Di-N-Butylphthalate	627	U	890	U	604	U	604	U
Di-N-Octyl Phthalate	1250	U	1780	U	1210	U	1210	U
Fluoranthene	627	U	890	U	604	U	604	U
Fluorene	627	U	890	U	604	U	604	U
Hexachlorobenzene	627	U	890	U	604	U	604	U
Hexachlorobutadiene	627	U	890	U	604	U	604	U
Hexachlorocyclopentadiene	2510	U	3560	U	2420	U	2420	U
Hexachloroethane	2510	U	3560	U	2420	U	2420	U
Indeno(1,2,3-cd)pyrene	2450	J	17800	U	612	J	743	J
Isophorone	1250	U	1780	U	1210	U	1210	U
Naphthalene	550	J	424	J	499	J	598	J
Nitrobenzene	627	U	890	U	604	U	604	U
N-Nitrosodimethylamine	2510	UJ	3560	UJ	2420	UJ	242	UJ
N-Nitroso-Di-N-Propylamine	627	UJ	890	U	604	U	604	U
N-Nitrosodiphenylamine	1250	UJ	1780	UJ	1210	UJ	1210	UJ
Pentachlorophenol	6270	UJ	8900	UJ	6040	UJ	6040	UJ
Phenanthrene	717		553	J	655		766	
Phenol	24200		5890	J	2500		2990	
Phenol, 4-Nonyl-	1250	U	1780	U	1210	U	1210	U
Pyrene	731		842	J	860		1040	
Retene	627	U	890	U	604	U	604	U
Tri(2-chloroethyl) phosphate	1480	J	890	U	974	U	604	U
Triethyl citrate	4800	NJ	6330	J	283	J	302	J

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Appendix F. Glossary, Acronyms, and Abbreviations

Aerobic: A biological process which occurs in the presence of oxygen.

Anaerobic: A biological process which occurs in the absence of oxygen.

Analyte: Parameter. Water quality constituent being measured.

Anoxic: Depleted of oxygen.

Anthropogenic: Human-caused.

Bioaccumulate: Build up in the food chain.

Biosolids: Organic, semi-solid material derived from municipal sewage sludge. It can be beneficially recycled but must meet strict quality standards for pathogens, animal attraction, and pollutant concentrations.

Conductivity: A measure of water's ability to conduct an electrical current. Conductivity is related to the concentration and charge of dissolved ions in water.

Clean Water Act: A federal act passed in 1972 that contains provisions to restore and maintain the quality of the nation's waters.

Effluent: An outflowing of water from a natural body of water or from a man-made structure. For example, the treated outflow from a sewage treatment system.

Grab sample: A discrete sample from a single point in the water column or sediment surface.

Hydraulic retention time (HRT): The theoretical time required to displace the contents of a tank or unit at a given rate of discharge (volume divided by the rate of discharge).

Influent: Water flowing into a natural body of water or man-made structure.

Mean cell residence time (MCRT): The average time that a given unit of cell mass stays in the activated sludge aeration tank. It is usually calculated as the total mixed liquor suspended solids in the aeration tank divided by the combination of solids in the effluent and solids wasted

Method detection limit (MDL): MDL is defined as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero. (Federal Register, October 26, 1984)

National Pollutant Discharge Elimination System (NPDES): National program for issuing, modifying, revoking and reissuing, terminating, monitoring, and enforcing permits, and imposing and enforcing pretreatment requirements under the Clean Water Act. The NPDES program regulates discharges from wastewater treatment plants, large factories, and other facilities that use, process, and discharge water back into lakes, streams, rivers, bays, and oceans.

Nutrient: Substance such as carbon, nitrogen, and phosphorus used by organisms to live and grow. Too many nutrients in the water can promote algal blooms and rob the water of oxygen vital to aquatic organisms.

Parameter: A physical chemical or biological property whose values determine environmental characteristics or behavior. Analyte.

Pathogen: Disease-causing microorganisms such as bacteria, protozoa, viruses.

Personal care products (or toiletries): Products used for personal hygiene or beautification. Personal care includes products as diverse as chapstick, colognes, cotton swabs, deodorant, eye liner, facial tissue, hair clippers, lipstick, lotion, makeup, mouthwash, nail files, pomade, perfumes, personal lubricant, razors, shampoo, shaving cream, skin cream, toilet paper, cleansing pads and wipes, lip gloss, toothbrushes, and toothpaste, to give a few examples.

pH: A measure of the acidity or alkalinity of water. A low pH value (0 to 7) indicates that an acidic condition is present, while a high pH (7 to 14) indicates a basic or alkaline condition. A pH of 7 is considered to be neutral. Since the pH scale is logarithmic, a water sample with a pH of 8 is ten times more basic than one with a pH of 7.

Pharmaceuticals: Medical substances including prescription or over-the-counter drugs, diagnostic agents, nutraceuticals, and excipients. Pharmaceuticals can be used for humans, pets, livestock, or aquaculture.

Pharmaceuticals and personal care products (PPCPs): Refers, in general, to any product used by individuals for personal health or cosmetic reasons or used by agribusiness to enhance growth or health of livestock. PPCPs comprise a diverse collection of thousands of chemical substances, including prescription and over-the-counter therapeutic drugs, fragrances, and cosmetics, as well as veterinary drugs.

Potable use: Water of sufficiently high quality that it can be consumed.

Reconnaissance survey: Sampling survey.

Reporting limit: The RL is the lowest concentration at which an analyte can be detected in a sample and its concentration can be reported with a reasonable degree of accuracy and precision. The published method and laboratory replicate determinations are used to define "reasonable"; the value is a laboratory specified number and may change over time. This value may be the same or higher than the detection limit. When a sample has to be diluted before analysis, either because of matrix problems or to get the instrument response within the linear dynamic range, the RL is raised by a factor corresponding to the dilution factor.

Removal efficiency: A measure of the effectiveness of a process in removing a constituent, such as biological oxygen demand or total suspended solids (TSS). Removal efficiency is calculated by subtracting the effluent value from the influent value and dividing it by the influent value. Multiply the answer by 100 to convert to a percentage.

Semi-volatile organic compound (SVOCs): refers generally to any organic compound that is volatile or semi-volatile (evaporating or vaporizing under normal conditions). This is a very broad set of chemicals. Definitions vary depending on the particular context. The compounds generally include, but are not limited to; fuels, solvents, scents, propellants, drugs, precursors, or pesticides.

Solids retention time (SRT): The average time of retention of suspended solids in a biological waste treatment system, equal to the total weight of suspended solids leaving the system, per unit of time.

Sorption: Sorption refers to the action of both absorption and adsorption taking place simultaneously. As such, it is the effect of gases or liquids being incorporated into a material of a different state *and* adhering to the surface of another molecule.

Total suspended solids: The suspended particulate matter in a water sample as retained by a filter.

Turbidity: A measure of water clarity. High levels of turbidity can have a negative impact on aquatic life.

Watershed: A drainage area or basin in which all land and water areas drain or flow toward a central collector such as a stream, river, or lake at a lower elevation.

Acronyms and Abbreviations

Axys	Axys Analytical Laboratory, Inc.
BC	British Columbia, Canada
BITP	Budd Inlet Treatment Plant
CAS #	Chemical Abstract Service registry number
CFR	Code of Federal Regulations
DMR	Discharge Monitoring Report
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency (also USEPA)
GAC	Granular activated carbon
GC	Gas Chromatography
GC/MS/MS	Gas Chromatography Tandem Mass Spectrometry
GIS	Geographic Information System software
GPS	Global Positioning System
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
K_{ow}	octanol-water partition coefficient
LOTT	Lacey, Olympia, Tumwater, Thurston County Alliance
MBR	membrane bioreactors
MEL	Manchester Environmental Laboratory (Ecology)
MGD	million gallons per day
MQO	measurement quality objectives
MS/MSD	matrix spike/matrix spike duplicate
NPDES	National Pollutant Discharge Elimination System (see Glossary above)
OPR	ongoing precision and recovery
PAC	powdered activated carbon
PAH	polycyclic aromatic hydrocarbon
PPCPs	pharmaceutical and personal care products (see Glossary above)
QA	quality assurance
QC	quality control
RCW	Revised Code of Washington

RO	reverse osmosis
RPD	relative percent difference
RWP	Reclaimed Water Plant
SRT	solids retention time (see Glossary above)
SVOC	semi-volatile organic compound (see Glossary above)
TCEP	tri(2-chloroethyl) phosphate
TOC	total organic carbon
TSS	total suspended solids (see Glossary above)
USGS	U.S. Geological Survey
UV	ultraviolet
WAC	Washington Administrative Code
WRF	Wastewater Research Facility
WWTP	wastewater treatment plant

Treatment codes

AD	Secondary effluent from aeration ditch treatment
AS	Secondary effluent from activated sludge treatment
AS+N	Final effluent from activated sludge treatment operated to provide nitrification
CA+F	Chemical addition and filtration applied to secondary effluent
EBNR	Secondary effluent with enhanced biological nutrient removal
EBNR+F	Enhanced biological nutrient removal and tertiary filtration
EBNR+MF	Enhanced biological nutrient removal and tertiary membrane filtration

Units of measurement

dw	dry weight
g	gram
kg	kilogram
mg/Kg	milligrams per kilogram (parts per million)
mg/L	milligrams per liter (parts per million)
ng/g	nanograms per gram (parts per billion)
ng/Kg	nanograms per kilogram (parts per trillion)
ng/L	nanograms per liter (parts per trillion)
µg/L	micrograms per liter (parts per billion)
ppb	parts per billion
pptr	parts per trillion
µg/g	micrograms per gram (parts per million)
ug/Kg	micrograms per kilogram (parts per billion)
°C	degrees centigrade
°F	degrees fahrenheit