Quality Assurance Project Plan

Pharmaceuticals and Personal Care Products in Wastewater Treatment Systems

by

Brandi Lubliner, Engineer In-Training
Melanie Redding, Licensed Geologist/Hydrogeologist
Steve Golding, Professional Engineer

Environmental Assessment Program
Washington State Department of Ecology
Olympia, Washington 98504-7710

and

Dave Ragsdale, Engineer
Office of Water and Watersheds
U.S. Environmental Protection Agency
Olympia, Washington 98503

August 2008

Publication Number 08-03-112

DEPARTMENT OF ECOLOGY
State of Washington

Publication and Contact Information

This plan is available on the Department of Ecology's website at www.ecy.wa.gov/biblio/0803112.html.

Data for this project will be available on Ecology's Environmental Information Management (EIM) website at www.ecy.wa.gov/eim/index.htm. Search User Study ID, BRWA0005.

Ecology's Project Tracker Code for this study is 09-219.

303(d) Listings Addressed in this Study: None

Waterbody Number: Puyallup River (WA-10-1070); Chambers Creek (WA-12-1110); Dalco Passage/Poverty Bay (WA-PS-0280); and Inner Budd Inlet (WA-13-0030.

LLID Number: Puyallup River (1224252472685); Chambers Creek (1225831471872); and South Puget Sound (1224819475188).

For more information contact:

Carol Norsen
Environmental Assessment Program
P.O. Box 47600
Olympia, WA 98504-7600
E-mail: CNOR461@ecy.wa.gov
Phone: 360-407-7486

Washington State Department of Ecology - www.ecy.wa.gov/

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

One of the co-authors of this report is a licensed hydrogeologist. A signed and stamped copy of the report is available upon request.

Quality Assurance Project Plan

Pharmaceuticals and Personal Care Products in Wastewater Treatment Systems

August 2008

Approved by:

Signature:	Date: August 2008
Dave Ragsdale, Client Field Sampling, U.S. Environmental Protection Agency	
Signature:	Date: August 2008
Brandi Lubliner, Author, Project Manager, and EIM Data Engineer, EAP	
Signature:	Date: August 2008
Melanie Redding, Co-Author, EAP	
Signature:	Date: August 2008
Steve Golding, Field Sampling, EAP	
Signature:	Date: August 2008
Dale Norton, Supervisor, Toxics Studies Unit, EAP	
Signature:	Date: August 2008
Martha Maggi, Supervisor, Groundwater/Forests & Fish Unit, EAP	
Signature:	Date: August 2008
Will Kendra, Manager, Statewide Coordination Section, EAP	
Signature:	Date: August 2008
Stuart Magoon, Director, Manchester Environmental Laboratory, EAP	
Signature:	Date: August 2008
Bill Kammin, Ecology Quality Assurance Officer	

Signatures are not available on the Internet version
EAP - Environmental Assessment Program
EIM - Environmental Information Management system

Table of Contents

Abstract

This Quality Assurance Project Plan is provided for monitoring pharmaceuticals and personal care products (PPCPs) and nutrients in the influent, effluent, and biosolids from four municipal wastewater treatment plants. The four selected facilities offer different types of wastewater treatment methods as well as varying levels of treatment.

The purpose of this effort is to (1) characterize the concentrations of the contaminants entering the wastewater treatment systems, (2) assess the extent to which the contamination is treated in each facility, and (3) compare contaminant removal between wastewater treatment technologies.

This work is funded through a grant from the U.S. Environmental Protection Agency, the Puget Sound Partnership, and the Washington State Department of Ecology (Ecology).

Each study conducted by Ecology must have an approved Quality Assurance Project Plan. The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completion of the study, a final report describing the results will be posted to the Internet.

Introduction

Background

Pharmaceuticals and personal care products (PPCPs) are used in everyday life. A review of the literature indicates that chemicals in these products can be found in surface water, groundwater, soils, fish, birds, livestock, pets, and humans including newborns. This is likely the result of their 1) stable chemical nature; 2) frequent use in medications, cosmetics, cleaners, and aerosols; and 3) presence in toilets, showers, and laundry. PPCPs eventually make their way into our domestic wastewater systems.

Pharmaceuticals (prescription drugs) are important medical tools that are essential to many people. The general population is becoming increasingly medicated. In 2004, almost half of the United States population was taking at least one prescription drug. Approximately 62% of doctor visits result in at least one prescription being written (National Institute on Drug Abuse, 2005). The Washington State Department of Ecology (Ecology) estimated that the average person in the U.S. uses 10.8 prescriptions per year, and in Washington State, the average person uses 8.5 prescriptions per year (PH:ARM Pilot Team, 2007).

Humans typically excrete 50% to 90% of the active ingredients in pharmaceuticals that are ingested, either as un-metabolized parent compounds or their metabolites (Holtz, 2006). PPCP compounds enter the wastewater treatment system. The system can be a municipal wastewater treatment facility, an on-site sewage system, or a reclaimed water treatment facility. Wastewater effluents are discharged to streams, lakes, estuaries, and groundwater where some PPCPs impact water quality and aquatic life. This issue is an emerging environmental concern.

It is estimated that once sold, 25 to 33% of the unused pharmaceuticals are disposed of by consumers either to a landfill or wastewater treatment plant (WWTP). This rate, which was extrapolated from data generated in Germany and Australia, is supported by a consumer survey conducted in King County in 2005. In King County, 36% of residents stated that they disposed of pharmaceuticals in the trash and 29% disposed of pharmaceuticals in the sink or toilet (PH:ARM Pilot Team, 2007).

Preventing unintended exposure to PPCPs in the environment is a challenging problem. Pharmaceuticals will always be used by humans. The major pathway for PPCPs to enter the environment is through discharges of municipal effluent, but the extent to which PPCPs are removed by treatment processes is not well understood. Proper disposal such as a product take-back-program is one option to reducing pharmaceuticals in the environment.

Although the concentrations of the PPCPs found in the environment are typically less than therapeutic doses, effects of constant low-level exposure to aquatic organisms are only beginning to be researched. Recent studies have found that PPCPs can cause feminization in fish (Orlando et al., 2004) and alligators (Guillette et al., 2000). PPCPs can also affect the behavior and migratory patterns of salmon (Lower Columbia River Estuary Partnership, 2007). The pharmaceutical diclofenac was found to be the direct cause of near extinction of the vulture population in India (Oaks et al., 2004).

Wastewater Systems

In Washington State, human waste is typically treated by on-site sewage systems, or WWTPs that may employ up to three levels of treatment.

On-Site Sewage Systems – Septic Tanks

On-site sewage systems are regulated by county health departments, and data on the number of systems in Washington State are not readily available. Nationwide, approximately 25% of households use on-site sewage systems to treat their domestic wastewater (Swartz et al., 2006). These systems serve over 22 million homes and businesses, discharging approximately 15 billion liters per day to the subsurface. This makes on-site sewage systems one of the most prevalent sources of groundwater contaminants (Conn et al., 2006).

Wastewater Treatment Plants

Primary and Secondary Treatment

There are 321 municipal WWTPs in Washington State, all of which provide primary and secondary treatment (Jones, 2008). The typical municipal WWTP collects raw sewage and other liquid wastes (e.g., toilets, showers, laundry, dishes, and food washing) from many sources. These sources include homes, schools, hospitals, nursing homes, rehabilitation centers, commerce, and industry, to name a few.

Primary treatment is mechanical removal of grit, garbage, rocks, fats, oils, and grease followed by settling in a large, round tank known as a primary clarifier. Secondary treatment is the biological reduction of organic materials such as fecal matter, food waste, soap, and detergent. This is done by bacteria and protozoa consuming the soluble organic nutrients in an aerobic environment. Aerobic treatment is followed by a secondary clarifier to settle out sludge.

Sludge is a byproduct of all processed wastewater. Generally, wastewater sludge is processed by a digester, a tank that breaks down the sludge. Digesters are of two types, aerobic and anaerobic. Bacteria in an anaerobic digester create heat that reduces the concentration of disease-causing organisms. Sludge can be dewatered and sold as a biosolid fertilizer, to reduce the amount taken to the landfill. Approximately 50% of all the biosolids nationally are land applied, which equates to less than 1% of the nation's agricultural lands (Kinney et al., 2006b).

Tertiary Treatment and Reclaimed Water

WWTPs that operate an additional level of treatment, called tertiary treatment, to remove nitrogen, phosphorus, or unsettled solids, will often use this reclaimed water for irrigation. Most tertiary treatment technologies follow the primary and secondary technologies at a standard WWTP. However, there are a few stand-alone tertiary treatment facilities that produce reclaimed clean water. There are 21 WWTPs in Washington State producing reclaimed water.

Not all reclaimed water treatment facilities employ the same type of treatment, nor are they all federally regulated facilities. Some reclaimed wastewater plants are regulated by state discharge permits only. Redundancy requirements are prescribed based on the beneficial use of the water and the potential for human contact. In the United States, treated wastewater is not applied to crops directly consumed by people. It is still unknown whether the biosolids sludge will transfer organic contaminants to crops.

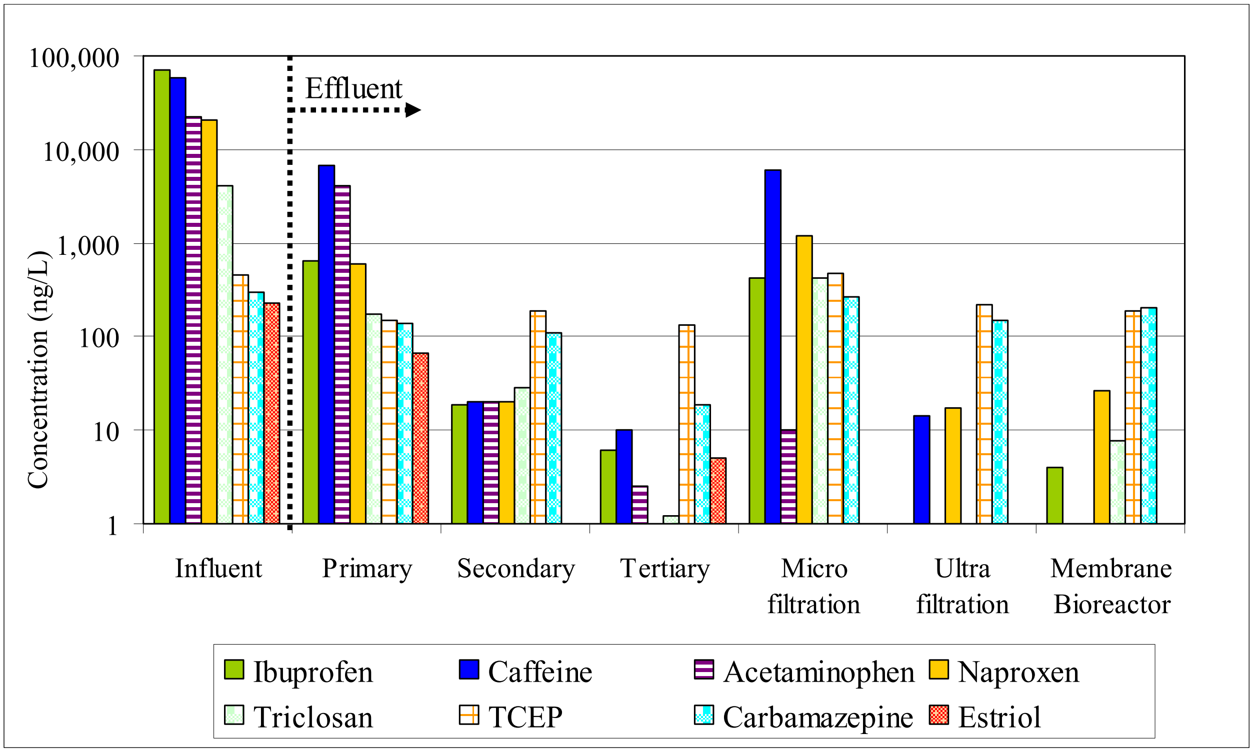
The traditional processes commonly used to treat municipal wastewater effluents do not effectively reduce all PPCPs. However, recent research documented that enhanced biological nutrient removal can reduce a significant portion of the PPCPs contained in municipal effluent (Kimura et al., 2005).

Previous Studies

PPCP treatment is dependent on the individual compound chemistry. Some treatment processes are efficient in removing certain chemicals but ineffective at treating others.

Secondary treatment provides a biologically oxidative environment where compounds susceptible to microbial degradation are treated (Khan et al., 2004). The literature suggests that activated sludge (a common secondary treatment method) is effective for removing some PPCPs but is not effective for all. Ternes (1998) found 80% of the 38 monitored PPCP compounds were found in at least one effluent sample.

Figure 1 illustrates the influent and effluent concentrations of a few PPCP compounds that have been tested in the literature. This figure is a compilation of several research studies.



Adapted from Snyder et al. (2006); Drury et al. (2006); Ternes et al. (2002); and Heberer et al. (2004).

Figure 1. Values for Influent and Treatment of Eight PPCPs by Different Treatment Technologies. (Redding, unpublished).

Several tertiary treatment processes are reported to be excellent at removing PPCPs from the effluent. Typical removal processes include adsorption, filtration, volatilization, photo-degradation, biodegradation, chemical alteration, and plant or animal utilization.

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

Table 1. Model-Predicted Removal Rates of 50 Pharmaceuticals (Khan and Ongerth, 2004).

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

This model assumes that wastewater will undergo primary settling, secondary aeration, and clarification in an activated sludge sewage treatment plant. They determined the majority of pharmaceutical removal will occur in the aeration tank. Additionally it was noted that pharmaceuticals were removed more efficiently during secondary clarification by biodegradation, than during primary settling.

Kasprzyk-Hordern et al. (2006) evaluated the gross removal efficiency of PPCPs by WWTPs. They used a new analytical technique to detect low concentrations in wastewater: ultra-high performance liquid chromatography-positive/negative electrospray tandem mass spectrometry. Fifty-three PPCPs were identified in influent and effluent of the WWTPs. PPCP categories and a gross description of removal through the WWTPs are presented in Table 2.

Table 2. Percent Removal of PPCPs by Wastewater Treatment Plants (Kasprzyk-Hordern et al., 2006).

Pharmaceuticals	
Antibacterial drugs	little reduction
Anti-inflammatory/analgesics	roughly 50% reduction
Antiepileptic drugs	no removal
Beta-blockers	roughly 75% reduction
Lipid-regulating drugs	roughly 50% reduction
H2-receptor antagonists	no removal
Personal care products	
Sunscreen agents	reduction by factor of 10*
Preservatives	reduction by greater than factor of 10
Disinfectants/antiseptics	varying from factors of 2 to 20

* 4-Benzophenone was the exception, with only 25% reduction.

It is unclear whether the PPCPs are actually broken down or are simply settled out in the biosolids. The concern is that the sludge might contain active untreated compounds which once land applied are available to be transported into surface or groundwater. For example, nonylphenol is believed to transfer from water to sludge via the treatment process.

Project Description

The traditional processes commonly used to treat municipal wastewater do not effectively reduce all PPCPs. However, recent research has documented that enhanced biological nutrient removal is effective at reducing a significant portion of the PPCPs contained in effluent from municipal WWTPs. The goals of this project are to characterize the concentrations of PPCPs in influent, effluent, and biosolids and to evaluate what PPCPs are removed by the different wastewater treatment processes. The effects from enhanced nutrient removal on PPCP concentrations will be evaluated.

This project will evaluate concentrations of PPCPs in influent, effluent, and biosolids at four treatment facilities. Nitrogen, phosphorus, and total suspended solids concentrations for the influent and effluent of all the WWTPs will be studied. PPCP concentrations from the discharges of conventional secondary treatment and tertiary treatment WWTPs will be compared.

Influent and effluent samples will be collected from two WWTPs that provide secondary treatment and two WWTPs that provide biological nutrient removal (tertiary treatment). The Chambers Creek WWTP in Pierce County and the Puyallup WWTP are the secondary treatment plants for this study. The only WWTP discharging into the Puget Sound watershed that currently provides treatment to remove nutrients (nitrogen) is the LOTT Alliance, Budd Inlet Treatment Plant in Olympia. A second nutrient (phosphorus) removal WWTP was selected to evaluate PPCP removal by chemical addition and filtration treatment. This plant is in Hayden, Idaho. Figure 2 illustrates the general location of the four WWTPs in this study.

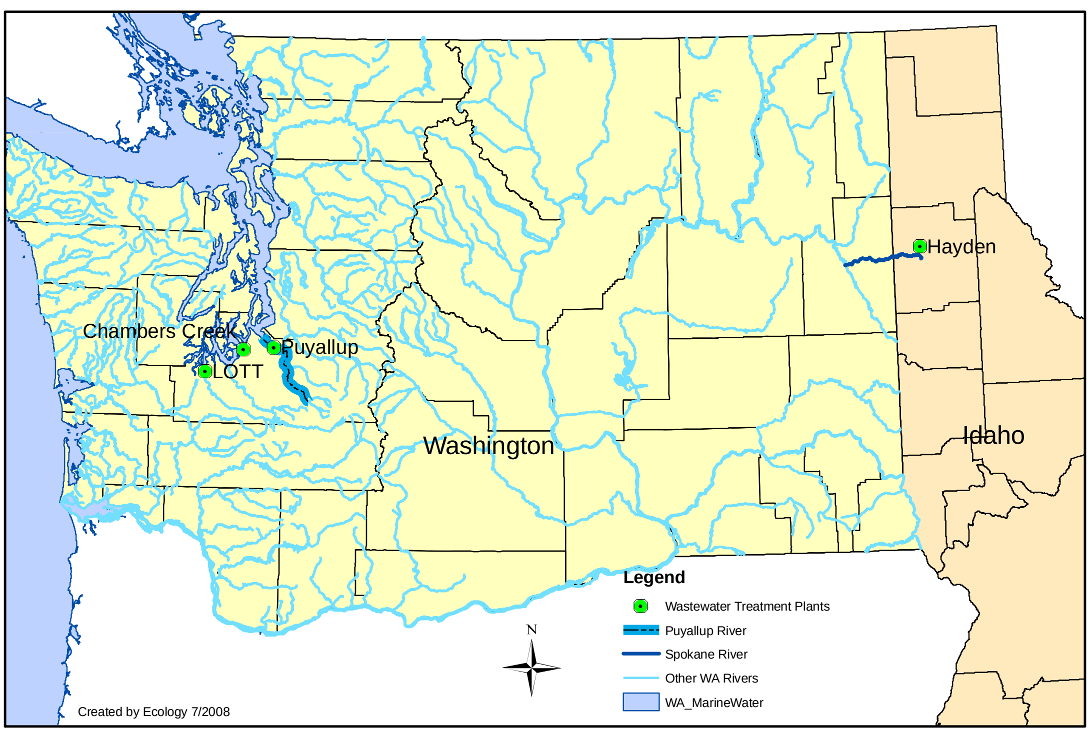


Figure 2. Locations of WWTP Study Sites.

Biosolids will be collected from the three Puget Sound WWTPs to measure PPCPs captured in the solids generated by the treatment process.

Pierce County Regional Chambers Creek WWTP

Located in the city of University Place, the Chambers Creek Regional WWTP serves more than 65,000 households and 2,000 businesses in the cities of DuPont, Lakewood, Tacoma, University Place, Steilacoom, and unincorporated areas such as Parkland, Spanaway, Frederickson, and South Hill. Average dry weather flow is 13.85 million gallons per day (MGD). Average monthly flow is 19.83 MGD.

The facility is an activated sludge plant. Step screens and grit tanks are followed by primary clarifiers, five aeration basins, and secondary clarifiers. Ultra-violet (UV) disinfection is used. Following anaerobic treatment of sludge, a filter press dewaters the solids, producing biosolids that are sent to the fertilizing manufacturing facility where further processing produces a Class A pelletized fertilizer for residential and commercial use.

City of Puyallup WWTP

The City of Puyallup WWTP operates an activated sludge plant which has a maximum monthly flow of 5.61 MGD. The sewer system is separated, with no combined stormwater. The National Pollutant Discharge Elimination System (NPDES) permit provides limits for ammonia.

After grit removal, the inflow is routed to two primary clarifiers, then to two aeration basins. Zones within the aeration basins achieve nitrification of ammonia (NH_3) to nitrate (NO_3^-) followed by denitrification of nitrate to release nitrogen gas to the atmosphere. Secondary clarifiers settle solids and release effluent for UV disinfection.

Settled sludge from the clarifiers is centrifuged to remove water, and then anaerobically digested. This is followed by further removal of water with a belt filter press, producing biosolids that are hauled for application on agricultural fields.

Lacey-Olympia-Tumwater-Thurston County (LOTT) WWTP

The LOTT Budd Inlet WWTP has a design capacity of 28 MGD. Effluent is principally discharged to Budd Inlet in South Puget Sound. In general, the treatment train is as follows; grit removal, primary clarification, anoxic zone 1, aeration, anoxic zone 2, and then final aeration. The anoxic zones within the aeration basins achieve nitrification of ammonia to nitrate followed by denitrification of nitrates to release nitrogen gas to the atmosphere. Secondary clarifiers settle solids and release effluent for UV disinfection and release of final effluent.

There are loops for return flows to the first and second anoxic zones to enhance nitrification and denitrification. The supernatant from the primary clarifier is routed through the first anoxic zone. The influent ammonia in the supernatant passes through the first anoxic zone to the first aeration basin where nitrification takes place. A splitter box after the first aeration basin returns 4/5 of the aerated supernatant back to the first anoxic zone for denitrification (nitrogen removal): $NO_3^- > N_2$ (atmospheric nitrogen). The remaining 1/5 from the splitter box is routed to the second anoxic zone where it is denitrified. Food for the bacteria in the second anoxic zone is added in the form of a proprietary product containing sugars.

The flow through the second anoxic zone consists of all plant flow plus return activated sludge. This flow is routed to the final aeration basin for polishing. The flow then enters a secondary clarifier. Discharge from the clarifier is routed to UV disinfection, leaving the plant as final effluent.

A portion of the LOTT final effluent is tertiary treated to produce reclaimed water and is used for irrigation of public land in Olympia. Tertiary treated effluent is produced by running effluent through a single-stage, continuous back-washing sand filter. Poly-aluminum chloride is added to aid filtration effectiveness. Filtration is followed by chlorination before the reclaimed water is used for irrigation.

Removed solids are routed to dissolved flotation thickeners, anaerobic digesters, and then dewatered through a centrifuge for land application.

Hayden Area Regional WWTP

The Hayden WWTP, operated by the Hayden Area Regional Sewer Board, serves the greater Hayden, Idaho area. Permitted plant flow is 1.6 MGD. All of the tertiary treated effluent is used for silviculture irrigation during the summer. During other times of the year, the effluent is discharged into the Spokane River.

Treatment at the Hayden WWTP consists of screening and grit removal, oxidation ditches, secondary clarification, and chlorine disinfection. Sludge from the process is aerobically digested and dewatered by a belt filter press. Plant processes include denitrification in non-summer months. The plant facilitates tertiary treatment, removing phosphorus with the "Blue Pro" proprietary process. This consists of sand filtration through sand coated with hydrous ferric oxide.

Influent concentrations are typically 7 to 9 mg/L phosphorus. Final effluent from the secondary treatment process is typically about 4 mg/L. A long-term, steady-state study was conducted from December 2005 through February 2006 to evaluate the tertiary filtration effluent concentrations. The monthly averages for effluent phosphorus concentrations during the study were:

- 0.036 mg/L in December (second stage filtration not optimized)
- 0.009 mg/L in January
- 0.016 mg/L in February

Table 3 provides a summary of the four WWTPs selected for this study.

Table 3. Summary of Wastewater Treatment Plants Selected for Study.

WWTP	Treatment	Location	Receiving Water
LOTT	Tertiary – Biological nitrification/denitrification for enhanced nitrogen removal	Olympia, WA	Marine – Budd Inlet in Puget Sound
Chambers Creek	Secondary – Activated sludge	University Place, WA	Marine – Puget Sound
Puyallup	Secondary – Activated sludge with denitrification	Puyallup, WA	Freshwater – Puyallup River which flows into Puget Sound
Hayden	Tertiary – Some denitrification, proprietary sand filtration, and advanced oxidation for phosphorus removal	Hayden, ID	Freshwater – Spokane River

Organization, Schedule, and Costs

Organization

EPA's Region 10 Office of Water and Watersheds, and Ecology's Environmental Assessment Program, will conduct this project (Table 4).

Table 4. Project Organization and Responsibilities.

Staff (EAP unless stated otherwise)	Title	Responsibilities
Brandi Lubliner Toxic Studies Unit Statewide Coordination Section (360) 407-7140	Project Lead	Writes the QAPP, manages the contract with labs, conducts field sampling at LOTT, enters data into EIM, and oversees writing of the report.
Steven Golding Toxic Studies Unit Statewide Coordination Section (360) 407-6701	Field Sampling	Helps with the QAPP, conducts field sampling at Chambers Creek WWTP, and writes methods section of the report.
Dave Ragsdale EPA, Region 10 (360) 407-6589	EPA Client, Field Sampling	Procures grant funding, reviews the QAPP, coordinates field sampling at Hayden and Puyallup, coordinates QA review of data, and writes the discussion section of the report.
Melanie Redding Groundwater/Forest and Fish Unit Statewide Coordination Section (360) 407-6524	EAP Co-Author	Helps with the QAPP introduction. Assists with the discussion section of the draft and final report.
Dale Norton Toxic Studies Unit Statewide Coordination Section (360) 407-6765	Unit Supervisor	Approves the budget. Reviews and approves the QAPP.
Martha Maggi Groundwater/Forest and Fish Unit Statewide Coordination Section (360) 407-6453	Unit Supervisor	Reviews and approves the QAPP. Reviews the draft report.
Will Kendra Statewide Coordination Section (360) 407-6698	Section Manager	Reviews project and budget. Reviews and approves the QAPP.
Stuart Magoon Manchester Environmental Laboratory (360) 871-8801	Director	Reviews and approves the QAPP.
William R. Kammin Quality Assurance Officer (360) 407-6964	Ecology Quality Assurance Officer	Reviews and approves the QAPP.
Cynthia Tomey Quality Assurance Officer AXYS Analytical Services Ltd. (250) 655-5811	Project Manager	Reviews the QAPP. Manages analytical services at AXYS.

EAP – Environmental Assessment Program
QAPP – Quality Assurance Project Plan
EIM – Environmental Information Management system

Schedule

Table 5: Anticipated Schedule.

Project Schedule	
Field Work	August 19 or 26, 2008
Laboratory Analyses Completed	December 2008
Environmental Information System (EIM) Data Set	
Data Engineer	Brandi Lubliner
EIM User Study ID	BRWA0005
EIM Study Name	Pharmaceuticals and Personal Care Products in Wastewater Treatment Systems
EIM Completion Due	June 2009
Final Report	
Author Lead	Brandi Lubliner
Draft to Supervisor	March 2009
Draft to Client/Peer	April 2009
External Draft	April 2009
Report Final Due (original)	June 2009

Costs

The analytical cost for this project is estimated to be $64,918 (Table 6). The cost estimate includes MEL's 50% discount for samples analyzed at MEL. The contracted laboratory analyses are not held to MEL's 25% surcharge due to the project lead's direct contracting with AXYS. Shipping costs are estimated to be $5,000.

Table 6. Laboratory Cost Estimate for Analyzing PPCPs in WWTPs Samples.

Analytical Method	Number of Samples[a]	Cost per Sample	Total
AXYS Analytical Laboratory Ltd.			
Biosolids by EPA Method 1698	4	$ 1,300	$ 5,200
Biosolids by EPA Method 1694	4	$ 1,425	$ 5,700
Wastewater by EPA Method 1698	16	$ 1,250	$ 20,000
Wastewater by EPA Method 1694	16	$ 1,300	$ 20,800
		Subtotal	$ 51,700
Manchester Environmental Laboratory			
Analyze all biosolids and water samples by EPA Method 8270	20	$ 600	$ 12,000
Nutrients (NH3, NO3-, NO2-, TPN, Ortho-PO43-, TP)	14	$ 76	$ 1,064
Total suspended solids	14	$ 11	$ 154
		Subtotal	$ 13,218
		Total	$ 64,918

[a] Includes blank, duplicates, and matrix quality control samples.
TP – Total phosphorus

Experimental Design

This project will provide information about the occurrence and removal of PPCP pollutants in municipal wastewater. The Puget Sound Partnership funded half of this study; therefore, WWTPs that discharge directly or indirectly to the Puget Sound were selected as the focus. Three of the four plants were chosen based on their location on or near Puget Sound.

The only tertiary WWTP discharging into the Puget Sound watershed is the LOTT Alliance, Budd Inlet Treatment Plant. To provide a reference to compare these results, the same sampling will be conducted at two secondary WWTPs located on or near Puget Sound: Chambers Creek and Puyallup.

The Hayden WWTP in Idaho was chosen to evaluate the effects of phosphorus removal by chemical addition and filtration treatment techniques. Many WWTPs proximal to Puget Sound discharge into freshwaters where phosphorus removal and anticipated reduction in PPCPs could help protect the freshwater, and ultimately the marine waters of Puget Sound, from PPCPs.

Specific collection points for influent, effluent, and biosolids samples will be determined in consultation with the WWTP operators, Ecology, and EPA. Figure 3 illustrates the typical flow process for a WWTP.

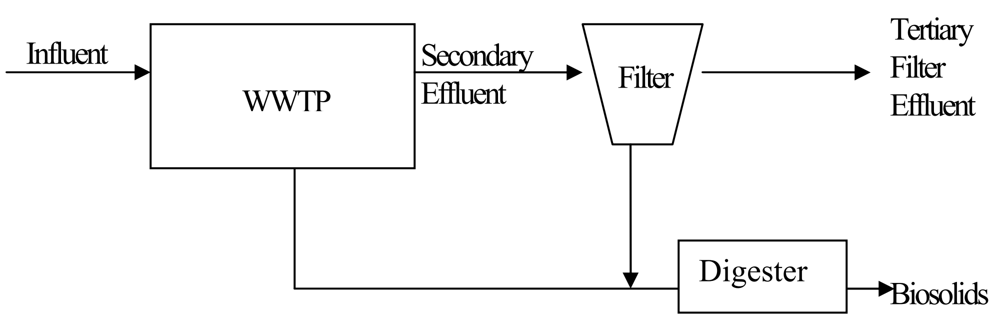


Figure 3. Wastewater Treatment Plant Sampling Schematic for Secondary and Tertiary Systems

A single sampling event is planned for August 2008. Influent, effluent, and tertiary filter effluent samples will be manually composited from three grabs taken over an eight-hour period. The composites will be taken by hand using glass jars cleaned to EPA quality assurance/quality control (QA/QC) specifications (EPA, 1990). Three grabs of 1/3 liter each will be used to fill a 1-liter jar for each analysis over the 8-hour timeframe. The grabs will be hand collected to avoid contamination that could occur with an auto-sampler.

Biosolids samples will be collected all at one time by allowing the belt filter to pour the sample into 8-oz. glass jars with Teflon lid liners, cleaned to EPA QA/QC specifications (EPA, 1990).

All samples will be chilled immediately in the field to 4°C and transported on ice in a cooler. Biosolids samples will be frozen in a -20°C freezer overnight prior to being sent on ice in coolers to the respective analytical laboratories. Influent and effluent flow data will be obtained from WWTP records.

Field personnel will wear powder-free nitrile gloves at all times during sample collection, and they will follow standard health and safety procedures. Water and biosolids samples will be held in a secure cooler for transport to Ecology's MEL or AXYS by Fed-Ex. Chain of custody will be maintained.

Table 7 shows the number of samples to be analyzed.

Table 7. Number of PPCP Field Samples per Wastewater Treatment Plant.

Wastewater Plant	Influent	Secondary Effluent	Tertiary Effluent	Biosolids
LOTT	2 [a]	2 [a]	2 [a]	2 [a]
Chambers Creek	1	1	NA	1
Puyallup	1	1	NA	1
Hayden	1	1	1	NA

[a] Includes sample and sample duplicate
NA –Not applicable

Measurement Procedures

Low-level analyses for PPCPs and steroids/hormones will follow EPA Method 1694 and 1698, approved by EPA in December 2007. Base/neutral and acid compounds with estrogenic properties will also be measured using EPA Method 625/SW-846 Method 8270. These methods were chosen because they provide low reporting limits with the highest degree of quality assurance.

AXYS Analytical Services Ltd. (AXYS) will analyze PPCPs and steroids/hormones. EPA and AXYS co-developed Methods 1694 and 1698:

- **EPA Method 1694**: *Pharmaceuticals and Personal Care Products in Water Soil, Sediment and Biosolids by HPLC/MS/MS.* This is a high performance liquid chromatography combined with tandem mass spectrometry (HPLC/MS/MS) using isotope dilution and internal standard quantitation techniques.

- **EPA Method 1698**: *Steroids and Hormones in Water, Soil, Sediment, and Biosolids by HRGC/HRMS.* This method uses an isotope dilution and internal standard high resolution gas chromatograph combined with high resolution mass spectrometry (HRGC/HRMS).

Other researchers have shown low detection limits can be achieved for a wide range of target compounds (Spongberg and Witter, 2008; Kasprzyk-Hordern et al., 2006).

During development of Methods 1694 and 1698, EPA and AXYS assessed five wastewater plant effluents for laboratory performance. The PPCP concentrations measured in these samples can be found in Table 14 of Method 1694 and Table 7 of Method 1698 (EPA 2007a, b).

Tables A-1 and A-2 in Appendix A list the PPCPs that will be evaluated in this project.

Manchester Environmental Laboratory will analyze the base neutral and acid (BNAs) organics by EPA Method 625/SW-846 Method 8270.

- **EPA Method 8270**: This list of compounds includes many chemicals that are not considered pharmaceuticals or personal care products. However, through this method, MEL will be able to analyze for PPCPs that are of high interest to the Puget Sound Partnership. These are PPCP compounds such as:

bisphenol A	multiple phthalates	Tri(2-chloroethyl)
4-nonylphenol	ethyl citrate	phosphate (TCEP)

Table A-3 lists the BNAs that will be analyzed by EPA Method 625/SW-846 Method 8270.

The anticipated concentrations for Method 8270 BNAs are unknown. 4-nonylphenol has been analyzed at MEL before in sediment samples. The water matrix will be new for MEL. Bisphenol A is a new compound for MEL. Method development costs are included in the costs per sample.

MEL will analyze for nutrients and total suspended solids (TSS). These conventional parameters are essential for this project to understand the WWTP treatment efficiency, and they will serve as a basis for comparing the PPCP treatment efficiencies. Sample containers, preservation, and holding times are shown in Table 8.

Table 8. Methods, Containers, Preservation, and Holding Times for PPCP Samples.

Method or Parameter	Matrix	Container[a]	Preservation[b]	Holding Time[b]
1694: PPCPs	Wastewater	1 liter amber glass, Teflon lid	Cool to 4°C	2-7 days
	Biosolids	8 oz. glass, Teflon lid	Freeze	2-7 days
1698: Steroids/ Hormones	Wastewater	1 liter amber glass, Teflon lid	Cool to 4°C	2-7 days
	Biosolids	8 oz. glass, Teflon lid	Freeze	2-7 days
8270: BNAs	Wastewater	1 liter amber glass, Teflon lid	Cool to 4°C	7 days
	Biosolids	8 oz. glass, Teflon lid	Cool to 4°C	7 days
SM 4500NH3H: Ammonia	Wastewater	125 mL polyethylene pre-acidified	Cool to 4°C, pre-acidified with H_2S0_4	28 days
SM4500NO3I: Nitrate +Nitrite				
SM4500PI: Total Phosphorus				
SM4500NO3B: Total Persulfate Nitrogen				
Orthophosphate	Wastewater	125 mL amber wide-mouth polyethylene	Cool to 4°C, filter through 0.45um pore	48 hours
% Solids	Biosolids	4 oz. Glass, Teflon lid	Cool to 4°C	7 days

[a] Sample containers will be provided by AXYS Analytical Services Ltd for Methods 1694 and 1698 and by Manchester Environmental Laboratory for Method 8270.

[b] AXYS and EPA have not conducted formal preservation nor hold-times studies for Methods 1694 and 1698. These are the recommendations given by AXYS on June 30, 2008.

Quality Objectives

Quality objectives for this project are to obtain high quality data so that uncertainties are minimized and results are comparable to other studies using these methods. These objectives will be achieved through careful attention to the sampling, measurement, and quality control (QC) procedures described in this plan.

The lowest concentrations of interest shown in Table 9 are the "practical quantitation limits" attainable with these methods. Ranges are presented due to the large number of chemicals analyzed. Additionally, there are currently no criteria or guidelines in existence to compare resulting data to. Ecology understands that the target concentrations in each sample are unknown and the interferences within each sample may cause analytical problems.

Table 9. Analytical Methods and Practical Quantitation Limits.

Method or Parameter	Matrix	Field Samples [a]	Practical Quantitation Limit
1694: PPCPs	Wastewater	16	2-10 ng/L
	Biosolids	4	0.1-100 µg/Kg
1698: Steroids/Hormones	Wastewater	16	2-10 ng/L
	Biosolids	4	0.1-100 µg/Kg
8270: BNAs	Wastewater	14	2-10 ng/L
	Biosolids	4	0.1-100 µg/Kg
Ammonia	Wastewater	14	10 µg/L
Nitrate + Nitrite	Wastewater	14	10 µg/L
Total Persulfate Nitrogen	Wastewater	14	25 µg/L
Orthophosphate	Wastewater	14	3 µg/L
Total Phosphorus	Wastewater	14	1 µg/L
Total Suspended Solids	Wastewater	14	1 mg/L
% Solids	Biosolids	4	% Wet Weight

[a] Including field duplicates and field blanks.

As part of the method development, measurement quality objectives were published (EPA 2007a, b) and are shown in Table 10. These objectives are used by the laboratory to evaluate performance of the staff, instrumentation, and QC procedures. The values in Table 10 are the method specifications from clean reference matrices (i.e., water or sand). Actual samples of influent, effluent, and biosolids may not meet these objectives for all analytes due to the matrix interferences present in these samples.

All three of these methods are "performance based." This means that AXYS and MEL may modify the method to improve performance (e.g., to overcome interferences or improve the accuracy or precision of the results), provided that they meet all performance requirements in the published method. High concentrations or interferences may cause the method to perform poorly which could result in higher detection limits. In this case the analytical laboratory will dilute the sample and re-run it to find an acceptable signal within the range of the instrument's calibrations.

Table 10. Measurement Quality Objectives for the Three Methods to Detect PPCPs.

EPA Method	Matrix	Initial Precision and Recovery (%)	Continuing Calibration (%)	Laboratory Control Samples (%)	Labeled Compound Recovery (%)
1694[a]: PPCPs	Wastewater	6-180	70-130	5 - 200	5 - 200
	Biosolids	6-180	70-130	5 - 200	5 - 200
1698[b]: Steroids/ Hormones	Wastewater	6-180	70-130	5 - 200	5 - 200
	Biosolids	6-180	70-130	5 - 200	5 - 200
8270[c]: BNAs	Wastewater	60-140	80-120	50 - 150	20-150 [d]
	Biosolids	60-140	80-120	50 - 150	20-150 [d]

[a] Overall range of all the analytes of Method 1694. See Table 12 of EPA Method 1694 for the range of each individual analyte. Note that these performance statistics are based on clean matrices.

[b] Overall range of all the analytes of Method 1698. See Table 5 of EPA Method 1698 for the range of each individual analyte. Note that these performance statistics are based on clean matrices.

[c] Overall range of all the analytes of Method 8270. Note that these performance statistics are based on clean matrices.

[d] Surrogates are used, not labeled compounds.

Initial Precision and Recovery of internal standards refers to a reference matrix spiked with labeled compounds to establish precision and recovery prior to the first run on an instrument. *Continuing Calibration* is the mid-point calibration standard used to verify calibration. *Ongoing Precision and Recover* standard is a method blank with known spike amounts analyzed like a sample. Labeled compounds are spiked into the field samples, and the recovery percentage is a measurement quality objective.

Laboratory control samples contain known amounts of analytes and indicate bias due to sample preparation and calibration. Results of field duplicate samples provide estimates of analytical precision, through the process of comparing the relative percent difference (RPD) in the sample values. Field and laboratory RPDs are not specified for this project due to the wide range in expected concentrations, potential for matrix interferences, and the relative newness of these methods. MEL and AXYS are expected to meet all QC requirements published in the EPA methods being used for this project.

Quality Control Procedures

Field

Field QC samples for this project will include duplicate samples, bottle blanks (water), and matrix spikes (Table 11). Duplicates will provide estimates of a combined field and analytical variability. An influent, effluent, and biosolids sample will be field duplicated and filled in a side-by-side manner at LOTT. The field blank sample is used to evaluate contamination arising from sample containers or sample handling in the field.

Table 11. Field Quality Control Samples for each Parameter Monitored at Wastewater Treatment Plants.

Methods or Parameter	Duplicate Water Sample	Duplicate Biosolid Sample	Field Blank
1694: PPCPs	1/project	1/project	1/project
1698: Steroids/Hormones	1/project	1/project	1/project
8270: BNAs	1/project	1/project	1/project
Ammonia	1/project	NA	1/project
Nitrate + Nitrite	1/project	NA	1/project
Total Persulfate Nitrogen	1/project	NA	1/project
Orthophosphate	1/project	NA	1/project
Total Phosphorus	1/project	NA	1/project
Total Suspended Solids	1/project	NA	NA

Laboratory

The QC procedures routinely followed by MEL and AXYS, shown in Table 12, are for the three organic chemistry methods used to detect PPCPs. The conventional parameters of nutrient and TSS laboratory QC routinely followed by MEL will be satisfactory for purposes of this project. MEL's full quality control procedures are documented in the Lab Users Manual (MEL, 2005). The laboratory will be able to assess laboratory bias in sample results.

Table 12. Laboratory Quality Control Samples for the Three Methods to Detect PPCPs.

Parameter	Check Standard/ Laboratory Control Sample	Method Blanks	OPR[a] Standards/ Labeled Compounds	Matrix Spike
1694: PPCPs	1/batch	1/batch	all samples	1/project
1698: Steroids/ Hormones	1/batch	1/batch	all samples	1/project
8270: BNAs	1/batch	1/batch	all samples	1/project

[a] Ongoing precision and recovery.

The matrix spike is an extra field sample taken at LOTT that the laboratory will spike with a known amount of target compounds and then measure the recovery of those analogues to assess matrix interferences.

Total variation (field plus lab) will be assessed by collecting duplicate samples for all parameters for 20% of samples. These duplicates will be used to assess whether the data quality objectives for precision were met. If the objectives were not met, the data will be qualified. MEL and AXYS routinely analyze duplicate sample analyses in the laboratory for QC purposes. The difference between field and laboratory variability is a measure of the sample field variability.

Laboratories will not be able to directly assess bias from field procedures. However, bias will be minimized by strictly following standard protocols.

Data Verification and Review

Data Verification

Field data and observations will be recorded on waterproof paper. MEL and AXYS will each prepare case narratives for each data set. The data package from MEL and AXYS will include a case narrative discussing any problems with the analyses, corrective actions taken, changes to the referenced method, and an explanation of data qualifiers. The data package will also include all associated QC results. This information is needed to evaluate the accuracy of the data and to determine whether the measurement quality objectives have been met. This will include results for all laboratory control samples, method blanks, standards and labeled compounds, and laboratory duplicates included in the sample batch.

A Quality Assurance (QA) review of all laboratory data and case narratives will be conducted by Environmental Protection Agency-Office of Science and Technology or their contractor. This will include a verification that (1) methods and protocols specified in this QA Project Plan were followed, (2) all calibrations, checks on quality control, and intermediate calculations were performed for all samples, and (3) the data are consistent, correct, and complete, with no errors or omissions. Evaluation criteria will include the acceptability of holding times, instrument calibration, procedural blanks, spike sample analyses, precision data, laboratory control sample analyses, and appropriateness of data qualifiers assigned.

To determine if measurement quality objectives have been met, the project lead will review results for initial precision and recovery, continuing calibration, laboratory control samples, duplicate samples, and labeled compound recovery. The field and method blank results will be examined to verify there was no significant contamination of the samples. To evaluate whether the targets for reporting limits have been met, the results will be examined for *non-detects* to determine if any values exceed the lowest concentration of interest.

The project lead will review the laboratory data packages, verify the report, and assess the usability of the data. Based on these assessments, the data will be either accepted, accepted with appropriate qualifications, or rejected and re-analysis considered.

Data Quality (Usability) Assessment

Once the data have been verified, the project lead will determine if the data can be used to make the calculations, determinations, and decisions for which the project was conducted. If the results are satisfactory, data analysis will proceed.

Data analysis will include, but not necessarily be limited to, compiling summary statistics and constructing plots to (1) examine the distribution of the concentrations detected in the samples, (2) compare levels in the influent versus effluent, and (3) compare levels of reduction between the WWTPs and treatment methods.

Data Management Procedures

All project data will be entered into Excel spreadsheets. All entries will be independently verified for accuracy by Ecology's Environmental Assessment Program.

All project data will be entered into Ecology's Environmental Information Management system (EIM). Data entered into EIM follow a formal Data Verification Review Procedure where data are reviewed by the project manager of the study, the person entering the data, and an independent reviewer from Ecology's Environmental Assessment Program.

Audits and Reports

MEL participates in performance and system audits of their routine procedures. Results of these audits are available on request. Ecology's Accreditation Program establishes whether the laboratory has the capability to provide accurate, defensible data. AXYS is accredited by Ecology for other parameters. The accreditation involves an evaluation of the laboratory's quality system, staff, facilities and equipment, test methods, records, and reports.

The PPCPs and steroids/hormones analyses contracted to AXYS will be evaluated by the project lead. Because these methods are still in the process of being published, Ecology's Quality Assurance Officer has waived the requirement of accreditation for this project.

The following reports will be prepared for this project:

1. The data will be provided to the project lead in printed and electronic formats.
2. A draft technical report will be prepared jointly by EPA and Ecology's Environmental Assessment Program staff on or before April 2009. The project lead is Brandi Lubliner.
3. A final technical report is anticipated in June 2009.
4. The project data will be entered into Ecology's EIM on or before June 2009.

References

Conn, K.E., L.B. Barber, G.K. Brown, and R.L. Siegrist, 2006. Occurrence and Fate of Organic Contaminants During Onsite Wastewater Treatment, Environmental Science and Technology, Vol. 40, No. 23, pp 7358-7366.

Drury, D.D., S. A. Snyder, and E.C. Wert, 2006. Using Ozone Disinfection for EDC Removal, Water Environment Foundation, pp 1249-1258.

EPA, 1990. Specifications and Guidance for Obtaining Contaminant-Free Sample Containers. U.S. Environmental Protection Agency. OSWER Directive #93240.0-05.

EPA, 2007a. Method 1694: Pharmaceuticals and Personal Care Products in Water, Soil, Sediment, and Biosolids by HPLC/MS/MS. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue, NW, Washington, DC 20460. EPA-821-R-08-002. December 2007. www.epa.gov/waterscience/methods/method/files/1694.pdf

EPA, 2007b. Method 1698: Steroids and Hormones in Water, Soil, Sediment, and Biosolids by HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology Engineering and Analysis Division (4303T), 1200 Pennsylvania Avenue, NW, Washington, DC 20460. EPA-821-R-08-003. December 2007. www.epa.gov/waterscience/methods/method/files/1698.pdf

Guillette, L.J., D. A. Crain, M.P. Gunderson, S.A.E. Kools, M.R. Milnes, E.F. Orlando, A.A. Rooney, and A.R. Woodward, 2000. Alligators and Endocrine Disrupting Contaminants: A Current Perspective. American Zoologist, Vol. 40, No. 3, pp 438-452.

Heberer, T., A. Mechlinski, B. Fanck, A. Knappe, G. Massmann, A. Pekdeger, and B. Fritz, 2004. Field Studies on the Fate and Transport of Pharmaceutical Residues in Bank Filtration, Ground Water Monitoring and Remediation, Vol. 24, No. 2, pp 70-77.

Holtz, S., 2006. There Is No Away. Pharmaceuticals, Personal Care Products, and Endocrine-Disrupting Substances: Emerging Contaminants Detected in Water, Canadian Institute for Environmental Law and Policy, Toronto, Ontario, 82 p.

Jones, C., 2008. Personal communication, Washington State Department of Ecology, Municipal Wastewater Treatment, Southwest Regional Office, Olympia, WA.

Kasprzyk-Hordern B., R.M. Dinsdale, and A.J. Guwy, 2008. Multiresidue methods for the analysis of pharmaceuticals, personal care products and illicit drugs in surface water and wastewater by solid-phase extraction and ultra performance liquid chromatography-electrospray tandem mass spectrometry. Analytical and Bioanalytical Chemistry. 391(4):1293-308.

Khan, S.J. and J.E. Ongerth, 2004. Modeling of Pharmaceutical Residues in Australian Sewage by Quantities of Use and Fugacity Calculations, Chemosphere, 54, pp 355-367.

Kimura, K., H. Hara, and Y. Watanabe, 2005. Removal of Pharmaceutical Compounds by Submerged Membrane Bioreactors (MBRs), Desalination, 178, pp 135-140.

Kinney, C.A., E.T. Furlong, S.D. Zaugg, M.R. Burkhardt, S.L. Werner, J.D. Cahill, and G.R. Jorgensen, 2006. Survey of Organic Wastewater Contaminants in Biosolids Destined for Land Application, Environmental Science and Technology, Vol. 40, No. 23, pp 7207-7215.

Lower Columbia River Estuary Partnership, 2007. Lower Columbia River and Estuary Ecosystem Monitoring: Water Quality and Salmon Sampling Report. Portland, OR.

MEL, 2005. Manchester Environmental Laboratory Lab Users Manual. Eighth Edition. Manchester Environmental Laboratory, Washington State Department of Ecology, Manchester, WA.

MEL, 2006. Manchester Environmental Laboratory Quality Assurance Manual. Manchester Environmental Laboratory, Washington State Department of Ecology, Manchester, WA.

National Institute on Drug Abuse, 2005. Prescription Drugs, Abuse and Addiction, NIH Publication No. 05-4881, Research Report Series, 12 p.

Oaks, J.L., M. Gilbert, M.Z. Virani, M. Arshad, S. Mahmood, A. All, and A.A. Khan, 2004. Diclofenac Residues as the Cause of Vulture Population Decline in Pakistan. Nature. Vol. 427, pp 630 - 633.

Orlando, E.F., A.S. Kolok, G.A. Binzcik, J.L. Gates, M.K. Horton, C.S. Lambrigth, L.E. Gray, A.M. Soto, and L.J. Guillette, 2004. Endocrine-Disrupting Effects of Cattle Feedlot Effluent on an Aquatic Sentinel Species, the Fathead Minnow. Environmental Health Perspectives. 112:3, pp 353-358

Ott, W., 1995. Environmental Statistics and Data Analysis. Lewis Publishers, New York, NY.

PH:ARM Pilot Team, 2007. Disposal of Medications from Residential Consumers: Issues, Barriers, and Opportunities, 33p.

Redding, M., 2008. (Unpublished) Literature Review of Pharmaceuticals and Personal Care Products. Environmental Assessment Program, Washington State Department of Ecology, Olympia, WA.

Snyder, S.A., S. Adham, A.M. Redding, F.S. Cannon, F.S., J. DeCarolis, J. Oppenheimer, E.C. Wert, and Y. Yoon, 2006. Role of Membranes and Activated Carbon in the Removal of Endocrine Disruptors and Pharmaceuticals, Desalination, Vol. 202, pp 156-181.

Spongberg A.L. and J.D. Witter, 2008. Pharmaceutical compounds in the wastewater process stream in Northwest Ohio. Sci Total Environ. 397(1-3):148-57.

Swanson, T., 2007. Standard Operating Procedure (SOP) for Hydrolab® DataSonde® and MiniSonde® Multiprobes, Version 1.0. Washington State Department of Ecology, Olympia, WA. SOP Number EAP033. www.ecy.wa.gov/programs/eap/quality.html

Swartz, C.H., S. Reddy, M.J. Benotti, H. Yin, L.B. Barber, B.J. Brownawell, and R.A. Rudel, 2006. Steroid Estrogens, Nonylphenol Ethoxylate Metabolites and Other Wastewater Contaminants in Groundwater Affected by a Residential Septic System on Cape Cod, MA., Environmental Science and Technology, Vol. 40, No. 16, pp 4894-4902.

Ternes, T.A., 1998. Occurrence of Drugs in German Sewage Treatment Plants and Rivers, Water Resources, Vol. 32, No. ll, pp 3245-3260.

Ternes, T.A., M. Meisenheimer, D. McDowell, F. Sacher, H-J Brauch, B. Haist-Gulde, G. Preuss, U. Wilme, and N. Zulei-Seibert, 2002. Removal of Pharmaceuticals During Drinking Water Treatment, Environmental Science and Technology, Vol. 36, No. 17, pp 3855-3863.

Appendices

Appendix A. Chemical Compounds Per EPA Methods

Tables A-1 and A-2 are the chemical compounds listed in EPA Methods 1694 and 1698.

Table A-1. EPA Method 1694: PPCPs in Water, Soil, Sediment, and Biosolids.

Chemical Name
Acetaminophen
Caffeine
Carbamazepine
Cimetidine
Codeine
Cotinine
Dehydronifedipine
Diltiazem
Diphenhydramine
Erythromycin
Fluoxetine
Gemfibrozil
Hexachlorobenzene
Lincomycin
Metformin
Miconazole
Ranitidine
Salbutamol (Albuterol)
Sulfamethoxazole
Thiabendazole
Triclosan
Trimethoprim
Warfarin
4-Epianhydrochlortetracycline (EACTC)
4-Epianhydrotetracycline (EATC)
4-Epichlortetracycline (ECTC)
4-Epioxytetracycline (EOTC)
4-Epitetracycline (ETC)
Ampicillin
Anhydrochlortetracycline (ACTC)
Anhydrotetracycline (ATC)
Carbadox
Cefotaxime
Chlortetracycline (CTC)
Ciprofloxacin
Clarithromycin
Clinafloxacin
Cloxacillin
Demeclocycline
Doxycycline
Enrofloxacin
Erythromycin
Erythromycin anhydrate
Flumequine
Isochlortetracycline (ICTC)
Lomefloxacin
Minocycline
Naproxen
Norfloxacin
Norgestimate
Ofloxacin
Ormetoprim
Oxacillin
Oxolinic acid
Oxytetracycline (OTC)
Penicillin G
Penicillin V
Roxithromycin
Sarafloxacin
Sulfachloropyridazine
Sulfadiazine
Sulfamerazine
Sulfamethizole
Sulfanilamide
Tetracycline (TC)
Triclocarban
Virginiamycin

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

Chemical Name
Androstenedione
Androsterone
Bisphenol A propane-d6
Campesterol
Cholestanol
Cholesterol and Cholesterol-d7
Coprostanol
Desmosterol
Desogestrel
Diethylstilbestrol-d8
17α-Dihydroequilin
Epi-Coprostanol
Equilenin
Equilin
Ergosterol
17α-Estradiol
17α-Ethynyl Estradiol and17α-Ethynyl Estradiol-d4
17β-Estradiol and 17β-Estradiol-d4
α-Estradiol-3-benzoate
Estriol
Estrone
Mestranol and Mestranol-d4
Norethindrone and Norethindrone-d6
Norgestrel and Norgestrel-d6
Progesterone and Progesterone-d9
beta-Sitosterol
beta-Stigmastanol
Stigmasterol
Testosterone

Table A-3 is a modified list of compounds that will be analyzed by EPA Methods 8270 at MEL.

Table A-3. EPA Method 625/SW-846 Method 8270: Base/Neutral and Acid Extractable Organics**.**

Chemical Name
1,4-Dichlorobenzene
4-nonylphenol
Bis-phenol A
Caffeine
Cholesterol
Coprostanol
Fluoranthene
Hexachlorobenzene
Isophorone
4-Methylphenol (p-cresol)
Tri(2-chloroethyl) phosphate TCEP
1,2,4-Trichlorobenzene
1,2-Dichlorobenzene
1,2-Diphenylhydrazine
1,3-Dichlorobenzene
1-Methylnaphthalene
2,2'-Oxybis[1-chloropropane]
2,4,5-Trichlorophenol
2,4,6-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
3,3'-Dichlorobenzidine
3B-Coprostanol
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
Benzoic Acid
Benzyl Alcohol
Bis(2-Chloroethoxy)Methane
Bis(2-Chloroethyl)Ether
Bis(2-Ethylhexyl) Phthalate
Butylbenzylphthalate
Carbazole
Chrysene
Dibenzo(a,h)anthracene
Dibenzofuran
Diethylphthalate
Dimethylphthalate
Di-N-Butylphthalate
Di-N-Octyl Phthalate
Fluorene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Indeno(1,2,3-cd)pyrene
Naphthalene
Nitrobenzene
N-Nitrosodimethylamine
N-Nitroso-Di-N-Propylamine
N-Nitrosodiphenylamine
Pentachlorophenol
Phenanthrene
Phenol
Pyrene
Retene

Appendix B. EPA Method 1694

EPA Method 1694 can be found at the following website:
www.epa.gov/waterscience/methods/method/files/1694.pdf

Appendix C. EPA Method 1698

EPA Method 1698 can be found at the following website:
www.epa.gov/waterscience/methods/method/files/1698.pdf

Appendix D. List of Acronyms

Following are acronyms and abbreviations used frequently in this report.

AXYS	AXYS Analytical Services Ltd.
BNA	base neutrals and acid
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management system
EPA	U.S. Environmental Protection Agency
LOTT	Lacey-Olympia-Tumwater-Thurston County
MEL	Manchester Environmental Laboratory
MGD	million gallons per day
$NO_3^- > N_2$	atmospheric nitrogen
PPCPs	pharmaceuticals and personal care products
QA	quality assurance
QC	quality control
RPD	relative percent difference
TSS	total suspended solids
UV	ultra-violet
WWTP	wastewater treatment plant