Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Process Water and Groundwater at Three Reclaimed Water Treatment Plants

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Cover photo: LOTT Martin Way Reclaimed Water Treatment Plant: Permit compliance monitoring point for Class A reclaimed water (Michael Friese, Department of Ecology)

Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Process Water and Groundwater at Three Reclaimed Water Treatment Plants

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Table of Contents

Page

List of Figures and Tables

Page

Figures

Tables

Abstract

Pharmaceuticals, personal care products, hormones, and sterols were analyzed in the spring and fall of 2011 in reclaimed water and groundwater samples collected at three reclaimed water facilities in Washington: LOTT Martin Way (Lacey), Quincy, and Yelm. Using reclaimed water to recharge groundwater is a main component of the facilities selected for study.

The objectives of this study were to: (1) increase understanding of the occurrence of these chemicals in reclaimed water, (2) assess their presence/absence in wells at groundwater recharge sites, and (3) identify potential indicator chemicals for future monitoring.

A total of 73 of the 145 compounds analyzed were detected in reclaimed water. The most frequently observed were drugs (or their metabolites) used to treat high blood pressure, followed by antidepressants and antibiotics. Relatively few hormones or sterols were detected, partly due to interferences and elevated background levels during the analyses.

A total of 15 compounds were found in groundwater samples obtained from established monitoring wells within the recharge area. With a few exceptions, measured concentrations in groundwater were lower than in the corresponding reclaimed water sample, by about a factor of 2 on average.

Carbamazepine (anti-epileptic), meprobamate (tranquilizer), and sulfamethoxazole (antibiotic) were consistently detected in both reclaimed water and groundwater monitoring wells at all three facilities. These pharmaceuticals appear to be good tracers of reclaimed water. The majority of compounds (80%) identified in reclaimed water do not appear to persist in groundwater at readily detectable levels.

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Background

Pharmaceuticals and personal care products (PPCPs) encompass a wide array of prescription and over-the-counter drugs for humans and animals, as well as products such as stimulants (coffee, nicotine), disinfectants, and insect repellants. Of related interest are natural and synthetic hormones and sterols. Steroid hormones include sex hormones such as estrogens, progesterone, and testosterone. The most well known animal sterol is cholesterol.

The detection of these chemicals at low levels in surface water, groundwater, soils, and drinking water has led to concerns that they may have adverse impacts on humans or animals (EPA, 2010). Their continual introduction into the environment causes a pseudo-persistence that might not otherwise exist (Halling-Sorenson et al., 1998).

Humans typically excrete 50% to 90% of the active ingredients in ingested pharmaceuticals, either unmetabolized or as metabolites (McGovern and McDonald, 2003). A King County, Washington study found that about 30% of residents dispose of drugs in the sink or toilet (Grasso, 2009). These chemicals enter on-site sewage systems, municipal wastewater treatment plants (WWTPs), and reclaimed water treatment facilities (RWTFs).

Reclaimed water is used water that has been treated to a high level so it can be beneficially reused. Tertiary-treated reclaimed water benefits from a set of treatment processes that provide a higher level of treatment and reliability than conventional secondary-treatment WWTPs. The water released from a RWTF is designed to meet the quality standards for its intended use. In Washington State, each RWTF is issued a Reclaimed Water Permit to produce and distribute reclaimed water in accordance with special and general conditions stipulated in the permit. Washington currently has 321 municipal WWTPs and 24 RWTFs.

With the increased difficulty in securing additional water supplies for growing communities, cities are using reclaimed water for non-potable uses to help extend water supplies. For example, reclaimed water is distributed to public and private entities for commercial and industrial uses, applied to land for irrigation at agronomic rates, and used to recharge groundwater via surface percolation at permitted locations.

In 2008, the Washington State Department of Ecology, Environmental Assessment Program (Ecology EAP) conducted a field study to assess the removal of PPCPs, hormones, sterols, and other chemicals by various treatment technologies at five WWTPs (Lubliner et al., 2010). Three of the five facilities produce reclaimed water: Lacey-Olympia-Tumwater-Thurston County Clean Water Alliance (LOTT) Martin Way Reclaimed Water Plant, LOTT Budd Inlet Reclaimed Water Plant, and the Hayden Wastewater Research Facility in Idaho. Substantially fewer chemicals were detected in reclaimed water than in conventional WWTP effluent.

The Ecology Water Quality Program requested that EAP do follow-up sampling for PPCPs, hormones, and sterols at selected reclaimed water facilities. The objectives were to: (1) expand Ecology’s understanding of the occurrence of these chemicals in reclaimed water, (2) assess their presence/absence in wells at groundwater recharge sites, and (3) identify potential indicator chemicals for future monitoring.

Project Description

Reclaimed water and groundwater samples were collected at the following three Washington RWTFs during the spring and fall of 2011 (Figure 1):

- LOTT Martin Way Reclaimed Water Plant and Hawks Prairie Ponds/Recharge Basins (Lacey)
- Quincy Reclaimed Water Plant
- Yelm Water Reclamation Facility (fall only)

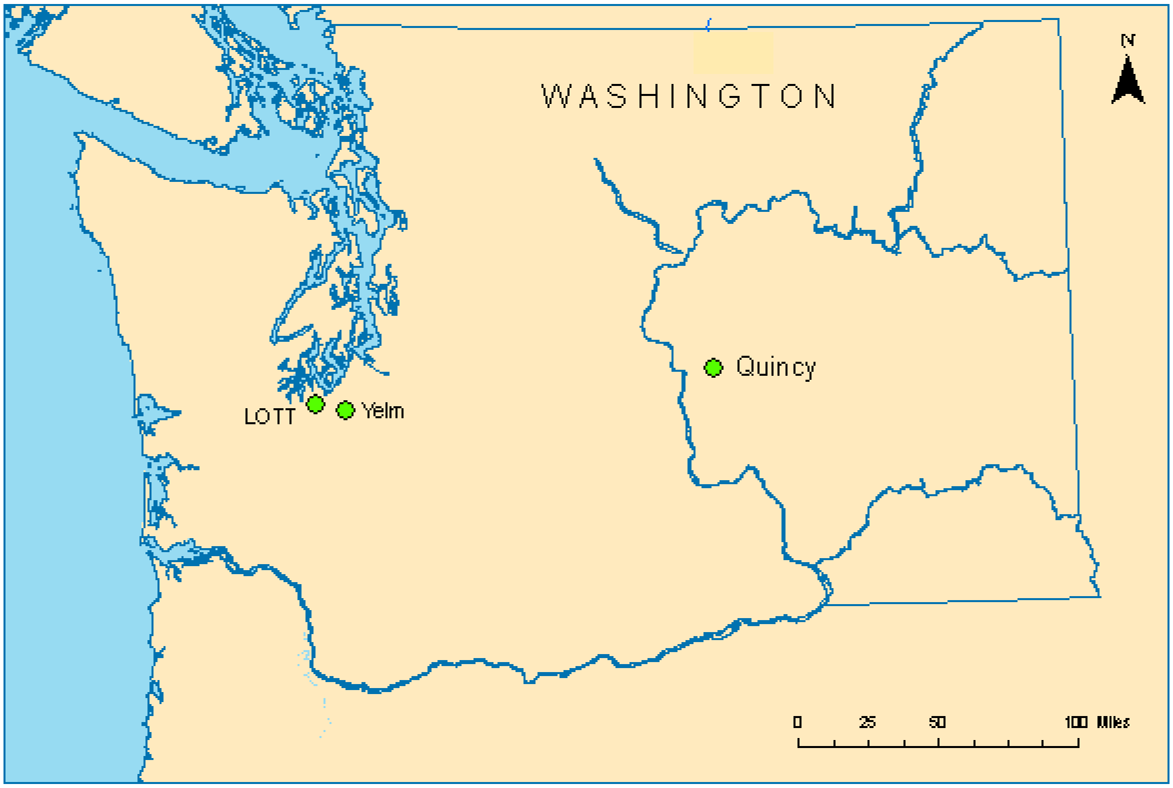

Figure 1. Location of Reclaimed Water Treatment Facilities Sampled for Pharmaceuticals, Personal Care Products, Hormones, and Sterols in 2011.

Reclaimed water and three monitoring wells were sampled at each plant once during the spring and once during the fall. The completed project provided data on two reclaimed water and six groundwater samples each from LOTT and Quincy. In the spring, Yelm was not producing reclaimed water because of difficulties meeting their discharge limits for total nitrogen. Yelm began producing and applying reclaimed water in August. One reclaimed water and three groundwater samples were collected at Yelm in the fall.

The samples were analyzed for 118 PPCPs and 27 synthetic or naturally-occurring hormones and sterols. Isotope dilution methods were used to achieve detection limits in the low parts per trillion range (nanograms per liter, ng/L).

PPCP, hormone, and sterol samples were analyzed by AXYS Analytical Services Ltd. (AXYS) in Sidney, British Columbia, through a contract with the Ecology Manchester Environmental Laboratory (MEL). This study followed a Quality Assurance Project Plan (Johnson and Marti, 2011).

Sampling Design

Facility Selection

RWTFs that have groundwater recharge basins as a major component of their operations were selected for sampling, in consultation with regional Ecology water quality staff. Each of the selected facilities had been operating for five years or more and had an established groundwater monitoring program within the infiltration area, as required by the Reclaimed Water Permit. The three plants differ in wastewater treatment processes and in geologic setting of the infiltration areas. A description of treatment processes and other background information is provided for each facility in Table 1 and Appendix A.

Table 1. Background Information on Reclaimed Water Treatment Facilities Sampled in 2011.

Facility Name	Location	Permit No.	Year Permit Authorized	Treatment Process	Average Monthly Flow (MGD)	Receiving Water
LOTT Martin Way Reclaimed Water Plant & Hawks Prairie Ponds/Recharge Basins	Lacey, WA	ST000 6206	2006	Enhanced biological nitrogen removal (EBNR) with membrane filtration. (Membrane biological reactors; MBRs). Sodium hypochlorite disinfects the treated water before distribution.	2.0 (capacity) 0.6 (2011)	Constructed wetland ponds and groundwater recharge through eight infiltration basins.
City of Quincy Reclaimed Water Plant	Quincy, WA	ST000 5278	2006	Two activated sludge lagoons using sequencing batch reactor (SBR) technology to remove nitrogen and attain secondary treatment standards. The SBR discharges to an equalization basin that reduces peak flows to a lower, more uniform flow. Advanced treatment includes chemical coagulation, continuous backwash upflow sand filters, and disinfection with ultraviolet (UV) light.	1.5	Recharge groundwater through six infiltration basins.
City of Yelm Water Reclamation Facility	Yelm, WA	WA00 40762	2005	SBR technology for secondary treatment (biological oxidation) and nitrogen removal. Advanced treatment follows with chemical coagulation, upflow sand filters, and chlorine disinfection.	1.0 (capacity)	Constructed surface and submerged wetlands that polish the reclaimed water before it recharges the groundwater.

LOTT: Lacey-Olympia-Tumwater-Thurston County Clean Water Alliance
MGD: million gallons per day

After treating to meet Washington State Class A standards, reclaimed water is piped to the recharge infiltration area. At the LOTT and Yelm facilities, reclaimed water is first sent through a series of constructed wetlands to allow natural treatment processes to act on the water prior to it being piped to the recharge basins for infiltration into the ground.

The infiltration areas for LOTT and Yelm are geologically characterized by thick sequences of unconsolidated glacial deposits which include highly permeable sands and gravels. Depth to groundwater differs between the two sites, ranging from approximately 15-25 feet at Yelm to more than 90 feet at LOTT. Geology of the Quincy facility, located in eastern Washington, is substantially different. It is characterized by a sequence of unconsolidated and poorly consolidated deposits of fluvial and lacustrine sand and silt with some fluvial gravel. Depth to groundwater is approximately 9-15 feet. Further description of the infiltration areas geology is provided in Appendix A.

Reclaimed Water

The reclaimed water produced by each facility was sampled at the point of compliance for the Reclaimed Water Permit. Appendix B has a description of the sites where reclaimed water samples were obtained.

Groundwater

Existing monitoring wells were used for this project. Wells were selected based on previous monitoring results and in consultation with Ecology regional water quality staff and facility operators. Three wells were sampled at each facility. Where possible, one well outside the recharge area (up-gradient) and two wells within the recharge area (down-gradient) were sampled. This was achieved at the Quincy and Yelm facilities. No identifiable wells outside of the recharge area were available at LOTT.

All down-gradient wells were intentionally selected next to the recharge infiltration basins to characterize PPCP, hormone, and sterol concentrations in the shallow groundwater soon after infiltration of reclaimed water. Previous monitoring data for conventional water quality parameters indicated that the selected wells at each facility were the most affected by the reclaimed water infiltrate. Well locations are shown in Appendix A.

Timing and Number of Samples

Reclaimed Water

Some PPCP data had already been obtained on reclaimed water through the Lubliner et al. (2010) study. For this reason and due to budget constraints, reclaimed water samples were limited to one from each facility during the spring (May) and fall (October) of 2011, coinciding with the groundwater sampling described below. As previously noted, this project was only able to sample Yelm during the fall because no reclaimed water was produced in the spring.

Groundwater

Groundwater samples were collected in the spring and fall to correspond with potential seasonal water table changes. Six groundwater samples each were collected from the LOTT and Quincy facilities, and three samples were collected from Yelm, for a total of 15 samples, as shown in Table 2. Well locations are shown in Appendix A, Figures A-1, A-2, and A-3, respectively.

Table 2. Groundwater Sample Locations.

Facility	Spring 2011		Fall 2011	
	Up-Gradient	Down-Gradient	Up-Gradient	Down-Gradient
LOTT	None	LOTTMW-1 LOTTMW-8 LOTTMW-9	None	LOTTMW-2 LOTTMW-8 LOTTMW-9
Quincy	Quinwo-1	Quinw-26 Quinw-60	Quinwo-1	Quinw-26 Quinw-60
Yelm	Not Sampled		YELMW-1	YELMW-3 YELMW-4

Groundwater samples outside the recharge area were collected at each facility to provide water quality data on the aquifer before infiltration of reclaimed water. Spring results indicated that the well initially selected as up-gradient for LOTT (LOTTMW-1) was actually within the recharge area. Because an alternate up-gradient well was not available and well MW-1 was dry during the fall sampling, well LOTTMW-2, located in the center of the recharge area, was sampled in its place. Up-gradient wells for Quincy and Yelm were Quinwo-1 and YELMW-1.

Groundwater samples were also collected from monitoring wells within the recharge area. Selected wells were located adjacent to the recharge infiltration basins to determine the presence or absence of pharmaceutical products in shallow groundwater soon after reclaimed water infiltration.

Quality Control Samples

Field quality control samples for this project consisted of duplicate reclaimed water and groundwater samples and field blanks. Three duplicate samples and three blank samples were analyzed. These samples are described in more detail under Methods and Data Quality.

Chemical Analyses

All reclaimed water and groundwater samples were analyzed for PPCPs, hormones, and sterols. Ancillary parameters included total suspended solids (TSS), turbidity, and chloride.

PPCPs were analyzed by high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS) following EPA Method 1694. Hormones and sterols were analyzed by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) following EPA Method 1698. The samples were analyzed by AXYS. The same methods and laboratory were used in Lubliner et al. (2010). AXYS played a major role in developing these methods for EPA.

Methods 1694 and 1698 provide low detection limits for a large number of compounds: 118 PPCPs, 17 hormones, and 10 sterols (Appendix C). For the present effort, an expanded list of PPCPs was analyzed (118 vs. 72 compounds in the Lubliner study). Target detection limits were 10 ng/L or less for most PPCPs and hormones. Sterols had higher detection limits of 25 – 1,000 ng/L but are also typically encountered at higher concentrations.

TSS, turbidity, and chloride were analyzed in the reclaimed water. TSS and turbidity are general indicators of water quality and are among the water quality parameters each facility is required to monitor as part of their Reclaimed Water Permit. Chloride is a useful indicator of the presence of reclaimed water in groundwater because it behaves conservatively in the environment. Chloride can migrate through groundwater relatively unaltered, making it a good tracer. Groundwater samples were analyzed for chloride but not TSS or turbidity.

Summary

Table 3 summarizes the sampling design for Ecology's 2011 reclaimed water study.

Table 3. Sampling Design (number of samples).

Season/Facility	Date	Reclaimed Water Samples	Groundwater Samples	Quality Control Samples		Total Samples
				Field Duplicates	Field Blanks	
Spring 2011						
LOTT	05/11	1	3	1	1*	6
Quincy	05/24	1	3	0	0	4
Yelm	Not producing reclaimed water – no samples					
					Total samples =	10
Fall 2011						
LOTT	10/05	1	3	0	0	4
Quincy	10/20	1	3	1	1*	6
Yelm	10/06	1	3	0	1†	5
					Total samples =	15

*transfer blank
†pump blank

Methods

Sampling Procedures

Sample containers, preservation, and holding times for reclaimed water and groundwater samples are shown in Table 4.

Table 4. Sample Containers, Preservation, and Holding Times.

Analysis	Sample Size	Container	Field Preservation	Holding Time
PPCPs	1 L*	HDPE	Cool to $\leq$ 4 °C	48 hours (refrigerated) 30 days (frozen) 40 days (extraction)
Hormones/Sterols	1 L[†]	HDPE	Cool to $\leq$ 4 °C	48 hours (refrigerated) 30 days (frozen) 40 days (extraction)
Total Suspended Solids	1 L	Poly bottle	Cool to $\leq$ 4 °C	7 days
Turbidity	500 mL	Poly bottle	Cool to $\leq$ 4 °C	48 hours
Chloride	500 mL	Poly bottle	Cool to <4 °C	28 days

*Four liters/sample provided to AXYS
[†] Two liters/sample provided to AXYS
HDPE: High-density polyethylene

EPA has not conducted formal holding time studies for PPCPs, hormones, or sterols. Freezing of aqueous samples is encouraged to minimize degradation. The holding times in Table 4 are those followed by AXYS for this project. The PPCP samples were frozen at Ecology headquarters after collection. The hormones/sterols samples were frozen at AXYS unless extracted within 48 hours.

Reclaimed Water

To maximize comparability between datasets, sampling procedures for reclaimed water followed the Lubliner et al. (2010) study. Individual grab samples were taken by hand in the morning, mid-day, and early afternoon, and composited by equal volume into appropriate sample containers (Table 4). A high density polyethylene PPCP bottle was used to take the grabs.

Sample bottles for PPCPs and hormones/sterols were provided by AXYS. TSS, turbidity, and chloride bottles were obtained from MEL. Nitrile gloves were worn by field personnel collecting and packaging the samples. Care was taken not to contaminate the samples with extraneous material.

Duplicate reclaimed water samples were prepared on two occasions, once at LOTT and once at Quincy (see Table 3). This was done by distributing the contents of each grab among two complete sets of sample containers.

Reclaimed water samples were kept in plastic bags on ice in coolers during the compositing period. Each sample was assigned a unique sample number obtained from MEL.

The samples were returned to Ecology headquarters the day after collection. PPCP samples were frozen; hormones/sterols samples were refrigerated. The PPCP and hormones/sterols samples were later shipped in coolers with blue ice to AXYS by overnight FedEx to arrive within holding times. The TSS, turbidity, and chloride samples were refrigerated for transport to MEL. Chain-of-custody procedures were followed.

Groundwater

Established monitoring wells were sampled at each of the RWTFs. All wells were sampled using low-flow sampling methods. The LOTT wells have permanently installed dedicated submersible pumps. These wells were sampled by LOTT and Ecology staff using procedures described in the *Hawks Prairie Groundwater Recharge Project Groundwater Sampling Quality Assurance Plan* (Stormon, 2011). Ecology sampled the Quincy and Yelm wells with a stainless-steel submersible pump in accordance with Ecology Standard Operating Procedure (SOP) EAP078 (Marti, 2011).

Static groundwater levels were measured at each well prior to purging and sampling. Measurements were taken with a calibrated electric well probe according to SOP EAP052 (Marti, 2009).

Ideally, monitoring wells for a recharge facility should be screened across the water table so that samples can be collected just below the water table surface where recharge water and groundwater mix. This is preferred because groundwater chemistry can change substantially with depth below the water table. For optimal sampling, the well pump intake should be placed just below the water table in the wells screened interval. This was achieved at the Quincy and Yelm facilities where measured water levels are below the top of the well screen and the pump intakes were placed just below the water table for sampling. Because of the groundwater mounding created by the recharge water at LOTT, not all well screens are at the water table. Also, in some wells the permanently installed pump intakes are substantially below the water table.

All wells were purged at a rate of 1-liter/minute or less. Purge water was routed through a continuous flow cell where temperature, pH, specific conductance, and dissolved oxygen were monitored and recorded at regular intervals. Purging continued until field parameter readings stabilized (Table 5).

Table 5. Field Methods and Well Purging Criteria.

Field Measurements	Instrument Type	Method	Accuracy	Stabilization Criteria
Water Level	Solinst Water Level Meter	SOP EAP052	±0.03 feet	Not applicable
pH	YSI ProPlus Quatro Cable	EPA Method 150.1	±0.2 std units	±0.1 standard unit
Temperature	YSI ProPlus Quatro Cable	EPA Method 150.1	±0.2 °C	±0.1 °C
Dissolved Oxygen	YSI ProPlus Quatro Cable	EPA Method 360.1	±0.2 mg/L	±0.2 mg/L for values > 2 mg/L
Specific Conductance	YSI ProPlus Quatro Cable	EPA Method 120.1	±10 umhos/cm	±10 umhos/cm for values <1000 umhos/cm or ±20 umhos/cm for values >1000 umhos/cm

SOP: Standard Operating Procedure
EAP: Ecology's Environmental Assessment Program
EPA: U.S. Environmental Protection Agency

Groundwater samples were collected from each well at the completion of purging. The flow cell was disconnected, and the samples were collected directly from the pump's discharge tubing into appropriate sample containers (Table 4). Nitrile gloves were worn by field personnel collecting and manipulating the samples. Care was taken not to contaminate the samples with extraneous material.

Ecology's submersible pump was decontaminated between wells by circulating laboratory-grade detergent/water through the pump followed by a clean deionized water rinse, with each cycle lasting five minutes.

Duplicate samples were collected from well LOTTMW-8 in the fall by splitting the pump discharge between two sets of sample bottles.

Sample handling and delivery to laboratories followed the same procedures as for reclaimed water.

Laboratory Procedures

Table 6. Laboratory Procedures.

Analysis	Reporting Limit	Analytical Method	Laboratory
PPCPs	Appendix C	HPLC/MS/MS (EPA Method 1694)	AXYS
Hormones/Sterols	Appendix C	HRGC/HRMS (EPA Method 1698)	AXYS
Total Suspended Solids	1 mg/L	Standard Methods 2540D	MEL
Turbidity	1 NTU	Standard Methods 2130B	MEL
Chloride	0.1 mg/L	EPA Method 300.0	MEL

AXYS: AXYS Analytical Services LTD
MEL: Manchester Environmental Laboratory

Each analytical method is described briefly below.

EPA Method 1694: *Pharmaceuticals and Personal Care Products in Water Soil, Sediment and Biosolids by HPLC/MS/MS.* Method 1694 determines PPCPs in environmental samples by high performance liquid chromatography combined with tandem mass spectrometry using isotope dilution and internal standard quantitation techniques. (www.epa.gov/waterscience/methods/method/files/1694.pdf)

EPA Method 1698: *Steroids and Hormones in Water, Soil, Sediment, and Biosolids by HRGC/HRMS.* Method 1698 determines hormones and sterols in environmental samples by isotope dilution and internal standard high resolution gas chromatography combined with high resolution mass spectrometry. (www.epa.gov/waterscience/methods/method/files/1698.pdf)

LOTT and Yelm use chlorine disinfection in their treatment process (see Table 1). Free chlorine can oxidize certain PPCPs, hormones, and sterols, causing a reduction in concentration (Snyder et al., 2007). AXYS treats with 50 mg of ascorbic acid when significant amounts are present. AXYS tested all project samples for free chlorine, and none was detected.

All samples were filtered prior to extraction. The hormones and sterols analyses were run as whole water by combining separate extracts of the filtrate and particulate fraction. Whole water analysis is not an option for PPCP analysis because the presence of silicate-based particulates (sand, silts) can potentially eliminate some List 1 and List 5 surrogates.

Standard Methods 2540D: *Total Suspended Solids Dried at 103-105^{o}C.* A well-mixed sample is filtered through a weighed standard glass-fiber filter, and the is residue retained on the filter dried to a constant weight.

Standard Methods 2130B: *Turbidity: Nephelometric Method.* The intensity of light scattered by a sample under defined conditions is compared with the intensity of light scattered by a standard reference suspension.

EPA Method 300.0: *Chloride.* The water sample is injected into a stream of carbonate-bicarbonate eluant and separated on the basis of relative affinities for a low capacity, strongly basic chromatographic column. A conductivity detector determines the separated anions.

Data Quality

Data Review and Verification

MEL reviewed and verified all the chemical data for this project. For the TSS, turbidity, and chloride data generated by MEL, final review was performed by the unit supervisor or an analyst experienced with the method. Quality assurance and quality control at MEL are described in MEL (2008, 2012).

MEL's quality assurance coordinator and organics unit supervisor reviewed the PPCP, hormones, and sterols analyses contracted to AXYS laboratory. The organics review followed National Functional Guidelines for Superfund Organic Methods Data Review (EPA, 2005).

MEL prepared written case narratives assessing the precision and bias of these data. The reviews include a description of analytical methods and an assessment of holding times, calibration, blanks, internal standard recoveries, ion abundance ratios, laboratory control samples, duplicate samples, and matrix spike recoveries, as appropriate.

Flags were added by AXYS to draw attention to quality control conditions that may affect the data. MEL interprets the effect on data quality and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Environmental Information Management (EIM) guidelines.

With few exceptions, laboratory results for this project met acceptance criteria for the analyses conducted and the data are usable as qualified. MEL's data reviews are provided in Appendix D. Noteworthy problems encountered in analyzing these samples are summarized below:

- The initial extract for the List 1 & 5 PPCPs (see Appendix C) for spring collected samples proved unusable. Additional sample was extracted 43 and 56 days past the suggested sample hold time. All results for these samples were qualified as estimates. Because these samples had been stored frozen, degradation of target analytes is anticipated to have been low. EPA has not established a holding time for PPCPs.
- Due to inferences and background issues, reporting limits for certain hormones and sterols sometimes varied substantially between spring and fall, affecting detection frequency. This occurred for several of the LOTT and Quincy samples where reporting limits differed by an order of magnitude: LOTT effluent and well LOTTMW-8; Quincy effluent and wells Quinw-26 and -60. Reporting limits for all PPCPs, hormones, and sterols samples are included in Appendices C and E of this report
- AXYS reports that background levels of sterols are variable and often high. Results for these analytes tend to show poor duplication.

Appendix E has results for all project samples including method blanks, field blanks, and duplicate samples. The project data can also be accessed through Ecology's EIM (www.ecy.wa.gov/eim).

Method Blanks

Laboratory method blanks were included with each sample batch analyzed for PPCPs, hormones, and sterols.

Low levels of some target compounds were detected in the method blanks, particularly hormones and sterols (Appendix E). In cases where the concentration measured in a sample was at least 5 times greater than the blank, the blank result was considered insignificant relative to the native concentration in the sample, and the data were used without further qualification (EPA, 2005). Where the sample concentration was less than 5 times the blank, the result was flagged as not detected. A limit of 10 times was used for cholesterol, stigmasterol, and *B*-sitosterol due to elevated blanks.

MEL did not flag tentatively identified compounds less than 5 times the blank as not detected. For purposes of the present report, however, these "NJ" qualified data are considered not detected; see Appendix E for those data.

Field Blanks

The potential for contamination arising from sampling procedures, sample containers, preservation, or transport was assessed with field blanks. Two types of blanks were analyzed: transfer blanks and a pump blank. AXYS provided the water used to prepare the blanks ("Seastar Ultrapure Water Blank" in one-gallon amber glass bottles).

Transfer blanks were prepared by pouring blank water into sample containers at the same time, manner, and location that the reclaimed water samples were being composited. The transfer blank was intended to mimic the sample compositing procedure for reclaimed water. One transfer blank was analyzed for spring and one for fall.

A pump blank for the monitoring well sampling system was prepared by pumping blank water through Ecology's submersible pump to determine if field cleaning procedures were sufficient to prevent cross-contamination of samples. The blank was prepared by pouring blank water into a decontaminated glass beaker while the pump was running. One gallon of blank water was pumped through the system and discarded before filling the sample containers. A pump blank was analyzed for the fall only.

Six compounds were detected in the field blanks at concentrations greater than 5 times the corresponding method blank and at concentrations that exceeded some of the reclaimed water and groundwater samples (Table 7). Although the higher of the reclaimed water and groundwater concentrations recorded for these compounds may be real, the field blanks raised questions about potential for bias due to contamination. With the exception of thiabendazole, these compounds are reported here as being not detected in any of the reclaimed water or groundwater samples.

Table 7. Compounds Detected at Elevated Concentrations in Field Blanks (ng/L, parts per trillion).

Compound	Type and Date of Field Blank	Concentrations					
		Field Blank		Lab Blank		Range of Reclaimed Water Detections	Range of Groundwater Detections
Thiabendazole	Transfer* – Fall 2011	59		3	U	16 – 115,000	2.7 U – 9.0 U
Campesterol	Transfer[†] – Spring 2011	54		7.2	J	7.7 – 854	4.5 J – 331 J
B-Stigmastanol	"	18	J	3.1	J	27 – 444	7.6 NJ – 87 J
Stigmasterol	"	538		48		211 – 50,900	74 J – 2,550 NJ
Cholestanol	Pump** – Fall 2011	12	J	0.89	NJ	8.0 – 572	2.2 J – 235 J
Cholesterol	"	777		49	J	666 – 1,970	251 - 574

*prepared at LOTT

[†] prepared at Quincy

**prepared at Yelm

U: The analyte was not detected at or above the reported sample quantitation limit.

J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ: The analysis indicates the presence of an analyte that has been "tentatively identified," and the associated numerical value represents its approximate concentration.

In view of the high thiabendazole concentration consistently detected in reclaimed water from one facility, Quincy, and possible link to use in the local area, this compound is considered detected for that site only. All of the Table 7 compounds are included in Appendix E and flagged for potential field blank contamination where appropriate. Thiabendazole is discussed in more detail later in this report.

Variability of the Data

Short-term variability in reclaimed water quality was minimized by the use of composite samples. As discussed later in this report, the quality of reclaimed water changes from day to day. Therefore, the results reported here provide minimal insights into long-term variations in PPCP, hormone, and sterol concentrations in reclaimed water.

The monitoring wells were fully purged before sampling to obtain water samples representative of the surrounding aquifer. This also reduces the potential variability created by mixing stagnant well casing water with incoming groundwater. As with the reclaimed water, long-term groundwater data could likewise be affected by varying composition and loading rates of the reclaimed water, as well as seasonal fluctuation in the water table levels.

Precision estimates were obtained by analyzing duplicate samples prepared in the field, as previously described. Three samples were analyzed in duplicate: two reclaimed water samples and one groundwater sample. Because the duplicates were split samples rather than separate grabs collected at different times, the results primarily reflect analytical variability. Complete results for the duplicates are included in Appendix E.

The duplicates provided precision data on 79 compounds detected in one or more sample pairs. The results are summarized in terms of relative percent difference (RPD) in Figure 2. RPD is the difference between duplicates expressed as a percent of the mean value.

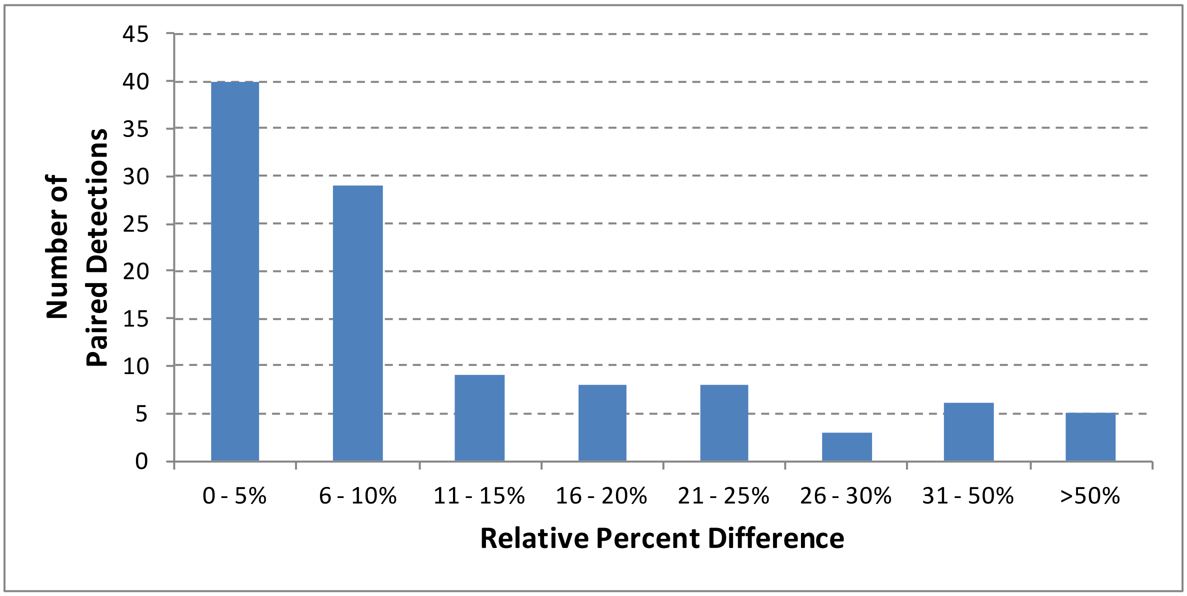

Figure 2. Summary of Precision Estimates from Duplicate Reclaimed Water and Groundwater Samples.

Duplicate analyses agreed with 15% on average, with a median RPD of 7%. In most instances (90%), the duplicates had an RPD of 30% or better. There was poor agreement between duplicates for the sterols campesterol and stigmasterol (RPDs of 135% and 146%, respectively). As already noted, AXYS often encounters poor duplication for sterols due to a variable and sometimes high analytical background.

There were 10 additional instances not depicted in Figure 2 where a compound was detected in only one of the sample duplicates. In most cases, the concentrations in question were in the region of the detection limit (Appendix E).

The average of duplicate results is used in the remainder of this report. Where one sample in a duplicate pair was non-detect, the detected result was used.

Results and Discussion

Reclaimed Water

Permit Parameters

Table 8 has the results on reclaimed water samples analyzed for selected parameters limited by the Reclaimed Water Permits issued to LOTT, Quincy, and Yelm. The flow data were obtained from the facility's discharge monitoring report (DMRs). Chloride does not have a permit limit, but was analyzed for potential use in interpreting the groundwater data.

Table 8. Results for Permit Parameters and Chloride in Reclaimed Water Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011.

Facility	Date (2011)	Flow (MGD)	TSS (mg/L)	Turbidity (NTU)	Chloride (mg/L)
LOTT	May 11	0.69	1 U	0.5 U	58
	October 5	0.56	1 U	0.8*	61
	Permit Limits	2.0	30/45[†]	0.2/0.5	NA
Quincy	May 24	1.22	4	1.3	114
	October 20	1.08	3	1.1	105
	Permit Limits	1.5	15/23	2/5**	NA
Yelm	October 6	0.32	1 U	1.0	64
	Permit Limits	1.0	30/NA	2/5	NA

MGD: million gallons per day
TSS: total suspended solids
NTU: nephelometric turbidity units
U: not detected at or above the reported result
NA: not applicable
*see discussion below
[†] average monthly/average weekly
**limit applies prior to disinfection

Flow, TSS, and turbidity were within allowable limits at all facilities at the time of this study. TSS was slightly elevated at Quincy compared to LOTT and Yelm, both in the spring and in the fall.

The October result for turbidity in LOTT's reclaimed water appears to be slightly above the permit limits. However, the instrument used to measure turbidity in these samples was not sensitive enough to accurately gauge compliance in the vicinity of 0.2-0.5 NTU. LOTT's in-situ instrument measured 0.05-0.06 NTU throughout that same day, and the LOTT laboratory result was 0.03 NTU in a grab sample (Karla Fowler, LOTT, personal communication). The reason for this large discrepancy is unknown. LOTT Martin Way consistently meets its permit limit for turbidity.

Pharmaceuticals, Personal Care Products, Hormones, and Sterols

Concentrations Detected

Reclaimed water was analyzed for 145 pharmaceuticals, personal care products, hormones, and sterols. Seventy-three compounds were reliably detected and quantified; 61 pharmaceuticals, 6 personal care products, 1 hormone, and 5 sterols (Table 9). The complete results for project samples showing all compounds analyzed can be found in Appendix E. Appendix F lists compounds that were not detected.

Thirty-four compounds were consistently found in reclaimed water across all facilities (Table 10). Fifteen of these – all pharmaceuticals – were detected in every sample, and an additional 19 were detected in at least one sample from each plant.

The most frequently observed compounds were 8 drugs (or metabolites) used to treat high blood pressure (antihypertensives). The next most commonly encountered were antidepressants and antibiotics (or metabolites), 5 and 4 compounds, respectively. Cocaine – which is more often used illegally than prescribed – was detected in all samples, as was one of its metabolites (benzoylecgonine). A second illegal drug, amphetamine, was found in Yelm reclaimed water.

Frequently detected compounds classed as personal care products included an insect repellant (DEET), a disinfectant primarily used in deodorant soaps (triclocarban), and nicotine, by way of a metabolite (cotinine). Hormones and sterols detected at all facilities were limited to 17*a*-dihydroequilin (estrogen replacement therapy), coprostanol (fecal sterol), and epicoprostanol (metabolite of coprostanol). Variable reporting limits likely resulted in underestimating the occurrence of hormones and sterols.

Concentrations of the target chemicals detected in this study spanned several orders of magnitude. Compounds present at concentrations of 100 ng/L or more in at least one reclaimed water sample are shown in Figures 3 and 4. Among the pharmaceuticals, concentrations exceeding 1,000 ng/L (one part per billion) were found for thiabendazole (fungicide), metformin (antidiabetic), theophylline (anti-asthmatic), meprobamate (tranquilizer), and 2-hydroxy-ibuprofen (ibuprofen metabolite). Several personal care products and sterols also exceeded 1,000 ng/L: *B*-sitosterol (plant sterol), coprostanol, caffeine, and a caffeine metabolite (1,7-dimethylxanthine). Many of the higher-level compounds were also among the most frequently detected.

Table 9. Summary of Results for Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Reclaimed Water Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011 (ng/L; parts per trillion).

Chemical	Class	LOTT				Quincy				Yelm	
		Spring*		Fall		Spring		Fall*		Fall	
Pharmaceuticals											
Acetaminophen	antipyretic	30	UJ	89	U	22	UJ	**32**		235	U
Albuterol	anti-asthmatic	0.6	U	1.9	U	**16**		**13**	**J**	**11**	
Alprazolam	anti-anxiety	**4.6**	**J**	**4.1**		1.3	UJ	0.57	U	**4.5**	
Amitriptyline	antidepressant	**1.2**	**J**	**0.73**		**21**	**J**	**32**		**31**	
10-hydroxy-amitriptyline	amitriptyline metabolite	**0.33**	**J**	1.0	U	**3.1**	**J**	**17**	**J**	**2.1**	
Amlodipine	antihypertensive	3.0	UJ	2.9	U	6.6	UJ	**5.3**		2.3	U
Amphetamine	stimulant	6.9	U	2.8	U	4.8	U	11	UJ	**15**	**NJ**
Atenolol	antihypertensive	**837**		**260**		**321**		**502**		**453**	
Atorvastatin	cholesterol lowering	3.0	U	2.8	U	**5.3**		**10**		2.2	U
Azithromycin	antibiotic	**14**	**J**	8.9	U	**189**	**J**	**71**		24	U
Betamethasone	anti-inflammatory	**5.1**	**J**	10	U	6.6	UJ	2.9	U	8.4	UJ
Carbamazepine	anti-epileptic	**477**	**J**	**298**	**J**	**170**	**J**	**514**		**216**	**J**
Cimetidine	antacid	1.5	UJ	1.1	UJ	**20**		**90**		0.9	U
Ciprofloxacin	antibiotic	37	UJ	36	U	**68**	**J**	**208**		94	U
Clarithromycin	antibiotic	**7.2**	**J**	9	U	**19**	**J**	**66**		**43**	
Cocaine	stimulant	**0.8**	**J**	**0.44**		**7.2**	**J**	**0.51**		**0.7**	
Benzoylecgonine	cocaine metabolite	**13**	**J**	**29**		**42**	**J**	**16**		**17**	
Codeine	analgesic	5.9	U	6.4	UJ	**14**	**J**	**41**		**28**	
Dehydronifedipine	nifedipine† metabolite	**16**	**J**	**8.3**		**6.7**	**J**	**2.5**		9.4	U
Diazepam	anti-anxiety	**1.6**	**J**	**1.8**		1.3	UJ	0.6	U	**2.1**	
Diltiazem	antihypertensive	0.6	UJ	1.8	U	**43**	**J**	**86**		**23**	
Desmethyldiltiazem	diltiazem metabolite	**0.6**	**J**	REJ		**12**	**J**	**14**		**81**	
Diphenhydramine	antihistamine	**7.8**	**J**	3.6	UJ	**166**	**J**	**353**	**J**	**127**	**J**
Doxycycline	antibiotic	12	U	40	U	**22**		**41**		32	UJ
Enalapril	antihypertensive	0.6	U	0.6	U	0.41	U	**2.1**		0.45	U

Chemical	Class	LOTT				Quincy				Yelm	
		Spring*		Fall		Spring		Fall*		Fall	
Erythromycin-H_2O	erythromycin** metabolite	**6.1**	**J**	1.8	U	**19**	**J**	**18**		**12**	
Fluoxetine	antidepressant	**31**	**J**	**13**	**J**	**12**	**J**	**8.3**		**27**	**J**
Norfluoxetine	fluoxetine metabolite	**3.7**	**J**	3.0	U	6.6	UJ	2.9	U	**4.2**	
Furosemide	diuretic	81	U	79	U	**263**		**444**		63	U
Gemfibrozil	antihyperlipidemic	**90**		**17**		**142**		**543**		**64**	
Glyburide	antidiabetic	**7.7**		6.0	U	4.3	U	5.7	U	4.7	U
Hydrochlorothiazide	diuretic	**224**	**J**	40	UJ	**117**	**J**	**84**		**147**	**J**
Hydrocodone	analgesic	3.0	U	2.8	UJ	**17**		**22**		**43**	**J**
Ibuprofen	anti-inflammatory	**126**		**80**		21	U	**171**		24	U
2-Hydroxy-ibuprofen	ibuprofen metabolite	162	U	159	U	**389**		**1,170**		125	U
Meprobamate	tranquilizer	**488**	**J**	**380**		**44**	**J**	**31**		**1,420**	
Metformin	antidiabetic	**1,090**		**804**		**3,320**		**1,030**		**2,080**	
Metoprolol	antihypertensive	**274**	**J**	**281**		**742**	**J**	**344**		**597**	
Miconazole	antifungal agent	3.0	UJ	8.9	U	2.2	UJ	**3.4**		24	U
Naproxen	anti-inflammatory	**11**		6.0	U	**101**		**253**		**24**	**J**
Norverapamil	anti-arrhythmic	**16**	**J**	**2.4**		**0.84**	**J**	**0.58**		**3.2**	
Ofloxacin	antibiotic	3.0	UJ	8.9	U	**24**	**J**	**12**		**47**	
Oxacillin	antibiotic	6.0	UJ	18	U	4.4	UJ	**14**		47	U
Oxycodone	analgesic	**13**		3.8	U	**20**		**41**		**55**	
Paroxetine	antidepressant	8.0	UJ	7.9	U	18	UJ	**10**		6.3	U
Propoxyphene	analgesic	**0.8**	**J**	0.6	U	1.3	UJ	0.57	U	**2.7**	
Propranolol	antihypertensive	**11**	**J**	4.0	U	**73**	**J**	**87**		**86**	
Ranitidine	antacid	1.2	U	1.1	U	**386**		**336**		1.8	UJ
Sertraline	antidepressant	**2.9**	**J**	**0.80**		**11**	**J**	**18**		**7.4**	
Sulfadiazine	antibiotic	3.0	UJ	8.9	U	**35**	**J**	**30**		24	U
Sulfadimethoxine	antibiotic	2.9	UJ	1.8	U	**7.6**	**J**	**62**		4.7	U
Sulfamethazine	antibiotic	4.0	UJ	5.8	UJ	**15**	**J**	6.8	UJ	9.4	U
Sulfamethoxazole	antibiotic	**40**	**J**	**20**		**505**	**J**	**164**		**155**	

Chemical	Class	LOTT				Quincy				Yelm	
		Spring*		Fall		Spring		Fall*		Fall	
Tetracycline	antibiotic	12	U	12	U	**38**		**34**		12	UJ
4-Epitetracycline	tetracycline metabolite	12	U	12	U	**26**		**33**		16	UJ
Thiabendazole	antifungal	REJ		REJ		**1,040**	**J**	**115,000**	**J**	REJ	
Theophylline	anti-asthmatic	**235**	**J**	**180**		265	UJ	**1,635**		**331**	
Triamterene	diuretic	**28**	**J**	1.9	U	**51**	**J**	**62**		**90**	**J**
Trimethoprim	antibiotic	**4.1**	**J**	8.9	U	**313**	**J**	**172**		**46**	
Valsartan	antihypertensive	**282**	**J**	**55**		**19**	**J**	**385**		**127**	
Verapamil	antihypertensive	**0.5**	**J**	0.30	U	**8.3**	**J**	**3.2**		**1.2**	
Personal Care Products											
Caffeine	stimulant	30	UJ	89	U	**79**	**J**	**1,210**		235	U
1,7-Dimethylxanthine	caffeine metabolite	120	UJ	357	U	88	UJ	**1,975**		940	U
Cotinine	nicotine metabolite	**26**		**23**		**31**		**212**		**17**	
DEET	insect repellant	26	UJ	**93**		**685**	**J**	**35**		**24**	
Triclocarban	disinfectant	**16**		**14**		4.3	U	**40**		**42**	
Triclosan	disinfectant	125	U	119	U	88	U	**169**		94	U
Hormones											
17*a*-Dihydroequilin	sex hormone	**13**	**J**	**15**		**64**		179	U	**12**	**J**
Sterols											
Desmosterol	cholesterol metabolite	1,090	UJ	109	U	**141**	**NJ**	2,250	U	**22**	**J**
Coprostanol	fecal sterol	1,080	UJ	**436**		**989**		**2,995**	**J**	**18**	**J**
Epicoprostanol	coprostanol metabolite	1,150	UJ	**50**	**J**	**85**	**J**	**137**	**J**	**11**	**J**
Ergosterol	fungi sterol	1,100	UJ	**10**	**NJ**	110	UJ	2,260	UJ	111	UJ
B-Sitosterol	plant sterol	1,090	UJ	**215**	**J**	1,120	U	**11,805**	**J**	110	UJ

*mean of duplicate samples. †antihypertensive **antibiotic

Bold: Analyte was detected.

U: The analyte was not detected at or above the reported sample quantitation limit.

UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.

J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ: The analysis indicates the presence of an analyte that has been "tentatively identified," and the associated numerical value represents its approximate concentration.

REJ: Data rejected due to field blank contamination.

Table 10. Pharmaceuticals, Personal Care Products, Hormones, and Sterols Most Consistently Detected in Reclaimed Water Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011 (ng/L, parts per trillion; N=5).

Chemical	Class	Maximum		Minimum	
100% detection frequency (detected in all samples)					
Amitriptyline	antidepressant	**32**		**0.73**	
Atenolol	antihypertensive	**837**		**260**	
Carbamazepine	anti-epileptic	**514**		**170**	**J**
Cocaine	stimulant	**7.2**	**J**	**0.44**	
Benzoylecgonine	cocaine metabolite	**42**	**J**	**13**	**J**
Fluoxetine	antidepressant	**31**	**J**	**8.3**	
Gemfibrozil	antilipemic	**543**		**17**	
Meprobamate	tranquilizer	**1,420**		**31**	
Metformin	antidiabetic	**3,320**		**804**	
Metoprolol	antihypertensive	**742**	**J**	**274**	**J**
Norverapamil	anti-arrhythmic	**16**	**J**	**0.58**	
Sertraline	antidepressant	**18**		**0.80**	
Sulfamethoxazole	antibiotic	**505**	**J**	**20**	
Valsartan	antihypertensive	**385**		**19**	**J**
Cotinine	nicotine metabolite	**212**		**17**	
80% detection frequency (found in at least one sample at each facility)					
10-Hydroxy-amitriptyline	amitriptyline metabolite	**17**	**J**	**0.33**	**J**
Clarithromycin	antibiotic	**66**		**7.2**	**J**
Dehydronifedipine	nifedipine* metabolite	**16**	**J**	**2.5**	
Desmethyldiltiazem	diltiazem* metabolite	**81**		**0.56**	**J**
Diphenhydramine	antihistamine	**353**	**J**	3.6	UJ
Erythromycin-H_2O	erythromycin† metabolite	**19**	**J**	1.8	U
Hydrochlorothiazide	diuretic	**224**	**J**	40	UJ
Naproxen	anti-inflammatory	**253**		6.0	U
Oxycodone	analgesic	**55**		3.8	U
Propranolol	antihypertensive	**87**		4.0	U
Theophylline	anti-asthmatic	**1,635**		**180**	
Triamterene	diuretic	**90**	**J**	1.9	U
Trimethoprim	antibiotic	**313**	**J**	**4.1**	**J**
Verapamil	antihypertensive	**8.3**	**J**	0.30	U
DEET	insect repellant	**685**	**J**	**24**	
Triclocarban	disinfectant	**42**		4.3	U
17*a*-Dihydroequilin	sex hormone	**64**		**12**	**J**
Coprostanol	fecal sterol	**2,995**	**J**	**18**	**J**
Epicoprostanol	coprostanol metabolite	**137**	**J**	**11**	**J**

*antihypertensive. †antibiotic **Bold:** Analyte was detected. U: The analyte was not detected at or above the reported sample quantitation limit. J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

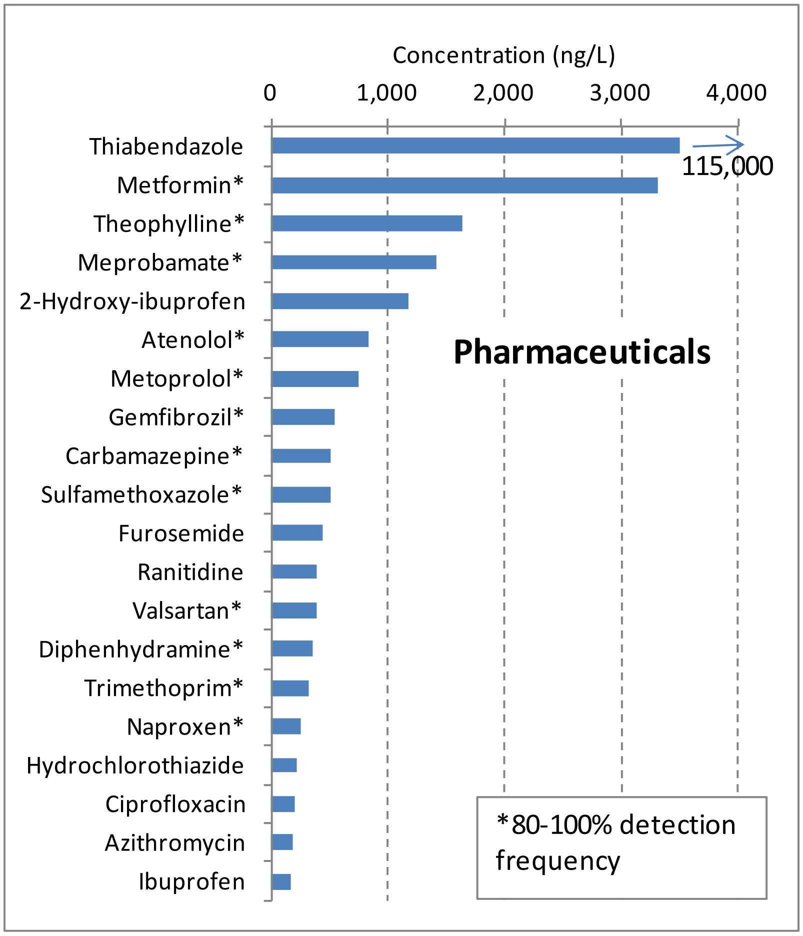

Figure 3. Maximum Concentrations of Pharmaceuticals in Reclaimed Water Samples from LOTT, Quincy, and Yelm (ng/L, parts per trillion; N =5).

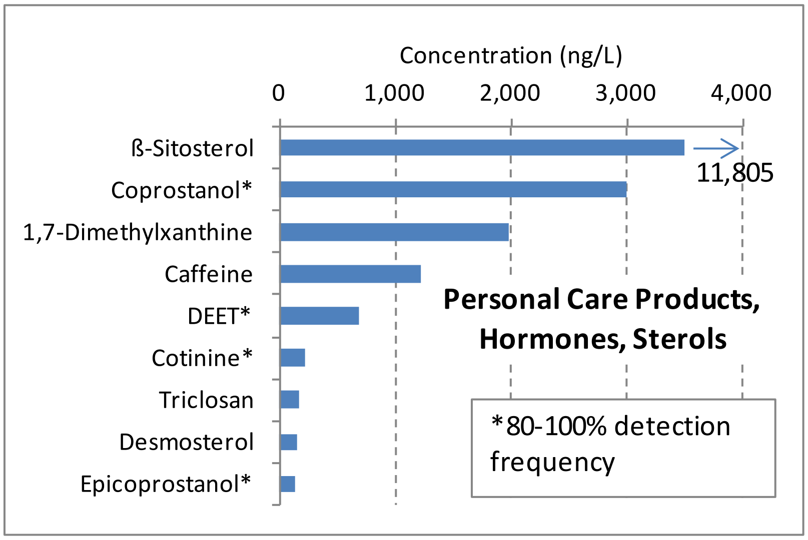

Figure 4. Maximum Concentrations of Personal Care Products, Hormones, and Sterols in Reclaimed Water Samples from LOTT, Quincy, and Yelm (ng/L, parts per trillion; N=5).

As described earlier in this report, thiabendazole was detected at 59 ng/L in a field blank for this project. The blank in question was prepared at the Quincy facility during the fall reclaimed water collection. Only slightly lower levels of thiabendazole were also found in the LOTT and Yelm reclaimed water samples (25-33 ng/L). Not having enough evidence to confirm the latter results as being accurate, these samples were rejected as potentially subject to contamination from sampling procedures or during sample transport.

Both of the Quincy reclaimed water samples had much higher thiabendazole concentrations, 1,040 ng/L in the spring and 115,000 ng/L in the fall, well above concentrations that could reasonably be attributed to contamination. The fall sample was analyzed in duplicate and the results agreed closely (111,000 and 119,000 ng/L).

Thiabendazole is a fungicide. Although it has some medicinal applications, major use is on fruits and vegetables. Thiabendazole salt is also used as a preservative in paints, carpets, adhesives, and textiles (EPA, 2002). Its detection in Quincy's reclaimed water could reflect local agriculture sources – the predominant land use in and around the service area – or it could be a relic of past use on facility property.

Thiabendazole was not detected in groundwater at Quincy, LOTT, or Yelm. EPA (2002) concluded that the extremely high soil-water partitioning coefficients values of thiabendazole tend to reduce its potential to leach through soils and contaminate groundwater.

Facility Differences

The number of reclaimed water samples analyzed for this study was too limited to draw strong conclusions about differences between facilities. Higher concentrations of measured parameters were generally found in the reclaimed water produced by Quincy and Yelm than at LOTT.

Figure 5 plots the fall data for the 15 compounds consistently detected in reclaimed water. In these samples, Quincy had 8 of the 15 highest concentrations, followed by Yelm with 6 of the highest concentrations. Only Quincy and LOTT were sampled in the spring, at which time Quincy recorded 9 of the highest concentrations for these 15 compounds.

The membrane bioreactors and longer solids retention time at LOTT may result in a higher removal efficiency than the sequencing batch reactors at Quincy and Yelm. Biological treatment systems with a longer solids age retain the slow growing organisms that can better degrade complex organic compounds (Karla Fowler, LOTT, personal communication). Other evidence of improved water quality can be seen in the lower TSS and turbidity levels in LOTT's reclaimed water compared to Quincy or Yelm (see Table 8). Personal habits of those in the LOTT service area may also be different.

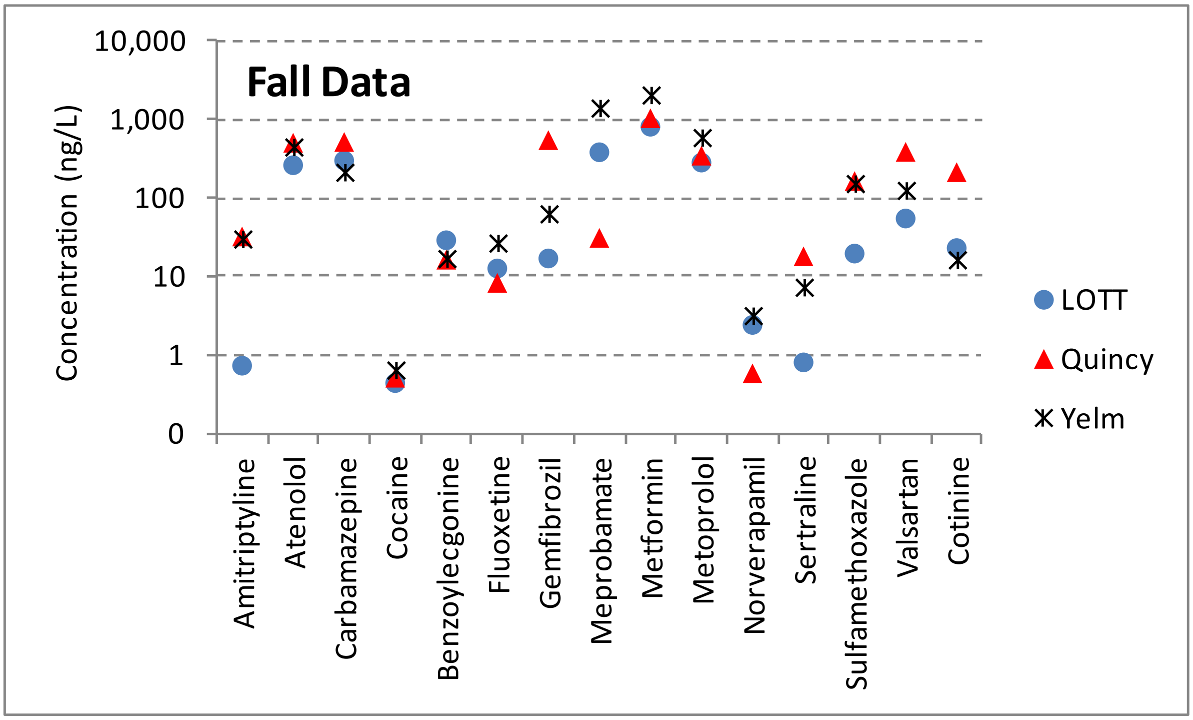

Figure 5. Relative Concentrations of the 15 Compounds Consistently Detected in Reclaimed Water, Fall 2011 (ng/L, parts per trillion; log scale).

Seasonal Patterns

Data for both spring and fall were obtained only for LOTT and Quincy. Looking again at the 15 top compounds, spring exhibited higher concentrations for 70% of the results, typically by a factor of 2 or more. Two possible reasons for higher PPCP content in the spring are the effect of lower temperatures on biochemical reaction rates and lower hydraulic retention time due to increased flow-through rates. Here again, however, only a few (four) samples were analyzed. A similar pattern was not seen in the groundwater data from these two facilities.

A recent study of tertiary treated wastewater in California showed there can be substantial variability in pharmaceuticals, personal care products, and estrogens over the course of even a daily cycle (Nelson et al., 2011). Some compounds exhibited standard deviations exceeding 100% of their daily means (e.g., sulfamethoxazole, trimethoprim); others had less extreme cycles (e.g., atenolol, gemfibrozil). Day-to-day and seasonal differences were also apparent, although the patterns were not defined. Only a few chemicals were relatively refractory (e.g., carbamazepine, fluoxetine). Nelson et al. cautioned against using limited numbers of samples to base conclusions about concentration levels and patterns.

Comparison with 2008 Data

The only other source of data on pharmaceuticals, personal care products, hormones, or sterols in reclaimed water from these facilities is restricted to a single LOTT reclaimed water sample in August 2008 from the Lubliner et al. (2010) study. The 2008 study also conducted a duplicate analysis of the reclaimed water produced by LOTT's Budd Inlet facility which serves the same community. The same laboratory and methods were used in 2008 as in the current study. Results for compounds analyzed in common by these two efforts are compared in Table 11.

Eleven of the 14 chemicals detected in the 2008 LOTT Martin Way reclaimed water sample were also detected in the present study, the exceptions being desmosterol, thiabendazole, and carbadox. The 2011 data on desmosterol suffered from poor detection limits in the spring. Thiabendazole was detected at 30-33 ng/L – similar to the 2008 result of 24 ng/L – but these data were rejected due to a contaminated field blank. Carbadox is an antimicrobial agent.

Eight compounds were found in Martin Way reclaimed water in 2011 but not in 2008. For the most part, the 2011 concentrations were in the vicinity of detection limits achieved in 2008. An additional 24 compounds determined in the present study were not analyzed in 2008.

For those compounds detected at Martin Way in both 2011 and 2008, most concentrations are in remarkably good agreement, given that studies were conducted three years apart. In a number of instances, the concentrations differ by a factor of 2 or less. Overall, these data suggest that, at least for LOTT Martin Way, most of these compounds do not exhibit extreme variability over time.

All of the compounds reported in the 2008 reclaimed water samples from the LOTT Budd Inlet plant were also detected in their Martin Way samples or in the current study. For the most part, the concentrations were not markedly different.

Table 11. Comparison of LOTT Reclaimed Water Samples Analyzed in the Present Study and by Lubliner et al. (2010) (ng/L, parts per trillion).

Source:	Present Study				Lubliner et al. (2010)			
Facility	LOTT Martin Way				LOTT Martin Way		LOTT Budd Inlet*	
Sampling Date:	May 2011		Oct 2011		Aug 2008		Aug 2008	
Detected at LOTT Martin Way in 2011 and 2008								
Carbamazepine	**477**	**J**	**298**	**J**	**917**		**1,600**	
Dehydronifedipine	**16**	**J**	**8.3**		**14**		84	UJ
Diphenhydramine	**7.8**	**J**	3.6	UJ	**4.1**		17	UJ
Fluoxetine	**31**	**J**	**13**	**J**	**62**		**42**	
Gemfibrozil	**90**		**17**		**47**		5.7	UJ
Ibuprofen	**126**		**80**		**74**		**30**	
Metformin	**1,090**		**804**		**1,760**	**J**	**542**	**J**
Sulfamethoxazole	**40**	**J**	**20**		**104**		**73**	**J**
Cotinine	**26**		**23**		**29**		22	UJ
Triclocarban	**16**	**J**	**14**		**103**		2.7	U
Coprostanol	1,080	UJ	**436**		**7.1**	**J**	**34**	
Detected at LOTT Martin Way in 2011 Only								
Azithromycin	**14**	**J**	8.9	U	6.9	U	**12**	**J**
Clarithromycin	**7.2**	**J**	9	U	5.0	UJ	4.5	U
Erythromycin-H_2O	**6.1**	**J**	1.8	U	3.6	U	**9.1**	
Trimethoprim	**4.1**	**J**	8.9	U	12	U	**73**	**J**
17*a*-Dihydroequilin	**13**	**J**	**15**		0.85	U	0.46	U
Epicoprostanol	1,150	UJ	**50**	**J**	2.0	U	**14**	**J**
Ergosterol	1,100	UJ	**10**	**NJ**	1.5	U	**1.7**	
B-Sitosterol	1,090	UJ	**215**	**J**	11	U	10	U
Detected at LOTT Martin Way in 2008 Only								
Carbadox	3.0	UJ	8.9	U	**27**		65	UJ
Thiabendazole	REJ		REJ		**24**		**22**	
Desmosterol	1,090	UJ	109	U	**7.1**	**J**	**148**	

Note: Twenty-four additional compounds were detected at LOTT Martin Way in present study but not analyzed by Lubliner et al. (see Table 8)
*mean of duplicates
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
REJ: Rejected due to field blank contamination.

Other Reclaimed Water Data

Table 12 compares results of the present study with similar data from other western states. Concentrations are summarized for chemicals commonly detected among these investigations. In many instances, the concentrations reported in discharges from reclaimed water facilities in Idaho and California are broadly comparable to present study findings for LOTT, Quincy, and Yelm. No reclaimed water data could be located for Oregon facilities.

Table 12. Comparison of Concentrations Reported for Selected, Frequently Detected Chemicals in Other Recent Studies of Reclaimed Water (ng/L, parts per trillion).

Location:	Washington	Idaho	California	
Reference:	Present Study	Lubliner et al. (2010)	Nelson et al. (2011)	SWRCB (2010)
No. of samples:	5	1	36*	5 - 23**
Atenolol	260 - 837	- -	487 - 929	1,780
Carbamazepine	170 - 514	918	223 - 297	900
DEET	24 - 685	- -	148 - 357	1,520
Erythromycin-H_2O	<1.8 - 19	168	76 - 110	- -
Fluoxetine	8.3 - 31	58	18 - 22	31
Gemfibrozil	17 - 543	1,230	215 - 773	3,550
Meprobamate	31 - 1,420	- -	- -	430
Metoprolol	274 - 742	- -	245 - 277	- -
Naproxen	<6.0 - 253	251	11 - 41	851
Propranolol	<4.0 - 87	- -	19 - 25	25
Sulfamethoxazole	20 - 505	2.0	18 - 265	1,400
Triclocarban	<4.3 - 42	52	200 - 231	- -
Trimethoprim	4.1 - 313	294	<10 - 59	- -

*range of means
**90th percentile
- - no data
SWRCB: State Water Resources Control Board

As noted elsewhere in this report, hormone levels were not well characterized by the present study. Due to the low hormone levels in treated wastewater, this shortcoming is commonly encountered in similar investigations.

Drewes et al. (2006) concluded that secondary and tertiary treatment was efficient at removing hormones in water reclamation processes. They found that estrogenic activity remaining after treatment was primarily accounted for by 17*B*-estradiol and estrone, whereas androgenic activity was caused by testosterone. None of these compounds were reliably quantified at LOTT, Quincy, or Yelm. Testosterone was detected in Quincy groundwater (see following discussion).

Groundwater

Field Measurements

Field measurements were taken at each site to assess groundwater conditions before and during well purging. Depth-to-water was measured in each monitoring well prior to purging. Measurements are listed in Appendix E, Table E-1. Water levels were within the screen interval of the Quincy and Yelm wells and only one of the LOTT wells. Measured water levels were above the screen interval in the other three LOTT wells.

Samples were collected after field parameter readings stabilized. End of purge pH, dissolved oxygen, specific conductance, and temperature readings are listed in Table E-1. Stabilized field parameter measurements were within expected ranges for groundwater of each area.

Chloride

Groundwater samples were analyzed for chloride as an indicator of the presence of reclaimed water. Although chloride is naturally occurring, there are many anthropogenic sources, such as wastewater, that make chloride a useful indicator of human-induced groundwater quality changes. Chloride is a good indicator parameter because it can migrate through groundwater relatively unaltered.

Chloride results are presented in Table 13. The reclaimed water chloride concentrations have been included for comparison.

Table 13. Chloride Concentrations in Reclaimed Water and Groundwater Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011 (mg/L, parts per million).

Spring	Fall	Spring	Fall	Spring	Fall	Spring	Fall
LOTT							
Reclaimed Water		LOTTMW-8		LOTTMW-9		LOTTMW-1	LOTTMW-2
58	61	47	54	36	55	50	20
Quincy							
Reclaimed Water		Quinwo-1†		Quinw-26		Quinw-60	
114	105	6.7	10	129	123	97	133
Yelm							
Reclaimed Water		YELMW-1†		YELMW-3		YELMW-4	
--	64	--	8.6	--	44	--	57

-- Not sampled.
† Up-gradient well

With the exception of well LOTTMW-2, chloride concentrations in the other three LOTT wells are similar to those found in the reclaimed water samples.

Chloride concentrations in Quincy well Quinwo-1, which is outside the recharge area, were low, increasing to concentrations similar to those reported in the reclaimed water samples in the two wells within the recharge area.

Chloride was detected in up-gradient well YELMW-1 at a concentration of 8.6 mg/L, increasing to 44 mg/L and 57 mg/L in the down-gradient wells. Concentrations for the wells within the recharge area are also similar to those in the reclaimed water.

Pharmaceutical, Personal Care Products, Hormones, and Sterols

Of the 67 PPCPs found in process water from the three reclaimed water facilities, only 11 were detected in groundwater samples from the recharge basins. Two hormones and two sterols were also reliably identified. All project groundwater results can be found in Appendix E. Compounds that were not detected in any of the samples are listed in Appendix F.

A summary of the groundwater results for pharmaceuticals, personal care products, hormones, and sterols are presented in Tables 14 (LOTT), 15 (Quincy), and 16 (Yelm).

LOTT

Nine pharmaceuticals were detected in groundwater samples from LOTT, four of which were reported in every sample: benzoylecgonine, carbamazepine, meprobamate, and sulfamethoxazole. Alprazolam (anti-anxiety) was found in all but one sample.

Dehydronifedipine (nifedipine metabolite) was detected in all three spring samples and sulfanilamide (antibiotic) in three of the four sampled wells.

Two sterols, desmosterol (cholesterol metabolite) and *B*-sitosterol (plant sterol), were identified. *B*-Sitosterol was detected in well LOTTMW-8 at relatively high concentrations. No hormones were reliably found in any of the LOTT groundwater samples.

As previously discussed, at the time of this study there was no suitable up-gradient well at the LOTT facility. This is partly due to the mounding effect the recharge infiltration basins have on local groundwater flow directions. Well LOTTMW-1 was initially selected because it would be considered up-gradient for the regional groundwater flow direction. This well was dry during the fall sampling. For comparison, well LOTTMW-2 was sampled in the fall. Although this well is located in the center of the recharge basin, fewer compounds were reported.

Table 14. Summary of Results for Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Groundwater Samples Collected at LOTT in May and October, 2011 (ng/L, parts per trillion).

Chemical	Concentrations											
	LOTTMW-8				LOTTMW-9				LOTTMW-1		LOTTMW-2	
	Spring		Fall*		Spring		Fall		Spring		Fall	
Pharmaceuticals												
Alprazolam	**1.1**	**J**	**0.7**		**1.2**	**J**	**1.4**		**1.4**	**J**	0.6	U
Amphetamine	3	U	3	U	3	U	**6.0**	**NJ**	3.8	U	2.9	U
Benzoylecgonine	**1.9**	**J**	**1.5**		**1.3**	**J**	**1.9**		**1.4**	**J**	**1**	
Betamethasone	3	UJ	10	U	3.3	UJ	9.8	U	**3.2**	**J**	9.8	U
Carbamazepine	**184**	**J**	**185**	**J**	**122**	**J**	**206**		**187**	**J**	**56**	
Dehydronifedipine	**4.3**	**J**	3.6	U	**3.9**	**J**	3.5	U	**4.5**	**J**	3.5	U
Meprobamate	**30**	**J**	**28**		**18**	**J**	**34**		**18**	**J**	**12**	
Sulfamethoxazole	**324**	**J**	**422**		**275**	**J**	**438**		**497**	**J**	**95**	
Sulfanilamide	**34**	**J**	**112**		28	UJ	**118**		**61**	**J**	88	U
Personal Care Products												
DEET	7.9	UJ	**15**		8.8	UJ	11	U	8.6	UJ	3.8	U
Sterols												
Desmosterol	109	UJ	106	U	116	UJ	106	U	549	UJ	**21**	**J**
B-Sitosterol	**10,300**	**J**	**1,240**	**J**	1,480	UJ	113	UJ	698	UJ	109	UJ

* mean of duplicate samples
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been "tentatively identified," and the associated numerical value represents its approximate concentration.

Quincy

At the Quincy facility, five pharmaceuticals were identified in groundwater samples collected from the two wells within the recharge area. Carbamazepine and sulfamethoxazole were detected in both the spring and fall. Meprobamate and sulfanilamide were also consistently reported. Sulfadimethoxine was detected once at a concentration near the reporting limit. No pharmaceuticals were found in up-gradient well Quinwo-1.

Two hormones, progesterone (tentatively identified) and testosterone, were each identified once in well Quinw-26. These are the only hormones found in any of the groundwater samples for this study.

Desmosterol and *B*-sitosterol were also identified in Quincy groundwater samples. Desmosterol was the only compound tentatively identified in up-gradient well Quinwo-1. *B*-Sitosterol was again detected at relatively high concentrations in the down-gradient wells. The inconsistent presence of *B*-sitosterol in the reclaimed water samples may indicate a source that is unrelated to the treatment facility. The Quincy facility is surrounded by agricultural areas.

Table 15. Summary of Results for Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Groundwater Samples Collected at Quincy in May and October, 2011 (ng/L, parts per trillion).

Chemical	Concentrations											
	Up-gradient				Down-gradient							
	Quinwo-1				Quinw-26				Quinw-60			
	Spring		Fall		Spring		Fall		Spring		Fall	
Pharmaceuticals												
Carbamazepine	3.1	UJ	2.9	U	**454**	**J**	**431**		**288**	**J**	**332**	
Meprobamate	8.2	UJ	7.8	U	**19**	**J**	**11**		7.2	UJ	7.9	U
Sulfadimethoxine	2.1	UJ	0.6	U	2.7	UJ	1.6	UJ	2.2	UJ	**2.0**	
Sulfamethoxazole	4.1	UJ	1.2	U	**260**	**J**	**41**	**J**	**19**	**J**	**59**	
Sulfanilamide	31	UJ	29	U	**45**	**J**	**48**		27	UJ	**56**	
Hormones												
Progesterone	22	U	24	U	**13**	**NJ**	486	U	29	U	474	U
Testosterone	9	U	9.7	U	9.2	U	**24**	**J**	12	U	190	U
Sterols												
Desmosterol	**4.3**	**NJ**	120	U	**3.4**	**J**	2,410	U	**7.4**	**NJ**	2,350	U
B-Sitosterol	111	U	120	UJ	628	U	**13,400**	**J**	320	U	**4,290**	**J**

Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been "tentatively identified," and the associated numerical value represents its approximate concentration.

Yelm

At the Yelm facility, four pharmaceuticals were found in groundwater samples collected from the two wells within the recharge area. Carbamazepine, meprobamate, and sulfamethoxazole were detected in both down-gradient wells. No pharmaceuticals were reported in up-gradient well YELMW-1.

The insect repellant DEET was also found in the two down-gradient wells.

B-Sitosterol was detected in all three wells at relatively high concentrations. *B*-Sitosterol was the only compound indentified in up-gradient well YELMW-1. *B*-Sitosterol was not found in the corresponding reclaimed water sample. The Yelm recharge area is a well maintained public park. The source of the *B*-sitosterol may be unrelated to the infiltration of the treated reclaimed water.

No hormones were reliably detected in any of the Yelm groundwater samples.

Table 16. Summary of Results for Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Groundwater Samples Collected at Yelm in October 2011 (ng/L, parts per trillion).

Chemical	Concentrations					
	Up-gradient		Down-gradient			
	YELMW-1		YELMW-3		YELMW-4	
	Fall		Fall		Fall	
Pharmaceuticals						
Alprazolam	0.5	U	0.6	U	**0.8**	
Carbamazepine	7.7	U	**77**		**118**	**J**
Meprobamate	6.9	U	**152**		**190**	
Sulfamethoxazole	3.1	U	**26**		**39**	
Personal Care Products						
DEET	3	U	**19**		**21**	
Sterols						
B-Sitosterol	**376**	**J**	**367**	**J**	**762**	**J**

Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

Detection Frequencies

Although a large number of PPCPs were reported in the reclaimed water samples, these chemicals were found infrequently and generally at much lower concentrations in the groundwater samples from within the recharge area. Detection frequencies and concentration ranges for the 15 chemicals identified in the groundwater samples are presented in Table 17. Only two of the 15 compounds were detected in either of the wells outside the recharge area.

Carbamazepine, meprobamate, sulfamethoxazole, sulfanilamide, and *B*-sitosterol were the most frequently detected chemicals in groundwater. These chemicals also had some of the highest concentrations, as shown in Table 17 and Figure 6. Dehydronifedipine, benzoylecgonine, alprazolam, desmosterol, and DEET were also regularly detected in groundwater from at least one facility, but at lower concentrations.

Table 17. Detection Frequency and Concentration Range of Pharmaceuticals, Personal Care Products, Hormones, and Sterols Detected in Groundwater Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011 (ng/L, parts per trillion; N = 15).

Chemical	Class	Overall Detection Frequency*	Concentrations (ng/L)			
			Maximum		Minimum	
Pharmaceuticals						
Alprazolam	anti-anxiety	40%	**1.4**	**J**	0.5	U
Amphetamine	stimulant	7%	**6.0**	**NJ**	2.5	U
Benzoylecgonine	cocaine metabolite	40%	**1.9**	**J**	0.5	U
Betamethasone	anti-inflammatory	7%	**3.2**	**J**	2.7	UJ
Carbamazepine	anti-epileptic	80%	**454**	**J**	2.9	UJ
Dehydronifedipine	nifedipine† metabolite	20%	**4.5**	**J**	1.2	U
Meprobamate	tranquilizer	67%	**190**		6.9	U
Sulfadimethoxine	antibiotic	7%	**2.0**		0.6	U
Sulfamethoxazole	antibiotic	80%	**497**	**J**	1.2	U
Sulfanilamide	antibiotic	47%	**118**		27	UJ
Personal Care Products						
DEET	insect repellant	20%	**21**		2.5	UJ
Hormones						
Progesterone	sex hormone	7%	486	U	**13**	**NJ**
Testosterone	sex hormone	7%	**24**	**J**	8.6	U
Sterols						
Desmosterol	cholesterol metabolite	27%	**21**	**J**	**3.4**	**J**
B-Sitosterol	plant sterol	47%	**13,400**	**J**	109	UJ

* Detection frequency based on all 15 groundwater samples.
† antihypertensive
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been “tentatively identified,” and the associated numerical value represents its approximate concentration.

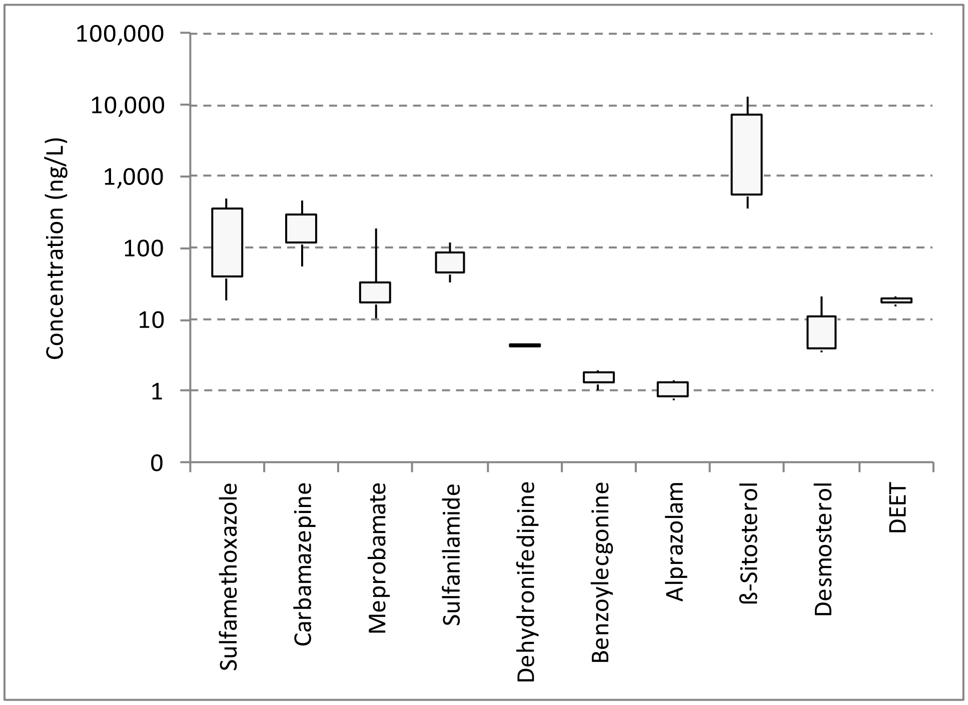

Figure 6. Concentration Range of Frequently Detected Pharmaceuticals, Personal Care Products, Hormones, and Sterols in Groundwater Samples Collected at LOTT, Quincy, and Yelm in May and October, 2011 (ng/L, parts per trillion; N=15).

Figures 7 and 8 present the maximum concentrations of the 15 chemicals indentified in the groundwater samples.

As mentioned before, of the pharmaceuticals, sulfamethoxazole, carbamazepine, meprobamate, and sulfanilamide had the greatest detection frequency and concentrations of the 15 compounds found in the groundwater samples. With the exception of sulfanilamide, these chemicals were also found in all the reclaimed water samples collected for this study.

Amphetamine, betamethasone, and sulfadimethoxine were each detected once out of the 15 groundwater samples collected over the study period. These compounds were reported at relatively low concentrations (Table 17).

Progesterone and testosterone were the only two hormones reliably identified in the groundwater samples. Each compound was detected once in groundwater and was not found in any of the reclaimed water samples. As discussed previously, high and variable reporting limits could have resulted in underestimating the occurrence of hormones for this project.

Sterols may also be underreported due to variable reporting limits. Desmosterol and *B*-sitosterol are the only two reliably identified sterols in groundwater. Desmosterol was found in the reclaimed water at all three facilities. It was detected in groundwater samples at LOTT and Quincy at much lower concentrations. *B*-Sitosterol was detected in approximately half the groundwater samples and at the highest concentrations reported for the study. *B*-Sitosterol was inconsistently reported in reclaimed water.

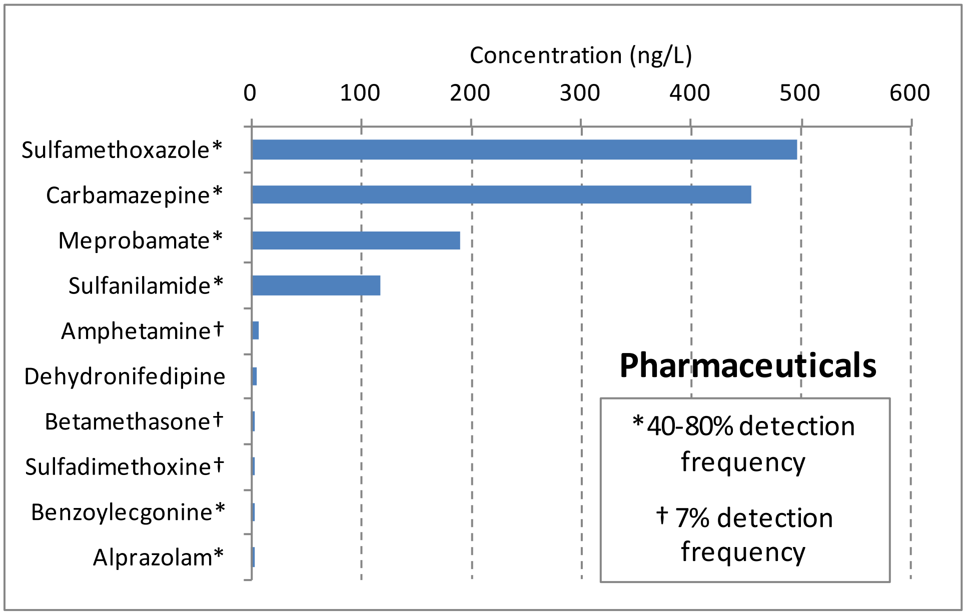

Figure 7. Maximum Concentrations of Pharmaceuticals in Groundwater Samples Collected at LOTT, Quincy, and Yelm (ng/L, parts per trillion; N=15).

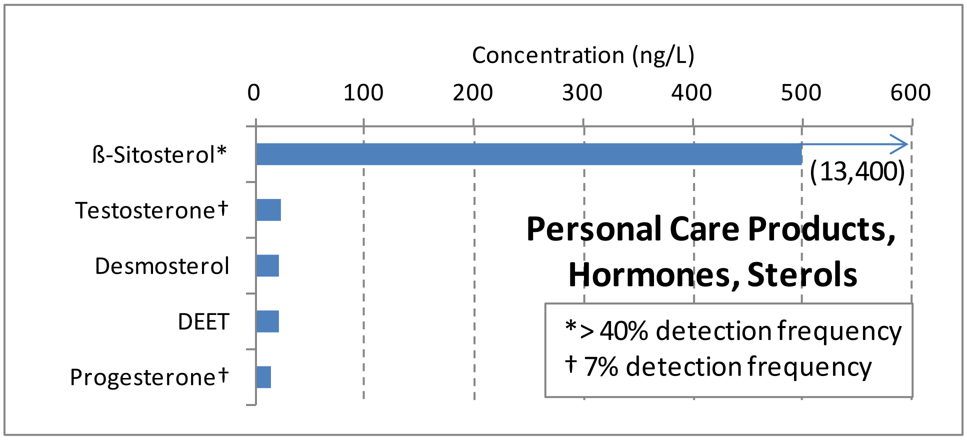

Figure 8. Maximum Concentrations of Personal Care Products, Hormones, and Sterols in Groundwater Samples Collected at LOTT, Quincy, and Yelm (ng/L, parts per trillion; N=15).

Facility Differences

As with reclaimed water, the number of groundwater samples collected during this study are too limited to draw many conclusions about differences between facilities. Figures 9 and 10 plot the maximum concentrations of compounds found more than once (i.e., >7% detection frequency).

In the spring, eight of the compounds shown in Figure 9 were reported in samples collected from LOTT and five at Quincy. Four compounds (sulfamethoxazole, carbamazepine, meprobamate, sulfanilamide) were found at both locations. Of those four, three of the highest concentrations were detected at LOTT.

In the fall, nine compounds were found at LOTT, five at Quincy, and six at Yelm, as shown in Figure 10. Sulfamethoxazole, carbamazepine, meprobamate, and *B*-sitosterol were again reported at all locations. Of those four, two of the highest concentrations were detected in Quincy, one at LOTT, and one at Yelm. As in the fall, sulfanilamide was detected at both LOTT and Quincy at similar concentrations. Sulfanilamide was not reported in any of the Yelm samples.

Overall, the most compounds and the highest concentrations have been reported in groundwater samples collected at LOTT. There is not enough data and too many variables to explain why LOTT has the lowest number of compounds and concentrations found in reclaimed water but the greatest number of compounds and concentrations found in groundwater.

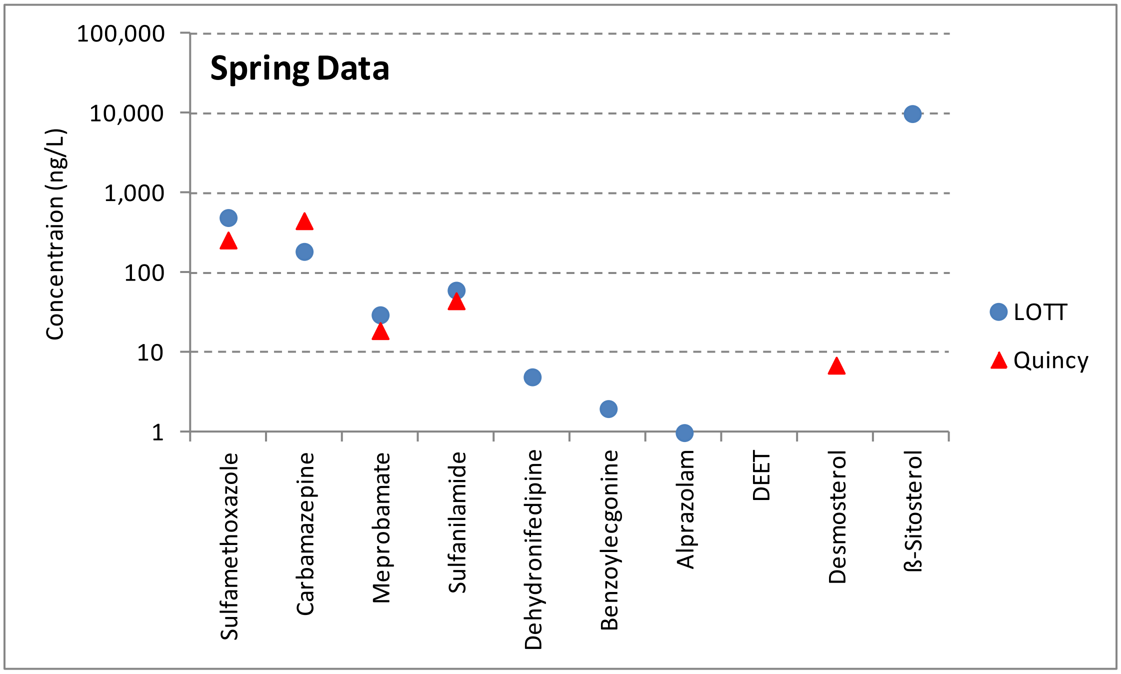

Figure 9. Relative Concentrations of Compounds Consistently Detected in Groundwater, Spring 2011 (ng/L, parts per trillion; log scale).

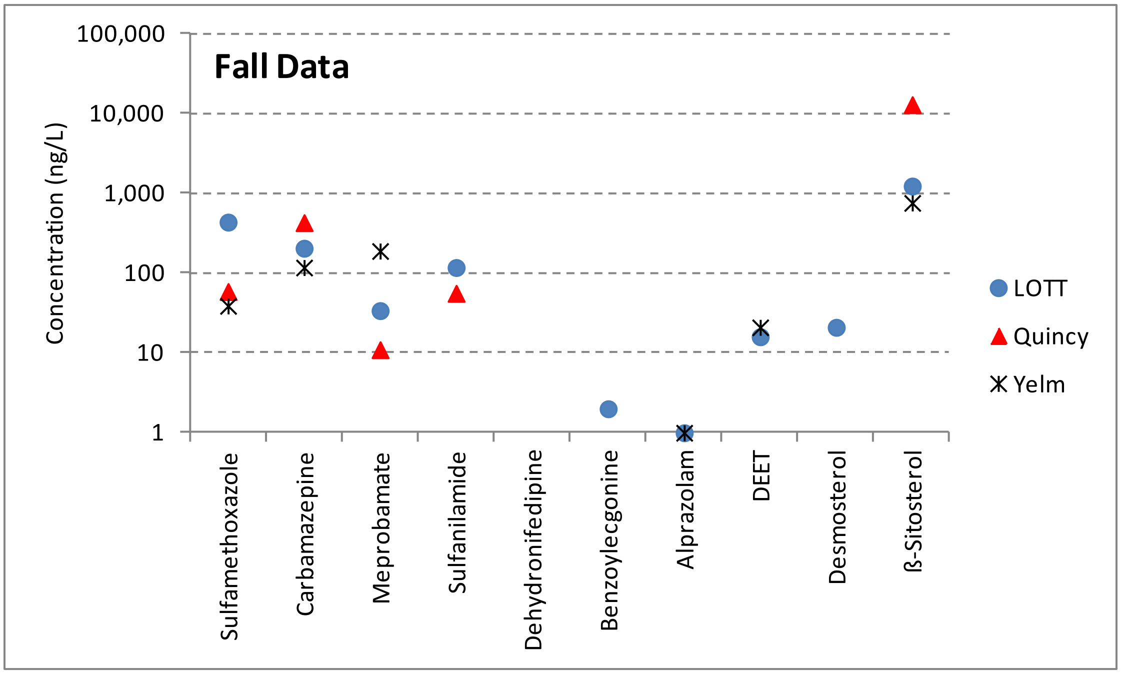

Figure 10. Relative Concentrations of Compounds Consistently Detected in Groundwater, Fall 2011 (ng/L, parts per trillion; log scale).

Seasonal Patterns

Based on the limited data collected from the LOTT and Quincy facilities, there was no clear pattern in the analytes that were detected in both the spring and fall sampling. In general, for analytes that were detected during both sample events, concentrations appear similar. But, as with the reclaimed water samples, the number of groundwater samples collected during this study is too limited to draw strong conclusions about seasonal patterns in compound detections or concentrations. A longer monitoring period is required to determine if concentrations are affected by factors such as varying composition and loading rates of reclaimed water and fluctuations in the seasonal high and low water table levels.

Other Reclaimed Water / Groundwater Studies

Results from this project seem to support what has been determined in previous studies, that many PPCPs are sufficiently removed during tertiary wastewater treatment and groundwater recharge process (Drewes et al., 2003; Kinney et al., 2006b; Hinkle et al., 2009; Lubliner et al., 2010).

Drewes et al. (2003) found that pharmaceuticals such as ibuprofen, naproxen, and gemfibrozil have a high potential for biodegradation during groundwater recharge. Other compounds such as carbamazepine and sulfamethoxazole appear to have a low removal rate during wastewater treatment and leach through the subsurface to the groundwater relatively unaltered (Kinney et al., 2006b; Drewes et al., 2003).

Studies suggest that how a chemical behaves in the subsurface is compound-specific and may be influenced by interaction with subsurface materials through processes such as sorption, degradation, or dispersion. Kinney et al. (2006b) found factors that may determine a chemical’s response in the subsurface include the physical properties of the soil, depth to the water table, and the ability of microfauna to degrade the pharmaceuticals.

Composition and concentrations of the pharmaceuticals in the treated effluent, as well as the recharge frequency, are also important factors in determining the possible presence of a pharmaceutical in the groundwater. Composition of the effluent is expected to fluctuate with seasons and changes in treatment operation efficiency.

Co-occurrence of Pharmaceuticals, Personal Care Products, Hormones, and Sterols in Reclaimed Water and Groundwater

The co-occurrence of PPCPs, hormones, and sterols in reclaimed water and groundwater at LOTT, Quincy, and Yelm is examined in Table 18. Concentrations in monitoring wells within the recharge area are compared with those measured in corresponding reclaimed water samples. The Quincy and Yelm up-gradient wells have not been included since no PPCPs were detected.

The pharmaceuticals carbamazepine, meprobamate, and sulfamethoxazole were detected at elevated concentrations in all, or almost all, samples at each facility. Alprazolam had detections in both reclaimed water and groundwater at LOTT and Yelm but not in any samples from Quincy. Three additional pharmaceuticals co-occurred at LOTT: benzoylecgonine, dehydronifedipine, and, to a limited extent, betamethasone. One pharmaceutical in reclaimed water – sulfadimethoxine – had a single trace detection in Quincy groundwater.

The insecticide DEET co-occurred in reclaimed water and groundwater at LOTT and Yelm, but not Quincy, despite relatively high concentrations measured in Quincy's reclaimed water. No additional personal care products were detected in groundwater during this study.

Compounds detected in groundwater, but not in reclaimed water, included sulfanilamide, progesterone, and testosterone. This may be due to higher detection limits in reclaimed water samples (progesterone/testosterone) or could indicate reclaimed water variability (sulfanilamide). None of these compounds were detected in up-gradient wells, indicating the sources are likely site related.

Table 18. Co-occurrence of Pharmaceuticals, Personal Care Products, Hormones, and Sterols in Reclaimed Water and Groundwater at LOTT, Quincy, and Yelm, 2011 (ng/L, parts per trillion).

Chemicals Detected in Groundwater	LOTT								Quincy						Yelm		
	Reclaimed Water		Groundwater						Reclaimed Water		Groundwater				Reclaimed Water	Groundwater	
	Spring	Fall	LOTTMW-8 Spring	LOTTMW-8 Fall	LOTTMW-9 Spring	LOTTMW-9 Fall	LOTTMW-1 Spring	LOTTMW-2 Fall	Spring	Fall	Quinw-26 Spring	Quinw-26 Fall	Quinw-60 Spring	Quinw-60 Fall	Fall	YELMW-3 Fall	YELMW-4 Fall
Pharmaceuticals																	
Carbamazepine	**477**	**298**	**184**	**185**	**122**	**206**	**187**	**56**	**170**	**514**	**454**	**431**	**288**	**332**	**216**	**77**	**118**
Meprobamate	**488**	**300**	**30**	**28**	**18**	**34**	**18**	**12**	**44**	**31**	**19**	**11**	<7.2	<7.9	**1,420**	**152**	**190**
Sulfamethoxazole	**40**	**20**	**324**	**422**	**275**	**438**	**497**	**95**	**505**	**164**	**260**	**41**	**19**	**59**	**155**	**26**	**39**
Alprazolam	**4.6**	**4.1**	**1.1**	**0.73**	**1.2**	**1.4**	**1.4**	<5.9	<1.3	<0.57	<0.54	<0.59	<0.54	<0.59	**4.5**	<0.55	**0.75**
Benzoylecgonine	**13**	**29**	**2.0**	**1.5**	**1.3**	**1.9**	**1.4**	**1.0**	**42**	**16**	<0.54	<0.59	<0.54	<0.59	**17**	<0.55	<0.54
Dehydronifedipine	**16**	**8.3**	**4.3**	<3.6	**3.9**	<3.5	**4.5**	<3.5	**6.7**	**2.5**	<3.6	<1.2	<3.6	<1.2	<9.4	<3.3	<3.3
Betamethasone	**5.1**	<10	<3.0	<10	<3.3	<9.8	**3.2**	<9.8	<6.3	<2.9	<2.7	<2.9	<2.7	<2.9	<8.4	<9.2	<9.1
Sulfadimethoxine	<2.9	<1.8	<2.5	<2.0	<3.5	<1.8	<2.2	<1.8	**7.6**	**62**	<2.7	<1.6	<2.2	**2.0**	<4.7	<1.6	<1.6
Sulfanilamide	<30	<89	**34**	**112**	<28	**118**	**61**	<89	<22	<29	**45**	**48**	<27	**56**	<235	<83	<82
Amphetamine	<6.9	<2.8	<3.0	<3.0	<3.0	**6.0**	<3.8	<2.9	<4.8	<11	<2.5	<6.9	<4.4	<7.2	**15**	<3.0	<3.0
Personal Care Products																	
DEET	<26	**93**	<79	**16**	<8.8	<11	<8.5	<3.8	**685**	**35**	<4.9	<3.3	<2.5	<6.0	**24**	**19**	**21**
Hormones																	
Progesterone	<220	<26	<441	<21	<24	<21	<111	<22	<22	<453	**14**	<486	<30	<474	<22	<22	<22
Testosterone	<88	<23	<177	<8.6	<9.4	<8.6	<44	<8.8	<29	<`81	<9.2	**24**	<12	<190	<29	<8.8	<8.7
Sterols																	
Coprostanol	<1,080	**436**	<109	<106	<116	<106	<549	**21**	**989**	**2,995**	**3.4**	<2,410	**7.4**	<2,350	**18**	<109	<107
ß-Sitosterol	<1,090	**215**	**10,300**	**1,240**	<1,480	<113	<698	<109	<1,120	**11,805**	<628	**13,400**	<320	**4,290**	<110	**367**	**762**

Bold: Analyte was detected.
Shading: Concentrations of compounds detected in both reclaimed water and groundwater.
J flags for estimated values have been omitted, and
U flags for non-detects are replaced with a less-than sign (<).

Table 19 groups the chemicals detected in groundwater or frequently detected in reclaimed water according to how often they were observed in one or both types of samples. Given their presence in reclaimed water and lack of detection in up-gradient wells, application of reclaimed water is the probable source of most of the groundwater chemicals with a high rate of co-detection, these being carbamazepine, meprobamate, sulfamethoxazole, alprazolam, benzoylecgonine, dehydronifedipine, and DEET. *B*-Sitosterol may also have localized animal or plant sources unrelated to facility discharges. As shown in Table 18 and elsewhere in this report, the majority (80%) of compounds detected in the reclaimed water produced by the LOTT, Quincy, and Yelm facilities do not appear to persist in groundwater at readily detectable levels.

Table 19. Detection Location for Certain Pharmaceuticals, Personal Care Products, Hormones, and Sterols at LOTT, Quincy, and Yelm, 2011.

Consistently Co-detected in Reclaimed Water and Groundwater	Some Co-detections in Reclaimed Water and Groundwater	Frequently Detected but in Reclaimed Water Only	Detected in Groundwater Only
Carbamazepine	Alprazolam	Amitriptyline	Sulfanilamide
Meprobamate	Benzoylecgonine	Atenolol	Progesterone
Sulfamethoxazole	Betamethasone	Clarithromycin	Testosterone
	Dehydronifedipine	Cocaine	
	Sulfadimethoxine	Desmethyldiltiazem	
	DEET	Diphenhydramine	
	Coprostanol	Erythromycin-H_2O	
	B-Sitosterol	Fluoxetine	
		Gemfibrozil	
		Hydrochlorothiazide	
		10-Hydroxy-amitriptyline	
		Metformin	
		Metoprolol	
		Naproxen	
		Norverapamil	
		Oxycodone	
		Propranolol	
		Sertraline	
		Theophylline	
		Triamterene	
		Valsartan	
		Verapamil	
		Cotinine	
		Triclocarban	
		Trimethoprim	
		17*a*-Dihydroequilin	
		Epicoprostanol	

Figure 11 compares reclaimed water and groundwater concentrations for the few chemicals consistently quantified in both types of samples (see Table 18). The figure plots groundwater: reclaimed water ratios for individual samples. Ratios greater than 1 indicate that groundwater exceeded reclaimed water.

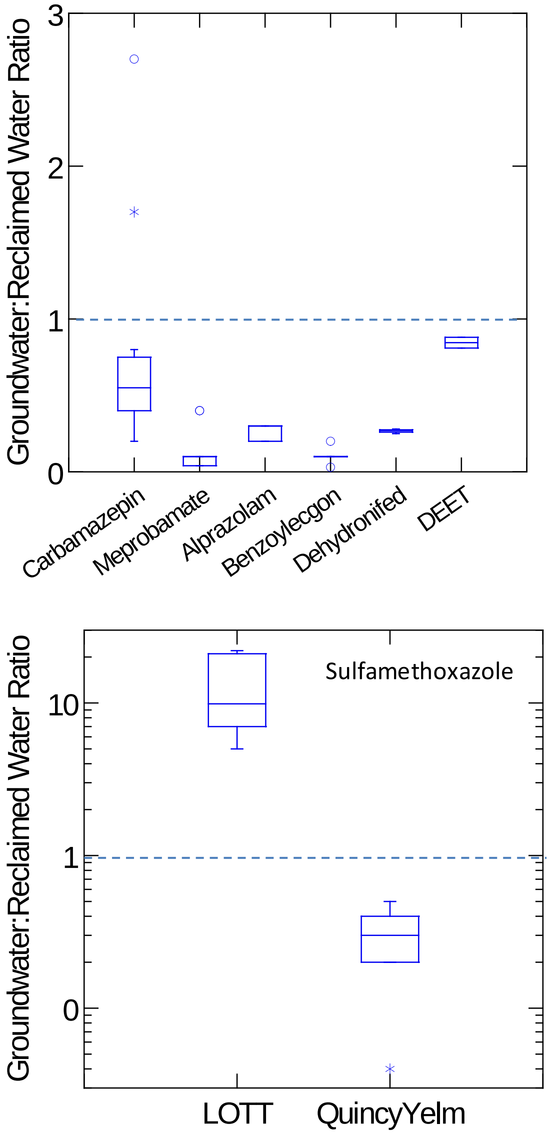

Figure 11. Groundwater vs. Reclaimed Water Concentrations for Selected Chemicals Detected at LOTT, Quincy, and Yelm, 2011.

Alprazolam, benzoylecgonine, and dehydronifedipine data from LOTT; DEET data from Yelm; two meprobamate non-detects in Quincy groundwater not plotted; sulfamethoxazole graph log scale.

The concentration ratios provide a perspective on the extent to which these chemicals are being degraded post-discharge or diluted in groundwater at these facilities. Ratios substantially greater than 1 may indicate that the concentrations measured in reclaimed water for the present study are low compared to the long-term average.

In most cases, measured groundwater concentrations were lower than in the corresponding reclaimed water sample, by about a factor of 2 on average. DEET levels in Yelm groundwater most closely approached those in reclaimed water, based on the limited results obtained. In two instances (Quincy), a slightly higher carbamazepine concentration was recorded in groundwater than in reclaimed water.

Much different results were obtained for sulfamethoxazole at LOTT which had higher rather than lower concentrations in groundwater compared to the reclaimed water. Lubliner et al. (2010) reported considerably more sulfamethoxazole in LOTT reclaimed water than was found in the present study (104 vs. 20-40 ng/L, see Table 11).

Excluding the LOTT results for sulfamethoxazole, the relative concentrations observed in reclaimed water and groundwater in the present study suggest the following order of decreasing presence in groundwater from these chemicals: DEET > carbamazepine > dehydronifedipine > alprazolam > sulfamethoxazole (Quincy/Yelm) > meprobamate > benzoylecgonine.

Indicator Chemicals

One objective of this project was to identify indicator compounds that could be used as tracers of pharmaceuticals, personal care products, hormones, or sterols in groundwater at reclaimed water recharge sites. A useful indicator is widely used, relatively persistent, and readily analyzed.

The compounds that appear most useful as groundwater tracers at LOTT, Quincy, and Yelm are carbamazepine, meprobamate, and sulfamethoxazole. These three compounds were detected in all reclaimed water samples and in all groundwater samples within the recharge area at each facility, both in the spring and in the fall. The concentrations were also substantial, often greater than 100 ng/L. From this standpoint, they meet the criterion of readily analyzed.

Carbamazepine and sulfamethoxazole are two of the most commonly found pharmaceuticals in the environment (Benotti, 2006). These compounds appear to have a low removal rate during wastewater treatment and leach through the subsurface to groundwater relatively unaltered (Kinney et al., 2006b; Drewes et al., 2003).

None of the personal care products, hormones, or sterols identified in this study were detected consistently enough to be useful groundwater indicator compounds. Interferences and blank contamination issues compromised some of the hormones and sterols data.

A number of PPCPs were detected at elevated levels in reclaimed water but did not appear to be important groundwater contaminants. Several of these have been identified by other investigators as useful indicator compounds in human wastewater.

By way of summary, Table 20 lists the potentially useful indicator compounds identified for reclaimed water or groundwater at LOTT, Quincy, and Yelm, including references to corroborating studies. Over half of these substances – 6 out of 10, including carbamazepine, sulfamethoxazole, and meprobamate – could be captured by analyzing Method 1694 target lists 1 and 5 (see Appendix C) at approximately half the cost of a full PPCP analysis.

Several additional pharmaceuticals mentioned in the scientific literature as indicator chemicals for human wastewater were consistently detected at elevated concentrations in Quincy's reclaimed water only. These include diltiazem, diphenhydramine, ranitidine, thiabendazole, and caffeine (Lubliner et al., 2010; Kinney et al, 2006a; Motzer, 2006; Barnes et al., 2002; Glassmeyer et al., 2005).

PPCPs will only be present in reclaimed water if they are being used by people in the community. Animal production can also be a source of some of these chemicals, particularly antibiotics and fungicides. Variances in effluent quality could be the result of advances in pharmaceutical technology/formulation, prescription and use practices, and the health of the community. These types of factors have been noted in other studies as to why some compounds were detected and then no longer present.

Table 20. Potential Indicator Compounds Identified at LOTT, Quincy, and Yelm in 2011.

Chemical (Method 1694 analyte list)	Class	Potentially Useful Indicator in:		Detection Frequency	Concentration Range (ng/L)	Identified as Indicator in Other Studies (refs)
		Reclaimed Water	Ground-water			
Carbamazepine (#1)	anti-epileptic	x	x	100%	170 - 514	1, 2, 3, 4, 5
Sulfamethoxazole (#1)	antibiotic	x	x	100%	20 - 505	1, 2, 3, 4, 6
Meprobamate (#5)	tranquilizer	x	x	88%	31 - 1,420	7, 8
Metformin (#4)	antidiabetic	x		100%*	804 - 3,320	10
Atenolol (#4)	antihypertensive	x		100%*	260 - 837	7
Metoprolol (#5)	antihypertensive	x		100%*	274 - 742	11
Gemfibrozil (#3)	antihyperlipidemic	x		100%*	17 - 543	1
Cotinine (#4)	nicotine metabolite	x		100%*	17 - 212	1, 2, 3, 6
Fluoxetine (#1)	antidepressant	x		100%*	8.3 - 31	1, 5
DEET (#5)	insect repellant	x		80%*	24 - 685	2, 3, 6, 7

References:
1. Kinney et al. (2006)
2. Glassmeyer et al. (2005)
3. Motzer (2006)
4. Benotti et al. (2006)
5. Lubliner et al. (2010)
6. Barnes et al. (2002)
7. Benotti et al. (2009)
8. Drewes (2012)
9. Johnson et al. (2004)
10. Scheurer et al. (2009)
11. Kunkel and Radke (2011)

*detected in reclaimed water samples, not detected in groundwater.

Conclusions

A low-level (parts per trillion) analysis for 145 pharmaceuticals and personal care products (PPCPs), hormones, and sterols in reclaimed water from the LOTT Martin Way, Quincy, and Yelm reclaimed water plants detected and reliably quantified 73 compounds. Fifteen of these were also found in samples obtained from monitoring wells located in areas where reclaimed water is used to recharge groundwater. PPCPs were not detected in the two up-gradient wells sampled during this 2011 study. Thus, most of the compounds (80%) observed in reclaimed water do not appear to persist at readily detectable levels in the groundwater at these locations.

Carbamazepine (anti-epileptic), meprobamate (tranquilizer), and sulfamethoxazole (antibiotic) were consistently detected in both reclaimed water and monitoring wells within the recharge area at all three facilities. Concentrations from the reclaimed water ranged from 20 – 1,420 ng/L (parts per trillion), while concentrations in the groundwater ranged from 10 – 500 ng/L. In most cases, measured groundwater concentrations were lower than those in corresponding reclaimed water samples, by about a factor of 2 on average.

Other chemicals frequently detected, but in reclaimed water only, included metformin (antidiabetic), atenolol (antihypertensive), metoprolol (antihypertensive), gemfibrozil (antihyperlipidemic), cotinine (nicotine metabolite), fluoxetine (antidepressant), and DEET (insect repellant). Compounds found in groundwater but not in reclaimed water were limited to sulfanilamide (antibiotic), progesterone, and testosterone (sex hormones). The occurrence of hormones and sterols was likely underestimated in general due to variable and sometimes high detection limits.

Results from this project seem to support what has been determined in previous studies, that many PPCPs are sufficiently removed during tertiary wastewater treatment and groundwater recharge.

Recommendations

- Carbamazepine, meprobamate, and sulfamethoxazole are recommended as the most useful indicators for tracing pharmaceuticals in the recharge areas at the LOTT Martin Way, Quincy, and Yelm reclaimed water plants.

- Other potentially useful indicators for reclaimed water, but not groundwater, include metformin, atenolol, metoprolol, gemfibrozil, cotinine, fluoxetine, and DEET.

- The elevated concentrations of thiabendazole detected in Quincy’s reclaimed water should be confirmed and the sources evaluated, as appropriate.

- Improved data – consistently low reporting limits and reduced analytical background – are needed on hormones and sterols in reclaimed water and groundwater at recharge areas.

- Additional groundwater sampling should be conducted at monitoring wells farther down-gradient of recharge basins to assess the fate of detected PPCPs with distance and time from point of recharge/infiltration.

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Appendices

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Appendix A. LOTT, Quincy, and Yelm Reclaimed Water Plant Descriptions, Recharge Infiltration Area Geology, and Groundwater Sample Locations in 2011

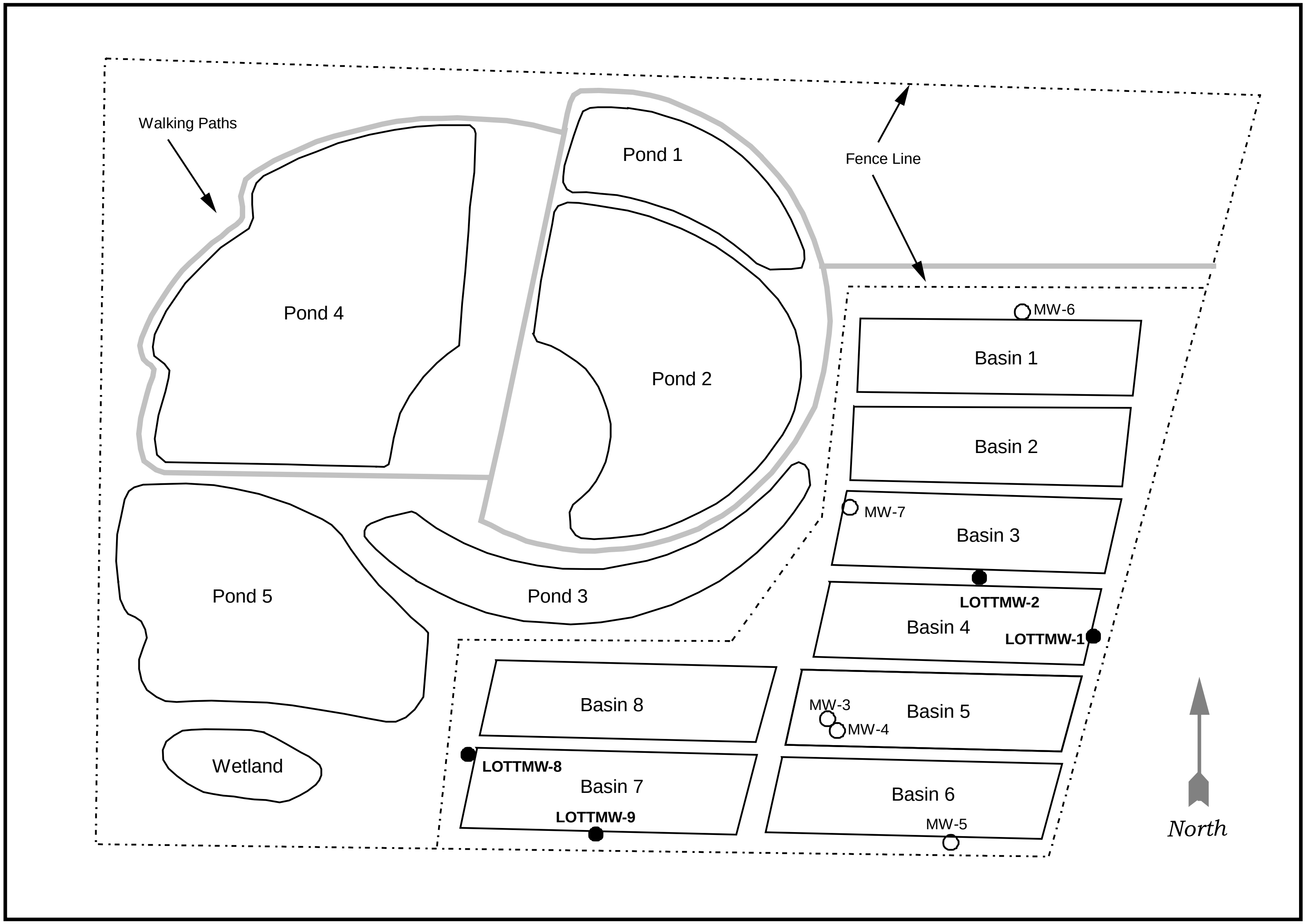
LOTT Martin Way Reclaimed Water Treatment Plant (RWTP) and Hawks Prairie Ponds/ Recharge Basins

Treatment to meet the Washington State Class A standards at the Martin Way facility is accomplished using two-stage biological nutrient removal in bioreactors. After biological treatment, the water 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 limitation. Solids removed during water treatment at the Martin Way RWTP are routed back into the sewer main where the solids then flow to the Budd Inlet wastewater treatment plant.

The Class A reclaimed water produced at the Martin Way RWTP is sent through three miles of pipe to the Hawks Prairie Reclaimed Water Ponds and Recharge Basins. The reclaimed water circulates through a series of five constructed wetland ponds. The water is then transferred to one of eight rectangular infiltration basins to recharge the groundwater.

Reclaimed water not used for the constructed wetland ponds and groundwater recharge is available to the cities of Lacey and Olympia for landscape irrigation, commercial and industrial processes, environmental enhancements, and other uses.

Geology of the LOTT Hawks Prairie reclaimed water recharge area is characterized by a thick sequence of unconsolidated glacial deposits which include the Vashon recessional outwash, Vashon Till, Vashon advance outwash, and the Kitsap Formation. The recessional outwash is a highly permeable, sandy gravel that is approximately 20 feet thick. Regionally, the recessional outwash is underlain by the Vashon Till. The till is a low permeability unit consisting of unsorted sand, gravel, and boulders in a matrix of silt and clay. However, studies of the recharge area have found this layer to be generally absent. Beneath the Vashon outwash/till is the relatively permeable Vashon advance outwash and a local unit, the Martin Way gravel. The surficial aquifer is located in this unit at approximately 90 feet below ground surface. The Kitsap Formation, a low permeable silty sand and clay unit, forms the lower boundary of the shallow aquifer. The regional groundwater flow direction of the surficial aquifer is generally to the west. Infiltration of the recharge water has created groundwater mounding under the eastern infiltration basins.



(Figure based on Brown and Caldwell 2010 drawing. Figure not drawn to scale.)

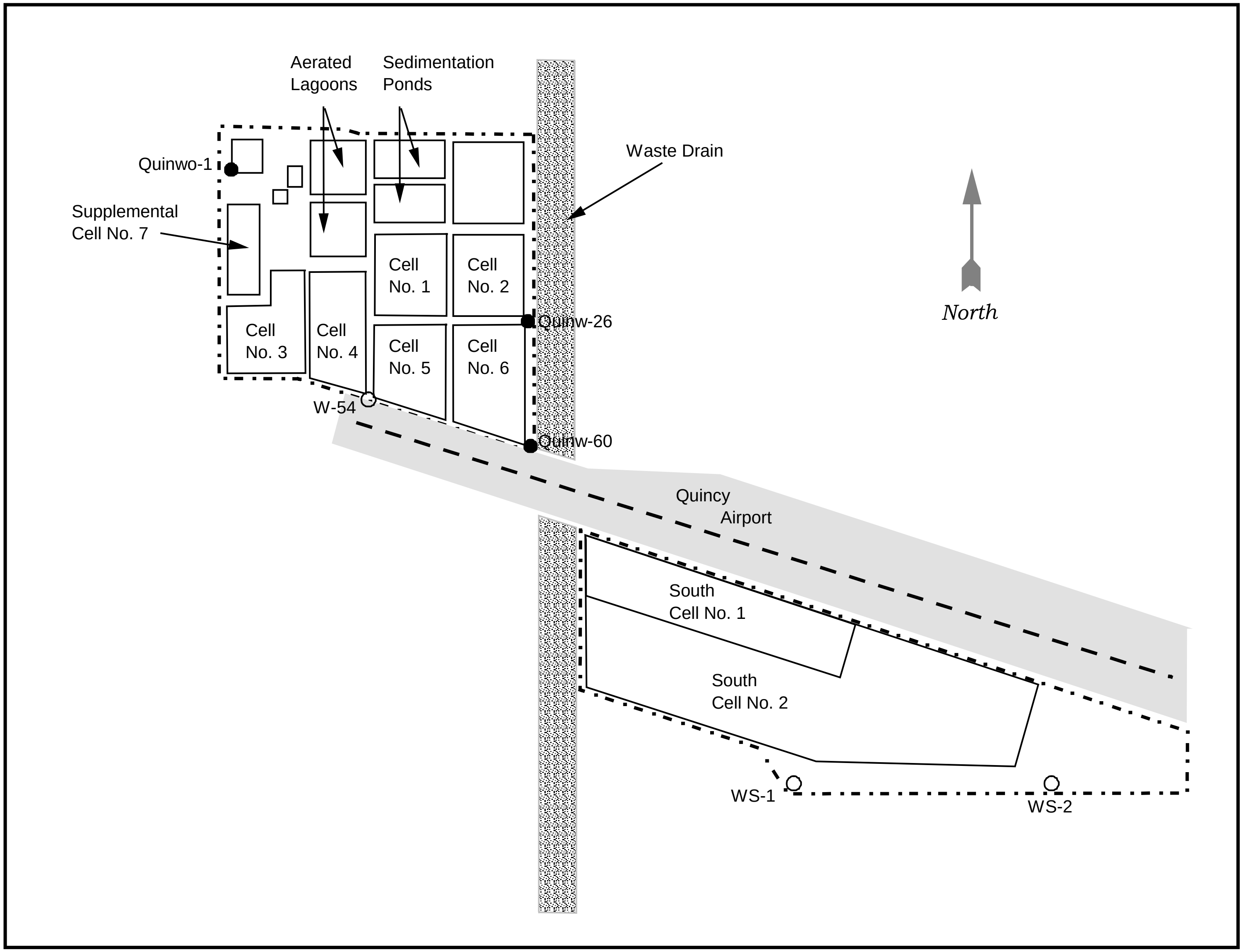
Figure A-1. LOTT Hawks Prairie Reclaimed Water Satellite – Groundwater Sample Locations.

Quincy Reclaimed Water Plant

Water treatment at Quincy consists of two activated sludge lagoons using sequencing batch reactor (SBR) technology to remove nitrogen and attain federal secondary treatment standards. The SBR discharges to an equalization basin that reduces peak flows to a lower more uniform flow. The reduced flow rate allows smaller sizing of the advanced treatment units that provide Class A reclaimed water. Advanced treatment consists of chemical coagulation, continuous backwash up-flow sand filtration, and ultraviolet light disinfection prior to discharge to six infiltration cells. The infiltration cells are located near the water reclamation facility. Because the clay soils in Quincy slow percolation rates, the six infiltration cells require over 15 acres of land. Flows to each cell are intermittent so that the basins have time to drain. Treated water is applied in a cycle of two to four days of infiltration, followed by four to ten days of no application or recharge.

Geology of the Quincy facility, located in eastern Washington, is substantially different from the other two study sites. The geology is characterized by a sequence of unconsolidated and poorly consolidated deposits of fluvial and lacustrine sand and silt, some fluvial gravel, and basalt bedrock (Grolier and Bingham, 1971). The thickness of unconsolidated deposits ranges from about 50 to 100 feet. Caliche deposits within the unconsolidated deposits are common. The Yakima Basalts of the Columbia River Group underlies the unconsolidated deposits and is continuous beneath the study area.

The upper-most aquifer consists of unconsolidated deposits and the upper portion of the underlying basalt. It is saturated to within 20 feet of the surface. General groundwater flow direction is to the east. Groundwater elevations were not directly influenced by the West Canal.



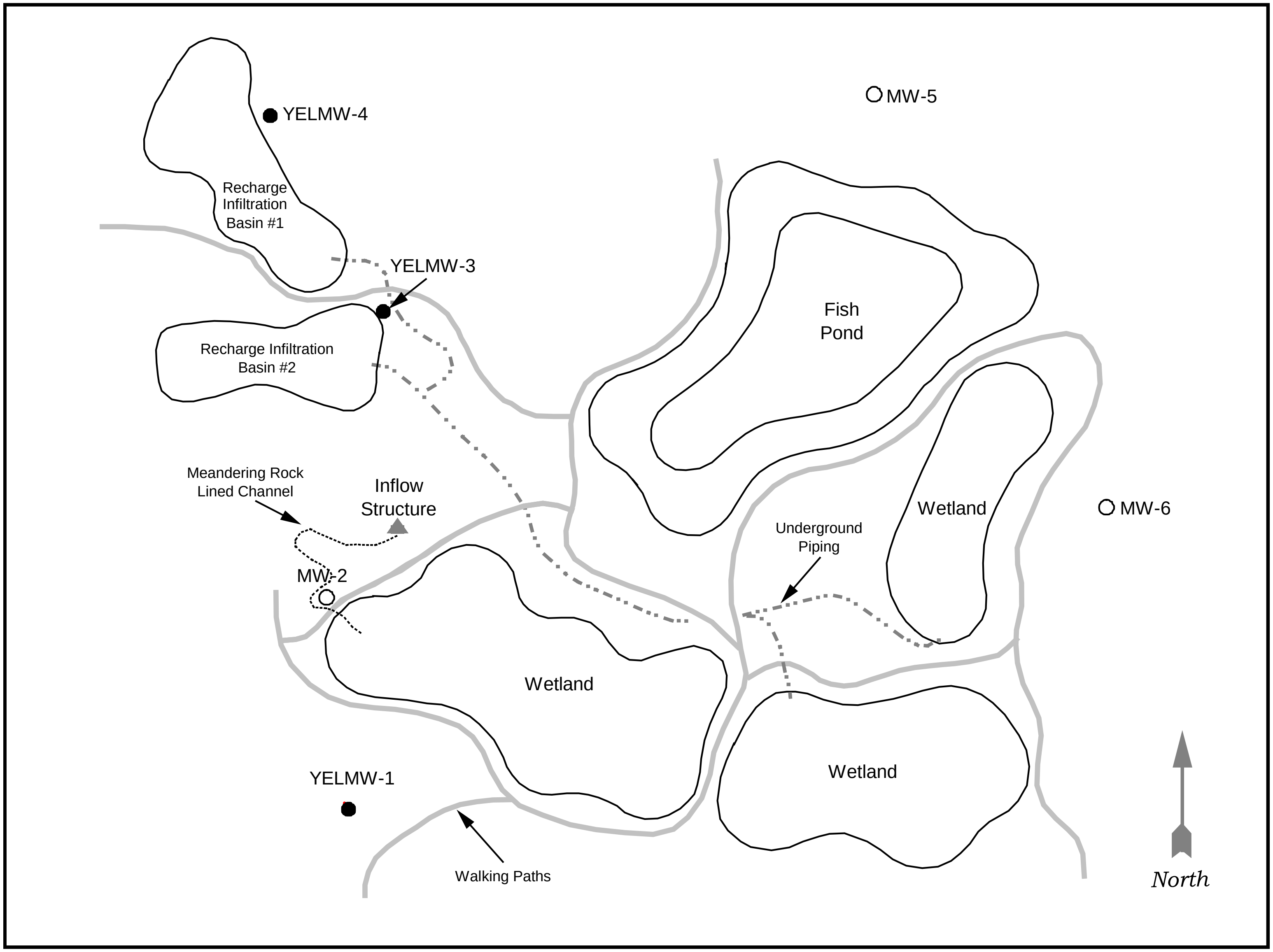
(Figure based on Gray & Osborne, Inc drawing. Figure not drawn to scale.)

Figure A-2. Quincy Reclaimed Water Plant – Groundwater Sample Locations.

Yelm Water Reclamation Facility

Treatment at the Yelm plant is also by sequencing batch reactor (SBR) for secondary treatment (biological oxidation) and nitrogen removal. Water from the SBRs flows by gravity to an equalization basin with five surface aerators. Water is then pumped from the equalization basin to the tertiary treatment area. Tertiary treatment consists of chemical coagulation, flocculation and settling, rapid-sand filtration, and chlorine disinfection. If the quality of the water meets the State’s Water Reclamation Criteria and there is a demand for reuse water, the reclaimed water is pumped to its beneficial use which includes irrigation projects throughout the city of Yelm. If there is insufficient demand for the reuse water, it is piped to Cochrane Memorial Park where it circulates through a series of constructed surface and submerged wetlands that allow natural treatment processes to act on the water prior to infiltration into the ground.

Geology of the Yelm recharge area at Cochran Memorial Park is similar to LOTT’s Hawks Prairie satellite, as both facilities are located in the Puget Sound lowlands. The Vashon recessional outwash is nearly continuous beneath the recharge area and ranges up to 38 feet thick. The surficial aquifer is located in this unit at approximately 25 feet below ground surface. Regionally, the Vashon till underlies the recessional outwash and is typically 10 to 30 feet thick in the Yelm area. However, this unit is also absent in some areas. Recharge area monitoring well completion depths are above the possible till unit. The Vashon advance outwash underlies the recharge area and occurs at approximately 50 feet below ground surface. The Kitsap Formation forms the lower aquitard in the region. Generally, groundwater flow appears to be northward towards the Nisqually River; however, localized flow patterns will develop as a result of variations in infiltration and recharge.



(Figure based on Skillings Connolly drawing. Figure not drawn to scale.)

Figure A-3. Yelm Water Reclamation Facility – Groundwater Sample Locations at Cochrane Park.

Appendix B. Location, Date, and Time of Reclaimed Water Samples Collected by Ecology in 2011

Facility	Sampling Point	Date	Time of Grabs for Composite Sample			Sample Numbers
LOTT	"CLA composite sample point"; collected from NPDES compositor pump after purging. 47.049 N x 122.802 W	11-May-11	0900	1130	1500	1105051-1, -2*, -3†
		5-Oct-11	0730	1100	1400	1110038-1
Quincy	"Municipal effluent"; collected from channel at NPDES compositor intake. 47.215 N x 119.845 W	24-May-11	0815	1100	1315	1105040-4
		20-Oct-11	0845	1115	1300	1110053-1, -2*, 3†
Yelm	Fecal coliform sample point for NPDES compliance; collected at end of chlorine contact channel. 46.950 N x 122.594 W	6-Oct-11	0845	1115	1400	1110038-2

CLA: Class A

*duplicate sample

†transfer blank

Appendix C. Chemicals Analyzed and Reporting Limits (ng/L; parts per trillion)

Table C-1. PPCPs Analyzed by EPA Method 1694.

Chemical	CAS	Spring Reporting Limit	Fall Reporting Limit
List 1 - Acid Extraction in Positive Ionization			
Acetaminophen	103-90-2	22.1 - 35.9	28.6 - 235
Azithromycin	83905-01-5	2.44 - 3.59	2.86 - 23.5
Caffeine	58-08-2	22.1 - 35.9	28.6 - 235
Carbadox	7-5-6804	2.21 - 3.59	2.86 - 23.5
Carbamazepine	298-46-4	2.21 - 3.59	2.86 - 23.5
Cefotaxime	63527-52-6	110 - 229	5.8 - 8.15
Ciprofloxacin	85721-33-1	11 - 50.3	11.4 - 94
Clarithromycin	81103-11-9	2.21 - 3.59	2.86 - 23.5
Clinafloxacin	105956-97-6	44.5 - 114	13.5 - 313
Cloxacillin	61-72-3	4.56 - 7.17	5.72 - 47
Dehydronifedipine	67035-22-7	2.95 - 4.78	1.14 - 9.4
Digoxigenin	1672-46-4	11 - 153	11.5 - 172
Digoxin	20830-75-5	8.84 - 14.3	11.4 - 94
Diltiazem	42399-41-7	0.542 - 0.717	0.572 - 4.7
1,7-Dimethylxanthine	611-59-6	88.4 - 143	114 - 940
Diphenhydramine	58-73-1	0.884 - 1.43	1.14 - 9.4
Enrofloxacin	93106-60-6	4.42 - 7.17	5.72 - 47
Erythromycin-H20	114-07-8	1.47 - 2.39	0.572 - 4.7
Flumequine	42835-25-6	2.61 - 5.45	2.91 - 78.3
Fluoxetine	54910-89-3	2.21 - 3.59	2.86 - 23.5
Lincomycin	154-21-2	4.42 - 7.17	5.72 - 47
Lomefloxacin	98079-51-7	5.45 - 9.88	5.72 - 47
Miconazole	22916-47-8	2.21 - 3.59	2.86 - 23.5
Norfloxacin	70458-96-7	27.6 - 96.3	28.6 - 235
Norgestimate	35189-28-7	17.5 - 23.9	5.72 - 47
Ofloxacin	82419-36-1	2.21 - 3.59	2.86 - 23.5
Ormetoprim	6981-18-6	0.884 - 1.43	1.14 - 9.4
Oxacillin	66-79-5	4.42 - 7.17	5.72 - 47
Oxolinic acid	14698-29-4	1.08 - 3.68	1.14 - 9.4
Penicillin G	61-33-6	4.42 - 7.17	5.74 - 157
Penicillin V	87-08-1	14.7 - 23.9	5.72 - 47
Roxithromycin	80214-83-1	0.442 - 0.717	0.572 - 4.7
Sarafloxacin	98105-99-8	22.1 - 35.9	28.6 - 235
Sulfachloropyridazine	80-32-0	2.21 - 3.59	2.86 - 23.5
Sulfadiazine	68-35-9	2.21 - 3.59	2.86 - 23.5
Sulfadimethoxine	122-11-2	2.08 - 3.45	0.572 - 4.7
Sulfamerazine	127-79-7	2.95 - 4.78	1.14 - 9.4
Sulfamethazine	57-68-1	2.95 - 4.78	1.14 - 10.4

Chemical	CAS	Spring Reporting Limit	Fall Reporting Limit
Sulfamethizole	144-82-1	2.95 - 4.78	1.14 - 12.3
Sulfamethoxazole	723-46-6	3.61 - 4.78	1.14 - 9.4
Sulfanilamide	63-74-1	22.1 - 35.9	28.6 - 235
Sulfathiazole	72-14-0	2.49 - 3.59	2.86 - 23.5
Thiabendazole	148-79-8	2.71 - 3.59	2.86 - 288
Trimethoprim	738-70-5	2.21 - 3.59	2.86 - 23.5
Tylosin	1401-69-0	8.84 - 14.3	11.4 - 94
Virginiamycin	11006-76-1	4.42 - 7.17	5.72 - 48.4
List 2 – Tetracyclines in Positive Ionization			
Anhydrochlortetracycline	4497-08-9	25 - 34.5	29.2 - 128
Anhydrotetracycline	4496-85-9	25.7 - 31.3	29.1 - 110
Chlortetracycline	57-62-5	9.04 - 12.9	10.3 - 40
Demeclocycline	127-33-3	21.3 - 31.3	23.5 - 100
Doxycycline	564-25-0	8.83 - 12.5	11.5 - 44
4-Epianhydrochlortetracycline	158018-53-2	85.1 - 125	116 - 440
4-Epianhydrotetracycline	4465-65-0	27.2 - 38.4	25.7 - 56.9
4-Epichlortetracycline	14297-93-9	21.3 - 31.3	23.5 - 40.6
4-Epioxytetracycline	14206-58-7	10.9 - 16.6	10.3 - 43.2
4-Epitetracycline	23313-80-6	10.9 - 16.5	10.3 - 43.9
Isochlortetracycline	514-53-4	8.51 - 12.5	9.4 - 13.2
Minocycline	10118-90-8	120 - 162	94 - 132
Oxytetracycline	79-57-2	8.51 - 12.5	10.3 - 13.2
Tetracycline	60-54-8	8.51 - 12.5	10.3 - 26.2
List 3 - Acid Extraction in Negative Ionization			
Bisphenol A	80-05-7	1420 - 2080	783 - 1100
Furosemide	54-31-9	68.7 - 83.4	62.7 - 88.1
Gemfibrozil	25812-30-0	2.5 - 3.13	2.35 - 3.31
Glipizide	29094-61-9	8.51 - 12.5	9.4 - 13.2
Glyburide	10238-21-8	4.26 - 6.25	4.7 - 6.61
Hydrochlorothiazide	58-93-5	28.4 - 41.7	31.3 - 44
2-hydroxy-ibuprofen	51146-55-5	114 - 303	125 - 181
Ibuprofen	15687-27-1	21.3 - 31.3	23.5 - 33
Naproxen	22204-53-1	5.12 - 6.25	4.7 - 6.61
Triclocarban	101-20-2	4.26 - 6.25	4.7 - 6.61
Triclosan	3380-34-5	87.7 - 129	94 - 132
Warfarin	81-81-2	2.13 - 3.13	2.35 - 3.3
List 4 - Basic Extraction in Positive Ionization			
Albuterol	18559-94-9	0.413 - 0.64	0.569 - 2.13
Amphetamine	300-62-9	2.06 - 3.2	2.23 - 14.5
Atenolol	29122-68-7	1.02 - 2.24	1.14 - 7.6

Chemical	CAS	Spring Reporting Limit	Fall Reporting Limit
Atorvastatin	134523-00-5	2.07 - 3.2	2.23 - 3.48
Cimetidine	51481-61-9	0.826 - 1.53	0.894 - 5.34
Clonidine	4205-90-7	2.06 - 3.2	2.56 - 4.88
Codeine	76-57-3	4.13 - 6.67	5.91 - 31
Cotinine	486-56-6	2.06 - 3.2	2.56 - 3.48
Enalapril	75847-73-3	0.413 - 0.64	0.447 - 0.959
Hydrocodone	125-29-1	2.06 - 3.2	2.23 - 10
Metformin	657-24-9	5.12 - 12.7	5.85 - 59.1
Oxycodone	76-42-6	1.2 - 5.5	1.14 - 10.6
Ranitidine	66357-35-5	0.934 - 1.28	1.03 - 4.68
Triamterene	396-01-0	0.714 - 1.06	1.73 - 2.32
List 5 - Acid Extraction in Positive Ionization			
Alprazolam	28981-97-7	0.542 - 1.33	0.47 - 1.05
Amitriptyline	50-48-6	0.542 - 1.33	0.51 - 0.743
Amlodipine	88150-42-9	2.71 - 6.63	2.26 - 3.17
Benzoylecgonine	519-09-5	0.542 - 1.33	0.47 - 0.863
Benztropine	86-13-5	0.542 - 1.33	0.47 - 0.661
Betamethasone	378-44-9	2.71 - 6.63	2.87 - 11
Cocaine	50-36-2	0.271 - 0.663	0.235 - 0.401
DEET	134-62-3	0.271 - 0.663	0.783 - 1.1
Desmethyldiltiazem	--	0.271 - 0.663	0.257 - 0.38
Diazepam	439-14-5	0.542 - 1.33	0.47 - 0.661
Fluocinonide	356-12-7	10.8 - 26.5	9.4 - 13.2
Fluticasone propionate	80474-14-2	3.61 - 8.84	3.43 - 8.27
Hydrocortisone	50-23-7	108 - 265	103 - 197
10-hydroxy-amitriptyline	1159-82-6	0.271 - 0.663	0.291 - 1.1
Meprobamate	57-53-4	7.23 - 17.7	6.27 - 8.81
Methylprednisolone	83-43-2	7.23 - 17.7	20.9 - 29.4
Metoprolol	51384-51-1	2.76 - 20.7	2.57 - 15.6
Norfluoxetine	83891-03-6	2.71 - 6.63	2.35 - 3.3
Norverapamil	67018-85-3	0.271 - 0.663	0.235 - 0.33
Paroxetine	61869-08-7	7.23 - 17.7	6.27 - 8.81
Prednisolone	50-24-8	10.8 - 26.5	10.6 - 24.6
Prednisone	53-03-2	36.1 - 88.4	38.9 - 220
Promethazine	60-87-7	0.723 - 1.77	0.627 - 0.881
Propoxyphene	469-62-5	0.542 - 1.33	0.47 - 0.661
Propranolol	525-66-6	3.61 - 8.84	3.13 - 4.4
Sertraline	79617-96-2	0.723 - 1.77	0.627 - 0.881
Simvastatin	79902-63-9	36.1 - 88.4	31.3 - 44
Theophylline	58-55-9	108 - 265	103 - 143

Chemical	CAS	Spring Reporting Limit	Fall Reporting Limit
Trenbolone	10161-33-8	7.23 - 17.7	6.38 - 9.15
Trenbolone acetate	10161-34-9	0.542 - 1.33	0.47 - 0.903
Valsartan	137862-53-4	7.23 - 17.7	6.27 - 8.81
Verapamil	52-53-9	0.271 - 0.663	0.235 - 0.33

Table C-2. Sterols and Hormones Analyzed by EPA Method 1698.

Chemical	CAS	Spring Reporting Limit	Fall Reporting Limit
Sterols			
Campesterol	474-62-4	0.847 - 5.43	1.79 - 249
Cholestanol	80-97-7	0.239 - 2.63	0.41 - 37
Cholesterol	57-88-5	0.612 - 5.39	1.45 - 198
Coprostanol	360-68-9	0.332 - 1.92	0.969 - 132
Desmosterol	313-04-2	1.05 - 15.5	1.63 - 463
Epicoprostanol	516-92-7	0.364 - 2.1	1.1 - 135
Ergosterol	57-87-4	1.06 - 14.8	0.677 - 267
B-Sitosterol	83-46-5	0.43 - 5.12	1.7 - 328
B-Stigmastanol	19466-47-8	0.396 - 4.38	1.68 - 273
Stigmasterol	83-48-7	0.753 - 9.39	2.11 - 428
Hormones			
17*B*-Estradiol	50-28-2	0.0709 - 1.69	0.195 - 7.37
17*a*-Estradiol	57-91-0	0.0674 - 1.67	0.183 - 7.95
17*a*-Ethinyl Estradiol	57-63-6	0.09 - 2.84	0.321 - 15.1
B-Estradiol-3-Benzoate	50-50-0	1.61 - 16.3	0.32 - 103
17*a*-Dihydroequilin	651-55-8	0.197 - 57.1	0.224 - 21.3
Androstenedione	63-05-8	4.68 - 134	5.54 - 95.8
Androsterone	53-41-8	0.104 - 13.9	0.0305 - 6.09
Desogestrel	54024-22-5	0.253 - 44.3	0.148 - 24.3
Equilenin	517-09-9	0.0634 - 15	0.241 - 20.8
Equilin	474-86-2	0.434 - 25.2	0.36 - 31.4
Estriol	50-27-1	0.143 - 2.91	0.272 - 30.5
Estrone	53-16-7	0.274 - 7.7	0.317 - 21.5
Mestranol	72-33-3	0.144 - 22.1	0.247 - 28.6
Norethindrone	68-22-4	0.424 - 51.4	0.287 - 36.5
Norgestrel	6533-00-2	0.858 - 11.7	0.639 - 69.7
Progesterone	57-83-0	1.29 - 19.4	2.64 - 162
Testosterone	58-22-0	1.14 - 28.9	0.778 - 59.5

Appendix D. Manchester Environmental Laboratory and Reviews and Case Narratives

Data Qualifiers

Code	**Definition**
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
REJ	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	The analyte was not detected at or above the reported sample quantitation limit.
UJ	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately measure the analyte in the sample.

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Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

September 15, 2011

Subject:	PPCPs in Reclaimed Water - 2011
Samples:	1105040-01 – 04, 1105051-01 - 06
Officer:	Art Johnson
By:	John Weakland

Pharmaceuticals and Personal Care Products

Review of LCMS/MS data from Axys Analytical

Analytical Method(s)

The samples were extracted and analyzed following a modification of EPA Method 1694. Data were reviewed for qualitative and quantitative accuracy. Axys' case narrative discussing the quality of the analytical data, analyte lists and analyte control limits are included in Appendix A.

Holding Times

EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, sample extraction should occur within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

To reduce analytical costs, the two sets of samples were batched together. All of the samples from WO 1105051 sampled on May 11, 2011 for List 2, 3 & 4 analytes (see Appendix A) were extracted 12 days past the suggested hold time of 7 days. However, the results for these compounds were not qualified due to the hold time exceedance.

Significant problems were encountered for the List 1 & 5 analytes (see Appendix A) and reported results were extracted 43 and 56 days past the suggested 7 day sample hold time. All of the results for all of the reported samples were qualified J if detected and UJ if not detected.

Initial Calibration

The initial calibration (ICAL), Initial Calibration Verification (ICV), and back calculations (BC) were within AXYS' QC limits with the following exceptions.

The ICAL for Hydrochlorothiazide and Cefotaxime contained only 4 calibration points. The samples were qualified by MEL according to Table 1 below.

Table 1

Compound	Sample IDs	Qual
Cefotaxime	WG37012-101 (Method Blank), 1105040-01,02, 03,04 1105051-01,01 DUP,03,04,05,06	UJ
Hydrochlorothiazide	WG36613-101 (Method Blank), 1105040-01,02,03 1105051-03,04,05,06	UJ
	1105040-04, 1105051-01,01 DUP	J

Continuing Calibration

All continuing calibration verifications (CCVs) were within Axys' QC limits.

Method Blanks

The method blanks were reasonable and acceptable with the following exceptions.

Erythromyacin-H2O was detected below the Method Reporting Limit (MRL). With the exception of the duplicate, all of the detected results were greater than 5 times the amount found in the method blank and were therefore not qualified. Since the concentration of the duplicate was similar to the native sample, the duplicate was not qualified.

DEET, Methylprednisolone and Amphetamine were detected in Axys' method blanks. If the amount detected in the sample was above the MRL, but less than 5 times the blank amount, the result was raised to amount found in the sample and qualified U, not detected. Samples were qualified by MEL according to Table 2 below. Note that DEET and Methylprednisolone results were additionally qualified UJ due to the hold time exceedance.

Table 2

Compound	Sample IDs	Qual
DEET	1105040-01,02,03, 1105051-01,01 DUP,03,04,05,06	UJ
Methylprednisolone	1105040-01,02,03, 1105051-01,01 DUP,04	UJ
Amphetamine	1105040-03,04, 1105051-01,01 DUP,03,04	U

Surrogates

The surrogate (Extraction Internal Standards) percent recoveries were within Axys' QC limits with the following exceptions.

Several surrogate recoveries were above Axys' QC limits, indicating a possible high bias. While the method is recovery-corrected, any detected analytes associated with the out of control surrogate were qualified according to Table 3 below. Undetected analytes were not qualified.

The recovery of d7-DEET was below Axys' QC limits, indicating a possible low bias. While the method is recovery-corrected, the associated analytes were qualified according to Table 3 below.

Table 3

Compound	Sample IDs	Qual
Carbamazepine	1105051-05	J
Dehydronifedipine	1105051-05	J
Fluoxetine	1105040-04	J
Triamterene	1105040-04, 1105051-01,01 DUP	J
10-hydroxy-amitriptyline	1105040-04	J
Noverapamil	1105040-04	J
Propranolol	1105040-04	J
Sertraline	1105040-04	J
DEET	WG37012-101 (Method Blank)	J

Laboratory Control Samples (LCS)

The percent recoveries of the LCS (Axys refers to these samples as OPR – Ongoing Precision and Recovery, and as "Spiked Matrix" in the EDD) were within Axys' QC limits with the following exceptions.

Several analyte recoveries were above Axys' QC limits indicating a possible high bias. If the analyte was not detected in the sample no qualification was necessary. The codeine result for sample 1105040-04 was qualified as an estimate; J. Since the DEET result for the method blank was qualified for other reasons, no further qualification was necessary.

Duplicate Samples

The RPDs of detected analytes for the sample and duplicate were within Axys' QC limits with the following exceptions.

The RPD of Methylprednisolone exceeded QC limits. However, the analyte was qualified for other reasons, and no further qualification of the data is necessary.

Qualitative Identification

All reported samples met Axys' criteria for qualitative identification with the following exceptions. There were instrumental interferences preventing the accurate identification of Amphetamine for all of the samples. The method blank was qualified NJ, tentatively identified at the estimated result. The samples were already qualified U due to the amount found in the method blank, and no further qualification of the data is necessary.

Comments

Axys defines it's MRL as the concentration of the lowest calibration standard (Result Value EQL in the EDD) or the Sample Detection Limit (SDL) (Result EDL in the EDD), whichever is greater. Axys' SDL is determined individually for every sample by converting the area equivalent of 3 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical workup.

Several analytes had SDLs greater than the MRL. In those instances, Axys raised the MRL to the calculated SDL, and the sample result was qualified UJ, not detected at the estimated reporting limit.

The EDD contains 6 tabs with the first tab containing the combined results followed by tabs with each of the 5 individual analyte lists. MEL's reporting and qualification of the results is included in the EDD as extra highlighted columns and titled MEL Amended Result Reported Value and MEL Amended Result Data Qualifier. A third highlighted column was added to the combined results to show which Axys analyte list the reported analyte belongs to.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

September 20, 2011

Subject:	Pharmaceuticals in Reclaimed Water
LIMS ID:	1105040 and 1105051
Laboratory:	AXYS Analytical Services, Ltd.
Project Officer:	Art Johnson and Pam Marti
By:	Karin Feddersen

Data Review for Analysis of Sterols and hormones

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias following AXYS' in-house method MLA-068.

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 GCIMS and GC/HRMS"***, based on EPA Method 1698. A method summary is provided in AXYS' case narrative.

Results have been reported in nanograms per Liter (ng/L).

Several groups of congeners coelute. Each IUPAC # of the congeners in the coeleution is listed on the report, separated by a slash "/" with a single value. This reported value is a sum total of all the coeluting congeners.

Results were reported down to the level of the instrument's "Sample Detection Limit" (SDL). The SDL values reflect levels that are approximately 2.5 times the signal-to-noise ratio. This is the same criterion as is used for the Method Detection Limit (MDL), described by 40CFR.

A number of congeners were qualified with a "J" because the concentration was below the lowest calibration standard (this value is in the "Result Value EQL"; Estimated Quantitation Limit; column). Non-detect results are reported to the EDL and are estimated "UJ". Results derived from responses outside the calibration range are considered estimates.

There were instrumental interferences preventing the accurate identification of several analytes. AXYS flagged these results with a "K". This flag has been amended to "NJ" in the EDD.

Holding Times

EPA Method 1698 states: "EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, begin sample extraction within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

If the sample will not be extracted within 48 hours of collection, the laboratory should adjust the pH of aqueous samples to 5.0 to 9.0 with sodium hydroxide or sulfuric acid solution. Record the volume of acid or base used."

Sample extracts are to be stored in the dark at less than -10 °C and analyzed within 40 days of extraction.

The sample coolers were verified to be at <4 °C upon receipt at the contract lab. The samples were subsequently stored at -20°C until extraction.

Samples for work order 1105051 were collected on May 11, and for 1105040 on May 24. All samples were extracted on May 27th and analyzed within 40 days of extraction. Results for samples from work order 1105051 have been qualified as estimates, as a negative bias may have resulted due to the 7-day holding time being exceeded.

Initial Calibration (ICAL) Curve and Verification (CAL VER) Standards

All verification standards are within of 65 - 135 % for Equilin, 50 -150 % for Ergosterol, and 70 to 130% for all others. Because the labeled compounds are present in each calibration standard at the same concentration, linearity of the labeled compounds is not assessed.

All the ion abundance ratios and relative retention times (RRT) were within QC criteria. The ratio for each analyte in the ICAL and CAL VER standards must be within 30 % of mid-level ICAL standard. Labeled analyte RRTs must be within ±15 sec. of the mid-level I-CAL standard. Sample RTS are compared to the RRT of the CAL VER.

Bracketing Standard Calibration

AXYS employs a procedure of quantification for Hormones and Sterols that varies from all EPA methods. Instead of quantifying the results from the initial calibration curve, they analyze the mid-point standard before and after each sequence of 12 samples. They determine the average relative response factor (RRF) from these two "bracketing" calibration standards and use it to quantify analyte results.

Each RRF for opening and closing calibrations over a 12 hour period agreed to within ±20% of the mean (i.e. ≤40 RPD between RRFs for the opening and closing calibrations).

AXYS has compared recoveries for a low level standard that has a concentration below the lowest standard in the calibration curve, using the bracketing standards and the initial calibration. This method of quantification has therefore been deemed acceptable for these samples.

Labeled Standard Recoveries

All field samples and QC samples were spiked with labeled Extraction Internal Standards (EIS) prior to extraction; and with labeled recovery standards after extraction and prior to cleanup.

Recoveries for all EIS compounds in these samples were within method QC limits.

Ion abundance ratios

Each congener reported as detected met the isotopic abundance ratio and retention time criteria for positive identification with several exceptions. AXYS flagged these exceptions with “K”. The K has been changed to the qualifier “NJ”, except where the analyte was also detected in the blank. These results have been raised to the level of the EQL and qualified as non-detects, “U”.

Blanks

The blank is labeled: WG36617-101 i.

Certain target compounds were detected in the laboratory blanks. At the time these samples were analyzed, AXYS appeared to be having a period of fairly high levels of sterols. All other batches also experienced a relatively high level of blank contamination. Where these congeners were also detected in the samples; the sample result was flagged with a “B” by the contract laboratory.

Where the sample concentration was less than five times the blank concentration, the B has been replaced with a “U”. Results in the sample less than the EQL have been raised to the level of the EQL and qualified with a “U”.

In cases where the sample concentration for a congener was greater than five times that of the blank, the blank result is considered insignificant relative to the native concentration detected in the sample. No qualification is warranted in these situations. B flags have been removed from these sample results.

A limit of 10 times has been used for Cholesterol, Stigmasterol and beta-Sitosterol.

In addition, the “B” has been deleted from sample results in the case of “NJ”-qualified blank results.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR is labeled: WG36617-102 i.

All target and labeled compound recoveries were within AXYS’ quality control limits (listed in the method summary of the accompanying case narrative provided by AXYS.)

Laboratory Duplicate Samples

Duplicate aliquots of sample LOTTEFF (1105051-1, -2) were prepared and analyzed. The Relative Percent Difference (RPD) between the results was above 40% for Campesterol, Stigmasterol, and beta-Sitosterol. These results have already been qualified as estimates in the sample and its duplicate for holding time exceedances.

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Manchester Environmental Laboratory
7411 Beach Drive East, Port Orchard Washington 98366

January 11, 2012

Subject: PPCPs in Reclaimed Water - 2011
Samples: 1110038-01 – 05, 07 - 10
Project Officer: Art Johnson
By: John Weakland

Pharmaceuticals and Personal Care Products

Review of LCMS/MS data from Axys Analytical

Analytical Method(s)

The samples were extracted and analyzed following a modification of EPA Method 1694. Data were reviewed for qualitative and quantitative accuracy. Axys' case narrative discussing the quality of the analytical data, analyte lists and analyte control limits are included in Appendix A.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and to add qualifiers, as appropriate and that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD contains 6 tabs with the first tab containing the combined results and MEL-amended result values and qualifiers. There are two highlighted columns titled MEL Amended Result Reported Value and MEL Amended Result Data Qualifier. These amended values should be used instead of the original values provided by the contract lab. Where cells in the amended qualifiers and values fields are blank, the original result values and qualifiers should be used. A third highlighted column was added to the combined results to show which Axys analyte list the reported analyte belongs to.

Holding Times

The EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, sample extraction should occur within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

The samples were extracted 11 - 12 days past the suggested hold time of 7 days. However, the results for these compounds were not qualified due to the hold time exceedance.

Initial Calibration

The initial calibration (ICAL), Initial Calibration Verification (ICV), and back calculations (BC) were within AXYS' QC limits with the following exceptions.

The ICAL for Hydrochlorothiazide and the Cefotaxime contained only 4 calibration points. The samples were qualified by MEL according to Table 1 below.

Table 1

Compound	Sample IDs	Qual
Cefotaxime	WG37947-101 (Method Blank), 1110038-01, 02 ,03 04, 05, 05 Dup, 07, 08, 09, 10	UJ
Hydrochlorothiazide	WG37947-101 (Method Blank), 1110038-01, 03, 04 05, 05 Dup, 07, 08, 09, 10	UJ
	1110038-02	J

The ICV for some of the analytes exceeded QC limits indicating a high bias. However, the analytes were not detected in the samples and therefore no qualification of the data was necessary.

Continuing Calibration

All continuing calibration verifications (CCVs) were within Axys' QC limits with the following exceptions.

Some of the analyte responses exceeded QC limits. However, in these cases the analytes were not detected in the samples and therefore no qualification of the data was necessary.

The CCVs for Carbamazepine, Digoxigenin and Naproxen exceeded QC limits and the samples were qualified according to Table 2 below.

Table 2

Compound	Sample IDs	Qual
Carbamazepine	1110038-01, 02, 09	J
Digoxigenin	1110038-01, 02, 09	UJ
Naproxen	1110038-02	J

Method Blanks

The method blanks were reasonable and acceptable with the following exceptions.

DEET and Diltiazem were detected in Axys' method blanks. If the amount detected in the sample was above the MRL, but less than 5 times the amount found in the associated method blank, the result was raised to amount found in the sample and qualified U, not detected. Samples were qualified by MEL according to Table 3 below.

Table 3

Compound	Sample IDs	Qual
DEET	1110038-03, 04, 05, 07, 10	U
Diltiazem	1110038-10	U

Surrogates

The surrogate (Extraction Internal Standards) percent recoveries were within Axys' QC limits with the following exceptions.

Several surrogate recoveries were above Axys' QC limits, indicating a possible high bias. However, the associated analytes were not detected in the sample and therefore no qualification was necessary.

The percent recoveries of c13-Trimethoprim, d5-Fluoxetine and d4-Clonidine were above Axys' QC limits, indicating a possible high bias. While the method is recovery-corrected, the detected analytes associated with the out of control surrogates were qualified according to Table 4 below.

The recovery of d3-Cimetidine for sample 1110038-01 was below Axys' QC limits, indicating a possible low bias. While the method is recovery-corrected, the associated analytes were qualified according to Table 4 below. In addition, there was no recovery of d4-Promethazine for sample 1110038-01. The associated analytes were qualified according to Table 4 below.

Table 4

Compound	Sample IDs	Qual
Carbamazepine	1110038-05	J
Fluoxetine	1110038-01, 02	J
Triamterene	1110038-02	J
Cimetidine	1110038-01	UJ
Promethazine	1110038-01	REJ
Desmethyldiltiazem	1110038-01	REJ

Laboratory Control Samples (LCS)

The percent recoveries of the LCS (Axys refers to these samples as OPR – Ongoing Precision and Recovery, and as “Spiked Matrix” in the EDD) were within Axys’ QC limits with the following exceptions.

Several recoveries were above Axys’ QC limits, indicating a possible high bias. However, if the analytes were not detected in any of the samples no qualification of the data was necessary. The fluoxetine results were already qualified and no further qualification was necessary.

The recoveries of Diphenhydramine and Hydrocodone were below Axys’ QC limits indicating a possible low bias. The sample results were qualified according to Table 5 below.

Table 5

Compound	Sample IDs	Qual
Diphenhydramine	WG37947-101 (Method Blank), 1110038-01, 03, 04 05, 05 Dup, 07, 08, 09, 10	UJ
	1110038-02	J
Hydrocodone	WG37947-101 (Method Blank), 1110038-01, 03, 04 05, 05 Dup, 07, 08, 09, 10	UJ
	1110038-02	J

Duplicate Samples

The RPDs of detected analytes for the sample and duplicate (1110038-05 and 05 Dup) were within Axys’ QC limits.

Qualitative Identification

All reported samples met Axys’ criteria for qualitative identification with the following exceptions.

There were instrumental interferences preventing the accurate identification of Amphetamine in samples 1110038-02 and 04. Therefore, the samples were qualified NJ, tentatively identified at the estimated result.

Comments

Axys defines it’s MRL as the concentration of the lowest calibration standard (Result Value EQL in the EDD) or the Sample Detection Limit (SDL) (Result EDL in the EDD), whichever is greater. Axys’ SDL is determined individually for every sample by converting the area equivalent of 3 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical workup.

Several analytes had SDLs greater than the MRL. In those instances, Axys raised the MRL to the calculated SDL and flagged the EDD results UJ, not detected at the estimated reporting limit.

During the initial analysis of the List 1 analytes, poor surrogate recoveries prevented the accurate quantitation of the data and was attributed to matrix interferences. Therefore, the samples were diluted and reanalyzed, with the exception of Cefotaxime. As a result, the reporting limits for List 1 analytes for all of the samples except for the Method Blank, Lab Control Sample and 1110038-10 (Equip Blk) were raised 3 to 10 times the method reporting limit.

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Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

January 12, 2012

Subject:	PPCPs in Reclaimed Water - 2011
Samples:	1110053-01, 03 - 06
Project Officer:	Art Johnson
By:	John Weakland

Pharmaceuticals and Personal Care Products

Review of LCMS/MS data from Axys Analytical

Analytical Method(s)

The samples were extracted and analyzed following a modification of EPA Method 1694. Data were reviewed for qualitative and quantitative accuracy. Axys' case narrative discussing the quality of the analytical data, analyte lists and analyte control limits are included in Appendix A.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and to add qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD contains 6 tabs with the first tab containing the combined results and MEL-amended result values and qualifiers. There are two highlighted columns titled MEL Amended Result Reported Value and MEL Amended Result Data Qualifier. These amended values should be used instead of the original values provided by the contract lab. Where cells in the amended qualifiers and values fields are blank, the original result values and qualifiers should be used. A third highlighted column was added to show the Axys analyte list the reported analyte belongs to.

Holding Times

The EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, sample extraction should occur within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

The samples were extracted within the suggested hold time of 7 days.

Initial Calibration

The initial calibration (ICAL), Initial Calibration Verification (ICV), and back calculations (BC) were within AXYS' QC limits with the following exceptions.

Cefotaxime was unstable during the analysis and all sample results were qualified REJ.

Continuing Calibration

All continuing calibration verifications (CCVs) were within Axys' QC limits.

Method Blanks

The method blanks were reasonable and acceptable with the following exceptions.

DEET was detected in Axys' method blank. If the amount detected in the samples was above the Method Reporting Limit (MRL), but less than 5 times the amount found in the associated method blank, the result was raised to the amount found in the sample and qualified U, not detected. Samples were qualified by MEL according to Table 1 below.

Table 1

Compound	Sample IDs	Qual
DEET	1110053-03, 04, 05, 06	J

Surrogates

The surrogate (Extraction Internal Standards) percent recoveries were within Axys' QC limits with the following exceptions.

Several surrogate recoveries were above Axys' QC limits, indicating a possible high bias. However, the associated analytes were not detected in the sample and therefore no qualification was necessary.

The percent recovery of 13c6-Sulfamethoxazole was above Axys' QC limits, indicating a possible high bias. While the method is recovery-corrected, the detected analytes associated with the out of control surrogates were qualified according to Table 2 below.

The recovery of d3-Cimetidine was below Axys' QC limits, indicating a possible low bias. While the method is recovery-corrected, the associated analytes were qualified according to Table 2 below. The recovery of d3-Cocaine for the LCS (WG38057-102) was below QC limits. However, the analyte was not qualified because the percent recovery of the unlabeled compound was within QC limits.

Table 2

Compound	Sample IDs	Qual
Sulfamethoxazole	1110053-06	J
Cimetidine	Method Blank (WG38057-101)	UJ

Laboratory Control Samples (LCS)

The percent recoveries of the LCS (Axys refers to these samples as OPR – Ongoing Precision and Recovery, and as "Spiked Matrix" in the EDD) were within Axys' QC limits with the following exceptions.

Several recoveries were above Axys' QC limits, indicating a possible high bias. However, if the analytes were not detected in any of the samples, no qualification of the data was necessary.

The recoveries of Cloxacin, Diphendyramine, Tylosin were below Axys' QC limits indicating a possible low bias. The sample results were qualified according to Table 3 below.

Table 3

Compound	Sample IDs	Qual
Cloxacin	WG38057-101 (Method Blank), 1110053-01, 01 DUP, 03, 04, 05, 06	UJ
Diphenhydramine	WG38057-101 (Method Blank), 1110053-03, 04, 05, 06	UJ
	1110053-01, 01 DUP	J
Tylosin	WG38057-101 (Method Blank), 1110053-01, 01 DUP, 03, 04, 05, 06	UJ

Duplicate Samples

The RPDs of detected analytes for the sample and duplicate (1110053-01 and 01 Dup) were within Axys' QC limits with the following exceptions.

The RPD of Albuterol exceeded QC limits so the analyte was qualified J, estimated value, for samples 1110053-01 and 1110053-01 DUP.

Qualitative Identification

All reported samples met Axys' criteria for qualitative identification.

Comments

Axys defines it's MRL as the concentration of the lowest calibration standard (Result Value EQL in the EDD) or the Sample Detection Limit (SDL) (Result EDL in the EDD), whichever is greater. Axys' SDL is determined individually for every sample by converting the area equivalent of 3 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical workup.

Several analytes had SDLs greater than the MRL. In those instances, Axys raised the MRL to the calculated SDL and flagged the EDD results UJ, not detected at the estimated reporting limit.

A portion of Axys’ method blank was inadvertently added to 1110053-04 prior to extraction. To account for the loss, a correction factor was applied to the associated surrogate recoveries and MRL. Since the method adjusts for losses, no qualification of the sample was necessary.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

February 13, 2012

Subject:	PPCP in Reclaimed Water
LIMS ID:	1110038
Laboratory:	AXYS Analytical Services, Ltd.
Project Officer:	Art Johnson & Pam Marti
By:	Karin Feddersen

Data Review for Analysis of Sterols and hormones

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias following AXYS' in-house method MLA-068.

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 GCIMS and GC/HRMS"***, based on EPA Method 1698. A method summary is provided in AXYS' case narrative.

Results have been reported in nanograms per Liter (ng/L).

Several groups of congeners coelute. Each IUPAC # of the congeners in the coeleution is listed on the report, separated by a slash "/" with a single value. This reported value is a sum total of all the coeluting congeners.

Detected results were reported down to the level of the instrument's "Estimated Detection Limit" (EDL). AXYS refers to this limit as the Sample Detection Limit (SDL). The EDL values reflect levels that are 3 times the signal-to-noise ratio. This criterion is similar to that used for the Method Detection Limit (MDL), described by 40CFR as 2.5 times the signal-to-noise ratio.

These results were qualified with a "J" because the concentration was below the lowest calibration standard (this value is in the "Result Value EQL"; Estimated Quantitation Limit; column). Results derived from responses outside the calibration range are considered estimates. Non-detect results are reported to the EQL and are not estimated.

The EQLs for several results were below the instrument's "Estimated Detection Limit" (EDL). These reporting limits have been amended to the EDL. These limits are estimated values.

AXYS flags the analytes stigmasterol, β-sitosterol, and β-stigmasterol with "MAX" in all samples, indicating that the reported value is an estimated maximum. This is a new flag that AXYS has recently introduced. The background levels of these analytes are variable and typically high; results for these analytes in samples show poor duplication. A "J" qualifier is used for these samples. Since this flag indicates a high bias only, non-detects are not qualified, unless there are other reasons to qualify the samples as described in one of the sections below. The OPR is not qualified, as QC samples are not given qualifiers.

A disturbance of the mass ion used to monitor instrument performance (lock-mass) greater than method specifications was observed at several different retention times, preventing the accurate identification of several analytes. AXYS flagged these results with a “K”. This flag has been amended to “NJ” in the EDD.

AXYS has no long term recovery data is available for Ergosterol in the OPR. Ergosterol results have been flagged “H”; estimated. The “H” has been replaced with a “J” for detected results and “UJ” for non-detects.

Holding Times

EPA Method 1698 states: “EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

“Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, begin sample extraction within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

“If the sample will not be extracted within 48 hours of collection, the laboratory should adjust the pH of aqueous samples to 5.0 to 9.0 with sodium hydroxide or sulfuric acid solution. Record the volume of acid or base used.”

Sample extracts are to be stored in the dark at less than -10 °C and analyzed within 40 days of extraction.

The sample coolers were verified to be at <4 °C upon receipt at the contract lab. The samples were subsequently stored at -20°C.

These samples were collected on October 5 and 6. All were extracted on October 13 and analyzed on October 27, within 40 days of extraction. Samples 1110038-01, 03, -04, -05, and -06 (duplicate of -05) were extracted past the holding time at 8 days after collection. However, AXYS states that all samples are frozen upon receipt, and analyzed immediately upon thawing.

Initial Calibration (ICAL) Curve and Verification (CAL VER) Standards

All verification standards are within of 65 - 135 % for Equilin, 50 -150 % for Ergosterol, and 70 to 130% for all others. Because the labeled compounds are present in each calibration standard at the same concentration, linearity of the labeled compounds is not assessed.

All the ion abundance ratios and relative retention times (RRT) were within QC criteria. The ratio for each analyte in the ICAL and CAL VER standards must be within 30 % of mid-level ICAL standard. Labeled analyte RRTs must be within ±15 sec. of the mid-level I-CAL standard. Sample RTS are compared to the RRT of the CAL VER.

Bracketing Standard Calibration

AXYS employs a procedure of quantification for Hormones and Sterols that varies from all EPA methods. Instead of quantifying the results from the initial calibration curve, they analyze the mid-point standard before and after each sequence of 12 samples. They determine the average relative response factor (RRF) from these two “bracketing” calibration standards and use it to quantify analyte results.

Each RRF for opening and closing calibrations over a 12 hour period agreed to within ±20% of the mean (i.e. ≤40% RPD between RRFs for the opening and closing calibrations).

The bracketing calibration is >40% RSD for Estriol-tris and β-Estradiol-3-Benzoate. However, these analytes were not detected in any of the samples.

AXYS has demonstrated acceptable recoveries of a low level standard, below the initial calibration curve, using the bracketing standards. This method has therefore been deemed acceptable for these samples.

Labeled Standard Recoveries aka Extraction Internal Standards (EIS)

All field samples and QC samples were spiked with EIS prior to extraction; and with labeled cleanup standards after extraction and prior to cleanup.

Recoveries for all EIS compounds in these samples were within method QC limits, with several exceptions. AXYS flagged these analytes with a “V”. The V has been deleted, as no qualifiers are added to QC sample results.

Recoveries for D4-17 β-estradiol and D4-17 α-ethinylestradiol in the method blank fell below the lower method control limit. Low EIS recoveries in the method blank may indicate low bias and thus unremarked contamination. β-Sitosterol, 17 α-Ethinyl-Estradiol, β-Stigmastanol, and β-Estradiol-3-benzoate are quantitated by these EIS. These analytes were qualified with “J” for detected analytes and “UJ” for non-detects in the method blank. Since these analytes were not detected in several samples with acceptable EIS recoveries, the system was demonstrated to be free of contamination from these analytes.

D6-Norethindrone recoveries were high in 1110038-01, 1110038-02, and 1110038-09. D6-Norgestrel recoveries were high in 1110038-01 and 1110038-02. Norethindrone, Norgestrel, Estriol, and β-Estradiol 3-benzoate are quantitated by these EIS. Congeners that may have been biased high have not been flagged if the affected congener was not detected. Since these analytes were not detected in any of the samples, no qualification was warranted.

Ion abundance ratios

Each congener reported as detected met the isotopic abundance ratio and retention time criteria for positive identification with several exceptions. AXYS flagged these exceptions with “K”. The K has been changed to the qualifier “NJ”, except where the analyte was also detected in the blank. Those results have been raised to the level of the EQL and qualified as non-detects, “U”.

Blanks

The blank is labeled: WG37918-101.

Certain target compounds were detected in the laboratory blanks. Where these congeners were also detected in the samples at a concentration greater than *10* times that in the blank; the sample result was flagged with a “B” by the contract laboratory.

Where the sample concentration was less than *5* times the blank concentration, the B has been replaced with a “U”. Results in the sample less than the EQL have been raised to the level of the EQL and qualified with a “U”.

Sample results above the EQL for β-sitosterol and stigmasterol, (originally flagged with “MAX”) have been qualified with “UJ”, as these analytes are typically biased high, according to AXYS.

In cases where the sample concentration for a congener was greater than *five* times that of the blank, the blank result is considered insignificant relative to the native concentration detected in the sample. No qualification is warranted in these situations. B flags have been removed from these sample results.

In addition, the B has been removed from positively detected sample results (no K flag) in the case of “NJ”-qualified blank results.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR is labeled: WG37918-102.

Target analyte and labeled compound recoveries were within AXYS’ quality control limits.

Laboratory Duplicate Samples

Sample 1110038-05 was prepared and analyzed in duplicate (1110038-06). In the duplication analysis, the relative percent differences (RPD) for campesterol, stigmasterol, and β-sitosterol were high (>40%). Detected results for campesterol have been qualified “J”.

The high RPDs for stigmasterol and β-sitosterol are typical (these analytes are flagged "MAX"). The “MAX” has been replaced with “J”.

Manchester Environmental Laboratory

7411 Beach Drive East, Port Orchard Washington 98366

February 13, 2012

Subject: PPCP in Reclaimed Water
LIMS ID: 1110053
Laboratory: AXYS Analytical Services, Ltd.
Project Officer: Art Johnson & Pam Marti
By: Karin Feddersen

Data Review for Analysis of Sterols and hormones

Summary

Data from these analyses were reviewed for qualitative and quantitative precision and bias following AXYS' in-house method MLA-068.

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 GCIMS and GC/HRMS"***, based on EPA Method 1698. A method summary is provided in AXYS' case narrative.

Results have been reported in nanograms per Liter (ng/L).

Several groups of congeners coelute. Each IUPAC # of the congeners in the coeleution is listed on the report, separated by a slash "/" with a single value. This reported value is a sum total of all the coeluting congeners.

Flags are added by the contract laboratory to draw attention to QC conditions that may affect the data. Manchester Environmental Laboratory (MEL) interprets the effect on the quality of the data and adds qualifiers, as appropriate, that are consistent with MEL and Ecology Information Management (EIM) guidelines.

The EDD includes some MEL-amended result values and qualifiers. These amended values should be used instead of the original values provided by the contract lab.

In addition, where the qualifiers are unchanged from the contract laboratory, they have been copied over to the MEL Amended field. In effect these MEL QA review qualifiers become the final qualifiers.

Detected results were reported down to the level of the instrument's "Estimated Detection Limit" (EDL). AXYS refers to this limit as the Sample Detection Limit (SDL). The EDL values reflect levels that are 3 times the signal-to-noise ratio. This criterion is similar to that used for the Method Detection Limit (MDL), described by 40CFR as 2.5 times the signal-to-noise ratio.

A number of congeners were qualified with a “J” because the concentration was below the lowest calibration standard (this value is in the “Result Value EQL”; Estimated Quantitation Limit; column). Non-detect results are reported to the EDL and are estimated “UJ”. Results derived from responses outside the calibration range are considered estimates.

There were instrumental interferences preventing the accurate identification of several analytes. AXYS flagged these results with a “K”. This flag has been amended to “NJ” in the EDD.

Samples QUINCY EFF {#1110053-01), QUINCYEFF (#1110053-01) (Duplicate), QUINW-60 (#1110053-05), and QUINW-26 (#1110053-06) were diluted and instrumentally re-analyzed to minimize matrix interferences and improve chromatography. The dilution analysis was successful and all target concentrations are reported from the diluted extract (indicated by suffix 'N' on AXYS ID). AXYS entered a “Y” into the “Re-Analysis Flag” column, and flagged these results with “D”. Since “D” is not a qualifier used in LIMS, it has been deleted from the sample results in the EDD.

AXYS flags the analytes stigmasterol, β-sitosterol, and β-stigmasterol with “MAX” in all samples, indicating that the reported value is an estimated maximum. This is a new flag that AXYS has recently introduced. The background levels of these analytes are variable and typically high; results for these analytes in samples show poor duplication. A “J” qualifier is used for these samples. Since this flag indicates a high bias only, non-detects are not qualified, unless there are other reasons to qualify the samples as described in one of the sections below. The OPR is not qualified, as QC samples are not given qualifiers.

AXYS has no long term recovery data is available for Ergosterol in the OPR. Ergosterol results have been flagged “H”; estimated. The “H” has been replaced with a “J” for detected results and “UJ” for non-detects.

Holding Times

EPA Method 1698 states: “EPA has not conducted formal holding time studies for these analytes to date. Use the information below as guidance. Exceeding these default holding times does not invalidate the sample results.

Anecdotal evidence suggests that some analytes may degrade rapidly in aqueous samples. Therefore, begin sample extraction within 7 days of collection (within 48 hours is strongly encouraged). Extracts should be analyzed within 40 days of extraction. Freezing of aqueous samples is encouraged to minimize degradation, in which case, samples should be extracted within 48 hours of removal from the freezer.

If the sample will not be extracted within 48 hours of collection, the laboratory should adjust the pH of aqueous samples to 5.0 to 9.0 with sodium hydroxide or sulfuric acid solution. Record the volume of acid or base used.”

Sample extracts are to be stored in the dark at less than -10 °C and analyzed within 40 days of extraction.

The sample coolers were verified to be at 0 °C upon receipt at the contract lab. The samples were subsequently stored at -20°C.

These samples were extracted on October 27, 2011, seven days after collection. These samples were analyzed December 6, 34 days after extraction and December 14, 42 days after extraction.

Initial Calibration (ICAL) Curve and Verification (CAL VER) Standards

All verification standards are within of 65 - 135 % for Equilin, 50 -150 % for Ergosterol, and 70 to 130% for all others. Because the labeled compounds are present in each calibration standard at the same concentration, linearity of the labeled compounds is not assessed.

All the ion abundance ratios and relative retention times (RRT) were within QC criteria. The ratio for each analyte in the ICAL and CAL VER standards must be within 30 % of mid-level ICAL standard. Labeled analyte RRTs must be within ±15 sec. of the mid-level I-CAL standard. Sample RTS are compared to the RRT of the CAL VER.

Bracketing Standard Calibration

AXYS employs a procedure of quantification for Hormones and Sterols that varies from all EPA methods. Instead of quantifying the results from the initial calibration curve, they analyze the mid-point standard before and after each sequence of 12 samples. They determine the average relative response factor (RRF) from these two "bracketing" calibration standards and use it to quantify analyte results.

Each RRF for opening and closing calibrations over a 12 hour period agreed to within ±20% of the mean (i.e. ≤40 RPD between RRFs for the opening and closing calibrations).

AXYS has demonstrated acceptable recoveries of a low level standard, below the initial calibration curve, using the bracketing standards. This method has therefore been deemed acceptable for these samples.

Labeled Standard Recoveries aka Extraction Internal Standards (EIS)

All field samples and QC samples were spiked with EIS prior to extraction; and with labeled recovery standards after extraction and prior to cleanup.

Recoveries for all EIS compounds in these samples were within method QC limits, with several exceptions.

Recoveries for D4-17 β-estradiol and D4-17 α-ethinylestradiol in the method blank fell below the lower method control limit. Low EIS recoveries in the method blank may indicate low bias and thus unremarked contamination. β-Sitosterol, 17 α-Ethinyl-Estradiol, β-Stigmastanol, and β-Estradiol-3-benzoate are quantitated by these EIS. These analytes were qualified with "J" for detected analytes and "UJ" for non-detects in the method blank. Since these analytes were not detected in several samples with acceptable EIS recoveries, the system was demonstrated to be free of contamination from these analytes.

None of the analytes that use the affected labeled compounds for quantification were detected in these samples. Corresponding results have been qualified with "UJ".

Ion abundance ratios

Each congener reported as detected met the isotopic abundance ratio and retention time criteria for positive identification with several exceptions. AXYS flagged these exceptions with "K". The K has been changed to the qualifier "NJ", except where the analyte was also detected in the blank. The results have been raised to the level of the EQL and qualified as non-detects, "U".

Blanks

The blank is labeled: WG38063-101.

Certain target compounds were detected in the laboratory blanks. Where these congeners were also detected in the samples; the sample result was flagged with a “B” by the contract laboratory.

Where the sample concentration was less than five times the blank concentration, the B has been replaced with a “U”. Results in the sample less than the EQL have been raised to the level of the EQL and qualified with a “U”.

In cases where the sample concentration for a congener was greater than five times that of the blank, the blank result is considered insignificant relative to the native concentration detected in the sample. No qualification is warranted in these situations. B flags have been removed from these sample results.

In addition, sample results are not qualified in the case of “NJ”-qualified blank results.

On-going Precision and Recovery (OPR) or Laboratory Control Sample (LCS)

The OPR is labeled: WG38063-102.

Target analyte and labeled compound recoveries were within AXYS’ quality control limits.

Laboratory Duplicate Samples

Sample 1110038-05 was prepared and analyzed in duplicate. AXYS states that “In the duplication analysis, the relative percent difference for β-sitosterol was high (>40%). The high RPD for
β-sitosterol are typical (these analytes are flagged "MAX").” In addition, Stigmasterol and β-Stigmastanol are considered to be highly variable and have thus also been flagged. The “MAX” has been replaced with “J” for detected results, except in the case of the OPR, as QC sample results are not qualified. Congeners that may have been biased high have not been qualified if the affected congener was not detected.

Appendix E. Analytical Results

Table E-1. Summary of Field Parameter Results for Groundwater, May and October, 2011.

Well Sample ID	Total Depth (feet)[1]	Depth-to-Water (feet) [1]		pH (standard units)		Dissolved Oxygen (mg/L)		Specific Conductance (umhos/cm)		Temperature	
		5/11	10/11	5/11	10/11	5/11	10/11	5/11	10/11	5/11	10/11
LOTT											
LOTTMW-1	115.06	83.49	--	6.7	--	2.0	--	353	--	15.3	--
LOTTMW-2	107.00	--	88.52	--	6.5	--	6.3	--	256	--	13.9
LOTTMW-8	124.30	103.75	108.02	6.7	6.6	4.0	8.7	351	456	16.1	16.9
LOTTMW-9	108.70	91.91	98.25	6.7	6.6	5.9	6.5	301	468	13.9	13.3
Quincy											
Quinwo-1	21.81	14.76	12.66	7.8	7.7	4.7	3.2	615	729	13.1	14.5
Quinw-26	30.05	18.16	14.81	7.4	7.3	1.8	4.4	1272	1343	18.3	17.3
Quinw-60	30.65	18.10	15.58	7.2	7.2	2.9	1.6	1157	1305	18.2	16.3
Yelm											
YELMW-1	36.62	--	26.24	--	6.3	--	8.4	--	160	--	12.3
YELMW-3	33.53	--	24.62	--	6.2	--	6.4	--	314	--	13.9
YELMW-4	36.46	--	24.49	--	6.3	--	4.7	--	389	--	13.3

[1] Measured from top of casing

-- Not sampled.

Table E-2. Pharmaceuticals and Personal Care Products Analytical Results for LOTT Martin Way Reclaimed Water Plant/Hawks Prairie Satellite, May and October, 2011.

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
List 1 - Acid Extraction in Positive Ionization																
Acetaminophen	30.1	UJ	30.4	UJ	29.1	UJ	28.4	UJ	89.3	U	88.5	U	89.9	U	88.5	U
Azithromycin	**14**	**J**	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Caffeine	30.1	UJ	30.4	UJ	29.1	UJ	28.4	UJ	89.3	U	88.5	U	89.9	U	88.5	U
Carbadox	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Carbamazepine	**493**	**J**	**187**	**J**	**184**	**J**	**122**	**J**	**298**	**J**	**56.3**		**186**	**J**	**206**	
Cefotaxime	183	UJ	122	UJ	117	UJ	114	UJ	7.35	UJ	7.28	UJ	7.39	UJ	7.28	UJ
Ciprofloxacin	37.2	UJ	16.8	UJ	17.8	UJ	11.4	UJ	35.7	U	35.4	U	36	U	35.4	U
Clarithromycin	**8.75**	**J**	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Clinafloxacin	114	UJ	61.1	UJ	51.5	UJ	48.7	UJ	119	U	35.4	U	36	U	35.4	U
Cloxacillin	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
Dehydronifedipine	**15.8**	**J**	**4.5**	**J**	**4.31**	**J**	**3.94**	**J**	**8.28**		3.54	U	3.6	U	3.54	U
Digoxigenin	153	UJ	37.5	UJ	15.5	UJ	17	UJ	114	UJ	39.9	UJ	40.4	UJ	62.2	UJ
Digoxin	12	UJ	12.2	UJ	11.7	UJ	11.4	UJ	35.7	U	35.4	U	36	U	35.4	U
Diltiazem	0.601	UJ	0.608	UJ	0.583	UJ	0.568	UJ	1.79	U	1.77	U	1.8	U	1.77	U
1,7-Dimethylxanthine	120	UJ	122	UJ	117	UJ	114	UJ	357	U	354	U	360	U	354	U
Diphenhydramine	**7.93**	**J**	1.22	UJ	1.17	UJ	1.14	UJ	3.57	UJ	3.54	UJ	3.6	UJ	3.54	UJ
Enrofloxacin	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
Erythromycin-H20	**6.51**	**J**	2.03	UJ	1.94	UJ	1.89	UJ	1.79	U	1.77	U	1.8	U	1.77	U
Flumequine	5.45	UJ	3.04	UJ	4.22	UJ	2.84	UJ	29.8	U	29.5	U	30	U	29.5	U
Fluoxetine	**33.2**	**J**	3.04	UJ	2.91	UJ	2.84	UJ	**12.6**	**J**	8.85	U	8.99	U	8.85	U
Lincomycin	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
Lomefloxacin	9.22	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
Miconazole	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Norfloxacin	96.3	UJ	50.1	UJ	50.8	UJ	41.5	UJ	89.3	U	88.5	U	89.9	U	88.5	U
Norgestimate	20	UJ	20.3	UJ	19.4	UJ	18.9	UJ	19.1	UJ	17.7	U	18	U	17.7	U
Ofloxacin	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Ormetoprim	1.2	UJ	1.22	UJ	1.17	UJ	1.14	UJ	3.57	U	3.54	U	3.6	U	3.54	U
Oxacillin	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
Oxolinic acid	3.68	UJ	1.22	UJ	1.17	UJ	1.24	UJ	3.57	U	3.54	U	3.6	U	3.54	U
Penicillin G	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	59.6	U	17.7	U	18	U	17.7	U
Penicillin V	20	UJ	20.3	UJ	19.4	UJ	18.9	UJ	17.9	U	17.7	U	18	U	17.7	U
Roxithromycin	0.601	UJ	0.608	UJ	0.583	UJ	0.568	UJ	1.79	U	1.77	U	1.8	U	1.77	U
Sarafloxacin	30.1	UJ	30.4	UJ	29.1	UJ	28.4	UJ	89.3	U	88.5	U	89.9	U	88.5	U
Sulfachloropyridazine	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Sulfadiazine	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Sulfadimethoxine	2.88	UJ	2.17	UJ	2.5	UJ	3.45	UJ	1.79	U	1.77	U	1.98	U	1.77	U
Sulfamerazine	4.13	UJ	4.05	UJ	3.88	UJ	3.79	UJ	3.57	U	3.54	U	3.6	U	3.54	U
Sulfamethazine	4.01	UJ	4.05	UJ	3.88	UJ	3.79	UJ	5.77	UJ	3.54	U	3.6	U	3.54	U
Sulfamethizole	4.01	UJ	4.05	UJ	3.88	UJ	3.79	UJ	3.57	U	3.54	U	3.6	U	3.54	U
Sulfamethoxazole	**39.3**	**J**	**497**	**J**	**324**	**J**	**275**	**J**	**19.5**		**95.1**		**422**		**438**	
Sulfanilamide	30.1	UJ	**60.7**	**J**	**34.1**	**J**	28.4	UJ	89.3	U	88.5	U	**105**		**118**	
Sulfathiazole	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Thiabendazole*	*30.3*	*REJ*	3.04	UJ	2.91	UJ	2.84	UJ	*16.4*	*REJ*	8.85	U	8.99	U	8.85	U
Trimethoprim	**4.06**	**J**	3.04	UJ	2.91	UJ	2.84	UJ	8.93	U	8.85	U	8.99	U	8.85	U
Tylosin	12	UJ	12.2	UJ	11.7	UJ	11.4	UJ	35.7	U	35.4	U	36	U	35.4	U
Virginiamycin	6.01	UJ	6.08	UJ	5.83	UJ	5.68	UJ	17.9	U	17.7	U	18	U	17.7	U
List 2 – Tetracyclines in Positive Ionization																
Anhydrochlortetracycline	34.5	UJ	30.6	U	29.4	U	30.2	U	99.3	U	98.3	U	99.9	U	102	UJ
Anhydrotetracycline	31.3	U	30.6	U	29.4	U	30.2	U	99.3	U	98.3	U	99.9	U	98.4	U
Chlortetracycline	12.5	U	12.7	UJ	11.7	U	12.1	U	14.8	UJ	12.6	UJ	12	U	12.8	UJ

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Demeclocycline	31.3	U	30.6	U	29.4	U	30.2	U	29.8	U	29.5	U	30	U	29.5	U
Doxycycline	12.5	U	12.2	U	11.7	U	12.1	U	39.7	U	39.3	U	40	U	39.4	U
4-Epianhydrochlortetracycline	125	U	122	U	117	U	121	U	397	U	393	U	400	U	394	U
4-Epianhydrotetracycline	31.3	U	30.6	U	29.4	U	30.2	U	32	UJ	29.5	U	30	U	30.5	UJ
4-Epichlortetracycline	31.3	U	30.6	U	29.4	U	30.2	U	29.8	U	29.5	U	30	U	29.5	U
4-Epioxytetracycline	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	12	U	11.8	U
4-Epitetracycline	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	12	U	11.8	U
Isochlortetracycline	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	12	U	11.8	U
Minocycline	158	UJ	145	UJ	139	UJ	141	UJ	119	U	118	U	120	U	118	UJ
Oxytetracycline	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	127	U	11.8	U
Tetracycline	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	12	U	11.8	U
List 3 - Acid Extraction in Negative Ionization																
Bisphenol A	2080	U	2040	U	1960	U	2010	U	993	U	983	U	999	U	984	U
Furosemide	83.4	U	81.5	U	78.3	U	80.5	U	79.4	U	78.7	U	79.9	U	78.7	U
Gemfibrozil	**86.8**		3.06	U	2.94	U	3.02	U	**16.9**		2.95	U	3	U	2.95	U
Glipizide	12.5	U	12.2	U	11.7	U	12.1	U	11.9	U	11.8	U	12	U	11.8	U
Glyburide	**7.17**		6.12	U	5.87	U	6.04	U	5.96	U	5.9	U	5.99	U	5.9	U
Hydrochlorothiazide	**190**	**J**	40.8	UJ	39.2	UJ	40.2	UJ	39.7	UJ	39.3	UJ	40	UJ	39.4	UJ
2-Hydroxy-ibuprofen	303	UJ	163	U	157	U	161	U	159	U	157	U	160	U	157	U
Ibuprofen	**140**		30.6	U	29.4	U	30.2	U	**80.2**		29.5	U	30	U	29.5	U
Naproxen	**10.6**		6.12	U	5.87	U	6.04	U	5.96	U	5.9	U	5.99	U	5.9	U
Triclocarban	**15.9**		6.12	U	5.87	U	6.04	U	**14.2**		5.9	U	5.99	U	5.9	U
Triclosan	129	U	126	U	121	U	124	U	119	U	118	U	120	U	118	U
Warfarin	3.13	U	3.06	U	2.94	U	3.02	U	2.98	U	2.95	U	3	U	2.95	U
List 4 - Basic Extraction in Positive Ionization																
Albuterol	0.589	U	0.64	U	0.599	U	0.598	U	1.89	U	1.94	U	1.98	U	1.99	U
Amphetamine	6.92	U	3.81	U	3	U	2.99	U	2.84	U	2.91	U	2.98	U	**6.04**	**NJ**

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Atenolol	**849**		1.28	U	1.2	U	1.46	UJ	**260**		2.29	UJ	2.07	UJ	2.18	UJ
Atorvastatin	2.95	U	3.2	U	3	U	2.99	U	2.84	U	2.91	U	2.98	U	2.99	U
Cimetidine	1.53	UJ	1.28	U	1.2	U	1.2	U	1.14	UJ	1.17	U	1.19	U	1.19	U
Clonidine	2.95	U	3.2	U	3	U	2.99	U	4.64	UJ	2.91	U	3.2	U	4.18	UJ
Codeine	5.89	U	6.67	UJ	5.99	U	6.29	UJ	6.37	UJ	9.83	UJ	7.38	UJ	8.77	UJ
Cotinine	**26.8**		3.2	U	3	U	2.99	U	**22.9**		2.91	U	2.98	U	2.99	U
Enalapril	0.589	U	0.64	U	0.599	U	0.598	U	0.568	U	0.583	U	0.595	U	0.597	U
Hydrocodone	2.95	U	3.2	U	3	U	2.99	U	2.84	UJ	2.91	UJ	2.98	UJ	2.99	UJ
Metformin	**1120**		6.64	UJ	9.96	UJ	5.98	U	**804**		10.9	UJ	15.5	UJ	24.1	UJ
Oxycodone	**12.7**		1.28	U	1.2	U	1.2	U	3.79	U	3.89	U	3.97	U	3.98	U
Ranitidine	1.18	U	1.28	U	1.2	U	1.2	U	1.14	U	1.17	U	1.19	U	1.19	U
Triamterene	**26.1**	**J**	1.01	UJ	0.945	UJ	1.06	UJ	1.89	U	1.97	UJ	1.98	U	1.99	U
List 5 - Acid Extraction in Positive Ionization																
Alprazolam	**4.42**	**J**	**1.43**	**J**	**1.14**	**J**	**1.23**	**J**	**4.13**		0.59	U	**0.725**		**1.39**	
Amitriptyline	**1.09**	**J**	0.631	UJ	0.583	UJ	0.568	UJ	**0.729**		0.59	U	0.599	U	0.59	U
Amlodipine	3.01	UJ	3.04	UJ	2.91	UJ	2.84	UJ	2.86	U	2.83	U	2.88	U	2.83	U
Benzoylecgonine	**12.5**	**J**	**1.39**	**J**	**1.95**	**J**	**1.32**	**J**	**28.8**		**1**		**1.51**		**1.86**	
Benztropine	0.601	UJ	0.608	UJ	0.583	UJ	0.568	UJ	0.596	U	0.59	U	0.599	U	0.59	U
Betamethasone	**5.06**	**J**	**3.18**	**J**	2.98	UJ	3.28	UJ	9.93	U	9.83	U	9.99	U	9.84	U
Cocaine	**0.926**	**J**	0.304	UJ	0.291	UJ	0.284	UJ	**0.438**		0.295	U	0.3	U	0.295	U
DEET	31.2	UJ	8.58	UJ	7.86	UJ	8.82	UJ	**92.9**		3.84	U	**15.5**		10.6	U
Desmethyldiltiazem	**0.67**	**J**	0.304	UJ	0.291	UJ	0.284	UJ		REJ	0.295	U	0.3	U	0.295	U
Diazepam	**1.75**	**J**	0.608	UJ	0.583	UJ	0.568	UJ	**1.77**		0.59	U	0.599	U	0.59	U
Fluocinonide	12	UJ	12.2	UJ	11.7	UJ	11.4	UJ	11.9	U	11.8	U	12	U	11.8	U
Fluticasone propionate	4.01	UJ	4.05	UJ	3.88	UJ	3.79	UJ	3.97	U	3.93	U	4	U	3.94	U
Hydrocortisone	120	UJ	122	UJ	117	UJ	114	UJ	119	U	118	U	120	U	118	U
10-hydroxy-amitriptyline	**0.328**	**J**	0.304	UJ	0.291	UJ	0.284	UJ	0.993	U	0.983	U	0.999	U	0.984	U

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Meprobamate	**466**	**J**	**17.8**	**J**	**30**	**J**	**18.3**	**J**	**380**		**12**		**26.9**		**34.4**	
Methylprednisolone	26.1	UJ	10.7	UJ	7.77	UJ	7.58	UJ	26.5	U	26.2	U	26.6	U	26.2	U
Metoprolol	**272**	**J**	3.04	UJ	5.12	UJ	3.73	UJ	**281**		3.65	UJ	3	U	5.76	UJ
Norfluoxetine	**3.43**	**J**	3.04	UJ	2.91	UJ	2.84	UJ	2.98	U	2.95	U	3	U	2.95	U
Norverapamil	**16.4**	**J**	0.304	UJ	2.91	UJ	0.284	UJ	**2.41**		0.295	U	0.3	U	0.295	U
Paroxetine	8.02	UJ	8.11	UJ	7.77	UJ	7.58	UJ	7.94	U	7.87	U	7.99	U	7.87	U
Prednisolone	12	UJ	12.2	UJ	11.7	UJ	11.4	UJ	19.1	UJ	11.8	U	14.7	UJ	12.4	UJ
Prednisone	40.1	UJ	40.5	UJ	38.8	UJ	37.9	UJ	97.7	UJ	58.8	UJ	44.7	UJ	68.4	UJ
Promethazine	0.802	UJ	0.811	UJ	0.777	UJ	0.758	UJ		REJ	0.787	U	0.799	U	0.787	U
Propoxyphene	**0.689**	**J**	0.608	UJ	0.583	UJ	0.568	UJ	0.596	U	0.59	U	0.599	U	0.59	U
Propranolol	**9.96**	**J**	4.05	UJ	3.88	UJ	3.79	UJ	3.97	U	3.93	U	4	U	3.94	U
Sertraline	**3.07**	**J**	0.811	UJ	0.777	UJ	0.758	UJ	**0.803**		0.787	U	0.799	U	0.787	U
Simvastatin	40.1	UJ	40.5	UJ	38.8	UJ	37.9	UJ	39.7	U	39.3	U	40	U	39.4	U
Theophylline	**240**	**J**	122	UJ	117	UJ	114	UJ	**180**		118	U	120	U	118	U
Trenbolone	8.02	UJ	8.11	UJ	7.77	UJ	7.58	UJ	9.01	UJ	7.87	U	9.06	UJ	7.87	U
Trenbolone acetate	0.601	UJ	0.608	UJ	0.583	UJ	0.568	UJ	0.596	U	0.59	U	0.599	U	0.59	U
Valsartan	**280**	**J**	8.11	UJ	7.77	UJ	7.58	UJ	**54.5**		7.87	U	7.99	U	7.87	U
Verapamil	**0.458**	**J**	0.304	UJ	0.291	UJ	0.284	UJ	0.298	U	0.295	U	0.3	U	0.295	U

*Parameter was detected in QA blank. Parameter value was rejected.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-3. Hormones and Sterols Analytical Results for LOTT Martin Way Reclaimed Water Plant/Hawks Prairie Satellite, May and October, 2011.

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Hormones																
17*B*-Estradiol*	88	UJ	*0.656*	*NJ*	*0.384*	*NJ*	*0.49*	*NJ*	8.77	U	8.81	UJ	8.51	UJ	8.53	UJ
17*a*-Estradiol	88.7	UJ	44.5	UJ	177	UJ	9.45	UJ	8.85	U	8.88	U	8.58	U	8.6	U
17*a*-Ethinyl Estradiol*	88.3	UJ	*0.573*	*NJ*	*0.475*	*NJ*	*0.621*	*NJ*	8.81	U	8.84	UJ	8.55	UJ	8.57	UJ
B-Estradiol-3-Benzoate	88.9	UJ	44.6	UJ	178	UJ	9.46	UJ	8.86	U	8.89	U	8.6	U	8.62	U
17*a*-Dihydroequilin	**14**	**J**	43.8	UJ	175	UJ	9.29	UJ	**14.8**		8.73	U	8.45	U	8.46	U
Androstenedione	220	UJ	111	UJ	441	UJ	23.5	UJ	95.8	UJ	22.1	U	21.3	U	23.2	UJ
Androsterone	88.5	UJ	44.4	UJ	177	UJ	9.43	UJ	8.83	U	8.86	U	8.57	U	8.58	U
Desogestrel	88.3	UJ	44.3	UJ	177	UJ	9.41	UJ	18.6	UJ	8.84	U	8.55	U	8.57	U
Equilenin	91.4	UJ	45.9	UJ	183	UJ	9.74	UJ	9.12	U	9.15	UJ	8.85	UJ	8.87	U
Equilin	87.2	UJ	43.7	UJ	174	UJ	9.28	UJ	12.6	UJ	8.73	U	8.44	U	8.45	U
Estriol	88.9	UJ	44.6	UJ	178	UJ	9.46	UJ	8.86	U	8.89	U	8.6	U	8.62	U
Estrone	88.2	UJ	44.3	UJ	177	UJ	9.4	UJ	9.73	UJ	8.83	U	8.54	U	8.56	U
Mestranol*	177	UJ	44.4	UJ	177	UJ	*0.592*	*NJ*	*2.08*	*J*	8.87	U	8.57	U	8.59	U
Norethindrone	177	UJ	44.3	UJ	177	UJ	9.41	UJ	8.81	U	8.84	U	8.55	U	8.57	U
Norgestrel	88.7	UJ	44.5	UJ	177	UJ	9.45	UJ	8.85	U	8.88	U	8.58	UJ	8.6	UJ
Progesterone	220	UJ	111	UJ	441	UJ	23.5	UJ	26	UJ	22	U	21.3	U	21.4	U
Testosterone	88.3	UJ	44.3	UJ	177	UJ	9.41	UJ	22.5	UJ	8.84	U	8.55	U	8.57	U
Sterols																
Campesterol*	443	UJ	222	UJ	*235*	*J*	*51.3*		*69.5*		44.3	U	*27*	*J*	*4.47*	*J*
Cholestanol*	440	UJ	221	UJ	44	UJ	46.8	UJ	*160*		*9.94*	*J*	*2.18*	*J*	*3.12*	*J*
Cholesterol*	1100	UJ	552	UJ	349	UJ	181	UJ	*666*		*574*		108	U	168	U
Coprostanol	1080	UJ	542	UJ	108	UJ	115	UJ	**436**		108	U	105	U	105	U
Desmosterol	1090	UJ	549	UJ	109	UJ	116	UJ	109	U	**21.1**	**J**	106	U	106	U
Epicoprostanol	1150	UJ	576	UJ	115	UJ	122	UJ	**50.3**	**J**	115	U	111	U	111	U

Analyte	LOTTEFF		LOTTMW-1		LOTTMW-8		LOTTMW-9		LOTTEFF		LOTTMW-2		LOTTMW-8		LOTTMW-9	
	May 2011								October 2011							
Ergosterol	1100	UJ	551	UJ	110	UJ	117	UJ	**10**	**NJ**	110	UJ	106	UJ	106	UJ
B-Sitosterol	1090	UJ	698	UJ	**10300**	**J**	1480	UJ	**215**	**J**	109	UJ	**1240**	**J**	113	UJ
B-Stigmastanol*	1100	UJ	551	UJ	*87*	*J*	*22.8*	*NJ*	*26.6*	*J*	110	UJ	*12.3*	*J*	106	UJ
Stigmasterol*	450	UJ	226	UJ	*2220*	*J*	*487*		*211*	*J*	45.1	UJ	*270*	*J*	43.7	UJ

*Parameter was detected in QA blank. Parameter was considered non-detect in report.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-4. Pharmaceuticals and Personal Care Products Analytical Results for Quincy Reclaimed Water Plant, May and October, 2011.

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
List 1 - Acid Extraction in Positive Ionization																
Acetaminophen	22.1	UJ	27.2	UJ	27.1	UJ	30.7	UJ	**31.1**		29.3	U	29.6	U	29.1	U
Azithromycin	**189**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**79.2**		2.93	U	2.96	U	2.91	U
Caffeine	**78.7**	**J**	27.2	UJ	27.1	UJ	30.7	UJ	**1390**		29.3	U	29.6	U	29.1	U
Carbadox	2.21	UJ	2.72	UJ	2.71	UJ	3.07	UJ	2.88	U	2.93	U	2.96	U	2.91	U
Carbamazepine	**170**	**J**	**454**	**J**	**288**	**J**	3.07	UJ	**511**		**431**		**332**		2.91	U
Cefotaxime	155	UJ	229	UJ	137	UJ	138	UJ		REJ		REJ		REJ		REJ
Ciprofloxacin	**67.7**	**J**	29.3	UJ	28.7	UJ	12.3	UJ	**202**		14.8	UJ	15.9	UJ	13.5	UJ
Clarithromycin	**18.9**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**64.5**		2.93	U	2.96	U	2.91	U
Clinafloxacin	72.6	UJ	67.2	UJ	66.9	UJ	66.2	UJ	53.8	UJ	47.7	UJ	31.3	UJ	26.7	UJ
Cloxacillin	4.56	UJ	5.45	UJ	5.42	UJ	6.14	UJ	6.87	UJ	5.87	UJ	5.91	UJ	5.82	UJ
Dehydronifedipine	**6.68**	**J**	3.63	UJ	3.61	UJ	4.09	UJ	**2.59**		1.17	U	1.18	U	1.16	U
Digoxigenin	134	UJ	85.8	UJ	63.1	UJ	39.5	UJ	11.5	U	11.7	U	11.8	U	11.6	U
Digoxin	8.84	UJ	10.9	UJ	10.8	UJ	12.3	UJ	11.5	U	11.7	U	11.8	U	11.6	U
Diltiazem	**43**	**J**	0.545	UJ	0.542	UJ	0.614	UJ	**84.7**		0.587	U	0.591	U	0.582	U
1,7-Dimethylxanthine	88.4	UJ	109	UJ	108	UJ	123	UJ	**2040**		117	U	118	U	116	U
Diphenhydramine	**166**	**J**	1.09	UJ	1.08	UJ	1.23	UJ	**345**	**J**	1.17	UJ	1.18	UJ	1.16	UJ
Enrofloxacin	4.42	UJ	5.45	UJ	5.42	UJ	6.14	UJ	6.49	UJ	5.87	U	5.91	U	5.82	U
Erythromycin-H20	**19.3**	**J**	1.82	UJ	1.81	UJ	2.05	UJ	**20**		0.587	U	0.591	U	0.582	U
Flumequine	2.61	UJ	3.68	UJ	3.29	UJ	3.07	UJ	10.8	UJ	2.93	U	4.11	UJ	2.91	U
Fluoxetine	**11.7**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**7.67**		2.93	U	2.96	U	2.91	U
Lincomycin	4.42	UJ	5.45	UJ	5.42	UJ	6.14	UJ	5.78	UJ	5.87	U	5.91	U	5.82	U
Lomefloxacin	9.88	UJ	5.45	UJ	5.8	UJ	6.14	UJ	26.1	UJ	7.63	UJ	6.53	UJ	5.82	U
Miconazole	2.21	UJ	2.72	UJ	2.71	UJ	3.07	UJ	**3.54**		2.93	U	2.96	U	2.91	U
Norfloxacin	77.5	UJ	74.8	UJ	39.5	UJ	30.7	UJ	62.3	UJ	29.3	U	29.6	U	29.1	U

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
Norgestimate	17.4	UJ	18.2	UJ	18.1	UJ	20.5	UJ	5.77	U	5.87	U	5.91	U	5.82	U
Ofloxacin	**24.4**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**10.4**		3.03	UJ	2.96	U	2.91	U
Ormetoprim	0.884	UJ	1.09	UJ	1.08	UJ	1.23	UJ	1.15	U	1.17	U	1.18	U	1.16	U
Oxacillin	4.42	UJ	5.45	UJ	5.42	UJ	6.14	UJ	**14.7**		5.87	U	5.91	U	5.82	U
Oxolinic acid	2.14	UJ	2.03	UJ	1.08	UJ	1.23	UJ	4.81	UJ	1.17	U	1.18	U	1.16	U
Penicillin G	4.42	UJ	5.45	UJ	5.42	UJ	6.14	UJ	5.77	U	5.87	U	5.91	U	5.82	U
Penicillin V	14.7	UJ	18.2	UJ	18.1	UJ	20.5	UJ	8.99	UJ	5.87	U	5.91	U	5.82	U
Roxithromycin	0.442	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.577	U	0.587	U	0.591	U	0.582	U
Sarafloxacin	22.1	UJ	29.8	UJ	27.1	UJ	30.7	UJ	65	UJ	29.3	U	29.6	U	29.1	U
Sulfachloropyridazine	2.21	UJ	2.72	UJ	2.71	UJ	3.07	UJ	2.88	U	2.93	U	2.96	U	2.91	U
Sulfadiazine	**34.7**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**29.4**		2.93	U	2.96	U	2.91	U
Sulfadimethoxine	**7.58**	**J**	2.67	UJ	2.17	UJ	2.08	UJ	**59.8**		1.63	UJ	**2.02**		0.582	U
Sulfamerazine	2.95	UJ	3.63	UJ	3.61	UJ	4.09	UJ	2.8	UJ	1.17	U	1.18	U	1.16	U
Sulfamethazine	**14.8**	**J**	3.63	UJ	3.61	UJ	4.09	UJ	6.84	UJ	1.4	UJ	2.21	UJ	1.2	UJ
Sulfamethizole	2.95	UJ	3.63	UJ	3.61	UJ	4.09	UJ	12.3	UJ	3.14	UJ	1.18	U	1.16	U
Sulfamethoxazole	**505**	**J**	**260**	**J**	**18.6**	**J**	4.09	UJ	**167**		**41.3**	**J**	**58.8**		1.16	U
Sulfanilamide	22.1	UJ	**44.7**	**J**	27.1	UJ	30.7	UJ	28.8	U	**48.2**		**55.9**		29.1	U
Sulfathiazole	2.49	UJ	2.72	UJ	2.71	UJ	3.07	UJ	5.7	UJ	2.93	U	2.96	U	2.91	U
Thiabendazole	**1040**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**119000**	**J**	2.93	U	2.96	U	2.91	U
Trimethoprim	**313**	**J**	2.72	UJ	2.71	UJ	3.07	UJ	**174**		2.93	U	4.04	UJ	2.91	U
Tylosin	8.84	UJ	10.9	UJ	10.8	UJ	12.3	UJ	11.5	UJ	11.7	UJ	11.8	UJ	11.6	UJ
Virginiamycin	4.42	UJ	5.45	UJ	5.42	UJ	6.14	UJ	48.4	UJ	6.91	UJ	7.47	UJ	5.82	U
List 2 – Tetracyclines in Positive Ionization																
Anhydrochlortetracycline	25	UJ	28.5	UJ	32.5	UJ	30.2	U	128	UJ	58.5	UJ	57.5	UJ	46.6	UJ
Anhydrotetracycline	27.4	UJ	25.7	U	28.8	U	30.2	U	43.2	UJ	29.3	U	33.7	UJ	29.1	U
Chlortetracycline	9.04	UJ	11.6	UJ	12.9	UJ	12.1	U	38.5	U	39.1	U	39.4	U	38.8	U
Demeclocycline	21.3	U	25.7	U	28.8	U	30.2	U	96.1	U	97.8	U	98.5	U	97	U

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
Doxycycline	**21.7**		10.3	U	11.5	U	12.1	U	**42.1**		11.7	U	11.8	U	11.6	U
4-Epianhydrochlortetracycline	85.1	U	103	U	115	U	121	U	281	UJ	118	UJ	118	U	116	U
4-Epianhydrotetracycline	38.4	UJ	28.3	UJ	30.4	UJ	30.2	U	56.9	UJ	29.3	U	38.6	UJ	29.1	U
4-Epichlortetracycline	21.3	U	25.7	U	28.8	U	30.2	U	40.6	UJ	29.3	U	31	UJ	29.1	U
4-Epioxytetracycline	12.6	UJ	16.6	UJ	13.3	UJ	12.1	U	43.2	UJ	25.3	UJ	23.1	UJ	14	UJ
4-Epitetracycline	**26.1**		16.5	UJ	13.6	UJ	12.1	U	43.9	UJ	16.4	UJ	18.3	UJ	17.9	UJ
Isochlortetracycline	8.51	U	10.3	U	11.5	U	12.1	U	11.5	U	11.7	U	11.8	U	11.6	U
Minocycline	120	UJ	136	UJ	143	UJ	155	UJ	115	U	117	U	118	U	116	U
Oxytetracycline	8.51	U	10.3	U	11.5	U	12.1	U	11.5	U	11.7	U	11.8	U	11.6	U
Tetracycline	**38**		10.3	U	11.5	U	12.1	U	**36.3**		11.7	U	11.8	U	11.6	U
List 3 - Acid Extraction in Negative Ionization																
Bisphenol A	1420	U	1720	U	1920	U	2020	U	961	U	978	U	985	U	970	U
Furosemide	**263**		68.7	U	76.7	U	80.7	U	**485**		78.2	U	78.8	U	77.6	U
Gemfibrozil	**142**		2.57	U	2.88	U	3.02	U	**542**		2.93	U	2.96	U	2.91	U
Glipizide	8.51	U	10.3	U	11.5	U	12.1	U	11.5	U	11.7	U	11.8	U	11.6	U
Glyburide	4.26	U	5.15	U	5.75	U	6.05	U	5.77	U	5.87	U	5.91	U	5.82	U
Hydrochlorothiazide	**117**	**J**	34.3	UJ	38.4	UJ	40.3	UJ	**98.3**		39.1	U	39.4	U	38.8	U
2-Hydroxy-ibuprofen	**389**		137	U	153	U	161	U	**1310**		156	U	158	U	155	U
Ibuprofen	21.3	U	25.7	U	28.8	U	30.2	U	**172**		29.3	U	29.6	U	29.1	U
Naproxen	**101**		5.15	U	5.75	U	6.05	U	**262**		5.87	U	5.91	U	5.82	U
Triclocarban	4.26	U	5.15	U	5.75	U	6.05	U	**40.8**		5.87	U	5.91	U	5.82	U
Triclosan	87.7	U	106	U	119	U	125	U	**158**		117	U	118	U	116	U
Warfarin	2.13	U	2.57	U	2.88	U	3.02	U	2.88	U	2.93	U	2.96	U	2.91	U
List 4 - Basic Extraction in Positive Ionization																
Albuterol	**15.6**		0.508	U	0.602	U	0.593	UJ	**10.5**	**J**	0.595	U	0.591	U	0.569	U
Amphetamine	4.81	U	2.54	U	4.41	U	2.71	U	11.4	UJ	6.88	UJ	7.22	UJ	6.31	UJ
Atenolol	**321**		1.09	UJ	1.3	UJ	1.23	UJ	**542**		1.19	U	1.18	U	1.14	U

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
Atorvastatin	**5.34**		2.54	U	3.01	U	2.71	U	**10.8**		2.98	U	2.95	U	2.85	U
Cimetidine	**20.3**		1.02	U	1.2	U	1.09	U	**90.5**		1.19	U	1.18	U	1.14	U
Clonidine	2.06	U	2.54	U	3.01	U	2.71	U	2.98	U	2.98	U	2.95	U	2.85	U
Codeine	**14**	**J**	5.08	U	6.02	U	5.43	U	**30.4**		6.4	UJ	5.91	U	13	UJ
Cotinine	**30.6**		2.54	U	3.01	U	2.71	U	**215**		2.98	U	2.95	U	2.85	U
Enalapril	0.413	U	0.508	U	0.602	U	0.543	U	**2.66**		0.595	U	0.804	UJ	0.569	U
Hydrocodone	**17.4**		2.54	U	3.01	U	2.71	U	**22.1**		2.98	U	2.95	U	2.85	U
Metformin	**3320**		11.1	UJ	12.3	UJ	6.09	UJ	**1030**		36	UJ	37.3	UJ	23	UJ
Oxycodone	**20.3**		1.61	UJ	1.78	UJ	1.28	UJ	**37.4**		1.19	U	1.18	U	1.14	U
Ranitidine	**386**		1.02	U	1.2	U	1.09	U	**336**		1.52	UJ	4.68	UJ	1.28	UJ
Triamterene	**51.1**	**J**	0.828	UJ	0.904	UJ	0.771	UJ	**64.5**		1.98	U	1.97	U	1.9	U
List 5 - Acid Extraction in Positive Ionization																
Alprazolam	1.33	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.577	U	0.587	U	0.591	U	0.582	U
Amitriptyline	**20.7**	**J**	0.545	UJ	0.542	UJ	0.614	UJ	**29.2**		0.587	U	0.591	U	0.582	U
Amlodipine	6.63	UJ	2.72	UJ	2.71	UJ	3.07	UJ	**5.48**		2.82	U	2.84	U	2.79	U
Benzoylecgonine	**41.6**	**J**	0.545	UJ	0.542	UJ	0.614	UJ	**16.8**		0.587	U	0.591	U	0.582	U
Benztropine	1.33	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.577	U	0.587	U	0.591	U	0.582	U
Betamethasone	6.33	UJ	2.72	UJ	2.71	UJ	3.67	UJ	2.88	U	2.93	U	2.96	U	2.91	U
Cocaine	**7.19**	**J**	0.272	UJ	0.271	UJ	0.307	UJ	**0.484**		0.293	U	0.296	U	0.291	U
DEET	**685**	**J**	4.91	UJ	2.45	UJ	3.97	UJ	**34.7**		3.13	U	5.95	U	2.49	U
Desmethyldiltiazem	**11.9**	**J**	0.272	UJ	0.271	UJ	0.307	UJ	**14.4**		0.293	U	0.296	U	0.291	U
Diazepam	1.33	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.577	U	0.587	U	0.591	U	0.582	U
Fluocinonide	26.5	UJ	10.9	UJ	10.8	UJ	12.3	UJ	11.5	U	11.7	U	11.8	U	11.6	U
Fluticasone propionate	8.84	UJ	3.63	UJ	3.61	UJ	4.09	UJ	5.57	UJ	3.91	U	3.94	U	3.88	U
Hydrocortisone	265	UJ	109	UJ	108	UJ	123	UJ	153	UJ	117	U	118	U	116	U
10-hydroxy-amitriptyline	**3.13**	**J**	0.272	UJ	0.271	UJ	0.337	UJ	**16.6**		0.293	U	0.296	U	0.291	U
Meprobamate	**43.9**	**J**	**19.2**	**J**	7.23	UJ	8.18	UJ	**33.4**		**10.6**		7.88	U	7.76	U

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
Methylprednisolone	17.7	UJ	8.56	UJ	9.08	UJ	18.9	UJ	26.6	U	26.1	U	26.3	U	25.9	U
Metoprolol	**742**	**J**	6.18	UJ	3.16	UJ	3.07	UJ	**339**		4.46	UJ	6.81	UJ	3.56	UJ
Norfluoxetine	6.63	UJ	2.72	UJ	2.71	UJ	3.07	UJ	2.88	U	2.93	U	2.96	U	2.91	U
Norverapamil	**0.841**	**J**	0.272	UJ	0.271	UJ	0.307	UJ	**0.484**		0.293	U	0.296	U	0.291	U
Paroxetine	17.7	UJ	7.26	UJ	7.23	UJ	8.18	UJ	**8.53**		7.82	U	7.88	U	7.76	U
Prednisolone	26.5	UJ	10.9	UJ	10.8	UJ	12.3	UJ	11.5	U	11.7	U	11.8	U	11.6	U
Prednisone	88.4	UJ	36.3	UJ	36.1	UJ	40.9	UJ	199	UJ	50.4	UJ	60.8	UJ	52.9	UJ
Promethazine	1.77	UJ	0.726	UJ	0.723	UJ	0.818	UJ	0.769	U	0.782	U	0.788	U	0.776	U
Propoxyphene	1.33	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.577	U	0.587	U	0.591	U	0.582	U
Propranolol	**72.9**	**J**	3.63	UJ	3.61	UJ	4.09	UJ	**88.7**		3.91	U	3.94	U	3.88	U
Sertraline	**11.3**	**J**	0.726	UJ	0.723	UJ	0.818	UJ	**18.4**		0.782	U	0.788	U	0.776	U
Simvastatin	88.4	UJ	36.3	UJ	36.1	UJ	40.9	UJ	38.5	U	39.1	U	39.4	U	38.8	U
Theophylline	265	UJ	109	UJ	108	UJ	123	UJ	**1570**		117	U	118	U	116	U
Trenbolone	17.7	UJ	7.26	UJ	7.23	UJ	8.18	UJ	7.69	U	7.82	U	7.88	U	7.76	U
Trenbolone acetate	1.33	UJ	0.545	UJ	0.542	UJ	0.614	UJ	0.903	UJ	0.587	U	0.591	U	0.582	U
Valsartan	**19**	**J**	7.26	UJ	7.23	UJ	8.18	UJ	**400**		7.82	U	7.88	U	7.76	U
Verapamil	**8.31**	**J**	0.272	UJ	0.271	UJ	0.307	UJ	**3.47**		0.293	U	0.296	U	0.291	U

Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-5. Hormones and Sterols Analytical Results for Quincy Reclaimed Water Plant, May and October, 2011.

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
Hormones																
17*B*-Estradiol*	8.8	U	9.14	U	*0.506*	*NJ*	*0.394*	*NJ*	181	U	194	U	189	U	9.64	U
17*a*-Estradiol	8.87	U	9.22	U	11.9	U	9.01	U	182	U	196	U	191	U	9.72	U
17*a*-Ethinyl Estradiol*	8.83	U	9.18	U	11.8	U	*0.619*	*NJ*	182	U	195	U	190	U	9.68	U
B-Estradiol-3-Benzoate	8.89	U	9.24	U	11.9	U	9.03	U	183	U	196	U	191	U	9.74	U
17*a*-Dihydroequilin	**63.6**		9.07	U	11.7	U	8.86	U	179	U	193	U	188	U	9.56	U
Androstenedione	134	UJ	70	UJ	94.6	UJ	47.6	UJ	453	U	486	U	474	U	24.2	U
Androsterone	11.4	UJ	9.2	U	11.8	U	8.99	U	182	U	195	U	190	U	9.7	U
Desogestrel	33	UJ	13	UJ	11.8	U	8.97	U	182	U	195	U	190	U	9.68	U
Equilenin	15	UJ	11.1	UJ	12.2	U	9.29	U	188	U	202	U	197	U	10	U
Equilin	25.2	UJ	9.06	U	11.7	U	8.86	U	179	U	192	U	188	U	9.55	U
Estriol	8.89	U	9.24	U	11.9	U	9.03	U	183	U	196	U	191	U	9.74	U
Estrone	8.82	U	9.17	U	11.8	U	8.96	U	181	U	195	U	190	U	9.67	U
Mestranol	22.1	UJ	9.21	U	11.9	U	9	U	182	U	195	U	191	U	9.71	U
Norethindrone	15	UJ	10.7	U	11.8	U	8.97	U	182	U	195	U	190	U	9.68	U
Norgestrel	8.87	U	9.22	U	11.9	U	9.01	U	182	U	196	U	191	U	9.72	U
Progesterone	22	U	**13.5**	**NJ**	29.5	U	22.4	U	453	U	486	U	474	U	24.1	U
Testosterone	28.9	UJ	9.18	U	11.8	U	8.97	U	182	U	**24.1**	**J**	190	U	9.68	U
Sterols																
Campesterol*	*273*		46	U	59.3	U	45	U	*859*	*NJ*	*331*	*J*	953	U	48.5	U
Cholestanol*	*264*		45.7	U	58.8	U	44.7	U	*557*	*J*	970	U	946	U	48.2	U
Cholesterol*	*1840*		211	U	480	U	650	U	*1940*	*J*	465	U	2370	U	212	U
Coprostanol	**989**		112	U	145	U	110	U	**2910**		2380	U	2330	U	118	U
Desmosterol	**141**	**NJ**	**3.39**	**J**	**7.44**	**NJ**	**4.29**	**NJ**	2250	U	2410	U	2350	U	120	U
Epicoprostanol	**84.7**	**J**	119	U	154	U	117	U	**128**	**J**	2530	U	2470	U	126	U
Ergosterol	110	UJ	114	UJ	147	UJ	112	UJ	2260	UJ	2420	UJ	2360	UJ	120	UJ

Analyte	QuincyEff		Quinw-26		Quinw-60		Quinwo-1		QuincyEff		Quinw-26		Quinw-60		Quinwo-1	
	May 2011								October 2011							
B-Sitosterol	1120	U	628	U	320	U	111	U	**14800**	**J**	**13400**	**J**	**4290**	**J**	120	UJ
B-Stigmastanol*	*82.1*	*J*	114	U	147	U	111	U	*444*	*NJ*	2420	U	2360	U	120	U
Stigmasterol*	*5420*		167	U	92.3	U	45.7	U	*51400*	*J*	*2550*	*NJ*	*810*	*NJ*	49.3	UJ

*Parameter was detected in QA blank. Parameter was considered non-detect in report.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-6. Pharmaceuticals and Personal Care Products Analytical Results for Yelm Water Reclamation Facility, October 2011.

Analyte	YelmEFF		YELMW-1		YELMW-3		YELMW-4	
List 1 - Acid Extraction in Positive Ionization								
Acetaminophen	235	U	77.2	U	82.9	U	81.5	U
Azithromycin	23.5	U	7.72	U	8.29	U	8.15	U
Caffeine	235	U	77.2	U	82.9	U	81.5	U
Carbadox	23.5	U	7.72	U	8.29	U	8.15	U
Carbamazepine	**216**	**J**	7.72	U	**76.6**		**118**	**J**
Cefotaxime	5.8	UJ	6.35	UJ	6.82	UJ	6.7	UJ
Ciprofloxacin	94	U	30.9	U	33.2	U	32.6	U
Clarithromycin	**42.6**		7.72	U	8.29	U	8.15	U
Clinafloxacin	313	U	80.9	UJ	33.2	U	109	U
Cloxacillin	47	U	15.4	U	16.6	U	16.3	U
Dehydronifedipine	9.4	U	3.09	U	3.32	U	3.26	U
Digoxigenin	172	UJ	37.4	UJ	33.2	U	65.3	UJ
Digoxin	94	U	30.9	U	33.2	U	32.6	U
Diltiazem	**22.9**		1.54	U	1.66	U	1.63	U
1,7-Dimethylxanthine	940	U	309	U	332	U	326	U
Diphenhydramine	**127**	**J**	3.09	UJ	3.32	UJ	3.26	UJ
Enrofloxacin	47	U	15.4	U	16.6	U	16.3	U
Erythromycin-H20	**12.3**		1.54	U	1.66	U	1.63	U
Flumequine	78.3	U	25.7	U	27.6	U	27.2	U
Fluoxetine	**27.1**	**J**	7.72	U	8.29	U	8.15	U
Lincomycin	47	U	15.4	U	16.6	U	16.3	U
Lomefloxacin	47	U	15.8	UJ	16.6	U	16.3	U
Miconazole	23.5	U	7.72	U	8.29	U	8.15	U
Norfloxacin	235	U	77.2	U	82.9	U	81.5	U
Norgestimate	47	U	15.4	U	16.6	U	16.3	U
Ofloxacin	**46.5**		7.72	U	8.29	U	8.15	U
Ormetoprim	9.4	U	3.09	U	3.32	U	3.26	U
Oxacillin	47	U	15.4	U	16.6	U	16.3	U
Oxolinic acid	9.4	U	3.09	U	3.32	U	3.26	U
Penicillin G	157	U	15.4	U	16.6	U	54.4	U
Penicillin V	47	U	15.4	U	16.6	U	16.3	U
Roxithromycin	4.7	U	1.54	U	1.66	U	1.63	U
Sarafloxacin	235	U	77.2	U	82.9	U	81.5	U
Sulfachloropyridazine	23.5	U	7.72	U	8.29	U	8.15	U
Sulfadiazine	23.5	U	7.72	U	8.29	U	8.15	U
Sulfadimethoxine	4.7	U	1.54	U	1.66	U	1.63	U

Analyte	YelmEFF		YELMW-1		YELMW-3		YELMW-4	
Sulfamerazine	9.4	U	3.09	U	3.32	U	3.26	U
Sulfamethazine	9.4	U	3.09	U	3.32	U	3.26	U
Sulfamethizole	9.4	U	3.09	U	3.32	U	3.26	U
Sulfamethoxazole	**155**		3.09	U	**25.8**		**39.3**	
Sulfanilamide	235	U	77.2	U	82.9	U	81.5	U
Sulfathiazole	23.5	U	7.72	U	8.29	U	8.15	U
Thiabendazole*	*25.2*	*REJ*	7.72	U	8.29	U	8.15	U
Trimethoprim	**46.2**		7.72	U	8.29	U	8.15	U
Tylosin	94	U	30.9	U	33.2	U	32.6	U
Virginiamycin	47	U	15.4	U	16.6	U	16.3	U
List 2 – Tetracyclines in Positive Ionization								
Anhydrochlortetracycline	87.3	UJ	85.5	U	92.1	U	96.4	UJ
Anhydrotetracycline	78.3	U	85.5	U	92.1	U	90.6	U
Chlortetracycline	12	UJ	10.3	U	11.1	UJ	10.9	U
Demeclocycline	23.5	U	25.7	U	27.6	U	27.2	U
Doxycycline	31.8	UJ	34.3	U	36.8	U	36.2	U
4-Epianhydrochlortetracycline	313	U	343	U	368	U	362	U
4-Epianhydrotetracycline	30.6	UJ	25.7	U	27.6	U	27.7	UJ
4-Epichlortetracycline	23.5	U	25.7	U	27.6	U	27.2	U
4-Epioxytetracycline	14.7	UJ	10.3	U	11.1	U	12.4	UJ
4-Epitetracycline	15.6	UJ	10.3	U	11.1	U	10.9	U
Isochlortetracycline	9.4	U	10.3	U	11.1	U	10.9	U
Minocycline	94	U	103	U	111	U	109	U
Oxytetracycline	10.8	UJ	10.3	U	11.1	U	10.9	U
Tetracycline	12	UJ	10.3	U	11.1	U	10.9	U
List 3 - Acid Extraction in Negative Ionization								
Bisphenol A	783	U	858	U	921	U	906	U
Furosemide	62.7	U	68.6	U	73.7	U	72.5	U
Gemfibrozil	**63.9**		2.57	U	2.76	U	2.72	U
Glipizide	9.4	U	10.3	U	11.1	U	10.9	U
Glyburide	4.7	U	5.15	U	5.53	U	5.44	U
Hydrochlorothiazide	**147**	**J**	34.3	UJ	36.8	UJ	36.2	UJ
2-Hydroxy-ibuprofen	125	U	137	U	147	U	145	U
Ibuprofen	23.5	U	25.7	U	27.6	U	27.2	U
Naproxen	**24.1**	**J**	5.15	U	5.53	U	5.44	U
Triclocarban	**42.2**		5.15	U	5.53	U	5.44	U
Triclosan	94	U	103	U	111	U	109	U
Warfarin	2.35	U	2.57	U	2.76	U	2.72	U
List 4 - Basic Extraction in Positive Ionization								
Albuterol	**10.7**		2.11	U	2	U	1.71	U

Analyte	YelmEFF		YELMW-1		YELMW-3		YELMW-4	
Amphetamine	**14.8**	**NJ**	3.17	U	3	U	2.56	U
Atenolol	**453**		1.53	UJ	2.16	UJ	1.28	UJ
Atorvastatin	2.23	U	3.17	U	3	U	2.56	U
Cimetidine	0.894	U	1.27	U	1.2	U	1.03	U
Clonidine	2.71	UJ	4.66	UJ	3.31	UJ	2.56	U
Codeine	**28.4**		6.33	U	6.1	UJ	6.81	UJ
Cotinine	**16.6**		3.17	U	3	U	2.56	U
Enalapril	0.447	U	0.633	U	0.599	U	0.513	U
Hydrocodone	**42.6**	**J**	3.17	UJ	3	UJ	2.56	UJ
Metformin	**2080**		8.66	UJ	13.5	UJ	21.4	UJ
Oxycodone	**54.6**		4.22	U	4	U	3.42	U
Ranitidine	1.75	UJ	1.27	U	1.2	U	1.03	U
Triamterene	**89.8**	**J**	2.11	U	2.13	UJ	1.73	UJ
List 5 - Acid Extraction in Positive Ionization								
Alprazolam	**4.51**		0.515	U	0.553	U	**0.752**	
Amitriptyline	**30.5**		0.515	U	0.553	U	0.696	UJ
Amlodipine	2.26	U	2.47	U	2.65	U	2.61	U
Benzoylecgonine	**17.2**		0.515	U	0.553	U	0.544	U
Benztropine	0.47	U	0.515	U	0.553	U	0.544	U
Betamethasone	8.36	UJ	8.58	U	9.21	U	9.06	U
Cocaine	**0.655**		0.257	U	0.276	U	0.272	U
DEET	**23.5**		3	U	**19.4**		**21**	
Desmethyldiltiazem	**81.3**		0.257	U	0.276	U	0.272	U
Diazepam	**2.08**		0.515	U	0.553	U	0.544	U
Fluocinonide	9.4	U	10.3	U	11.1	U	10.9	U
Fluticasone propionate	6.59	UJ	3.43	U	3.68	U	3.62	U
Hydrocortisone	197	UJ	103	U	111	U	109	U
10-hydroxy-amitriptyline	**2.1**		0.858	U	0.921	U	0.906	U
Meprobamate	**1420**		6.86	U	**152**		**190**	
Methylprednisolone	20.9	U	22.9	U	24.6	U	24.2	U
Metoprolol	**597**		2.57	U	3.91	UJ	4.92	UJ
Norfluoxetine	**4.16**		2.57	U	2.76	U	2.72	U
Norverapamil	**3.23**		0.257	U	0.276	U	0.272	U
Paroxetine	6.27	U	6.86	U	7.37	U	7.25	U
Prednisolone	24.6	UJ	10.6	UJ	12.3	UJ	17.9	UJ
Prednisone	195	UJ	47.7	UJ	85.3	UJ	55.1	UJ
Promethazine	0.627	U	0.686	U	0.737	U	0.725	U
Propoxyphene	**2.73**		0.515	U	0.553	U	0.544	U
Propranolol	**86**		3.43	U	3.68	U	3.62	U
Sertraline	**7.42**		0.686	U	0.737	U	0.725	U

Analyte	YelmEFF		YELMW-1		YELMW-3		YELMW-4	
Simvastatin	31.3	U	34.3	U	36.8	U	36.2	U
Theophylline	**331**		103	U	111	U	109	U
Trenbolone	6.38	UJ	6.86	U	7.37	U	7.25	U
Trenbolone acetate	0.47	U	0.515	U	0.553	U	0.544	U
Valsartan	**127**		6.86	U	7.37	U	7.25	U
Verapamil	**1.18**		0.257	U	0.276	U	0.272	U

*Parameter was detected in QA blank. Parameter value was rejected.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-7. Hormones and Sterols Analytical Results for Yelm Water Reclamation Facility, October 2011.

Analyte	YelmEFF		YELMW-1		YELMW-3		YELMW-4	
Hormones								
17*B*-Estradiol	8.9	UJ	8.54	U	8.77	U	8.59	U
17*a*-Estradiol	8.97	UJ	8.61	U	8.84	U	8.66	U
17*a*-Ethinyl Estradiol	8.93	UJ	8.58	U	8.81	U	8.63	U
B-Estradiol-3-Benzoate	8.98	UJ	8.63	U	8.86	U	8.68	U
17*a*-Dihydroequilin	**11.5**	**J**	8.47	U	8.7	U	8.52	U
Androstenedione	52.1	UJ	21.4	U	22	U	30.1	UJ
Androsterone	8.95	UJ	8.59	U	8.82	U	8.65	U
Desogestrel	24.3	UJ	8.58	U	8.81	U	8.63	U
Equilenin	9.25	UJ	8.88	U	9.12	U	8.93	U
Equilin	8.81	UJ	8.46	U	8.69	U	8.52	U
Estriol	8.98	UJ	8.63	U	8.86	U	8.68	U
Estrone	8.92	UJ	8.57	U	8.8	U	8.62	U
Mestranol	8.96	UJ	8.6	U	8.83	U	8.65	U
Norethindrone	8.93	UJ	8.58	U	8.81	U	8.63	U
Norgestrel	8.97	UJ	8.61	U	8.84	U	8.66	U
Progesterone	22.3	UJ	21.4	U	22	U	21.5	U
Testosterone	29.1	UJ	8.58	U	8.81	U	8.63	U
Sterols								
Campesterol*	*7.68*	*NJ*	*10.4*	*J*	*10.1*	*J*	*22.8*	*J*
Cholestanol*	*7.99*	*J*	*4.71*	*NJ*	*3.16*	*J*	*5.03*	*J*
Cholesterol*	181	UJ	*251*		185	U	167	U
Coprostanol	**17.9**	**J**	105	U	108	U	106	U
Desmosterol	**22.2**	**J**	106	U	109	U	107	U
Epicoprostanol	**11.4**	**J**	111	U	114	U	112	U
Ergosterol	111	UJ	107	UJ	109	UJ	107	UJ
B-Sitosterol	110	UJ	**376**	**J**	**367**	**J**	**762**	**J**
B-Stigmastanol*	111	UJ	107	U	*7.62*	*NJ*	*11.9*	*NJ*
Stigmasterol*	45.5	UJ	*74.2*	*J*	*89.2*	*J*	*200*	*J*

*Parameter was detected in QA blank. Parameter was considered non-detect in report.

Bold: Analyte was detected.

U: The analyte was not detected at or above the reported sample quantitation limit.

UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.

J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-8. Quality Assurance Data for Pharmaceuticals and Personal Care Products (ng/L), May 2011

Analyte	LOTTEFF		LOTTEFF Duplicate		LOTTEFF Mean		LOTTEFF RPD (%)		Transfer Blank-LOTT		Lab Blank Spring	
	May 2011											
List 1 - Acid Extraction in Positive Ionization												
Acetaminophen	30.1	UJ	35.9	UJ	--		--		27.6	UJ	30	UJ
Azithromycin	**14**	**J**	**14.5**	**J**	14.25	J	3.5		2.76	UJ	3	UJ
Caffeine	30.1	UJ	35.9	UJ	--		--		27.6	UJ	30	UJ
Carbadox	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ
Carbamazepine	**493**	**J**	**461**	**J**	477	J	6.7		2.76	UJ	3	UJ
Cefotaxime	183	UJ	206	UJ	--		--		110	UJ	120	UJ
Ciprofloxacin	37.2	UJ	50.3	UJ	--		--		11	UJ	14	UJ
Clarithromycin	**8.75**	**J**	**5.58**	**J**	7.16	J	44		2.76	UJ	3	UJ
Clinafloxacin	114	UJ	107	UJ	--		--		44.5	UJ	53.7	UJ
Cloxacillin	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Dehydronifedipine	**15.8**	**J**	**15.7**	**J**	15.75	J	0.6		3.68	UJ	4	UJ
Digoxigenin	153	UJ	119	UJ	--		--		11	UJ	13.7	UJ
Digoxin	12	UJ	14.3	UJ	--		--		11	UJ	12	UJ
Diltiazem	0.601	UJ	0.717	UJ	--		--		0.552	UJ	0.6	UJ
1,7-Dimethylxanthine	120	UJ	143	UJ	--		--		110	UJ	120	UJ
Diphenhydramine	**7.93**	**J**	**7.57**	**J**	7.75	J	4.6		1.1	UJ	1.2	UJ
Enrofloxacin	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Erythromycin-H20	**6.51**	**J**	**5.63**	**J**	6.07	J	14		1.84	UJ	**1.24**	**J**
Flumequine	5.45	UJ	5	UJ	--		--		2.76	UJ	3	UJ
Fluoxetine	**33.2**	**J**	**29.2**	**J**	31.2	J	13		2.76	UJ	3	UJ
Lincomycin	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Lomefloxacin	9.22	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Miconazole	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ
Norfloxacin	96.3	UJ	72.9	UJ	--		--		27.6	UJ	30	UJ
Norgestimate	20	UJ	23.9	UJ	--		--		18.4	UJ	20	UJ
Ofloxacin	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ
Ormetoprim	1.2	UJ	1.43	UJ	--		--		1.1	UJ	1.2	UJ
Oxacillin	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Oxolinic acid	3.68	UJ	1.67	UJ	--		--		1.1	UJ	1.2	UJ
Penicillin G	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
Penicillin V	20	UJ	23.9	UJ	--		--		18.4	UJ	20	UJ
Roxithromycin	0.601	UJ	0.717	UJ	--		--		0.552	UJ	0.6	UJ
Sarafloxacin	30.1	UJ	35.9	UJ	--		--		27.6	UJ	30	UJ
Sulfachloropyridazine	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ
Sulfadiazine	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ

Analyte	LOTTEFF		LOTTEFF Duplicate		LOTTEFF Mean		LOTTEFF RPD (%)		Transfer Blank-LOTT		Lab Blank Spring	
	May 2011											
Sulfadimethoxine	2.88	UJ	3.04	UJ	--		--		2.73	UJ	2.29	UJ
Sulfamerazine	4.13	UJ	4.78	UJ	--		--		3.68	UJ	4	UJ
Sulfamethazine	4.01	UJ	4.78	UJ	--		--		3.68	UJ	4	UJ
Sulfamethizole	4.01	UJ	4.78	UJ	--		--		3.68	UJ	4	UJ
Sulfamethoxazole	**39.3**	**J**	**39.7**	**J**	39.5	J	1.0		3.68	UJ	4	UJ
Sulfanilamide	30.1	UJ	35.9	UJ	--		--		27.6	UJ	30	UJ
Sulfathiazole	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	UJ
Thiabendazole*	*30.3*	*REJ*	*33.2*	*REJ*					2.76	UJ	3	UJ
Trimethoprim	**4.06**	**J**	**4.06**	**J**	4.06	J	0.0		2.76	UJ	3	UJ
Tylosin	12	UJ	14.3	UJ	--		--		11	UJ	12	UJ
Virginiamycin	6.01	UJ	7.17	UJ	--		--		5.52	UJ	6	UJ
List 2 – Tetracyclines in Positive Ionization												
Anhydrochlortetracycline	34.5	UJ	30.4	UJ	--		--		27.2	U	30	U
Anhydrotetracycline	31.3	U	30.3	U	--		--		27.2	U	30	U
Chlortetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
Demeclocycline	31.3	U	30.3	U	--		--		27.2	U	30	U
Doxycycline	12.5	U	12.1	U	--		--		10.9	U	12	U
4-Epianhydrochlortetracycline	125	U	121	U	--		--		109	U	120	U
4-Epianhydrotetracycline	31.3	U	30.3	U	--		--		27.2	U	30	U
4-Epichlortetracycline	31.3	U	30.3	U	--		--		27.2	U	30	U
4-Epioxytetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
4-Epitetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
Isochlortetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
Minocycline	158	UJ	162	UJ	--		--		121	UJ	131	UJ
Oxytetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
Tetracycline	12.5	U	12.1	U	--		--		10.9	U	12	U
List 3 - Acid Extraction in Negative Ionization												
Bisphenol A	2080	U	2070	U	--		--		1810	U	2000	U
Furosemide	83.4	U	80.9	U	--		--		72.6	U	80	U
Gemfibrozil	**86.8**		**93.9**		90.4		7.9		2.72	U	3	U
Glipizide	12.5	U	12.1	U	--		--		10.9	U	12	U
Glyburide	**7.17**		**8.32**		7.75		15		5.44	U	6	U
Hydrochlorothiazide	**190**	**J**	**258**	**J**	224	J	30		36.3	UJ	40	UJ
2-Hydroxy-ibuprofen	303	UJ	162	U	--		--		145	U	160	U
Ibuprofen	**140**		**111**		126		23		27.2	U	30	U
Naproxen	**10.6**		**10.6**		10.6		0.0		5.44	U	6	U
Triclocarban	**15.9**		**16.7**		16.3		4.9		5.44	U	6	U
Triclosan	129	U	125	U	--		--		112	U	124	U

Analyte	LOTTEFF		LOTTEFF Duplicate		LOTTEFF Mean		LOTTEFF RPD (%)		Transfer Blank-LOTT		Lab Blank Spring	
	May 2011											
Warfarin	3.13	U	3.03	U	--		--		2.72	U	3	U
List 4 - Basic Extraction in Positive Ionization												
Albuterol	0.589	U	0.606	U	--		--		0.512	U	0.6	U
Amphetamine	6.92	U	7.87	U	--		--		5.33	U	**2.68**	**NJ**
Atenolol	**849**		**824**		837		3		1.02	U	1.2	U
Atorvastatin	2.95	U	3.03	U	--		--		2.56	U	3	U
Cimetidine	1.53	UJ	1.46	UJ	--		--		1.02	U	1.2	U
Clonidine	2.95	U	3.03	U	--		--		2.56	U	3	U
Codeine	5.89	U	6.06	U	--		--		5.12	U	6	U
Cotinine	**26.8**		**26**		26.4		3		2.56	U	3	U
Enalapril	0.589	U	0.606	U	--		--		0.512	U	0.6	U
Hydrocodone	2.95	U	3.03	U	--		--		2.56	U	3	U
Metformin	**1120**		**1060**		1090		5.5		5.12	U	6	U
Oxycodone	**12.7**		**13.8**		13.25		8.3		1.65	UJ	1.59	UJ
Ranitidine	1.18	U	1.21	U	--		--		1.02	U	1.2	U
Triamterene	**26.1**	**J**	**29.8**	**J**	27.9	J	13		0.877	UJ	0.959	UJ
List 5 - Acid Extraction in Positive Ionization												
Alprazolam	**4.42**	**J**	**4.69**	**J**	4.56	J	5.9		0.552	UJ	0.6	U
Amitriptyline	**1.09**	**J**	**1.21**	**J**	1.15	J	10		0.552	UJ	0.6	U
Amlodipine	3.01	UJ	3.59	UJ	--		--		2.76	UJ	3	U
Benzoylecgonine	**12.5**	**J**	**13**	**J**	12.75	J	3.9		0.552	UJ	0.6	U
Benztropine	0.601	UJ	0.717	UJ	--		--		0.552	UJ	0.6	U
Betamethasone	**5.06**	**J**	**5.06**	**J**	5.06	J	0.0		2.76	UJ	3	U
Cocaine	**0.926**	**J**	**0.681**	**J**	0.8	J	31		0.276	UJ	0.3	U
DEET	31.2	UJ	26.1	UJ	--		--		5.1	UJ	**25.7**	**J**
Desmethyldiltiazem	**0.67**	**J**	**0.456**	**J**	0.56	J	38		0.276	UJ	0.3	U
Diazepam	**1.75**	**J**	**1.39**	**J**	1.57	J	23		0.552	UJ	0.6	U
Fluocinonide	12	UJ	14.3	UJ	--		--		11	UJ	12	U
Fluticasone propionate	4.01	UJ	4.78	UJ	--		--		3.68	UJ	4	U
Hydrocortisone	120	UJ	143	UJ	--		--		110	UJ	120	U
10-hydroxy-amitriptyline	**0.328**	**J**	0.438	UJ	--		--		0.276	UJ	0.33	UJ
Meprobamate	**466**	**J**	**510**	**J**	488	J	9		7.36	UJ	8	U
Methylprednisolone	26.1	UJ	13.2	UJ	--		--		7.36	UJ	**47.6**	
Metoprolol	**272**	**J**	**276**	**J**	274	J	1.5		2.76	UJ	3	U
Norfluoxetine	**3.43**	**J**	**3.99**	**J**	3.71	J	15		2.76	UJ	3	U
Norverapamil	**16.4**	**J**	**15.5**	**J**	15.9	J	5.6		0.276	UJ	0.3	U
Paroxetine	8.02	UJ	9.57	UJ	--		--		7.36	UJ	8	U
Prednisolone	12	UJ	14.3	UJ	--		--		11	UJ	12	U

Analyte	LOTTEFF		LOTTEFF Duplicate		LOTTEFF Mean		LOTTEFF RPD (%)		Transfer Blank-LOTT		Lab Blank Spring	
	May 2011											
Prednisone	40.1	UJ	47.8	UJ	--		--		36.8	UJ	40	U
Promethazine	0.802	UJ	0.957	UJ	--		--		0.736	UJ	0.8	U
Propoxyphene	**0.689**	**J**	**0.882**	**J**	0.79	J	24		0.552	UJ	0.6	U
Propranolol	**9.96**	**J**	**12.5**	**J**	11.23	J	23		3.68	UJ	4	U
Sertraline	**3.07**	**J**	**2.78**	**J**	2.93	J	9.9		0.736	UJ	0.8	U
Simvastatin	40.1	UJ	47.8	UJ	--		--		36.8	UJ	40	U
Theophylline	**240**	**J**	**229**	**J**	234.5	J	4.7		110	UJ	120	U
Trenbolone	8.02	UJ	9.57	UJ	--		--		7.36	UJ	8	U
Trenbolone acetate	0.601	UJ	0.717	UJ	--		--		0.552	UJ	0.6	U
Valsartan	**280**	**J**	**284**	**J**	282	J	1.4		7.36	UJ	8	U
Verapamil	**0.458**	**J**	**0.625**	**J**	0.542	J	31		0.276	UJ	0.3	U

*Parameter was detected in QA blank. Parameter value was rejected.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been “tentatively identified,” and the associated numerical value represents its approximate concentration.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-9. Quality Assurance Data for Hormones and Sterols (ng/L), May 2011

Analyte	LOTTEFF		LOTTEFF Duplicate		LOTTEFF Mean		LOTTEFF RPD (%)		Transfer Blank-LOTT		Lab Blank Spring	
	May 2011											
Hormones												
17*B*-Estradiol*	88	UJ	88.6	UJ	--		--		**0.335**	**NJ**	**0.401**	**NJ**
17*a*-Estradiol	88.7	UJ	89.3	UJ	--		--		9.18	UJ	**0.095**	**J**
17*a*-Ethinyl Estradiol*	88.3	UJ	88.9	UJ	--		--		**0.423**	**NJ**	**0.395**	**NJ**
B-Estradiol-3-Benzoate	88.9	UJ	89.5	UJ	--		--		9.2	UJ	8.02	U
17*a*-Dihydroequilin	**14**	**J**	**12.9**	**J**	13.5	J	8.2		9.04	UJ	7.87	U
Androstenedione	220	UJ	222	UJ	--		--		22.8	UJ	19.9	U
Androsterone	88.5	UJ	89.1	UJ	--		--		9.17	UJ	7.98	U
Desogestrel	88.3	UJ	88.9	UJ	--		--		9.15	UJ	7.97	U
Equilenin	91.4	UJ	92.1	UJ	--		--		**0.275**	**NJ**	**0.302**	**NJ**
Equilin	87.2	UJ	87.8	UJ	--		--		9.03	UJ	7.86	U
Estriol	88.9	UJ	89.5	UJ	--		--		9.2	UJ	8.02	U
Estrone	88.2	UJ	88.8	UJ	--		--		9.14	UJ	7.96	U
Mestranol*	177	UJ	178	UJ	--		--		**0.686**	**NJ**	**0.584**	**NJ**
Norethindrone	177	UJ	178	UJ	--		--		9.15	UJ	7.97	U
Norgestrel	88.7	UJ	89.3	UJ	--		--		9.18	UJ	8	U
Progesterone	220	UJ	222	UJ	--		--		22.8	UJ	19.9	U
Testosterone	88.3	UJ	88.9	UJ	--		--		9.15	UJ	7.97	U
Sterols												
Campesterol*	443	UJ	446	UJ	--		--		**53.8**		**7.19**	**J**
Cholestanol*	440	UJ	443	UJ	--		--		45.5	UJ	**2.33**	**J**
Cholesterol*	1100	UJ	1110	UJ	--		--		324	UJ	**169**	
Coprostanol	1080	UJ	1090	UJ	--		--		112	UJ	**2.4**	**J**
Desmosterol	1090	UJ	1100	UJ	--		--		113	UJ	98.7	U
Epicoprostanol	1150	UJ	1160	UJ	--		--		119	UJ	**1.22**	**J**
Ergosterol	1100	UJ	1110	UJ	--		--		114	UJ	99	UJ
B-Sitosterol	1090	UJ	1100	UJ	--		--		1800	U	**212**	
B-Stigmastanol*	1100	UJ	1100	UJ	--		--		**17.7**	**J**	**3.06**	**J**
Stigmasterol*	450	UJ	453	UJ	--		--		**538**		**48.2**	

*Parameter was detected in QA blank. Parameter was considered non-detect in report.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ: The analysis indicates the presence of an analyte that has been “tentatively identified”, and the associated numerical value represents its approximate concentration.

Table E-10. Quality Assurance Data for Pharmaceuticals and Personal Care Products (ng/L), October 2011

Analyte	LOTTMW-8		LOTTMW-8 Duplicate		LOTTMW-8 Mean		LOTTMW-8 RPD (%)		Equipment Blank		Lab Blank Fall	
	October 2011											
List 1 - Acid Extraction in Positive Ionization												
Acetaminophen	89.9	U	99.1	U	--		--		28.6	U	30	U
Azithromycin	8.99	U	9.91	U	--		--		2.86	U	3	U
Caffeine	89.9	U	99.1	U	--		--		28.6	U	30	U
Carbadox	8.99	U	9.91	U	--		--		2.86	U	3	U
Carbamazepine	**186**	**J**	**184**		185	J	1.1		2.86	U	3	U
Cefotaxime	7.39	UJ	8.15	UJ	--		--		7.05	UJ	7.4	UJ
Ciprofloxacin	36	U	39.6	U	--		--		11.4	U	12	U
Clarithromycin	8.99	U	9.91	U	--		--		2.86	U	3	U
Clinafloxacin	36	U	44.9	UJ	--		--		40.2	UJ	40	U
Cloxacillin	18	U	19.8	U	--		--		5.72	U	6	U
Dehydronifedipine	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Digoxigenin	44.6	UJ	40.4	UJ	--		--		15	UJ	22.1	UJ
Digoxin	36	U	39.6	U	--		--		11.4	U	14	UJ
Diltiazem	1.8	U	1.98	U	--		--		0.607	U	**0.753**	
1,7-Dimethylxanthine	360	U	396	U	--		--		114	U	120	U
Diphenhydramine	3.6	UJ	3.96	UJ	--		--		1.14	UJ	1.2	UJ
Enrofloxacin	18	U	19.8	U	--		--		5.72	U	6	U
Erythromycin-H20	1.8	U	1.98	U	--		--		0.572	U	0.6	U
Flumequine	30	U	33	U	--		--		9.53	U	10	U
Fluoxetine	8.99	U	9.91	U	--		--		2.86	U	3	U
Lincomycin	18	U	19.8	U	--		--		5.72	U	6	U
Lomefloxacin	18	U	19.8	U	--		--		5.72	U	6	U
Miconazole	8.99	U	9.91	U	--		--		2.86	U	3	U
Norfloxacin	89.9	U	99.1	U	--		--		28.6	U	30	U
Norgestimate	18	U	19.8	U	--		--		5.72	U	6	U
Ofloxacin	8.99	U	9.91	U	--		--		2.86	U	3	U
Ormetoprim	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Oxacillin	18	U	19.8	U	--		--		5.72	U	6	U
Oxolinic acid	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Penicillin G	18	U	19.8	U	--		--		19.1	U	20	U
Penicillin V	18	U	19.8	U	--		--		5.72	U	6	U
Roxithromycin	1.8	U	1.98	U	--		--		0.572	U	0.6	U
Sarafloxacin	89.9	U	99.1	U	--		--		28.6	U	30	U
Sulfachloropyridazine	8.99	U	9.91	U	--		--		2.86	U	3	UJ
Sulfadiazine	8.99	U	9.91	U	--		--		2.86	U	3	U

Analyte	LOTTMW-8		LOTTMW-8 Duplicate		LOTTMW-8 Mean		LOTTMW-8 RPD (%)		Equipment Blank		Lab Blank Fall	
	October 2011											
Sulfadimethoxine	2.32	UJ	1.98	U	--		--		0.572	U	0.6	U
Sulfamerazine	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Sulfamethazine	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Sulfamethizole	3.6	U	3.96	U	--		--		1.14	U	1.2	U
Sulfamethoxazole	**422**		**422**		422		0.0		1.14	U	1.2	U
Sulfanilamide	**105**		**119**		112		12		28.6	U	30	U
Sulfathiazole	8.99	U	9.91	U	--		--		2.86	U	3	U
Thiabendazole*	8.99	U	9.91	U	--		--		2.86	U	3	U
Trimethoprim	8.99	U	9.91	U	--		--		2.86	U	3	U
Tylosin	36	U	39.6	U	--		--		11.4	U	12	U
Virginiamycin	18	U	19.8	U	--		--		5.72	U	6	U
List 2 – Tetracyclines in Positive Ionization												
Anhydrochlortetracycline	99.9	U	110	U	--		--		95.3	U	100	U
Anhydrotetracycline	99.9	U	110	U	--		--		95.3	U	100	U
Chlortetracycline	12	U	13.2	U	--		--		11.4	U	12	U
Demeclocycline	30	U	33	U	--		--		28.6	U	30	U
Doxycycline	40	U	44	U	--		--		38.1	U	40	U
4-Epianhydrochlortetracycline	400	U	440	U	--		--		381	U	400	U
4-Epianhydrotetracycline	30	U	33	U	--		--		28.6	U	30	U
4-Epichlortetracycline	30	U	33	U	--		--		28.6	U	30	U
4-Epioxytetracycline	12	U	13.2	U	--		--		11.4	U	12	U
4-Epitetracycline	12	U	14.3	UJ	--		--		11.4	U	12	U
Isochlortetracycline	12	U	13.2	U	--		--		11.4	U	12	U
Minocycline	120	U	132	U	--		--		114	U	120	U
Oxytetracycline	12	U	13.2	U	--		--		11.4	U	12	U
Tetracycline	12	U	13.2	U	--		--		11.4	U	12	U
List 3 - Acid Extraction in Negative Ionization												
Bisphenol A	999	U	1100	U	--		--		953	U	1000	U
Furosemide	79.9	U	88.1	U	--		--		76.2	U	80	U
Gemfibrozil	3	U	3.3	U	--		--		2.86	U	3	U
Glipizide	12	U	13.2	U	--		--		11.4	U	12	U
Glyburide	5.99	U	6.61	U	--		--		5.72	U	6	U
Hydrochlorothiazide	40	UJ	44	UJ	--		--		38.1	UJ	40	UJ
2-Hydroxy-ibuprofen	160	U	176	U	--		--		152	U	160	U
Ibuprofen	30	U	33	U	--		--		28.6	U	30	U
Naproxen	5.99	U	6.61	U	--		--		5.72	U	6	U
Triclocarban	5.99	U	6.61	U	--		--		5.72	U	6	U
Triclosan	120	U	132	U	--		--		114	U	120	U

Analyte	LOTTMW-8		LOTTMW-8 Duplicate		LOTTMW-8 Mean		LOTTMW-8 RPD (%)		Equipment Blank		Lab Blank Fall	
	October 2011											
Warfarin	3	U	3.3	U	--		--		2.86	U	3	U
List 4 - Basic Extraction in Positive Ionization												
Albuterol	1.98	U	2.13	U	--		--		1.95	U	2	U
Amphetamine	2.98	U	3.51	UJ	--		--		2.93	U	3	U
Atenolol	2.07	UJ	3.37	UJ	--		--		2.5	UJ	1.28	UJ
Atorvastatin	2.98	U	3.2	U	--		--		2.93	U	3	U
Cimetidine	1.19	U	1.28	U	--		--		1.17	U	1.2	U
Clonidine	4.88	UJ	3.2	U	--		--		3.77	UJ	4.04	UJ
Codeine	9.47	UJ	7.38	UJ	--		--		7.63	UJ	9.03	UJ
Cotinine	2.98	U	3.2	U	--		--		2.93	U	3	U
Enalapril	0.595	U	0.64	U	--		--		0.585	U	0.6	U
Hydrocodone	2.98	UJ	3.2	UJ	--		--		2.93	UJ	3	UJ
Metformin	15.5	UJ	25.1	UJ	--		--		5.85	U	11.6	UJ
Oxycodone	3.97	U	4.27	U	--		--		3.9	U	4	U
Ranitidine	1.19	U	1.28	U	--		--		1.17	U	1.2	U
Triamterene	1.98	U	2.13	U	--		--		1.95	U	2.13	UJ
List 5 - Acid Extraction in Positive Ionization												
Alprazolam	1.05	UJ	**0.725**		--		--		0.572	U	0.6	U
Amitriptyline	0.599	U	0.743	UJ	--		--		0.572	U	0.6	U
Amlodipine	2.88	U	3.17	U	--		--		2.74	U	2.88	U
Benzoylecgonine	**1.51**		**1.55**		1.53		2.6		0.572	U	0.6	U
Benztropine	0.599	U	0.661	U	--		--		0.572	U	0.6	U
Betamethasone	9.99	U	11	U	--		--		9.53	U	10	U
Cocaine	0.3	U	0.33	U	--		--		0.286	U	0.3	U
DEET	10.9	U	**15.5**		--		--		1.62	U	**2.91**	
Desmethyldiltiazem	0.3	U	0.33	U	--		--		0.286	U	0.3	U
Diazepam	0.599	U	0.661	U	--		--		0.572	U	0.6	U
Fluocinonide	12	U	13.2	U	--		--		11.4	U	12	U
Fluticasone propionate	4	U	4.4	U	--		--		3.81	U	4	U
Hydrocortisone	120	U	132	U	--		--		114	U	120	U
10-hydroxy-amitriptyline	0.999	U	1.1	U	--		--		0.953	U	1	U
Meprobamate	**26.9**		**29.1**		28		7.9		7.62	U	8	U
Methylprednisolone	26.6	U	29.4	U	--		--		25.4	U	26.7	U
Metoprolol	3	U	4	UJ	--		--		2.86	U	3	U
Norfluoxetine	3	U	3.3	U	--		--		2.86	U	3	U
Norverapamil	0.3	U	0.33	U	--		--		0.286	U	0.3	U
Paroxetine	7.99	U	8.81	U	--		--		7.62	U	8	U
Prednisolone	16.4	UJ	14.7	UJ	--		--		11.4	U	12	U

Analyte	LOTTMW-8		LOTTMW-8 Duplicate		LOTTMW-8 Mean		LOTTMW-8 RPD (%)		Equipment Blank		Lab Blank Fall	
	October 2011											
Prednisone	44.7	UJ	68.2	UJ	--		--		40.8	UJ	44.1	UJ
Promethazine	0.799	U	0.881	U	--		--		0.762	U	0.8	U
Propoxyphene	0.599	U	0.661	U	--		--		0.572	U	0.6	U
Propranolol	4	U	4.4	U	--		--		3.81	U	4	U
Sertraline	0.799	U	0.881	U	--		--		0.762	U	0.8	U
Simvastatin	40	U	44	U	--		--		38.1	U	40	U
Theophylline	120	U	132	U	--		--		114	U	120	U
Trenbolone	9.15	UJ	9.06	UJ	--		--		7.62	U	8	U
Trenbolone acetate	0.599	U	0.661	U	--		--		0.572	U	0.6	U
Valsartan	7.99	U	8.81	U	--		--		7.62	U	8	U
Verapamil	0.3	U	0.33	U	--		--		0.286	U	0.3	U

*Parameter was detected in QA blank. Parameter value was rejected.
Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

Table E-10 (cont.). Quality Assurance Data for Pharmaceuticals and Personal Care Products (ng/L), October 2011

Analyte	QuincyEff		QuincyEff Duplicate		QuincyEff Mean		QuincyEff RPD (%)		Transfer Blank		Lab Blank Fall	
	October 2011											
List 1 - Acid Extraction in Positive Ionization												
Acetaminophen	**31.1**		**33.3**		32.2		6.8		29.2	U	30	U
Azithromycin	**79.2**		**63.5**		71.4		22		2.92	U	3	U
Caffeine	**1390**		**1030**		1210		30		29.2	U	30	U
Carbadox	2.88	U	2.87	U	--		--		2.92	U	3	U
Carbamazepine	**511**		**517**		514		1.2		2.92	U	3	U
Cefotaxime		REJ		REJ			--			REJ		REJ
Ciprofloxacin	**202**		**214**		208		5.8		11.7	U	12	U
Clarithromycin	**64.5**		**67.2**		65.9		4.1		2.92	U	3	U
Clinafloxacin	53.8	UJ	82.9	UJ	--		--		13.5	UJ	16.5	UJ
Cloxacillin	6.87	UJ	5.74	UJ	--		--		5.84	UJ	6	UJ
Dehydronifedipine	**2.59**		**2.49**		2.54		3.9		1.17	U	1.2	U
Digoxigenin	11.5	U	11.5	U	--		--		11.7	U	12	U
Digoxin	11.5	U	11.5	U	--		--		11.7	U	12	U
Diltiazem	**84.7**		**87.4**		86.1		3.1		0.584	U	0.6	U
1,7-Dimethylxanthine	**2040**		**1910**		1975		6.6		117	U	120	U
Diphenhydramine	**345**	**J**	**360**	**J**	353	J	4.3		1.17	UJ	1.2	UJ
Enrofloxacin	6.49	UJ	6.16	UJ	--		--		5.84	U	6	U
Erythromycin-H20	**20**		**16.6**		18.3		19		0.584	U	0.6	U
Flumequine	10.8	UJ	8.64	UJ	--		--		2.92	U	3.15	UJ
Fluoxetine	**7.67**		**8.87**		8.27		15		2.92	U	3	U
Lincomycin	5.78	UJ	5.74	U	--		--		5.84	U	6	U
Lomefloxacin	26.1	UJ	23.7	UJ	--		--		5.84	U	6	U
Miconazole	**3.54**		**3.24**		3.39		8.8		2.92	U	3	U
Norfloxacin	62.3	UJ	44.5	UJ	--		--		29.2	U	30	U
Norgestimate	5.77	U	5.74	U	--		--		5.84	U	6	U
Ofloxacin	**10.4**		**13.2**		11.8		24		2.92	U	3	U
Ormetoprim	1.15	U	1.15	U	--		--		1.17	U	1.2	U
Oxacillin	**14.7**		**13.9**		14.3		5.6		5.84	U	6	U
Oxolinic acid	4.81	UJ	3.83	UJ	--		--		1.17	U	1.2	U
Penicillin G	5.77	U	5.74	U	--		--		5.84	U	6	U
Penicillin V	8.99	UJ	8.95	UJ	--		--		5.84	U	6	U
Roxithromycin	0.577	U	0.574	U	--		--		0.584	U	0.6	U
Sarafloxacin	65	UJ	63.1	UJ	--		--		29.2	U	30	U
Sulfachloropyridazine	2.88	U	2.87	U	--		--		2.92	U	3	U
Sulfadiazine	**29.4**		**29.6**		29.5		0.7		2.92	U	3	U

Analyte	QuincyEff		QuincyEff Duplicate		QuincyEff Mean		QuincyEff RPD (%)		Transfer Blank		Lab Blank Fall	
	October 2011											
Sulfadimethoxine	**59.8**		**63.6**		61.7		6.2		0.584	U	0.6	U
Sulfamerazine	2.8	UJ	2.73	UJ	--		--		1.17	U	1.2	U
Sulfamethazine	6.84	UJ	10.4	UJ	--		--		1.17	U	1.22	UJ
Sulfamethizole	12.3	UJ	2.27	UJ	--		--		1.17	U	1.2	U
Sulfamethoxazole	**167**		**160**		163.5		4.3		1.17	U	1.2	U
Sulfanilamide	28.8	U	28.7	U	--		--		29.2	U	30	U
Sulfathiazole	5.7	UJ	6.58	UJ	--		--		2.92	U	3	U
Thiabendazole	**119000**	**J**	**111000**	**J**	115000	J	7		**59**		3	U
Trimethoprim	**174**		**169**		171.5		2.9		2.92	U	3	U
Tylosin	11.5	UJ	11.5	UJ	--		--		11.7	UJ	12	UJ
Virginiamycin	48.4	UJ	36.4	UJ	--		--		5.84	U	6	U
List 2 – Tetracyclines in Positive Ionization												
Anhydrochlortetracycline	128	UJ	99	UJ	--		--		29.2	U	30	U
Anhydrotetracycline	43.2	UJ	40.1	UJ	--		--		29.2	U	30	U
Chlortetracycline	38.5	U	38.3	U	--		--		38.9	U	40	U
Demeclocycline	96.1	U	95.7	U	--		--		97.3	U	100	U
Doxycycline	**42.1**		**40.6**		41.4		3.6		11.7	U	12	U
4-Epianhydrochlortetracycline	281	UJ	213	UJ	--		--		117	U	120	U
4-Epianhydrotetracycline	56.9	UJ	51.3	UJ	--		--		29.2	U	30	U
4-Epichlortetracycline	40.6	UJ	34	UJ	--		--		29.2	U	30	U
4-Epioxytetracycline	43.2	UJ	42.5	UJ	--		--		11.7	U	12	U
4-Epitetracycline	43.9	UJ	**33.1**		--		--		11.7	U	12	U
Isochlortetracycline	11.5	U	11.5	U	--		--		11.7	U	12	U
Minocycline	115	U	115	U	--		--		117	U	120	U
Oxytetracycline	11.5	U	11.5	U	--		--		11.7	U	12	U
Tetracycline	**36.3**		**30.9**		33.6		16		11.7	U	12	U
List 3 - Acid Extraction in Negative Ionization												
Bisphenol A	961	U	957	U	--		--		973	U	1000	U
Furosemide	**485**		**402**		444		19		77.8	U	80	U
Gemfibrozil	**542**		**543**		542.5		0.2		2.92	U	3	U
Glipizide	11.5	U	11.5	U	--		--		11.7	U	12	U
Glyburide	5.77	U	5.74	U	--		--		5.84	U	6	U
Hydrochlorothiazide	**98.3**		**69.1**		83.7		35		38.9	U	40	U
2-Hydroxy-ibuprofen	**1310**		**1030**		1170		24		156	U	160	U
Ibuprofen	**172**		**169**		170.5		1.8		29.2	U	30	U
Naproxen	**262**		**244**		253		7.1		5.84	U	6	U
Triclocarban	**40.8**		**38.3**		39.6		6.3		5.84	U	6	U
Triclosan	**158**		**179**		168.5		12		117	U	120	U

Analyte	QuincyEff		QuincyEff Duplicate		QuincyEff Mean		QuincyEff RPD (%)		Transfer Blank		Lab Blank Fall	
	October 2011											
Warfarin	2.88	U	2.87	U	--		--		2.92	U	3	U
List 4 - Basic Extraction in Positive Ionization												
Albuterol	**10.5**	**J**	**16.2**	**J**	13.4	J	42		0.597	U	0.76	UJ
Amphetamine	11.4	UJ	14.5	UJ	--		--		5.47	UJ	7.01	UJ
Atenolol	**542**		**462**		502		16		1.19	U	1.39	U
Atorvastatin	**10.8**		**9.9**		10.4		8.7		2.99	U	3.48	U
Cimetidine	**90.5**		**89.5**		90		1.1		1.29	UJ	5.34	UJ
Clonidine	2.98	U	3	U	--		--		2.99	U	3.48	U
Codeine	**30.4**		**51.9**		41.2		52		9.05	UJ	6.96	U
Cotinine	**215**		**209**		212		2.8		2.99	U	3.48	U
Enalapril	**2.66**		**1.48**		2.07		57		0.597	U	0.696	U
Hydrocodone	**22.1**		**22.5**		22.3		1.8		2.99	U	3.48	U
Metformin	**1030**		**1030**		1030		0.0		7.18	UJ	8.07	UJ
Oxycodone	**37.4**		**44.6**		41		18		1.19	U	1.39	U
Ranitidine	**336**		**336**		336		0.0		1.61	UJ	1.39	U
Triamterene	**64.5**		**59.8**		62.2		7.6		1.99	U	2.32	U
List 5 - Acid Extraction in Positive Ionization												
Alprazolam	0.577	U	0.574	U	--		--		0.584	U	0.6	U
Amitriptyline	**29.2**		**35.2**		32.2		19		0.584	U	0.6	U
Amlodipine	**5.48**		**5.1**		5.29		7.2		2.8	U	2.88	U
Benzoylecgonine	**16.8**		**15.8**		16.3		6.1		0.584	U	0.6	U
Benztropine	0.577	U	0.574	U	--		--		0.584	U	0.6	U
Betamethasone	2.88	U	2.87	U	--		--		2.92	U	3	U
Cocaine	**0.484**		**0.541**		0.513		11		0.292	U	0.3	U
DEET	**34.7**		**36.2**		35.5		4.2		2.42	U	**1.98**	
Desmethyldiltiazem	**14.4**		**14.3**		14.35		0.7		0.292	U	0.3	U
Diazepam	0.577	U	0.574	U	--		--		0.584	U	0.6	U
Fluocinonide	11.5	U	11.5	U	--		--		11.7	U	12	U
Fluticasone propionate	5.57	UJ	8.27	UJ	--		--		3.89	U	4	U
Hydrocortisone	153	UJ	178	UJ	--		--		117	U	120	U
10-hydroxy-amitriptyline	**16.6**		**17.1**		16.9		3		0.292	U	0.3	U
Meprobamate	**33.4**		**28.4**		30.9		16		7.78	U	8	U
Methylprednisolone	25.6	U	25.5	U	--		--		25.9	U	26.7	U
Metoprolol	**339**		**349**		344		2.9		2.92	U	3	U
Norfluoxetine	2.88	U	2.87	U	--		--		2.92	U	3	U
Norverapamil	**0.484**		**0.68**		0.582		34		0.292	U	0.3	U
Paroxetine	**8.53**		**10.6**		9.6		22		7.78	U	8	U
Prednisolone	11.5	U	11.5	U	--		--		11.7	U	12	U
Prednisone	199	UJ	220	UJ	--		--		38.9	U	40	U

Analyte	QuincyEff		QuincyEff Duplicate		QuincyEff Mean		QuincyEff RPD (%)		Transfer Blank		Lab Blank Fall	
	October 2011											
Promethazine	0.769	U	0.765	U	--		--		0.778	U	0.8	U
Propoxyphene	0.577	U	0.574	U	--		--		0.584	U	0.6	U
Propranolol	**88.7**		**86**		87.4		3.1		3.89	U	4	U
Sertraline	**18.4**		**17.6**		18		4.4		0.778	U	0.8	U
Simvastatin	38.5	U	38.3	U	--		--		38.9	U	40	U
Theophylline	**1570**		**1700**		1635		8		117	U	120	U
Trenbolone	7.69	U	7.65	U	--		--		7.78	U	8	U
Trenbolone acetate	0.903	UJ	0.83	UJ	--		--		0.584	U	0.6	U
Valsartan	**400**		**369**		385		8		7.78	U	8	U
Verapamil	**3.47**		**2.95**		3.21		16		0.292	U	0.3	U

Bold: Analyte was detected.
U: The analyte was not detected at or above the reported sample quantitation limit.
UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.
J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
REJ: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Table E-11. Quality Assurance Data for Hormones and Sterols (ng/L), October 2011

Analyte	LOTTMW-8		LOTTMW-8 Duplicate		LOTTMW-8 Mean		LOTTMW-8 RPD (%)		Equipment Blank		Lab Blank Fall	
	October 2011											
Hormones												
17*B*-Estradiol*	8.51	UJ	8.72	UJ	--		--		8.77	U	**1.23**	**NJ**
17*a*-Estradiol	8.58	U	8.79	U	--		--		8.84	U	8	U
17*a*-Ethinyl Estradiol*	8.55	UJ	8.75	UJ	--		--		8.81	U	**1.3**	**NJ**
B-Estradiol-3-Benzoate	8.6	U	8.8	U	--		--		8.86	U	8.02	U
17*a*-Dihydroequilin	8.45	U	8.65	U	--		--		**0.253**	**NJ**	7.87	U
Androstenedione	21.3	U	21.8	U	--		--		22	U	**12.4**	**NJ**
Androsterone	8.57	U	8.77	U	--		--		8.82	U	**0.151**	**J**
Desogestrel	8.55	U	8.75	U	--		--		8.81	U	7.97	U
Equilenin	8.85	UJ	9.06	U	--		--		9.12	U	**1.02**	**NJ**
Equilin	8.44	U	8.64	U	--		--		8.69	U	7.86	U
Estriol	8.6	U	8.8	U	--		--		8.86	U	8.02	U
Estrone	8.54	U	8.74	U	--		--		8.8	U	7.96	U
Mestranol*	8.57	U	8.78	UJ	--		--		8.83	U	**0.896**	**NJ**
Norethindrone	8.55	U	8.75	U	--		--		8.81	U	7.97	U
Norgestrel	8.58	UJ	8.79	UJ	--		--		8.84	U	**0.822**	**NJ**
Progesterone	21.3	U	21.8	U	--		--		22	U	19.9	U
Testosterone	8.55	U	8.75	U	--		--		8.81	U	7.97	U
Sterols												
Campesterol*	*27*	*J*	*5.23*	*J*	*16.12*	*J*	135		**6.78**	**NJ**	39.9	U
Cholestanol*	*2.18*	*J*	*2.06*	*J*	*2.12*	*J*	5.7		**11.8**	**J**	**0.89**	**NJ**
Cholesterol*	108	U	109	UJ	--		--		**777**		**49.3**	**J**
Coprostanol	105	U	107	U	--		--		108	U	97.5	U
Desmosterol	106	U	108	U	--		--		109	U	98.7	U
Epicoprostanol	111	U	114	U	--		--		114	U	104	U
Ergosterol	106	UJ	109	UJ	--		--		109	UJ	99	UJ
B-Sitosterol	**1240**	**J**	161	UJ	--		--		109	U	**41.2**	**J**
B-Stigmastanol*	*12.3*	*J*	109	UJ	--		--		109	U	99	UJ
Stigmasterol*	*270*	*J*	*42*	*J*	*156*	*J*	146		44.9	U	**8.36**	**J**

*Parameter was detected in QA blank. Parameter was considered non-detect in report.

Bold: Analyte was detected.

U: The analyte was not detected at or above the reported sample quantitation limit.

UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.

J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ: The analysis indicates the presence of an analyte that has been “tentatively identified”, and the associated numerical value represents its approximate concentration.

Table E-11 (cont.). Quality Assurance Data for Hormones and Sterols (ng/L), October 2011

Analyte	QuincyEff		QuincyEff Duplicate		QuincyEff Mean		QuincyEff RPD (%)		Transfer Blank		Lab Blank Fall	
	October 2011											
Hormones												
17*B*-Estradiol*	181	U	288	U	--		--		9.31	UJ	7.94	UJ
17*a*-Estradiol	182	U	291	U	--		--		9.38	UJ	8	UJ
17*a*-Ethinyl Estradiol*	182	U	289	U	--		--		9.34	UJ	7.97	UJ
B-Estradiol-3-Benzoate	183	U	291	U	--		--		9.4	UJ	8.02	UJ
17*a*-Dihydroequilin	179	U	286	U	--		--		9.23	UJ	7.87	UJ
Androstenedione	453	U	722	U	--		--		23.3	U	19.9	U
Androsterone	182	U	290	U	--		--		9.36	U	**0.118**	**NJ**
Desogestrel	182	U	289	U	--		--		9.34	U	7.97	U
Equilenin	188	U	300	U	--		--		9.67	UJ	8.25	UJ
Equilin	179	U	286	U	--		--		9.22	UJ	7.86	UJ
Estriol	183	U	291	U	--		--		9.4	U	8.02	U
Estrone	181	U	289	U	--		--		9.33	UJ	7.96	UJ
Mestranol*	182	U	290	U	--		--		9.37	U	7.99	U
Norethindrone	182	U	289	U	--		--		9.34	U	7.97	U
Norgestrel	182	U	291	U	--		--		9.38	U	8	U
Progesterone	453	U	722	U	--		--		23.3	U	19.9	U
Testosterone	182	U	289	U	--		--		9.34	U	7.97	U
Sterols												
Campesterol*	*859*	*NJ*	*849*	*J*					46.8	U	39.9	U
Cholestanol*	*557*	*J*	*587*	*J*					46.5	U	**1.86**	**J**
Cholesterol*	*1940*	*J*	*2000*	*J*					232	U	**139**	
Coprostanol	**2910**		**3080**	**J**	2995	J	5.7		114	U	97.5	U
Desmosterol	2250	U	3580	U	--		--		116	U	98.7	U
Epicoprostanol	**128**	**J**	**146**	**J**	137	J	13		121	U	104	U
Ergosterol	2260	UJ	3600	UJ	--		--		116	UJ	99	UJ
B-Sitosterol	**14800**	**J**	**8810**	**J**	11805	J	51		116	UJ	**51.8**	**J**
B-Stigmastanol*	*444*	*NJ*	3600	U					116	UJ	99	UJ
Stigmasterol*	*51400*	*J*	*50400*	*J*					47.6	U	**15.1**	**J**

*Parameter was detected in QA blank. Parameter was considered non-detect in report.

Bold: Analyte was detected.

U: The analyte was not detected at or above the reported sample quantitation limit.

UJ: The analyte was not detected above the reported *estimated* sample quantitation limit.

J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ: The analysis indicates the presence of an analyte that has been "tentatively identified", and the associated numerical value represents its approximate concentration.

Appendix F. Chemicals Not Detected in Reclaimed Water or Groundwater during Present Study

Method 1694 for PPCPs: 50 of 118 analytes

Anhydrochlortetracycline
Anhydrotetracycline
Benztropine
Bisphenol A
Carbadox
Cefotaxime
Chlortetracycline
Clinafloxacin
Clonidine
Cloxacillin
Demeclocycline
Digoxigenin
Digoxin
Enrofloxacin
4-Epianhydrochlortetracycline
4-Epianhydrotetracycline
4-Epichlortetracycline
4-Epioxytetracycline
Flumequine
Fluocinonide
Fluticasone propionate
Glipizide
Hydrocortisone
Isochlortetracycline
Lincomycin
Lomefloxacin
Methylprednisolone
Minocycline
Norfloxacin
Norgestimate
Ormetoprim
Oxolinic acid
Oxytetracycline
Penicillin G
Penicillin V
Prednisolone
Prednisone
Promethazine
Roxithromycin
Sarafloxacin
Simvastatin
Sulfachloropyridazine
Sulfamerazine
Sulfamethizole
Sulfathiazole
Trenbolone
Trenbolone acetate
Tylosin
Virginiamycin
Warfarin

Method 1698 for Hormones and Sterols: 19 of 27 analytes

Hormones

17 *B*-Estradiol
17*a*-Estradiol
17*a*-Ethinyl Estradiol
B-Estradiol-3-Benzoate
Androstenedione
Androsterone
Desogestrel
Equilenin
Equilin
Estriol
Estrone
Mestranol
Norethindrone
Norgestrel

Sterols

Campesterol*
Cholestanol*
Cholesterol*
B-Stigmastanol*
Stigmasterol*

*Detections rejected due to field blank contamination

Appendix G. Glossary, Acronyms, and Abbreviations

Glossary

Analgesic: A class of drugs used to relieve pain.

Anti-arrhythmic: A class of drugs used to suppress abnormal rhythms of the heart

Antihistamine: A class of drugs used to clear congestion and block allergic reactions

Antihyperlipidemic: A class of drugs used to reduce lipid levels in the blood.

Antihypertensive: A class of drugs used to lower blood pressure

Antipyretic: A class of drugs used to reduce fever

Diuretic: A class of drugs that promotes the formation of urine.

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.

Median: A measure of central tendency where one-half (50%) of the observations lie above that value and one-half lie below that value.

Parameter: Water quality constituent being measured (analyte). A physical, chemical, or biological property whose values determine environmental characteristics or behavior.

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 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.

Total suspended solids (TSS): The suspended particulate matter in a water sample as retained by a filter.

Turbidity: A measure of the amount of suspended silt or organic matter in water. High levels of turbidity can have a negative impact on aquatic life.

90th percentile: A statistical number obtained from a distribution of a data set, above which 10% of the data exists and below which 90% of the data exists.

Acronyms and Abbreviations

AXYS	AXYS Analytical Services LTD
DEET	N,N-diethyl-meta-toluamide, an active ingredient in many insect repellants
Ecology	Washington State Department of Ecology

EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
GIS	Geographic Information System software
LOTT	Lacey, Olympia, Tumwater, and Thurston County Clean Water Alliance
MEL	Manchester Environmental Laboratory
NPDES	National Pollutant Discharge Elimination System permitting program
PPCPs	Pharmaceuticals and personal care products
RPD	Relative percent difference
RWTF	Reclaimed Water Treatment Facility
SOP	Standard operating procedures
SWRCB	State Water Resources Control Board
TSS	Total suspended solids
USGS	U.S. Geological Survey
WAC	Washington Administrative Code
WRIA	Water Resource Inventory Area
WWTP	Wastewater treatment plant

Units of Measurement

°C	degrees centigrade
L	liters
mg	milligrams
MGD	million gallons per day
mg/L	milligrams per liter (parts per million)
mg/L/hr	milligrams per liter per hour
mL	milliliters
ng/L	nanograms per liter (parts per trillion)
NTU	nephelometric turbidity units
umhos/cm	micromhos per centimeter